بیست و هفتمین سمینار شیمی تجزیه انجمن شیمی ایران

۱ تا ۳ شهریور ۱۴۰۱

23 _ 25 August 2022

دانشگاه زنجان

دانشکده علوم _ گروه شیمی

محورهای سمینار

- روشهای جداسازی
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27th Iranian Seminar of Analytical Chemistry

23-25 August 2022

By:

University of Zanjan, Zanjan, Iran

In collaboration with:

Iranian Chemical Society

In The Name Of Allah, The Most Gracious, The Merciful

This book has been designed and edited by:
Elham Mohammadi (Ph.D. Student of Organic Chemistry)
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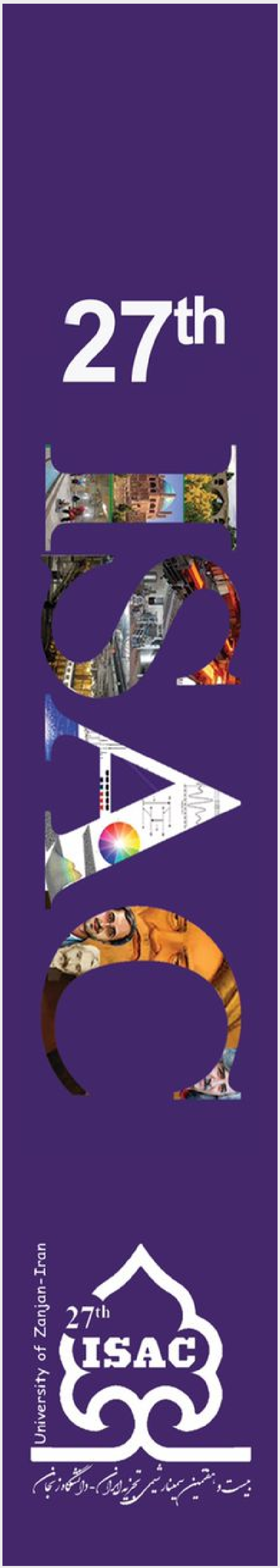

Editorial Note:

Dear Friends and Colleagues
It was our great pleasure to organize the 27th Iranian Seminar of Analytical Chemistry (27th ISAC) on Aug. 23-25, 2022, at the Department of Chemistry of the University of Zanjan (Zanjan, Iran). This annual event has been organized under scientific supervision of the Iranian Chemical Society. About 300 academic, industrial researchers and students, from 26 provinces of Iran, with about 350 papers were participated in this scientific event. The participants presented their last scientific findings in the field of the topics considered for 27th ISAC, in two oral and poster sections. Forty-six oral presentations were presented by the Iranian and international scientists, included 6 plenary and keynote talks. Besides, a panel concerned the basic sciences challenges in Iran, as 2022 is nominated as the International Year of Basic Sciences, was formed by the invitation of Iranian expert scientists in the field of Chemistry, Physics, and Mathematics. Four workshops were presented the roles of analytical chemistry in quotidian life. Some analytical chemistry instruments and chemicals supplier companies presented their potentials in the seminar’s exhibition.

This event has been successfully held thanks to the collaboration of the presidency, vice-presidencies, dean of Faculty of Science of the University of Zanjan. The invaluable helps of our colleagues and students in the department of chemistry are sincerely acknowledged.

Industrial companies, Department of Environment of Zanjan Province, Institute for Advanced Studies in Basic Sciences, Cultural Heritage, Handicrafts and Tourism of Zanjan Province, Municipality of Zanjan City, Zanjan Industrial Estates Company, and Institute of Standards and Industrial Research of Zanjan Province, are profoundly appreciated for their support.

Mohammad Reza Yaftian

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August 2022

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Keynote and Plenary Talks (Time table)

Keynote Speakers	Title of speech	Date	Time (Zanjan zone)
Prof. Farzaneh Shemirani- University of Tehran, Iran	Smart Liquids to Improve Extraction Systems: the state of-the-art advances	01/06/01 (23-08-2022)	10:15 -11:15
Prof. Spas D. Kolev- University of Melbourne, Australia	Online Separation and Preconcentration of Au(III) in Bead Injection Analysis Using Micro Core-shell Polymer Inclusion Beads	01/06/01 (23-08-2022)	11:45 -12:30
Prof. Mustafa Soylak- Erciyes University, Türkye	Metal organic frameworks ad various nano composites for solid phase microextraction of trace analytes from environmental samples	01/06/01 (23-08-2022)	17:00 -17:45
Prof. Hossein Kazemian- University of Northern British Columbia, Canada	Establishing a Self-Sufficient (self-supporting) University Laboratory: Challenges and Opportunities	01/06/02 (24-08-2022)	08:00 - 08:45
Prof. Parviz Norouzi- University of Tehran, Iran	Modern electrochemical instrumentation for nano-faradic measurements: Design and Application	01/06/02 (24-08-2022)	17:00 - 17:45

Plenary Speaker	Title of speech	Date	Time (Zanjan zone)
Prof. M. F. Mousavi- Tarbiat Modares University, Iran	Development of Advanced Materials for Supercapacitors, Batteries, and Water Splitting for contributing to a better world	01/06/03 25-08-2022	08:00- 09:00

Time Table

Tuesday August 23, Morning

8:30-10:15	10:15-11:15	11:15-11:45	11:45-12:30	12:30-14:00	-
Opening Ceremony (Ghadir Hall)	Keynote Lecture (Ghadir Hall)	Coffee Break & Exhibition	Keynote Lecture (Ghadir Hall)	Lunch and Pray	-

Afternoon

14:00-15:30	15:30-16:30	16:30-17:00	17:00-17:45	17:45-19:15	19:15-
Oral Session (Ghadir & Sohrvardi Halls)	Poster Session	Coffee Break & Exhibition	Keynote Lecture (Ghadir Hall)	Oral Session (Ghadir & Sohrvardi Halls)	City Tour

Wednesday August 24 Morning

8-8:45	-8:45-9:45	9:45-10:15	10:15-11:45	11:45-12:30	12:30-14:00
Keynote Lecture (Ghadir Hall)	Poster Session	Coffee Break & Exhibition	Oral Session (Ghadir & Sohrvardi Halls)	--	Lunch and Pray

Afternoon

14:00-15:30	15:30-16:30	16:30-17:00	17:00-17:45	17:45-21:00	
Oral Session	Poster Session	Coffee Break & Exhibition	Keynote Lecture (Ghadir Hall)	Soltanieh Tour	

Thursday August 25

8:00-9:00	9:00-10:00	10:00-10:30	10.30-12:00	12:00-13:00	13:00-14:30
Plenary Talk (Ghadir Hall)	Poster Session	Coffee Break & Exhibition	Oral Session (Ghadir & Sohrvardi Halls)	Closing Ceremony Ghadir (Hall)	Lunch and Pray

Keynotes

Smart Liquids to Improve Extraction Systems: the state of-the-art advances

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Abstract: The advancement of smart materials has substantially affected the sample preparation techniques. Recently, different smart materials like carbon-based materials, magnetic nanoparticles, and ferrofluids have been used to separate analytes from different matrices. Among these, ferrofluids have attracted considrerable attention due to their liquidity and magnetic controllability, facilitating their recovery during the extraction process. Ferrofluids are dispersions of magnetic powders in a carrier liquid with a great magnetic response. Ionic liquids (ILs) possess unique physical and chemical properties, such as excellent solubility, negligible vapour pressure, and high thermal stability. Ionic liquid (IL)-based materials have been used as efficient in sample preparation due to their unique physico-chemical properties and ability to interact with analytes through multiple interactions. This article provides information about the applications, properties, and also recent advances of ferrofluids and type of IL-based adsorbents. We will talk about the future outlook in the development of ferrofluids.

Metal organic frameworks and various nanocopposites for solid phase microextraction of trace analytes from environmental samples

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Abstract: Metal organic frameworks (MOFs) are organic-inorganic hybrid crystalline porous materials. MOFs have an extraordinarily large internal surface area. Metal organic framework are very popular structures in the area of storage of gases including hydrogen and carbon dioxide, water absorption, catalysis and drug delivery. The properties of MOFs such as their high surface areas, their stability to acids and bases make them attractive for solid phase microextraction. The preparation of the nanocomposite adsorbents containing MOFs is also an attractive part of solid phase microextraction studies. In this presentation, the main strategies on the usage of MOFs and nanocomposites for the solid phase microextraction of organic and inorganic species at trace levels have been discussed with some examples from our research group.

Online Separation and Preconcentration of Au(III) in Bead Injection Analysis Using Micro Core-shell Polymer Inclusion Beads

Y. Zhang, R. W. Cattrall, S. D. Kolev*

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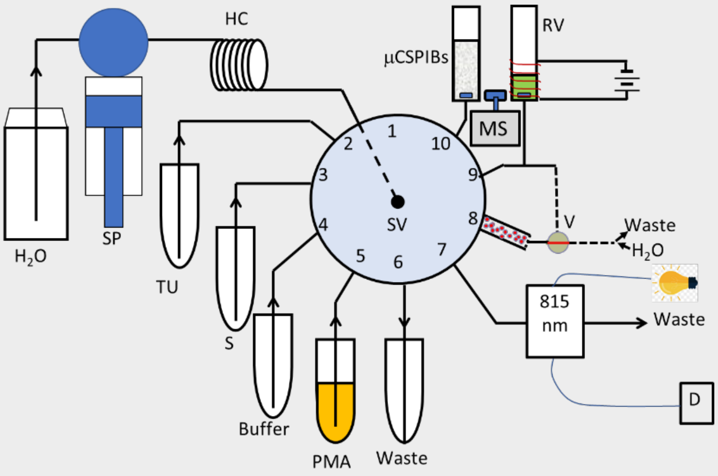
Graphical Abstract



Abstract: Micro core-shell polymer inclusion beads (µCSPIBs), composed of poly(vinyl chloride) (PVC) as the base polymer, Aliquat 336 (a mixture of quaternary alkylammonium chlorides) as the extractant/carrier and 1-tetradecanol as a plasticizer, were fabricated by a novel microfluidic technique and used for improving the selectivity and sensitivity of Au(III) determination in a bead injection analysis (BIA) system. This determination was based on the catalytic effect of Au(I) on the reduction of phosphomolybdic acid by thiourea. The analytical procedure involved first loading the separation column of the SIA system with µPIBs where Au(III) was separated from the sample matrix as a result of its extraction in the form of [AuCl4]- by Aliquat 336 into the µPIBs. This was followed by stripping the extracted Au(III) from the µPIBs into a receiving solution of thiourea where Au(III) was reduced to Au(I). The receiving solution was then mixed with a solution of phosphomolybdic acid which was reduced by thiourea to phosphomolybdenum blue and the latter was detected spectrophotometrically. The SIA method was optimized and successfully applied to the determination of Au in aqua regia digests of electronic scrap. The results were in good agreement with those obtained by inductively coupled plasma-optical emission spectrometry.

Modern electrochemical instrumentation for Non-faradic measurements: Design and Application

P. Norouzi

Center of Excellence in Electrochemistry, School of Chemistry, College of Science, University of Tehran, Tehran, Iran

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Abstract: The design of the most of the commercial potentiostat rely on the faradaic current, and non-faradaic current are limited or omitted. Such electrochemical (EC) instruments extensively limits application of EC measurement for non-redox compounds, which most of them cannot be measured. This is mainly due to submission DC part of the current in data processing result of redox reaction even in AC and square wave (SW) voltammetry. Here, it is intended to present an overview for describing the new advanced methods for non-faradic measurements along with its applications. These include the extension of the EC methods employed to detect nonredox molecules, protein and cells in medical, environmental and industrial interests. Also, our homemade electronic circuit for a Potentiostat, software equipped with FFT data processing, which include the basic theory of the non-faradic measurements in advanced EC methods. It will show that how non-faradaic measurements could be used for study of macroscopic processeces on the electrodes in solution interface. In fact, when an altering potential (AC or SW) excitation waveform was applied to the electrode, based on the electrical properties of compounds, non-faradic signal can be calculated and completely characterized. The electrode responses (which are frequency depended) represent the intrinsic properties of the system permittivity, impedance and capacitance. The selective or sensitive measurements based on capacitance, phase shift and admittance or impedance could be used for such compounds. Some of the methods offer EC measurements without need of a reference electrode. This could be amenable to miniaturize EC systems for detection of bio/molecules in the gas phase, the fluidic solution (for example, blood sample) and bacterial contain, which is noninvasive.

Challenges and Opportunities of Establishing a Self-Sustaining University Laboratory: The case of Northern Analytical Lab Services (NALS) at the University of Northern British Columbia (UNBC)

H. Kazemian

Head of The Northern Analytical Lab Services (NALS), Northern BC's Environmental & Climate Solutions Innovation Hub, University of Northern British Columbia (UNBC), Prince George, BC, Canada

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Abstract: The struggle to equip, maintain, operate, upgrade, and expand a university research and service laboratory has long been a challenge in academia. Securing and maintaining appropriate funding for research laboratories is the central obstacle most scientists face in their careers. All research laboratories require financial support for their consumable, to subsidize lab equipment, and to pay research staffs' salaries and benefits. There is new and promising model that might provide a sustainable solution to this everlasting problem. Some North American universities- particularly small size universities- are considering the establishment of central equipment facilities. The main mandates of these centralized instrumental analysis laboratories is to provide research and testing services the university researchers under a fee-for-service (FFS) model. While the FFS model might help the central labs to cover the cost of their consumables and partial cost of equipment maintenance, however, they need to diversify their source of funding to be able to operate, maintain and upgrade the facility sustainably.Therefore, revenue generation through conducting client-based research and development projects as well as providing routine and none-routine testing services, are being considered as viable options. To attract external clients (both from industries and local communities), a university-based lab facilities might need to pursue accreditation efforts as an effective trust-building measure with their clients and service users. To become an accredited facility, they need to invest on infrastructure as well as hiring (and retaining) highly qualified personnel (e.g., analytical lab specialists, quality assurance officer).In this talk, successes, and challenges of the Northern Analytical Lab Services (NALS) as the central equipment facility at the University of Northern British Columbia (UNBC) will be shared and discussed. Under new leadership in 2016, the NALS was created as a Central Equipment Lab to address the growing need for analytical services at the University of Northern British Columbia in Prince George, BC. The NALS is home to an extensive suite of analytical science instrumentation that enables a broad spectrum of in-house, provincially, federally, and internationally accredited biological, chemical, and physical analyses. NALS services are available to the private, public and non-profit sectors to help meet their research, development, analytical and quality assurance needs. In 2022, PacifiCan, a new Canadian federal agency, invested 1.92 M to establish the "Northern BC's Environmental & Climate Solutions Innovation Hub" in the NALS that will allow for the increased and expedited development and prototyping of new materials and technologies. This will help solve pressing environmental concerns in Northern B.C. in sectors including water management, mining, forestry, and bioenergy. As of October 2021, NALS is accredited to ISO 17025:2017 with the Standards Council of Canada. As of May 2021 NALS also accredited by the Eastern Water Quality Association for E.coli and coliforms in water. This means the NALS is committed to providing high quality and defensible data by participating and passing regular proficiency testing rounds.

Sources for more info: a) https://www.nalslab.ca/ b) https://www.canada.ca/en/pacific-economic-development/news/2022/06/government-of-canada-announces-support-to-the-university-of-northern-british-columbia-to-establish-the-environmental-solutions-innovation-hub.html

Plenary Talk

Development of Advanced Materials for Supercapacitors, Batteries, and Water Splitting for Contributing to a Better World

Mir F. Mousavi

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Abstract: Ever-increasing demands for portable electronics along with the inevitable demand for clean renewable energy have considerably pushed the development of next-generation energy storage and conversion devices with the characteristics of a high capacity/capacitance, high energy, and high power, fast charging capability, and long cycling stability. As a remedy to these challenges and to build a better world, we have developed some advanced electrode materials for electrochemical energy storage and conversion devices. In one approach, we used a facile and scalable laser-scribing method to prepare graphene films and fabricate a multifunctional system [1]. The laser-scribed graphene (LSG) in the multifunctional integrated system not only serves as a substrate-free micro-supercapacitor (μSC), but also serves as a thin-film humidity sensor, a resistor, and a near field communication (NFC) antenna for internet-of-things (IoT)-linked wireless communication with a smartphone. We also have reported a simple hydrothermal method for conjugation of thionine (Th) and Nile Blue (NB) as positively charged and readily available planar aromatic redox dyes to graphene aerogel via π-π stacking interaction [2, 3]. This non-covalent conjugation method induced extra stability to the Th(NB)-GA macromolecules and resulted in excellent structural stabilities and significantly higher supercapacitive performances. In another approach, we electrosynthesized an interpenetrating network of polyaniline and lignosulfonate (PANI-LS) on carbon fibers as a high-performance supercapacitive energy storage material [4]. We also electrosynthesized a trilayer metal-organic framework (MOF) in which Fe, Co, and Ni layers are electrodeposited in a layer-by-layer (LbL) assembled method [5]. The LbL assembled MOF displayed excellent energy storage performance as a cathode of a zinc-air battery that outperforms the commercial noble metal benchmarks and serves as a supercapacitive material as well. The trimetallic Fe-Co-Ni MOF also demonstrated excellent trifunctional electrocatalytic activities toward the hydrogen evolution reaction (HER, $\eta_{j=10}$ = 116 mV), oxygen evolution reaction (OER, $\eta_{j=10}$ = 254 mV), and oxygen reduction reaction (ORR, half-wave potential = 0.75 V vs. RHE). We also used layered-double hydroxides (LDH) and their composites with graphene-based materials for the development of a variety of energy storage and conversion devices [6, 7]. In a comprehensive review, we also surveyed the standard performance metrics of batteries and supercapacitors that would help the researchers of the energy storage community to evaluate the energy storage devices with well-established criteria [8]. We hope these intriguing approaches will inspire further studies and paves the way toward a more secure energy future.

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Invited Talks

On Essential Data Points and Their Relative Importance for Efficient Data Reduction

S. Valizade[1], S. Khodadadi[2], J. Mohammad Jafari[2] and H. Abdollahi[2*]
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Abstract: The term data reduction methods are defined as different mathematical algorithms that can be used to decrease big data dimensionality and/or data size by maintaining information. The extreme observations in a multidimensional data set for unraveling its underlying structure can be considered for efficient data reduction. These extreme observations are corresponding to actual signal vectors (data points) which are the most linearly dissimilar and they are representative of the most independent observed patterns in data set. Detection of these essential data points allows reproducing all the measured information and removing redundancies in data points which brings simplicity and computational speed. The new term Data Point Importance (DPI) defines an easily calculable value corresponding to each row or column of data matrix to reflect its impact for keeping the pattern of the data structure. Usually a lot of data points have DPIs equal or very close to zero that they do not carry on useful information about keeping the data pattern. DPI values for some of the data points are significant and they have been sorted regarding to their importances [1-3]. The basic idea for definition of essential points (EP) and also the concept behind the new term data point importance (DPI) will be explained. Application of both strategy in effiecient data reduction in several simulated and real experimental data will be discussed.

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Fluorescence Sensing Based on Carbon Dot-Metallic Deep Eutectic Solvent and Perylene Bisimide Nanocubes

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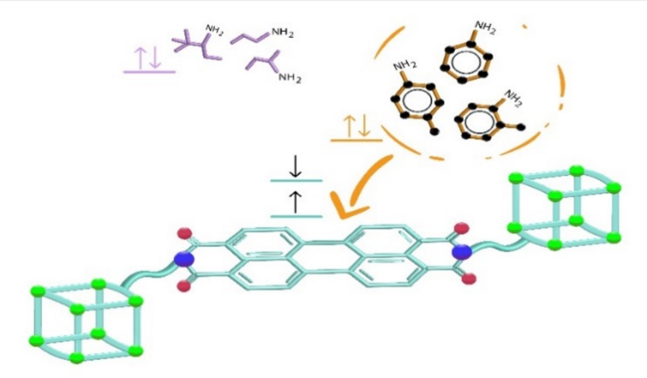
Graphical Abstract



Abstract: As an answer to the scientific challenges of the 21st century, our modern world today is increasingly shaped by optical sensors. Remarkable photo-stability, high-flexible surface modification, good water solubility, low cost and toxicity, and excellent biocompatibility are the key issues in developing optical sensors. Research and development of innovative concepts for high-performance sensors are in our research group, providing solutions for a widespread range of applications, such as food quality control [1], cancer detection [2,3] and drug monitoring.

In this contribution, the preparation of a miniaturized fluorescent probe based on the ultrasound–assisted formation of carbon dots (CDs) in a metallic deep eutectic solvent (MDES) is discussed. In-situ synthesis of CDs-chelated metals in the presence of the target analyte facilitates a kinetically fluorescence emission with no need to multi-step analysis. Simultaneous implementation of MDES as a green solvent and passivation agent also practically paves the way to minimize the hazardous chemicals consumption. Chelation of the divalent metal cations on the synthesized CDs and their strong interactions with the imidazole ring presumably provides a fluorescence quenching pathway induced by 4-methylimidazol (4-MeI) in commercial dark soft drinks. Also, the synthesis and characterization of a nanocubic morphology of perylene bisimide (PBI) derivative in which imide nitrogen atoms substituted by bulky polyhedral oligomeric silsesquioxane (POSS) to prevent π stacking is disclosed. The synthesized compound shows an intense red emission in solid state with an appropriate quantum yield efficiency. This nanocubic material is suitable for highly selective and highly sensitive (ng L^{-1} level) detection of gaseous aniline derivatives which are particularly vital in lung cancer detection. Our investigation showed that revealed sensor is more suitable than previously reported β-cyclodextrin grafted or bay area twisted PBI systems for photoinduced electron transfer reaction which has considered as a main pathway for quenching of PBI species in many applications including aniline derivatives sensing.

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Analysis of strongly Multi-collinear Spectroscopic Data

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Abstract: In chemistry, multivariate calibration methods are widely applied for investigating multi-component spectroscopic data and regression with strongly correlated data with ‘‘small observations and large predictors’’ is an issue of importance in spectroscopic analysis. Interpretation of the multiple regression equation depends implicitly on the assumption that the predictor variables are not strongly interrelated. This interpretation may not be valid if there are strong linear relationships among the predictor variables and the condition of severe nonorthogonality is referred to as the problem of collinear data, or multicollinearity.

It is recommended that one should be very cautious in regression analysis in the presence of multicollinearity. Thus, one of the challenges for analysis of spectral data is how to deal with the colinearty problem and data analysis methods should be explored for this problem and I will focus on three questions on the collinearity:

1. How does multicollinearity affect statistical inference and prediction?
2. How can multicollinearity be detected?
3. What can be done to resolve the difficulties associated with multicollinearity?

Roadmap for evaluation of univariate linear calibration curve in analytical chemistry

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Abstract: Assessment of the adequacy of a proposed linear calibration curve is necessarily subjective in chemical analysis. If the influential points in calibration are not identified and discarded, the constructed model will not have much validity and doesn't warrant the accuracy and precision of prediction step. Recognizing of influential points, outlier data, and discarding them is one of the steps in data processing that has been considered in various sciences. The influential points can be arisen from, I) bad design of calibration set II) gross error in doing experiments. So, we aimed to extract a map that recognizes the following issues: A) The existence of data with high regression coverage that is far from the rest and will strongly affect the accuracy of the calibration equation, high leverage points. B) Large error in the experimental process: the recorded signal does not match the desired concentration. C) Points with concentration lower than limit of quantification which calculated by considering standard error of regression instead of standard deviation of blank.

The efficiency of proposed roadmap will be reviewed and this will give a new perspective on the calibration equation to avoid common mistakes in analytical chemistry. To achieve the above goal, visual and statistically significant tests will be used and all tests will be performed in a simple Microsoft Excel environment.

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Smartphones and RGB Analysis: The Recent Developments in Analytical Chemistry

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Abstract: Today's attractive aspects of in situ analysis have pushed the analytical chemists' community toward design of portable and integrated analysis systems. The bottle-neck in creation such systems are returned to the quantification step. Already owned by the majority of the population in the world, smartphones equipped with high-resolution cameras, the ability to connect to the internet and install apps, fast processing systems, etc., are an inseparable part of today's life. Thus, a smartphone camera as an accessible and portable detector for analysis can be a convenient way to achieve this goal. According to literature, applications of smartphones in analytical chemistry can be classified to optical detection (including colorimetric, and various types of luminescence detections, pixelation as well as label-free detection), electrochemical detection, barcode reading, chemometric applications and smartphone imaging with fluorescence microscopy. Here, it is tried to have a brief discussion on these applications with focus on RGB analysis as well as their advantages and disadvantages.

Mesoporous silica magnetite nanocomposites for dispersive solid phase adsorption of pollutants from environmental samples

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Abstract: From the first discovery of mesoporous silica materials in 1992, they are known as an essential group of nanoporous materials that are synthesized in both basic and acidic environments using supramolecular species as templates and structure directing agent. Magnetic mesoporous nanocomposites considering different morphologies, structures, and particle sizes are classified as: monodisperse magnetic nanocrystals encapsulated in mesoporous nanospheres; microspheres encapsulating magnetic cores into aligned mesoporous silica shells; magnetic nanoparticles encapsulated in the pores or cages of mesoporous silica and rattle-type magnetic nanocomposites. In recent years, many studies described the applicability of these magnetic mesoporous silica materials (MCM-n, SBA-n, KIT-n, FSM-n,…) for the magnetic separation of inorganic and organic analytes/pollutants to extract or preconcentrate them by magnetic dispersive solid phase extraction (for liquid samples) or matrix solid phase dispersion (for powder samples) methods and for magnetically assisted removal of chemicals from polluted samples. The large specific surface areas, high chemical and mechanical stability, tunable pore sizes and volumes, easy separation by applying a magnetic field and fasil functionalization of their surface make them as a good candidate for application as novel adsorbents in the separation or removal of organic and inorganic compounds.

Export of Articles and Import of Equipment in the Field of Sensors; And the Problems Behind

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Abstract: As we all know, over the past few decades, the country's growth rate for article printing has increased, and we have overtaken many other countries, especially our neighbors. Among other academic disciplines, chemistry has been at the forefront of this movement. Researchers across the country are particularly interested in developing a variety of sensors. Iranians have written more than 18,000 papers in the subject of sensors, according to Scopus data. Of course, in addition to chemists, researchers from other fields including physics, chemical engineering, material science, electronics, etc. have also been active in this area. Unfortunately, despite the great success in publishing articles, we have not made much progress in the technology of applying these ideas. For example, more than 500 articles on the design of glucose measurement sensors have been reported by Iranian researchers. Meanwhile, only one domestic company has produced a glucometer instrument. More than 50 models of glucometers and 60 models of blood sugar test strips are imported from other countries. It is natural that a lot of foreign currency is taken out of the country to import this equipment.

What is the problem? In this lecture, we are trying to provide solutions by presenting the topic and enumerating some of the problems that are of interest to us academics. We hope to help solve this problem with the solutions provided by respected researchers after this lecture.

Desulfurization and Denitrogenation of Fossil Fuels: A Report on Our Current Activities and Future Plans

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Abstract: Fossil fuels consumption leads to sulfur and nitrogen oxide gases emission, which harms the environment. Strict fines are imposed if this emission is not controlled and reduced. Hydrodesulfurization (HDS) and hydrodenitrogenation (HDN) are adopted to remove sulfur and nitrogen compounds in petroleum refining systems. The Ni-Mo/Al_2O_3 and Co-Mo/Al_2O_3 catalysts are applied as catalysts in these process. The HDS and HDN have little effect on the aromatic sulfur and nitrogen compounds removal. Removing these compounds through HDS and HDN needs strict conditions (high temperature and pressure) [1]. Hence, the development of efficient methods for the removal of aromatic sulfur compounds has attracted the attention of researchers. For this purpose, liquid-liquid extraction desulfurization (LLEDS) [2], adsorption desulfurization (ADS) [3], biodesulfurization (BDS) [4], oxidative desulfurization (ODS) [5], and electrochemical desulfurization (ECDS) [6] methods are applied as the complementary methods in HDS process.

In this seminar, a report of our activities related to the application of liquid-liquid extraction methods, the use of synthetic adsorbents, magnetic adsorbents, carbon adsorbents prepared from agricultural wastes, the integration of liquid-liquid absorption and extraction methods, the integration of electrochemical oxidation methods and liquid-liquid extraction and assessment of three-dimensional electrochemistry and photoelectrochemistry in the removal of aromatic sulfur and nitrogen compounds in fossil fuels are presented.

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Exploratory Analysis of Different Types of Two Dimensional Metabolomics Data

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Abstract: Metabolomics has been successfully applied in many fields including clinical research, drug discovery, toxicology, and phytochemistry. There are three main questions which should be answered from analysis of metabolomics data. 1) Which biomarkers are responsible in appearing a specific phenotype? 2) Are different factors and their interactions important in appearing a specific phenotype in biological systems? And finally 3) how the concentration of specified metabolites are changed when a biological system is exposed to a stress? In fact extracting relevant biological information from large data sets is a major challenge in this field and comprehensive analysis of targeted and untargeted metabolomics data is still a major challenge.

Gas chromatography-mass spectrometry (GC-MS)-based metabolomics is ideal for identifying and quantitating small molecular metabolites (<650 daltons). This unit shows that on GC-MS-based metabolomics easily allows integrating targeted assays for absolute quantification of specific metabolites with untargeted metabolomics to discover novel compounds. The raw GC-MS data showing the three dimensional nature of the data (intensity as a function of elution time and mass). These data can be used in three formats: vectorized data, Total ion chromatogram (TIC) data and peak area data. Peak area data can be obtained with MCR-ALS method.

In this study exploratory analysis was applied on different types of a 2 factor simulated GC-MS data. The data were simulated in different conditions including the presence of selectivity in spectral mode for all components, one components and when there is not selectivity for any components in data. It was shown that different results were obtained when different forms of data is applied. PCA was applied as the exploratory method in this study.

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Structural Engineering of 2D Nanomaterials: Electrochemical Energy Storage Applications

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Abstract: Ultrathin two-dimensional (2D) nanomaterials such as transition metal dichalcogenides are fundamentally and technologically intriguing. The tunable electronic structure of 2D nanomaterials, especially metal dichalcogenides, makes them attractive for a variety of applications. They have been investigated as chemically active electrocatalysts for hydrogen evolution and hydro-sulfurization, as well as electrically active materials in opto-electronics. Their morphologies and properties are also useful for energy storage applications such as electrodes for ion batteries and supercapacitors. This representation explains the effect of interlayer spacing, tailoring the cracks and defects, and phase changes of the 2D nanomaterials on the excellent enhancement of their energy storage performance, especially in the K- and Na-ion batteries and supercapacitors. Moreover, different strategies will be introduced to do desirable engineering of 2D metal dichalcogenide nanomaterials to make energy storage devices with higher power and energy densities.

Metal-Organic Frameworks (MOFs): Application in the separation sciences

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Abstract: Metal-organic frameworks (MOFs) have received great attention due to their fascinating structures and intriguing utilization in diverse applications [1]. Here, the recent advances in the design and synthesis of MOFs with hierarchical porosity across the micro- and mesoporous for application as adsorbent for SPE and SPME methods as well as stationary phase of chromatography are summarized. Some examined MOFs have exhibited a good selectivity for adsorption of different compounds that is not seen in other materials, such as zeolites and activated carbon [2]. Recently, porous MOFs have been introduced good alternatives as stationary phases for chromatography (including gas- and liquid-phase), because they are crystalline and have high pore volume as well as tunable pore sizes/shapes. It has been demonstrated that some MOFs have exceptional thermal stability and high selectivity for application as separation medium in high-resolution gas chromatography [3]. Reverse shape selectivity and molecular sieving effect have been explored for capillary gas chromatographic separation of alkane isomers and benzene homologues on UIO-66 coated capillary column [4]. In addition, MOFs have been used as a new excellent separation medium in liquid chromatography due to their uniform morphology, good dispersion, and solvent stability. In a work, MIL-100(Fe) was explored as a novel stationary phase for both normal phase and reverse-phase high performance liquid chromatography [5]. The preparation of pure crystalline phase MOF monolith with a porous structure containing micropores, mesopores and macropores has been also reported for utilization as liquid chromatography stationary phase [6]. The presence of macropores may be particularly useful in improving mass transport for applications involving liquid phase. In addition, fabrication of MIL-100(Fe) coated open tubular capillary columns for use in capillary electrochromatography has been reported for separation of neutral, basic and acidic compounds [7]. Also, some authors have presented chiral chromatographic columns for the separation of some racemic mixtures over enantiopure porous MOF stationary phases [8]. Herein, the applications of MOFs in separation sciences are reviewed.

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Challenges in the determining of gold content in ores

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Abstract: Gold has been considered as a precious element since ancient times and has been a very important factor for power, peace and war in human societies. Gold is found in various forms in nature, and the type of gold found in soil and rock causes various errors in determination methods. Determining the grade of gold in metal coins and jewelry is easier and with less error than determining the real content of gold in ores. The challenge of determining the gold content and the reputation of world-renowned laboratories such as Acme laboratory in Canada are in field of determining the real content of gold in mineral samples. In this short opportunity, after a brief review of the importance of gold throughout history, the introduction of different forms of gold in nature, different methods of determination of gold content in jewelry, electronic components and ores will be reviewed and the errors of each method will be described. Based on my research experience in Felez Pooyan Company (www.felezpooyan.com), the suitable ways to measure total gold content will be introduced, especially in mineral samples. It will be identified that the big errors of determination of cold content is related to refractory and invisible coated native golds. Leaching and release of refractory gold is a difficult process and requires a complex process and sub-micrometer particles of ore. The entry of invisible coated native gold into the analytical sample solution requires a large volume of the ore sample, while it has already been ball milled into fine particles (sub-micrometer or nanoparticles).

A brief perspective on photodegradationm mechanisms

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Abstract: In recent past decades, explosive increased in the world population induced critical interests toward environmental and energy issues. Thus, introducing/developing novel techniques/catalysts is a critical research filed for analytical/environmental chemists. A common technique in the above mentioned issues is heterogeneous photocatalysis based on the illumination of a typical semiconducting material for inducing photoinduced electrons (e) in the conduction band (CB) and holes (h) in its valence band (VB). Other reactive species are powerful hydroxyl and superoxide radicals due to the reaction of the holes with water molecules and the electrons with the dissolved oxygen. The e/h recombination drastically decreases the overall efficiency of the photocatalysis process. An efficient strategy to diminish the e/h recombination is coupling of two or more semiconductors for constructing heterojunction structures. Mechanisms suggested for transferring the charge carriers between the contacted semiconductors are commonly type (II)-heterojunction and direct Z-scheme pathways that will illustrate here. Commonly, direct Z-scheme mechanism produce powerful reducing and oxidizing centers than the second case, and it is more efficient in photocatalysis. Water splitting for hydrogen production and oxygen evolution is a use of photocatalysis in energy issue, while degradation and mineralization of organic pollutants is its application in environmental issue.

Orals

Oral Session			
Subject	**Title**	**Paper Code**	**Presenter**
Separation	Selective Solid- Liquid Separation of RDX and HMX Explosive Using Deep Eutectic Solvent Based on Zinc Acetate/Diethylene Glycol	Isac27-1055	J. Ghorbani
Separation	Residues of Multi-Class Pesticides from Cow and Human Milk samples from Iran using UHPLC-MS/MS and GC-ECD: A Probabilistic Health Risks study	Isac27-1083	V. Mahdavi
Chemometrics	Detection of COVID-19; A Smartphone-based Machine-Learning-Assisted ECL Immunoassay Approach with the Ability of RT-PCR CT Value Prediction	Isac27-1114	Morteza Hosseini
Electrochemistry	Safe and stable Li–CO2 battery using conductive ionic liquid of 2-hydroxyethylammonium acetate as electrolyte and MWCNT composite cathode	Isac27-1190	M. Hosseini
Separation	Plasma Electrolytic Deposition as a Novel Coating Technique for Preparation of Novel Solid-Phase Microextraction Sorbents	Isac27-1299	A. Aghakhani
Spectroscopy	Development of colorimetric platforms for Biosensing using programmable Assembly of AuNP-Core sphericalNucleic acids	Isac27-1359	A. Karami
Separation	Application of Nanostructured Sorbents for Separation /Preconcentration of Various Pollutants in food samples	Isac27-1351	M. Shirani
Separation	Determination and extraction of fluoxetine antidepressant drug using Dispersive Liquid-Liquid Microextraction coupled with Solid Phase Evaporation followed by Corona Discharge Ionization-Ion Mobility Spectrometer Apparatus	Isac27-1077	K. Azadkish
Electrochemistry	Electro-Catalytic Reduction of Carbon Dioxide on Cobalt Phthalocyanine/Nitrogen- Doped Ordered Mesoporous Carbon	Isac27-1015	S. Barat Abtahi
Electrochemistry	Simultaneous Determination of Pb2+ and Hg2+ at Food Specimens by a Melamine-based Covalent Organic Framework Modified Glassy Carbon Electrode	Isac27-1024	M. R. Jalali Sarvestani
Chemometrics	The Ability of Multispectral Images and Chemometrics for Ripeness Prediction of Kiwi Fruit	Isac27-1227	A. Khosravi
General	Investigating and comparing incentive mechanisms and awards to chemists in Iran and the world and its role in research bias	Isac27-1290	A. Yeganeh-Faal
Separation	Vortex-assisted dispersive solid-phase microextraction of Cd(II) using Zeolite imidazolate framework (ZIF-8) from environmental samples before FAAS determination	Isac27-1051	N. Vaezi
Separation	Development and Validation of HPLC Method for the Determination of D-Chiro Inositol and Myo-Inositol in Pharmaceutical Dosage Form	Isac27-1058	S. Noroozi
Spectroscopy	Silver NanoTriangles Optochronometric Tongue For Determination And Discrimination of Halide Ions	Isac27-1086	P. Keshavarzi
Separation	Magnetic dispersive micro solidphase extraction based carbon nanotubes/metalorganic framework as nano	Isac27-1095	A.R. Fakhari

	sorbent for trace determination of triazole fungicides in agricultural waste water and agricultural soil samples		
Electrochemistry	Application of 4-Ethynylaniline in Electrosynthesis as Both Nucleophile and Electrophile	Isac27-1104	S. Khazalpour
Separation	Bismuth Ions Extraction by Polymer Inclusion Membranes and Electrospun Polymer Fibers Containing Ionic Liquid Aliquat® 336	Isac27-1115	J. Rajabi
Drug delivery	Redox and pH-responsive nanocarriers based on Boltron® H40 and PEG for tumor triggered targeted paclitaxel delivery: preparation and in vivo/in vitro evaluation	Isac27-1120	E. Mohammadi
Separation	A Novel Electrospun-Based Sorbent For Electrically Assisted Solid Phase Micro Extraction Of Six Non-Steroid Anti-Inflammatory Drugs And Simultaneous Determination Of Them In Human Plasma Samples With HPLC-UV	Isac27-1160	F. Nejabati
Spectroscopy	Vis-NIR hyperspectral imaging coupled with chemometrics for turmeric authentication	Isac27-1170	F. S. Hashemi-Nasab
Chemometrics	Investigation of Correlation between Chemical Model Parameters in Rank Deficient Data	Isac27-1177	S. Vali Zade
Electrochemistry	Electrochemical investigation of the interactions of Hydroxychloroquine and Famotidine in the treatment of patients with Covid-19	Isac27-1278	Z. Raeisi
Spectroscopy	Colorimetric Semi-Quantitative MicroRNA Detection Using AuNP-core Hairpin Spherical Nucleic Acids	Isac27-1245	M. Hasani

Selective Solid- Liquid Separation of RDX and HMX Explosive Using Deep Eutectic Solvent Based on Zinc Acetate/Diethylene Glycol

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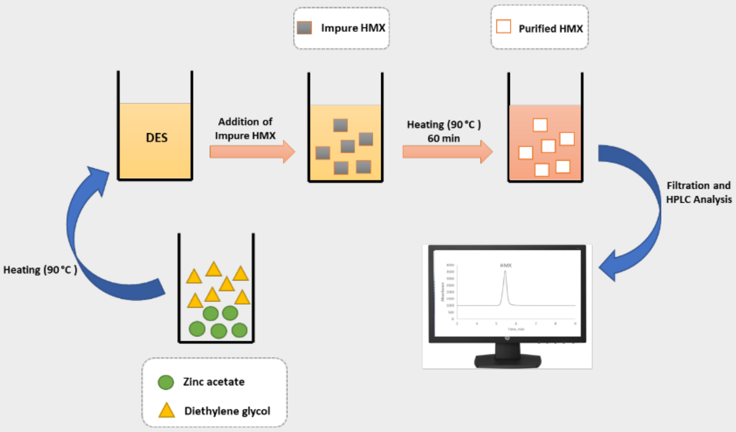
Graphical Abstract



A DES is a fluid generally composed of two or three cheap and safe components that are capable of self-association, often through hydrogen bond interactions, to form a eutectic mixture with a melting point lower than that of each individual component. These DESs exhibit similar physico-chemical properties to the traditionally used ionic liquids, while being much cheaper and environmentally friendlier. These solvents have been used in adsorption and separation processes as a substitute for conventional solvents [1,2]. In this study, deep eutectic solvent (DES) based on zinc acetate and diethylene glycol (DEG) was introduced for selective separation and purification of HMX explosive from RDX. The electrical conductivity and dielectric constant of different DES solutions were investigated by LCR meter (inductance-capacitance-resistance) in the range of 100 Hz - 1 MHz Solubility parameters such as activity coefficient, dissolution enthalpy and mixing enthalpy for RDX and HMX in the proposed solvent were also calculated. Using central composite design (CCD) in Minitab software, the solubility of two substances in DES with two parameters of temperature and zinc acetate concentration was modeled and optimized. Analysis of variance (ANOVA) showed that the experimental results of solubility have a good correlation with the predicted results. Experimental results also show that the solubility ratio of RDX to HMX varies in the range of 12 to 30 times, which makes it possible to provide a selective separation method for two substances that have similar chemical properties with a separation efficiency of more than 98% and a purity of HMX of more than Provides 99.5%. X-ray diffraction (XRD), high performance liquid chromatography (HPLC), laser-induced failure spectroscopy (LIBS) and FT-IR analysis were used to evaluate the product quality. According to the obtained results, this method can be used for selective, safe and green separation of HMX and RDX explosives.

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Residues of Multi-Class Pesticides from Cow and Human Milk Samples from Iran Using UHPLC-MS/MS and GC-ECD: A Probabilistic Health Risks Study

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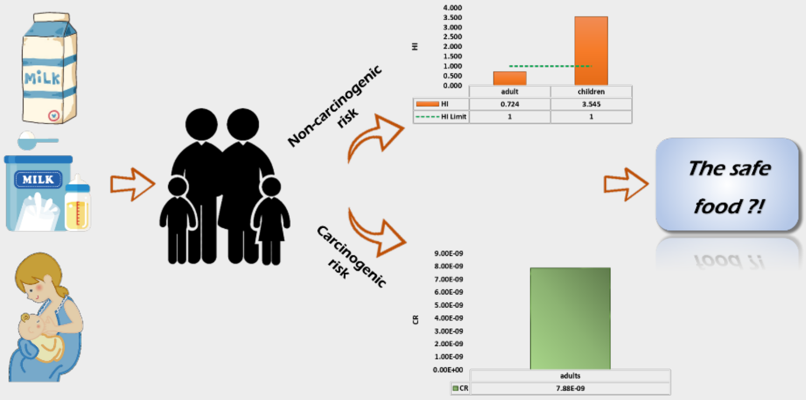
Graphical Abstract



Abstract: Raw, pasteurized, powdered cow milk and human milk samples from the capital city, Tehran were analyzed for investigation the residual of fifty important pesticides by gas chromatography with electron capture detector (GC-ECD) and mass detector for confirmation; and ultra-high performance liquid chromatography-tandem mass spectroscopy (UHPLC-MS/MS). No pesticide residue was evaluated in more than 91% of samples; in 3 human milk samples residue of organochlorine pesticides, p,p′-DDT and p,p′-DDD was recognized below MRLs. Only in 1 human sample residue of p,p′-DDE was more than codex MRL. Dimethoate residue was detected in 3 raw milk samples, more than EU MRL. HI in adults and children were 0.72 and 3.55, respectively. However, the health risk assessment based on HI demonstrated that adult consumers are not at considerable risk. HI, more than 1 in children confirms the risks of organochlorine (OCP) and organophosphorus (OPP) pesticide exposure threaten consumer health in Iranian children. In addition, there is no carcinogenic risk to milk consumers due to CR 7.88E-09.

This study aimed to evaluate the residual levels of pesticides and the health risks of pesticide exposure through milk consumption. Assessing the health risks of contaminants present in all types of milk using the Monte Carlo simulation showed that adults are not at significant non-carcinogenic and carcinogenic risk [1,2]. Nevertheless, due to dimethoate in raw milk, children are considerable at non-carcinogenic risk. Therefore, implement good farming practices on farms, improve the knowledge and consciousness of pesticide users, use safe further methods for pest control such as biotechnology-based, and use a rational program for application of pesticides, continuous monitoring of pesticides in crops, and tough government regulations on pesticide residues in food are recommended.

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Detection of COVID-19; A Smartphone-based Machine-Learning-Assisted ECL Immunoassay Approach with the Ability of RT-PCR CT Value Prediction

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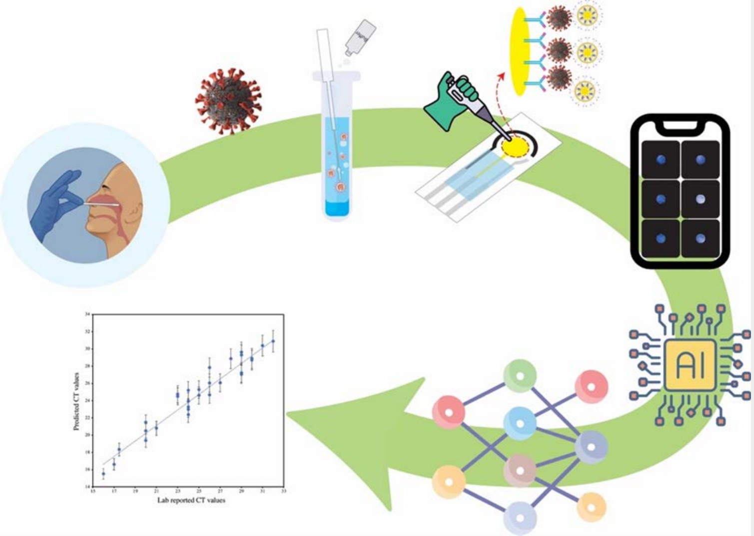
Graphical Abstract



Abstract: The unstoppable spread of SARS-CoV-2 has severely threatened public health over the past two years. The current ubiquitously accepted method for its diagnosis provides sensitive detection of the virus; however, it is relatively time consuming and costly not to mention the need for highly skilled personnel. There is a clear need to develop novel computer-based diagnostic tools to provide rapid, cost-efficient, and time-saving detection in places where massive traditional testing is not practical. Here, we develop an ECL-based detection system with a sensitivity comparable to that of RT-PCR. A concentration-dependent signal is generated upon the introduction of the virus to the electrode and is recorded with a smartphone camera. The ECL images are used to train machine learning algorithms, and a model using ANN for 45 samples was developed. The model demonstrated more than 90% accuracy in the diagnosis of 50 unknown samples, detecting upto a CT value of 32.

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Safe and stable Li–CO_2 battery using conductive ionic liquid of 2-hydroxyethylammonium acetate as electrolyte and MWCNT composite cathode

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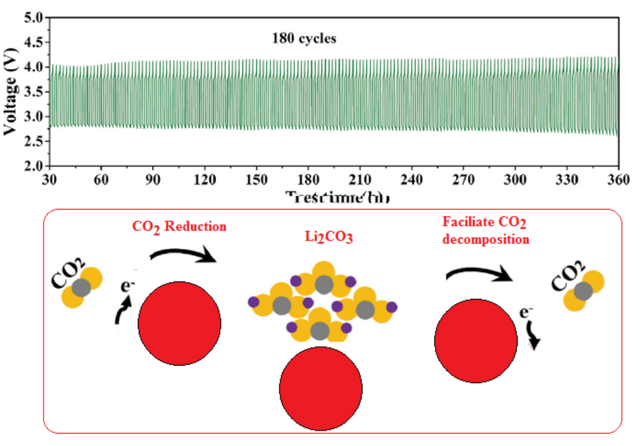

Abstract: As an emerging energy storage technology, the lithium-carbon dioxide battery has attracted worldwide attention in last decade, owing to its high theoretical specific energy density and recycling utilization of carbon dioxide from the atmosphere [1]. The involved electrochemical reaction in the Li-CO_2 battery is $4Li^+ + 3CO_2 + 4e = 2Li_2CO_3 + C$. Until now, its development has been enormously hindered by the sluggish kinetics of carbon dioxide reduction and evolution reactions. During the discharging step, the CO_2 is reduced on the cathode and, the main challenge is the high charge potential that arises from the electro-inactivity of the discharge product, Li_2CO_3, largely constraining the electrochemical performance and recharge ability [2]. The high charge potential also induces the inevitable decomposition of the aprotic electrolyte, thus making Li_2CO_3 incompletely decomposed. This usually results in the low energy efficiency, poor cycling performance, and poor rate capability of existing Li-CO_2 batteries. Many efforts have been devoted to developing highly efficient catalysts, for which many carbons based materials and aprotic electrolytes are still the best choice for facilitating the Li_2CO_3 decomposition [3]. To achieve good rate capability, the catalysts should greatly enhance CO_2 reduction and Li_2CO_3 nucleation, furthermore the electrolyte must to be very conductive. Here, we demonstrates a Li–CO_2 rechargeable battery with ionic liquid of 2-hydroxyethylammonium acetate ([2-HEA][AC]) as electrolyte and multi-walled carbon nanotubes as cathodic material. The physicochemical properties of ionic liquid are evaluated using FTIR and HNMR analysis. The ionic conductivity of the [2-HEA][AC] is measured by electrochemical impedance spectroscopy and found to be $\sim 6.46 \times 10^{-3}$ S.cm^{-1}. Further, the porous morphology of the carbon cathodes with MWCNTs is examined by SEM & TEM techniques. The electrochemical performance of battery is evaluated by galvanostatic tests at a current density of 50 mA g^{-1} with a cut-off capacity of 500 mAh g^{-1} for 180 cycles. The test results signify the high performance and good cyclic stability. The Li–CO_2 cell has delivered a maximum capacity of 4988 mAh g^{-1}.

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Plasma Electrolytic Deposition as a Novel Coating Technique for Preparation of Solid-Phase Microextraction Sorbents

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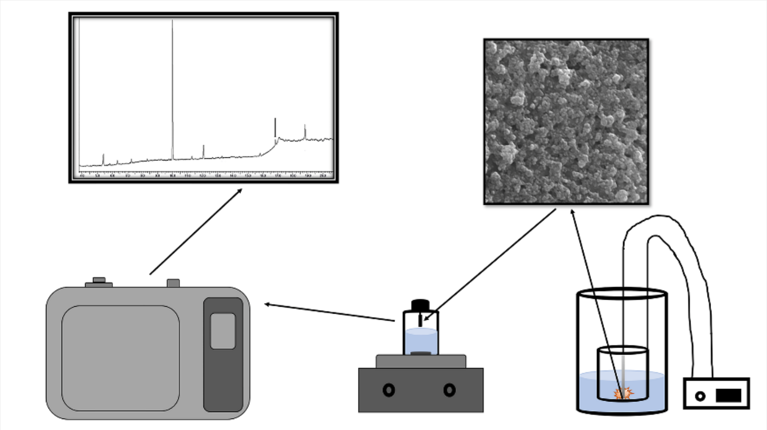
Graphical Abstract



Abstract: Novel solid-phase microextraction coatings were prepared using cathodic and anodic plasma electrolytic deposition (PED) techniques on the titanium wire and used to measure the content of nerolidol in water and water distillate of bitter orange' blossoms with GC-FID. PED is an attractive technique for metal surface modification, which can be used in cathodic PED configuration to coat the metal surface with carbon materials, while in the anodic configuration it causes surface etching and thus significantly increases surface porosity [1]. PED technique works based on contact glow discharge phenomena at atmospheric pressure. Within electrolyte solution, plasma glow discharge produces inside the bubbles formed around the smaller electrode which is surrounded by a liquid electrolyte. In the anodic configuration, an adherent oxide such as alumina, silica, or titania film is produced [2]. In the cathodic configuration, nano-crystalline graphite films form in an electrolyte composed of ethanol and phosphate buffer [3]. In this research, in the cathodic mode, a dense carbonaceous layer was deposited on the Ti rode. On the other hand, in anodic mode, the Ti surface etched by the PED process and a highly porous surface was created. Effective parameters in the extraction and desorption were investigated. The optimal conditions include microextraction of 50 mL of an aqueous solution containing 35% (w/v) NaCl at 45 °C for 45 min using anodic coating followed by desorption using 50 μL hexane in an ultrasonic bath for 5 min. The developed method has a linear dynamic range of 1 to 1000 ng mL^{-1}. The limit of detection (S/N=3) and the limit of quantification (S/N=10) of the developed method were 0.3 and 1 ng mL^{-1}, respectively. The relative standard deviation of the method is less than 16%. The prepared adsorbent can be used many times due to its good mechanical stability. The developed method was used for the determination of nerolidol, as a product quality indicator, in the water distillate of bitter orange's blossom. The developed method is a simple, convenient, sensitive and reproducible that can be used for the micro-extraction of volatile organic compounds in various aqueous and food matrices.

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Development of Colorimetric Platforms for Biosensing Using Programmable Assembly of AuNP-Core Spherical Nucleic Acids.

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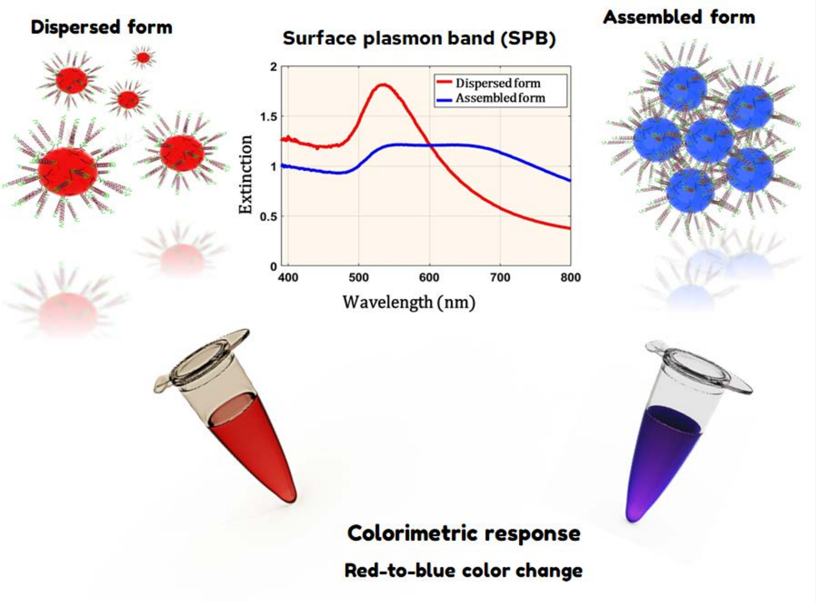
Graphical Abstract



Abstract: DNA molecules have a polymer language with four alphabets that can be designed with an arbitrary sequence, programmed base by base [1] Rather than directing biological processes, synthetic forms of DNA are now utilized to program the assembly of nanoparticles. Spherical nucleic acids (SNAs) are an emerging class of three-dimensional nanostructures typically made by arranging linear nucleic acids at high density around a nanoparticle core. [2, 3] Programmable assembly of SNAs is a target-responsive and sequence-specific molecular recognition event. In cases where SNAs are composed of gold nanoparticles (called AuNP-core SNAs), the programmable assembly events lead to generating naked-eye-observable colorimetric responses. The last two decades have seen a growing trend toward developing AuNP-core SNAs as medical diagnostic probes. This study provides greater insight into the utilization of DNA-programmable assembly of gold nanoparticles as a colorimetric transduction event for molecular diagnosis. Recently, we developed several strategies based on the target-responsive assembly of AuNP-core SNAs for the "naked-eye" colorimetric detection of severe acute respiratory syndrome coronavirus 2 (SARS-CoV-2) RNA. [4-6] Conventional polymerase chain reaction (PCR), which requires minimal laboratory equipment (a simple thermal cycler), was modified for molecular diagnosis via the 5′-exonuclease activity of the DNA polymerase. Due to public health interest in the management of infectious diseases using simple, accessible, and affordable laboratory equipment, the DNA-based programmable assembly strategies have a valuable potential to design biosensors for point-of-care testing.

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Application of Nanostructured Sorbents for Separation/Preconcentration of Various Pollutants in Food Samples

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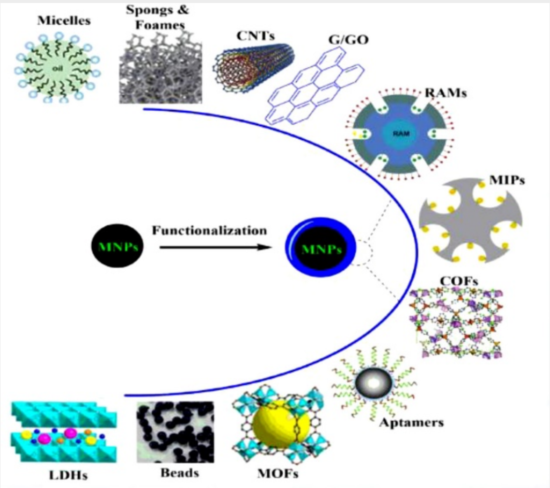
Graphical Abstract



Abstract: Food analysis is a very important branch of analytical chemistry, able to provide information about chemical composition, processing, quality control (QC) and contamination of foodstuffs, ensuring compliance with food and trade laws [1]. Pesticides belong among typical food contaminants that are monitored in a wide range of food commodities. Other contaminants may represent various veterinary drugs, especially antibiotics and hormones, in animal-based food, or mycotoxins, allergens, and food additives. Sorbents consist of nano-to micro-sized particles are being rapidly developed. Sorbent materials are of a great scientific and technological interests owing to their tendency to interact with specific substances and also their ability that can be efficiently separated from a mixture. Nowadays, sorbents, especially nanostructured ones, are widely used in sample preparation techniques and also for removal of pollutants from the environment. Application of sorbents considerably simplifies the sample pretreatment process without filtering and centrifugation. It also eases recycle and reuse of the sorbent. Nanocmposite sorbents such as NPs, nanocomposites, are generally used for preconcentration purposes. However, sponges, foams, hydrogel, and beads are generally used for removal purposes. Synthesis of novel magnetic hybrid sorbents by using advanced nanostructured materials such as MOFs, LDHs, G/GO, CNTs, COFs, RAM, MIPs and aptamers are being developed to find more efficient sorbents and introduce different extraction mechanisms at the same time. It is worthy to note that the preparation of novel nanocomposites are attractive approaches which leads to an efficient and selective extraction of target analytes form complex matrices without any pretreatment steps. Efficiency and simplicity of MSPE, QuEChERS, IT-SPME, SBSE and DLLME techniques can be greatly improved by using nanostructured sorbents [2]. In addition, automation of these techniques are other attractive trends. Finally, the preparation of novel and emerging sorbents with high adsorption capacity as possessing different functional groups to remove a wide range of contaminants especially in food samples.

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Determination and extraction of fluoxetine antidepressant drug using Dispersive Liquid-Liquid Microextraction coupled with Solid Phase Evaporation followed by Corona Discharge Ionization-Ion Mobility Spectrometer Apparatus

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Abstract: The MCM-41@SiO_2 solid phase was used as a porous adsorbent for preconcentration of fluoxetine antidepressant drug (as a model compound) and totally evaporation of the extraction solvents obtained by dispersive liquid liquid microextraction method. To detect of the analyte molecules, corona discharge ionization-ion mobility spectrometer (CD-IMS) was applied. The MCM-41@SiO_2 was synthesized as an adsorbent for coating on the solid phase fiber. In order to increase the extraction efficiency of the fluoxetine drug, some variables including, extraction solvent and its volume, disperser solvents and its volume, sample solution pH, desorption temperature, and evaporation time of the solvent from the solid phase fiber were chosen and optimized. Some analytical parameters including, limit of detection (LOD), limit of quantification (LOQ), linear dynamic range (LDR) with determination coefficient, and relative standard deviations (RSDs) were calculated at the optimized conditions. The LOD (S/N=3); 3 µg L^{-1}, LOQ (S/N=10); 10 µg L^{-1}, LDR; 10-200 µg L^{-1}, intra- and inter-day RSDs (n=3); 2.5%, 9.6% for 10 µg L^{-1}, 1.8%, 7.7% for 150 µg L^{-1}, respectively. To investigate the ability of the hyphenated method, some biological samples such as human urine, blood plasma, and saliva samples were selected and the relative recovery values were calculated.

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Electro-Catalytic Reduction of Carbon Dioxide on Cobalt Phthalocyanine/Nitrogen-Doped Ordered Mesoporous Carbon

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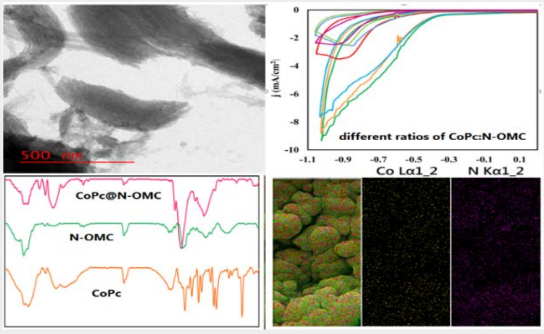
Graphical Abstract



Abstract: Ever-increasing dependence on fossil fuels consumption, has resulted in continuous enhancing the carbon dioxide emissions in recent decades. Therefore, in order to protect the life and slow-down the rate of climate change it is necessary to provide solutions to either reduce carbon dioxide emission or explore novel sustainable protocols for conversion of carbon dioxide coming from non-renewable resources to useful and preferentially non-hazardous materials since. However, carbon dioxide with its non-polar linear structure is chemically stable and difficult to reduce. Several strategies are exerted to reduce the amount of carbon dioxide. Among current methodologies, electro-catalytic CO_2 reduction is a green and cost-effective method. Electrochemical reduction of carbon dioxide is usually performed in an aqueous solution in which water provides required protons for electron transfer. However, electrolysis process is always accompanied with the release of hydrogen, which strongly and potentially prevents the reduction of carbon dioxide [1]. The use of catalysts and catalytic reactions are of particular importance to reduce energy consumption and to increase the selectivity in the chemicals as well as electro-chemical processes. To date, a broad spectrum of catalysts has been employed in electro-catalytic CO_2 reduction with different successes to hopefully obtain more suitable catalytic materials demonstrating more appropriate activities and selectivities. Nitrogen-doped ordered mesoporous carbon (N-doped OMC) exhibits superior chemical and electrochemical performance as a result of their unique properties derived from the nitrogen lone-pair electrons [2, 3]. These OMCs have several attractive features such as high accessible surface area, tunable porous structures, tailorable dopant configurations and chemical and thermal stability. On the other hand, metal phthalocyanines can be elegantly used as the catalysts for CO_2 reduction reaction (CRR). Metal phthalocyanines have many advantages including easy synthesis, high chemical stability, adjustable electronic structure, as well as the ability to combine with carbon structures to form the effective catalytic composites [4, 5]. In the current work a composite of CoPc/N-doped OMC was prepared as catalyst to modify electrode surface for CO_2 reduction. In this direction, N-doped OMC was fabricated by the carbonization of ionic liquid (1-methyl-3-phenethyl-1H-imidazolium hydrogen sulfate) and guanine using hard templating with ordered mesoporous silica SBA-15. CoPc was uniformly anchored on the resulting carbon surface to increase current density, reduce overvoltage and cause stability of the electrode. The resulted modified electrode exhibited suitable faradic efficiency in CRR.

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Simultaneous Determination of Pb^{2+} and Hg^{2+} at Food Specimens by a Melamine-based Covalent Organic Framework Modified Glassy Carbon Electrode

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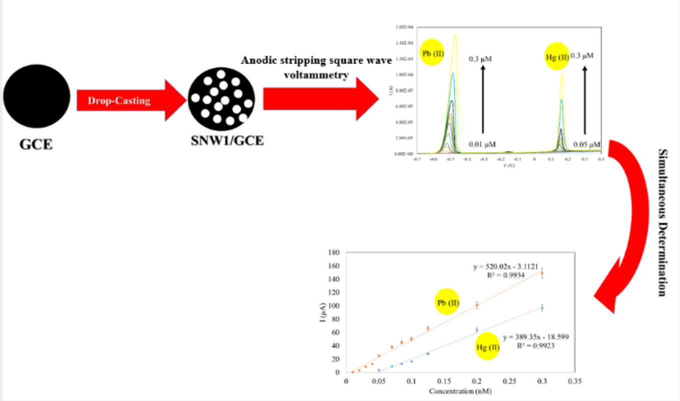
Graphical Abstract



Abstract: Establishing an analytical technique for the simultaneous determination of Pb^{2+} and Hg^{2+} at edible products is of great importance. Therefore, in this research, the applicability of a recently synthesized melamine-based covalent organic framework (Schiff base network$_1$ (SNW_1)) as a novel modifier for the simultaneous measurement of Pb^{2+} and Hg^{2+} was investigated. At first, the complexation of SNW_1 with Pb^{2+} and Hg^{2+} ions was evaluated by density functional theory calculations and the results indicated the complexation process is spontaneous, exothermic, experimentally possible, and thermodynamically favorable. Then, the morphology, chemical composition and electroanalytical function of the developed modified glassy carbon electrode were completely characterized by Fourier-transform infrared spectroscopy (FT-IR), energy dispersive X-ray spectroscopy (EDS), scanning electron microscopy (SEM), cyclic voltammetry (CV), square wave voltammetry (SWV) and Electrochemical impedance spectroscopy. Afterward, all of the effective experimental factors including pH, supporting electrolyte type and instrumental parameters were optimized by one factor at a time method. Under optimized conditions, the designed sensor showed a linear response over the concentration ranges of 0.01-0.3 and 0.05-0.3 µmol L^{-1} for Pb^{2+} and Hg^{2+} respectively with a detection limit of 0.72 and 12.11 nmol L^{-1}. The selectivity of the developed sensor over a wide range of ionic species was examined and the findings indicated the electrode has an admissible selectivity towards both analytes. In the end, the designed electrochemical sensor was successfully employed for simultaneous measurement of Pb^{2+} and Hg^{2+} at different edible samples including drinking water, white rice, black tea, red pepper, and marine salt and the obtained results were compared with a standard reference analytical technique.

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The Ability of Multispectral Images and Chemometrics for Ripeness Prediction of Kiwi Fruit

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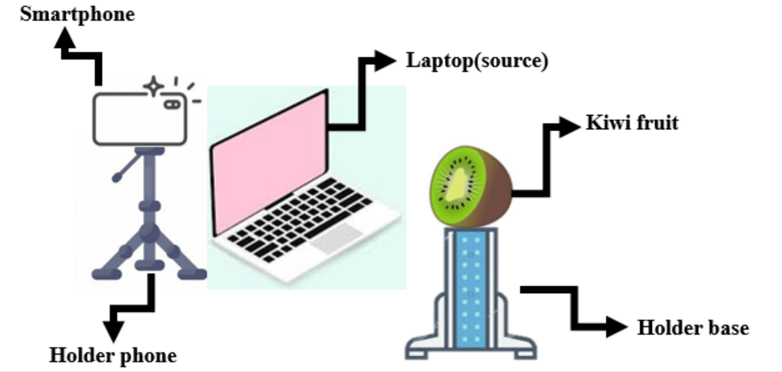
Graphical Abstract



Abstract: Spectral imaging combine the spectroscopic attributes of chemical measurements with those of imaging for chemical analysis. It has many applications in chemistry, biology, medicine, food science, and agriculture. In spectral imaging techniques, a spectrum is measured per pixel and the sample can be scanned without preparation [1]. Hyperspectral images (HSIs) are often composed of a large number of pixels a large number of variables to get sufficient spectral resolution and selectivity. However, in the multispectral image spectroscopic features are recorded within specific wavelength ranges. The wavelengths can be separated by filters or detected via the use of instruments that are sensitive to particular wavelengths. The size of the multispectral images can be very small but may carry more information than an RGB image. Kiwis are exceptionally high in vitamin C, a nutrient that helps protect our cells from oxidative damage and plays many other important roles in the body [3]. However, a visual inspection cannot detect its ripness easily, and squeezing is needed. On the other hand, the kiwis'ripeness can be changed over time until they over-ripeness In this study, we utilized a simple set-up including a laptop and a smartphone to monitor the kiwi's change over time. This set-up can collect multispectral data by changing the color of the laptop screen (9 colors). In each color of the screen, one RGB image is recorded by a smartphone. Each image is taken in a separate spectral band in the visible spectral region. By augmenting all of the unfolded RGB images, the multispectral image was generated. The multispectral images of kiwi fruit in 12 hours were used to predict time using Partial Least Squares Regression (PLSR). According to the results, the Root Mean Square Error values (RMSE) for the calibration (0.26) and cross-validation (1.8914) were the most optimal by selecting three factors. Therefore, three factors were used as the optimal number of factors to evaluate the PLSR model. The precision and accuracy of the model are adequate.

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Investigating and comparing incentive mechanisms and awards to chemists in Iran and the world and its role in research bias

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Graphical Abstract

Abstract: The role of incentive mechanisms and awards has been well-known and used in different societies since ancient times in order to guide and to lead and dominate a human group. Today, despite the passage of several thousand years, the use of these mechanisms in many areas of computer games, television advertising, elections and many other fields is inevitable. This mechanism has been used in educational and research processes as a result of this effectiveness. Several examples of these can be mentioned in the history of science. One of the brightest scientific periods in Iran was during the reign of the Buyid dynasty. The importance of science and books during the Buyid dynasty was so important and it has been shown that the relocation of the capital has been cancelled due to the impossibility of moving the library. This is the brightest and proudest period in Iran's history. The result of this system has been the encouragement of unique scholars such as Abu Ali Sina, Al-Shaykh al-Mufid, Al-Shaykh al-Saduq, Sayyid Razi, and many others. The libraries of Shiraz, Ray and Isfahan, which were built by the statesmen of the Al-Boya, have provoked the admiration of the world, and it is well-known that Ibn Sina had a great position as a minister with Shams al-Dawlah. This issue has also appeared in numerous government dynasties in Iran and the world, in all of these societies, the factors affecting this issue can be mentioned in politics, culture and economics. In leading societies, the external factor has been eliminated and the most important factors have been internal factors. Among these internal factors, it may be safe to say that politics and government played the most important role. In the last century, with the democratization of different societies and consequently, the scientific community, the process of selecting and encouraging scientific elites has been more influenced by scientific culture.

In this research, we have tried to study and compare the awards and incentive mechanisms in Iran and the world by focusing on chemistry and basic sciences in the last 100 years and the extent of its impact on the bias of scientific research in that field has been investigated. For example, awards of the Royal Society of Chemistry (35 items), awards of the American chemical society (34 items), awards of the Society of Chemical Industry (10 items and other countries such as Australia, Canada, Denmark, France, Germany, Europe and etc. were investigated. Due to the sensitivity of the present time period and pioneering chemistry in Iran, it has been tried to present this issue to the scientific community for a historical turnaround in an appropriate orientation based on global data and appropriate trends, and by providing solutions for a desirable and effective process.

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Vortex Assisted Dispersive Solid Phase Microextraction of Cd(II) using Zeolite Imidazolate Framework (ZIF-8) from Environmental Samples before FAAS Determination

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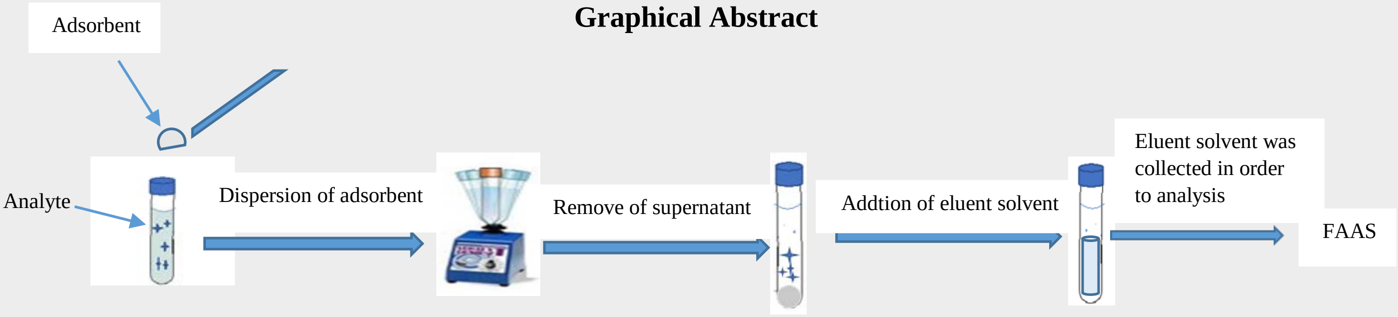
Graphical Abstract



Abstract: Cadmium as a highly toxic metal is released into the environment through paper production, metal processing, phosphate fertilizers, insecticides, and treatment of wastewater [1]. Cadmium also inhibits the body activities and is very toxic for kidney and other organisms [2]. Metal-organic frameworks (MOFs) have been intensely studied for the past few decades as an enormous family of highly tunable porous materials with promisingly applicable functionalities in adsorption, separation, catalysis, sensing, electrochemistry, and a great number of emerging purposes. Zeolitic imidazolate frameworks (ZIFs) are a subclass of metal-organic frameworks (MOFs) with isomorphic topologies of zeolites and coordinative composition of MOFs. They are fabricated via the self-assembly of transition metal ions and imidazolate linkers through tetrahedral coordination. Owning an approximate metal-imidazole-metal angle of 145° and analogous structures to aluminosilicate zeolites [3]. Therefore, ZIFs combine the advantages of both MOFs and natural zeolite. Due to their unique properties consisting of ultrahigh porosity, hydrophobicity, satisfactory thermo-chemical stability, and easy synthesis, ZIF-8 MOF nanoparticles are great candidate materials for adsorption applications [4]. In the current study, zeolitic imidazolate framework (ZIF)-8, was synthesized, fully characterized, and applied for the adsorption of Cd(II) in soli, vegetable juice, and water samples using vortex assisted dispersive solid phase microextraction method before determination by flame atomic absorption spectroscopy. The optimum conditions for this method were: pH of solution: 7, vortex mixing time for sorption:30 seconds, amount of adsorbent:10 mg, concentration of analyte 0.5 ppm, type and concentration of eluent: HCl and 0.03 molL-1, volume of eluent:3mL. Under the optimized conditions the limit of detection and relative standard deviation for the detection of cadmium ions were found to be 0.27 ppb and 0.81%, respectively.

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Development and Validation of HPLC Method for the Determination of D-Chiro Inositol and Myo-Inositol in Pharmaceutical Dosage Form

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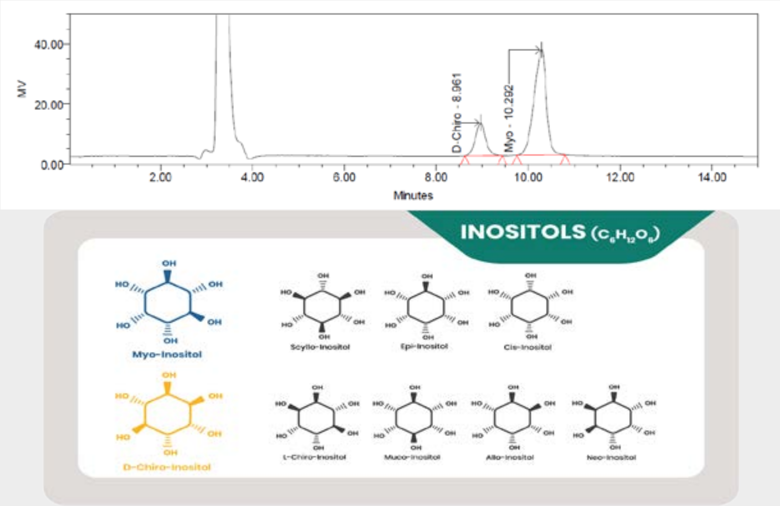
Graphical Abstract



Abstract: Polycystic ovary syndrome (PCOS) is one of the most common endocrine disorders in women of reproductive age and is characterized by menstrual abnormalities, clinical or biochemical hyperandrogenism, multiple abnormal cysts and enlarge ovaries [1]. Women affected by PCOS often suffer of insulin resistance and of a compensatory hyperinsulinemia which put them at risk of developing several metabolic disorders. Inositol is a six-carbon polyol which has been characterized as an insulin sensitizer: it exists as nine different isomers and among them D-chiro inositol and myo-inositol are the most represented and studied in physiology and physiopathology. In particular, D-chiro inositol (DCI) and myo-inositol (MI) glycans administration has been reported to exert beneficial effects at metabolic, hormonal and ovarian levels.The proposed HPLC method is a suitable technique for the simultaneous determination of D-chiro inositol (DCI) and myo-inositol (MI) in pharmaceutical dosage form. The development and validation was performed using a liquid chromatographic system which is equipped with RI detector. The chromatographic column used for separation was HECTOR-M NH2, 250×4.6 mm, 3µm. The mobile phase used for the separation was acetonitrile-water (75:25 % v/v). The column temperature was maintained at 55 °C and the detector at 50 °C. The injection volume was 10 µL and the flow rate was 1 mL/min. The RP-HPLC method has shown good resolution between D-chiro inositol (DCI) and myo-inositol (MI). The method was validated by determining system suitability, linearity, repeatability and intermediate precision, accuracy and robustness in accordance with the guideline of the International Conference on Harmonization (ICH) [2]. The calibration curves obtained were linear (R2=0.999) over the concentration range of 0.5-3 mg/mL and 1.8-10.8 mg/mL for DCI and MI respectively. The percent relative standard deviation values (%RSD) in the repeatability and intermediate precision study were less than 1%. The recovery values obtained were 99-101% and 95-96% for DCI and MI respectively. The results reveal that the developed method was guileless, commercial, fast and precise analytical method for the routine quantitative determination of D-chiro inositol (DCI) and myo-inositol (MI) in pharmaceutical dosage form without any interference from the excipients.

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Silver NanoTriangles Optochronometric Tongue for Determination And Discrimination of Halide Ions

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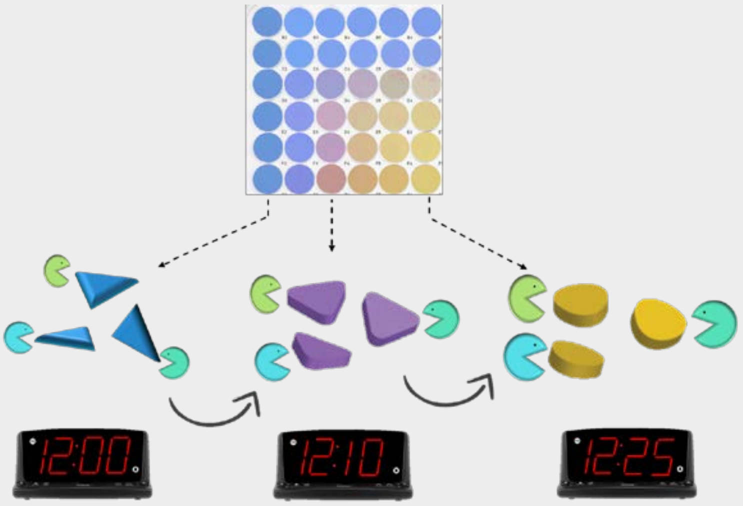
Graphical Abstract



Abstract: Halide ions including Cl^-, Br^- and I^- ions are known as critical species for human health, and their appropriate amount of them can be very useful for body. In contrast, the excess amount of halide ions may cause serious problems including neurological dysfunction and endemic goiter[1]. Due to the important roles of the halide ions in human health, the development of rapid and simple yet reliable probes for the detection of these species have attracted significant interest in recent years. Hence, in this study, Ag triangle nanoparticles (Ag TNPs) were exploited as colorimetric sensing element for the detection of halide ions [2, 3]. The halide ions (I^-, Br^- and Cl^-) at different concentrations can etch the sharp vertices of Ag TNPs and produce semi-spherical AgNPs. Consequently, changing the structures of Ag TNPs to AgNPs leads to a dramatic blue shift in the plasmon resonance band of Ag TNPs and changes the color from blue to yellow. Therefore, the spectra changes, as a result of etching the Ag TNPs produced a unique color and spectra variation for each halide ions. Given these halide ions have differential effect on the kinetic of etching Ag NTPs, a colorimetric sensor array has been developed to successfully discriminate halide ions with the help of pattern recognition analysis. Finally, the capability of the sensor in quantitative and qualitative analysis of the halide ions in tap water was investigated by which the practicality of the proposed strategy was successfully verified.

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Magnetic Dispersive Micro-Solid Phase Extraction Based Carbon Nanotubes/ Metal-Organic Framework as Nanosorbent for Trace Determination of Triazole Fungicides in Agricultural Waste Water and Agricultural Soil Samples

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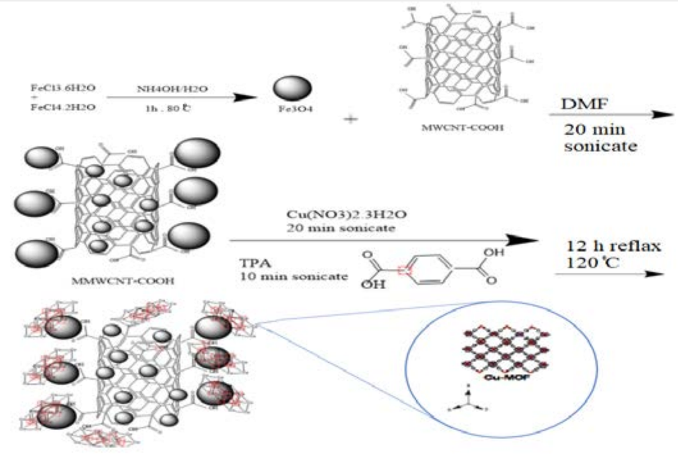

Abstract: Triazole fungicides (TFs) are systemic pesticides that are broadly used to prevent fungal sickness in agriculture. Hexaconazole, triticonazole, difenoconazole belong to the triazole group of fungicides and are widely used in vegetables and fruits fields to control diseases such as blast, sheath blight and false smut and fruits spots[1]. Thus, developing a fast, simple and reliable method to monitor the residues of fungicides in the environment is of prime importance[2].

In this research, the magnetic dispersive micro solid phase extraction (M-D-µ-SPE) method was used to pre-concentration and traces detection of triazole fungicides in agricultural waste water and agricultural soil samples. The nanocomposite based on magnetized carbon nanotubes/Cu-based MOF (M-MWCNT-Cu-BDC) synthesized and used as a nanosorbent. The nanocomposite was characterized by Fourier transform infrared spectroscopy, X-ray diffraction, vibrating-sample magnetometer, field emission scanning electron microscopy and energy-dispersive X-ray spectroscopy. The effective parameters were optimized in the method and under the optimized conditions (solution pH value: 7.0; amount of sorbent: 30 mg; extraction time: 30 min; desorption agent: ethanol and desorption time: 5 min), the limit of detection (LOD) was 0.45 ng ml-1 and the linear range was 1.50 to 750 ng ml-1 with a correlation coefficient higher than 0.993. The repeatability (%RSD) of the method for 5 replications ranged from 2.8 to 5.5%. Excellent preconcentration factor (PF = 91-94) and high recoveries (above 90%). Finally, this method applied to determination of triazole fungicides in agricultural waste water and agricultural soil samples.

M-D-µ-SPE method using a novel nanosorbent M-MWCNT-Cu-BDC was successfully implemented for the extraction and sorption of TFs from various real samples prior to the CD-IMS quantification. The presented sample preparation method is a new extraction method for separation and trace detection of TFs in environmental samples.

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Application of 4-Ethynylaniline in Electrosynthesis as Both Nucleophile and Electrophile

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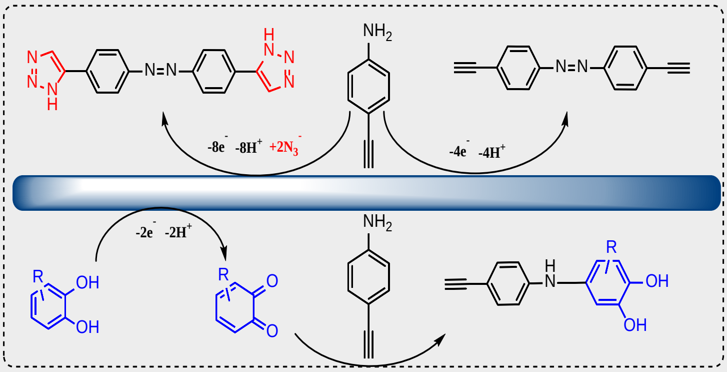

Abstract: Ethinyl anilines, as a group of heteroaryl acetylenes, are widely used in materials science, especially as a strong electron donor for applications in optoelectronic devices and their ability to the formation of a diazonium compound and doing the polymerization reaction [1-2]. Also, these compounds as primary amines play a pivotal role in the synthesis of various substrates [3-4]. In this study electrochemical oxidation of 4-ethynylaniline was studied in buffer solution/acetonitrile mixture in different pHs. Our electrochemical data assert that the product of oxidation of 4-ethynylaniline is unstable in acidic and alkaline solution, and can be hydrolyzed in strong acidic (pH: 1–3) and alkaline (pH: 9–10) solutions. In continues, the electrochemical synthesis of 1,2-bis(4-ethynylphenyl) diazene was carried out by electrochemical oxidation of 4-ethynylaniline in aqueous HCl buffer and in a simple undivided cell, using carbon anode. The electrochemical reaction of 4-ethynylaniline (4-EA) in the presence of sodium azide in buffered solutions with the various pHs (buffer solution /acetonitrile (80/20 v/v)) was investigated for the first time. Our electrochemical data assert that the product of oxidation of 4-ethynylaniline enters the chemical reaction with azide ion. In continues, after the 1,3 dipolar cycloaddition, electrochemical oxidation, dimerization, and third step electrochemical oxidation a triazole ring was produced as the desired product in a simple undivided cell and using carbon anode. Electrochemical oxidation of 3 and 4 substituted catechols in the presence of 4-ethynylanilin as a nucleophile in acetonitrile/ sodium acetate 0.15M (30/70) solution has been carried out in detail with employing the cyclic voltammetry and controlled potential coulometry methods. The results show that the *o*-benzoquinones derived from these catechols participate in Michael type reaction with this nucleophile to form the corresponding new *p*-quinone imines.

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Bismuth Ions Extraction by Polymer Inclusion Membranes and Electrospun Polymer Fibers Containing Ionic Liquid Aliquat® 336

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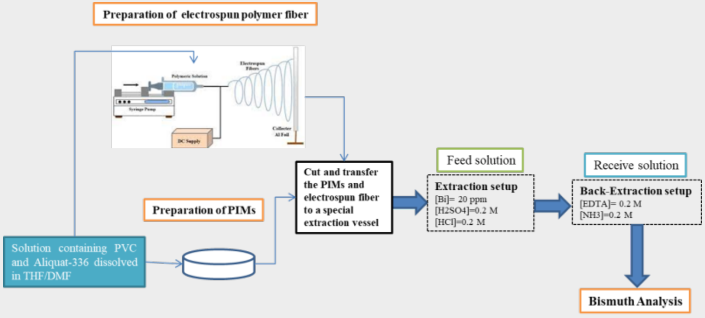

Abstract

Polymer inclusion membranes (PIMs) an approximately newly developed liquid membranes, are known to be an alternative method for solvent extraction [1]. PIMs are thin, homogenous with naked eyes and flexible film which fabricated by using a base polymer, an extractant and sometimes, a plasticizer. Electrospinning also, is innovate method to fabricate of polymer fibers (EPFs) [2]. The fibers have a high porosity and a large surface area to volume ratio. PIMs and fibers can be used in many applications. In this work, a PVC-based PIM and electrospun polymer fiber containing trioactylmethyl ammonium chloride (Aliquat® 336) as extractant were prepared. PIMs and EPFs with 70 wt% PVC and 30 wt% Aliquat® 336 were selected as optimum composition. The PIM and EPF were characterized by contact angle, TGA, SEM, AFM and BET analysis and employed for Bi(III) extraction. Morphological and thermal analysis showed that the EPFs have different physical, mechanical and surface properties compared to the PIMs. The presence of a porous structure in the EPFs enhances surface area comparing PIMs and increases bismuth extraction rate [3]. The factors influencing the extraction of bismuth were investigated. The results revealed that the presence of chloride ions is required for the bismuth extraction by Aliquat® 336 and highest extraction efficiency was obtained in 0.2 mol L^{-1} HCl and H_2SO_4. The effect of interfering ions on the extraction was also investigated. An EDTA/NH_3 solution was used to back-extract bismuth from PIMs and EPFs. Further research is shown that EPFs are more efficient than PIMs in the extraction and back-extraction consecutive cycles also, the maximum capacity of EPFs is much higher than the PIMs. This study showed that EPFs have a high performance and capacity for metal ions extraction.

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Redox and pH-responsive nanocarriers based on Boltorn® H40 and PEG for tumor triggered targeted paclitaxel delivery: Preparation and in vivo / invitro evaluation

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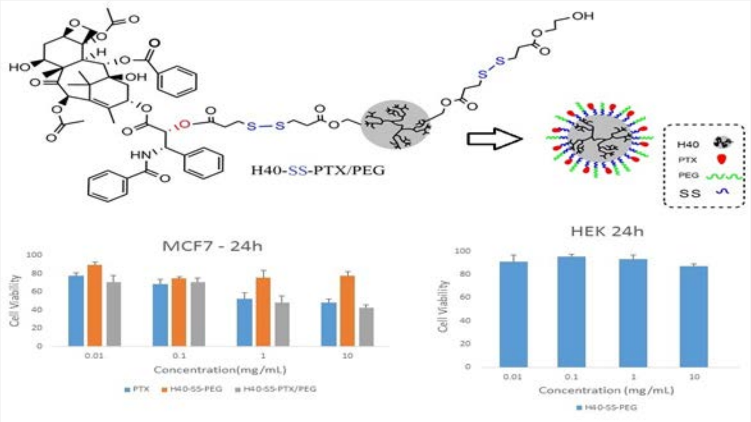

Abstract: The adoption of Boltorn H40, a commercially available dendritic polymer of Boltorn family containing multiple hydroxyl groups with various functionalities as a dendrimerbased starting core template for the generation of hyperbranched polymers, offers a straightforward solution to address this problem. In this contribution, hyperbranched polyester, boltorn® H40 (H40), a commercially available dendritic polymer of Boltorn family containing multiple hydroxyl groups with various functionalities as a dendrimer based starting core template for the generation of hyperbranched polymers, were used and characterized for tumor triggered targeted Drug delivery. Multi-arm star amphiphilic block copolymers with approximately 32 arms based on a hyperbranched polyester, boltorn® H40 (H40) amphiphilic star copolymers (H40-SS-PEG) with an a hyperbranched polyester, boltorn® H40 (H40), was used as the macroinitiator for the ring-opening polymerization and 3 3'-dithiodipropionic acid (DTDP) -a-poly(ethylene glycol) copolymer arms linked with acetal groups are synthesized using ring-opening polymerization and a copper (I)catalyzed alkyne-azide cycloaddition click reaction. The resulting multi-arm H40-DTDP (3 3'-dithiodipropionic acid) (H40-SS-COOH) was further reacted with carboxyl terminated poly(ethylene glycol) (PEG-COOH) to form H40-SS-PEG copolymers were synthesized as a nanocarrier for tumor-targeted paclitaxel delivery, anticancer drug PTX was covalently conjugated on the amphiphilic block copolymer arms by pH-responsive Disulfide Bonds.The block copolymer was characterized using 1H NMR, Fourier-transform infrared spectroscopy (FTIR), UV–visible spectrophotometry, Dynamic light scattering (DLS) and transmission electron microscopy (TEM) analysis. The results indicate that this multifunctional nanocarrier is a significant breakthrough in developing a drug delivery vehicle that combines drug targeting as well as sensing and therapy at the same time.

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A Novel Electrospun-Based Sorbent for Electrically Assisted Solid Phase Micro Extraction of Six Non-Steroid Anti-Inflammatory Drugs and Simultaneous Determination of Them in Human Plasma Samples with HPLC-UV

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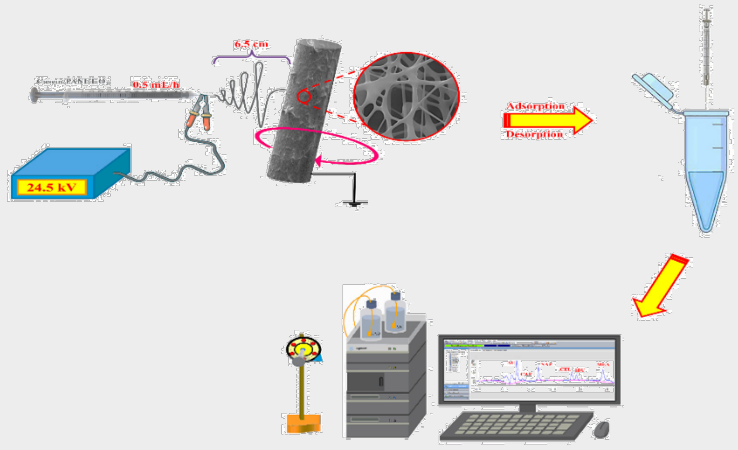

Abstract: Development of novel electrospun nanofibers as a sorbent for electrically assisted solid phase micro extraction (EA-SPME) of NSAIDs is essential for sample pre-treatment. Herein, firstly, a novel electrospun nanofibers (PVA/casein/PANI/TiO_2 nano particles) was fabricated for EA-SPME on pewter rod; and then, trace amounts of six NSAIDs (Acetaminophen, Caffeine, Naproxen, Celecoxib, Ibuprofen and mefenamic acid) were extracted. Finally, these drugs were determined from different human plasma samples with HPLC-UV. From the characterization of PVA/casein/PANI/TiO2 nano particles electrospun nanofibers with field emission-scanning electron microscopy (FE-SEM), energy-dispersive X-ray spectroscopy with elemental mapping analysis (EDS-Mapping), Fourier transform-infrared (FT-IR) and X-ray diffraction (XRD), the synthesis of composite nanofibers was approved. In addition, casein that was existed in composite nanofibers, has many functional groups and it is a polar molecule that we extracted it from expired milk with acetic acid but it could not preparate electrospun nanofibers alone and PVA must be mixed with it [1]. Due to the NSAIDs are polar molecules; so, there are interactions between electrospun nanofibers and analytes. Also, the electrochemical synthesis of PANI on platinum disk electrode by chronoamperometry and use of it in composite nanofibers, leads to that the used pewter rod in EA-SPME was more conductive for using of it as a working electrode at electrochemically desorption with chronoamperometry technique. But in this work the adsorption was done chemically [2]. Also, TiO_2 nanoparticles was used for increasing the porosity of electrospun nanofiber [3] but the selectivity of this technique did not change. The optimum percent of additive compound to casein in order to electrospinning, was reached from design-expert 12.0 software. Also, the effective factors on adsorption and desorption process were optimized by one at time (OAT) method. According to optimum conditions, the wide linear range was obtained with coefficient of determination (r^2) ≥ 0.99, low limits of detection based on S/N=3 and large enrichment factors. Finally, the efficiency of the EA-SPME-HPLC-UV method was evaluated for the determination of NSAIDs in human plasma samples with good recoveries.

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Vis-NIR hyperspectral imaging coupled with chemometrics for turmeric authentication

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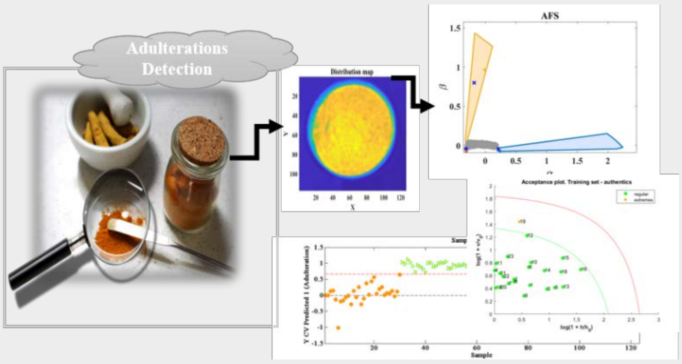
Graphical Abstract



Abstract: Ensuring food quality is a vital issue because food plays an essential role in human health [1]. Spices are precious and are often ground or powdered, which can cause many counterfeits by using additives and different colors [2]. One of the most popular spices in the world is turmeric (Curcuma longa), which is used in various fields such as food and medicine [3]. In the present contribution, the visible-near infrared hyperspectral imaging (Vis-NIR-HSI) system combined with different chemometric methods is proposed as a novel technique for authentication and detecting five common adulterants (corn flour, rice flour, starch, wheat flour, and zedoary) in turmeric powder. The Vis-NIR hyperspectral images of three datasets were recorded and arranged in different datasets including 23 authentic samples (dataset I), binary mixtures of pooled turmeric and each adulterant in seven concentration levels (1-35%) (dataset II) and mixtures of five plant materials and turmeric in different percentages by D-optimal design (dataset III). Then, multivariate curve resolution-alternating least squares (MCR-ALS) and mean-field independent component analysis (MF-ICA) were used as multivariate resolution methods to exploit pure spatial (distribution map) and spectral profiles of the pure components [4]. Due to the presence of rotational ambiguities, the performance of these two algorithms were compared by considering the area of feasible solutions (AFSs) using FACKPACK. The resolved spatial profiles (distribution maps) of turmeric was then used to find patterns of authentic samples by using principal component analysis (PCA). In the next step, data-driven soft independent modeling of class analogy (DD-SIMCA) was used for modelling of the distribution maps obtained by MCR-ALS and MF-ICA. On this matter, the model sensitivity and specificities obtained based on MCR-ALS resolved distribution maps of authentic turmeric samples were better than those obtained by MF-ICA. Finally, partial least squares-discriminant analysis (PLS-DA) was utilized for supervised classification and distinguishing between authentic and adulterated turmeric samples again by using distribution maps obtained by MCR-ALS and MF-ICA. Again good model accuracies were obtained for calibration and prediction sets (100% for MCR-ALS and 96% for MF-ICA). To test the applicability of the proposed method in mixed samples, dataset III was analyzed by the developed DD-SIMCA and PLS-DA models and good classification results were obtained. This was proof that the proposing model can detect adulterants in turmeric powder even at one percent of the adulteration level. Therefore, it is concluded that Vis-NIR HSI combined with chemometric methods is a powerful and novel technique for turmeric authentication and adulteration detection.

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Investigation of Correlation between Chemical Model Parameters in Rank Deficient Data

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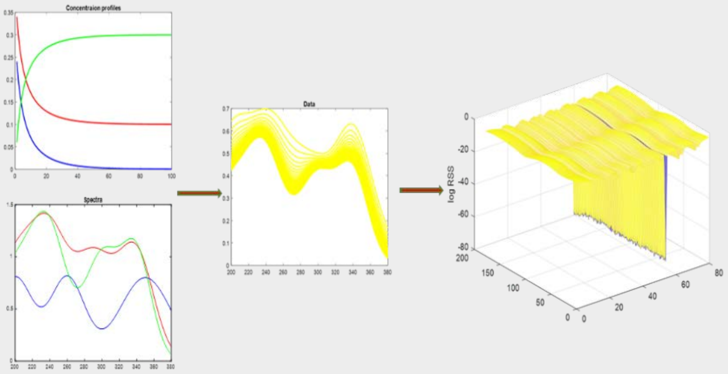
Graphical Abstract



Abstract: Nonlinear fitting is a method for calculation of chemical model parameters. A problem that complicates some applications of nonlinear modeling is correlation between the parameters of the model. Two interdependent parameters in a model fitting are said to be correlated, if the fitted value of each parameter depends on the fitted value of the other parameters. If correlation is very strong, convergence and /or unique estimates of each parameter may be difficult to achieve. In this condition often convergence cannot be obtained at all or requires a tedious search for a set of initial parameters from which convergence is attainable. Models with partial correlations may often be used successfully but degraded precision in parameters compared to an uncorrelated model are the costs that may have to be paid. The chemical model fitting can be more complex when there are linear dependencies between concentration profiles. This situation is known as rank deficiency in measurement chemical data. In such cases, there are several different strategies, to treat rank deficiency problems in the concentration matrix and to allow the fitting of pure component spectra. The correlation between model parameters in rank deficient systems and also the effects of different rank deficiency breaking methods on these correlations have not been investigated systematically.

In this research the relation between correlation of model parameters and rank deficiency in concentration profiles has been investigated. Different kinetics and equilibria models with inherent linear dependency in their concentration profiles have been simulated and evaluated. The influence of some different experimental situation such as different initial concentrations, known spectra as initial information, extent of reaction progress and different values of model parameters on the correlation of them have been evaluated.

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Electrochemical investigation of the interactions of Hydroxychloroquine and Famotidine in the treatment of patients with Covid-19

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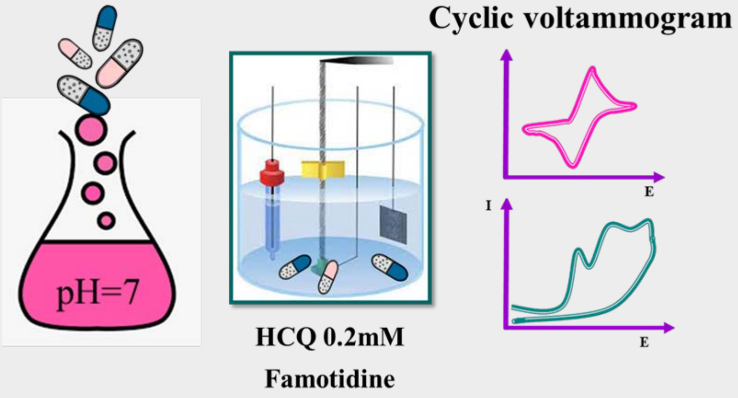

Abstract: Drug-drug interactions occur when two or more drugs react with each other. The necessity to pay attention to drug interactions has been felt more in recent years due to the prevalence of Coronavirus disease (Covid-19). This is an infectious disease caused by the SARS-CoV-2 virus. Most people infected with the virus will experience mild to moderate respiratory illness and recover without requiring special treatment. However, some will become seriously ill and require medical attention. Since Covid -19 has a viral cause, no specific drug has yet been designed for it, so the lack of coronavirus-specific antiviral drugs has led to several studies reusing previously approved drugs to treat SARS-CoV-2. Chloroquine (CQ) and hydroxychloroquine (HCQ) are synthetic 4-aminoquinolines. They are used to treat autoimmune diseases. Recently, international medical organizations have allowed the treatment of Covid -19 in some hospitalized patients with HCQ. HCQ increases the endosomal pH, which inhibits fusion between SARS-CoV-2 and the host cell membrane [1]. On the other hand, famotidine is used to treat and prevent ulcers in the stomach, but a recent clinical study showed that when high-dose famotidine was administered to hospitalized Covid -19 patients, it reduced intubation and mortality rate [2].

In this study the electrochemical behavior of HCQ in the presence of famotidine was investigated using cyclic voltammetry and controlled-potential coulometry techniques in aqueous buffer solution (pH~7). The results indicated that the peak structures and currents have changed and famotidine has reduced the HCQ current value in cyclic voltammetry. Corresponding products were electrochemically synthesized in aqueous solutions using a carbon electrode in an undivided cell. These products were identified by spectroscopic methods (FT-IR, ^{1}HNMR, ^{13}CNMR and MS) and chromatographic method (HPLC). Also, the homogeneous rate constants (k_{obs}), based on the suggested electrode mechanism were estimated by comparing the experimental cyclic voltammetric responses with those digital simulated results.

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Colorimetric Semi-Quantitative MicroRNA Detection Using AuNP-core Hairpin Spherical Nucleic Acids

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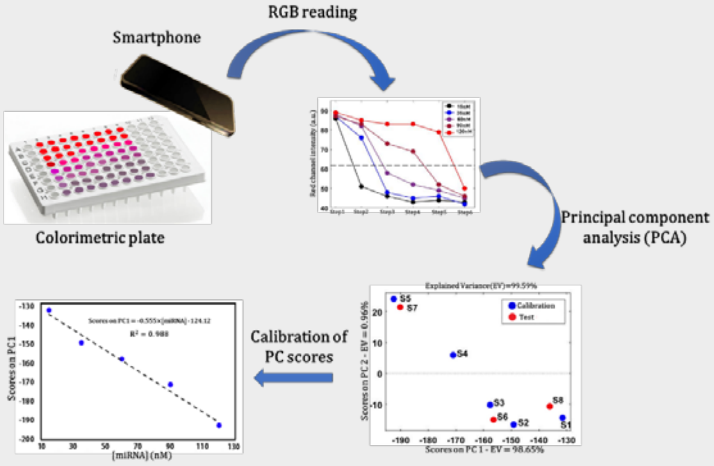
Graphical Abstract



Abstract: In recent years, increasing interest has focused on gold nanoparticle-core spherical nucleic acids (AuNP-core SNAs), in which AuNPs are densely functionalized with linear DNA or RNA [1]. AuNP-core SNAs can be programmed base by base to accurately assemble themselves, resulting in the SPR-related color change. This observable color change can provide appropriate platforms for target-responsive nucleic acid detection [2-4]. MicroRNAs (miRNAs) are a broad family of non-coding, single-stranded RNA molecules with a length of about 22 nucleotides that regulate a wide range of biological activities [5]. They have been found as biomarkers for the early identification and screening of cancer .So, MiRNA analysis is extremely useful in cancer treatment and monitoring. In this study, hairpin DNA-coated gold nanoparticles known as hairpin spherical nucleic acids (H-SNAs) are described to identify miRNA-30a as a candidate biomarker. The target miRNA induces single-component assembly of H-SNAs and provides a target-responsive colorimetric strategy. In the absence of the target miRNA a hairpin secondary structure is formed by a four-nucleotide palindromic sequence flanking the recognition loop on each side. However, the presence of the target miRNA which is complementary to the hairpin recognition loop breaks the stem-loop secondary structure, exposing the outer edge of the stem sequence (outer palindromic arm) and favoring single component assembly formation and subsequent red to blue colorimetric response. The colorimetric signal from the single-component assembly of H-SNAs was converted into measurable intensities associated to the three primary colors, red, green, and blue (RGB) using a smartphone. The data was then processed using the principal component analysis (PCA), and the first PC values were used as a calibration response and plotted against the concentration of the miRNA-30a in the samples. The obtained quantification limit was 5 nM and a linear response established for the range of 3-65 nM.

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Development of Electrospun Cellulose acetate /Polyacrylonitrile /Thymol /Mg-metal organic framework Composite Nanofibers-based Pipette-tip Micro-solid phase Extraction Coupled with HPLC-UV Analysis for the Quantification of Anti-cancer Drugs in Biological Fluids

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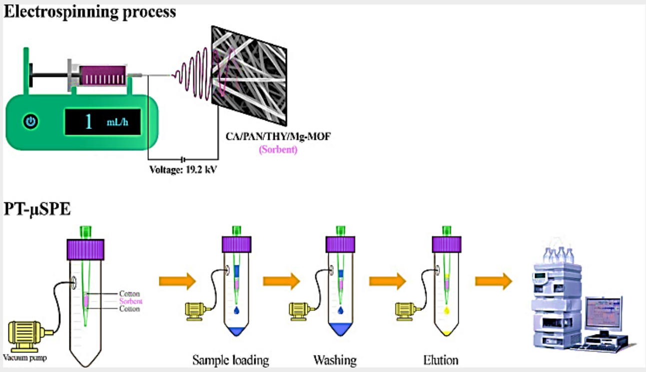
Graphical Abstract



Abstract: In cancer therapy, pharmacological drugs are utilized to avoid the growth and proliferation of cancers or metastasis. In recent years, targeted cancer therapies have become one of the dominant generations of cancer treatment. However, in this type of cancer therapy, tumor-selective drugs have not been developed yet; therefore, normal cells can be targeted as well by these cytotoxic drugs. As a result, precision dosing is conducted as a crucial step prior to prescribing such drugs in order to optimize the plasma concentration of them, and subsequently their clinical effects without altering the overall health condition of a patient. Quantifying trace levels of anti-cancer drug residues in biological fluids can be considered to provide valuable data for precision dosing [1]. In this study, a novel cellulose acetate /polyacrylonitrile /thymol /Mg-metal organic framework composite electrospun nanofiber was developed as a sorbent in pipette-tip micro-solid phase extraction for the rapid and simultaneous extraction of three anti-cancer drugs, including Letrozole, Gefitinib, and Riluzole in human biological fluids before separation and quantification by high-performance liquid chromatography-ultraviolet system. This nanosorbent was characterized by field emission scanning electron microscopy, energy-dispersive X-ray spectroscopy, X-ray diffraction, Fourier transform infrared spectroscopy, and nitrogen adsorption-desorption analysis. Incorporating polyacrylonitrile, thymol, and Mg-MOF into the cellulose acetate network confers appreciable properties to the sorbent, such as good mechanical stability, increased resistance versus water and chemicals, high porosity, and significant extraction ability. Under the optimum conditions, the linearity was achieved in the range of 0.1-1500.0 μg L^{-1} for Letrozole and Gefitinib, and 0.25-1500.0 μg L^{-1} for Riluzole with correlation coefficients ≥ 0.9996. Low limit of detections (0.03-0.10 μg L^{-1}), and limit of quantifications (0.10-0.33 μg L^{-1}) with low relative standard deviations ($\leq \mathbf{6.6}\%$ for intra-day and $\leq \mathbf{8.2}\%$ for inter-day), were also obtained. Eventually, this method was successfully used to extract the mentioned analytes from the biological samples with acceptable relative recoveries (80.4-94.4%, relative standard deviations = 5.0-8.7%).

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LC-MS/MS Method for the Quantification of Teriflunomide in Human Plasma: Application to patients with multiple sclerosis

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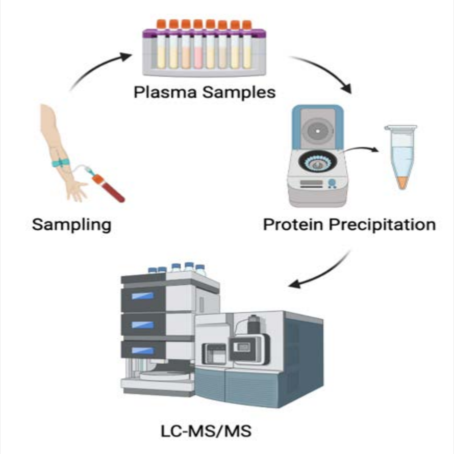

Abstract: Teriflunomide (TF) is a disease modifying antirheumatic drug of the isoxazole class which is primarily used for the treatment of rheumatoid arthritis and multiple sclerosis [1]. TF has a long half-life and the plasma clearance of this drug in plasma is very slow. Moreover, a non-teratogenic plasma concentration of TF was considered 20 ng/mL based on the animal data. Therefore, in case of safe pregnancy, the TF plasma concentration must reach below the medical decision point of 20 ng/mL through fast elimination methods [2,3]. Therefore, we aimed to develop and validate an LC–MS/MS method for the measurement of TF in human plasma. The chromatographic separation was achieved on a YMC ODS-AM column (150 mm × 3.0 mm, 3 µm) using a gradient mobile phase consisting of solvent A (water, containing 0.1% formic acid) and mobile phase B (acetonitrile: methanol: water, 18:1:1 v/v, containing 0.1% formic acid) with a run time of 12.0 min at a flow rate of 0.4 mL/min. Detection was carried out by a triple–quadrupole tandem mass spectrometer in multiple reaction monitoring (MRM) in negative mode (at m/z 269.1/160.0 and 269.1/82.0). The method has been fully validated based on the Guideline on Bioanalytical Method Validation. The weighted calibration curves were linear over the range of 4- 400 µg L^{-1} in plasma with relative standard deviations and relative errors lower than 15%. Intra- and inter-day precision and accuracy using quality control (QC) samples were within the generally accepted criteria for bioanalytical method. Method selectivity, specificity, matrix-effect, and autosampler stability also have been studied. The developed method was successfully applied to determine the TF plasma concentration in patients with multiple sclerosis who have eliminated the drug to get pregnant.

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Determination of Rhodamine 6G in Food Samples by Solid-Phase Extraction Method Using a Waste Natural Adsorbent

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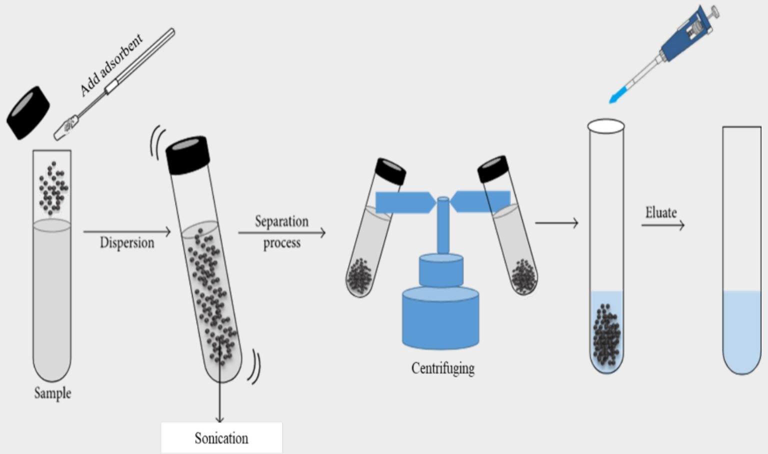
Graphical Abstract



Abstract: For many years, the food industry has utilized various types of additives such as synthetic dyes, artificial sweeteners, flavors, and preservatives in industrial products in order to improve the desired characteristics of the consumer. Using synthetic dyes in foodstuffs has been considered an earnest threat to people's health [1]. Although the use of these dyes in food is prohibited, due to easy-fast coloring, aesthetic and low-cost aspects are added to food to increase products sales. Determining the trace amount of synthetic dyes in food samples is considered one of the most important topics in analytical chemistry [2]. In this research, we employed the stone waste powder industrial (SWPI) as the adsorbent to detect and enrich trace amounts of Rhodamine 6G in food samples using the solid-phase extraction Uv-Vis spectrophotometry method. The impact of effective parameters upon rhodamine 6G sorption on SWPI was explored utilizing three levels of Box-Behnken design (pH, sorbent mass, and uptake time). The optimum condition was determined at 4.35, 0.1 g, and 45 min for pH, sorbent mass, and uptake time. Under optimum conditions; adsorption percent, linear range, the limit of detection, and limit of quantification were obtained at 98.03±1.06, 50-20000 $\mu g.L^{-1}$, 8.50 $\mu g.L^{-1}$, and 28.35 $\mu g.L^{-1}$, respectively. The enrichment factor was determined at 50 and relative standard deviations for 6 repetitions are achieved at 4.13 %. Saffron ice cream, jelly, orange juice, and tap water were employed as food samples. Finally, the results were depicted that the determination of rhodamine 6G using this extraction method is selective, sensitive, repeatable, fast, low-cost, eco-friendly, and high recoveries in the range 73.59-93.88 % from food samples.

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Determination of Trace Amounts of Aflatoxins by Dispersive Solid Phase Microextraction Using Layered Double Hydroxide Adsorbents Followed by High Performance Liquid Chromatography - Fluorescence Detector in Food Samples

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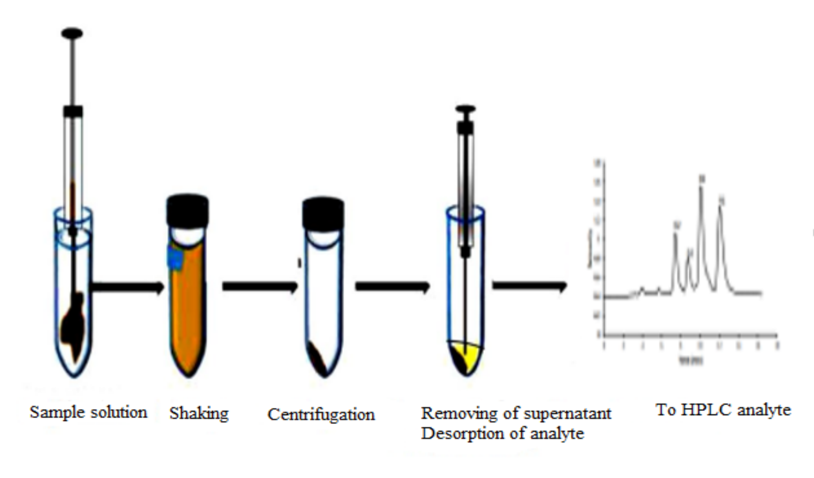

Abstract: The natural toxins of aflatoxins are produced by some kind of fungus. These fungi can grow on some plant foods derived from cereals or beans and lead to their contamination. The carcinogenicity of aflatoxin toxins has been confirmed by health authorities, even in small quantities. Therefore, it is important to measure these compounds. In this study, dispersive solid phase microextraction was used to extract and preconcentrate aflatoxin toxins from aqueous samples. As adsorbent in solid phase microextraction method, layer double hydroxide (LDH) modified with sodium dodecyl sulfonate surfactant were used. The used adsorbent, after preparation and characterization, has been used directly to extract target compounds. Final analysis was performed by liquid chromatography with fluorescence detector. Factors affecting the extraction, including the type and amount of adsorbent, solvent, sample pH and ionic strength, surfactant concentration, homogenization rate and time were investigated and optimized. Under optimal conditions, the method attained good precisions (relative standard deviation, RSD), enrichment factors and limits of detection (LODs ≤ 0.17µgL-1), S/N=3) in compared with other methods. Detection limit of 0.03, 0.08, 0.16 and 0.17 µgL-1 were obtained for aflatoxins B1, B2, G1 and G2, respectively. The reproducibility of the method was obtained based on the relative standard deviation in the range of 5 to 10%. The linear range of 0.2 to 100 µgL-1 for type B aflatoxin and 0.5 to 500 µgL-1 for type G aflatoxin and. The optimized method was successfully applied to the determination of four types aflatoxins in real samples.

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Synthesis of Polyoxometalate-rGO Composite Coated on the Inner Wall of Stainless Steel Tube as a Sorbent for Extraction of Some Selected Parabens Through Determination by HPLC-UV

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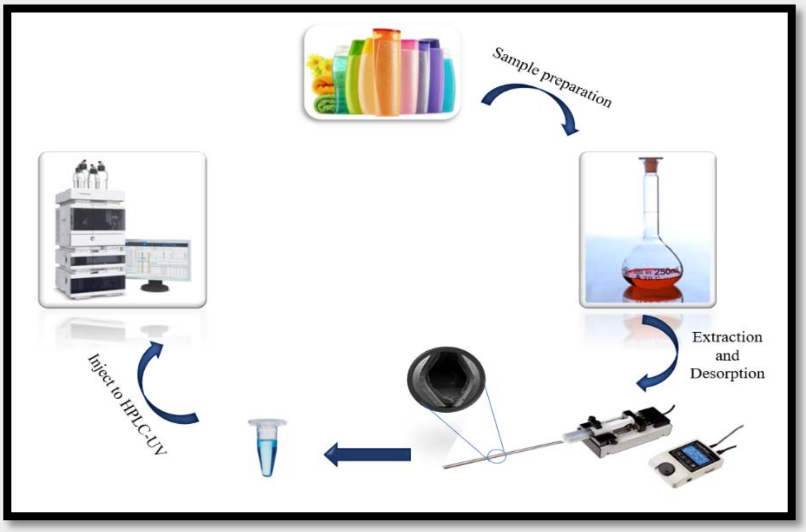

Abstract: Parabens are one of the harmful components of cosmetic products, which can affect the safety of cosmetic products. Moreover the concentration of parabens have to be controlled [1]. Therefore, an appropriate method should be developed to determine the concentration of parabens in the cosmetic samples. Here in, a new sorbent based on the combination of POMs with rGO have been synthesized. The prepared sorbent has been used for the extraction of the selected analyte. The extracted analytes were eluted by an appropriate eluent and analyzed by HPLC–UV. To perform the extraction method, the prepared sorbent has been accommodated on the inner surface of the stainless steel tube and the sample solution was passed through the sorbent. The extraction was performed by flow injection analysis method. Finally, the extracting device was used for the extraction and determination of parabens in different types of cosmetic samples. The obtained results indicated that the sorbent is appropriate for the extraction of parabens from cosmetic. Optimization of extraction factors was done using univariate optimization method. Under optimal conditions, RSDs of intra-day and inter-day standards was less than 4 % and 6.8 % for samples, respectively. This method was successfully used to determine some parabens in various cosmetic products.

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Determination of Cobalt Ions from the Environmental Samples using IL-ZIF(8) Followed by Atomic Absorption Spectrometry

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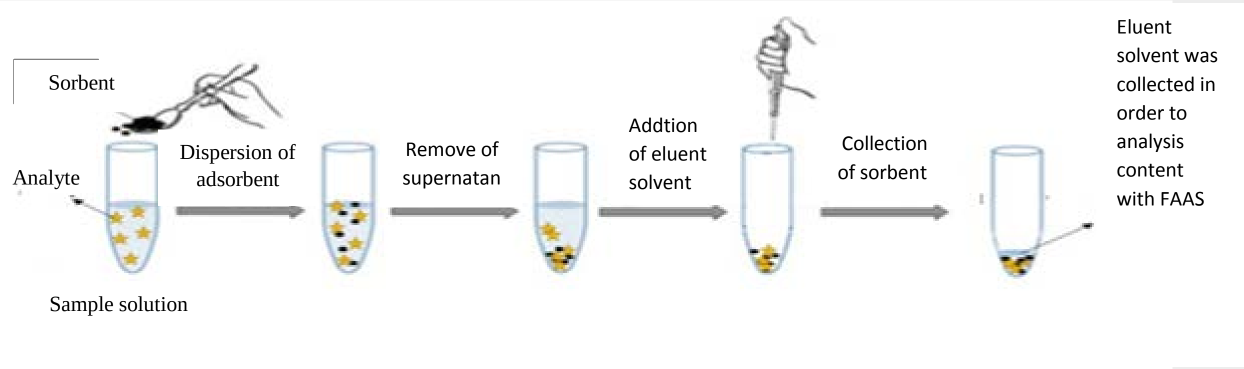

Abstract: Cobalt is widely used in various industries as alloys, catalysts, batteries, paints, drugs and ceramics, but at the same time, it is a significant pollutant of the environment because of its toxic effect on human health. Cobalt is a typical metal ion present in biological and environmental samples and has important roles in many physiological functions. However, at high levels, it can be toxic and can lead to toxic effects such as vasodilatation, cardiomyopathy, low blood pressure, and bone defects in humans and animals [1,2]. Therefore, the development of efficient, reliable determination methods for monitoring the level of cobalt concentration in natural waters is required. However, the determination of trace cobalt in biological and environmental samples is very difficult due to its extremely low concentration and the interfering effects of the matrix. Several methods have been reported for the separation and preconcentration of cobalt. A vortex-assisted dispersive solid-phase microextraction (VA-d-μ-SPE) method for separation and preconcentration of cobalt ions using IL-ZIF (8) prior to measurement by flame atomic absorption spectrometry was developed. Key parameters influencing the extraction efficiency of cobalt were examined and optimized. The optimum conditions for this method were: pH of solution: 7, vortex mixing time for sorption: 2 Minutes, amount of adsorbent, 10 mg, concentration of analyte 0.5 ppm; type and concentration of eluent, HCl and 0.01 mol L^{-1}, volume of eluent: 4mL; the limit of detection and relative standard deviation for the detection of cobalt ions were found to be 2.42 ppb and 0.75%, respectively. Finally, the developed method was successfully used for extraction and determination of cobalt ions in water, vegetable, and soil samples.

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Extraction of Co(II) from Thiocyanate Solutions Using Polymer Inclusion Membranes Composed of Cellulose Triacetate as the Base Polymer and Trioctylamine as Extractant

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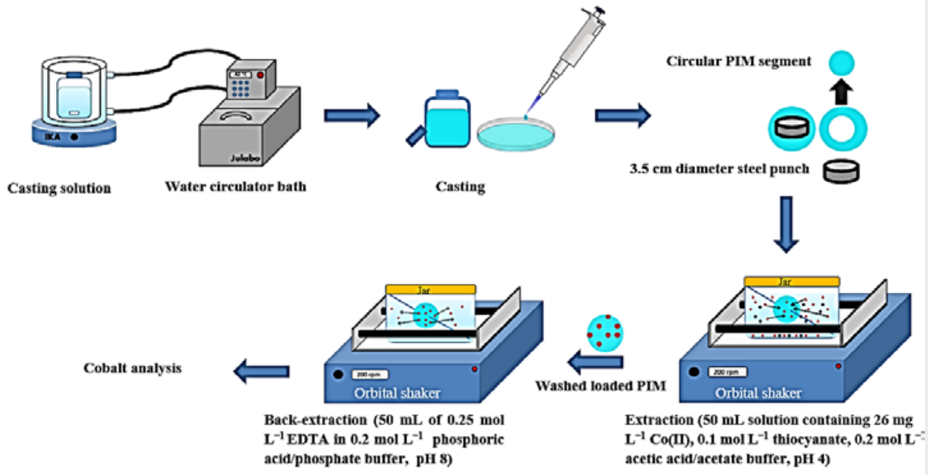

Abstract: Polymer inclusion membranes (PIMs) has oppened a new horizon for introducing new strategies for developing the environmentally friendly membrane based techniques for the separation-recovery of many environmentally and economically important metals [1]. They are known as alternatives for the solvent extraction techniques, in which the consumption of great amount of toxic, expensive, and flammable organic dilunets is indispensable. The characteristics of PIMs can be tuned by varying its consituents i.e. the base polymer (homopolymers, copolymers, polymers blends, and cross-linked polymers [2] and the extractant. In some cases, the type and the amount of the plasticizer used affects also the PIMs chracteristics [3]. First, in the present project, the optimal polymer inclusion membrane with suitable physical, mechanical and extraction properties including a combination of 40% w/w cellulose triacetate (CTA) polymer, 25% w/w trioctylamine (TOA) extractant and 35% w/w tributyl phosphate (TBP) plasticizer was selected. the membrane was capable to extract completely Co(II) from 50 ml of solution containing 26 mg L-1 Co(II) in 0.1 M ammonium thiocyanate solution and 0.2 M buffer acetic acid/acetate at pH=4 over time 2 hours and back extraction of Co(II) from 50 ml of solution containing 0.2 M buffer phosphoric acid/phosphate at pH=8 with 0.25 M EDTA for 16 hours. The selectivity of the investigated membrane toward Co(II) was evaluated by performing competitive extraction experiments of Co(II) in the presence of various cations and anions. The application of real aqueous samples and nickel-cadmium battery sample leached were investigated. The study to determine the extracted species confirmed the mechanism of ion pair formation $(TOAH+)_2(Co(SCN)_4)$. The maximum capacity of the membrane made in cobalt extraction is 19.75 mg of cobalt ions per gram of membrane.The kinetic and isotherm studies revealed that the sorption of Co(II) by the optimized membrane is a adsorption and chemisorption process.

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Dispersive Liquid-Liquid Microextraction Followed by Ion Mobility Spectrometry for Determination of Antifungal Drug: Optimization Using Response Surface Methodology

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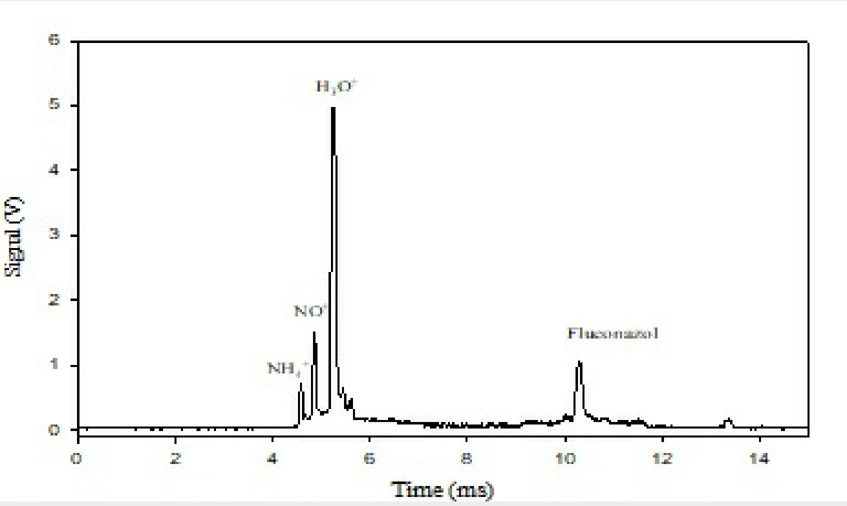

Abstract: Fluconazole is an antifungal drug, which is widely used in the treatment and prevention of manifold fungal infections. Determination of fluconazole in the different real samples can be essential, particularly in environmental studies [1]. Therefore a fast and sensitive analytical method can be very helpful. Ion mobility spectrometry (IMS) has almost all the features [2]. On the other hand, a fast and efficient microextraction method can obtain a lower detection limit and better cleanup in real samples. Consequently, in this study, a dispersive liquid-phase microextraction, assisted by a homogenizer, combined with ion mobility spectrometry has been applied for the determination of fluconazole. Figure 1 reveals the ion mobility spectrum of fluconazole. To find the optimization conditions and evaluate the proposed method, the response surface methodology (RSM) was employed. The optimum conditions were determined for the aqueous sample of 56 µl of dibromomethane and toluene with a volume ratio of 70:30 as the extraction solvent, sample solutions pH of 10, 9.5 w/v% of NaCl for adjusting the ionic strength, dispersing rate of 16000 rpm, and the homogenizing time of 4 s. The analytical performances of the method for this analyte were also studied. The method was linear in the range of 5 to 100 µg/L. Enrichment factor for the method was 545.7. The detection limit of the method was equivalent to 2.3 µg/L and the intra- and inter-day relative standard deviation were 4.3% and 4.5% respectively. The suggested method was investigated for the determination of fluconazole in real samples such as river water, and the relative recoveries for studied samples were obtained between 98 and 106%.

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Extraction of Methylene Blue (MB) from Water Solutions by a Poly(Vinyl Chloride) Based Polymer Inclusion Membrane Incorporating Bis (2-ethylhexyl)Phosphoric Acid As the Extractant

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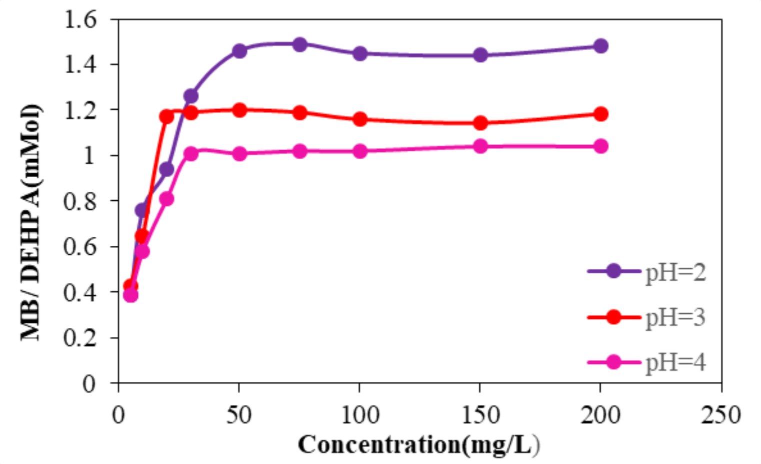

Abstract: Polymer inclusion membranes (PIMs) have attracted the attention of many researchers around the world due to their high capability in the process of extraction, separation and removal of metal ions and also having special privileges compared to other liquid membrane methods [2]. The present paper reports on the first application of a polymer inclusion membrane (PIMs) for the extraction of Methylene blue (MB). PIMs containing of 50% w/w poly(vinyl chloride) as the base polymer and 50% w/w bis(2-ethylhexyl)phosphoric acid (D2EHPA) as the extractant. The optimized PIM was able to efficiently extract 92% of MB from 50 ml water solution containing 20 mg/l^{-1} (MB) at pH=3 after 2h. Back extraction of MB from 50 ml of solution containing 0.15 mg/l^{-1} oxalic acid over time 2h. To achieve the highest efficiency, the parameters affecting the extraction process were investigated and optimized. These parameters were time, effect of shaking rate, pH, real sample, stability of the PIM, extraction stoichiometry. The investigated PIM exhibited excellent performance stability in 11 extraction/back-extraction cycles. The membrane was used for recovery of MB from some real samples. [1, 2]

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Bionanocomposites based on Halloysite Nanotubes-Zein and Evaluation of Their Performance for Thin Film Microextraction

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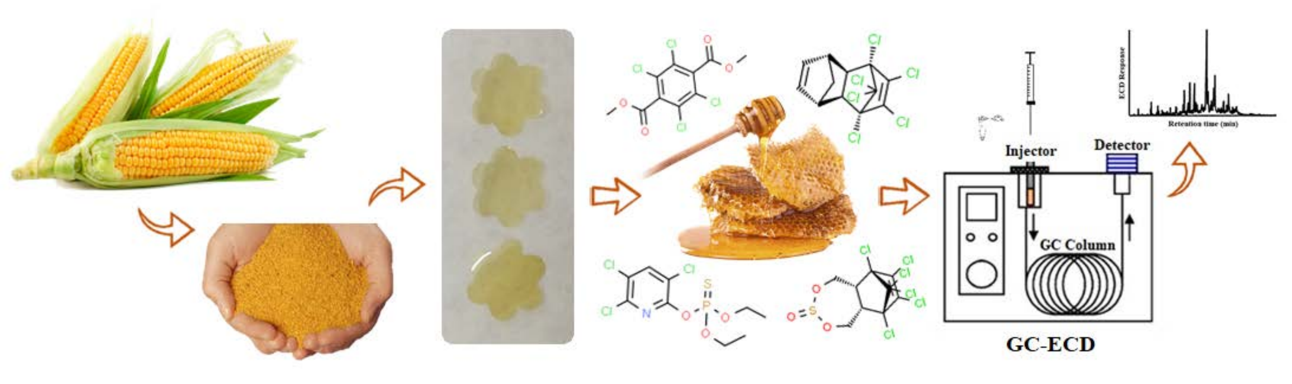

Abstract: In this work, a biocompatible sorbent based on halloysite nanotubes-zein bionanocomposite for direct-immersion thin film microextraction (DI-TFME) is introduced. Zein as a biodegradable, low cost and accessible polymer was used to prepare a flexible thin film with the possession of different interactions and good adsorption capacity. To enhance the mechanical and chemical stability of the film, halloysite nanotubes were added to the zein. The film was used for TFME of 16 pesticides from different chemical classes, including organochlorines and organophosphates followed by gas chromatography with electron capture detector (GC-ECD). To achieve the maximum efficiency and sensitivity, effective parameters such as the amount of halloysite nanotubes in bionanocomposite, type and volume of desorption solvent and desorption and extraction times were investigated and optimized. The linear dynamic range of the method was in the range of 0.1-300 μg L^{-1}. The limits of detection and limits of quantification of the method were in the range of 0.01-0.04 μg L^{-1} and 0.03-0.12 μg L^{-1}, respectively. The relative standard deviations for three replicates at the concentration levels of 5 and 50 μg L^{-1} were in the range of 3–12%. The method was validated for the TFME of pesticide residues in honey samples and recoveries for the spiked samples at two concentration levels of 5 and 50 μg L^{-1} were found to be in the range of 79-118%. The analytical results of the present method were comparable with some relevant methods currently used for the determination of pesticide residues. Besides, the prepared sorbent was environmentally friendly, easy to prepare, and inexpensive.

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Application of Organic Gas Steam- Liquid Extraction System for Extraction and Separation of Uranium from Water Samples as a New Efficient Method

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Abstract : In this study, for the first time the Organic Gas Steam-Liquid Extraction (OGS-LE) method is used as a simple, efficient and scalable to industrial application technique for the extraction and separation of uranium (VI) from aqueous samples. OGS-LE is done by a special handmade extraction cell. In this method, the organic solvent vapor produced in the evaporator unit is introduced into the aqueous sample by using nitrogen as a carrier gas. By inserting the vapor bubbles of the organic solvent into the aqueous sample, the organic solvent dissolves in water and the organic solvent concentration in water reaches supersaturation. During this process, equilibrium occurs between the dissolved organic solvent and the insoluble organic solvent, and it is collected on top of the aqueous phase. Uranium has been extracted with cyanex 272 and tetrabutylammonium bromide (TBAB) as extractant into n-heptane from the alkaline aqueous media by the OGS-LE method. Cyanex 272 and TBAB were used as the complexing ligand and the ion pairing reagent, respectively. The mechanism of extraction was proposed depending on the deprotonating of cyanex 272 and ionic interaction with the quaternary ammonium bases. Face Central Composite Design (FCCD) was used to evaluate the effect of various factors. Under the optimized conditions, uranium extraction could be completed in a single stage with the extraction efficiency of more than 90% from an aqueous solution containing alkali, alkaline earth and transition metal ions. The precision, obtained by performing five replicates under the optimized conditions, was 90.12 % ± 0.75 % (percentage of extraction ± RSD).

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Electrodeposition of Zn-Al-LDH intercalated with Ibuprofen on the Surface of the Graphite Pencil Substrate for Fiber Solid Phase Microextraction of some Nonsteroidal Anti-inflammatory Drugs and their *Determination by HPLC*

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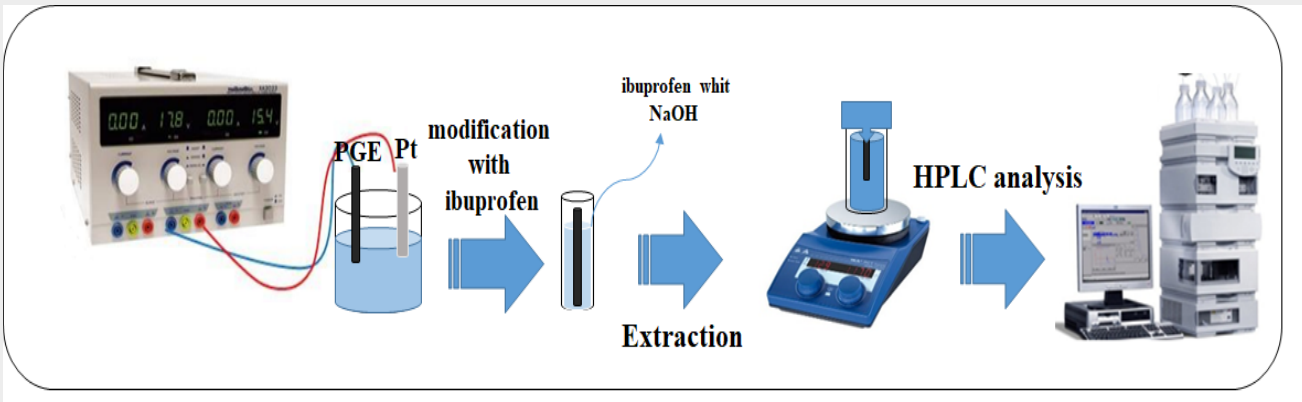
Graphical Abstract



Abstract: The main aim of the present study is the in-situ electrodeposition of Zn-Al-LDH on the surface of the conductive graphite pencil substrate together with the modification with ibuprofen. For this purpose, the electrodeposition method was employed using two electrode systems in the constant potential to produce Zn-Al-LDH on the surface of a pencil graphite substrate as working electrode. The electrochemically prepared Zn-Al-LDH will then be modified by ibuprofen [1]. The prepared modified adsorbent was used as the extracting fiber in the solid micro phase extraction (SPME) of selected drugs such as aspirin and naproxen from urine samples. The extracted analytes were eventually injected into the HPLC-UV for quantitative measurement and determination. In order to reach the maximum efficiency and obtain the optimal conditions for extraction of the selected analytes, the response surface methodology (RSM) was used. Based on the obtained results and the small RSD% (6.7%) for the method presented, it can be concluded that this absorbent has a high ability to pre-concentrations and selective extractions of testing analytes in biological samples.

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Simultaneous Synthesis of Dual LDH/Cobalt Oxide Sorbents on Pencil Graphite Substrate for Using in Solid Phase Microextraction of Some Environmental Pollutants and Quantitative Measurement via HPLC

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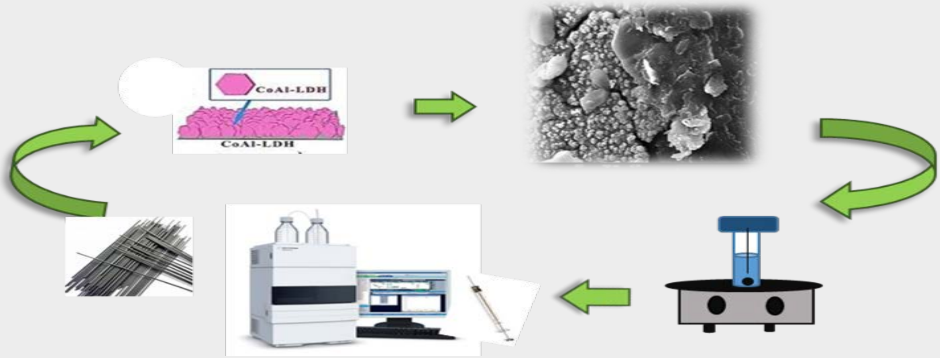

Abstract: Todays, toxic compounds such as toluene, benzene and phenolic compounds are known as carcinogens, which the presence of these compounds, even in low concentration, in the groundwater aquifers is dangerous and these types of pollutants are highly toxic and carcinogenic according to the WHO regulations [1]. In this context, microextraction methods can be used to pre-concentrate the desired compounds [2]. One of the most interested technique, which is used for the extraction of analytes is solid phase microextraction (SPME) [3], which is considered as a suitable method for pre-concentration and extraction of experimental species.

Recently, layered double hydroxide (LDH) and Co_3O_4 have solely been used as sorbents in the extraction phases in the SPME method. The mentioned sorbents have shown incredible properties for the extraction of some analytes. The main objective of the present study is to simultaneous synthesis of the both LDH and Co3O4 on the same time on the surface of a very cheap pencil graphite substrate. By using this sorbent, hydrogen bonding and the anion exchange as two different forces were lead to extraction of the analytes, which caused the selectivity of the method.

Herein, the LDH was accommodated on the surface of the pencil graphite electrode by the electrochemical method, and then, some part of the LDH were changed into the Co_3O_4 by thermal method. Finally, this fiber, which is containing the two adsorbents was used for the extraction and measurement of some of the desired species, including benzene, toluene and some chlorophenols in drinking water samples. The extracted compounds were finally eluted with an organic solvent and injected into the HPLC apparatus for measurement. Based on the obtained results, RSDs were higher than 5.6 % and for BTEX compounds were better than 5.8 %.

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From waste to health: Application of MOF modified walnut biochars for determination of twenty pesticides in wheat flour by ultra-high performance liquid chromatography-tandem mass spectrometry

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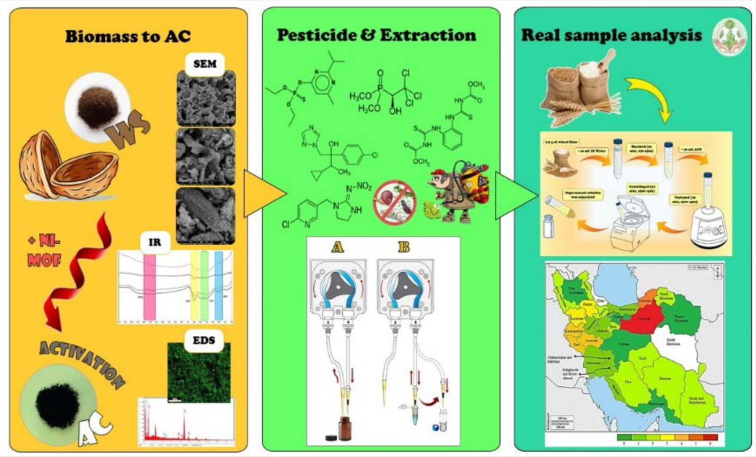

Abstract: Pesticides as an unavoidable part of crop cultivation have grown a great concern regarding food safety and wheat as one of the most important grains with high per capita consumption needs to be studied for possible contamination. In this research, walnut shell as high potential biomass has been used along with Ni-metal organic framework (Ni-MOF) and thermally treated to obtain high efficient biochar with application in pesticide detection. The wheat specified pesticides with a good range of polarity (0.5-4.4 Log P range) have been detected using the optimized walnut modified biochar as the sorbent and pipette tip-µ-solid phase extraction (PT-µ-SPE) method as the extraction setup. Modified biochars were characterized using regular methods and the role of Ni-MOF in the enhancement of obtained biochars was justified. In the following, the extraction setup parameters were optimized and the final method with 50%-Ni-MOF-walnut shell biochar as sorbent was evaluated for precision, accuracy, and other figures of merit. Accordingly, the precision of the method was evaluated by RSD values obtained for intraday, interday, and synthesis procedure for all twenty pesticides in the wheat matrix. The limit of detection (LOD) and quantification (LOQ) was also evaluated. Consequently, the method was applied for the analysis of 29 wheat samples collected from all over the country and was able to find several pesticides which were compared individually with the maximum residue limit (MRL) defined by the European pesticide database. As a result, a map illustrating the wheat pesticide analysis was obtained and a risk assessment study has been performed on the acquired results. The risk assessment study demonstrated that target pesticides in analyzed wheat samples cannot cause non-carcinogenic harm to adults but might have an impact on children as more vulnerable consumers.

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Improving the Separation of Thorium and Uranium Ions from Wastewater by Impregnating Amberlite XAD-4 Resin

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Graphical Abstract

Abstract: Thorium and uranium are one of the most important heavy metals because of the chemical toxicity and radioactivity. Excessive amounts of thorium and uranium have entered into environment through the activities of nuclear industry. The toxic nature of thorium and uranium ions, even at trace levels, has been a public health problem for many years. For this reason, removal of thorium and uranium ions from wastewater is of great importance [1]. The solid phase extraction technique is now routinely used in various research and application areas due to its characteristics and advantages over the classical solvent extraction or other pre-concentration techniques. Chelating resin sorption method is one of the most effective multielement pre-concentration methods. In this study, Amberlite XAD-4 resin for improve the separation and removal thorium and uranium ions from nuclear waste was investigated and in view of this point that the adsorptive properties of adsorbent can be improved [2, 3], impregnation of the XAD-4 surface by complexing agent bis(2-hydroxybenzaldehyde)-1,2-ethylendiimine was done. The results show the adsorptive properties of adsorbent can be improved by impregnation of the surface with complexing agent. The procedure was based on the adsorption of thorium and uranium ions on Amberlite XAD-4 resin loaded with a Schiff base prior to their determination by Inductive Coupled Plasma spectrometry. A comparison of the adsorption efficiency of the studied resin loaded with a Schiff base with those unloaded ones shows a shift for uptake of the metal ions vs. pH curves towards lower pH the values by applying the loaded resin with Schiff base. Also, $\Delta pH_{0.5\ Th/U}$ increases from 0.2 to 1.4 and a selective separation occurs for the thorium ion. The effect of parameters influencing such as aqueous phase pH, contact time and adsorbent dosage was investigated. Under optimal conditions (pH=5, adsorbent amount 0.1 g, contact time 45 min.) for thorium ions and (pH=6, adsorbent amount 0.1 g, contact time 60 min.) for uranium ions with initial concentration 20 mg/L were quantitatively removed from 20 mL of the sample solution (99.28% for thorium and 97.35% for uranium). The kinetic data corresponds well to the pseudo-second-order equation. This model predicts chemisorption for the adsorption. The adsorption data for studied ion were well fitted by the Frundlich isotherm.

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The Combination of Dispersive Liquid-Liquid Microextraction Method with Thin Film Evaporation for Extraction of Rivastigmine Drug in Biological Samples before Analyzing by Secondary Electrospray Ionization-Ion Mobility Spectrometry

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Abstract: The combination of dispersive liquid-liquid microextraction method with diffusive thin film as a surface for evaporation of solvent was used to extract and preconcentration of rivastigmine drug. Secondary electrospray ionization-ion mobility spectrometer apparatus was applied to identify of rivastigmine molecules. The thin film used in this method was prepared using electrospinning method. To measure the trace amount of rivastigmine and achieve the highest extraction efficiency, some effective parameters such as type of extraction and disperser solvents, volume of extraction and disperser solvents, centrifugation time, solvent evaporation time from the film surface and sample pH were studied. Type of extraction solvent; chloroform, type of disperser solvent; methanol, volume of extraction solvent; 90 microliters, disperser solvent volume; 1.5 ml, evaporation time from the film surface; 1 minute, centrifugation time; 5 minutes and the pH of the sample solution; 10 was obtained as the optimized conditions. Under the optimized conditions, the limit of detection (LOD) and linear dynamic range (LDR) were obtained 3 μg L^{-1}, 10-1000 μg L^{-1}, respectively. The intra- and inter-day relative standard deviations were calculated to be 4% and 6%, respectively. The capability of the proposed method for analyzing of rivastigmine drug in the urine, saliva, and plasma samples were investigated and the relative recovery values were calculated 101-113%.

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Extraction of some Antibiotics in Biological Samples via Combination of Fiber Solid Phase Microextraction based on Electrosynthesized Zn-Al-Layered Double Hydroxide and Three-Phase Hollow Fiber Liquid-Phase Microextraction and their quantification by High-Performance Liquid Chromatography

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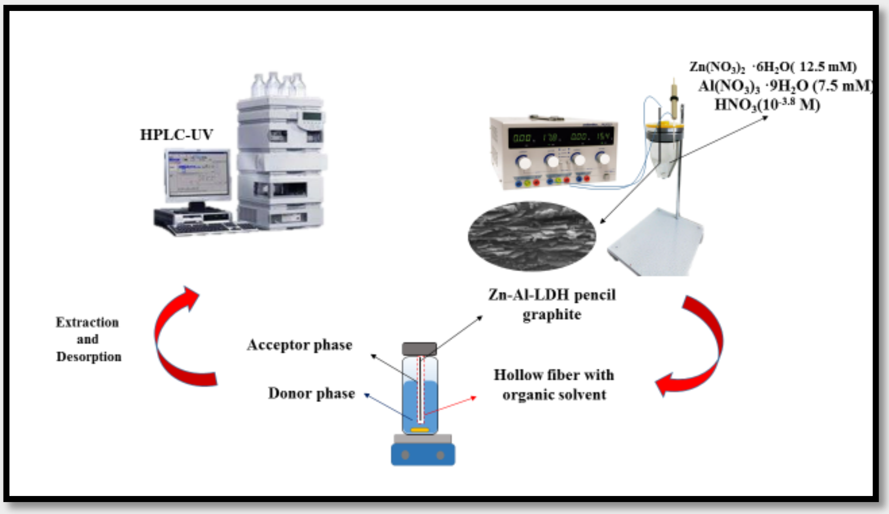
Graphical Abstract



Abstract: In the current study, solid phase microextraction (SPME) and hollow fiber three phase liquid microextraction (HF-LLLME) techniques have been combined to improve microextraction methods. The selective anion-exchange Zn-Al-layered double hydroxide (Zn-Al-LDH) as a fiber SPME has been electrodeposited on the surface of pencil graphite substrate [1]. The electrosynthesized Zn-Al-LDH sorbent was characterized by different methods such as Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), thermal gravimetric analysis (TGA) and then put into the lumen of HF which was filled by alkaline solution. The HF is utilized for achieving the excellent sample clean-up by preventing diffusion of interferences [2-3]. The pores of polypropylene hollow fiber are impregnated by an appropriate organic solvent. To obtain the high extraction efficiency, different parameters were optimized through response surface method. In the proposed method, HF-LLLME-SPME configuration was applied for extraction and determination selected antibiotics including amoxicillin, ciprofloxacin and cefixime in urine samples. The extracted antibiotics were analyzed through high performance liquid chromatography-ultraviolet detection (HPLC-UV). Under optimal conditions, the inter-day and intra-day RSDs were determined between 3.2-4.7%. The results confirmed the presented method would be useful for the analysis of different types of drugs in complicated biological samples.

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Application of Ferocarpholit Nano-Crystal as a Natural Adsorbent for Removal of Th(IV) and Eu(III) Ions from Aqueous Solution by Batch Process

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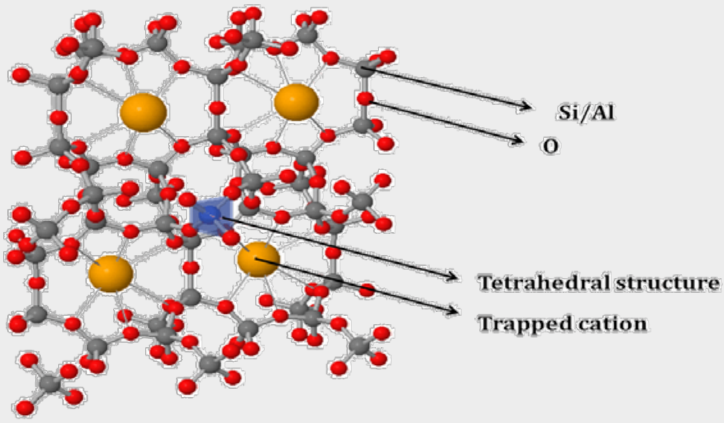

Abstract: Radioactive waste has been created by humans as a by-product of various endeavors since the discovery of radioactivity. Radioactive pollutants can be dangerous and it takes many years until radioactive substances are no longer considered dangerous. Therefore, to protect human health and the environment from hazards caused by radioactive waste and to avoid any undue burden to future generations, radioactive waste needs to be managed responsibly [1]. Zeolites due to easy access and high resistance against radiations for separating radioactive compounds of nuclear waste are used. Zeolites are crystalline hydrated aluminosilicates. Their structure is built up of tetrahedral SiO_4 and AlO_4 units bridged by oxygen atoms generating secondary building units. The trivalent aluminum in the structure generates a negative charge in the framework that imparts the ion exchange properties to the material [2-3]. There are great resources of natural zeolite in Iran. In this study, first, Ferocarpholit in rock form was supplied from Tabriz mines and were characterized by X-ray diffraction spectrometry (XRD), X-ray fluorescence spectroscopy (XRF), infrared spectroscopy (IR) and scanning electron microscopy (SEM) methods. Then, the adsorption behavior of Th(IV) and Eu(III) ions from aqueous solutions was studied. The adsorption efficiencies of the sorbent were found to be pH dependent. The maximum adsorption of Th(IV) and Eu(III) ions was obtained at pHs 4 (99.85%) and 5 (25.05%), respectively. Selective separation of these ions can be achieved by controlling the aqueous phase pH. However, the studied zeolite was shown to be an adsorbent with lower adsorption percentage towards Eu(III) ions. The other parameters such as the initial concentration of studied ions, contact time, mass of sorbent and temperature have been investigated. It is found that under optimum conditions i.e. pH=4, 10 min. by 0.01 g of the adsorbent, for Th(IV) ions, and pH=5, 60 min., 0.12 g of the adsorbent for Eu(III) ions, the metal ions can be quantitatively removed from 20 mL aqueous solutions containing 20 mgL^{-1} of metal ions. The results show that Th(IV) ions in less time and with lower adsorbent dose has the highest adsorption compared to Eu(III) ions. The experimental data showed a good agreement with second-order equation and Freundlich isotherm for studied ions.

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Comparison of absorption properties of Iranian natural zeolites in removal of Eu^{3+} ions from aqueous solution

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Abstract: Extraction and recovery of lanthanide metals from nuclear and metal-containing industrial waste streams is important both environmentally and economically. Otherwise, the consequence of the presence of such waste in the environment generates damaging effects on human societies and the earth's environment. Therefore, there is much interest concerning on the separation of these metal ions [1-2]. Zeolites are valuable inorganic crystalline materials, which are porous having wide variety of industrial applications due to their unique properties including molecular sieving, ion-exchange and catalytic behavior. Considerable attention has been paid on utilizing zeolitic nano-particles because their advantages over conventional micron sized materials. Studies have been shown that the particle size and morphology of the zeolitic materials play an important role in their properties and efficiencies as catalysts, adsorbents for separation scopes [3-4]. There are numerous natural zeolite minerals in Iran. In this communication we report the adsorption properties of clinoptilolite (a type of natural zeolites in Sabzevar and Semnan regions) and ferrocarpholite (a type of natural zeolites in Tabriz region) in removal Eu^{3+} ion from aqueous solution. The adsorption efficiencies of the sorbents were found to be pH dependent. The maximum adsorption of Eu^{3+}ion was obtained at pH 5 (25.05% for Tabriz, 55.18% for Semnan and 78.68% for Sabzevar). The selectivity of adsorption varies as Sabzevar>Semnan>Tabriz. However, the Tabriz zeolite was shown to be an adsorbent with lower adsorption percentage towards Eu^{3+} ion. In order to improve the adsorptive properties of zeolits, modification of the zeolite's surface by bis(2-hydroxybenzaldehyde)-1,2-diaminoethane ligand has been done. It is noteworthy that with the modification of nano-crystalline zeolite's surface, the uptake of metal ions significantly increase to 88.65%, 85.09% and 97.97% by adsorbents Tabriz, Semnan and Sabzevar, so that in the optimum conditions pH=5, 60 min. by 0.05 g of the adsorbent, Eu^{3+} ions can be quantitatively removed from 20 mL aqueous solutions containing 20 mgL^{-1} of metal ions in compared to the unmodified zeolites.

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Direct Immersion SPME Sampling of Phytohormones in Plants using an APTES-Chitosan Sorbent Followed by HPLC-UV Quantification

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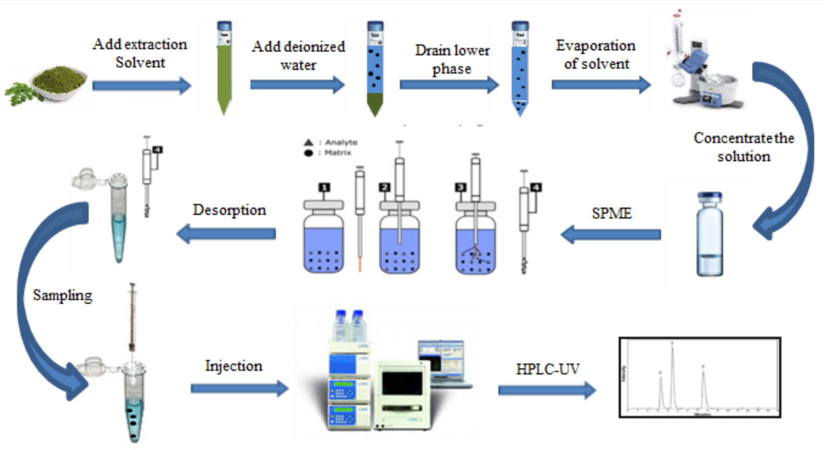
Graphical Abstract



Abstract: Herein, a simple technique for the extraction and quantification of the phytohormones of gibberellic acid (GA), indole-3-acetic acid (IAA), and abscisic (ABA), in plant samples is described [1]. A hybrid gel biopolymer, 3-aminopropyltriethoxysilane-chitosan (APTES-CS), was synthesized and coated on a activated titanium substrate and used for direct-immersion solid-phase microextraction (DI-SPME) of the phytohormones. The analytes were separated and determined using RP-HPLC after desorption from the fiber. Structural characteristics and surface morphology of the APTES-CS sorbent was studied using Fourier transform infrared spectroscopy (FT-IR) and scanning electron microscopy (SEM). Experimental variables affecting on the extraction efficiency were optimized through response surface methodology through a Box-Behnken design (BBD). The limits of detection were obtained 0.1 mg/L for GA and ABA, and 0.05 mg/L for IAA. Calibration graphs were linear over the range of 0.5 - 200 mg/L. The relative standard deviations (RSDs, n = 6) for intra-day and inter-days repeatability were obtained 4.32 - 6.3% and 1.31 - 7.33%, respectively. The developed technique was successfully used to analyze phytohormones in plant samples with average recoveries over the range of 80.9 - 118.17%. Activation of the fiber substrate created plenty of superficial reactive groups which ensured strong bonding between the substrate and the hybrid gel biopolymer. This fact led to an increased surface porosity, enhanced sorption capability, and high coating durability compare to the reported commercial and homemade fibers, employed for the DI-SPME sampling [2-4]. Fiber SPME coated with biopolymer APTES-CS used in this work showed high performance and low detection limit which makes it a simple, reliable, fast and cost effective method to determine phytohormones in the plant.

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Ultrasonic assisted dispersive solid phase microextraction of triazole fungicides from food samples using cellulose-based nanosorbent

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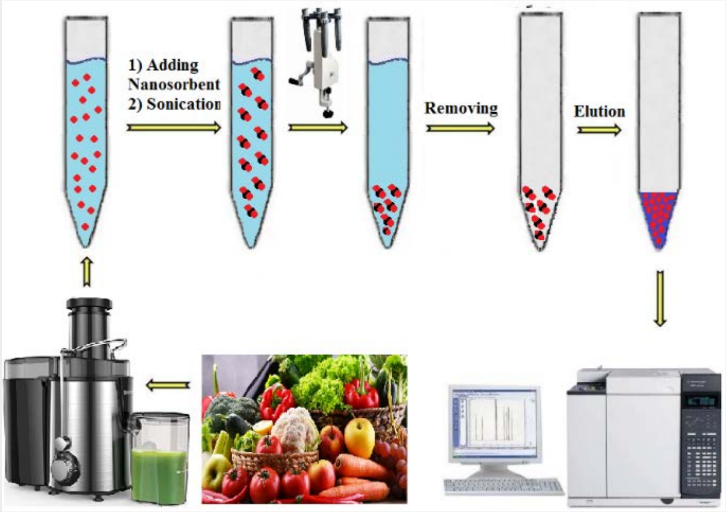
Graphical Abstract



Abstract: Herein, an ultrasound-assisted dispersive solid phase microextraction (UA-DSPME) approach has been described for trace level analysis of triazole fungicides in real samples. For this purpose, a new nanosorbent was prepared through modification of carboxymethylcellulose biopolymer with zinc-based metal-organic framework and graphene oxide, and fully characterized. Then, the effect of extraction parameters on extraction efficiency was optimized for the microextraction process. Finally, desorbed triazole fungicides with ethanol were determined using gas chromatography equipped with flame ionization detector. This technique provided good linearity (R2> 0.99), low detection limits (0.3 – 1.5 ng mL−1), high preconcentration factors (419 – 426), relative recoveries (91.6 – 102%), and high repeatability (RSD < 4.1%) at optimized conditions (amount of sorbent: 15 mg; pH of solution: 7.0; and extraction time; 4 min). Ultimately, this approach was applied to determine triazole fungicides in different water and food samples.

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Green Sample Preparation Based on Directly Suspended Droplet Microextraction Technique Using Deep Eutectic Solvent for Determination of Ultra-Trace Amounts of Titanium Dioxide in Drug Samples

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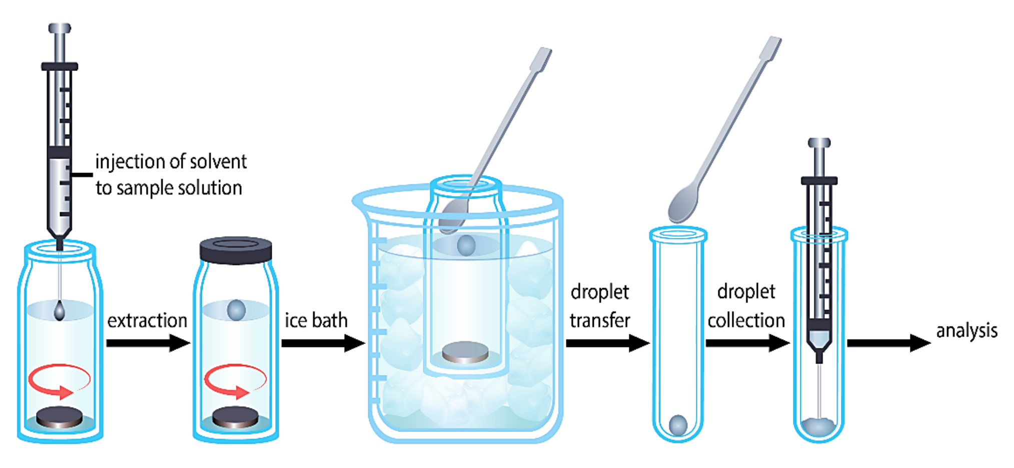

Abstract: Deep eutectic solvents (DESs) are green solvents that are known as sustainable solvents [1]. They are usually synthesized from non-toxic compounds that are biodegradable and biocompatible. Therefore, the purpose of the presented method was to develop a green, simple, quick, low-cost, and innovative method base on DSDME-SFOD microextraction by using a deep eutectic solvent for preconcentration of titanium dioxide from drug samples. In the present study, a simple and efficient directly suspended-solidified floating organic droplet microextraction (DS-SFOD) by using deep eutectic solvent (DES) has been proposed for extraction and determination of ultra-trace amount of titanium dioxide by spectrophotometric Uv-Vis. the capability of preparing deep eutectic solvent-based on tetra butyl ammonium bromide/acetic acid was considered in combination with the DSDME-SFOD microextraction method. The predominant experimental factors such as type and amount of deep eutectic solvent, extraction time, salt concentration, and temperature that affect the extraction efficiency of titanium were considered and optimized. By performing the process in optimal conditions, the calibration curves were linear in the range of 100 - 1000 ng/l. The limits of detection were 50 ng/l, respectively. The relative standard deviations were between 1-1.5% (n=6). The proposed method was successfully applied to drug samples. At the different spiking levels, recovery values ranged between 89 to 102% for drug samples that which indicates the negligible effect of the matrix on the extraction process [2].

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Novel magnetic nano sorbent modified with tannic acid for micro magnetic solid phase extraction of herbicides and determination by corona discharge ion mobility spectrometry

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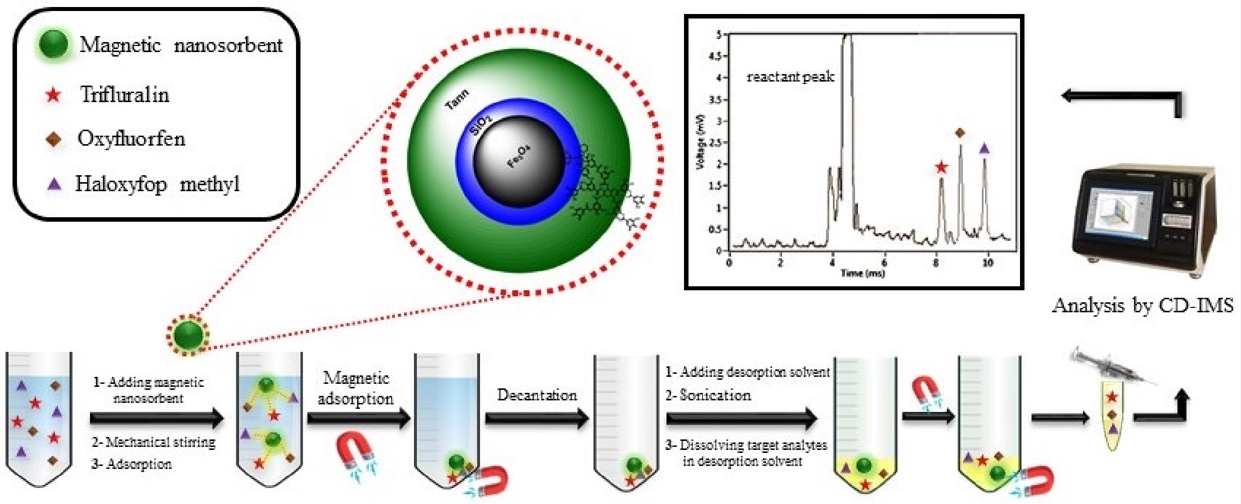

Abstract: Design and constructing proper magnetic silica core shell nanoparticles modified with tannic acid are coupled with corona discharge ion mobility spectrometry for high efficiency extraction and determination of trace amount of herbicides. Several techniques such as FT-IR, SEM, DLS, VSM, XRD and EDX have been used to verify the structural characterization of the magnetic nano sorbent. Micro magnetic solid phase extraction followed by corona discharge ion mobility spectrometry, μ-MSPE-CD-IMS was applied as low-cost, rapid, sensitive, and selective procedure for detection and quantitation of haloxyfop-methyl, oxyfluorfen and trifluralin in garlic, onion, soy bean, soil and agricultural waste water samples. The effective parameters of the procedure were optimized; Sorbent amount 5 mg, extraction time 20 minutes, desorption agent methanol, volume desorption agent 50 μL, desorption time 2 minutes, pH 7, salt addition 0% and temperature of solution 20C were achieved. LOD, RSD, LRD, and extraction recovery of the procedure were obtained (0.45-0.61 ppb), (<5.62%), (1.5-100 ppb), and (92-98%), respectively.

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Vortex-Assisted Dispersive Liquid-Liquid Microextraction Followed by Ion Mobility Spectrometry Analysis for Determination of Spirotetramat

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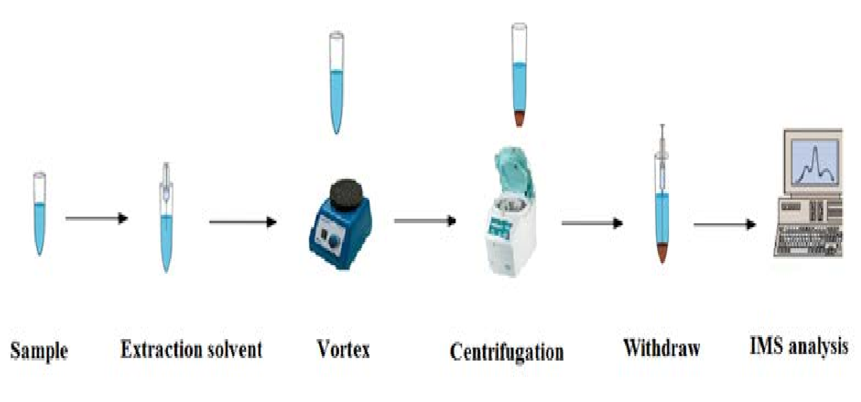

Abstract : Spirotetramat (SPT), as a new pesticide belonging to the cyclic ketoenoles displays a two-way systemicity property and can be easily transported within both the xylem and phloem. This insecticide is extensively used for controlling a broad spectrum of sucking insects such as pistachio psylla. However, the accumulation of SPT in environmental and food samples causing damages for human and animals [1-3]. Therefore, simple, rapid and sensitive techniques are needed to extract and measure SPT from different complex matrix. Ion mobility spectrometry (IMS) is a simple, cost- effective, rapid and high sensitive technique widely used in monitoring of different compound such as explosives and environmental pollutants. Thus, in the present study, a combination of vortex-assisted dispersive liquid-liquid microextraction method and IMS technique was used to extract and detection of SPT in water and food samples. In order to enhance sensitivity factors affecting the extraction process such as pH, reaction time, volume of extraction solvent were optimized. Under the instrumental optimum conditions, the proposed method provided linear dynamic range of 0.5 – 10 µg L^{-1} and limit of detection of 0.15 µg L^{-1}.

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Fabrication of PVC-based polymer inclusion membranes containing Cyphos® IL 101 to extract bismuth from chloride medium.

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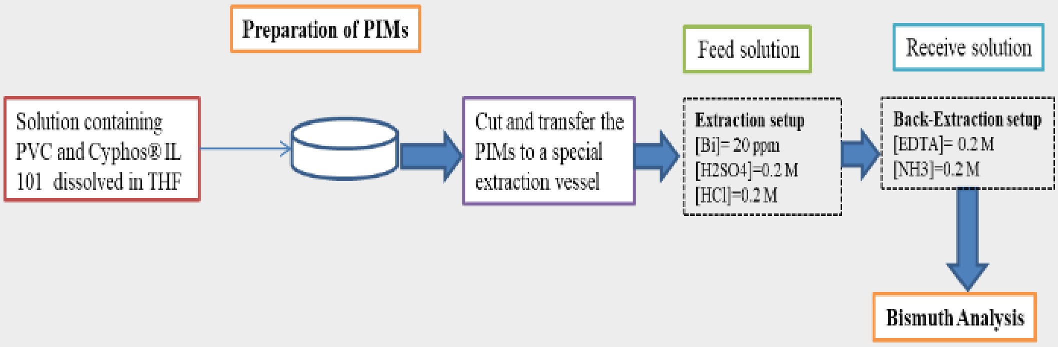

Abstract: Since bismuth has very low toxicity among heavy metals, it is often referred to as a "Green metal." [1]. Its interesting feature has attracted a great deal of attention in the field of medicinal chemistry. The present study attempted to extract bismuth using inclusion membranes containing ionic liquid. Polyvinyl chloride (PVC) and trihexyltetradecylphosphonium chloride (Cyphos® IL 101) was used as a base polymer and anion exchange extractor.[2] And with using the following materials, the optimization of a suitable membrane with acceptable quality for extraction was studied, followed by different characterization methods like TGA, SEM, and AFM on the optimized membrane. The membrane is optimized for bismuth extraction from chloride medium considering the factors such as the percentage of the extractor, acid concentration, and extraction time. The results of this study suggest that quantitative extraction of bismuth requires at least 40 percent of the extractor, 0.2 M hydrochloric acid and 8 hours of extraction time. Among several combinations of back-extraction reagents, EDTA and NH_3 exhibited the best results in removing bismuth from membranes. The study showed that the optimized membrane can do the process for a minimum of three cycles without losing efficiency. The effect of various plasticizers to enhance the performance of the studied membrane was also investigated. The present study demonstrated that PIMs containing Cyphos® IL 101 can be used as a promising material for metal ion extraction.

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Recovery of Bi(III) from hydrochloric acid solutions using a plasticized polymer inclusion membrane composed of CTA and TOA

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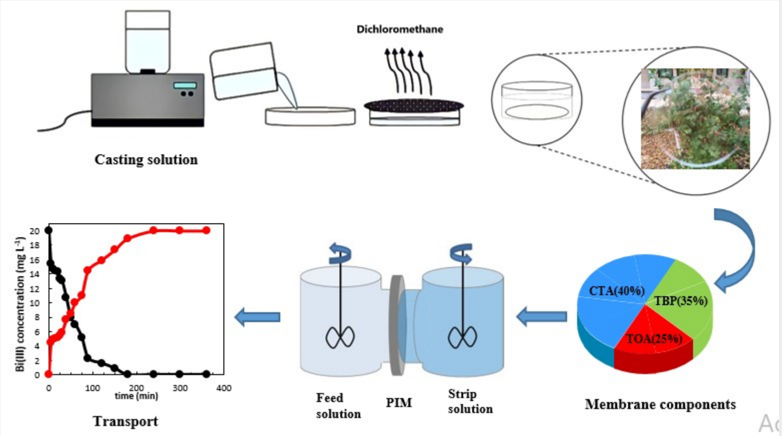
Graphical Abstract



Abstract: Polymer inclusion membranes (PIMs) are a type of liquid membranes with excellent potentials for the separation-recovery of both metal ions and organic compounds [1]. They are considered as environmentally friendly technique because the toxic and expensive organic solvents required for the solvent extraction methods are practically eliminated in PIMs techniques [2]. PIMs incorporate small amounts of an extractant within a polymer matrix, and in some cases a plasticizer/modifier can be added to improve the mechanical or extraction characteristics [3]. The present study is devoted to the application of a polymer inclusion membrane composed of 40 wt% cellulose triacetate polymer (CTA), 25 wt% trioctylamine (TOA) as the carrier, and 35% tributyl phosphate (TBP) as the plasticizer/modifier, for the transport of Bi(III) from chloride solutions. To achieve the highest efficiency, the parameters affecting the transport process were investigated and optimized. These parameters were time, initial concentration of Bi(III), phases stirring rate, the type and the concentration of the stripping reagent. Under optimized conditions, the membrane was capable to transport completely Bi(III) (initial concentration 20 mg/L) from the feed phase including 150 ml of 0.5 M hydrochloric acid solution into the receiving phase (150 ml of 2 M sodium carbonate solution) after 4 h. The selectivity of the investigated membrane toward Bi(III) was evaluated by performing competitive transport experiments of Bi(III) accompanied with copper(II), nickel(II), zinc(II), cadmium(II), iron(III), lead(II), cobalt(II), molybdenum(VI), chromium(III) and tungsten(II) cations and nitrate and sulfate anions. The potential of the studied membrane for multiuse in Bi(III) transport process was appraised and the strategies for improving this ability were checked by saturating the feed and aqueous phases with the membrane constituents. The membrane was used for the recovery of Bi(III) from some real samples.

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Synthesis and application of a porous carbon material as a new sorbent for solid phase extraction of vitamin B_{12}

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Abstract: Recently, many research studies have been focused on the development of new sorbent materials in solid phase extraction (SPE) procedure. Among different kinds of sorbents, carbon based materials appear as efficient sorbents because of their good interactions with organic compounds [1]. Also, porous materials due to having high surface area and high adsorption capacity are considered as interesting sorbent material. Recently, ionic liquids were used as precursor for preparation of carbon material via pyrolysis process [2]. Vitamin B_{12} is an essential nutrient for cell development and human growth. It acts as a coenzyme for normal DNA synthesis and plays an integral role in development of the myelin sheath [2]. The deficiency of vitamin B_{12} in humans is manifested by anemia and neuropathy [3]. It is very important to diagnose vitamin B_{12} at a sensitive level. Methods for determination of vitamin B_{12} remain limited due to their low sensitivity and poor selectivity. Here, a porous carbon material was synthesized by pyrolysis of an ionic liquid composed of citric acid and tetrabutyl ammonium bromide and applied as an efficient sorbent for extraction of vitamin B_{12} by dispersive solid phase extraction method and determination by HPLC/UV. The characteristics of the synthesized carbon material such as size, morphology, chemical composition, thermal stability and porosity with different techniques such as FE-SEM, EDX, XRD, TGA, BET and Raman and FT-IR spectroscopy were studied. Different variables affecting the extraction efficiency were investigated and optimized. In this method, the target analyte was extracted from 30 mL of the aqueous sample solution at pH 5.0 and 5% NaCl during 15 min, and desorption was performed by 300 µL methanol. The optimized methodology exhibited a good linearity between 1.0 and 250 $\mu g\ L^{-1}$ ($R^2 > 0.998$) with the relative standard deviation (RSD%) less than 4.9 ($n = 5$). The applicability of the method was demonstrated with the analysis of different samples including tap water and milk, and good results were obtained. Obtained results demonstrated proposed sorbent has advantages of rapid and efficient extraction of vitamin B_{12} due to the high porosity and surface area of the proposed sorbent.

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Alumina/graphene oxide nanocomposite as a new sorbent for extraction and preconcentration of three preservatives by dispersive solid phase extraction method

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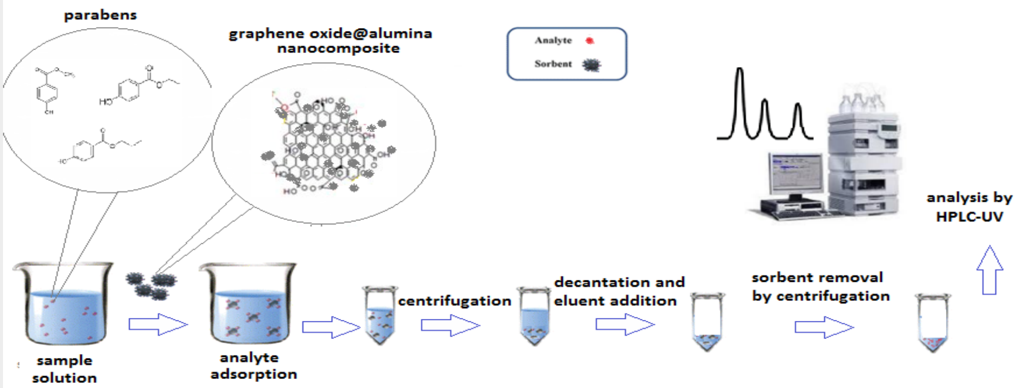
Graphical Abstract



Abstract: Recently, the development of new sorbent materials employed for solid phase extraction has received more attraction due to the need of improving the adsorption efficiency and selectivity [1]. Among different adsorbents, nanomaterials have attracted great interest because of their unusual mechanical, optical properties and high surface area. Graphene-based materials have been used as SPE sorbents for various pollutants because of their large surface area, high adsorption capacity for organic pollutants, and chemical and thermal stability [2]. The esters of p-hydroxybenzoic acid, commonly known as parabens, are worldwide applied as preservatives in a large number of personal care products and canned foods due to their antimicrobial and antifungal properties [3]. Considering the complexity of matrix and low concentration of parabens in real samples, a sample preparation step is required for isolation or enrichment of the analytes prior to the final analysis [4]. In the present study, a new nanosorbent, alumina-graphene oxide nanocomposite, has been successfully synthesized. The synthesized material was characterized by FT-IR spectroscopy, thermal gravimetric analysis (TGA), scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX) analysis. In the next step, the synthesized material was used as sorbent for solid-phase extraction of trace amounts of three preservatives (parabens) and followed by determination using HPLC/UV. Several variables affecting the extraction efficiency of the analytes i.e., amount of sorbent, salt concentration, extraction time, and desorption conditions were investigated and optimized by experimental design and one-at-a time methodologies. Under the optimum conditions, the analytical performance of the method showed good linear dynamic ranges (0.2-100 $\mu g\ L^{-1}$) and good limits of detection (0.03-0.05 $\mu g\ L^{-1}$), and the relative standard deviations (RSD%) were less than 4.4% (n = 5). Preconcentration factors of the target analytes were obtained in the range of 128-173. Finally, the applicability of the proposed method was evaluated by extraction of the parabens from some real samples including wastewater, cream, toothpaste, and juice samples, with recoveries from 88 to 109%, and relative standard deviations (RSDs%) less than 8.7 % (n = 3).

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Determination of Ni^{2+} and Cr^{3+} in Aqueous Samples by Graphite Furnace Atomic Absorption Spectrometry after Preconcentration by Organic Gas Steam Liquid Extraction

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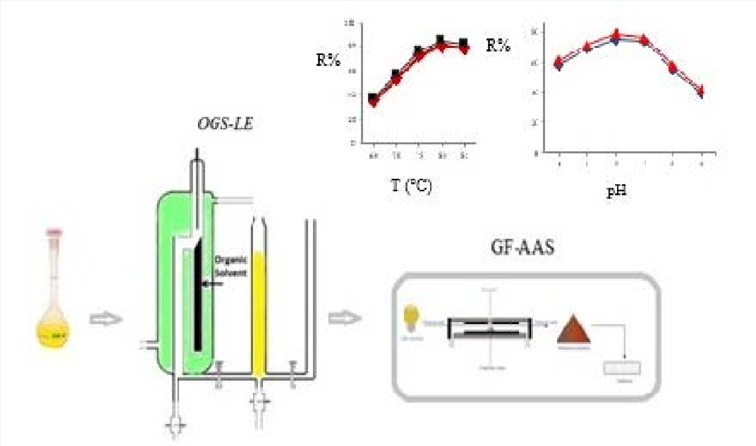
Graphical Abstract



Abstract: Heavy metals are well-known environmental pollutants due to their toxicity, persistence in the environment, and bioaccumulative nature [1]. The concentration of toxic heavy metals in water and food samples is fairly low and the interference due to matrix cannot be always eliminated. On the other hand, to cope with these problems, for real samples with complicated matrix, Preconcentration procedure is necessary before determination of metal ions [2]. In this study, organic gas steam liquid extraction method was proposed as a simple and sensitive method for the preconcentration of ultra-trace amounts of Ni^{2+} and Cr^{3+} in water as a prior step to their determination by graphite furnace atomic absorption spectrometry. For this purpose, ammonium pyrrolidine dithiocarbamate (APDC) as a complexing agent and methyl isobutyl ketone (MIBK) as extracting solvent was used, Also, in order to obtain a high extraction recovery, the effect of different parameters such as the volume of organic solvent, the molar ratio of the extracting agent relative to metal, the pH of the aqueous phase, the extraction time and the temperature of the heating chamber were investigated. Under the optimized conditions, the limit of detection (LOD), limit of quantification (LOQ) of this method, linear concentration range and percentage of the relative standard deviation were found. To evaluate the selectivity of the proposed method, the interference effects of some common ions including Na^{+}, K^{+}, Ca^{2+}, Mg^{2+}, Fe^{3+}, Co^{2+}, NO_3^- and Cl^-, on the recovery of Ni and Cr were investigated. The average recovery for the extraction of nickel and chromium was over 85%. The limit of detection was 0.38 and 0.25 μgL^{-1} for Ni^{2+} and Cr^{3+} respectively. The proposed method was successfully applied to the determination of Ni and Cr in water samples.

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Pre-concentration and determination of trace amounts of bismuth in human urine by electrospun nanofiber fabricated by PVC/Aliquat® 336

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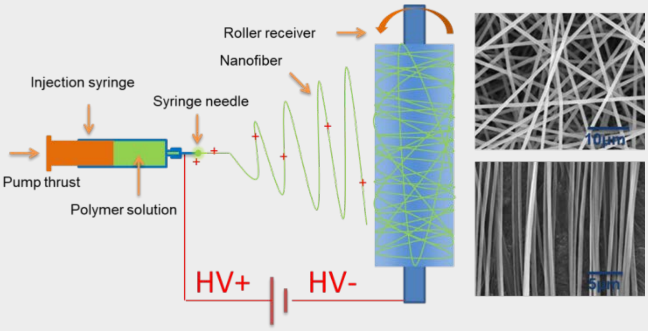
Graphical Abstract



Abstract: Electrospun polymer fibers including PVC and Aliquat 336, as the form round disks, have been used in a solid phase extraction method for the preconcentration followed by FAAS determination of trace amount of Bi(III) [1, 2]. To the best of our knowledge, no report has been presented so far based on the method presented in this paper for the preconcentration-determination of bismuth. In this research, for electrospinning of polymer fibers, polyvinyl chloride (PVC) was used as the base polymer and trioactylmethyl ammonium chloride (Aliquat® 336) was used as an anion exchange extractor [3]. First, the instrumental factors influencing the structure of polymer fibers were investigated. The potential, distance between the tip of the needle and the collector, and appropriate flow rate were determined. The extraction of bismuth was studied using electrospun fibers in chloride ion medium, and it was observed that the chloride ion concentration was extremely effective on the extraction, with the lowest necessary concentration being 0.5 M. The extraction flow rate was set to 250 ml/min as the minimum. A solution of EDTA /NH_3 was found to be the most efficient factor in the back-extraction of bismuth from electrospun fibers. The effects of zinc, lead, copper, cadmium, nickel, iron, cobalt, manganese, calcium, magnesium cations and nitrate, sulphate, chloride, thiosulfate, fluoride, and acetate anions on extraction and back-extraction were investigated, and the lowest quantity that was ineffective was determined. The evaluated polymer fibers were extracted and back-extracted at least four times without losing efficiency. The studied fibers were used to evaluate bismuth in human urine and river water samples using the standard addition method, and it was discovered that this methodology can quantify bismuth at a concentration of 5 μg/L.

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Fabrication and Characterization of Electrospun PVDF-HFP-Aliquat 336 Fiber for Eextract of Chromium from Aqueous Solutions

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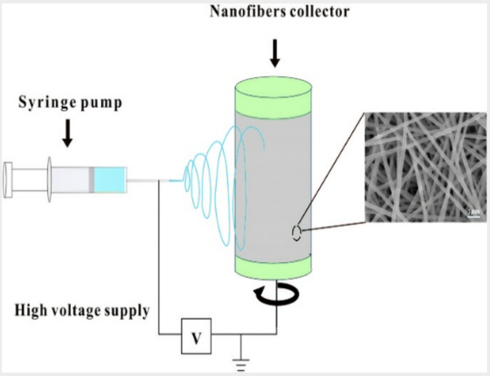
Graphical Abstract



Abstract: Hexavalent chromium is a heavy metal which is highly toxic to plants, animals and humans[1, 2]. Cr(VI)-containing compounds cause cancer of the respiratory tract, renal damage, allergy, and asthma in humans. Therefore, the Cr(VI) content of wastewaters must be reduced to a safe level before they can be discharged into the environment [3]. As a result, the current study attempted to remove Cr(VI) ions from aqueous samples using electrospun polymer fibers containing ionic liquid and bringing its amount to the World Health Organization's recommended value [4, 5]. Poly (vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP) was used as a base polymer in electrospun polymer fibers, and trioactylmethyl ammonium chloride (Aliquat® 336) was used as an anion exchange extractor. First, the instrumental factors influencing the structure of polymer fibers were investigated. The potential, distance between the tip of the needle and the collector, and appropriate flow rate were determined. Scanning electron microscopy (SEM) was used to determined fiber diameter and to observe the fiber morphology. The extraction of Cr(VI) from aqueous solution was studied using optimized polymer fibers, and the parameters affecting the extraction, such as the percentage of ligand in electrospun polymer fibers, solution pH, and flow rate, were determined. The mixture of sodium nitrate and nitric acid performed the best in the back-extraction of Cr(VI) from electrospun polymer fibers. The extraction and back-extraction of chromium using electrospun polymer fibers demonstrated that these fibers can be used in four complete cycles. According to the result of this research, electrospun polymer fibers have a high capability to remove chromium ions in low amounts.

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In-Situ Solvothermal Growth of MOF on The Capillary Fused Silica Aiming to Use as Stationary Phases in Gas Chromatography

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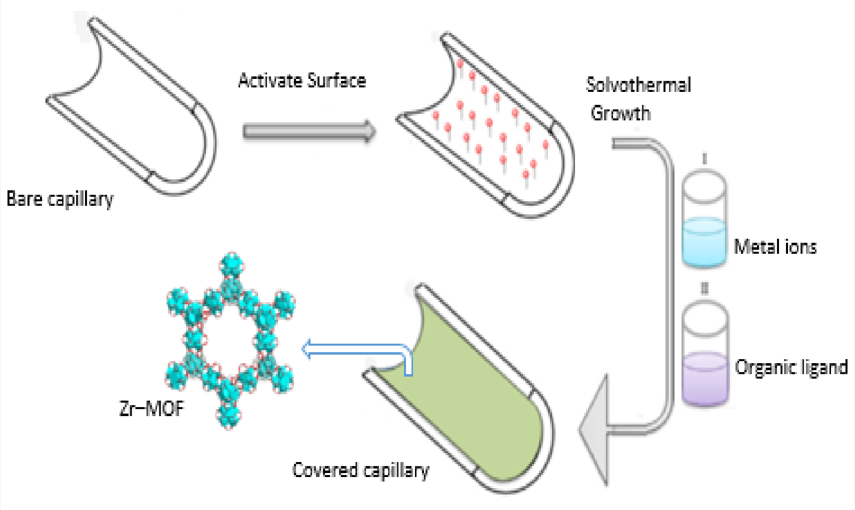
Graphical Abstract



Abstract: Metal–organic frameworks (MOFs) have received great attention as novel substrate in separation sciences because of their fascinating structures and unusual properties such as large surface-to-volume ratios, high levels of porosity, and selective adsorption of different materials. In the road to goals of this subject, several MOFs have been proposed to be as stationary phase in gas and liquid chromatography. In this work, we reported the simple and effective *in-situ* method for immobilization of special MOF on the surface of fused silica capillaries aiming to use as a stationary phase in the gas chromatography. The method includes *in-situ* solvothermal growth of MOF on the surface of the fused silica capillary. The results of the data obtained from SEM, FT-IR, XRD and BET have indicated that the MOF nanoparticles were successfully immobilized and bonded on of the surface of the fused silica capillary.

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Preparation of ZnS/MoS_2 film grown on cellulose paper for determination of cardiac drugs from aqueous solutions by thin film microextraction method

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Graphical Abstract

Abstract: In the current study, chemically modified cellulose paper was introduced as a novel extracting phase for thin film microextraction (TFME). ZnS/MoS_2 was used to modify the cellulose papers with hydrothermal conditions. The modified papers were evaluated as a sorbent for the extraction of Digoxin medicine from aqueous samples. High-performance liquid chromatography was used for the quantification of the extracted compounds. TEME parameters such as desorption condition, shaking rate, sample ionic strength and extraction time were investigated and optimized. Limit of detections were good and relative standard deviations was low under the optimized condition. The calibration curves were obtained in the bigger range with a good linearity. Human urine and Blood plasma samples were studied as real samples for the evaluation of the method.

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Optimization of Black Raspberry extract by Ultrasonic-assisted Extraction Method

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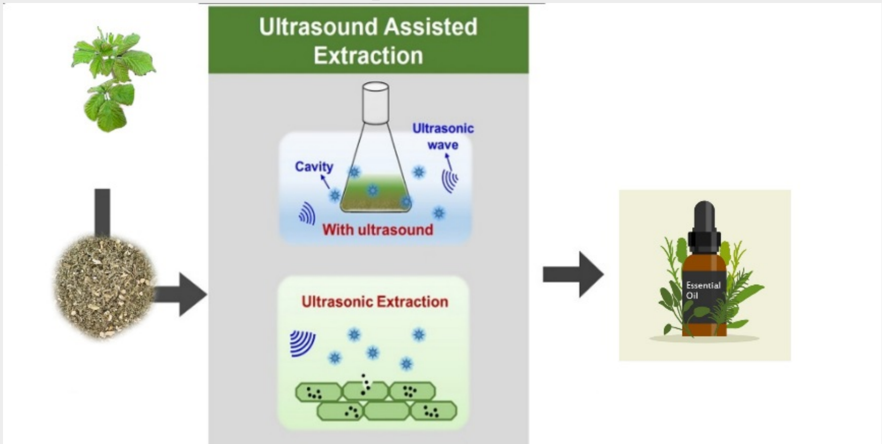
Graphical Abstract



Abstract: Safe and healthy nutrition has a beneficial effect on human well-being. Various foods, such as berries, are known to inhibit cancer-promoting pre-proliferative signals. Among fruit and berry crops, raspberries demonstrate one with the widest ranges of biologically active substances [1]. Ultrasonic-assisted extraction was employed for highly efficient separation of secondary metabolites from medicinal plants. Specifically, this extraction method was able to provide a higher content of beneficial compounds compared to conventional methods such as hot water bath, maceration, and soxhlet extraction, which have been used for many decades [2, 3]. The present study aimed to the optimizing of black raspberry extract using ultrasonic-assisted extraction method. Some of main parameters such as the kind of solvent, extraction time, power and cycle of ultrasonic, and plant to solvent ratio were optimized. The obtained results indicated that the optimized conditions were as follows: kind of solvent: methanol 80%; extraction time: 5 minute; ultrasonic power: 80%; ultrasonic cycle: 6; and plant to solvent ratio: 1:20 w/v. At optimum condition, the yield of extract was

128/1.043 mg/g dry weight (mg/g DW), whereas the yield of extract with maceration method was 21/1.006 mg/g DW. Also, total phenol and flavonoid contents as well as antioxidant activity of the extracts were studied. The analysis of total phenol and flavonoid contents was done using the Folin Ciocalteu and aluminum chloride methods, respectively. Also, DPPH (2,2-diphenyl-1-picryl-hydrazyl-hydrate) free radical was used to test the antioxidant property of the extracts. According to the results, the yield and biological activities of the extract was better than the extract obtained by classical method. So, using this method, the efficiency and biological activities of the extract were higher and can be used in food and pharmaceutical industries.

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A Novel MOF/COF Hybrid as Fiber Coating for HS-SPME Sampling of Polycyclic Aromatic Hydrocarbons from Environmental Samples

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Graphical Abstract

Abstract: Porous materials have intrigued a great deal of research interest in recent years due to their unique properties and widespread applications. In particular, so called advanced porous materials (APMs) [1] such as metal-organic frameworks (MOFs) [2,3], covalent organic frameworks (COFs) [4,5], polymers of intrinsic microporosity (PIMs), hypercrosslinked polymers (HCPs), porous aromatic frameworks (PAFs), and conjugated microporous polymers (CMPs) have been widely investigated in many fields of science and technology. Among them, COFs and MOFs have received increasing attention in various technological and scientific fields owing to the capability to control their pore size, shape, and chemical functionalities [6,7]. Herein, a novel Cu-MOF/COF hybrid was prepared and applied as a sorbent for headspace solid-phase microextraction sampling of polycyclic aromatic hydrocarbons (PAHs) in solid samples. The thermal behavior, morphology, and structure of the Cu-MOF/COF hybrid were characterized using thermogravimetric analysis (TGA), scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier transform infrared spectroscopy (FT-IR). The influential experimental parameters were investigated and optimized using a Box-Behnken design (BBD). The developed method showed low limits of detection (0.005-0.02 ng g^{-1}), and good linearities (0.1-1000 ng g^{-1}) for the PAHs. The calculated standard deviations were 3.7-11.4% (n = 6). The proposed sorbent was successfully applied for the analysis of PAHs in contaminated soil samples.

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Removal of Acid Fuchsin Dye by Using PVC-Based Polymer Inclusion Membranes Containing Bis(2-ethylhexyl)phosphoric Acid

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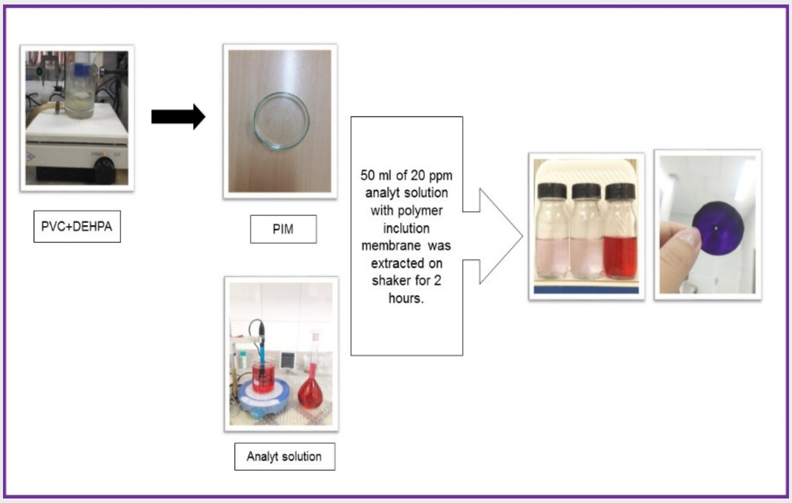
Graphical Abstract



Abstract: Organic dyes are known as important water and environmental pollutions [1]. This stimulates research groups to find efficient, cost effective and simple methods for the removal of dyes from contaminated environmental samples. Acid fuchsin (AF) is a toxic dye with a great carcinogenic potential [2]. Several techniques such as adsorption, ion exchange, photochemical degradation and membrane separation have been already employed for removing organic dyes from contaminated samples. Polymer inclusion membranes (PIMs) are composed of a base polymer, an extractant and often a plasticizer. The selected composition of PIMs paly a decisive role on their selectivity and efficiency towards a target species. It also determines the physical properties of PIMs [3]. The possibility to employ different type of extractants allows accessing to a great variety of selectivity for separation aims by PIMs [4]. In this communication, the extraction of AF from aqueous solutions by a non-plasticized polymer inclusion membrane is described. The optimized membrane composed of 50/50 wt% of polyvinyl chloride (PVC) base polymer and bis(2-ethylhexyl) phosphoric acid (DEHPA) as extractant. The prepared PIM was homogenous, transparent, and flexible. The average mass and thickness of a circular segment (3.5 cm in diameter) of the investigated PIMs were 0.09026±0.0059 g and 0.0801±0.0068 mm, respectively. The parameters affecting the extraction process such as pH, extraction time, shaking rate, type and concentration of the electrolyte and AF initial concentration were optimized. The results showed that an extraction efficiency of 96% is achieved from 50 mL of a solution containing 20 mg L^{-1} of AF adjusted to pH = 3 with 200 rpm shaking rate in 2 h. The loaded PIMs were stripped by using a solution of 0.5 M nitric acid. Isotherm and kinetic investigations of the process were studied. These studies indicated that the extraction is well described by the Langmuir isotherm, and pseudo-second order kinetic model suitably interpreted the process kinetics.

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Treatment of anionic azo dyes contaminated waters by using polymer inclusion membranes

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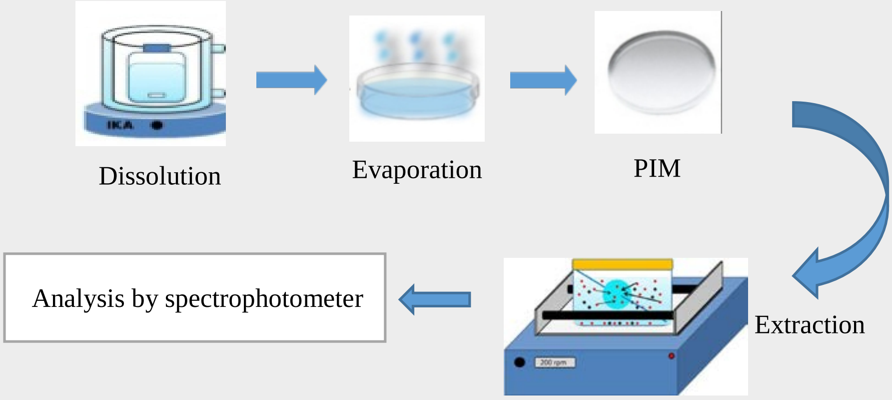
Graphical Abstract



Abstract: Dyes usually have synthetic origins and complex aromatic molecular structures. They are highly soluble in water. When these colored effluents enter the rivers or any other surface water systems, they upset biological activity. Aromatic azo dyes, with the azo group (N=N) comprise about half of the world dye market. Azo dyes are also a major class of organic compounds released by many industries such as textile, paper, plastic, leather, cosmetic, food and pharmaceutical industries. These dyes are released into the environment through industrial effluents which are hazardous to ecological systems and public health. Azo dye effluents are highly resistant to microorganisms [1]. Polymer inclusion membranes (PIMs) are a type of liquid membranes with high potentials, homogeneous, transparent, and flexible for separation metal ions, dyes and organic compounds [2]. Polymer inclusion membranes containing 40 wt% cellulose triacetate (CTA) as the polymer matrix, 25 wt% triethylamine (TOA) as extractant, and 35 wt% plasticizer modifier tributyl phosphate (TBP), for the extraction of Methyl orange (MO) from water solution. The optimized PIM was able to efficiently extract Methyl orange in 120 min from water solution. It was suggested that the presence, which was the main species extracted via an ion pair mechanism with protonated TOA by forming ($TOAH^+$)(MO^-) complex. The selectivity of the PIM towards Methyl orange was tested in the presence of SO_4^{2-}, Cl^-, NO_3^- and PO_4^{2-}. This PIM were used successfully for selective extraction of Methyl Orange from a various water samples. The loaded PIM could be quantitatively back extracted with 0.5 mol L^{-1} NH_4OH. The developed PIM was stable for four extraction/back extraction cycles without loss of its extraction capacity for Methyl Orange. It is found that addition of 1 drop extractant (TOA) and 4 drop plasticizer (TBP) to the PIM composition improves the stability so that the newly modified PIM can be used for eight extraction/back-extraction cycles. The kinetic and isotherm studies revealed that the sorption of Methyl orange by the optimized membrane is a chemisorption process.

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Polymer Inclusion Membrane Composed of Cellulose Triacetate and Trioctylamine for Removal of the Anionic Dye Congo Red

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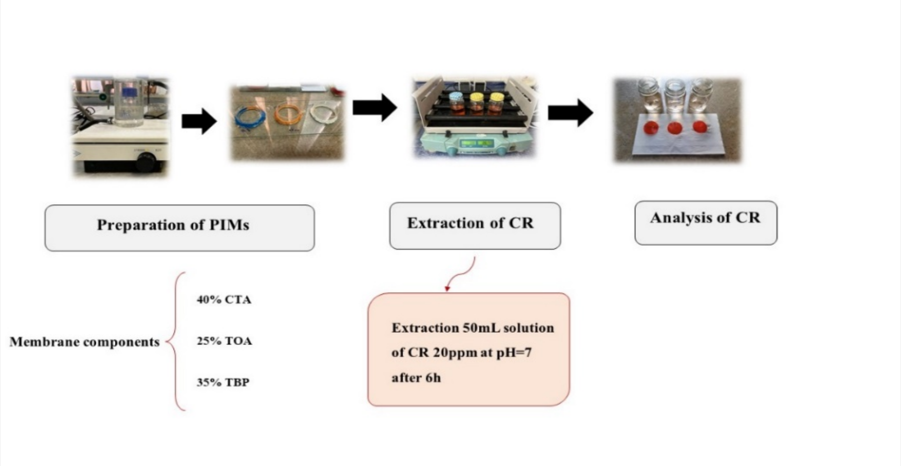

Abstract: Organic dyes are pollutants which widely used in several industries that finally dumped as wastewater, contributing to environmental and water pollution [1]. Synthetic dyes, including azo dyes, are considered to be more toxic, mutagenic, and carcinogenic in nature. Therefore, the removal of the dyes from wastewaters is a serious challenge. Congo red is an anionic type of the azo dyes [2]. A variety of the physical, chemical and biological techniques was employed for the removal of the pollutants from wastewaters. Membranes based process have attracted attention as a valuable technology for many industries. Polymer inclusion membranes (PIMs) are a type of liquid membranes which have been widely used for purifying wastewaters. PIMs are thin films that are formed by mixing an extractant, a base polymer and sometimes a plasticizer, in an organic solvent [3]. The films which are transparent, flexible, homogeneous and mechanically strength is suitable for the extraction experiments.

In the present study a PIM containing 40% w/w cellulose triacetate (CTA) as the base polymer, 25% w/w trioctylamine (TOA) as the extractant, and 35% w/w tributyl phosphate (TBP) as a plasticizer were fabricated, characterized and used for the removal of congo red from aqueous solution [3]. The average mass and thickness of a circular segments (3.5 cm in diameter) of the ten PIMs which employed in the experiments were 0.07306±0.00292 g and 0.0748±0.0054 mm, respectively. Sample solution pH, contact time, shaking rate, type and concentration of the electrolyte and initial concentration of dye solution, as effective parameters on the process efficiency, were evaluated. The results indicated that, highest extraction percentage was achieved from 50 mL of a solution containing 20 mg L^{-1} of congo red in pH = 7 with 200 rpm shaking rate in 6 h. Isotherm and kinetic of the process were studied and shown that the extraction isotherm and kinetic were well described by the Langmuir isotherm and pseudo-second order kinetic model, respectively.

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Synthesis and application of magnetic metal organic framework nanocomposite as an adsorbent for removal of citalopram from aqueous solutions

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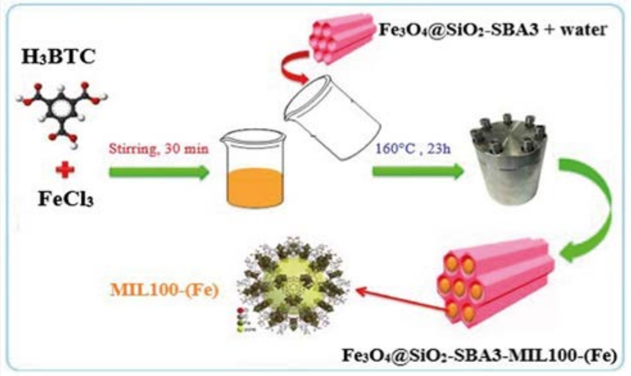

Abstract: Citalopram is an antidepressant belongs to a group of drugs called selective serotonin reuptake inhibitors (SSRIs). The popularity of citalopram has led to its excessive consumptions consequently. As a pharmaceutical pollutant, citalopram causes contamination when enters into the environment. The development and use of highly efficient methods for its removal from aqueous environments are of great importance for the environmental scientist [1, 2].

In the present study, a nanocomposite of meso-metal organic framework (Fe_3O_4@SiO_2-SBA3-MIL100-(Fe)/SO_3H, Meso-MOF) for removal of citalopram hydrobromide was prepared. Characterization of the synthesized Meso-MOF was performed by FESEM, FT-IR, XRD, and N_2 adsorption-desorption techniques. The results confirmed the synthesis of nanocomposites with particle size less than 30 nm. The obtained BET surface areas and the total pore volume of Meso-MOF were obtained as 119.69 m^2g^{-1} and 0.09964 cm^3g^{-1}, respectively. All of the experimental variables, influencing the citalopram hydrobromide removal efficiency were investigated using Central Composite Design. Under the optimum conditions (contact time = 20 min, pH = 10, adsorbent weight = 0.1 g, sample volume = 25 mL and ionic strength = 0), more than 95% of citalopram hydrobromide was removed.

The pseudo-first order ($R^2 = 0.6235$), pseudo-second order ($R^2 = 0.9827$), intra particle diffusion ($R^2 = 0.5354$) and Elovich kinetic ($R2 = 0.7235$) models were investigated, and the kinetic data followed the pseudo-second order kinetic model ($R^2 = 0.9827$, $Q_e = 64.23$ mg g^{-1}, $K_2 = 0.012$).

Compared with the Langmuir isotherm model ($R^2 = 0.9366$), the Freundlich model ($R^2 = 0.9887$), has a higher degree of fitting, which reveals that the adsorption of citalopram hydrobromide by Meso-MOF is more consistent with the theory of multi-layer adsorption.

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Bubble-in-drop microextraction of medicinal plant followed by gas chromatography-mass spectrometric analysis

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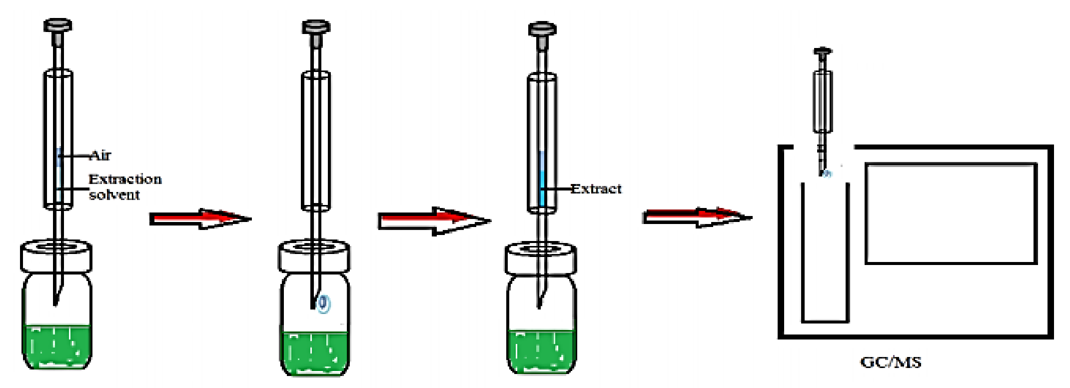

Abstract: Bubble-in-drop microextraction (BID) is a variation of single-drop microextraction in which an air bubble is deliberately introduced to the solvent to enhance extraction efficiency. Here, BID coupled with GC-MS was developed for the extraction, enrichment and determination of essential oils of medicinal plants [1]. This article investigates the effect of microwaves on the amount of volatile compounds Echinophora platyloba DC with two methods, headspace single-drop microextraction (HS-SDME) and Bubble-in-drop headspace microextraction (BID-SDME), for the analysis of essential oil [2]. Solvent selection, solvent volume, microwave power, irradiation time and sample mass were optimized by simplex method. Thirty two components were extracted and identified by use of this method, E-β ocimene (37.54%), R-D-decalactone (14.06%), α-pinene (8.35%) and nonane (6.85%) were the major constituents. It was observed that relative standard deviation (RSD) values for the main compounds were less than 9.5%.

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Zn MOF/COF Composite as an Effective Adsorbent for Solid-Phase Microextraction Sampling Of PAHs from Soil Samples

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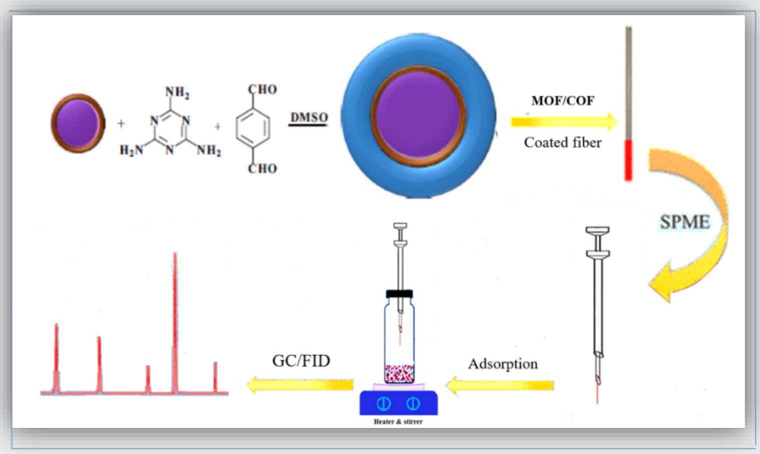

Abstract: By hybridization of zinc-based metal-organic framework-5 (MOF-5) and melamine-terephthaldehyde-based two-dimensional π-conjugated covalent organic framework (COF), a novel MOF-5/COF (M5C) nanocomposite was prepared. M5C was carried out for efficient solid-phase microextraction (SPME) sampling of ultratrace trace levels of polycyclic aromatic hydrocarbons (PAHs) from soil samples prior to their separation and determination by GC-FID [**1**, **2**]. The adsorbent was characterized by Fourier transform infrared spectrometry, X-ray diffractometry, Scanning electron microscopy, Thermal gravimetric analysis, and Nitrogen adsorption-desorption. A Box-Behnken design (BBD) was carried out to evaluate the variables affecting the extraction efficiency [**3**]. Under the optimum conditions, the proposed method showed low limits of detection (LODs, 0.006–0.54 μg g−1), wide linear ranges (0.02–2000 μg g−1), and good repeatability with relative standard deviations (RSDs) of 0.11–4.93% for single fiber and 0.42%–7.36% for fiber-to-fiber. The spiked recoveries at two levels of 20 and 50 μg g−1 were in the range of 76.5–112.0% with the RSDs less than 8.3%. Compared with commercial fibers, the homemade fiber displayed excellent extraction capacity. The Zn MOF/COF coating was stable over 120 successive cycles of extraction/desorption without significant loss of extraction efficiency. The method was successfully applied for the determination of PAHs in real soil samples.

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Design of a New Magnetic Solid Phase Microextraction system for simultaneous Separation and Preconcentration of Tetracycline and Chloramphenicol

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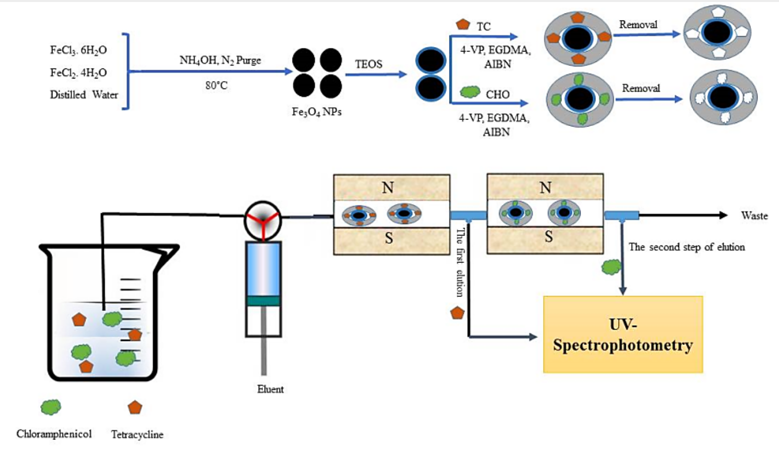

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Abstract: In this paper, a new, simple, low-cost, and selective magnetic solid phase microextraction system for simultaneous separation and preconcentration of tetracycline and chloramphenicol before their spectrophotometric determination has been designed based on as previous experiences [1-3]. In this method two molecularly imprinted polymer (MIP) for specific sorption of tetracycline and chloramphenicol is synthesized on the surface of magnetic Fe_3O_4 nanoparticles. Each sorbent is held separately by a magnet in different part of a loop. Then, the sample solution is passed through the loop causing selective extraction and preconcentration of each analyte on the respective sorbent. Finally, the two sections of the loop are separated and each analyte is eluted by a proper sorbent and quantified. The major parameters affecting the extraction, including the sample pH, sample flow rate, sorbent amount, sample volume, eluent concentration, and eluent volume were investigated. The optimum conditions were: pH 6.0; flow rate, 5 mL min^{-1}; amount of magnetic nanoparticles, 15 mg; sample volume, 50 mL, and eluent volume, 500 μL. Under optimized conditions, a preconcentration factor of 100 and detection limits of 0.027 and 0.058 μg L^{-1} were achieved for tetracycline and chloramphenicol, respectively. The method was successfully applied to the determination of the analytes in natural water and serum samples.

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Electrosynthesis of Cu-BDC Metal–Organic Framework on Pencil Graphite Substrate for DI-SPME of Some Pesticides and Determining Them Through High Performance Liquid Chromatography-Ultraviolet Detection

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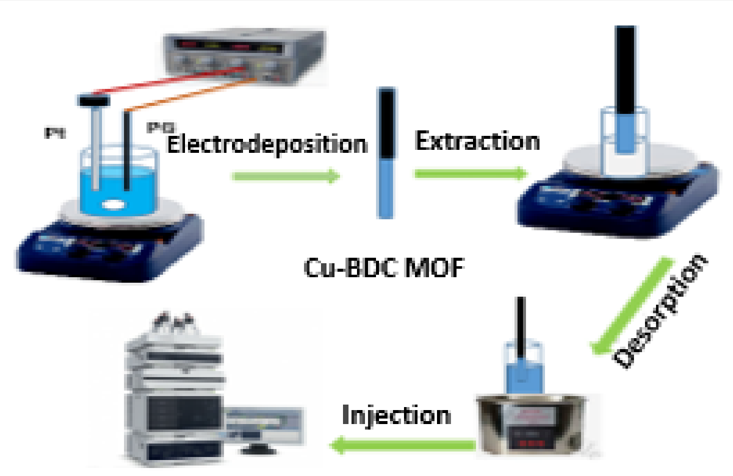

Abstract: In the current study, a fiber coating based on copper 1,4-benzenedicarboxylate metal–organic framework (Cu-BDC MOF) was fabricated on the surface of a pencil graphite (PG) substrate by an in-situ electrodeposition approach. The fiber was used as the sorbent in fiber solid-phase microextraction (SPME) of two chosen pesticides including abamectin and amitraz [1,2]. The extracted pesticides were quantified by high-performance liquid chromatography-ultraviolet detection (HPLC-UV). The electrosynthesis of the fiber coating was performed under a constant potential in a solution containing sodium nitrate as the probase, 1,4-Benzenedicarboxylic acid as the ligand and copper nitrate as the cation source. The coating was characterized using field emission scanning electron microscopy (FESEM), energy-dispersive X-ray spectroscopy (EDX), Fourier transform infrared spectroscopy (FT-IR), and X-ray Diffraction (XRD) analysis. The effective parameters on the electrosynthesis and extraction processes were optimized by experimental design. Under the optimized conditions, the extracted amitraz and abamectin were quantified over an appropriate linear range. Suitable limits of determination, appropriate limits of quantifications, satisfactory enrichment factors, acceptable absolute recoveries and acceptable spiking recoveries were achieved for amitraz and abamectin.

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Evaluation and Determination of Phthalates in Ten Samples of Lipstick and Ten Samples of Foundation Cream in Iran's Market

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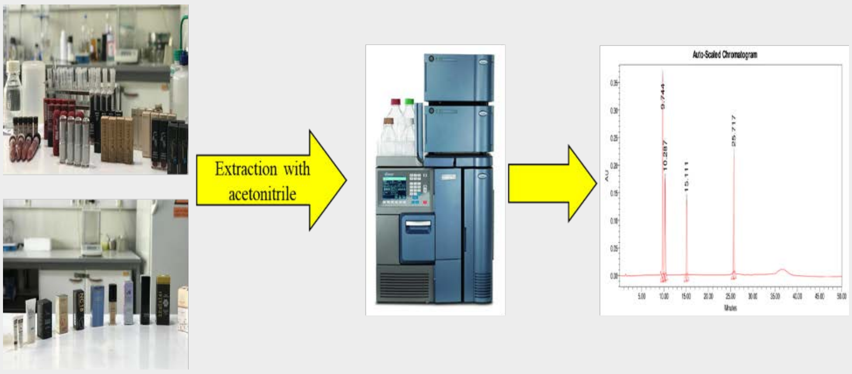

Abstract: From the past until now, beauty has had a special place among the world's people, especially women. Hence, a lot of money is spent on buying cosmetics across the globe [1]. One of the common frauds is phthalate, which uses as a plasticizer in products for softening, anti-cracking, etc. [2]. Four high-risk and high-consumption substances in cosmetics include Benzyl butyl phthalate (BBP), Dibutyl phthalate (DBP), Dicyclohexyl phthalate (DCHP), and Di (2-Ethylhexyl) phthalate (DEHP), have side effects on human health. A simple, sensitive, and selective analytical method was developed with High-performance liquid chromatography Photodiode-Array Detection to evaluate and determine these mentioned phthalates simultaneously in Lipstick and Foundation cream. A gradient program was set with an initial ratio of acetonitrile and water plus 10 µL trifluoroacetic acid (65:35) as the mobile phase. This ratio was changed at different times and separated four phthalates from each other. Standard calibration curves were drawn linearly for each phthalate under the optimum conditions. The limit of detection values of BBP is about 0.15 µg/mL, DBP and DCHP are about 0.075 µg/mL, and DEHP is 0.15 µg/mL. By two-way ANOVA method, days change and phthalate type's changed examined in three spike levels for lipstick and powder cream. The results during three days in each level and for both products showed that there was no significant difference between the measurement efficiencies. P-value in lipstick is 0.8 for the first level, 0.87 for the second level, and 0.28 for the third level, respectively; for foundation cream is 0.43, 0.14, and 0.91 for each level, respectively. The second factor studied was the change in phthalate type, which showed that the measurement efficiency changes significantly with the shift in phthalate type. The P-value of this factor in lipstick is 3.09×10^{-7}, 6.47×10^{-7}, and 1.73×10^{-10} for each level, respectively; for foundation cream in each level is 5.62×10^{-9}, 6.43×10^{-8}, and 4.28×10^{-9}, respectively. The study aims to evaluate and determine four high-consumption phthalates in Lipsticks and foundation creams in Iran's market. Also, it helps the regulatory organizations make limits and rules for the consumption of these ingredients in the industry. All results indicated that these cosmetics maybe have one of the described phthalates. They were used as an ingredient in the formulation or have migrated into it due to environmental conditions.

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Preparation of polymer inclusion membrane based on natural hydrophobic deep eutectic solvent for extraction of Co(II)

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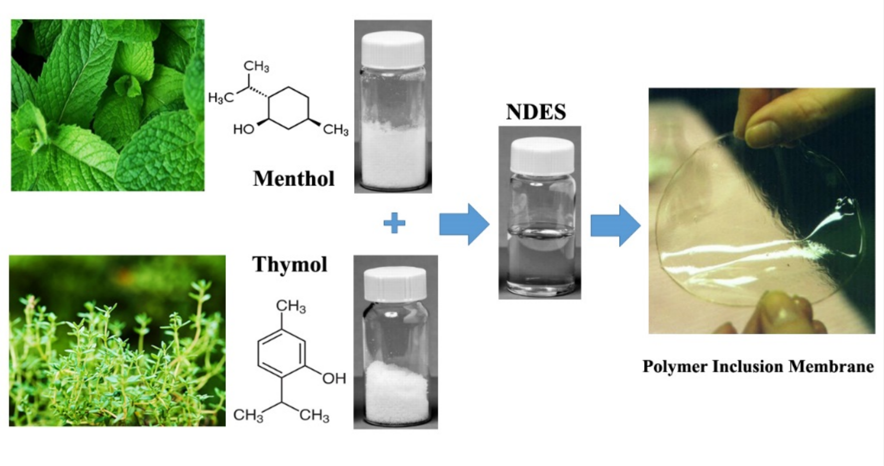

Abstract: In recent years, environmental-friendly extraction methods have been introduced as the alternative to conventional extraction procedures. In this particular field, a novel class of solvents known as deep eutectic solvents (DES) has arisen as a new and very promising tool. Compared with conventional organic solvents, natural deep eutectic solvents have attracted considerable attention due to the fact that they not only are eco-friendly, non-toxic, and biodegradable organic compounds but also have a low cost, being easy to produce in the own laboratory [1,2]. In this research, a natural deep eutectic solvent (NDES) has been synthesized by mixing thymol and menthol, and then used as carrier to prepare a homogenous and flexible polyvinylchloride (PVC)-based polymer inclusion membrane (PIM) for the recovery of Co(II) in aqueous system. A series of characterizations and conditions optimization experiments have been performed on the prepared PIMs. It was found that PIM composed of 60 wt% PVC and 40 wt% NDES had excellent selectivity and could achieve the quantitative extraction of Co(II) from aqueous solution. Under the optimal conditions, 98.1% of cobalt was extracted by the optimized PIM. Besides, the elaborated PIM had high reusability and stability, which still had an extraction efficiency of 92.4% after four consecutive extraction experiments. These results all confirm the advantages of the newly developed PIM in the extraction and recovery of cobalt, and it will be expected to realize a wide range of economic and practical value.

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Unambiguous Identification of Saxitoxin using LC-MS/MS & HPLC-FLD

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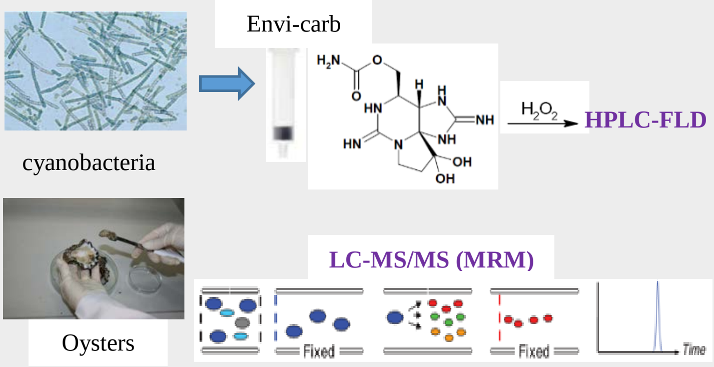

Abstract: Saxitoxin (STX) is a member of the paralytic shellfish neurotoxins that are produced by certain marine dinoflagellates and freshwater cyanobacteria [1]. These organisms are responsible for harmful algal blooms (HAB) or red tides. STX is transferred from toxin-producing algae to human through seafood such as shellfish, lobster, fish; or contaminated drinking water (from cyanobacteria) and cause intoxication. STX binds to the sodium channels in nerve cells, thereby blocking the influx of sodium ions into cells, preventing impulse-generation in the peripheral nerves and skeletal muscles, leading to paralysis, respiratory failure and death [2]. The lethal dose, LD_{50} (mouse, oral) is 263 μg/Kg. It is also being included in the both chemical and Biological Weapon Conventions (CWC & BWC) [3], so unambiguous identification of this neurotoxin is very critical. The HPLC-FLD data that usually is used for identification is not unequivocal, so for unambiguous identification, a data rich spectrometric technique (LC-MS/MS) is needed. The aim of this work was isolation of STX from some simple and complicated environmental samples and unambiguous identification using HPLC-FLD and LC-MS/MS (MRM) as confirmatory approach.

Sample preparation for simple water samples was developed using different solid phase extractions. Finally, ENVI-Carb cartridge was selected. For complicated matrix (such as orange juice, milk, sea water, algae and mussel), after protein precipitation using acetonitrile, at first lipophilic interfering materials were isolated using DSC-18 cartridge. Then, after solvent exchange to water, an ENVI-Carb was used for extraction of STX. Other interfering matrix components were washed with water. STX was eluted with 2% formic acid, 20% acetonitrile solution, concentrated and analyzed using LC-MS/MS (MRM) and HPLC-FLD (after hydrogen peroxide pre-column oxidation, Fluorescence detection (Ex=350 nm, Em=395 mn)).

After optimization of methods and instrument parameters, the on-column limit of detection (LOD) was determined 3 picogram. The results showed that STX (100 ppb, 1 gram of oyster homogenized material) was extracted and analyzed successfully. This method was used in the 6th OPCW (Organisation for The Prohibition of Chemical Weapons) international Biotoxin Sample Analysis Exercise and results showed that STX was identified successfully in the samples. The method can be used for analysis of various environmental samples and seafood.

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Dispersive liquid-liquid microextraction with deep eutectic solvents for the extraction and measurement of phenazopyridine by spectrophotometry and HPLC

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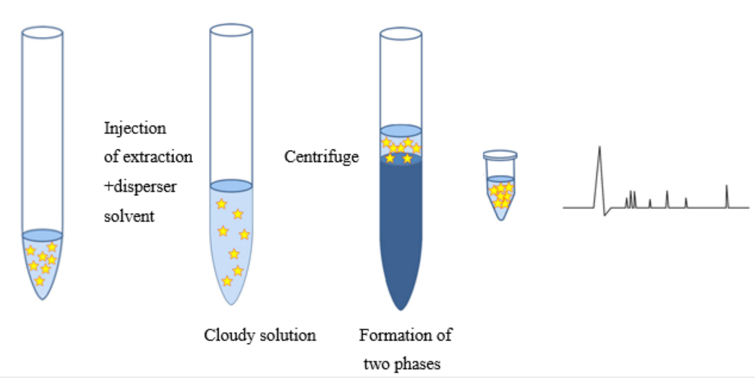

Abstract: Determining small amounts of drugs in biological fluids (plasma, blood serum, and urine) is one of the most important issues in the process of evaluating the effect of the drugs on the body. One of the most important steps in this process is the pretreatment of the test samples. The dispersive liquid-liquid microextraction (DLLME) technique is based on a ternary solvent system in which the first component contains an extractive organic solvent and the second one is a dispersing organic solvent that must be soluble in both aqueous and organic phases. The third component is a sample aqueous solution. The aim of this study is to obtain an efficient and reproducible method for preconcentration, extraction and measurement of phenazopyridine in biological samples of drug consumers [4]. Another goal of this research is the synthesis and use of green, simple and fast deep eutectic solvents as disperser in the DLLME method. In this study, a DLLME technique was designed for the extraction of phenazopyridine in urine. A mixture of TBABr salt and ethylene glycol with specific molar ratio were used to prepare the deep eutectic solvent to be used as the disperser [1], [3]. Undecanol was also used as the extraction solvent. Recoveries of 95%-98% were obtained for urinary phenazopyridine extraction by the designed method. A preconcentration factor of 32.6 and an RSD of about 5% were achieved. The proposed method is fast, low cost and environmentally friendly [2].

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Application of thin film microextraction by polymer inclusion membrane for determination of Hg^{2+}

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Graphical Abstract

Abstract: Mercury ions (Hg^{2+}) are the dominant mercury species in water, and have severe toxic effects on human health and the ecosystem. Due to the limitations of traditional analytical methods for mercury determination, such as complex pretreatment and costly instruments, numerous efforts have been made to develop rapid, low-cost, and portable sensors for Hg^{2+} determination [1]. In the past three decades, the development of optical sensors or optodes has progressed. This was primarily because of simplicity, portability and costless devices [2]. In this study, a thin film microextraction based on polymer inclusion membrane (PIM) was applied for preconcentration and measurement of mercury(II) ion. Optimized membrane was obtained containing 55% (w/w) polyvinyl chloride base polymer, 40% (2-ethylhexyl) phosphoric acid extractor and 5% diphenylcarbazide chromophore. The variables affecting microextraction efficiency, including pH of solution, ionic strength, stirring rate and extraction time, were also investigated and optimized. The optimize conditions were found at pH 11, 0.02 g/ml of sodium chloride added and extraction time of 30 minutes at room temperature. The analytical performance of the method was evaluated and the proposed method offered linear range between 5 to 100 μg L^{-1}, limits of detection of 1.4 μg L^{-1} and limit of quantitation of 5 μg L^{-1}, respectively. Good precision was demonstrated in terms of the relative standard deviations less than 5.2%. Selectivity of the method was evaluated at present of different possible interfering cations and anions. Finally, the proposed method was used for determination of mercury in tap water and fish meat samples. Relative recovery for tap water sample was determined in the range of 94-110%.

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Facile and Rapid Preparation of Magnetic Octadecylamine Nanocomposite and Its Application as a Capable Adsorbent in Magnetic Dispersive Solid Phase Extraction of Some Polycyclic Aromatic Hydrocarbons from Wastewater Samples

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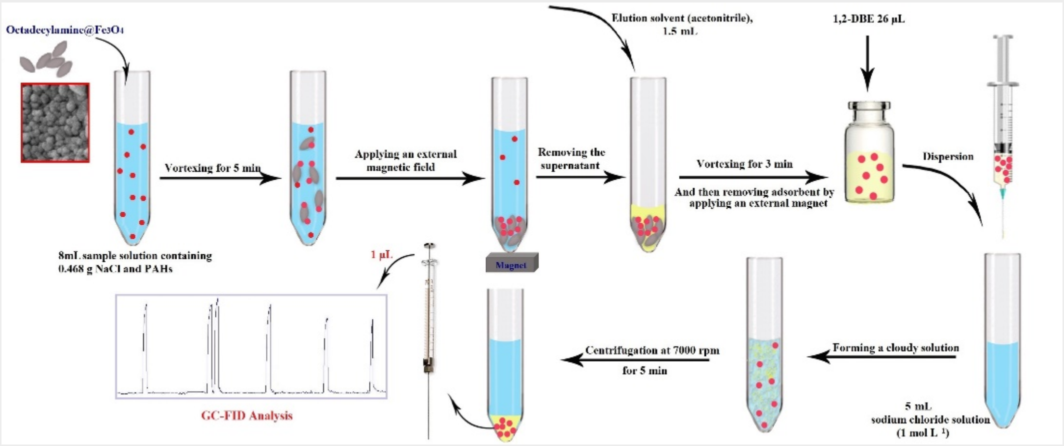
Graphical Abstract



Abstract: Polycyclic aromatic hydrocarbons (PAHs) are a class of perilous carcinogenic compounds comprising of two or more fused aromatic rings [1]. These compounds are prevailing environmental contaminants that are formed during the incomplete combustion of organic materials. It has been postulated that long–term exposure to PAHs is dramatically linked to human health threats such as lung cancers, reproductive abnormalities, DNA damage, and cardiopulmonary mortality [2]. Considering the stability and bioaccumulation risk of these compounds, development of a quick, sensitive, and simple analytical method for monitoring and determination of PAHs in various industrial and environmental wastewaters is highly demanded. In this research, a highly-effective and reliable sorbent based on octadecylamine@Fe_3O_4 was proposed and used in magnetic dispersive solid phase extraction (MDSPE) procedure. The MDSPE method was followed by dispersive liquid-liquid microextraction and employed for the extraction and preconcentration of some PAHs (acenaphthene, phenanthrene, anthracene, pyrene, acenaphtylene, and benzo[a]pyrene) from wastewater samples. The developed process was performed by dispersing the nanoparticles into an aqueous sample solution containing the target analytes by vortexing. Then the magnetic sorbent was collected by applying an external magnetic field followed by adding acetontrile to elute the analytes from the sorbent surface by vortexing for a few minutes. By utilizing an external magnet once again, the sorbent was separated, the attained eluent was mixed with an extraction solvent at µL–level and speedily injected into deionized water. After centrifugation, the enriched analytes were determined using a gas chromatograph equipped with flame ionization detection. It is worth mentioning that preparation of the utilized sorbent is effortless, convenient, and completed in nearly 2 h. Good figures of merit including high enrichment factors (506–676) and extraction recoveries (63–84%), wide linear ranges of the calibration curves, and low limits of detection (0.11–0.30 µg L^{-1}) and quantification (0.40–1.0 µg L^{-1}) were achieved in the present approach.

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Evaluating the Effect of Magnetic Field in Magnetic Fiber Headspace Solid-Phase Microextraction of Volatile Components Using Fe_3O_4@MCM-41/ MIL-101 (Fe)/ MIL-53 (Fe) Nanocomposite

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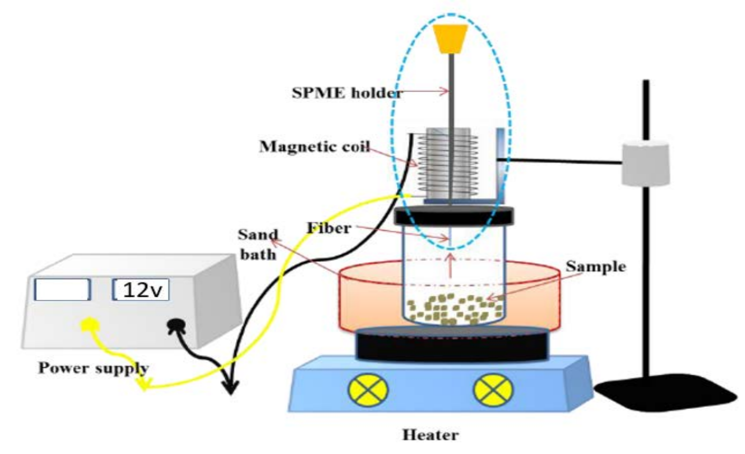
Graphical Abstract



Abstract: Solid-phase microextraction (SPME), a relatively efficient and versatile sample preparation method, was developed by Pawliszyn. The fiber-based SPME is simple, rapid, solvent free and cost effective [1]. To improve the capabilities and expand the applications of this technique, different configurations of SPME have been developed and promoted during the recent years. In addition, reinforced SPME techniques such as vortex-assisted [2], microwave-assisted [3] and solvent-assisted [4] techniques have been emerged recently. In this work, a technique has been developed for magnetic fiber headspace-solid phase microextraction (MF-HS-SPME) of plant volatile components. For this purpose, Fe3O4@MCM-41 (Mobil Composition of Matter No. 41; a nanoporous sorbent) /the porous MIL-101(Fe) Metal–organic framework/ MIL-53 (Fe) nanocomposite was prepared and used as the fiber coating. The resulting material was characterized by SEM, XRD, BET, TGA and FTIR. The extraction efficiency for the components of Nepeta elymaitica Bornm (one of the endemic species in the genus Nepeta in Iran) was compared with different systems. Comparison of permanent magnets and AC and DC magnetic fields indicated the superior effect of the AC field for the target analytes. Optimization of the extraction parameters including extraction temperature, extraction time, moisture content of the sample, desorption time and desorption temperature was carried out using a GC-MS instrument and a simplex method. The prepared fiber was stable in multiple injections with relative standard deviations of 5.3 to 10.9% for 6 replicates. The proposed method was successfully applied to the extraction and identification of volatile components of some Nepeta elymaitica Bornm samples. According to the results obtained by GC-FID, 1,8-cineol, nepetalactone (4a-α,7- α,,7a- α), E-caryophyllene, terpiene-4-ol and linalool were the main components of the plant.

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Salting-out assisted micro liquid-liquid extraction (SA-µ-LLE) of sertraline antidepressant from different real samples and determination by UV-Vis spectrophotometry

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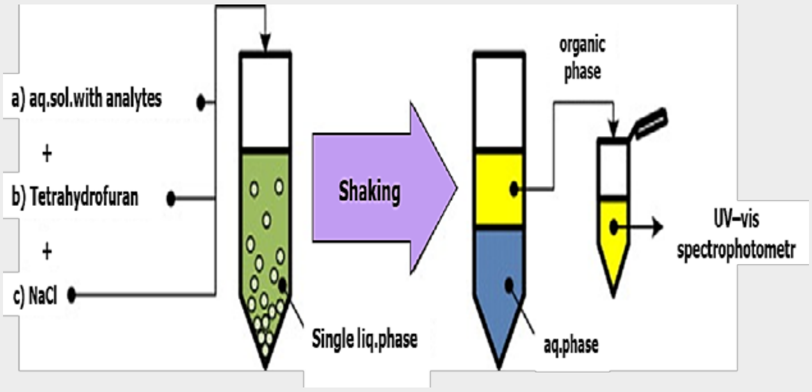

Abstract: Sertraline is an antidepressant with selective inhibition of serotonin reuptake, which decreases serum level of inflammatory factors in patients with depression [1]. Salting-out assisted micro liquid-liquid extraction (SA-µ-LLE) is a simple extraction method which used polar and simple solvents (such as acetonitrile) for extraction of different analytes [2]. In this research, SA-µ-LLE combined with UV–Vis spectrophotometry was applied for extraction and determination of sertraline. Sertraline is a secondary amine and it forms dithiocarbamate complex in the present of carbon disulfide (CS_2) and nickel(II) ion. The formed complex has maximum wavelength at 389 nm. The effective factors such as pH, type and volume of extraction solvent, effect of type and amount of salt, and derivatization parameters were investigated and optimized. The best condition was achieved by tetrahydrofuran as the extraction solvent, NaCl as the salt, at pH equal to 11. Under optimal conditions, the linear concentration was from 1 to 20 µg/ml, the intra- and enter-day precisions were 6.3 and 8.5% respectively. Finally, the method was applied for determination of sertraline in the pharmaceutical formulation and urine samples. The relative recoveries were in the range of 86 to 112%.

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Adsorption of Cr(VI) from aqueous environment onto metal-organic framework MOF-808: Synthesis, kinetics, equilibrium and thermodynamics

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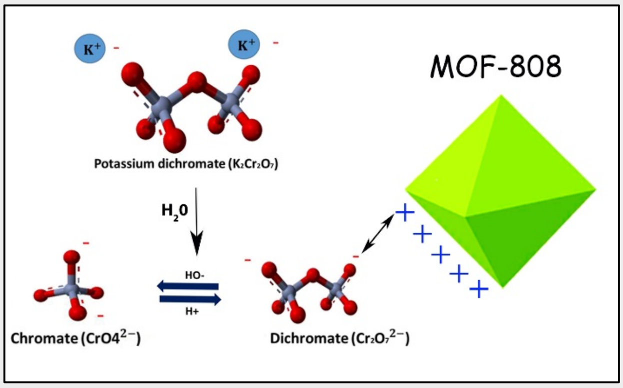

Abstract: Unlike the removal of organic contaminants from aqueous solution, heavy metals are not easily degraded and remain a persistent threat to the environment [1]. The Cr(VI), as one of the heavy metals is one kind of dangerous water pollutants authenticated by the World Health Organization (WHO) [2]. In recent years, numerous techniques have been developed for the removal of Cr(VI) from aqueous solution. Among various developed techniques for Cr(VI) removal, adsorption has attracted more attention due to its high efficiency, simplicity, low energy consumption and easy recyclability [3]. Metal organic framework (MOF) has received extensive attention due to its unique crystal structures and excellent properties [4]. In the present study, we have successfully synthesized water-stable MOF-808 material, which was developed into an effective Cr(VI) removal material from water. The experimental results suggest that MOF-808 fast response and high capture capability for Cr(VI). The maximum absorption capacity of MOF-808 at 298 K and pH = 5 was 280 mg/g for Cr(VI) and adsorption equilibrium was reached after 20 min. Pseudo-second-order kinetic (R^2 = 0.9945) and Langmuir isotherm model (R^2 = 0.9995) are more suitable for describing the adsorption process of Cr(VI) on MOF-808, which indicates that the process is a physically controlled monolayer adsorption. Thermodynamic investigation proposed spontaneous ($\Delta G° < 0$), disordered ($\Delta S° > 0$) and endothermic ($\Delta H° > 0$) for adsorption process. Besides, MOF-808 displayed an appropriate reusability for the elimination of Cr(VI) ions from their aqueous solutions for 6 successive cycles. In general, MOF-808 has been shown well adsorption properties and reusability, and our finding might provide a new idea for developing efficient and reusable adsorbent to remove Cr(VI) from wastewater.

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Spectrophotometric Determination of Iodate Based on Ultrasound-Assisted Emulsification Microextraction Using Deep Eutectic Solvent

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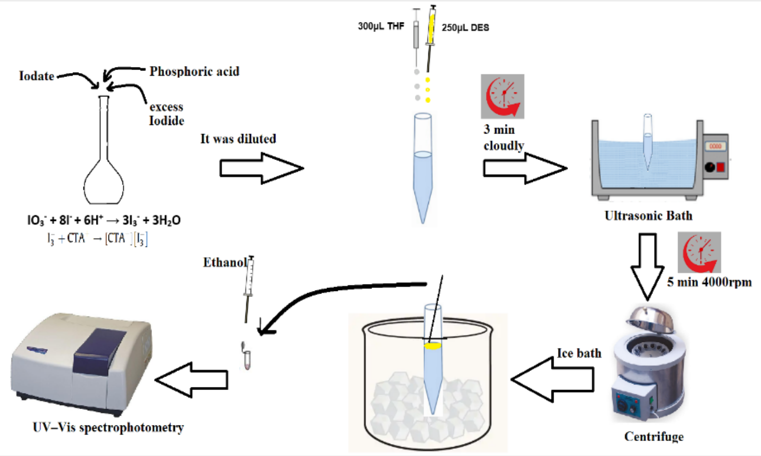

Abstract: Iodine is an essential element of the human body and is a fundamental part of the thyroid hormones that are essential to the development and differentiation of all cells. Iodine deficiency is an important public health issue for populations throughout the world, which can lead to developmental delays and other health problems (i.e., hypothyroidism, brain damage, and cretinism). The common method to prevent these iodine deficiency disorders is salt iodization due to its extensive consumption. Iodate is added to iodized salt as a source of iodine to prevent iodine deficiency disorders in many countries. Thus, iodate quantification in iodized edible salt is of great importance [1]. In 2021, Caleb et al. used smartphone digital image colorimetry combined with the solidification of floating organic drop-dispersive liquid-liquid microextraction for the determination of iodate in table salt. The method had a limit of detection of 0.2 µg g^{-1}. In 2022, Gupta et al. used dispersive liquid-liquid microextraction and diffuse reflectance-Fourier transform infrared spectroscopy for iodate determination in salt and food samples. The method had a limit of detection of 4.4 µg kg^{-1} [3].

In this study, an ultrasound-assisted deep eutectic solvent-based liquid-phase microextraction method combined with spectrophotometry has been developed for the determination of iodate. The method is based on the oxidation of iodide to iodine by iodate in the acidic media. The produced iodine reacts with the excess iodide to form triiodide ions. Triiodide is extracted into the DES phase (tetrabutylammonium chloride: decanoic acid, 1:3 molar ratio) in the presence of CTAB and was determined by a UV-Vis spectrophotometry at 355 nm. Under the optimized conditions, the method provided a detection limit of 17 µg L^{-1} and an enrichment factor of 250. The relative standard deviation at 140 µg L^{-1} concentration of iodate (n = 6) was found to be 2.4%. The method was applied for the determination of iodate in iodized salts.

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Simultaneous extraction and determination of Cd and Pb by syringe to syringe dispersive liquid phase microextraction-solidified floating organic drop combined electrothermal vaporization-inductively coupled plasma mass spectrometry

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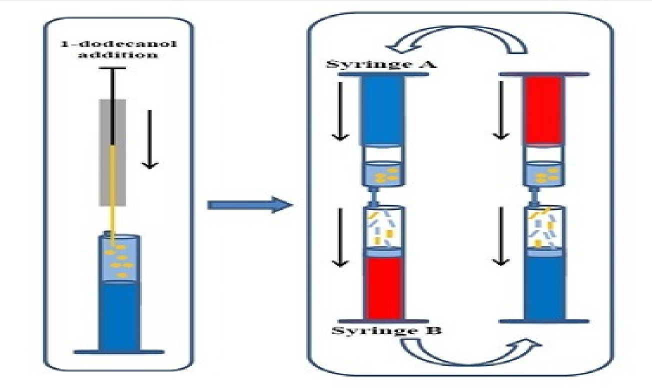

Abstract: A syringe to syringe dispersive liquid phase microextraction-solidified floating organic drop was induced and used for the ultra-trace simultaneous determination of cadmium and lead after extraction from environmental water samples. The extracted analytes will be determined by electrothermal vaporization (ETV)-inductively coupled plasma mass spectrometry (ICP-MS). The analytical parameters affecting the microextraction efficiency including the nature and volume of the extraction solvent, sample volume, pH, ionic strength, and concentration of reagents and the cycles of extraction will be optimized. Elements such as Cd and Pb have been widely studied and are usually present at low concentration in environmental, biological and food samples [1]. In the syringe-to-syringe DLPME (SS-DLPME) method, two disposal syringes are connected to each other through one needle. In this technique, the aqueous sample and the extraction solvent are placed in syringe 1 and then syringe 2 is connected to it. Then, the extraction solvent is dispersed in the aqueous sample through the consecutive injection between the two mentioned syringes. Also, in this published research [2], a new mode of DLPME that called syringe-to-syringe dispersive liquid phase microextraction-solidified floating organic drop (SS-DLPME-SFOD) is reported. In this method, SS-DLMPE-SFOD, a low toxic and low density organic solvent, such as 1-dodecanol is used as the extraction solvent with a melting point near the room temperature. This simplifies the solvent recovery at the end of extraction as the solvent solidified and floats on the top of the sample solution. This method is performed without a dispersive solvent, a vortex mixer or an ultrasound bath. It is well known that inductively coupled plasma mass spectrometry (ICP-MS) is the most favorable analysis tool for element and its speciation owing to its high sensitivity, wide linear dynamic range, and rapid multielement detection capability. In the present work, the SS-DLPME-SFOD is combined with ETV-ICP-MS for the preconcentration and simultaneous determination of Cd and Pb after the formation of the complex with 1-(2-pyridylazo)-2-naphthol. Various factors influencing separation, preconcentration and determination of the analytes will be investigated in detail. The purpose and applicability of this approach is to determine cadmium and lead in different water samples. As a part of the analytical results obtained for this method, we can refer to the calibration curves that were linear in the ranges of 0.01-22.00 ng L-1 and 0.02-25.00 ng L^{-1} with determination coefficients of 0.9975 and 0.9983 for Cd and Pb, respectively.

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Pertraction of Mo(VI) through a ternary polymer inclusion membrane composed of cellulose triacetate/trioctyl amine/tributyl phosphare

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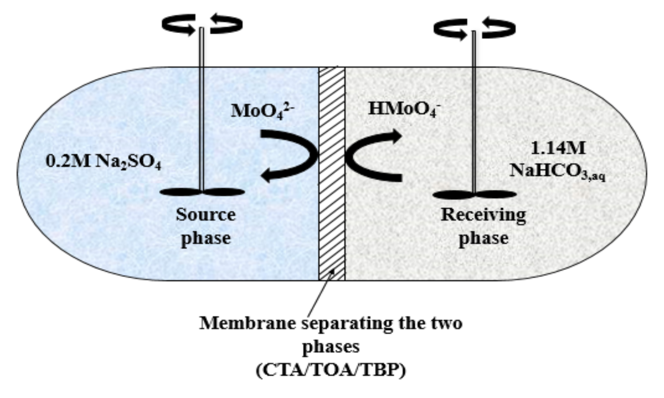
Graphical Abstract



Abstract: Even though SLM-based separation technique is an attractive alternative to conventional solvent extraction, these membranes suffer from poor stability. However, another type of liquid membranes called polymer inclusion membranes (PIMs) are known to be more stable than SLMs [1]. Beside the wide application of these membranes in separation studies, they have been shown to be very useful technique in analytical chemistry scopes [2]. In this communication, the potential of a tributyl phosphate (TBP) plasticized polymer inclusion membrane (PIM) including cellulose triacetate (CTA) base polymer and trioctylamine (TOA) as extractant has been appraised in the transport of molybdenum(VI) from sulfate solution, for the first time. The optimized membrane comprised of 40/25/35 wt% of CTA, TOA and TBP, respectively. It was transparent, flexible, homogenous, and mechanically stable. The optimized PIM has been employed in a two compartment pertraction cell for the transport-separation of Mo(VI) from acidic sulfate feed solution into the sodium hydrogen carbonate receiving phase. Under the optimized conditions (feed phase: pH 1.4, 0.2 M Na_2SO_4; receiving phase: 1.14 M $NaHCO_3$; stirring rate 650 rpm) a quantitative transport of Mo(VI) (initial concentration 20 mg L^{-1}) from the feed phase into the receiving phase has been achieved, after 24 h. It is reported that the main Mo(VI) species under the optimized feed phase conditions is H_2MoO_4 [3]. Thus, the reactions at the first and second interfaces can be respectively considered as those represented in Eqs. 1 and 2:

$$H_2MoO_{4,aq} + TOA_{PIM} \rightleftharpoons [TOAH^+][HMoO_4^-]_{PIM} \qquad (1)$$

$$[TOAH^+][HMoO_4^-]_{PIM} + 2NaHCO_{3,aq} \rightleftharpoons 2Na^+_{aq} + MoO_4^{2-}{}_{aq} + TOA_{PIM} + 2H_2O + 2CO_2 \qquad (2)$$

The highest permeability coefficient and initial flux of Mo(VI) were 5.9×10^{-6} m s^{-1} and 1.24×10^{-6} mol m^{-2} s^{-1}. The excellent selectivity demonstrated by the proposed system towards Mo(VI) in the presence of Zn(II), Co(II), Ni(II), Cd(II), Cr(III), Al(III), Cu(II), Bi(III), Fe(III), and V(V) species allowed considering the developed PIM for the recovery of Mo(VI) from spent catalysts used in hydrodesulphurization process in petroleum industries [4].

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Investigation of Some Effective Factors on the extraction of Uranium by Emulsion Liquid Membrane

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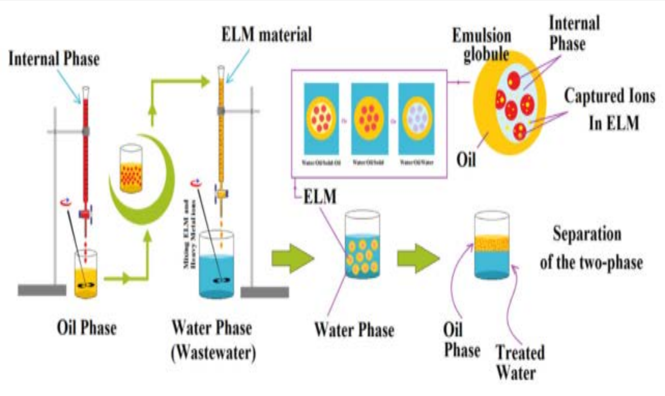

Abstract: In this study, the extraction of uranium from aqueous solution is studied using water-in-oil (W/O) emulsion liquid membrane (ELM) system. The ELM constitutes kerosene as an organic diluent, Di-(2-ethylhexyl) phosphoric acid (D2EHPA) as a carrier, sorbitan monooleate (Span-80) as a surfactant for stabilizing the emulsion phase, sulfuric acid as a stripping solution and potassium chloride (KCl) as a tracer [1]. Response surface methodology (RSM) based on central composite design (CCD) is carried out to explore the relationships between process variables namely initial pH of the donor phase, sulphuric acid concentration in the acceptor phase, surfactant concentration in the liquid membrane phase and mixing speed, and their interactions. The contours and 3D response surfaces of selective extraction of U were achieved [2].

The hydrodynamic parameters mentioned above were optimized for achieving the maximum uranium extraction with minimum emulsion breakage and swelling. The residence time required for adequate pertraction of uranium has been evaluated. The effect of other factors was also investigated on a case-by-case basis at points obtained on efficiency. The effect of the selected factors and the effect of their interactions were studied to obtain the optimal conditions. The results showed that the best extraction efficiency and emulsion stability is obtained at pH of donor phase=3; Span 80 volume percentage in the membrane phase= 2 % V/V; emulsion/donor phase volume ratio= 0.1; sulfuric acid concentration as a stripping solution is 0.6 molar, and mixing speed = 180 rpm. The role of pH as an important parameter in the ELM process for extraction of uranium was studied. With these conditions, the extraction efficiency of 99.923% was obtained within 15 min and the measured values of emulsion breakage and swelling were 0.083% and 0.424%, respectively.

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Optimization of Effective Parameters on the ligandless Extraction of Thorium (IV) Ions from Aqueous Solutions by Emulsion Liquid Membrane

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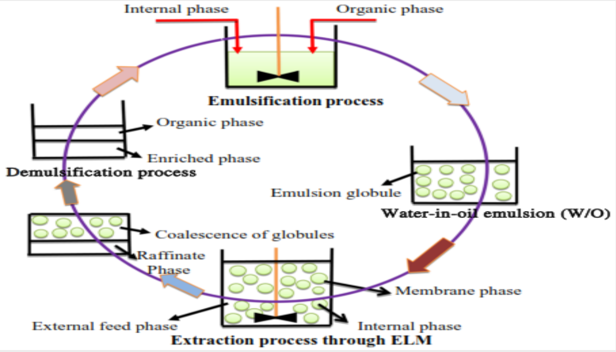

Abstract: The present paper introduces a technical feasibility study of one of the most advanced extraction techniques; emulsion liquid membrane (ELM) on the selective and very efficient transport of thorium (IV) ions from aqueous solutions. The study aims to give a comprehensive account of various parameters that affected the ELM formation and stability for increasing the extraction of thorium. In this work, the transport of thorium from aqueous solution occurs through the water-in-oil (W/O) emulsion liquid membrane that constitutes of kerosene as an organic diluent and sorbitan monooleate (Span-80) as a surfactant for stabilizing the emulsion phase, sulphuric acid as a stripping solution and potassium chloride as a tracer. The carrier is not used here for emulsification and for the transport of thorium. The influence of pH of the external aqueous phase, the concentration of the acid used in the receiving phase and the mixing speed on the process efficiency were examined and discussed with response surface methodology (RSM) based on central composite design (CCD) [1-4]. The hydrodynamic parameters mentioned above were optimized for achieving the maximum thorium extraction with minimum emulsion breakage and swelling. The optimized conditions showed that the best extraction efficiency and emulsion stability is obtained at pH of donor phase=5; Span 80 volume percentage in the membrane phase= 4 % V/V; the sulphuric acid concentration as a stripping solution is 0.6 molar; emulsion/donor phase volume ratio= 0.1, and the effect of volume ratios of the internal phase to the organic phase 1:1 (A/O) and mixing speed = 180 rpm. The role of pH as an important parameter in the ELM process for extraction of thorium was studied. With these conditions, the extraction efficiency of 97.84% was obtained within 15 min and the measured values of emulsion breakage and swelling were 0.051% and 0.260%, respectively.

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Separation and Determination of Diethyl phthalate in Water Samples Based on STA-16(Fe) as an Efficient Sorbent

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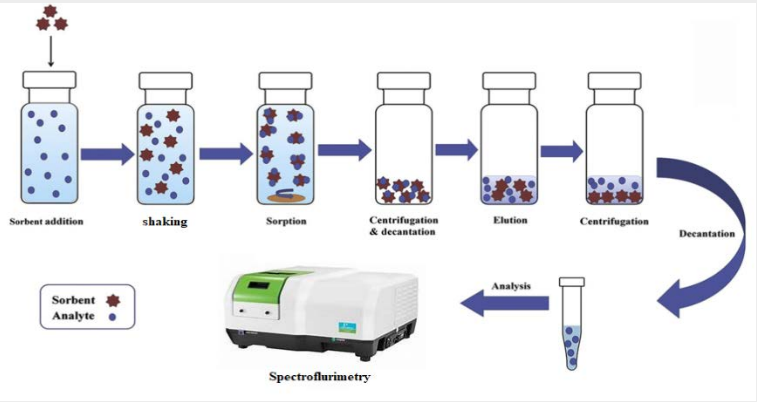
Graphical Abstract



Abstract: Diethyl phthalate (DEP) belongs to the family of phthalates or phthalic acid esters (PAEs). Diet is considered as the main source of PAE exposure [1, 2]. PAEs are known to be potential endocrine disruptors, accumulate in tissues, and are not well degradable. Therefore, determination of DEP is an important analytical task. In this study, STA-16 (Fe) was synthesized [3] and characterized by Fourier-transform infrared (FT-IR) spectroscopy and X-ray diffraction (XRD) analysis. STA-16 (Fe) as adsorbent, in dispersive micro-solid phase extraction (Dµ-SPE) as green, simple and efficient method was used for trace quantification of diethyl phthalate. Four independed parameters of pH, adsorbent amount, temperature and ultrasonic time were selected as factors influencing the process and optimized by central composite design (CCD) [4]. In Dµ-SPE method, a solution containing the appropriate amount of DEP was placed in a 30 mL centrifuge tube. The pH of the sample solution was adjusted to 7, then the adsorbent was dispersed in the sample solution by ultrasonic device and the adsorbed analytes were eluted with a 0.5 mL of acetonitrile-nitric acid 65% 0.2 M (50/50 ratio). By separating the adsorbent from the solution the analyte solution is transferred to the spectrofluorimetry cell. Under optimal conditions, linear range of 25-550 ng mL^{-1}, detection limit of 7.33 ng mL^{-1} and relative standard deviation (R.S.D) of 0.36% were obtained for the DEP. Finally, the proposed method was successfully used for preconcentration and determination of DEP in water samples.

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Deep Eutectic Solvent-Based Liquid-Liquid Microextraction Method for Separation and Determination of Tartrazine in Food Sample

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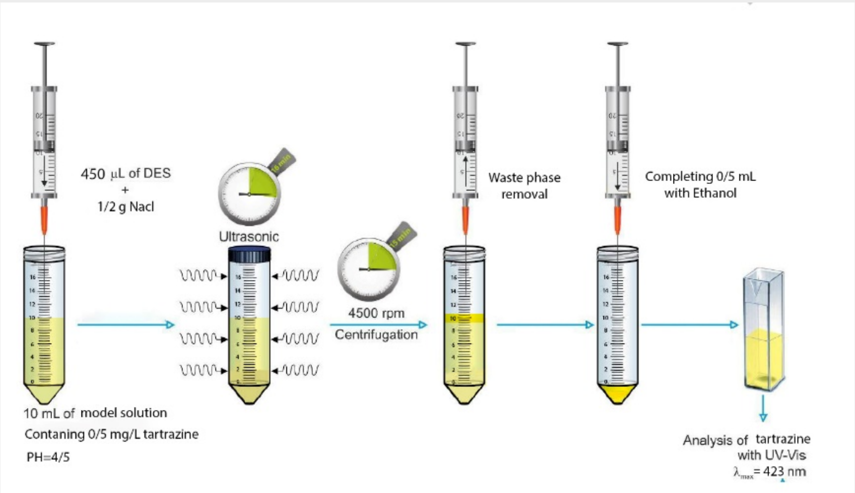
Graphical Abstract



Abstract: Tartrazine is an azo dye used in soft drinks, jellies, juices and other food products. It can cause many health problems including hyperactivity in children, allergies, migraine, asthma and eczema. Therefore, the determination of tartrazine in food samples in order to control and prevent the mentioned complications is very important. This study reports the application of deep eutectic solvents (DES), as a new generation of environmentally friendly solvents for liquid-liquid microextraction (LLME) of tartrazine in food samples [1, 2]. DES was formed by mixing of choline chloride and phenol with 1:4 molar ratio. Four parameters of pH, DES volume, ultrasonic time and amount of salt were selected as effective factors on the process and optimized by central composite design (CCD) [3]. In LLME-DES method, a certain amount of tartrazine was transferred to a 10 mL test tube. The pH of the analyte solution was adjusted to 4.5 by HCl and NaOH. Then, 450 μL of DES and 1.2 g salt (NaCl) was added to the test tube to perform the extraction process. The solution was placed in an ultrasonic bath at 45°C for 16 min. After centrifugation for 15 minutes, two aqueous and DES-rich phases were formed. The aqueous phase was removed using a syringe and the DES-rich phase remaining at the end of the test tube was diluted with 0.5 mL of ethanol to be measured by UV-Vis spectrophotometry at 423 nm. The analytical curve obtained for tartrazine was in the linear concentration range of 10 to 600 ng mL^{-1}, with limit of detection 2.89 ng mL^{-1} and the relative standard deviation (RSD) 4%. The proposed method was successfully applied for preconcentration and determination of tartrazine in food samples.

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Trace Quantification of Methyl Paraben in Cosmetic Samples Based on Fe-MIL-NH_2 Functionalized With Isatin Schiff Base

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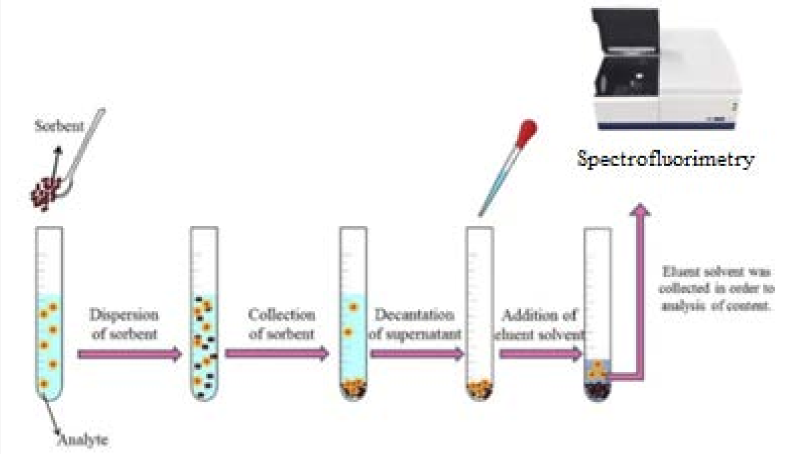

Abstract: Preservatives are substances used to enhance the quality or nutritional value of health, pharmaceutical and food flavors, and to prevent them from being broken down by microorganisms [1]. Methyl paraben and propyl paraben are effective antibacterial and antifungal agents that are commonly used as preservatives in food, beverages, cosmetics and pharmaceuticals [2]. Parabens are considered as an ideal preservative due to their antimicrobial activity, high resistance to pH changes, relatively safe use and low production cost. Due to the benefits of preservatives, they can cause problems such as disorders of the immune system and endocrine glands. Therefore determination of parabens is important [3]. In this study, Fe-MIL-101-NH_2 functionalized with isatin Schiff base was synthesized and characterized by Fourier transform infrared spectroscopy (FT-IR) and X-ray diffraction (XRD) analysis. Dispersive micro-solid phase extraction (Dμ-SPE) was used as a green, simple and efficient method for trace quantification of methyl paraben (MP). Four independent parameters of pH, adsorbent amount, temperature, and ultrasonic time were selected as factors influencing the process and optimized by central composite design (CCD) [4]. In Dμ-SPE method, a solution containing the appropriate amount of MP was placed in a 30 mL centrifuge tube. The pH of the sample solution was adjusted to 7, then the adsorbent was dispersed in the sample solution by ultrasonic device and the adsorbed analytes were eluted with a 0.7 mL of acetonitrile and transferred to the spectrofluorimetry cell. Under optimal conditions, linear range of 35-650 ng mL^{-1}, detection limit of 9.19 ng mL^{-1} and relative standard deviation (R.S.D) of 3.5% were obtained for the target analyte. Finally, the proposed method was successfully used for preconcentration and determination of methyl paraben in cosmetic samples.

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Synthesis of Fibrous Adsorbent Consisting of Two Types of Cu-MOF and Pd-MOF Using Electrochemical Method and Galvanic Replacement for Use in Fiber Solid Phase Micro-Extraction of Paraben Derivatives from Cosmetic and Their Measurement by HPLC

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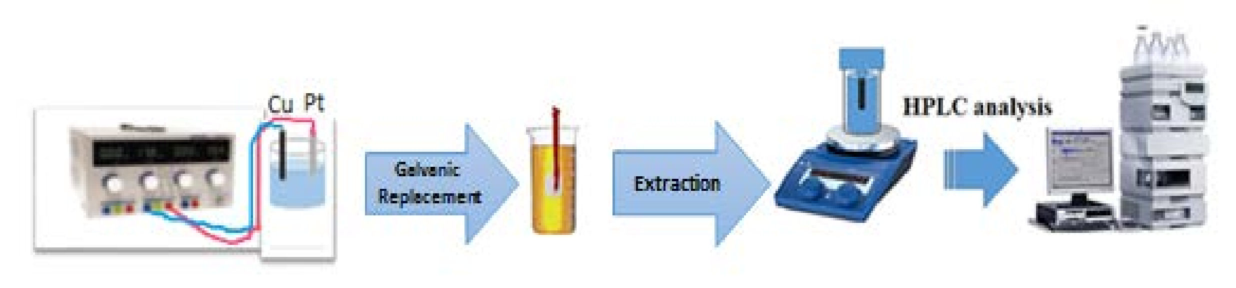

Abstract: The main aim of in this dissertation, we will try to place the porous copper foam on the copper substrate by electrochemical method [1], and finally, by replacing it in Pd2 + solution during the galvanic displacement method, a part of Cu on the surface will be displaced with Pd. Finally, by putting the fiber containing Pd and Cu in 2-hydroxypyrimidine solution (as a organic ligand) and synthesizing in aqueous solution, the Cu-MOF and Pd-MOF organic-metal framework is created on the surface. The porous foam mold as a substrate can create a polyhedral shape in the metal-organic framework. In addition, one of the special features of this MOF is that unlike other MOFs, it is not sensitive to moisture and is easily synthesized in aqueous medium, and finally the synthesized adsorbent will be used to extract the desired experimental species. The prepared modified adsorbent was used as the extracting fiber in the solid micro phase extraction (SPME) of selected paraben derivatives from cosmetic. The extracted analytes were eventually injected into the HPLC-UV for quantitative measurement and determination. In order to reach the maximum efficiency and obtain the optimal conditions for extraction of the selected analytes, one-at-a-time optimization was used. Based on the obtained results and the small RSD% (4.6%) for the method presented, it can be concluded that this absorbent has a high ability to pre-concentrations and selective extractions of testing analytes in cosmetic samples.

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Application of Zinc based Metal-Organic Framework reinforced porous hollow fiber for Solid Phase Micro Extraction of Oxyfluorfene and Ioxinyl Toxins from Environmental samples and Quantification via HPLC-UV

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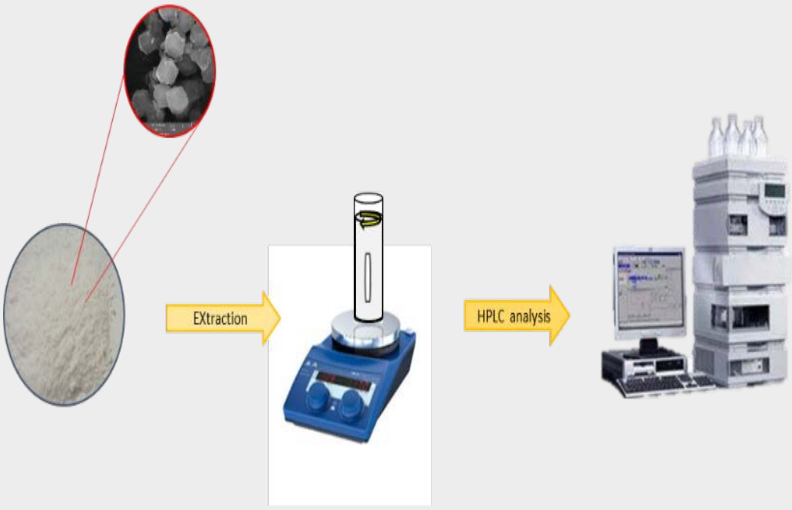

Abstract: Zeolitic imidazolate framework 8 (ZIF-8) reinforced hollow fiber solid phase microextraction combined with high performance liquid chromatography (HPLC) was used for the extraction and measurement of selective analytes including: ioxynil and oxyfluorfen in samples of garlic, onion and agricultural soil. The pores of the polypropylene fiber walls were filled by adsorbent with a simple, easy to use method and a very short time. The modified fiber was used to extract selective analytes and the extracted analytes were then eluted by using acetonitrile as the desorption solvent. The eluent was finally injected into HPLC-UV. In order to achieve the highest extraction efficiency for the selected analytes in the HF-SPME method, several parameters including extraction time, type of eluent, stirring rate, solvent volume and pH of sample solution were investigated for optimization [1]. In order to perform the optimization process, Plackett–Burman design (PBD) was performed. The Box Behnken Design (BBD) method was used to identify the optimal point for the influencing parameters affecting the extraction efficiency [2]. According to the results of optimization, the relative standard deviation (RSD%) of the method for selected analytes was obtained lower than 5.5% [3]. Finally, the method was successfully applied for the determination of the selected analytes in different types of garlic, onion and agricultural soil samples. Based on the results, it can be stated that the method has the ability of the extraction of the intended analytes in real samples.

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Highly effeicient removal of diazinon and malathione pesticides from fruit sample by using Cu doped Zeloitic Imidazolate Framework 8 (ZIF-8)

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Graphical Abstract

Abstract: The ZIF-8 substrate was constructed using the Solvothermal method with the central Zinc metal, and the Cu metal doping procedure was performed on the ZIF-8 substrate during the galvanic replacement process. The selected organophosphorous pesticides (OPPs), including diazinon and malathion, were extracted and determined in different types of real samples, including cherry, banana, and grape samples, and also agricultural water, using the adsorbent made during the process reinforced hollow fiber solid phase micro extraction combined with high performance liquid chromatography (HPLC) [1]. Pesticides, such as organochlorines, rodenticides, herbicides, fungicides, and fumigants, are used to prevent, eradicate, repel, or alleviate pests. OPPs, which from to the pesticide family, are the most widely used insecticides on the planet [2]. In this study, ZIF-8 was synthesized using a simple solvothermal approach, and the doping procedure was carried out using the galvanic replacement method. The resultant substance was then utilized as an adsorbent in the HF SPME procedure to extract relevant analytes. Select analytes were extracted using Cu@ZIF-8 metal doping absorbent, desorbed to methanol, and analyzed using HPLC UV in this methodology. In order to attain the maximum extraction efficiency, library studies and initial experiments are being used to assess the parameters affecting the extraction process. These parameters were already evaluated using the Placket Burnman experimental design, and then optimized using the Box Behnken Design, with optimal points established for each of the parametersThe relative standard deviation (RSD) of the procedure for diazinon and malathion was reported to be (2.7% - 0.7%) under optimal circumstances. Finally, based on the findings, it was demonstrated that the proposed approach has a high ability to extract and analyze selective analytes in a wide range of real samples [3].

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Mesoporous organosilicas with tyrosine framework as extractive phases for different pharmaceutical groups in biological samples prior to high performance liquid chromatography

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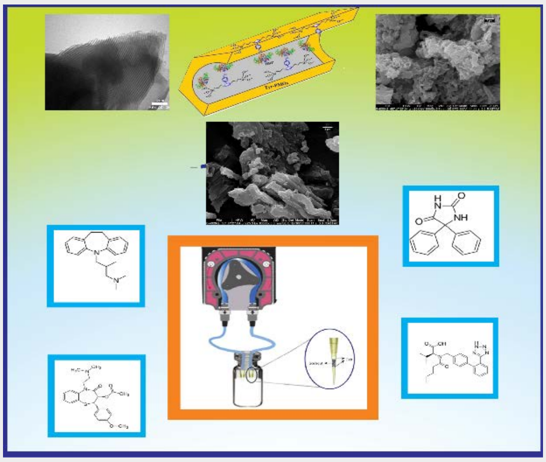
Graphical Abstract



Abstract: Sample preparation techniques are usually required prior to the instrumental analysis because of low samples concentrations and their complexity [1]. Mesoporous silica can be considered as promising materials in extraction due to their tunable pore size or shape, high surface area, thermal stability, and biocompatibility. Moreover, their performance can be improved via functionalization with various organic groups [2]. In this research, tyrosine, a natural abundant amino acid, was incorporated within the channels of mesoporous materials through three ways of grafting, co-condensation, and periodic mesoporous organosilicon (PMO). The applicability of the fabricated structures in pipette tip-µ-solid phase extraction (PT-µ-SPE) of different pharmaceutical groups such as anti- inflammatory, cardiovascular, antipsychotics, antiepileptics and antidepressants drugs were examined in conjunction with reversed phase liquid chromatography with UV detection. Amino acid-bridged periodic exhibited the best performance due to homogeneously distribution of organic unit mesoporous in the framework without any blockage of mesoporous channels. In the following, PMO materials were synthesized by varying the concentration of the tyrosine- based precursor in the initial mixture. The prepared structures were confirmed by NMR, FT-IR, MS, small-angle XRD, N_2 adsorption–desorption, TEM, SEM and EDX analysis. Under optimal conditions, the analytical performance of the developed method was also evaluated and precision, accuracy, and other figures of merit for selected analytes were achieved.

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Combination of Magnetic Dispersive Solid Phase Extraction and Liquid–Liquid Microextraction Followed by Gas Chromatography–Mass Spectrometry as an Efficient Analytical Method for the Quantification of Some Antidepressant Drugs in Biological Fluids

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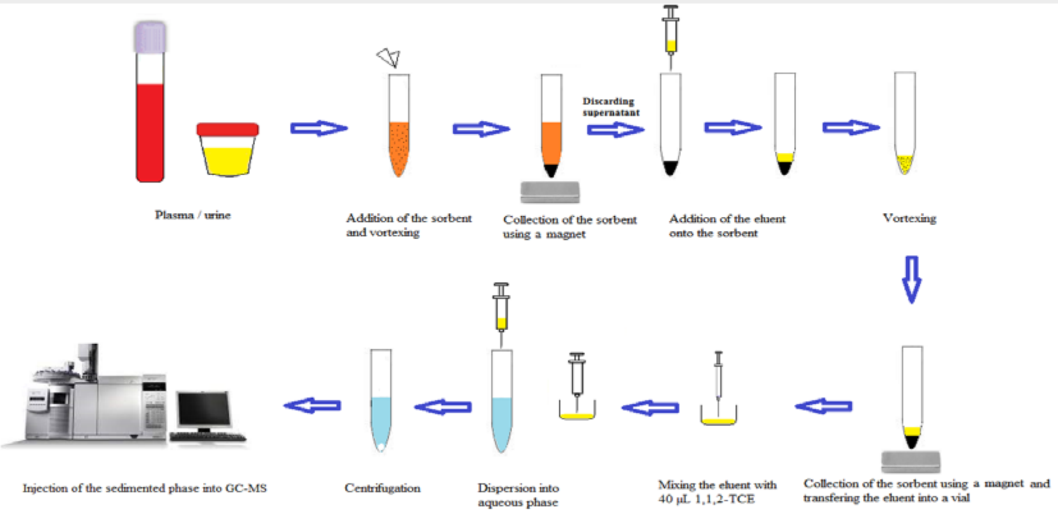

Abstract: The term depression describes a mood disorder that causes a strong sense of sadness and a lack of interest. Various factors are contributing to the increase in antidepressant drug consumption today, including economic stress and emotional problems [1]. A therapeutic drug monitoring program is important in the clinical pharmacology and forensic sciences since it can aid in the effective control of pharmacotherapy and drug poisoning. This point can be more important in the case of anti-depressant drugs (ADDs) because of their narrow therapeutic range [2]. Different methods are used to determine ADDs in different samples including high performance liquid chromatography, gas chromatography, and ion mobility spectrometry. This study developed a magnetic sorbent for the extraction of four ADDs, including fluoxetine, amitriptyline, imipramine, and clomipramine. In order to extract the target compounds from plasma and urine, the magnetic Fe_3O_4 nanoparticles coated with graphene were used as sorbent. First, a solution containing analytes and dissolved Na_2SO_4 was used to adsorb the tested ADDs on the sorbent. By using an external magnetic field, the sorbent particles containing the adsorbed analytes were separated from the solution. Then acetonitrile was used to desorb the analytes from the sorbent. The eluent containing analytes was separated from the sorbent, mixed with 1,1,2-trichloroethane (at µL level), and quickly injected into an ammonia solution containing dissolved NaCl. After centrifuging the formed cloudy solution, an aliquat of the sedimented organic phase was injected into a gas chromatograph equipped with a mass spectrometer.

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Separation of the racemic mixture of carvone using drift gas modifier by ion mobility spectrometry

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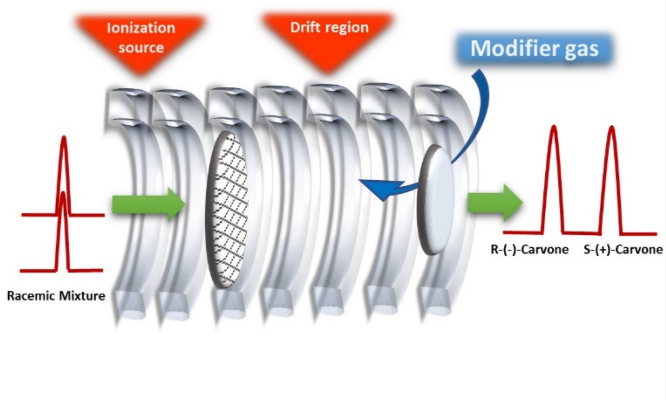
Graphical Abstract



Abstract: Ion mobility spectrometry (IMS) is an analytical technique that can separate gas-phase ions at atmospheric pressure. To this end, isomer separation (for structural and optical isomers) is a main advantage of IMS, while mass spectrometry fails for it. Enantiomer mixtures have similar physicochemical properties while they have different metabolic properties, so their separation is very important for their analysis. Here, the racemic mixture of carvone was separated by a drift gas modifier for qualitative and quantitative analysis using IMS [1,2]. Tert-butanol was selected as the modifier gas to separate the enantiomers mixture, and some effective parameters, such as flow rate of drift and modifier gas, and cell temperature were optimized. These parameters affect the ion cluster formation and collision cross-sections, influencing on the resolution between IMS isomers ion peaks. at the optimum condition, high resolution between R and S enantiomer of carvone was observed with a time difference of about 2 ms. The calibration curves were plotted using their standard solutions, and the linear dynamic ranges were obtained 50-500 mg. L^{-1} for both isomers.

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Syringe to syringe-dispersive liquid–phase micro extraction combined with UV-Vis spectrophotometry for pre concentration and determination of acetaminophen & codeine phosphate with the aid of experimental design

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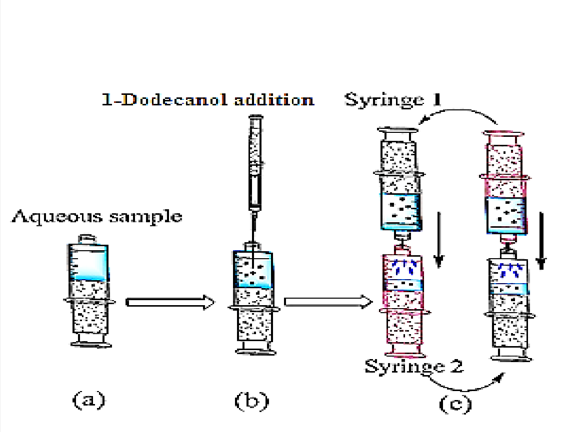

Abstract: This study was aimed at developing a new, technically simple method for pre concentration and determination of *acetaminophen* and *codeine phosphate combination* by syringe to syringe-dispersive liquid–phase micro extraction *technique* combined with UV-Vis spectrophotometry method and compared with common brand names include tylenol and panadol. The study assessed factors affecting on the extraction technique, extraction solvent [1], extraction solvent volume [2], pH of the solvent, effect of sample pH and centrifugation time, ionic strength on the extraction of target analytes, extraction time, effect of syringe to syringe numbers, temperature, sample to solvent ratio, sample characteristics and are the defining factors that effect extraction efficiency, effect of ionic strength on the extraction of target analytes, effect of syringe to syringe numbers, also effective factors were screened using a response surface methology (RSM) and optimized using a I-Optimal. Under the optimized experimental conditions, the calibration curve was a dynamic linear in the range for the assay of acetaminophen (5.0 – 25.0 mg L-1) and codeine phosphate (1.25 – 10.0 mg L-1) and the limit of detection (LOD) for the acetaminophen (0.15 mg L-1) and for the codeine phosphate (0.26 mg L-1) and limit of quantification (LOQ) for the acetaminophen (0.50 mg L-1) and for the codeine phosphate (0.87 mg L-1) with R2 = 0.9991 for acetaminophen and R2 = 0.9995 for codeine phosphate respectively. The absorption maxima of drugs in mixture were found to be at 243 nm for the both of acetaminophen and codeine phosphate respectively, in 1-dodecanol as extraction solvent. The proposed method was applied to the determination of codeine phosphate and acetaminophen in raw material and solid dosage forms (powder and granules tablets and capsules) and urine samples.

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Magnetic Dispersive Solid Phase Extraction of Some Polycyclic Aromatic Hydrocarbons from Honey Samples Using Iron (III) Oxinate Nanocomposite as an Efficient Sorbent

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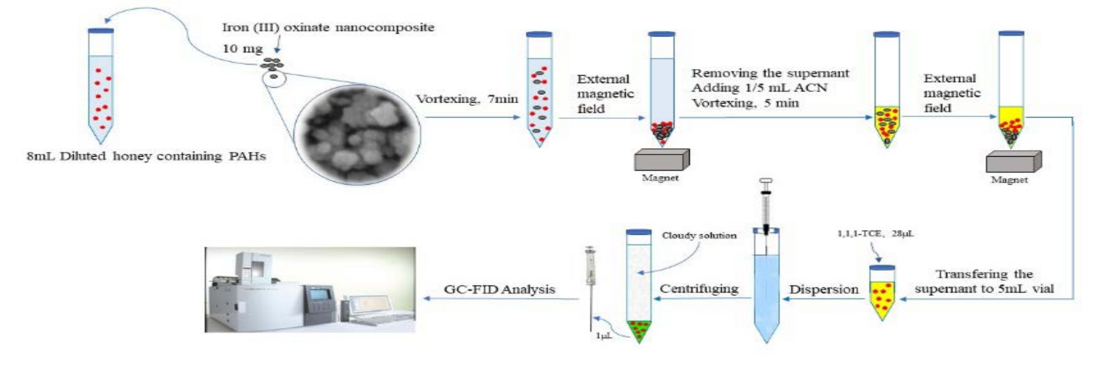

Abstract: Honey is a natural food product that is widely used by human for various purposes. This biologically active product contains important compounds such as minerals, vitamins, sugars, enzymes, proteins, antioxidants, and organic acids. Therefore, it has high nutritional and therapeutic properties [1–2]. In this work, iron (III) oxinate magnetic nanocomposite was synthesized and employed as an efficient sorbent for magnetic dispersive solid phase extraction of some polycyclic aromatic hydrocarbons from honey samples. Further preconcentration of the analytes was accomplished using dispersive liquid-liquid microextraction. The prepared sorbent was characterized using Fourier transform infrared spectrophotometer, X–ray diffractometer, vibrating sample magnetometer, energy dispersive X–ray, and scanning electron microscope. These analyses verified the successful formation of the magnetic sorbent. In the extraction process, briefly, the sorbent was added into an aqueous solution containing the analytes and vortexed well. After completing the adsorption process, the supernatant phase was separated in the presence of a magnet and the analytes loaded on the adsorbent were eluted by acetonitrile. Then, µL–level of 1,1,1–trichloroethane was mixed with the obtained acetonitrile phase and injected into NaCl solution. One microliter of the sedimented phase after centrifugation was injected into gas chromatograph–flame ionization detector. Under the best extraction conditions, a great repeatability (relative standard deviation equal or less than 5 and 6% for intra– and inter day precisions, respectively), acceptable extraction recoveries (59–84%), high enrichment factors (118–168), and low limits of detection and quantification (0.16–0.36 and 0.56–1.22 ng g^{-1}, respectively) were acquired.

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Introduction of A New Polymer Inclusion Membrane Based Upon Mixed Cellulose Triacetate/Poly Vinylidene Fluoride-*CO*-Hexafluoropropylene Polymers and Trioctylamine Extractant for the Extraction of Bi(III) From Chloride Solutions

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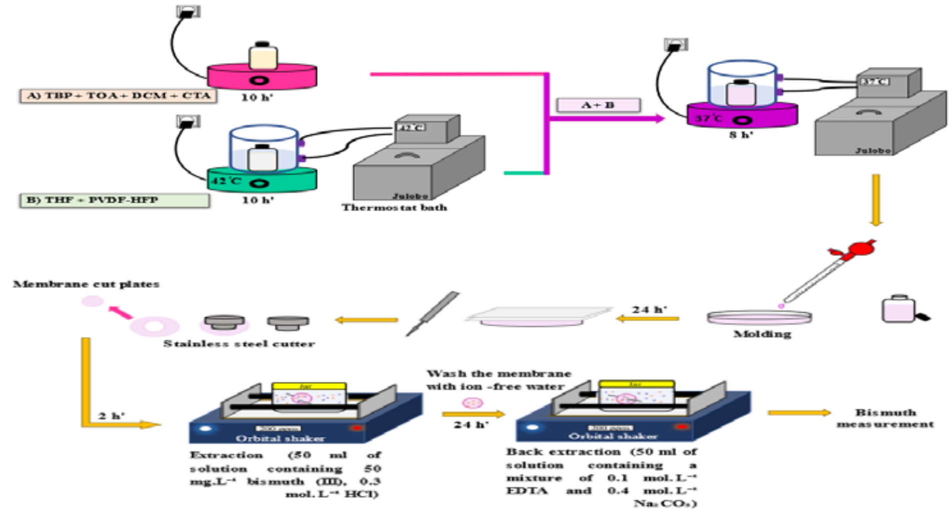
Graphical Abstract



Abstract: In the present study, initally several types of blend polymer inclusion membranes including CTA, PVDF, PVC and PVDF-HFP polymers containing tri octylamaine (TOA) extractant as well as nitrophenyl octyl ether (NPOE), dibutyl phthalate (DBP), tris(2-ethylhexyl) phosphate (TEHP), diorctyl phthalate (DOP) and tributyl phosphate (TBP) as plasticizer were prepared in different weight ratios. A membrane with 30 wt% of polymer CTA, 35 wt% by weight of polymer PVDF-HFP, 25 wt% of extractant TOA and 10 wt% of modifier TBP with suitable physical, mechanical and extraction properties was selected as the optimal membrane. The physical and mechanical properties of this membrane were investigated by measuring the contact angle, studying the elastic properties scanning electron microscopy, atomic force microscopy and thermogravimetric analysis [1, 2]. The studied membrane was capable of complete and selective extraction of Bi(III) ions from 50 ml of solution containing 20 mg.L^{-1} bismuth ion in 0.3 mol.L^{-1} hydrochloric acid solution for 120 minutes. By studying the stoichiometry of the extracted species, the mechanism of ion pair formation $(TOAH^{+})2$ $(BiCl_5^{2-})$ was confirmed. Complete Back extraction of the extracted ions was performed using a combination of 0.4 mol L^{-1} sodium carbonate and 0.1 mol L^{-1} ethylene diamine tetra acetic acid. The prepared membrane can be used for four continuous extraction and Back extraction steps without reducing the efficiency. Kinetic and isothermal studies of the extraction process were investigated and the results of these studies showed that the extraction data were well consistent with the quasi-second order model and proved the chemical adsorption of bismuth ions [2].

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Preparation of a Novel Polymer Inclusion Membrane Based on Industrial PVDF Polymer for the Extraction of the Bismuth(III)

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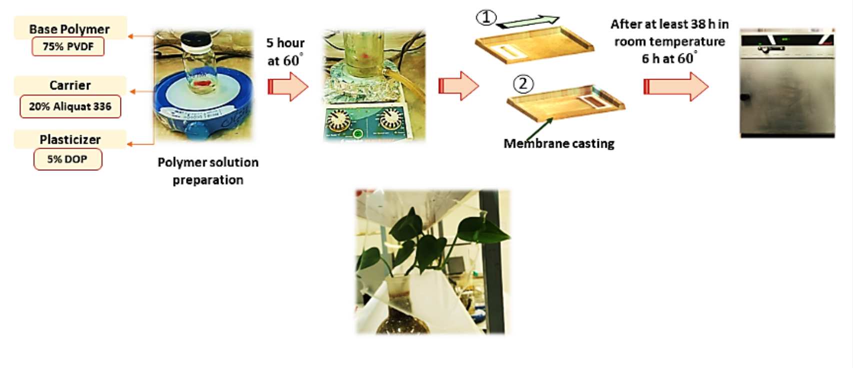

Preparation of polymer inclusion membrane by casting knife method

Abstract: Bismuth and its compounds have various medical and industrial applications. The abundance of bismuth ores is low therefore, the extraction/recovery of bismuth ions from other resources such as industry effluents is economically important [1]. Various techniques such as precipitation, ion exchange, solvent extraction, adsorption and filtration membrane was employed for separation/ extraction of the metal ions from wastewaters [2]. Among these methods membrane-based processes have attracted attention as a valuable technology. Polymer inclusion membranes (PIMs) are a type of liquid membranes which have been widely used for purifying wastewaters, due to this method is environmentally friendly, high reusability, lower cost, selective and provide extraction and back extraction simultaneous [3]. PIMs are thin films that are formed by mixing an extractant, a base polymer and sometimes a plasticizer, in an organic solvent [4].

In this work, polymer inclusion membranes based on industrial PVDF polymer and tricaprylmethylammonium chloride (Aliquat® 336) as ionic liquid extractant and dioctyl phthalate (DOP) as plasticizer by casting evaporation method were prepared. The PIM including 75 wt% PVDF, 20 wt% Aliquat® 336 and 5 wt% DOP selected as optimum composition because of its suitable physical and mechanical properties. Bismuth extraction experiments were performed by immersing a 3.5 cm diameter round disk, with average mass 0.070±0.008 g and average thickness 49.7±5.7 µm, of the PIMs in a chloride solution containing 20 mg L^{-1} of Bi(III). The results showed that the PIMs can efficiently extract Bi(III) from 50 mL solution containing 20 mg L^{-1} of Bi(III) and 0.2 mol L^{-1} hydrochloric acid.

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Identification and measurement of active ingredients of laurus nobilis leaf and antibacterial effects by MIC, MBC and disk diffusion methods

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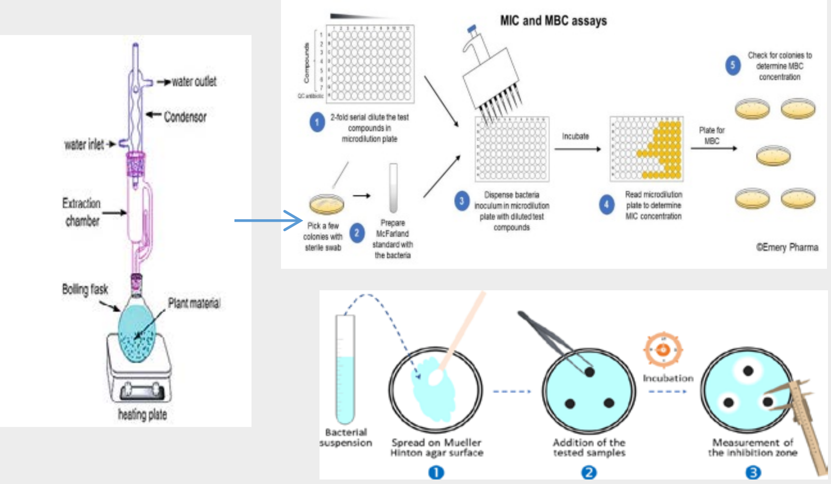

Abstract: Laurus nobilis L. from the Lauraceae family is one of the medicinal plants with many active ingredients whose extract has good antioxidant, antibacterial, antifungal and anti-inflammatory properties and can be used as a natural antioxidant or alternative to antibiotics in the treatment of some diseases. In this study, Laurus nobilis L. was collected from the north of Iran, dried and extracted with ethanol by Soxhlet apparatus, then poured on a watch glass for 48 hours to evaporate the solvent.The prepared dry samples were applied to disk diffusion test with concentrations of 100 mg.ml^{-1}, 50 mg.ml^{-1}, 25 mg.ml^{-1}, 12.5 mg.ml^{-1}, 6.25 mg.ml^{-1} and 3.125 mg.ml^{-1} in the culture medium containing BHI Agar. Also the tests of Minimum Bacteriocidal Concenteration (MBC) and Minimum Inhibitory Concenteration (MIC) were carried with concentrations of 400 mg.ml^{-1}, 200 mg.ml^{-1}, 100 mg.ml^{-1}, 50 mg.ml^{-1}, 25 mg.ml^{-1}, 12.5 mg.ml^{-1}, 6.25 mg.ml^{-1}, 3.125 mg.ml^{-1}, 1.56 mg.ml^{-1} and 0/78 mg.ml^{-1} in Broth culture medium containing BHI. The results were investigated after 24 hours of incubation. The amount of MIC effect of ethanolic extract of Laurus nobilis L. on Staphylococcus epidermis was 0.78 mg.ml^{-1} and 6.25 mg.ml^{-1} was obtained for Escherichia coli. Also in 3 higher concentrations, lethal property was obsereved for MBC method. According to the results, ethanolic extract of Laurus nobilis L. showed a better inhibitory and lethal effect on gram-positive bacteria Staphylococcus epidermis in comparison with gram-negative bacteria Escherichia coli. Finally, the ethanolic extract of Laurus nobilis L. can be used as raw material in the manufacture of cosmetic products due to its inhibitory and lethal properties of skin microorganisms.

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Extraction and identification of TNT Explosive Compound by Dispersive Liquid-Liquid Microextraction Combined with Silica Aerogel Thin Film Followed by Thermal Desorption Unit-Ion Mobility Spectrometry

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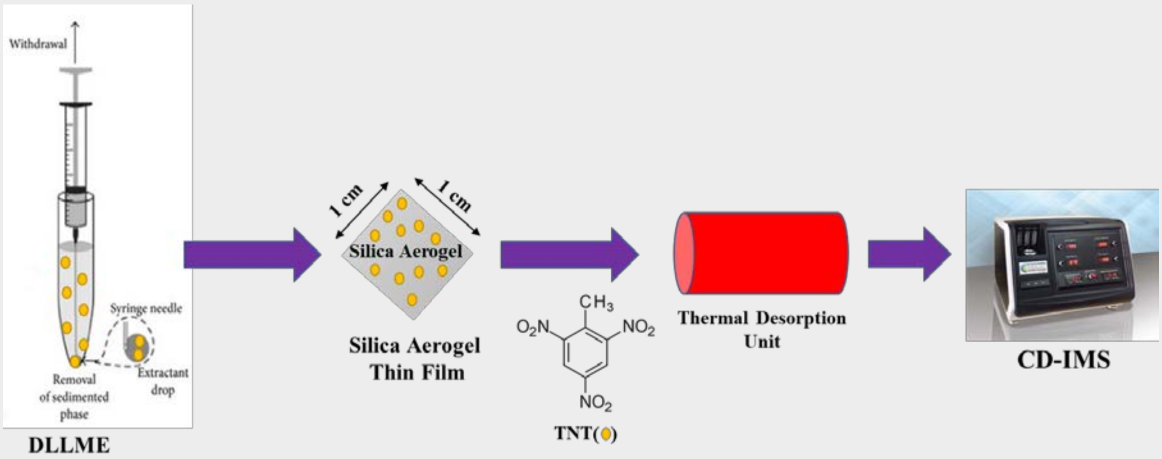

Abstract: Silica aerogel thin film prior to dispersive liquid-liquid microextraction method was applied for extracting and preconcentrating of trinitrotoluene (TNT) explosive compound. To evaporate and detection of the extracted TNT obtained by proposed method, ion mobility spectrometer equipped with thermal desorption unit was used. Electrospinning procedure was done for preparing of the thin film. Different effective parameters including extraction solvent, disperser solvent, centrifugation time, and evaporation time from thin film were optimized to increase the trace concentration of the TNT and the cleaning-up. Under the optimized conditions, the calibration curve was plotted by least square method and the linear dynamic range (LDR) as well as correlation coefficient were obtained 10-200 μg L^{-1} and 0.9970, respectively. According to the signal to noise ratio (S/N) 3 and 10, the limit of detection (LOD) and limit of quantification (LOQ) were calculated to be 3 μg L^{-1} and 10 μg L^{-1}, respectively. To investigate the repeatability of the proposed method, the relative standard deviation (RSD) was calculated 2% for the TNT concentration of 50 μg L^{-1}. To assess the ability of the method for extraction and identification of the analyte in the real sample, agricultural wastewater was used and the relative recovery value was obtained to be 114%.

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High throughput thin film microextraction of antifungal drug by highly porous polyvinylidene fluoride membrane sheet

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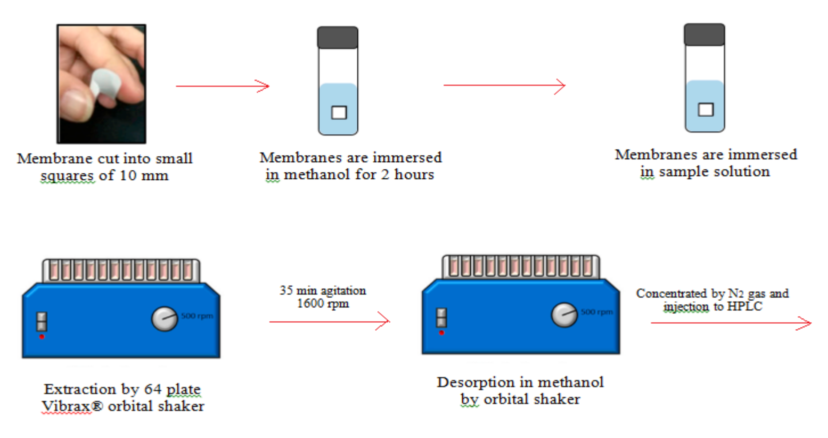

Abstract: In this study, highly porous polyvinylidene fluoride (PVDF) membrane sheet was introduced as extracting phase in high-throughput thin film microextraction (TFME) for the rapid and simultaneous extraction of three antifungal drug, including ketoconazole (KZ), clotrimazole (CZ), and miconazole (MZ) in aqueous samples (water, waste water and urine samples) prior to liquid chromatographic determination. The proposed thin film membrane was characterized by FE-SEM, AFM, ATR and BET. Highly porous thin film membrane was prepared by non-solvent induced phase inversion method and pore generation using Polyvinylpyrrolidone (PVP). Optimum extraction/desorption conditions were found as: Salt concentration: 4%w/v NaCl, stirring speed: 1400 rpm, pH 10, extraction temperature: 25 °C, extraction time: 40 min, desorption solvent: methanol and desorption time: 10 min. The figures of merit were obtained under the optimized conditions: the Linear Dynamic Ranges (LDRs): 6.70-100 μg L^{-1}, 4.18-100 μg L^{-1} and 10.32-100 μg L^{-1}, Limits of Detection (LODs): 1.38 μg L^{-1}, 2.21 μg L^{-1} and 5.42 μg L^{-1}, Limits of Quantification (LOQs): 6.70 μg L^{-1}, 4.18 μg $L^{-1}$and 10.32 μg L^{-1} for KZ, CZ and MZ respectively.

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High throughput thin film microextraction of anticancer drug by covalent organic framework based mixed matrix membrane

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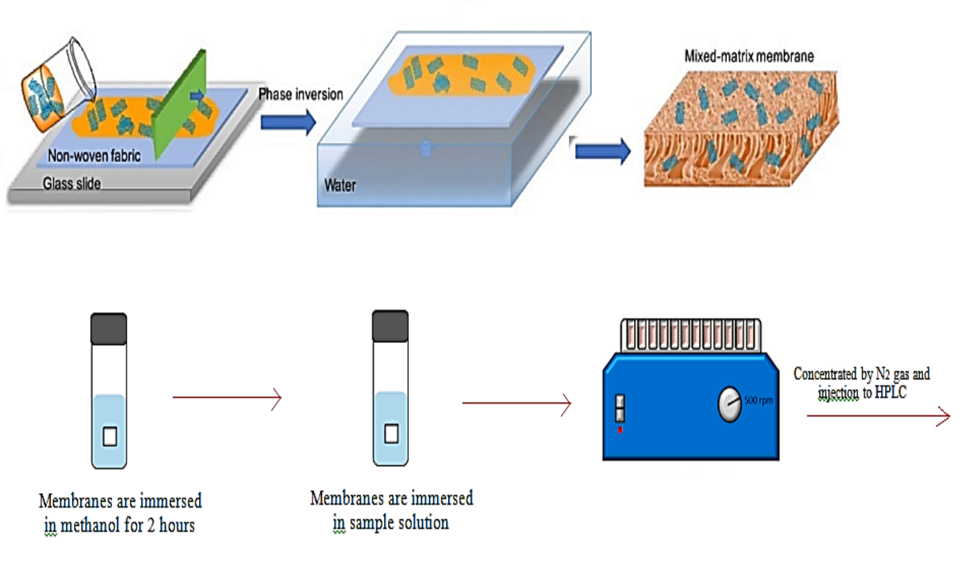
Graphical Abstract



Abstract: In this study, covalent organic framework based mixed matrix polyvinylidene fluoride (PVDF) membrane sheet was introduced as extracting phase in high-throughput thin film microextraction (TFME) for the rapid and simultaneous extraction of three anticancer drug, including Sorafenib (SORA), Erlotinib (ERLO) and Dasatinib (DASA) in aqueous samples (water, waste water and urine samples) prior to liquid chromatographic determination. The proposed thin film membrane and covalent organic framework was characterized by FE-SEM, AFM, ATR and BET. Mixed matrix membrane was prepared by non-solvent induced phase inversion method and pore generation using Polyvinylpyrrolidone (PVP). Optimum extraction/desorption conditions were found as: Salt concentration: 6%w/v NaCl, stirring speed: 1400 rpm, pH 11, extraction temperature: 25 °C, extraction time: 35 min, desorption solvent: methanol and desorption time: 10 min. The figures of merit were obtained under the optimized conditions: the Linear Dynamic Ranges (LDRs): 9.58-100 µg L^{-1}, 5.12-100 µg L^{-1} and 4.22-100 µg L^{-1}, Limits of Detection (LODs): 7.12 µg L^{-1}, 4.20 µg L^{-1} and 2.50 µg L^{-1}, Limits of Quantification (LOQs): 9.58 µg L^{-1}, 5.12 µg $L^{-1}$and 4.22 µg L^{-1} for SORA, ERLO and DASA respectively.

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Magnetic solid-phase extraction in combination with hollow fiber liquid phase microextraction for preconcentration and determination of trace amounts of chlorpyrifos pesticide in aqueous samples

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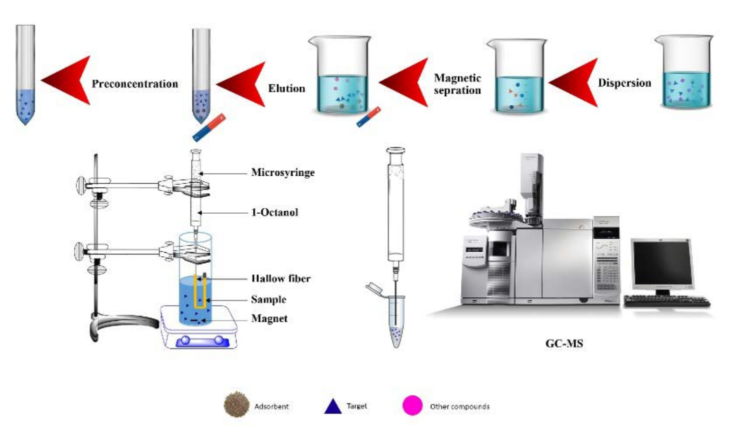
Graphical Abstract



Abstract: A novel sample pre-treatment approach based on the integration of magnetic solid-phase extraction (MSPE) and hollow fiber liquid phase microextraction (HF-LPME) prior to gas chromatography-mass spectroscopy (GC-MS) was developed for preconcentration and determination of trace amount of chlorpyrifos pesticide in aqueous samples. In this study, nanocomposites of Azolla modified with magnetite nanoparticles were prepared as a cheap natural adsorbent [1] for adsorption of chlorpyrifos as an organophosphate insecticide. The size and structure of magnetite modified Azolla nanocomposite (MANC) was studied by XRD, FT-IR, EDX, BET, FESEM, TEM and VSM. The MANC was used in magnetic solid phase extraction (MSPE) as adsorbent for sorption of Chlorpyrifos parameters prior to hollow fiber liquid phase microextraction (HF-LPME) [2]. All of affecting parameters during adsorption/desorption of chlorpryfos in MSPE procedure, such as sample volume, contact time, pH, and ionic strength, were studied and optimized (OA16) by Taguchi experimental design method. After extraction by MSPE method under the optimum conditions of adsorption (sample volume= 50 ml, contact time = 30 min, pH= 3, Sorbent weight= 0.05 gr, ionic strength= 0.01) and desorption (solvent= ethanol, solvent volume= 2 ml, contact time=10 min), 2 ml of the ethanolic solution containing the desorbed Chlorpyrifos was dispersed in 16 ml aqueous solutions of 10% NaCl in an extraction vial and the solution was extracted by HF-LPME process. After optimizing the experimental parameters by Taguchi experimental design method (OA9), the optimum parameters were obtained to have higher extraction efficiency (sample volume= 18 ml, contact time= 15 min, Salt amount= 10% w/v, extraction solvent=1-Octanol, extraction solvent volume= 30 µl). The final extract solution was analyzed by GC-MS. Thus the present MSPE-HF-LPME method is a good way to preconcentrate and determines trace amounts of Chlorpyrifos from the channel, well, and river water samples.

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Using an Efficient Al-Based MOF in a Two-Step Microextraction Procedure for the Extraction of Pesticides from Commercial Fruit Beverages

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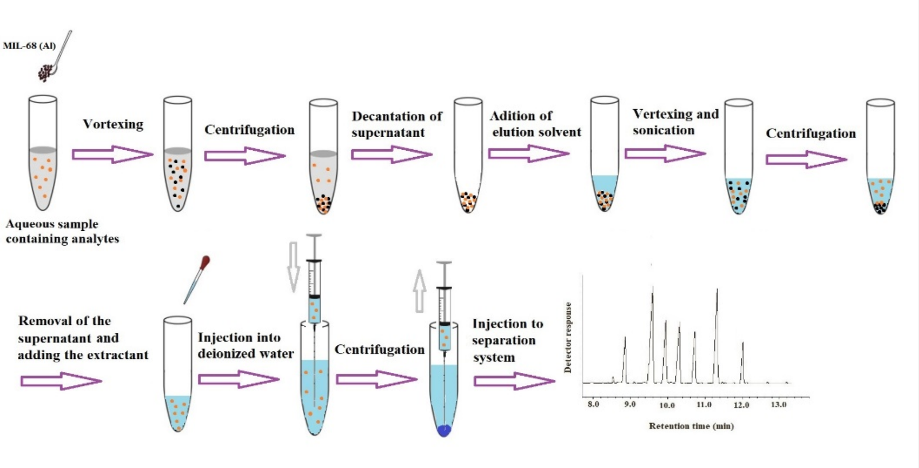

Abstract: Around 85% of the world's pesticide production is consumed in agriculture for overcoming pests, removing unwanted plants, and increasing crop quality and yield [1]. To conduct a food safety survey by analysing the pesticide content of various commercial beverages, a dispersive micro-solid-phase extraction procedure was performed using MIL-68 (Al) (a metal-organic framework based on aluminium and 1,4-benzenedicarboxylic acid) as an efficient adsorbent following a preconcentration step by dispersive liquid–liquid microextraction. In the extraction process, the adsorbent was added to an aqueous solution containing the mentioned pesticides and vortexed well. After centrifugation, the analytes loaded on the adsorbent were eluted using 2-propanol. Then, μL-level of 1,1,2-trichloroethane, as the extraction solvent, was mixed with the obtained 2-propanol phase and injected into deionised water containing dissolved Na_2SO_4 in which a cloudy solution appeared. One microlitre of the sedimented phase after centrifugation of the cloudy solution was injected into a gas chromatograph equipped with a flame ionisation detector. Satisfactory outcomes such as wide linear ranges (5.95–1000 $\mu g\ L^{-1}$), acceptable relative standard deviations (4.6–6.9% for intra- ($n = 6$) and 5.6–7.4% for inter-day ($n = 3$) precisions), high enrichment factors (228–350), reasonable extraction recoveries (45–70%), satisfactory relative recoveries (82–108%), and low limits of detection (1.15–1.80 $\mu g\ L^{-1}$) and quantification (3.80–5.95 $\mu g\ L^{-1}$) were obtained. This research demonstrates the successful application of MIL-68 (Al) for the analysis of some pesticides in fruit beverages. Also, using mg amount of the sorbent and μL volume of the extraction solvent makes the method precious. Moreover, using a green metal, aluminium, as the central ion of the synthesised metal-organic framework is another beneficial aspect of the research.

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Simultaneously Cooling Assisted Headspace and Fiber in Headspace Solid-Phase Microextraction Coupled to GC-MS for the determination of Pesticides in soil

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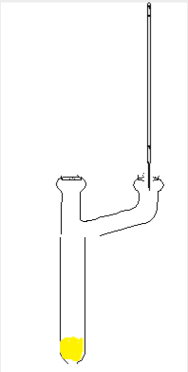
Graphical Abstract



Abstract: For volatiles, trapping and adsorption of analytes on solid phases is very important [1]. Also for semi-volatile analytes, low volatility is a challenge. To overcome these limitations, thermal desorption and use of an organic solvent to release analytes from their matrix often are suggested [1,2]. Thermal desorption is a simple and most effective approach that can enhance the mass transfer process and increase the vapor pressure of the analyte. Headspace solid-phase microextraction (HS-SPME) combined thermal desorption is a conventional method that is used for the analysis of volatile and semi volatile analytes [3]. However, applying high temperatures in this method can decrease the extraction from headspace because of reducing the partition coefficients of the analytes. This problem can be overcome by simultaneously heating the sample matrix and cooling the extraction phase. Acceleration of mass transfer and increasing the temperature gap between the sample and sorbent are achieved by this strategy [1]. The use of a cooling assisted (CA) method for SPME was first reported by Pawliszyn et al. [4]. In this paper, in addition to cooling the fiber, the head space containing the analyte around the adsorbent was cooled. This increases the concentration of analyte around the fiber. As a result, the efficiency of the method increases. The proposed method is evaluated for the extraction of Pesticides such as Fenvalerate and Chlorpyrifos. Optimization of the extraction parameters was carried out using a GC–MS instrument. To optimize the extraction parameters such as the moisture and temperature of the sample and fiber, and time of the extraction a simplex method was used as an easy and fast optimization technique. Under the optimized conditions, linear calibration curves were obtained for the analytes within a range of 1.2 ng g^{-1} up to 9000 ng g^{-1}, with determination coefficients that ranged from 0.989 to 0.991. The limits of detections were between 0.89 to 1 ng g^{-1} and the coefficients of variation for 5 replicates were between 2.6 to 4.1%. The method was successfully applied to the GC-MS analysis of pesticides in soil samples.

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Investigation of the Effect of γ-alumina Nanoparticles on the Elimination of Cationic Dyes from Industrial Effluents

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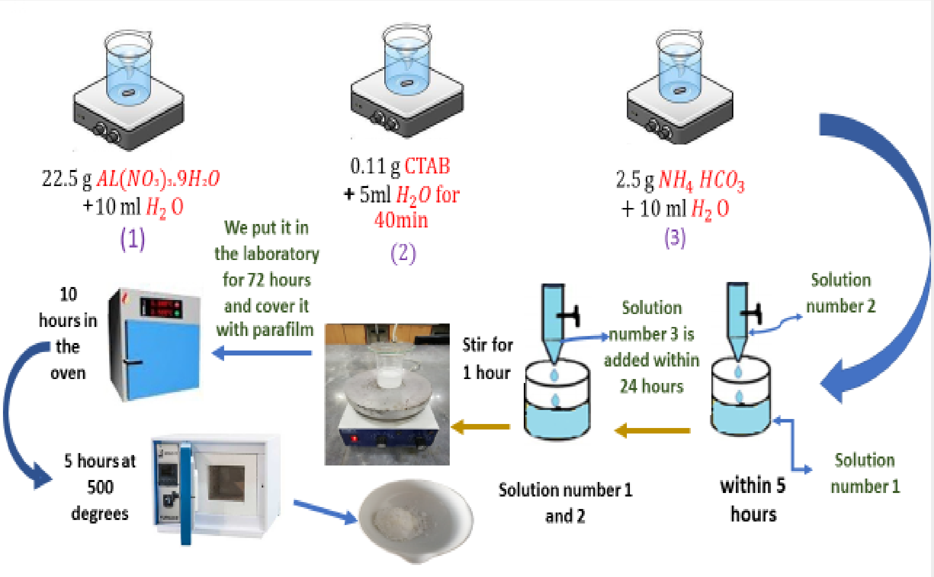
Graphical Abstract



Abstract: The present study investigates the removal of Basic Brown 1 (BB1) by surfactant-modified γ-alumina nanoparticles. The use of alumina nanoparticles with an anionic surfactant (sodium dodecyl sulfate (SDS)) as a new and efficient adsorbent has been successfully performed to remove cationic dyes from aqueous solutions of textile industrial effluents [1]. The SEM image, XRD pattern and FT-IR spectra was examined and the analysis showed that the surface modification of the nanoparticles was done correctly. The effect of different parameters such as contact time, initial concentration of dye, amount of adsorbent used, pH of solution, temperature, the interference of similar dyes and ions on the removal of BB1 were also investigated. The optimal conditions for dye removal were examined by the response surface methodology (RSM) using a four-factor four-level design. The results obtained showed that the contact time of 15 minute, pH of 6, initial concentration of 27.5 mg/l BB1, and adsorbent dose of 0.030g BB1 resulted in the most significant efficiency (93.12%) of BB1 removal. A spectrophotometric method was used to determine dye concentration. It was also found that the SDS-coated γ-alumina has shown higher adsorption efficiency for BB1 opposed to unmodified γ-alumina nanoparticles [2].

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Application of hollow fiber stir bar solid/liquid phase microextraction for determination of Chlorpyrifos in water and fruit juice samples

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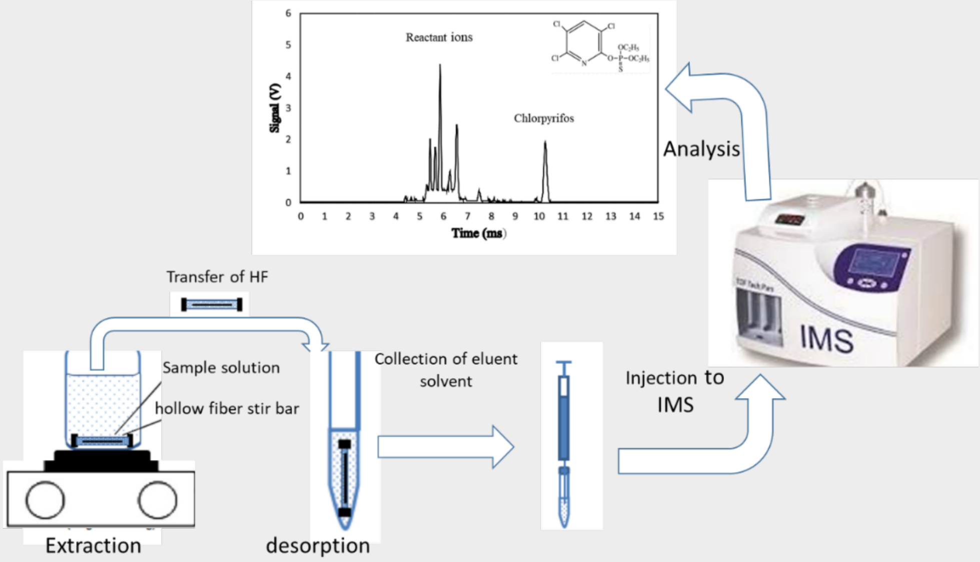

Abstract: Chlorpyrifos is an organophosphorous insecticide widely used for pest control in agriculture and with minimum degree for indoor applications. This pesticide can affect the human nervous system, so it is considered a toxic species for the human body [1]. Layered double hydroxides (LDHs) are a class of 2-dimensional layered materials. LDHs have high anion exchange capacities, with large surface area, and the interlayers having variable sizes. For these reasons, the application of LDHs as sorbents has been actively investigated [2]. In the past decades, the development of new extraction methods that integrate the extraction and stirring element in the same device have been become popular because of some advantages such as simplicity, no loss of analyte and enhancement in the extraction recovery [3]. In this regard, a design of hollow fiber supported liquid membrane so-called hollow fiber stir bar solid/liquid phase microextraction ((HF-SB-S/LPME) has been developed. Extraction device for the HF-SB-S/LPME procedure was prepared using a hollow fiber reinforced with acceptor phase and magnetic wire in the HF lumen. In the current study, acceptor phase was Fe3O4/Mg-Al layered double hydroxide dispersed in organic solvent of toluene, which was held in the pores and lumen of a porous polypropylene hollow fiber membrane while a magnetic wire in the HF lumen and applied for preconcentration and determination of Chlorpyrifos in water and fruit juice samples. The effective factors on extraction (pH, stirring rate, salt effect, the volume of donor phase and extraction time) were investigated and optimized. At the optimum conditions, the relative standard deviations (RSD) of the method were calculated 5.4% (as intra-day relative standard deviation) and 8.9% (as inter-day relative standard deviation) at 30 µg L^{-1} concentration level.

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Analytical Nanochemistry

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Environmentally-Friendly Green Approach for the Production of Zinc Nanocomplex from the Extract of *Citrus aurantifolia* Waste: to Evaluate Its Effect on the Antioxidant Activity of *Thymus daenensis*

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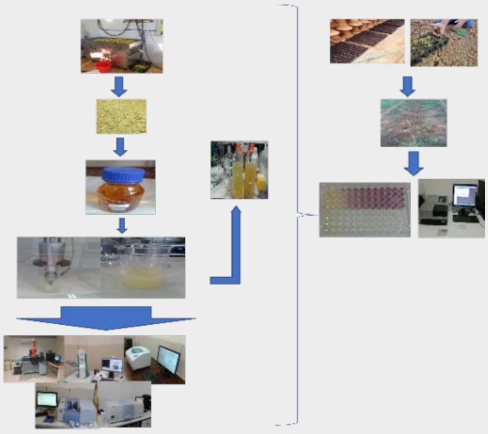
Graphical Abstract



Abstract: In the recent years, green synthesis of metal nanostructure (NS) is an interesting issue of the nanoscience and nanobiotechnology. There is a growing attention to biosynthesis the metal NS using organisms. Among these organisms, plants seem to be the best candidate for biosynthesis of NS. NS produced by plants are more stable than that in the case of other organisms. The present investigation was carried out to green synthesis of Zinc nanocomplex (Zn NC) by using the waste of *Citrus aurantifolia*, via ultrasonic probe (Oscillation of 90% for 30 min). The effectiveness of this new introduced Zn-fertilizer was evaluated on the antioxidant capacity of *T. daenensis* as the valuable and medicinal plant. The formation of NC was confirmed and characterized by UV/VIS spectroscopy, X-ray diffraction (XRD), Scanning electron microscopy (SEM), Dynamic light scattering (DLS), Fourier transform infrared spectroscopy (FTIR), Transmission electron microscopy (TEM) and Energy dispersive X-ray analysis (EDX). The TEM image indicated that the Zn NC size was 18-30 nm. The Zeta potential to test their stability resulted in a value of -48.8 mV. The spectrum of the XRD confirmed the hexagonal structure for Zn NC in which was similar to the previously reported pattern [1,2]. The EDX analysis showed the presence of carbon, oxygen and nitrogen in the Zn NC. The FTIR of the resultant material showed a characteristic Zn-O stretching at 525 cm^{-1}, which confirms the formation of Zn NC. The SEM image of Zn NC showed the morphology of Zn NS was hexagonal and quasi spherical. The antioxidant activity varied from 307.0 ± 2.3 µg/ml to 464.4 ± 1.4 µg/ml. Application of Zn NC increased the antioxidant activity of the *T. daenensis* extract in comparison to the control plant. Increasing the content of polyphenolics by the Zn NC could be a useful method to enhance antioxidant capacity and pharmaceutical properties of *T. daenensis*. This green synthesis approach showed that the waste of *C. aurantifolia* could be used as an effective stabilizing as well as reducing agent for the synthesis of Zn NC.

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Removal of Pb(II) from aqueous solutions using magnetic nanocomposite

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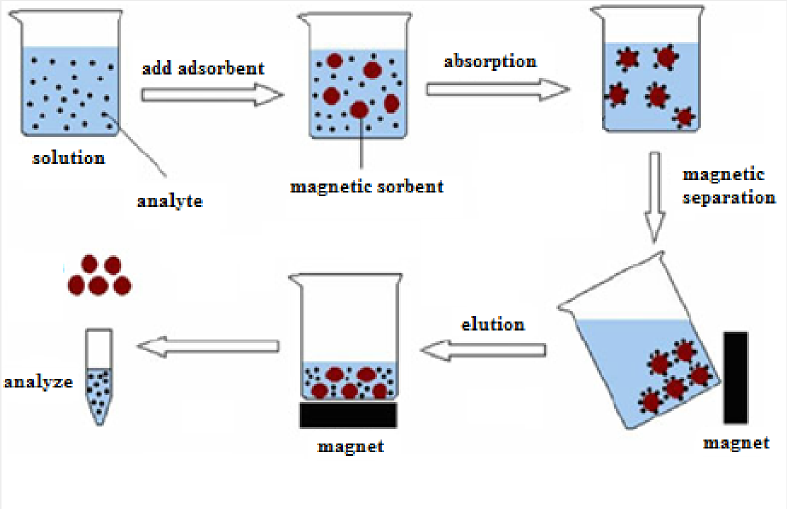

Abstract: In recent years, water pollution by heavy metal ions has been a widespread environmental problem because of non biodegradability, accumulation in environment and living body, discharge and harmful effects of these heavy metals [1]. Heavy metals are mostly originated from industrial activities resulting in the release of toxic metals into the environment directly or indirectly, reaching the air, soil and water sources [2]. Lead (Pb) and its compounds can result in damage of living organisms, depending on the level and duration of exposure [4]. Therefore, removal of toxic lead ions prior to disposal is very important task. In the present study, magnetic sepiolite nanocomposite was prepared via facile hydrothermal method. The formed nanocomposite was employed to removal of Pb(II) ions from water samples. The structure properties and morphology of the prepared nanocomposite were characterized by scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), X- ray diffraction (XRD) transmission electron microscopy (TEM), and vibrating sample magnetometer. The effect of several parameters including initial concentration of Pb(II) ions, pH, adsorbent dosage and contact time was examined to optimize the conditions for maximum removal of Pb(II) ions. Equilibrium data and isotherm constants were evaluated using Freundlich and Langmuir models. The maximum adsorption capacity under optimal conditions was calculated 96.15 mg g^{-1}. Kinetic data were analyzed using the first-order and second-order Lagergren equations. The pseudo-second-order kinetic model was fitted ($R^2 = 0.9998$), which indicates that removal of Pb(II) ions could be controlled by the chemical adsorption process. The as-prepared magnetic sorbent was easily reused through sequential adsorption-desorption cycles, indicating that the resulting nanocomposite has an acceptable stability and reusability. This study shows that the prepared nanocomposite can be utilized as an efficient and magnetically separable sorbent for the environmental treatment.

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An electrochemical sensor based on a carbon paste electrode modified with novel nanostructure modifier

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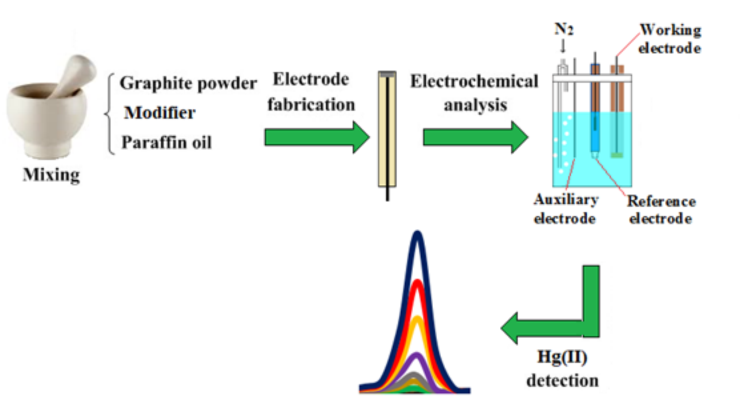

Abstract: Currently, there is an increasing demand to selectively sense heavy metal ions such as Hg(II), Cd(II) and Pb(II) due to their toxic effect on human health [1,2]. Among them, mercury is one of the most widely studied toxicant [3]. Even a trace amount of mercury intake can lead to serious central nervous system problems and damage vital human organs [4]. Therefore, it is of great importance to determine Hg in a varied range of samples especially water resources. In the present study, N and S doped carbon/sepiolite nanocomposite was prepared via a simple hydrothermal method. The electrochemical Hg(II) sensing capability of the prepared modifier was studied with the modification of an inexpensive carbon paste electrode (CPE). Differential pulse voltammetry (DPV) was applied for quantitative determinations. The effect of several parameters including solution pH, modifier dosage, accumulation potential and accumulation time on the electrochemical signal of the modified electrode were researched, and their values were determined as 5.0, 6.0% (w/w), 0.7 V and 200 s, respectively. The resulting electrode exhibited a linear relationship towards Hg(II) concentrations ranging from 0.4-0.85 μg L^{-1}. The detection limit was found to be 0.1 μg L^{-1} (S/N = 3) that is lower than the permitted value of Hg(II) reported by the Environmental Protection Agency limit for drinkable water. The relative standard deviation (RSD) for 7 successive measurements at different electrodes was also found to be 2.5%. The interference studies showed that the several common metal ions (e.g., Ag(I), Pb(II), Cd(II), Co(II), Ni(II), Zn(II), Cu(II), Mn(II) and Cr(III)) did not interfere with the quantitative mercury determination. The designed sensor was further utilized for the determination of mercury ions in real water samples with satisfactory results

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Citrate Capped Silver Nanoparticles as Naked-Eye Colorimetric Sensor for Sensitive and Rapid Detection of Cysteine in Biological and Environmental Samples

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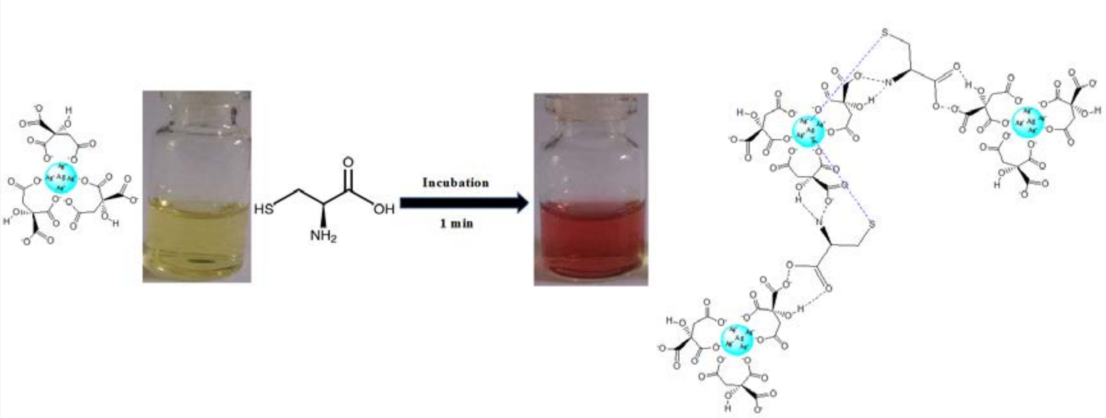
Graphical Abstract



Abstract: Cysteine (Cys) is an essential intracellular biothiol, which performs very important role in various biological process [1-3]. Once the content of Cys is out of the normal range, it will lead to a variety of diseases. Increased levels of Cys has been associated with vascular disease and neurotoxicity. Conversely, Cys deficiency can also lead to diverse diseases such as hematopoiesis decline, leukopenia, psoriasis, liver damage, weakness, slow growth rate, skin lesion, hair depigmentation, lethargy, edema, and neurological disorders [4]. Therefore, it is highly important to detect and quantify this biothiol with high sensitivity and selectivity. In this work, we have proposed a simple, very fast, easy synthesis and cost effective colorimetric sensor for sensitive detection of cysteine using citrate capped AgNPs (Cit/Ag NPs). The colorimetric recognition strategy was based on the significant change in the surface plasmon resonance (SPR) band of Cit/Ag NPs from 403 to 550 nm with cysteine. By adding cysteine to the Cit/Ag NPs solution, the color of solution rapidly changed from yellow to red. This is attributed to the aggregation of citrate-capped Ag NPs induced by cysteine due to formation of the strong covalent Ag-S bond and electrostatic binding. The proposed method was successfully applied for detection of cysteine in biological (human serum and urine) and environmental (tap and lake water) samples with significant recovery rates in the range of 95.5-105.1% and the relative standard deviation (RSD) lower than 5.5%. The interaction process between Cys and Cit/Ag NPs is very fast, so that the whole test can be completed within 1 minutes. Moreover, the colorimetric detection operation is very simple and cost-effective without any further modification process. Therefore, the Cit/Ag NPs as a chemical sensor in colorimetry assay is simple, very fast, easy synthesis, highly selective and sensitive, a single-step process and mainly cost-effective, providing a possibility to develop an analysis in real-time and an effective rapid detection way for monitoring and visualizing Cys.

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Photocatalytic Degradation of Methyl Green Dye in Aqueous Solution by Novel Magnetic Carboxymethyl Cellulose - Cysteine Stabilized ZnO Nanocomposites

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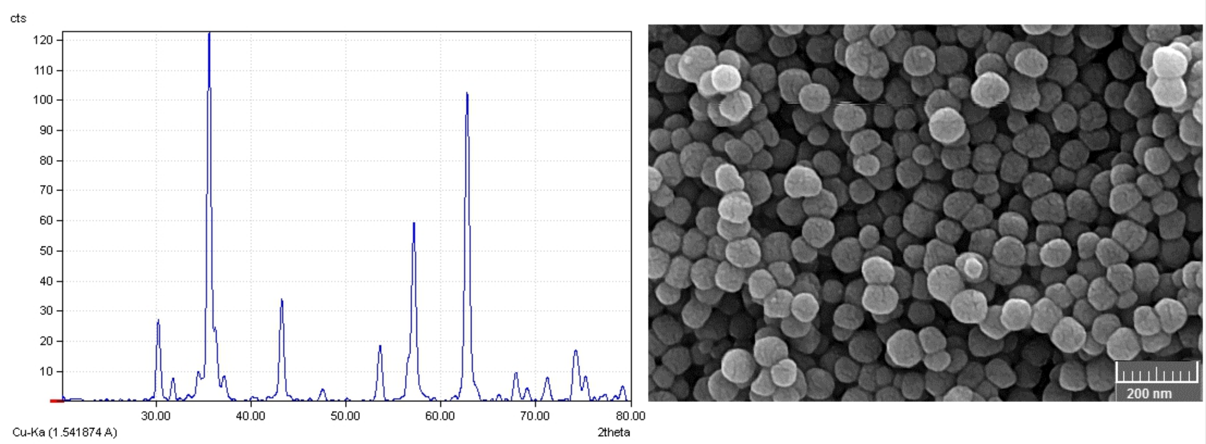
Graphical Abstract



Abstract: In recent years, interest in the field of magnetic polymeric nanocomposite with magnetic core and polymeric shell has increased considerably because of many advantages such as flexibility, good compatibility and functionalization with other materials, ease of processing, and tunable properties [1]. The nanocomposite properties are defined by chemical nature of polymeric matrix which served as surfactant or stabilizer, and the interaction between nanoparticles and polymeric matrix [2, 3]. In view of this, several natural and synthetic polymers (e.g., chitosan, graphene oxide, polypyrrole, polyaniline, polyacrylonitrile) are extensively studied as the organic shell in magnetic polymeric nanocomposite. Among the natural polymers, cellulose is the most abundant biopolymer, and is considered as a biodegradable, eco-friendly, and non-toxic polymer. However, the poor reactivity of cellulose and also its poor solubility limits its application. Chemical modifications of cellulose have improved the overall applications of its polymers. Carboxymethyl cellulose (CMC) is a cellulose derivative with carboxymethyl groups bonded to some of the hydroxyl groups on the cellulose backbone [4]. In order to maximize its functionality in existing applications, e.g. without employing any extraneous cross-linking agents, CMC is often modified. In this work, magnetic Carboxymethyl cellulose - Cysteine stabilized ZnO nanocomposites was successfully synthesized by an effective procedure without any toxic monomer and crosslinking agent. The asprepared nanocomposites were characterized in detailed, and their photocatalytic properties were investigated for the degradation of methyl green dye from aqueous solution. Hence, the effects of experimental parameters on the methyl green photocatalytic degradation by the nanocomposites were studied and the optimized conditions were obtained.

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Colorimetric-based artificial tongue for discrimination of neurodegenerative disorders biomarkers in the urine sample

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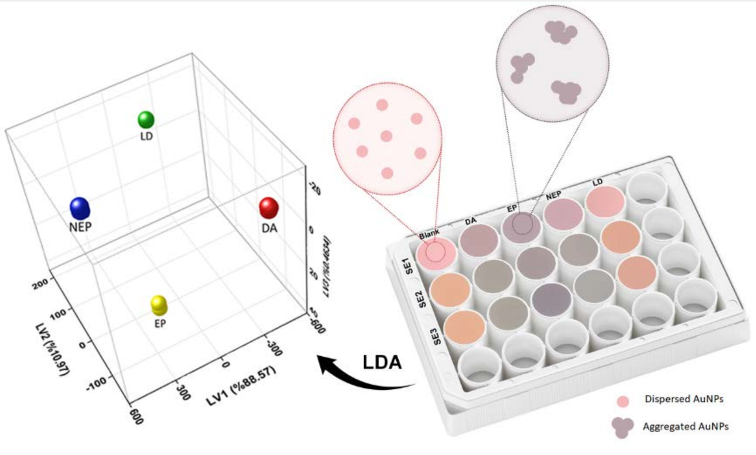
Graphical Abstract



Abstract: Catecholamine neurotransmitters (CNs), including dopamine (DA), epinephrine (EP), and norepinephrine (NE) are known as the main index of various neurological diseases such as Parkinson's [1]. On the other, levodopa (LD), the precursor of these biomarkers, is prescribed to slow the progression of Parkinson's disease [2]. Therefore, concurrent discrimination of DA, EP, NEP, and LD are extremely recommended in early-stage clinical diagnosis and evaluation of disease response to treatment. In this respect, in this research, a high-performance colorimetric artificial tongue has been proposed for the identification and discrimination of catecholamines and levodopa based on their ionic states in various buffering conditions and involvement in the aggregation of capped-gold nanoparticles. The amassed AuNPs led to a redshift in the localized surface plasmon resonance (LSPR) peak of AuNPs, altering the color of the solution, and creating a unique visual pattern for each analyte. Diverse gold-nanoparticle (AuNP)-aggregation behaviors and distinct fingerprint response patterns were checked out by linear discriminate analysis (LDA) in order to classify DA, EP, NEP, and LD. The proposed colorimetric array was successfully able to differentiate among solitary neurotransmitters as well as their mixtures with acceptable accuracy, sensitivity, and specificity. Besides, partial least square regression (PLSR), a multivariate calibration method, was applied for quantitation modeling. Then, the analytical figures of merit verified that the designed sensor array is precise and accurate in both the discrimination and quantification of the Analytes by the linear range of 0.6-9 µM (R^2 = 0.99) for DA, 0.1-10 µM (R^2 = 0.99) for EP, 0.1-9 µM (R^2 = 0.99) for NEP and 1-70 µM (R^2 = 0.99) for LD. Finally, the feasible potential of the array was validated in the human urine sample as one of the complex biological fluids.

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Synthesis of New Nanocomposite Based on Metal-Organic Framework Montmorillonite Consists of Cu_2O and Investigation of Its Antibacterial Properties

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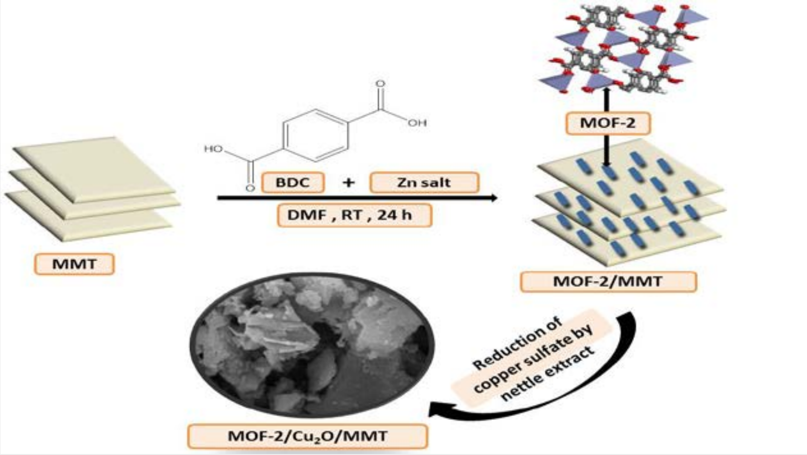
Graphical Abstract



Abstract: One of the metal-organic framework (MOF) materials called MOF-2 with the formula $[Zn_2(BDC)_2]$ has attracted the attention of researchers. Here, we describe the preparation and synthesis of high-quality MOF-2 in more stable and simple conditions such as room temperature [1]. On the other hand, Montmorillonite (MMT), a nano layered clay mineral that combines with this metal-organic framework to form a new environmentally friendly clay-based composite MOF-2/MMT using an in-situ growth technique created [2].

The synthesized composite modified by Cu_2O nanoparticles and new prepared nanocomposite (MOF-2/Cu_2O/MMT) was used for antibacterial tests. Cu_2O nanoparticles were used in composite in order to increment of antibacterial activity. The results show that the nanocomposite has been synthesized correctly and completely. The results showed MOF-2 were synthesized in the interlamellar space of MMT. Cu_2O nanoparticles were incorporated in composite by the simple green method. $CuSO_4$ salt and nettle leaf extract were used as a copper precursor and reducing agent, respectively [3].

The results of antibacterial testing of these compounds have also been announced. The antibacterial activity of MOF-2 and the mentioned composites was tested against gram-positive and gram-negative bacteria. The MOF-2, MOF-2/MMT and MOF-2/Cu_2O/MMT showed efficient antibacterial activity. Antibacterial activity can be related to release of cations from synthesized compounds such as Cu^{2+}, Zn^{2+}.

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Post synthetic modified MOF for nitrate removal from aqueous solution by MOF

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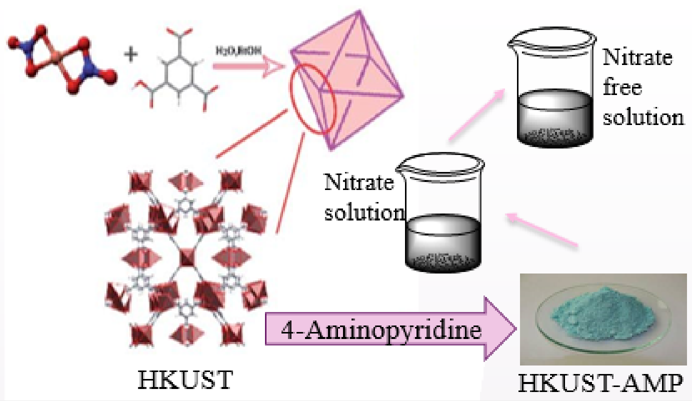
Graphical Abstract



Abstract: Metal-organic frameworks have been used because of their unique properties for various applications including adsorption. These compounds are a good base for loading different species because of their modifiable structure. In this project, modified metal-organic framework was used as adsorbent in adsorption of nitrate from aqueous solution. The HKUST framework was first synthesized and modified by the 4-aminopyridine functional group (HKUST-AMP), the sorbent used to remove nitrate from aqueous solution. The product characterized by Infrared spectroscopy, powder X-ray diffraction, elemental analysis and scanning electron microscopy, thermal gravimetry and absorption- desorption Nitrogen gas. Nitrate adsorption process from aqueous solution was investigated at 25°C, 30 min, pH=5 by UV-Visible spectroscopy. Nitrate removal from aqueous solution by adsorbent 70% was calculated.

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Zif-8/ AgNPs Nanocomposites incorporating of Electrospum Nanofiber Membrane for Removal of Heavy Metal Ions (Cd^{+2}, Cu^{+2}) from Water and investigation of It's Antibacterial properties

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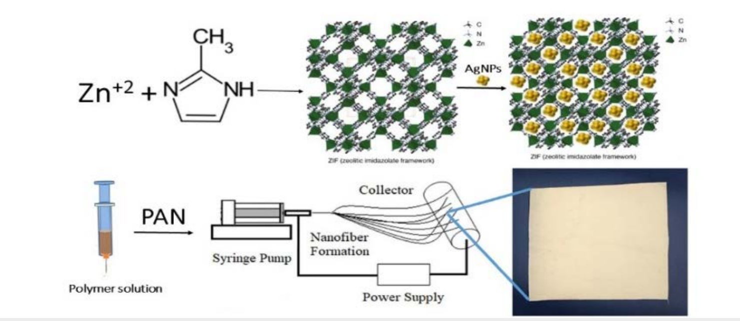
Graphical Abstract



Abstract: Metal-organic frameworks (MOFs) have been widely applied for pollutants removal in water. Ag NPs also can be considered as suitable antibacterial agent. However, aggregation of MOFs and AgNPs in solution and difficult collection after use are considered as two of the critical challenges for the wide applications of these nanomaterials. Effective and efficient removal of both heavy metal pollutants and bacterial contamination from water is an open issue. In this work, silver nanoparticles (AgNPs) were encapsulated in porous metal-organic frameworks of ZIF-8 (ZIF-8/AgPSs), Then Electrospun nanofiber composite membranes containing ZIF-8/AgNPs incorporated on polyacrylonitrile (PAN) nanofiber synthesized via electrospinning have been prepared. The obtained composite membranes were characterized by transmission electron microscopy (TEM), scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), Brunauer Emmett Teller (BET). The effects of different parameters influencing the removal efficiency including the concentration of sample and its volume, the amount of the sorbent, pH, the ionic strength, the time of extraction and the reusability of the sorbent were investigated and optimized. Under the optimized conditions, the linearity varied in range of 0.25–30 mg L^{-1} for Cu^{+2} and 0.1-20 mg L^{-1} with R^2 values higher than 0.998. The adsorption kinetic fitted well with pseudo-second-order model and its adsorption isotherm can be described by Langmuir model. The effectiveness of PAN-ZIF-8/AgNPs nanofibrous membrane was optimized in a batch system for the removal of Cd^{+2} and Cu^{+2} ions, and additionally characterized for its antimicrobial properties. The fiber nanocomposite exhibited efficient antibacterial activity, with maximum adsorption capacity of about 49.3 mg g^{-1} for Cd^{+2} and of about 51.6 mg g^{-1} for Cu^{+2} after 120 min. The applicability of the method was examined by analyzing the analytes in the water and wastewater samples. The prepared membrane appears to be promising for possible use in water purification systems.

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Cobalt (II) phthalocyanine dye removal from aqueous solution using cobalt ferrite nanoparticles as an efficient adsorbent

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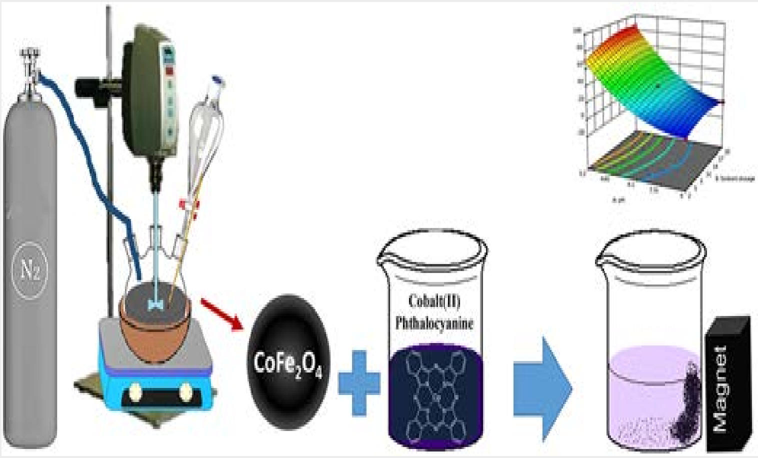
Graphical Abstract



Abstract: Cobalt ferrite nanoparticles ($CoFe_2O_4$ NPs) are used as an efficient adsorbent to remove cobalt (II) phthalocyanine (CoPc) dye from aqueous solutions. The characterization of adsorbent is investigated by field emission scanning electron microscopy (FE-SEM), X-ray diffraction (XRD), Fourier transform infrared (FT-IR) spectroscopy, energy dispersive X-ray spectroscopy (EDX), and the vibrating sample magnetometer (VSM) technique. To optimize the effective factors, response surface methodology (RSM) through using Box–Behnken design (BBD) is applied. By proper running of the Desirability function option in MINITAB software, the optimum conditions were found as pH 3.2, adsorbent mass (m) 11 mg, contact time of nine minutes (t), and initial dye concentration (C_d) of 30 mg L^{-1}. Isotherm studies of the adsorption process are carried out where the Langmuir isotherm shows the maximum monolayer capacity (q_{max}) is 431 mg g^{-1}. The kinetic studies including pseudo-first-order, pseudo-second-order and intra-particle diffusion models indicate that the pseudo-second-order kinetic model describes better the adsorption kinetic behavior. This study shows that $CoFe_2O_4$ NPs have excellent potential for the removal of CoPc dye from an aqueous solution.

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Visual Discrimination of Alcohols using Nanoplasmonic Sensor Array

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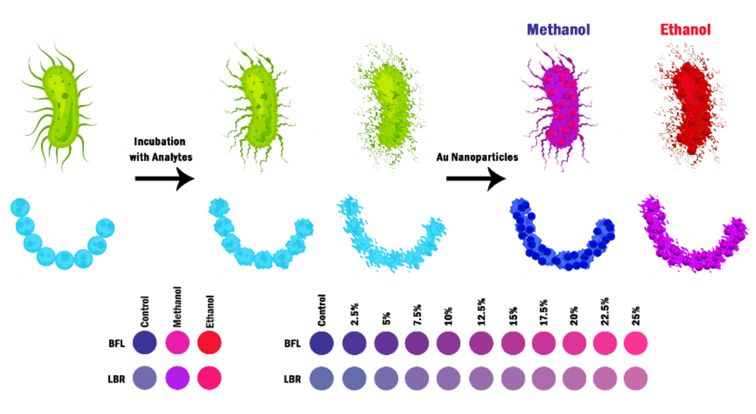

Abstract: Methanol is an extremely toxic compound engendering serious human poisoning when it comes to contact with skin, is swallowed or inhaled. Numerous methanol poisonings have been reported over the past three years of the pandemic, but notoriously, discrimination of methanol from other alcohols still remains a great challenge. Among the plenty of analytical techniques, colorimetric sensor arrays based on the plasmonic nanoparticles hold a great promise in naked-eye discrimination of compounds with high structural similarities and can properly meet the foregoing challenge. Herein, we intend to present a colorimetric sensor array based upon the assembly of the gold nanoparticles (AuNPs) onto two types of bacteria for discrimination and quantification of alcohols in aqueous media. As a matter of fact, the exposure of the bacteria to the alcohols precludes the assembly of AuNPs, thereby hindering the color variation from red to blue. The structural characteristics of each bacteria differ from one another, resulting in independent responses for each analyte. Different experimental conditions comprising the aggregation time, and incubation time were optimized both for the sensing elements. The inspection of visible spectra and RGB data by Linear Discriminant Analysis (LDA) revealed the potentiality of the presented approach in the discrimination of unicomponent and bicomponent samples of methanol and ethanol. Alternatively, Partial Least Square Regression (PLSR) was exploited as a multivariate regression technique for calibrating the spectral and RGB data with the concentration of the analytes. After all, the proposed strategy can open up a new avenue in the development of aggregation-based plasmonic sensors.

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Synthesis of chitosan-based intelligent nanocomposite hydrogel and investigation of its behavior for controlled release of metronidazole antibiotic

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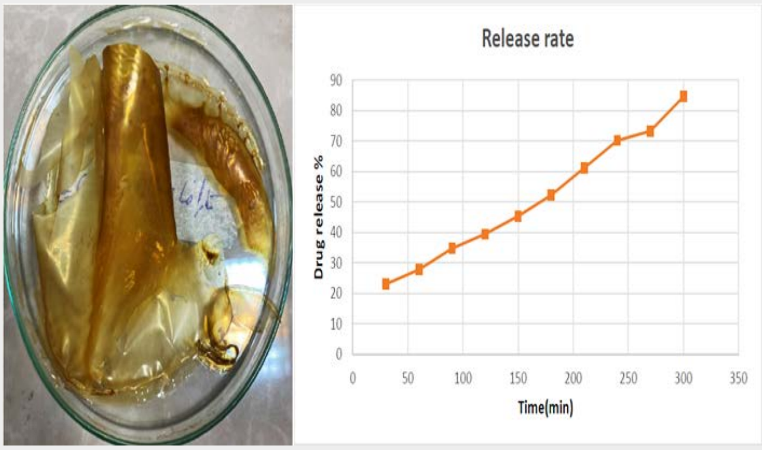

Abstract: The development of innovative drug delivery devices for different therapeutic agents has attracted the interest of many researchers [1-3]. In this study, chitosan/carbon nanotubes (Chit/CNT) nanocomposite hydrogel was prepared through grafting chitosan with acrylamide and acrylic acid monomers in the presence of methyl base acrylamide and thermal initiator of ammonium sulfate. The structure of the prepared hydrogel was characterized using FT-IR, TGA, and FE-SEM technics. The antibiotic metronidazole was then loaded into the Chit/CNT hydrogel. Afterward, the behavior of hydrogel in the release of the metronidazole was studied. The swelling property of the hydrogel and the effect of some parameters such as pH and temperature on the release rate were investigated. The amount of released drug from the hydrogel was determined by UV-Vis spectrophotometry. The results showed that the maximum drug release was obtained in the first 90 minutes under acidic pH of 4 and physiological temperature. The controlled and continuous release of the Chit/CNT nanocomposite hydrogel made it the best choice in smart drug delivery.

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Ocimum gratissimum: Phytochemical analysis and green synthesis of Ag/Ag_2O nanoparticles

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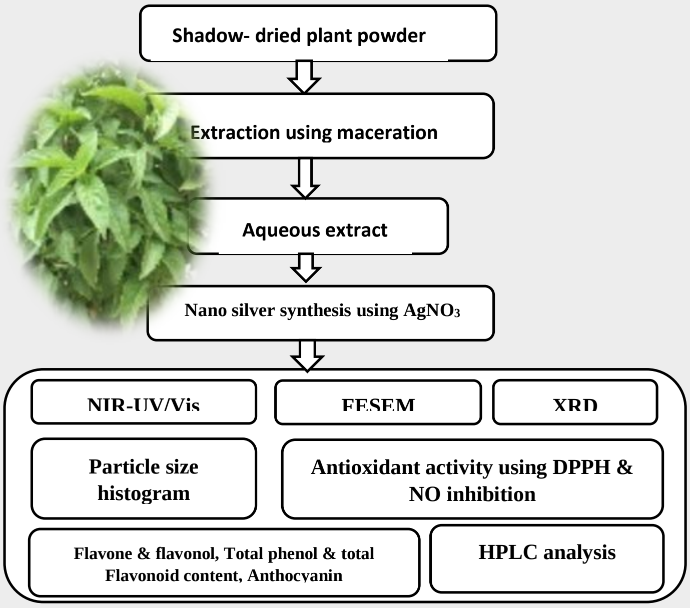

Abstract Biosynthesis of Ag/Ag_2O nanostructures was performed using a cheap and environmentally friendly method, where silver ions in $AgNO_3$ were reduced via co-precipitation using bio-reductants present in the aqueous extract of *Ocimum gratissimum* leaf [1]. In addition, the amount of phytophenols, total phenol, flavonoid content, anthocyanin, and flavone and flavonol content as well as NO and DPPH assay before and after nanoparticle synthesis were measured to obtain the amounts of active reductants or capping agents in the plant. The measured predominant phenolic compounds in this extract were sinapic acid and gallic acid. Moreover, the amount of total phenol content, total flavonoid content, flavone, and flavonol content, and total anthocyanin, were 113.8 µg eq gallic acid mg^{-1} extract, 10.3 µg eq gallic acid mg^{-1} extract, 0.01 µg eq gallic acid mg^{-1} DW and 7.4 mmol L^{-1}, respectively. The amount of IC50 in the extract, before and after synthesis of Ag/Ag_2ONPs, were 387.1 and 99.9 µg mL^{-1}, respectively, compared to the standard ascorbic acid [2]. The percentages of inhibition NO before and after the synthesis of Ag/Ag_2ONPs were 19.3% and 48.7%, respectively which implies that nanoparticle synthesis increases antioxidant activity in contrast to *O. gratissimum* extract. Scanning electron microscopy (SEM) of Ag/Ag_2ONPs showed the average particle size was 11.49 nm. The formation of nanoparticles was successfully confirmed using UV-Vis absorption spectroscopy and different properties of the resulting nanoparticles were determined using X-ray diffraction (XRD) and field emission scanning Electron microscopy (Fe-SEM) [3]. Naked observations also imply a color change of extract from light brown to dark brown that proves silver nanoparticle synthesis.

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Fluorescence sensor for guaifenesin detection in blood serum and pharmaceutical samples based on poly vinyl alcohol-borax hydrogel film modified by manganese doped zinc sulfide quantum dots

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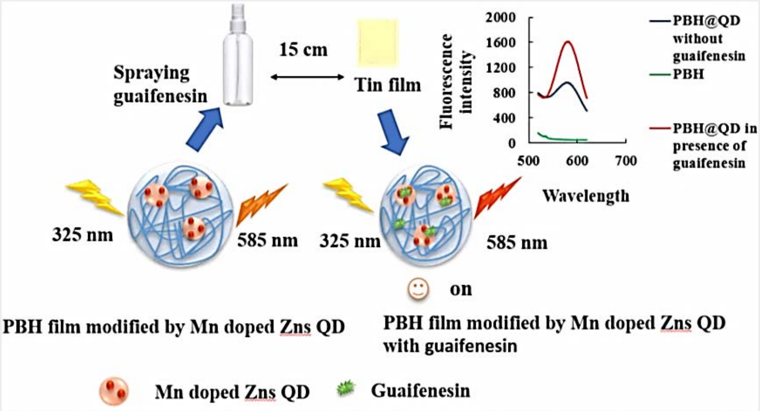

Abstract: Guaifenesin is a commonly used expectorant medicine to treat the symptoms of coughs caused by cold and mild upper respiratory tract infections such as pharyngitis, sinusitis and bronchitis. In veterinary medicine, guaifenesin is given intravenously to induce muscle relaxation when administering anesthesia. guaifenesin relaxes both laryngeal and pharyngeal muscles, allowing easier intubation. Guaifenesin (3-(2-methoxyphenoxy)-1,2-propanediol), or glyceryl guaiacolate ether (GGE), was originally derived from the guaiac tree [1,2]. In this work, a new turn-on fluorescence probe tin film sensor has been designed for guaifenesin monitoring based on embedding of l-cysteine capped manganese-doped zinc sulfide quantum dot into polyvinyl alcohol and borax hydrogel (PBH). The synthesized quantum dots are dissolved in a polyvinyl alcohol solution by dropwise adding the amounts of borax solution in the mixture. Quantum dots are trapped into the hydrogel frame simultaneously with the formation of a three dimensional network of PBH. The characterization and morphology of the new hydrogel nanocomposite tin film was investigated by energy dispersive X-ray microanalysis, TEM and average particle size analyzer. It was also observed that the fluorescence intensity of PBH functionalized by l-cysteine capped manganese-doped zinc sulfide quantum dot was enhanced by spraying guaifenesin on the film. This probe shows a linear calibration curve in the range of 0.5–10 μg mL^{-1} with a detection limit of 0.15 μg mL^{-1}. Moreover, relative standard deviation of the sensing platform was 1.15 and 1.07 % ($n = 10$) for two concentrations of 5 and 7 μg mL^{-1} respectively. The present sensing system was utilized for guaifenesin assay in human blood serums and regular guaifenesin with satisfactory results.

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Synthesis of reduced form of nanopolyaniline in a new method and its functionalization with chitosan for silver ion fluorescence measurement

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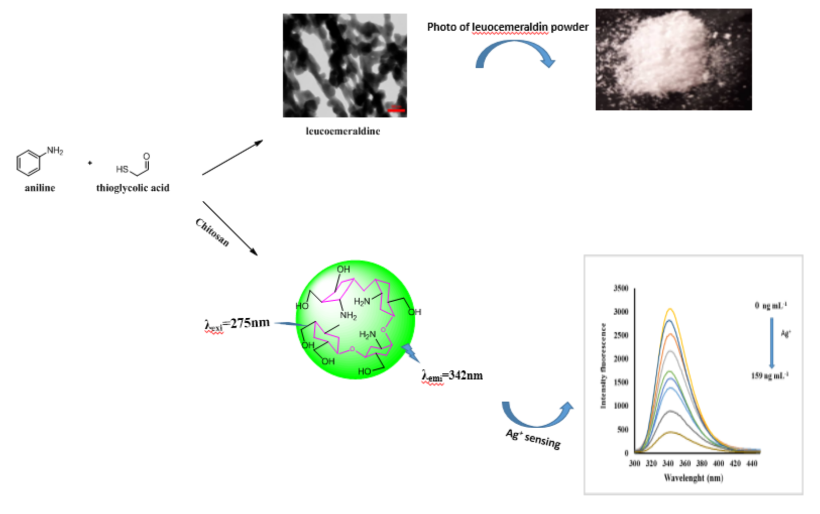
Graphical Abstract



Abstract In this work, we introduce a new and easy method for the synthesis of fluorescent nano probe. The nano probe was synthesized directly using aniline and thioglycolic acid. The formation of nanoparticles was characterized by particle size analysis and TEM images. Reduced form polyaniline (leucoemeraldin) is proposed for the nanoparticles name as the result of Fourier transform infrared (FTIR), X-ray diffraction (XRD), nuclear magnetic resonance (NMR) fluorescence, and ultraviolet-visible (UV-vis) spectroscopy studies. To make modifying the LE nanofiber, chitosan solution was severed as stabilizing and modifying agent accordingly the 0.6 -8 nm-sized nano sphere were gained, which exhibited enhanced fluorescence intensity. CS-LE nanosphere emitted bright blue fluorescence in 342 nm with excitation at 280 nm. CS-LE exhibited an exceptional fluorescence sensor for the application of silver ion (Ag^+) sensing. Experimental results showed that the decrease of the FL was proportional to the concentration of silver ion within the range of 6.4-159 ng mL-1 ($R^2 = 0.9953$) with a detection limit of 1.7 ng mL^{-1} under the optimized experimental conditions. Furthermore, the proposed method applies to the detection of Ag^+ in real aqueous samples, which is a detection system with excellent development prospects.

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Nanostructured Cobalt Compounds Derived From Terephthalate Metal-Organic Framework

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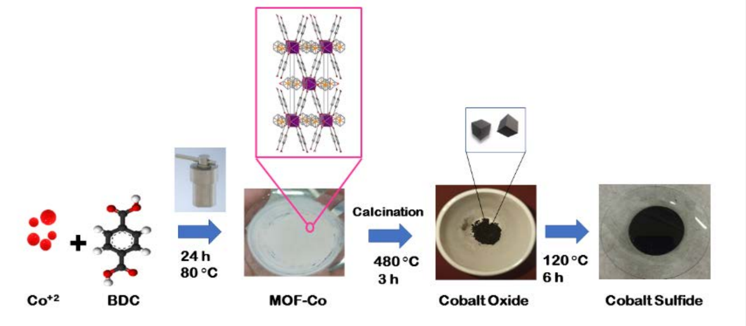

Abstract: Metal-organic frameworks (MOFs) are a group of high-surface adjustable structures with high porosity, low density, and high thermal stability that consists of organic ligands and metal ions. Lately, MOFs and their derivatives have become ideal electrochemical intermediates due to their properties, such as low cost, non-toxicity, and electrochemical oxidation-reduction properties for electrode modification in various applications [1-2]. Different compositions of MOF-derived crystals, e.g. metal oxides, sulfides, carbides, and their nanocomposites can be prepared in different sizes, shapes, and compounds of the desired metal ions and organic linkers with the advantages of high specific surface area and good porosity. Therefore, the MOF-derived compounds are widely used in electrochemical devices, especially sensors [3-4]. The study aimed to synthesize Co-MOF-derived porous cobalt compounds. In this case, Co-MOF was prepared by solvothermal method from cobalt acetate and benzene-1,4-dicarboxylic acid (terephthalic acid, BDC) as precursors in 1:1 ethanol-dimethylformamide (DMF) solvent at 80 °C for 24 hours. The resulting Co-MOF powder was converted to cobalt oxide nanostructures through calcination in a furnace at 480 °C for 3 hours. Finally, cobalt sulfide nanostructures were prepared from MOF-derived cobalt oxide nanostructures by means of thioacetamide in a sealed autoclave at 120 °C for 6 hours. The morphologies of all samples were characterized by field-emission scanning electron microscopy (FE-SEM). The chemical structure of the materials was characterized Fourier-transform infrared (FT-IR) and X-ray energy dispersive (EDX) spectroscopies. The crystal planes of the obtained samples were observed in powder X-ray diffraction (XRD) patterns. The study suggests that the obtained cobalt sulfide nanostructures can be used in different applications, especially electrochemical sensors, separation, and catalysis.

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One-pot Synthesis of Cobalt-Copper Bimetallic Metal-Organic Framework

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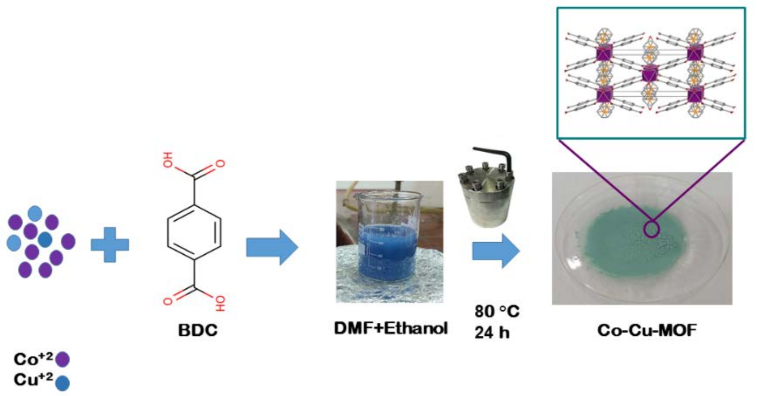

Abstract: Metal-organic frameworks (MOFs) are crystalline extended solid-state porous structures composed of metal ions or clusters as nodes linked by organic ligands. These periodic structures contain unique properties such as uniform porosity and extensively high surface area. MOFs have great potential for various applications from gas storage, separations, sensing, and catalysis, to energy harvesting and storage [1, 2]. Mixed-metal metal-organic frameworks (MM-MOFs) can be considered as MOFs having two metals anywhere in the structure. It is well-known that MM-MOFs mostly provide the advantage of the complexity and the synergism derived from the presence of different metal ions in the structure in comparison to homometallic MOFs [3]. MM-MOFs also presented superior performance and stability with respect to the corresponding single metal ones. Therefore, the current research has aimed to develop a porous efficient cobalt-copper bimetallic MOF by means of the solvothermal method. In this regard, cobalt acetate and copper acetate as metallic precursors, benzene-1,4-dicarboxylic acid (terephthalic acid, BDC) as ligand, and 1:1 ratio of ethanol : dimethylformamide (DMF) as solvents were added to a Teflon-lined stainless steel autoclave and stored in an oven at 80 °C for 24 hours. The morphology of the collected precipitants was characterized by field-emission scanning electron microscopy (FE-SEM) followed by X-ray energy dispersive spectroscopy (EDS) and elemental mapping analysis. Further, the metal-ligand bond was detected by the characteristic stretching peaks in Fourier-transform infrared spectroscopy (FT-IR). The crystal phases of the obtained samples were recorded in X-ray diffraction (XRD) patterns. The study suggests that the resulting material and its derivatives would be suitable for various applications such as electrochemical energy storage and catalysis.

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Design of Optoelectronic Tongue based on Core-Shell Plasmonic Nanostructures for Visual Detection and Discrimination of halide ions

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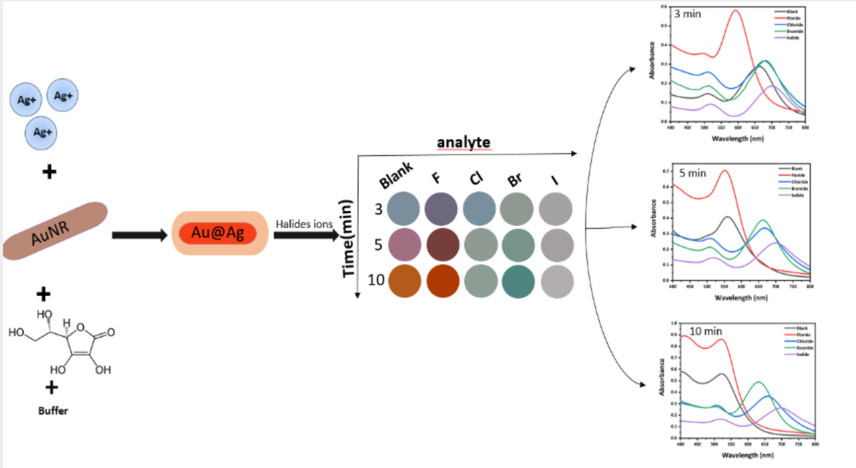
Graphical Abstract



Abstract: The purpose of this study is to provide an optoelectronic tongue for detection and discrimination of halide ions, one of the most important factors in environmental pollutants, using wide-color tonality of gold nanorods (AuNRs). Due to their high toxicity to mammals and organisms as well as corrosion ability, determination of halide ions in environmental waters are of high importance [1]. A group of nanostructures that are considered as sensory elements today due to their high absorption coefficient and sensitivity are AuNR@Ag. In these nanostructures, AuNRs first synthesized and silver ions are reduced by a reducing agent and placed in the form of shells on the surface of AuNRs. This sensor elements was used to determine many different analytes with growth or inhibition mechanism [2]. In this paper, inhibition of silver deposition on AuNRs by the targets have been utilized for development of a colorimetric sensor array for simultaneous identification and determination of halide ions in water samples. At first AuNRs with high LSPR wavelengths are synthesized by a modified seed-mediated growth approach. Then, at suitable pH condition, silver ions are reduced by ascorbic acid and coated on the surface of AuNRs. This phenomenon leads to decrease in aspect ratio of AuNRs and thus a blue shift in the longitudinal plasmon resonance of AuNRs to shorter wavelength. These nanostructures are used in the next step to detection of the analytes. In the presence of the targets, due to different interaction tendency of halide ions towards silver ions in terms of their solubility constants, redox potentials and complex formation constants and their interaction with surface of AuNRs as well, the thickness of silver deposited layer on the surface AuNRs decreases at different levels. This alteration in the thickness of silver layer on AuNRs; thus, a blue-shift in LSPR peak of AuNRs. These changes in the location of the LSPR peak in the visible region provide a wide-range of color tonalities from red to green, blue, and purple, enabling discrimination of target anions. The fingerprint absorption profile of the AuNRS@Ag in the presence of halide anions analyzed by linear discriminant analysis (LDA) and partial least squares (PLS) regression were employed for the qualitative and quantitative determination.

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Gold nanoparticles supported iron-based BTC (AuNPs/ Fe-BTC) Metal–organic frameworks as a fluorescence sensor for the selective detection of As (III) in contaminated waters

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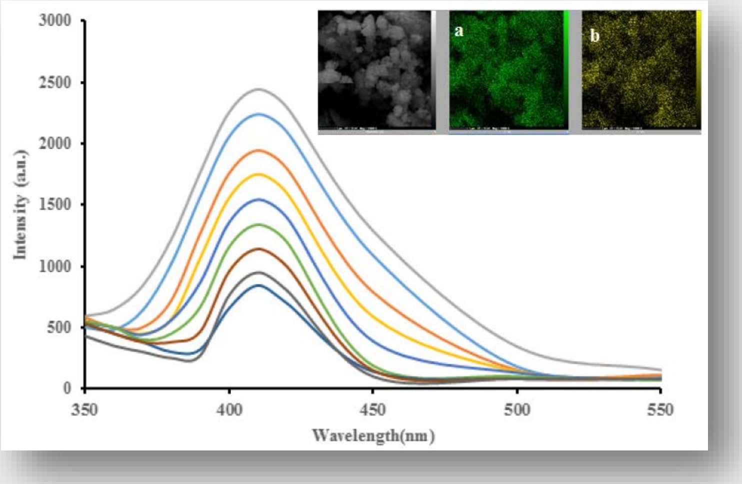

Abstract: Disposal of industrial effluents and wastewaters is considered as one of the most important challenges in the industrial world today. Due to the toxic ingredients of these effluents such as, heavy metal ions, dyes, organic compounds and other pollutants are the main cause of pollution of rivers, lakes and underground waters. Inorganic As(III) and As(V) are the major arsenic species in natural water resources with the former dominating in the reducing atmosphere while the latter is more stable in the oxidizing environment. World Health Organizations (WHO) prescribed 10 μg/L as the highest tolerance level for arsenic in drinking water [1, 2]. Fluorescence-based chemical sensors have attracted enormous research attention because of their rapid responses, high sensitivities, and the ease of preparation. In this regard, metal-organic frameworks (MOFs) have been recently developed as the sustainable platforms to detect trace amounts of contaminants present in water, due to their large specific surface areas, extremely high porosities, and tunable structures and functions [3]. In the present work, a gold-based fluorescent MOF was synthesized and applied as a highly sensitive and quickly responsive chemical sensor for As(III) detection in wastewater samples. Here, we applied a MOF, Fe-BTC (BTC= 1,3,5-benzenetricarboxylate), as a porous template to trap gold nanoparticles as arsenic-sensitive sites. The fluorescence intensity of this sensor increased significantly with increasing in As (III) concentration. The FT-IR, XRD, EDX-mapping and SEM were used to characterize the synthesized Au-Fe-BTC MOF. The main effective Factors on the performance of this sensor such as nanocomposite dose, ultrasonic time, pH value, type, and volume of buffer and reaction time were optimized by changing one single variable at a time method. Under optimized conditions, the calibration curve was linear in the concentration range of 30-80 μg/L of As (III) and limit of detection was 33 μg/L. Five real samples including two water samples of Karun River, Bahmanshir River, tap water and two wastewater effluent of petrochemical company were used for determination of As (III) and recovery percentages obtained were in the range of 95.4-105%.

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Sulfonamide‒functionalized melamine-based covalent organic framework for simultaneous removal of metal ions from aqueous solutions

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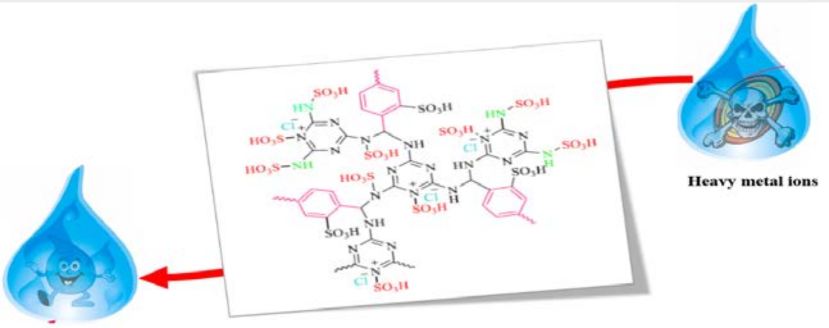

Abstract: Over the past decade, interest in the field of nanoporous materials as regular organic or inorganic frameworks has grown tremendously because of their excellent porous properties and broad applications. As an emerging kind of crystalline porous materials, covalent organic frameworks (COFs) pioneered by Yaghi and co-workers in 2005 are composed of two organic moieties via strong covalent bonds between light atoms (e.g., H, B, C, N, O, Si) to generate long-range ordered two-dimensional (2D) or three-dimensional (3D) network structures [1]. Disposal of industrial effluents and wastewaters is considered as one of the most important challenges in the industrial world today. Due to the toxic ingredients of these effluents such as, heavy metal ions and dyes, they are the main cause of pollution of rivers, lakes and underground waters. The most notorious heavy metals that cause significant environmental pollution are lead, chromium, mercury, cadmium, arsenic, zinc, copper, and nickel. Accumulation of these ions in the human body lead to various health consequences, such as: anorexia, gastrointestinal colic, anemia, neurasthenia, kidney and liver damage, and even cancer. Therefore, their removal before release into the environment using a suitable adsorbents is a necessity [2, 3].Herein, we report the sulfonamide-functionalized covalent organic framework (COF-SO3H) prepared from melamine and terephthalaldehyde as a new adsorbent for the removal of some heavy metal ions ions from environmental water and wastewater samples. The studied ions include Cu(II), Cd(II), Pb(II), Ni(II) and Zn(II). The physicochemical properties of COF-SO3H adsorbent was characterized by X-ray diffraction analysis (XRD), scanning electron microscopy (FESEM), Transmission electron microscopy (TEM), Energy-dispersive X-ray spectroscopy (EDAX), Thermo-gravimetric analysis (TGA) and Fourier-transform infrared spectroscopy (FT-IR) analysis. In this method, the batch removal process were designed by response surface methodology (RSM) based on a central composite design (CCD) model. Under the optimized conditions, the removal efficiency of metal ions were > 90 % at pH of 5.5, and maximum adsorption capacities of all ions was above 100 mg/g. Adsorption data showed that investigated ions uptake on COF-SO3H followed the Langmuir isotherm model equation and pseudo-second order kinetic model. The proposed method was successfully used to remove the studied ions from some environmental water samples and satisfactory results was achieved.

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Arabic Gum and Polyvinyl Alcohol Composite Decorated by Quercetin Nanoparticles as a Fluorimetric Film Shape Sensor for Meropenem Determination

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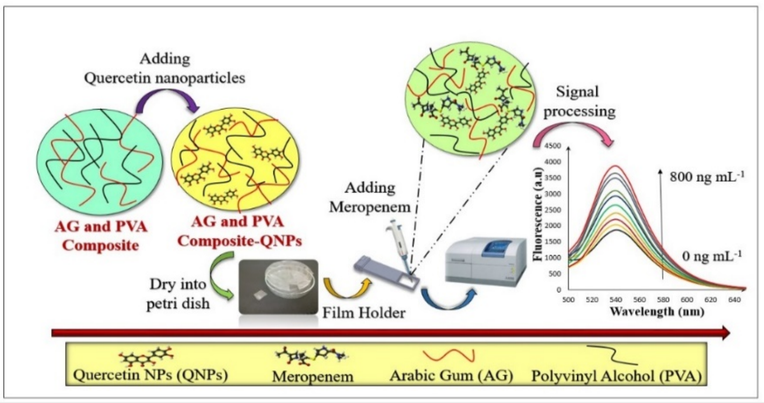

Abstract: Meropenem (MPN), chemically (4R,5S,6S)-3- [[(3S,5S)-5-dimethylcarbamoyl pyrrolidin-3-yl]-thio]-6-[(1R)-1-hydroxyethyl]-4-methyl-7-oxo-1- azabicyclo [3,2,0] hept-2-ene-2-carboxylic acid, with molecular formula ($C_{17}H_{25}N_3O_5S_1$) is a beta-lactam antibiotic belonging to the carbapenem group that has a very antimicrobial effect against a wide range of gram-positive and gram-negative bacteria that are resistant to other beta-lactam antibiotics. Meropenem is effective in treating a large number of infections such as urinary tract infections, respiratory tract infections, intra-abdominal infections and hospital bacterial infections. However, its excessive consumption leads to vomiting, nausea and headache [1-2-3]. Meropenem, like all beta-lactam antibiotics, binds to penicillin-binding proteins and inactivates them, inhibiting bacterial cell wall synthesis, thereby killing bacteria. This antibiotic has a very good penetration on most tissues and body fluids, such as lung tissue and the central nervous system [4]. In this research work, a new method was developed for the synthesis of arabic gum and polyvinyl alcohol composite decorated by quercetin nanoparticles as a fluorimetric film shape sensor to measure meropenem. In order to embed quercetin nanoparticles in the polymer composite substrate, quercetin nanoparticles were first synthesized and then added to the prepared composite solution under optimal conditions. The developed film sensor was placed in a holder made with 3D printer technology. The characteristics of arabic gum and polyvinyl alcohol composite and arabic gum and polyvinyl alcohol composite decorated by quercetin nanoparticles films, before and after of the addition of meropenem was studied by TEM, FT-IR and EDAX-Mapping. The fluorescence spectra showed that the maximum fluorescence intensity of arabic gum and polyvinyl alcohol composite decorated by quercetin nanoparticles was at λ_{em}=540 nm at λ_{ex}=380 nm. After adding meropenem to the sensor, the fluorescence intensity increased at the mentioned wavelengths. The meropenem solution with a certain concentration and volume was added on the film surface by microsampler. Finally, the difference in the fluorescence intensity of the fabricated film before and after the addition of meropenem was considered as an analytical signal for meropenem determination. The effect of different parameters on the fabrication of film-shaped fluorimetric sensors such as the concentration of polymer solutions, the volume of quercetin nanoparticles and the volume of glycerol were investigated. Factors affecting the measurement of meropenem such as pH, type and volume of buffer, volume of meropenem solution added on the sensor and time were also investigated. Under the obtained optimum conditions, the calibration graph was linear in the concentration range of 50-800 ng mL^{-1} with a correlation coefficient (r) of 0.9976 and the detection limit was 42.6 ng mL^{-1}. The relative standard deviation for eight replicate measurements of 100 and 400 ng mL^{-1} of meropenem was 3.53 and 1.42 %, respectively. The proposed method was successfully applied to the determination of meropenem in human urine, blood serum and pharmaceutical samples.

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Fe(III) doped TiO_2 nanoparticles prepared by planetary ball mill on Ni foam as efficient and novel electrocatalyst for oxygen generation

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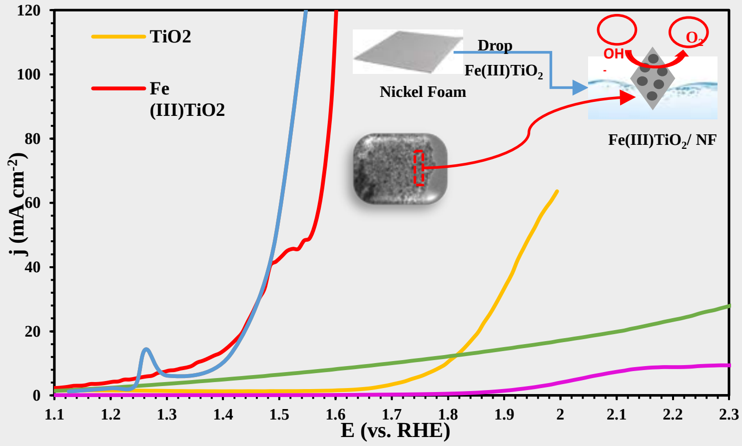

Abstract: Clean and renewable energy has gotten a lot of attention as a result of the growth of societies and the demand for more energy. Water splitting was one of these alternate energy sources. Because the oxidation of water is a delayed reaction, it's critical to use high-efficiency, low-cost electrocatalysts in the process [1-2]. This work presents the synthesis of Fe(III) TiO_2 by the planetary ball mill method. TiO_2 nanoparticles as anatase phase were synthesized using a solvothermal method and doped with Fe (III) by mechanochemical planetary ball mill method. The use of Fe(III) TiO_2 in a 3D porous Nickel Foam substrate, in which this conductive substrate can improve the electrocatalytic activity of the catalyst, electron transfer rate, and surface area. Nickel Foam also, the intelligent network structure substrate and conductive, provides the ideal electron path as the electrode affordable. The Fe(III)TiO_2/NF nano sensor demonstrates excellent stability and durability in aqueous environments, which makes it a very active, stable, low-cost, and promising catalytic system in the field of energy analysis, conversion, and storage. For structural and chemical characterization of the as-synthesized nanoparticles, techniques such as X-ray diffraction (XRD), energy dispersive X-ray (EDX), and transmission electron microscopy (TEM) were employed. XRD proved that the nanoparticles, after doping, consist of a mixture of anatase and brookite phases. The catalytic efficiency of OER was studied by linear sweep voltammetry (LSV), cyclic voltammetry (CV), and chronoamperometry (CA) techniques. The electrolysis of water using Fe(III)TiO_2/NF with a current density of 10 mA cm^{-2} can be achieved by a cell voltage of 1.35 (vs. RHE) volts in a solution of 0.1 M KOH. The catalyst synthesized to reach 10 mA cm^{-2} in oxygen evolution reaction only has 124 mV overpotentials.

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In Situ Synthesis of CuONP into Polyvinyl Alcohol-Borax Hydrogel Sensor for Determination of Tryptophan using a Smart Phone

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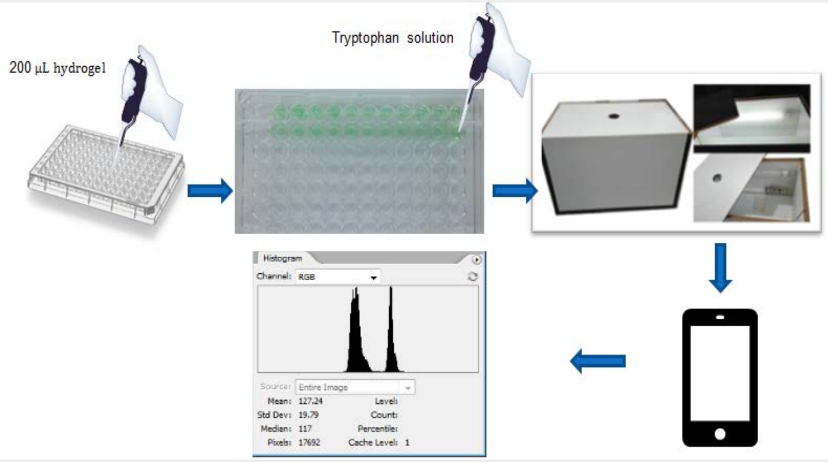

Abstract: Tryptophan is an essential amino acid consisting an α-amino group, an α-carboxylic acid group and a side chain indole which converts it to a non-polar aromatic amino acid [1]. The human body is unable to produce it and must be supplied through the consumption of foods containing L-tryptophan.When tryptophan is improperly metabolized, it produces a waste product in the brain that is toxic and causes hallucinations and delusions. Many methods are currently used to measure tryptophan, such as high performance liquid chromatography, spectrophotometry, and electrochemical methods. Chromatographic methods are often tedious and time consuming and require complex tools and a skilled operator, and are practically not simple methods. Spectroscopic methods involve difficult and slow steps with tryptophan modification by countless reagents [2]. Today, smartphone-based colorimetric quantification is becoming more and more widespread. In this work, a user-friendly, non-instrumental, cost-effective and portable method was developed that can rapidly determine tryptophan concentration by digital image processing-based colorimetric method. In this study, a red, green, blue (RGB) color sensor was used for quantitative optical analysis of tryptophan using copper oxide nanoparticles synthesized in borax polyvinyl alcohol hydrogel matrix. The hydrogel was characterized by EDX-MAP, FT-IR, UV–Vis spectrometry and SEM. By photographing the sensor and analyzing the images using Photoshop software, the relationship between the mean of the signals obtained by the RGB channels from the pixels of the experimental area for different concentrations of tryptophan. In order to increase the sensitivity and selectivity of the method, the effective factors in the tryptophan measurement were optimized using design expert software. Under optimal conditions, mean of RGB channels sensor was linearly related to the tryptophan concentration in the range of 5-100 ng mL^{-1} with a correlation coefficient of r=0.9967 and the detection limit was 1.43 ng mL^{-1}. From 7 repeated measurements at concentrations of 20 ng mL^{-1} and 60 ng mL^{-1} of tryptophan, the relative standard deviation was calculated to be 2.02% and 1.07%, respectively. The proposed sensor was successfully applied to the determination of tryptophan in chickpea, barley and milk samples.

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Extraction of Heavy Metal Ions from Water and Vegetable Samples Using DABCO-based Poly (Ionic Liquid) Functionalized Magnetic Nanoparticles

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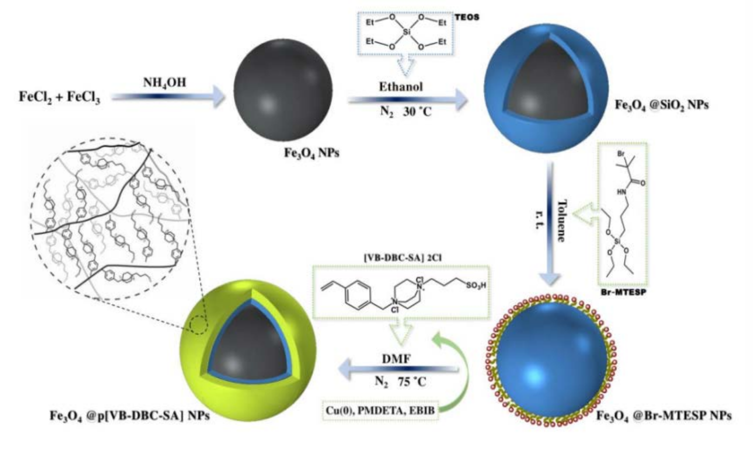

Abstract: A poly (ionic liquid) (PIL) functionalized magnetic nanoparticles methodology was developed and utilized as an efficient adsorbent for the simultaneous extraction of lead, cadmium, zinc, nickel, and copper ions from water and vegetable samples. The determination of target analytes was performed by electrothermal atomic absorption spectro0metry (ETAAS). The ionic liquid monomer (1-(3-sulfopropyl)-4-(4- vinylbenzyl)-1,4-diazabicyclo[2.2.2]octane-1,4-diium chloride) was synthesized for the first time. Then, this monomer was utilized to polymerize silica-coated iron oxide nanoparticles surface via the Cu(0)-mediated reversible-deactivation radical polymerization (RDRP) method. The synthesized nanomaterials were exhaustively characterized by SEM, TEM, FTIR, XRD, VSM, BET TGA, and XPS. Effective parameters on the extraction and enrichment of analytes were investigated by multivariate optimization methods using response surface methodology (RSM) based on central composite design (CCD). The validation experiments were carried out according to recommendations of the US Food and Drug Administration. The detection limits for water samples ranged between 3.8 to 11.4 ng.L^{-1}, and vegetable samples varied from 0.084 to 0.252 μg.kg^{-1}. The recovery ranged from 97.2 to 104.6% and 83.7 to 102.3% for water and vegetable samples, respectively. In particular, the purpose of this work was to validate a methodology to the analysis of Cd (II), Pb (II), Ni (II), Zn (II) and Cu (II) ions in water and vegetable samples by calculating measurement uncertainties using the bottom-up approach (GUM) in conjunction with in-house validation data. The relive measurement uncertainty ranged from 9.5 to 15.8%. The proposed method was successfully employed in the extraction and determination of heavy metal ions in the cucumber, tomato, pepper, and cabbage samples. Also, the developed method was applied to monitor the concentration levels of Cd (II), Pb (II), Ni (II), Zn (II) and Cu (II) in 52 bottled water samples. The proposed method is rapid, sensitive, environmentally friendly, and useful for monitoring the residues of heavy metal ions in water and vegetable samples.

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Cressa cretica leaves extract for the simple preparation method of CuO nanoparticles and its characterization

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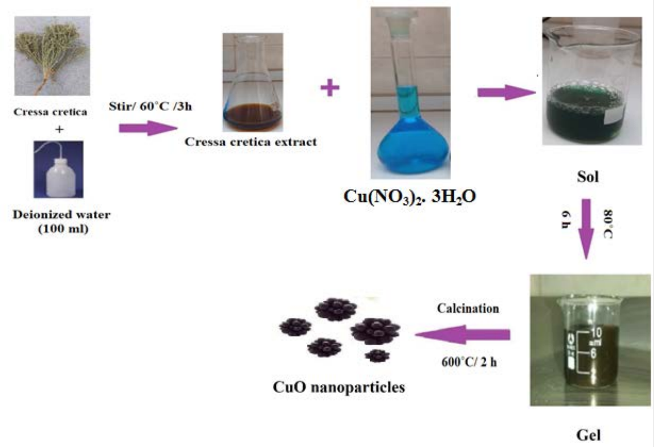

Abstract: In this work, we reported a simple method by a green approach for CuO NPs preparation in aqueous medium through Cressa cretica (C. cretica) leaves extract. This method gives some benefits that are harmless, low cost and simple preparation. In this study we used from c. cretica extract as a green reducing and as well as a capping agent and $Cu(NO_3)_2.3H_2O$ as a copper source. CuO nanoparticles were synthesized through a simple sol–gel route in the presence of C. cretica leaf extract without any surfactant and reducing agent. FTIR studies disclosed that hydroxyl, amide and amine groups of Cressa cretica leaf broth were liable for the formation and stabilization of the CuO nanoparticles [1].

The C. cretica plant leaves were washed thoroughly first with tap water to remove the adhering particles, then with distilled water. The purified C. cretica leaves were dried in shadow. The dried C. cretica leaves were crushed using mortar and pestle. The powdered C. cretica leaf was weighed (5.0 g) and mixed with 100 mL of distilled water. The mixture was stirred at 55 °C for 2 h and filtered and stored at 5 °C. After that, 30 ml of the plant extract solution was added to the 50 ml of 0.5M $Cu(NO_3)_2.3H_2O$ solution, drop by drop. The solution was stirred for 5 h in a paraffin bath at a constant temperature of 72 ˚C. Finally, at the plate's bottom, an extremely sticky, green-colored gel formed. The remaining product was dried for 4 h at 50 ˚C in an oven. Then, the sediments were calcined at 500 ˚C for 2 h, yielding a black powder. Furthermore, CuO NPs were characterized by XRD, FESEM, EDX, and UV–Vis spectrophotometery, Zeta potential and TGA/DTA techniques. XRD pattern was theoretically well-matched to CuO with a monoclinic structure. All diffraction peaks can be indexed as the typical monoclinic structure. Moreover, the well-defined and sharp CuO reflections in the observed XRD patterns verify the well-crystalline nature of CuO NPs. The result of thermogravimetric analysis of the precursor product showed that the proper calcination temperature was 500 °C. Finally, the results revealed that CuO NPs have spherical shape with the particle size range of 40 nm.

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Cost-effective removal of Cr(VI) ions from wastewater using supported melamine-based covalent organic framework

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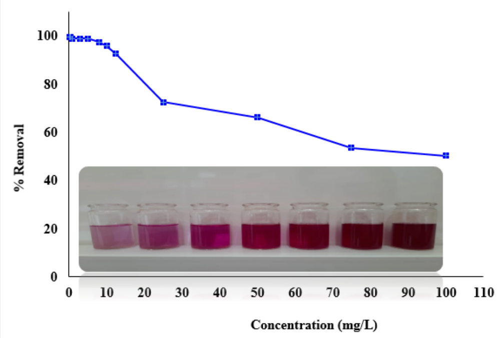

Abstract: Nowadays, environmental pollution with metal ions is one of the challenges in human societies. Heavy metals, which are inherently toxic, can be found naturally or through human activities in different hydrology and water sources, which may cause serious damage to the environment and require close monitoring of these species due to lack of treatment. Among heavy metal ions, chromium and its compounds enter the wastewater from many industries like metal plating, tanning, metal cleaning, dyeing, textile and refractory materials. Chromium (III) exists naturally in animal cells and is a vital element for maintaining the metabolism of glucose, lipids and proteins effective in mammals, whereas, it is well-known that Cr(VI) is a very dangerous metal pollutant with non-biodegradable and carcinogenic properties. Accumulation of Cr(VI) in the human body lead to various health consequences, therefore its removal before release into the environment using a suitable adsorbents is a necessity [1]. Covalent organic frameworks (COFs) are a class of materials that form two- or three- dimensional structures through reactions between organic precursors resulting in strong, covalent bonds to afford porous, stable, and crystalline materials. COFs have demonstrated a great potential in the removal of heavy metals due to its high adsorption capacity, large surface area, tunable porosity, porous structure, and recyclability. These features give COFs advantages over traditional adsorbents [2-3].

The aim of this work was to develop a new adsorbent for the removal of Cr(VI) ions from wastewater. In this regard, an amino acid supported melamine-based covalent organic framework was synthesized through a facile hydrothermal method. The structure and morphology of the synthesized COF-based adsorbent were characterized by different technique. The maximum adsorption capacity of the prepared adsorbent is 157.2 mg g^{-1} for removing Cr(VI) ions at ambient temperature and an optimum pH of 2.5. It was found that the adsorption data can be well fitted to the linear Langmuir model (R^2 = 0.996). The adsorption kinetics can also be well described by the linear pseudo-second-order model. To make a portable, highly sensitive, and low-cost device for in situ sensing of trace amounts of Cr(VI) ions, the smartphone colorimeter platform was developed using accessible equipment. The ability of suggested method with portable device can become an alternative to the conventional methods in field-analysis.

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Carbon Quantum Dots as a Novel Coating for Quartz Crystal Microbalance Sensing of Diethylamine Vapour

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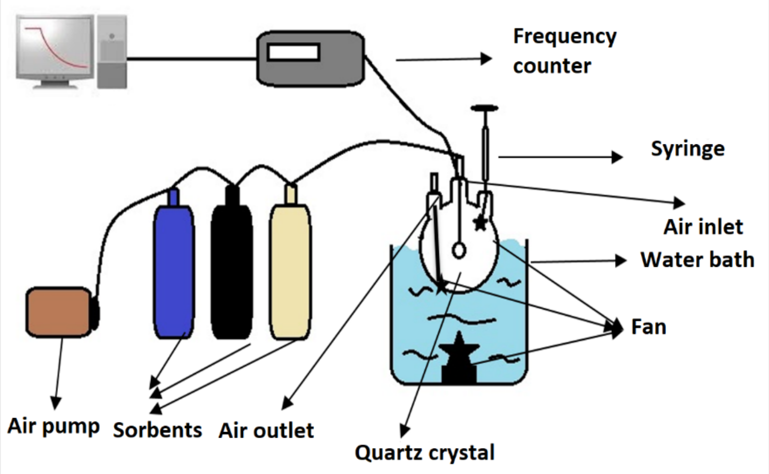
Graphical Abstract



Abstract: In this work, a quartz crystal microbalance (QCM) based mass sensor was developed for measurement of volatile organic compounds (VOCs). Quartz crystal microbalance (QCM) is a mass sensor which measure the frequency of a piezoelectric quartz crystal [1]. The frequency of the piezoelectric crystal is decreased upon an analyte adsorption on its surface which are proportional to the mass of adsorbed analytes [2]. QCM sensors are interested, due to their low cost, small dimension, portability and high sensitivity. In order to improve the sensitivity and selectivity of QCM sensors, different materials have been coated on their surface. In this research, the crystal surface of the QCM was coated for the first time with carbon quantum dots and applied for determination of diethylamine vapour. Carbon quantum dots were prepared from sugar and Galega dry extract by a microwave synthesis method and characterized by FE-SEM, EDX, AFM, UV-Vis, fluorescence and FTIR techniques. The prepared CQDs showed superior sensing results due of their high-water solubility, very small sizes (around 5-20 nm), various functional group such as hydroxyl and amino groups and forming homogenous and smooth thin film on the crystal surface. The developed sensor had a linear response over 70 to 200000 μgL^{-1} towards dimethylamine vapour. The LOD and LOQ of the developed method was 10 and 70 μgL^{-1}, respectively. The sensor relative standard error was less than 15%. The developed sensor with low cost, high efficiency, good sensitivity could be applied for sensing of amine compounds in the different chemical industries, environmental and biological applications.

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Fast and effective removal of cadmium and lead ions from aqueous environments using dithizone-modified mesoporous cellulose acetate

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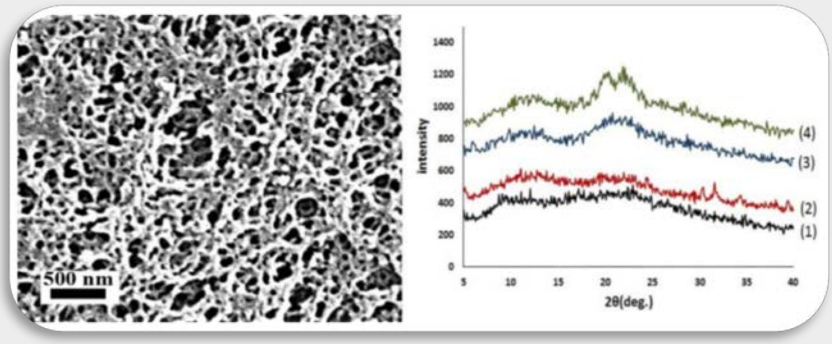
Graphical Abstract



Abstract: With the rapid industrial development, environmental pollution due to discharging trace metals into rivers and marine waters is getting worse. Lead and cadmium are two such trace metals enter into the environment. Existence these two trace metals at hazardous levels in water may attend to accumulate in living bodies thorough the food chain. Several techniques have been applied for the removal of lead and cadmium ions from wastewater such as chemical precipitation, coagulation-flocculation, ion exchange, adsorption, evaporation, biosorption and membrane filtration [1]. Nowadays, nano-adsorption techniques have been extended for water treatment. These techniques have some advantages over traditional treatment methods such as high area to volume ratio, flexibility to modify, selectivity, and feasibility to remove trace quantities of hazardous metals. Nanotechnology and nanoparticles are increasingly recognized for their potential applications in different branches of science. In the recent years, nanoparticles-based solid phase extraction (SPE) offers great possibilities for development of new analytical methodology, because of their unique properties, such as their large surface areas and high adsorption capacity. Cellulose acetate (CA) adsorbent derivatives like silver-loaded CA hollow fiber membranes, CA/polyethyleneimine blend microfiltration membranes, epoxy functionalized poly (ether-sulfone) incorporated CA ultrafiltration membranes, and so on have been recently applied to eliminate noxious compounds from water [2, 3]. The objective of the present study was to investigate the adsorption of lead and cadmium ions onto cellulose acetate mesoporous modified with dithizone, an agent that can form strong complexes with lead and cadmium ions. In this regard, a sorbent based on cellulose nanosponges modified with dithizone was prepared and characterized using various instrumental techniques. In this method, the batch removal process were designed by response surface methodology (RSM) based on a central composite design (CCD) model. Under the optimized conditions, the removal efficiency of metal ions were > 90 % at pH of 5.5, and maximum adsorption capacities of investigated ions was above 150 mg/g. Adsorption data showed that investigated ions uptake on CA@dithizone followed the Langmuir isotherm model equation and pseudo-second order kinetic model. The proposed method was successfully used to remove the cadmium and lead ions from some environmental water samples and satisfactory results was achieved.

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Supported Ru on Periodic Mesoporous Organosilica as Efficient Catalyst for Electrochemical Water Oxidation under Alkaline medium

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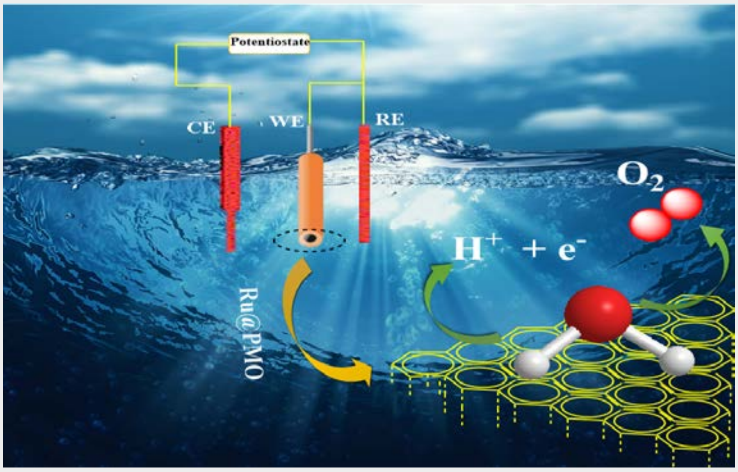

Abstract: Electrochemical water splitting is an appealing technology to produce high-purity hydrogen as a clean and sustainable energy carrier. The efficiency of water splitting largely depends on the intrinsic activity, selectivity, and stability of the electrocatalysts. Hence, soaring scientific endeavors have been made to develop high-performance electrocatalysts and uncover the underling reaction mechanisms. Ruthenium (Ru)-based nanomaterials are most promising for water splitting due to their favorable intrinsic activity, wide pH window, and high stability. In this study, a rationally designed Ru@PMO porous heterocatalyst containing ionic liquid framework (to stabilizing the metal on the substrate) were used for electrochemical water oxidation in alkaline medium (1 M KOH). This synthesized material catalyze water oxidation (WO), when it immobilized in carbon paste electrodes, with performances that exceed those of the benchmark system past catalysts, as probed by cyclic voltammetry (CV) and chrono amperometry (CA). This catalyst undergo continuous activation after polishing the surface of the electrode. The results herein reported highlight the remarkable potentialities of this catalyst. Ru@PMO oxidizes water with potential equal to 1 V vs. NHE by 1.4 mA. Cm^{-2} current. Then the current gets to 10 mA. Cm^{-2} at the potential equal to 1.06 V. The we observed maximum current equal to 197 mA at 2 V vs. NHE, that is much more than 3.60 mA. Cm^{-2} current density of Pt/C catalyst in PEM electrolyser at the same potential. It’s notable that this catalyst and also the metal free PMO (periodic mesoporous organosilica) substrate can oxidized water under the Nernst potential of OER half reaction.

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Ru and RuO2 Catalysts Supported on Ordered Mesoporous Organosilica for Electrochemical Water Oxidation in acidic medium

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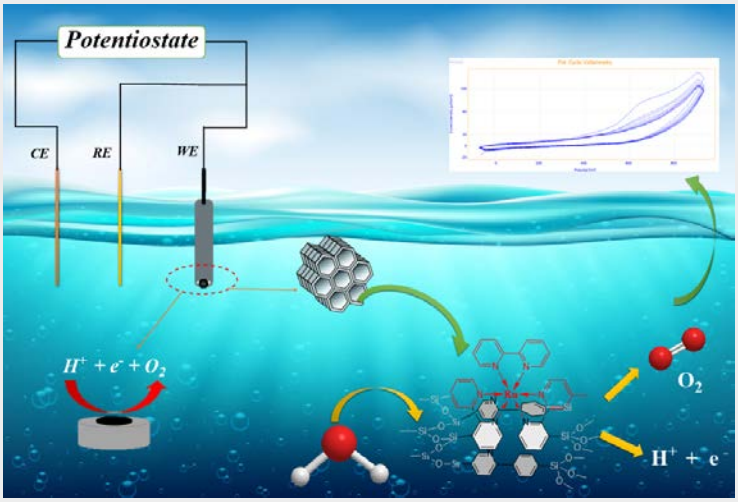

Abstract: Electrochemical water splitting constitutes one of the most promising strategies for converting water into hydrogen-based fuels, and this technology is predicted to play a key role in our transition towards a carbon-neutral energy economy. To enable the design of cost-effective electrolysis cells based on this technology, new and more efficient anodes with augmented water splitting activity and stability will be required. Herein, we report two active Ru@PMO and RuO2@PMO porous heterocatalysts containing ionic liquid framework (to stabilizing the metal on the substrate) were used for electrochemical water oxidation in acidic media (0.5 M H_2SO_4). These synthesized materials catalyze water oxidation (WO), when they immobilized in carbon paste electrodes, with performances that exceed those of the benchmark system past catalysts, as probed by cyclic voltammetry (CV) and chrono amperometry (CA). These catalysts undergo continuous activation after polishing the surface of the electrodes. The results herein reported highlight the remarkable potentialities of these two catalysts. Ru@PMO and RuO_2@PMO respectively reach to 14 mA. Cm^{-2} and 31 mA.Cm^{-2} at 962 mV vs. NHE and maximum current equal to 171 mA and 282 mA at 2 V vs. NHE, that is much more than 3.60 mA. Cm^{-2} current density of Pt/C catalyst in PEM electrolyser at the same potential. It's notable that these two catalysts and also the metal free PMO (periodic mesoporous organosilica) substrate can oxidized water under the Nernst potential of OER half reaction.

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Electrochemical Water Oxidation under Neutral Medium with Ruthenium Base Catalysts

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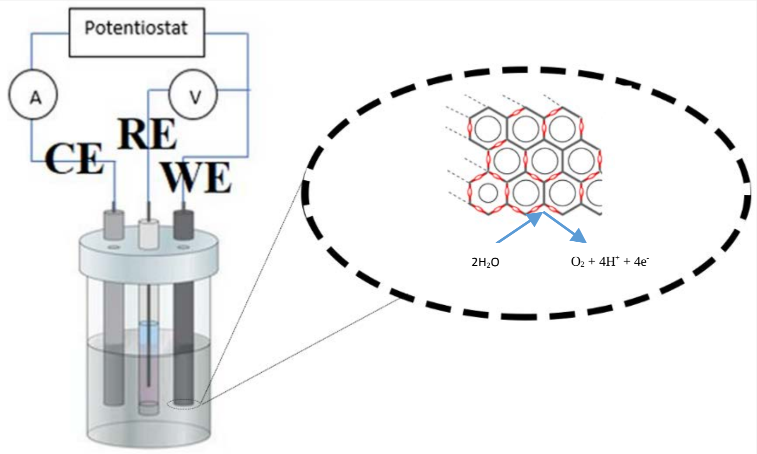

Abstract: Conversion of solar energy into hydrogen-based fuels is considered to be one of the most promising alternatives to fossil fuels [1]. Water is an inexpensive and readily available natural source of hydrogen, which has made electrochemical water splitting an intensively pursued research field in the recent decades [2]. However, the anodic reaction that leads to the oxidation of water to molecular oxygen is plagued by a high activation barrier, which has steered research towards the development of active and durable water oxidation catalysts (WOCs) [3-6]. The number of catalysts that catalyze water oxidation at high rate with low overpotential is limited, while Ruthenium based catalysts can be particularly active [3-6]. We already knew that doping of nanoparticles on surfaces is an efficient method for designing heterogeneous water oxidation catalysts that lead to improve the properties of catalyst [7]. In this study, a rationally designed Ru@PMO porous heterocatalyst containing ionic liquid framework were used for electrochemical water oxidation. This synthesized material catalyzes water oxidation, when it immobilized in carbon paste electrodes, with performances that exceed those of the benchmark system past catalysts, as probed by cyclic voltammetry and chrono amperometry. This catalyst undergo a continuous activation after polishing the surface of electrode. The results herein reported highlight the remarkable potentialities of this catalyst. Ru@PMO oxidizes water with current equal to 10 mA. Cm^{-2} at 1.19 V vs. NHE and maximum current is equal to 58.59 mA at 2 V vs. NHE. While current density of Pt/C catalyst in PEM electrolyser is 3.60 mA. Cm^{-2} at 2 V vs. NHE [8]. It's notable that this catalyst and also the metal free periodic mesoporous organosilica (PMO) substrate can oxidize water under the Nernst potential of oxygen evolution reaction.

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Adsorption for the Purpose of the Removal Chromium(VI) Pollutants in Water

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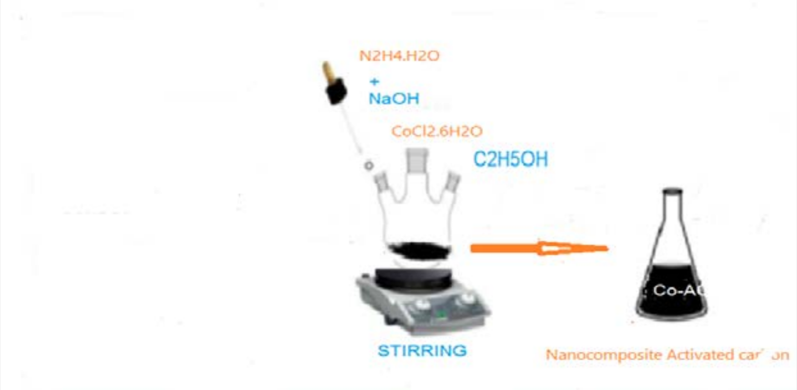
Graphical Abstract



Abstract: The removal of the Cr(VI) in wastewater is a critical because of its pisonousness and probable accumulation in the environment [1,2]. Cr(VI) has been classified as the priority contaminants through the US Environmental Protection Agency [3]. World Health Organization has set the permissible pollutants level for Cr(VI) at 0.1ppm in drinking water [4]. Exposed to such as chemicals can harm the central blood system, kidney, and respiratory system in case into human body. In recent years, adsorption has been successively employed in industrial procedure and wastewater treat involving Cr(VI) removal. In this study magnetic activated carbon/cobalt synthesized and examination for Cr(VI) ions removal. All parameters resulting the removal effectiveness such as pH, analyte concentration and adsorbent dosage, contact time was evaluated by UV-Vis spectrophotometer. The results showed that nanocomposites can be employed as a new adsorbent which has higher adsorption capacity and rapid adsorption rate in order to removal of Cr(VI).

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Synthesis of Cu-Nanocomposite Hydrogel as a Green Catalyst for Biginelli Reaction

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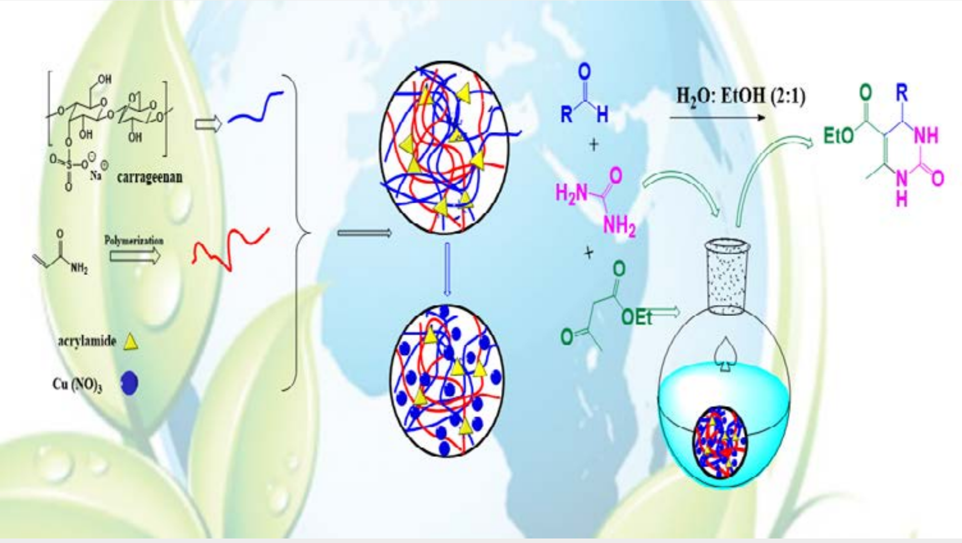
Graphical Abstract



Abstract: Biginelli Multicomponent reaction is one of the important reactions in synthetic organic chemistry which is due to the myriad of medicinal properties of its products. This reaction involves the combination of an aldehyde, a β-keto ester and urea. The use of catalyst results in higher reaction yields. Also, by using heterogeneous catalysts in the reaction, it can be easily recovered and separated from the products. Green nanocomposite hydrogel comprised of carrageenan, acrylamide (AAm), and N,N,N',N'-ethylene-bis(acrylamide) with copper ion was prepared as a heterogeneous catalyst and was used in Biginelli multicomponent reactions. The chemical structure and morphology of the hydrogel were investigated by FT-IR FE-SEM and XRD analyses, respectively. The use of a green solvent, high yields, short reaction time, straightforward reactions, easy recyclability, and simple separation of catalyst from products are the main features of the current method, which offers both financial and environmental benefits.

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Distinguishing of Autism Disorder by the Aptasensor Based on Screen Printed Carbon Electrode Modified with Gold Nanorods

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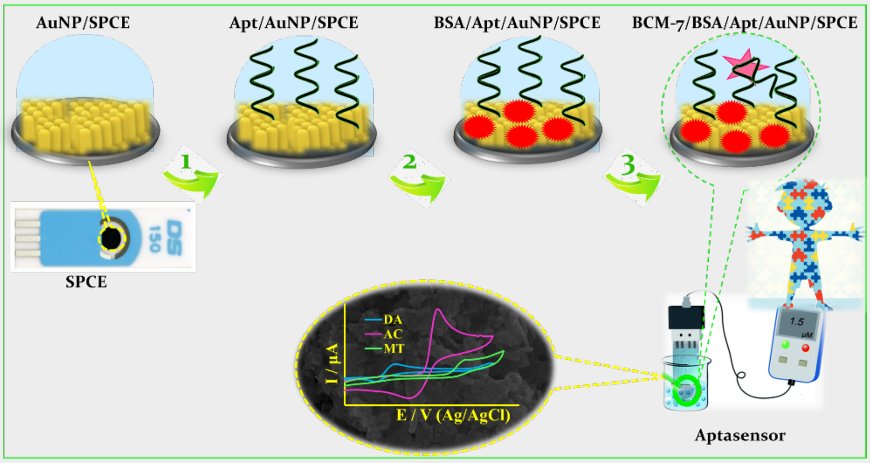
Graphical Abstract



Abstract: Autism spectrum disorder (ASD) is a complex neurodevelopmental disorder among children that is estimated to a ration of 1 in 160 in children all over the world by the world health organization (WHO) reports [1]. The higher levels of bovine β-casomorphin 7 (BCM-7) biomarker in autistic children's urine than in healthy children is correlated with the severity of the autistic symptoms. Despite some reported spectroscopic and chromatographic-based methods in BCM-7 detection, some practical disadvantages such as complexity, need for skilled expertise and even the inability to trace BCM-7 in the biofluids limits their applications [2,3]. To overcome these issues, developing an ultrasensitive and selective aptasensor for the accurately sensing of BCM-7 in human biofluids has great significance in ASD distinction. An electrochemical aptasensor has been developed based on a screen-printed carbon electrode (SPCE) modified with gold nanorod (AuNR). AuNR not only suggested a large specific surface area on the SPCE surface but also captured more Apt sequences by forming a chemisorption interaction between the amine-Au groups. By BCM-7 introducing onto the sensing interface, the Apt configuration was changed to form a BCM-7/Apt complex. The induced spatial barrier on the surface created a linear relationship with BCM-7 concentration increasing over a dynamic range of 1 fM to 25 nM with a limit of detection (LOD) of 334 aM. The well-evaluated applicability of the aptasensor in the presence of some interfering species and also in some human urine and plasma samples as actual samples can be promised the autism distinguish as early as infancy under clinical conditions [4]. The proposed strategy may be broadly applicable for fabricating other miniaturized biodevices because of utilizing cost-effective SPCEs to economically reduce the cost and replace the Apt sequence as the bioreceptor for different targets.

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Removal of Direct Black 38 Pigment Using SPE Method by LDH Al-Zn from Aqueous Samples

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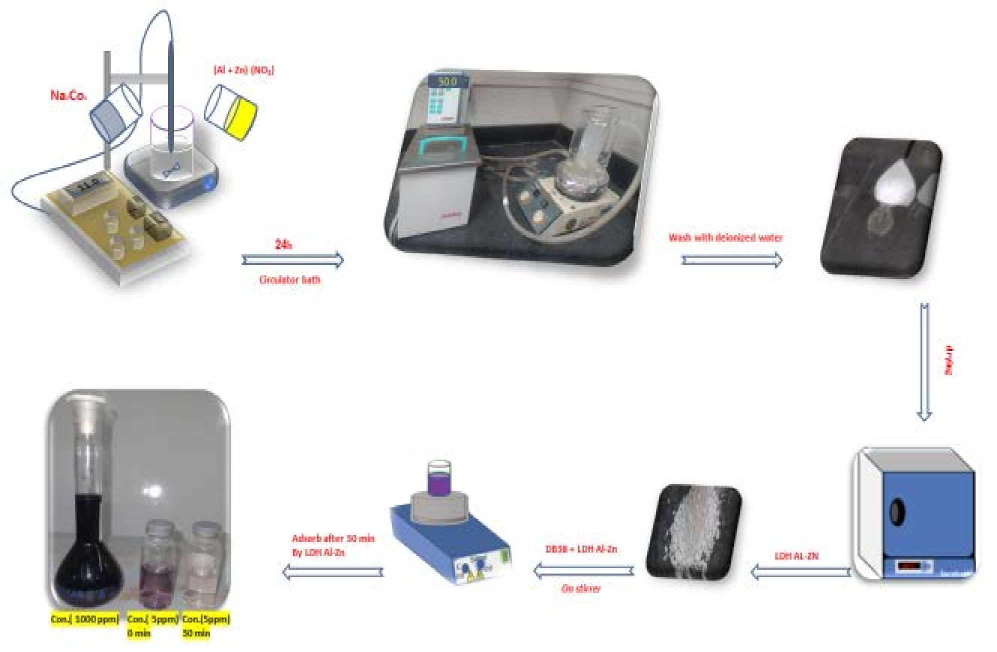
Graphical Abstract



Abstract: The pigments (dyes) especially Direct Black 38 being utilized in industrial activities are considered as potential organic pollutants having adverse effect on the environment, human and animal health. Hence, there is a fundamental need for identifying and treating environmental wastes [1]. Solid phase extraction technique (SPE) using different adsorbents is preferred for removal/ preconcentration of various analytes, due to its known advantages [2]. In recent years, layered-double hydroxides as tunable compounds have attracted the attentions of several researchers in different fields, including removal of various pollutants because of their advantages, such as relatively simple synthesis, non-toxicity, possessing a large specific surface area and high catalytic activity, biocompatibility [3]. In this research LDH AL-Zn with nitrate salt was synthesized, characterized and utilized as an adsorbent in the SPE method to remove and purify water sample containing Direct Black 38 pigment, for the first time. UV-Vis spectrophotometry as a simple and economical analytical technique rather than advanced analysis methods such as chromatography and electrophoresis was used for determination of analyte content in the samples. The effect of experimental parameters on the extraction efficiency was investigated and optimized as: sample pH=7, analyte concentration =5 mg/L, stirring rate = 300 rpm, equilibration time = 30 min. and adsorbent dose = 8 mg. Under the optimal conditions the extraction efficiency for the pigment was found to be more than 97%.

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Bioanalytical Chemistry

Ionic liquid based magnetic needle headspace single-drop microextraction combined with high performance liquid chromatography for the determination of chlorophenols in wastewater

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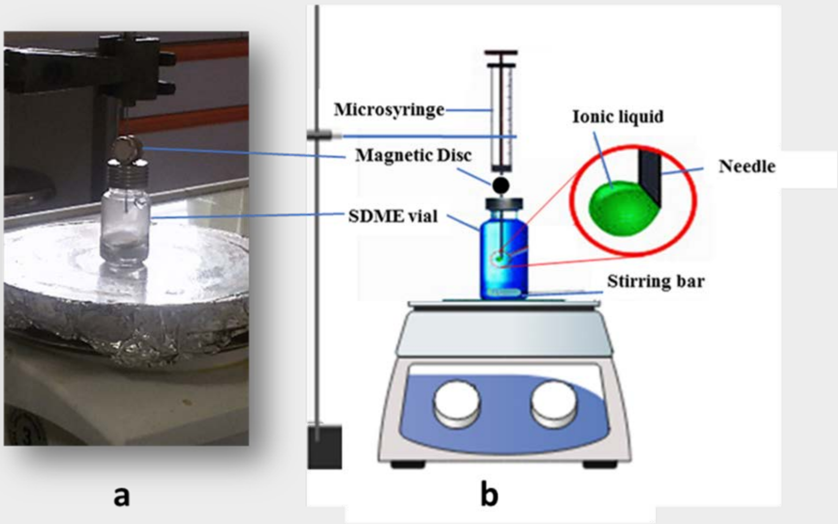
Graphical Abstract



Abstract: A magnetic needle headspace single-drop microextraction (MN-HS-SDME) method coupled to HPLC/UV has been developed. Trihexyl-(tetradecyl) phosphonium chloride was employed as an ionic liquid (IL) solvent for the headspace extraction of some chlorophenol compounds from wastewater samples. Despite of the non-magnetic character of the IL, a significant improvement in the extraction efficiency was obtained by magnetization of the SDME needle using a pair of permanent disc magnets. A simplex method for the fast optimization of the experimental conditions (e.g, stirring speed, ionic strength, pH, extraction time, and temperature) was used. The coefficients of determination (R^2) varied between 0.9943 and 0.9996, the limits of detection were from 0.004 to 0.007 µg ml^{-1} and, the recoveries were in the range of 88% to 120% for the studied analytes. The developed MN-HS-SDME HPLC/UV method was successfully applied to the determination of chlorophenols in industrial wastewater.

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Using of ultrasonic assisted dispersive liquid–liquid microextraction based on deep eutectic solvent for Simultaneous determination of illegal dyes sudan orange G and sudan red 7B followed by derivative UV-VIS spectrophotometry

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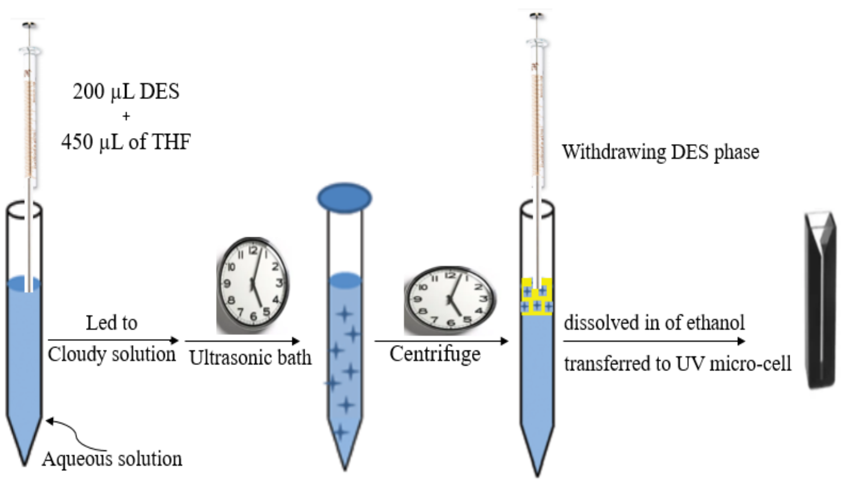
Graphical Abstract



Abstract: In this study, simultaneous preconcentration and determination of illegal dyes sudan orange G and sudan red 7B was performed using the method ultrasonic assisted dispersive liquid–liquid microextraction based on deep eutectic solvent with second derivative ultraviolet spectrophotometry. Deep eutectic solvent was synthesized by mixing TBAB (HBA) and decanoic acid (HBD). Under optimized conditions including 1:3 molar ratios for TBAB and decanoic acid, pH 5.0, 10 minutes Centrifugation, 200 µL DES as the extraction solvent, 450 µL of THF as the disperser solvent, 3 minutes ultrasonication, and 5.26 % (w/v) of NaCl, the relative standard deviations of intra-day (RSD, n=3) were found 3.5 and 5 % for SOG and SR7B, respectively. Also, the RSD of inter-day were 10.5 and 9 % for SOG and SR7B, respectively. The limit of detection for SOG and SR7B were obtained as 0.014 and 0.011 µg mL^{-1}. A good linearity range ($r^2 > 0.991$) was obtained in the range of 0.036 – 0.900 and 0.030 -1.000 µg mL^{-1} for SOG and SR7B, respectively. The preconcentration factor for both dyes were calculated 60 for all experiments. The effects of interfering ions and dyes were investigated. Finally, the applicability of the method was applied for different real samples, including in chili powder, lipstick, river water and also in well water that relative recovery percentage of 88 to 114 was obtained for these samples.

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Green-Prepared Nickel Oxide Nanoparticles Using Cressa cretica Leaf Extract

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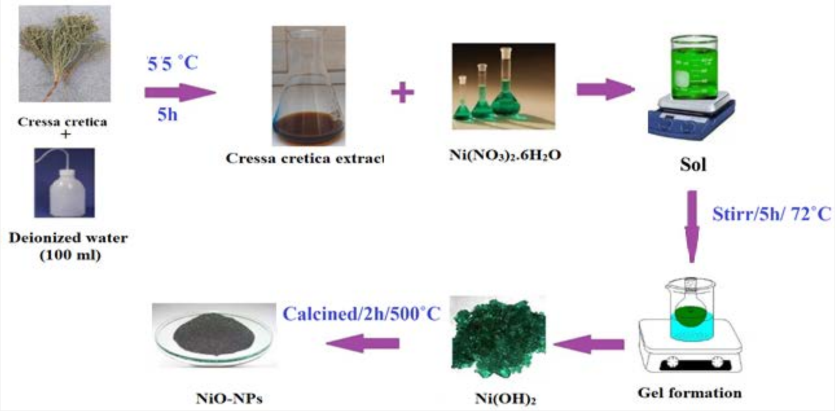
Graphical Abstract



Abstract: In this work, we aimed to develop a simple and cheap procedure for the synthesis of nickel oxide nanoparticles (NiO-NPs) using aqueous Cressa cretica (C. cretica) leaf extract as a green reducing and stabilizing agent and nickel nitrate hexahydrate as a nickel source [1]. In the present work, NiO nanoparticles were synthesized through a simple sol–gel route in the presence of C. cretica leaf extract without any surfactant and reducing agent.

The C. cretica plant leaves were washed thoroughly first with tap water to remove the adhering particles, then with distilled water. The purified C. cretica leaves were dried in shadow. The dried C. cretica leaves were crushed using mortar and pestle. The powdered C. cretica leaf was weighed (5.0 g) and mixed with 100 mL of distilled water. The mixture was stirred at 55 °C for 2 h and filtered and stored at 5 °C. After that, 30 ml of the plant extract solution was added to the 50 ml of 0.5M nickel (II) nitrate hexahydrate ($Ni(NO_3)_2.6H_2O$) solution, drop by drop. The solution was stirred for 5 h in a paraffin bath at a constant temperature of 72 ˚C. Finally, at the plate's bottom, an extremely sticky, green-colored gel formed. The remaining product was dried for 4 h at 50 ˚C in an oven. Then, the sediments were calcined at 500 ˚C for 2 h, yielding a black powder.

Also, NiO-NPs were identified by XRD, FT-IR, FESEM, EDS, UV-Vis, Zeta potential and TGA/DTA techniques. X-ray diffraction result revealed that the obtained product was nickel oxide with face-centered cubic structure. Analysis of FTIR spectra confirmed the composition of product. The result of thermogravimetric analysis of the precursor product showed that the proper calcination temperature was 500 °C. The results FESEM and EDS showed that NiO-NPs were pure with an average particle size of about 52 nm. NiO-NPs has zeta potentials of -19.1 mV at pH=7. The stability of produced particles in water is confirmed by this result [2].

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A colorimetric paper-based sensor with nanoporous SBA-16 for the sensing of Amino Acid cysteine in Urine

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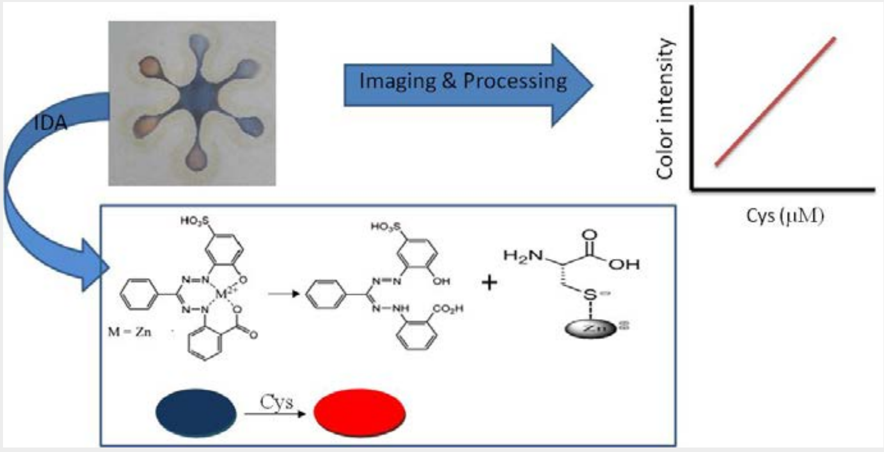
Graphical Abstract



Abstract: We described a method for the determination of cysteine in urine sample by using a microfluidic paper-based sensor modifies with SBA-16 [1]. Cysteine is an essential amino acid in humans. Cysteine metabolism problems like cystinuria, exist and are marked by increased cysteine levels in the blood or urine. SBA-16 mesoporous silica molecular was successfully synthesized and characterized by Fourier transform infrared spectroscopy, powder X-ray diffraction and used on the surface of paper based sensor [2]. The paper-based microfluidic sensor was made with a wax pen, a piece of filter paper and modified by mesoporous SBA-16 and applicated it to determine the quantity of cysteine Amino Acid in Urine sample. The sensor consist of six distribution channels: 6 circle zones with diameter of 6 mm on the ends of the channels and one circle zone with diameter of 8 mm in the center. The fabricated sensors for cysteine assay are operated based on an indicator displacement assay (IDA) [3]. The sensor was optimized for determination of cysteine. The image of the colorimetric assay was captured with a camera and was opened by Image J software in RGB color format so the image was inverted and the mean red values in detection zone were obtained by subtracting the blank value. The linear range of the calibration curve and the detection limit (S/N=3) were between 0.1 to 90 µM, 1.2 µM. The recoveries for the human urine samples analysis were in the range of 92%–111% for cysteine**.** So that the paper-based sensor method in this work could be used to determination the cysteine in urine.

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Marine Pathogens Classification Based on Raman Spectra Using Deep learning

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Abstract: Machine learning and deep learning approaches have been an important impact in different fields of chemistry, especially analytical chemistry, and it has lots of potential for the future. Using Raman spectra to identify and predict specific tasks has been a subject of interest [1]. Accurate determination and prediction of pathogens in the environment have been gathered in different researches to develop fast and accurate models to predict this specific property [2]. Therefore, we developed a deep learning model based on Raman spectra data that was gathered and published by Yu *et al* [2] to generate a more accurate model. The model architecture consists of a dense layer with an input shape of 1200, a batch normalization layer, and a dropback layer. These layers were repeated three times, and at the end, a single dense layer with softmax activation and output of eight was put to predict the class of the Marine Pathogens based on their Raman spectra data. The input data contains of 1200 rows. Our model outperformed the previous model by achieving the average accuracy for 5fold cross-validation of 99.91% and the recall of 99.9. For the random split model, data is separated into two different datasets. 70% on the training dataset and 30% on the test dataset; in this situation, the proposed model achieved 100% accuracy and the recall of 100% on the test dataset. For both of these process, a checkpoint method was used to save the best model based on its performance on the test dataset. The deep learning models were generated using TensorFlow version two. All the codes, data, and trained models are available free of charge at this GitHub repository.

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Identification and measurement of active ingredients aerial parts "Artemisia Annua" and antibacterial effects by MIC, MBC and Disk diffusion methods

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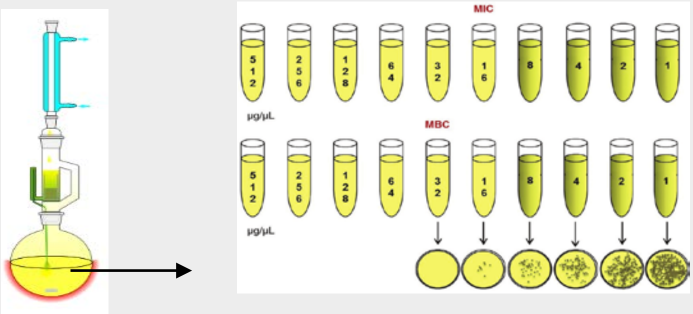

Abstract: More than 80% of the world's population relies on traditional medicine (herbal extracts or their active ingredients) for their health needs. In addition, more than 50% of all modern clinical drugs are of natural origin. Herbs may have the ability to resist against bacteria and many types of antibiotics. Totally, many plants have a high ability to fight bacterial and fungal pathogens [1]. Artemisia is a genus of small plants or shrubs found in temperate regions of northern Iran. This plant belongs to the important composite family (Asteraceae), one of the largest plant groups, which includes about 1000 genera and more than 20,000 species. The compounds in the Artemisia Annua have antimicrobial properties. The purpose of this research is investigation of the antifungal and antimicrobial effect of alcoholic extracts of plant Artemisia Annua on the microorganisms in the laboratory conditions [2].

In this study, Artemisia Annua, was dried at room temperature after collecting and confirming its species, and then extracted by Soxhlet apparatus with ethanol. Identification and study of plant compounds were performed using GC-Mass. Next, for complete removal of solvent, it was poured on a watch glass for 48 hours and dried [3]. Disk diffusion test was carried on the obtained extract by concentrations of 100 mg.mL^{-1}, 50 mg.mL^{-1}, 25 mg.mL^{-1}, 12.5 mg.mL^{-1}, 6.25 mg.mL^{-1} and 3.125 mg.mL^{-1} in the culture medium containing BHI Agar. Also Minimum Bactericidal Concentration (MBC) and Minimum Inhibitory Concentration (MIC) tests were carried by concentrations of 400 mg.mL^{-1}, 200 mg.ml^{-1}, 100 mg.mL^{-1}, 50 mg.ml^{-1}, 25 mg.mL^{-1}, 12.5 mg.mL^{-1}, 6.25 mg.mL^{-1}, 3.125 mg.mL^{-1}, 1.56 mg.mL^{-1} and 0.78 mg.mL^{-1} Broth in culture medium containing BHI and the results were evaluated after 24 hours of incubation. MIC effect of ethanolic extract of Artemisia Annua on Staphylococcus epidermis was 0.81 mg.mL^{-1} and for Escherichia coli it was obtained as 6.01 mg.mL^{-1}. Also in MBC method in 3 higher concentrations had lethal properties. The mechanism of action of the active ingredients of this plant is the destruction of the bacterial cell wall. According to the results of ethanolic extract of Artemisia Annua showed a better inhibitory effect on gram-positive bacteria Staphylococcus epidermis in comparison with gram-negative bacteria Escherichia coli and also has a lethal effect. Finally, the ethanolic extract of Artemisia Annua, can be used as raw material in the manufacture of cosmetic products due to its inhibitory and lethal properties of skin microorganisms.

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The efficient degradation of trifluralin via electrochemically generated sodium ferrate. A green and easily scalable method

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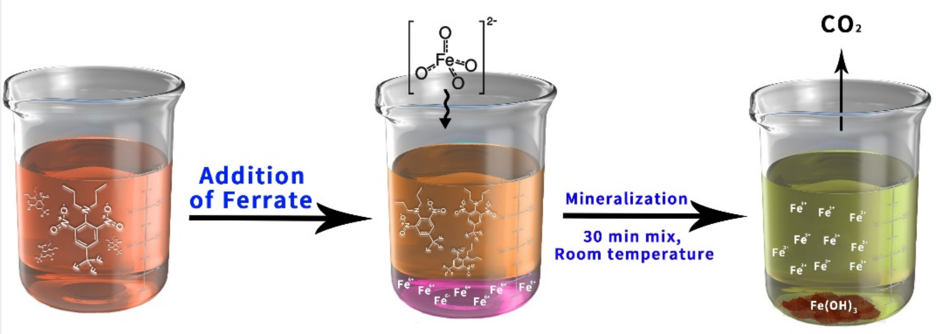
Graphical Abstract



Abstract: In today's world, water pollution has become a serious issue for the whole globe. Unfortunately, over the past decades, due to severe population growth, industrialization, agricultural activities, and climate changes, the quality of available water resources has gradually diminished, and most of them are polluted with various compounds [1]. Therefore, the water treatment processes are one of the important environmental concerns of government authorities and researchers. In the current decade, thousands of organic, inorganic and biological water pollutants are reported as dangerous compounds for humans' health, aquatic conditions, and the ecosystem [2]. Trifluralin (TRF) is a 2,6-dinitro herbicide. It has been extensively used in agricultural applications as a selective and pre-emergency compound in various plant cultures since the 1970s. Because of existence of nitro groups, TRF is a stable pollutant of food, groundwater, and surface water and there are also numerous reports evaluating TRF for genotoxicity and immunotoxicity [3]. To the best of our knowledge, conventional technologies of water treatment methods are not efficient enough to completely mineralize TRF to non-dangerous compounds under mild and economic conditions at a semi-industrial scale. However, the electrochemical processes offer several significant advantages over the analogous conventional techniques for the oxidation or the removal of contaminants, such as low energy requirements, in situ generation of some starting reactants, and high potential for process control [4]. On the other side, iron species are well-known abundant materials to perform scalable environmental-friendly applications (especially water treatment process). It must be noted that among the different forms of iron, Ferrate (Fe(VI)) salts have garnered much attention over the past several years because of their application in the coagulation/precipitation of toxic metals, the oxidative transformation of pollutants, and also the complete degradation of emerging contaminants [5]. Therefore in this research project, we firstly used the constant-current electrolysis (CCE) as a straightforward and cost-effective method for the generation of sodium ferrate (Na_2FeO_4) solution, and then we used the electrochemically generated ferrate for the degradation of TRF under the mild conditions. In the next part, the influence of several parameters on the degradation process of TRF and COD removal efficiency have been optimized. Finally, the obtained data from cyclic voltammogram, UV-Vis spectrum, and COD analysis of TRF have perfectly confirmed the full degradation of TRF to non-dangerous compounds.

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Coupled graphene oxide with Provskite nanomaterials as potential electrochemical modifier agent for precise detection of Morin within Kiwi samples

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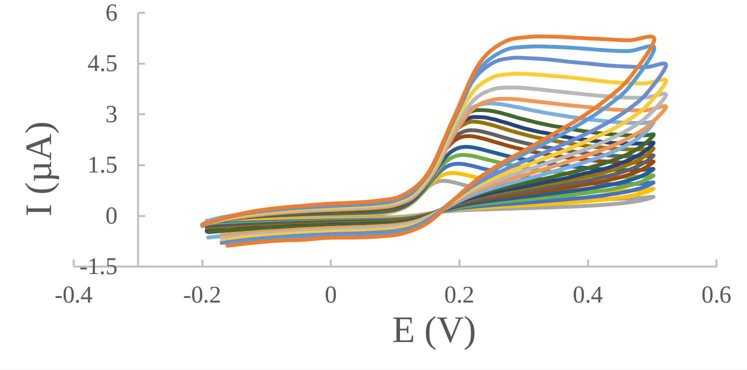
Graphical Abstract



Abstract: Flavonoids are considered to be natural antioxidants found in plants that have anti-viral, anti-inflammatory, anti-mutagenic, anti-carcinogenic and anti-stress activities. Morin is an important phytochemical Pentahydroxyflavone that contains a wide range of polyphenol compounds, mainly found in fruits, vegetables, and some Chinese herbs, due to its potential applications in the food, pharmaceutical, and cosmetic sectors. Have attracted a lot of attention.

In the present study, perovskite / graphene oxide nanosheets were used to modify the surface of a glassy carbon electrode to identify and measure the morphine composition in a true kiwifruit sample. To ensure the accuracy of the synthesis of the desired nanosheets, the structure and particle size of the mentioned compound and the modified electrode were examined by various identification techniques including SEM, XRD, FT-IR, EDS. The electrochemical behavior of Morin on the surface of the carbon glass electrode of the above modified glass was investigated using cyclic voltammetry techniques. A difference with a linear range of 10 to 110µM and a detection limit of 4.49µM was obtained. The results obtained from the sensor designed for Maureen measurement indicate appropriate parsing characteristics including low detection limits, wide linear range and good performance for Maureen measurement. Also, in order to check the accuracy, the recovery values of 97.5 indicate the high performance of the sensor designed in real samples. Stability, reproducibility and high reproducibility of the modified electrode are other prominent features of the designed sensor.

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Label-Free Impedimetric Aptasensor for Methamphetamine Detection based on Ceria Nanocomposite

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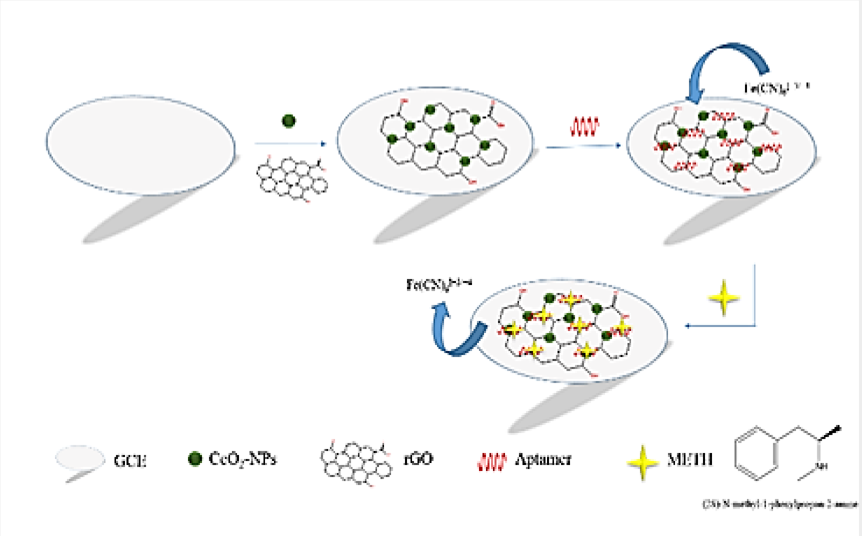
Graphical Abstract



Abstract: Methamphetamine (METH), a potent central nervous system stimulant, is the second most widely abused drug in the world [1]. Based on its chemical structure, it can be an electroactive species. However, its oxidation on the electrode surfaces is not facile. In this work, a novel aptasensor for methamphetamine (METH) detection is introduced. The structure of the METH, cerium oxide (called nanoceria; CeO_2 NPs) decorated on reduced graphene oxide (rGO) [2,3] was used as glassy carbon electrode modifier to study the redox behavior of METH. The aptamer selectivity and sensitivity has been enhanced by using the carbon nanomaterial. A suitable amount of CeO_2 and aptamer on rGO showed a synergistic electrocatalytic effect for optimizing the METH signal. Electrochemical impedance spectroscopy using $[Fe(CN)_6]^{-3/-4}$ redox probe was used as detection technique. The obtained system also exhibits a high outstanding repeatability and selectivity and Different experimental parameters were optimized to achieve the maximum response. Under the optimized experimental conditions, the lowest linear dynamic range for the proposed aptasensor were obtained 0.5 nM to 250 nM. In addition, the obtained results revealed that limits of detection (LOD) of the label-free impedimetric detection of METH was about 0.16 nM. The designed aptasensor exhibits an excellent repeatability and selectivity, and was effectively used to determine METH in spiked urine sample. At last, to be concluded that , wide linear range, low detection, high stability, simplicity, low cost, good reproducibility, compatibility with microfabrication, high sensitivity,large active surface, and fast response were obtained, showed proposed modified electrode can be potentially applied for the addictive drugs determination.

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The Role of Nitrogen Moieties in N-Doped Ordered Mesoporous Carbons for Spontaneous and Electrochemical Grafting of Redox Active Materials

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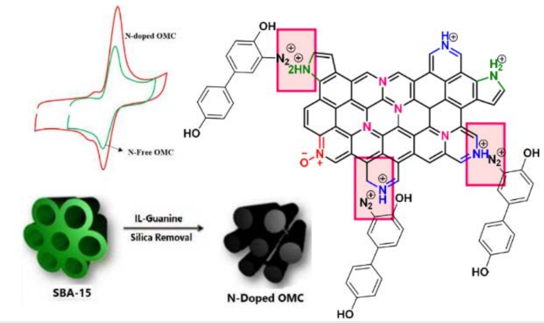

Abstract: Ordered mesoporous carbons (OMC) have been receiving continues attention for using in electrochemistry in recent years [1]. The unique capabilities of facilitating mass transfer, considerable electrical conductivity, tunable porous channels and high surface area together make them to be suitable candidate materials for fabrication of redox-active electrodes [2]. The chemical and structural properties of OMC can be improved by doping different heteroatoms such as nitrogen, phosphorus, sulfur, boron, and multi heteroatoms in their framework as the structure defects in order to enhance the corresponding performance in wither chemical or electrochemical applications. [3, 4]. Among heteroatoms, modification of the electrode surface using nitrogen-doped OMC has diverse implications in electro-analytical chemistry to fabricate redox-active electrodes with improvement of the electrode/electrolyte interfacial properties [5, 6]. The redox-active electrodes have a wide application in materials science, electroanalytical chemistry, electrocatalysis, biosensing, energy storage, and electronic and optoelectronic systems. The electro-active modified electrodes are constructed via either spontaneous adsorption or electrochemical grafting. In the later, the active redox molecules are covalently bonded to the surface of carboneous materials. The current study mostly deals with the role of nitrogen moieties in OMCs for more effective loading of redox active species through both adsorption and electro-grafting protocols. In this direction, a nitrogen-doped OMCs bearing high nitrogen content were fabricated by the carbonization of a mixture of ionic liquid (1-methyl-3-phenethyl-1H-imidazolium hydrogen sulfate) and Guanine using ordered mesoporous silica SBA-15 as hard template. A few electro-active moieties were then used as model to electrochemically functionalize the surface of the resulting N-riched OMC. In this way, a redox-active electrode was fabricated via grafting of various electro-active species with different electronic properties onto OMCs/GCE. Our investigation showed that not only the charge on the adjacent carbon atom can markedly influence the surface electro-grafting of redox-active materials, but nitrogen content also exerts a dramatic impact on electrical conductivity, defect site, the electronic nature of the carbons, etc. The mentioned properties enabled us to successfully prepare varied electrodes having different loading of electro-active species on the electrode surface. The outcome of the present work reveals changing the pH of the solution and the nature of substituents on electro-active species leads to significant differences in loading of redox-active molecules.

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Synthesis and characterization of layered double hydroxide bimetallic nanostructures and their application as supercapacitor

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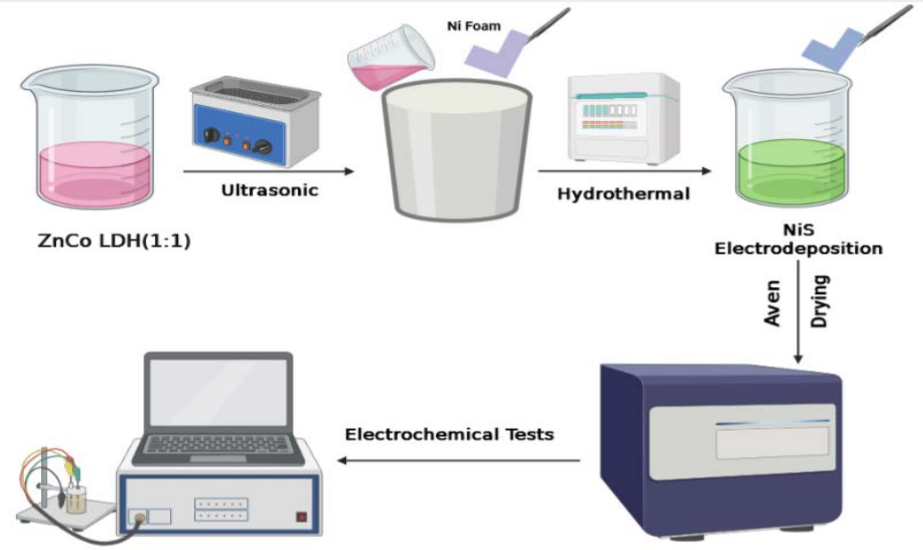

Abstract: Supercapacitors as one of the electrical energy storage devices differ from conventional capacitors due to their fast charge–discharge rates, longer life cycle, high power, and high energy density [1]. Recently several activities have been carried out to increase their efficiency in various fields, including more efficient electrode materials [2]. Layered double hydroxides (LDHs) have received scientific attractions due to their unique two-dimensional (2D) structure, band gap tunability, and composition flexibility [3]. In this study, a hierarchical ZnCo LDH@NiS core–shell nanostructures with a battery-like nature were synthesized by the hydrothermal and electrodeposition methods for hybrid supercapacitors. In a three-electrode cell, the ZnCo LDH@NiS core–shell acquires excellent capacity (2448 F g^{-1}, 297.2 mAh g^{-1} at 2 A g^{-1}), considerable rate performance and a good durability which is superior to the ZnCo LDH and NiS electrodes. Moreover, the asymmetric supercapacitor (ASC) which was fabricated with ZnCo LDH@NiS as a positive electrode and activated carbon as negative electrode demonstrated the maximum energy density of 38.016 Wh Kg^{-1}, power density of 412 W Kg^{-1} and desirable cyclability. The present synthesis provides a praiseworthy source for the fabrication of other kinds of metal LDH/sulfide electrode materials with core–shell structures for various applications.

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Detection of phthalic acid esters on Fe_3O_4@Azolla modified electrode with electrochemical impedance spectroscopy method

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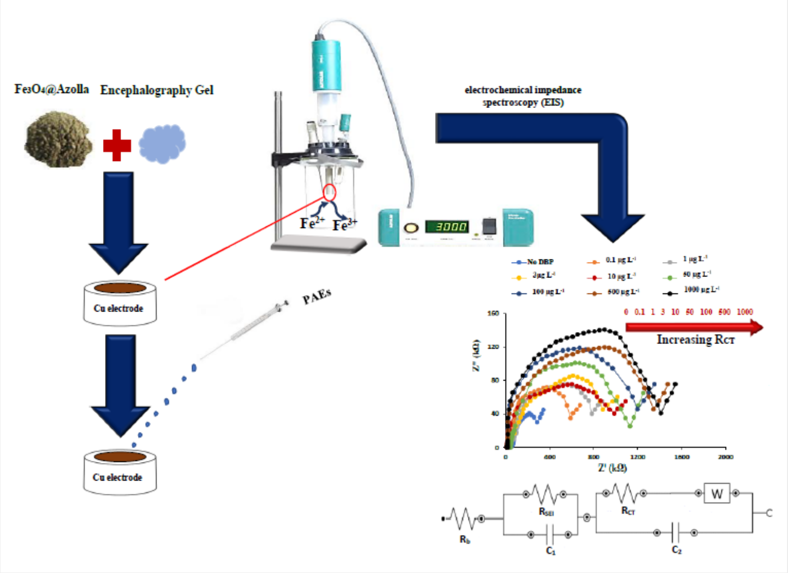

Abstract: Phthalic acid esters (PAEs) are very widely employed as additives for polymers in plastic, particularly in polyvinyl chloride (PVC) and polyethylene terephthalate (PET), which are applied in rubber, cellulose, and in the production of styrene as well [1]. PAEs help to improve the flexibility, transparency, and durability of articles manufactured with polymeric matrixes due to its low production cost. Unfortunately, some illegal food producers take advantage of the above feature of PAEs, adding PAEs instead of palm oil into food products to sustain the desired texture, resulting in severe harm to consumers [2]. Besides intentional addition, PAEs migrate from plastic packaging to the articles of consumption. In this work, a sensitive and efficient electrochemical sensor was introduced and the interaction of four PAEs including dibutyl phthalate (DBP), dimethyl phthalate (DMP), di(2-ethylhexyl) phthalate (DEHP) and dicyclohexyl phthalate (DCHP) in aqueous solutions with this sensor was separately investigated by electrochemical impedance spectroscopy (EIS). EIS analysis is a technique that measures resistance (R), capacitance (C), and inductance (L) by monitoring the current response while an AC voltage is applied to an electrochemical cell [3]. The surface of a copper electrode was modified by Fe_3O_4@azolla paste prepared using Fe_3O_4@azolla powder and the electroencephalography gel (EEG). Modifiers were characterized by FESEM and TEM methods. Due to the Nyquist plots, the charge transfer resistance (R_{CT}) of bare electrode and Fe_3O_4@azolla electrode were 468.8 and 285.1 kΩ. After separately injection of 3 μg L^{-1} DBP, DMP, DEHP and DCHP, R_{CT} were obtained as 593.1, 934.4, 529.6, 469.2 kΩ, respectively. The linear relationships were found between ΔR_{CT} and the logarithm of PAEs concentrations and the limit of detection (LOD) and limit of quantification (LOQ) values were in the ranges of 0.008-0.009 μg L^{-1} and 0.027-0.031 μg L^{-1}. According to the results, this sensor can be used to determine PAEs in real aqueous samples with good recovery percentages ranging from 93.3%-99.3% (RSD < 2.45 %).

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Voltammetric Determination of Pb^{2+} in the presence of Bi^{3+} at Food Samples by a Fe_3O_4@Shicff Base Network Modified Glassy Carbon Electrode

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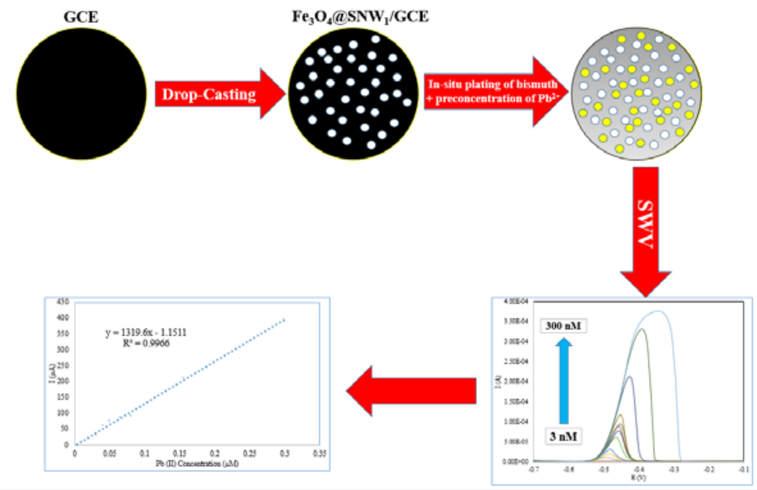
Graphical Abstract



Abstract: Lead is a carcinogenic and neurotoxic heavy metal that it is associated with several health issues at humans even at ultra-trace amounts like neuropathy, liver damage, hallucination, seizures, tremor and coma. In this respect, developing a sensitive, rapid, selective, economic and simple analytical method for its determination is of great importance. As a consequence, in this research, a highly sensitive electrochemical sensor was developed for the square wave anodic stripping voltammetric determination of Pb^{2+} at ultra-trace levels. A Glassy carbon electrode was modified with an in-situ electroplated bismuth film and the nanocomposite of a recently synthesized melamine based covalent organic framework (schiff base network$_1$ (SNW_1)) and Fe_3O_4 nanoparticles (Fe_3O_4@SNW_1). The obtained results exhibit clearly that combination of Fe_3O_4@SNW_1 and in-situ electroplated bismuth film enhances the sensitivity of the modified electrode towards Pb^{2+} remarkably. A Plackett-Burman design was implemented for screening experimental factors to specify the significant variables influencing the sensitivity of the electroanalytical method. Afterward, the effective factors were optimized using Box-Behnken design (BBD). Under optimized conditions, the proposed electrode showed a linear response towards Pb^{2+} in the concentration range of 0.003-0.3 µmol L^{-1} with the detection limit of 0.95 nmol L^{-1}. The selectivity of the fabricated electrode towards different ionic species were checked out and no serious interference was observed. At the end, the application of the designed sensor in the determination of Pb^{2+} at 10 different edible specimens were investigated and the obtained recovery values were in the range of (95.56-106.64%) indicating the successful performance of the designed sensor.

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Potentiometric Determination of Hg^{2+} Using a PVC Membrane Coated Graphite Electrode Based on Schiff Base Network$_1$ as a Novel Ionophore

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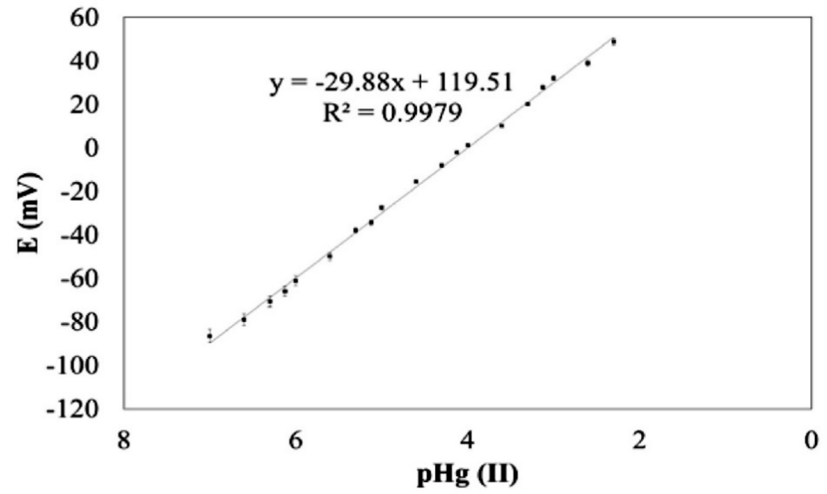
Graphical Abstract



Abstract: In this research, the complexation of a recently synthesized covalent organic framework (schiff base network$_1$) with Hg^{2+} was investigated by IR and FMO computations. The obtained theoretical results showed the complexation process is experimentally feasible, exothermic, spontaneous, and thermodynamically favorable and schiff base network$_1$ can be used as an ionophore for the development of a potentiometric sensor to determination of Hg^{2+}. Therefore, an Hg^{2+} selective PVC membrane coated graphite electrode was developed based on schiff base network$_1$. The best response was observed form the membrane composition of PVC (32%), Nitrobenzene (60%), sodium tetraphenylborate (2%), and schiff base network$_1$ (6%). The designed sensor showed an excellent Nernstian response to Mercury (II) (29.8 mV. Decade^{-1}) over a wide concentration range from 1×10^{-7} to 5×10^{-3} mol L^{-1} and the detection limit of the proposed potentiometric sensor was 80 nmol L^{-1} (16.05 ppb). The effect of different interfering species on the potentiometric response of the suggested electrode was scrutinized by matched potential method (MPM) and no interference was observed. The performance of the developed electrode in partially non-aqueous mediums was also checked out and the obtained results showed the electrode can also be utilized in systems containing %20 organic solvents (ethanol and acetone). The response time and lifespan of the electrode were < 10 S and 16 weeks respectively. In the end, the analytical performance of the fabricated sensor for the determination of Hg (II) in three different industrial effluent samples was scrutinized and the obtained results were compared with a standard spectrophotometric technique.

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Direct Borohydride Fuel Cell Without MEA: A New Electrocatalyst of Mix Metal Oxide as a Cathode and Ni-Co/rGO/NF as an Anode

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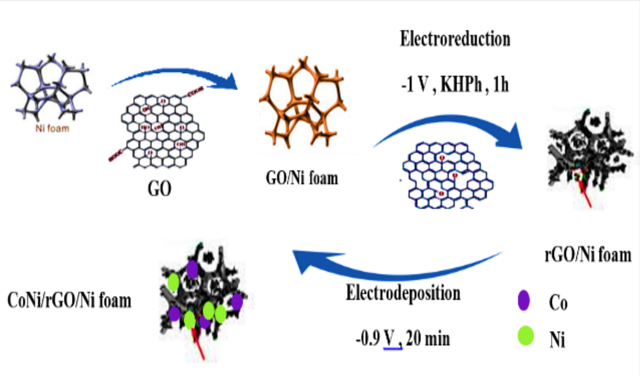
Graphical Abstract



Abstract: Direct Fuel cells (DFCs) are well-known as a clean and efficient energy production technology, and are an interest category of renewable and sustainable energy conversion devices that combine the advantages of combustion engines and batteries, while eliminating their major drawbacks. $NaBH_4$ has benefits such as high theoretical specific capacity (5.7 Ah g^{-1}), easy transportation, and high theoretical open-cell voltage (OCV) (1.64 V), high hydrogen content (weight content of 10.6%) and massively was produced from trimethyl borate and borax [1]. One of the major challenges in liquid fuel cells is the provision of Membrane Electrode Assembly (MEA) electrocatalysts. In the present study, an attempt is made to make a fuel cell with liquid borohydride fuel without immobilizing catalytic inks on the Nafion. Bimetallic Ni-Co nanoparticles on rGO/NF are fabricated by electrodeposition to be used as anode electrocatalysts for direct borohydride-hydrogen peroxide fuel cells (DBHPFCs) [2]. The physical characterization of synthesized material is investigated using Fourier transform infrared spectroscopy, scanning electron microscopy, energy-dispersive X-ray spectroscopy, and X-ray diffraction. The electrochemical half-cell tests are used to study their electrocatalytic properties toward borohydride oxidation in an alkaline solution. A single fuel cell of direct borohydride–hydrogen peroxide is assembled using mixture of metal oxides (MMO) as a cathode and Ni-Co/ rGO/NF as an anode. The cell performance was studied at different operation conditions. Maximum power density value of 336.53 mW cm^{-2} at 55 °C was obtained for Ni_{50}-Co_{50} /rGO/NF. The power efficiency of DBHPFCs in this study with commercial liquid fuel cells is comparable to that of catalytic inks immobilized on Nafion; instead, the cathode and anode preparation methods are so convenient and accessible that a Nafion without any preparation can be used multiple times. The construction of this type of cells without the use of catalytic inks on Nafion has been done for the first time by the electrochemical research team of energy production and storage systems of the Faculty of Chemistry, University of Tabriz.

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Ultrasensitive Electrochemical Aptasensor for Selective Label-Free Detection of *Acinetobcter Baumannii* Bacteria

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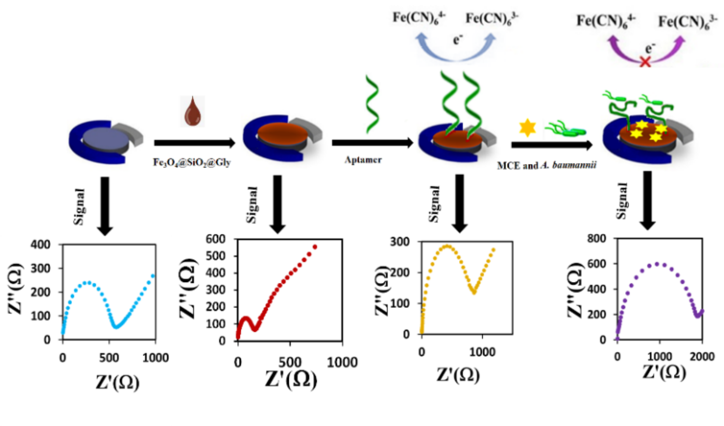
Graphical Abstract



Abstract: *Acinetobcter baumannii* (*A. baumannii*) is responsible for various nosocomial infections, which is known as clinically important opportunistic pathogen. Therefore, rapid detection of this pathogen is critical to prevent the spread of infection and appropriate treatment [1]. Various methods have been developed for the detection of *A. baumannii*, such as Antibody-based assays, bacterial culture, nucleic acid-based assays, biochemical assays, and surface-enhanced Raman scattering [2]. Most of them are accurate but are accompanied by several problems in on-site detection, such as procedures being complicated and cumbersome, time is long, sensitivity is limited, highly sophisticated instruments and expensive, skilled manpower and the cost is high [2]. Therefore, to combat the aforementioned challenges, there is an urgent need for new powerful tools that are rapid and sensitive with the ability to detect *A. baumannii* on-site and that can guide the concerned person for taking appropriate control measures. Among various detection techniques, electrochemical methods have been recognized as one of the most promising technologies due to their high sensitivity, fabrication simplicity, accuracy, cost-effectiveness, selection, the possibility of mass production and ease of modification [3]. In the past few years, aptasensors were used for detecting the type of some bacteria [4]. In this work, an electrochemical aptasensor was developed for the ultrasensitive detection of *A. baumannii* by electrochemical impedance spectroscopy (EIS). The aptamer was linked to the surface of a carbon screen-printed electrode (CSPE) modified with the nanocomposite $Fe_3O_4@SiO_2@Gly$ for selective and label-free detection of *A. baumannii* by covalent bonding. The results of EIS studies showed that the constructed electrochemical aptasensor can specifically detect *A. baumannii* in the concentration range of from 1.0×10^3–1.0×10^8 CFU mL^{-1} and with a detection limit of 150 CFU mL^{-1} (S/N=3). In addition to its sensitivity, the biosensor exhibits high selectivity over other pathogens. Therefore, a simple, inexpensive, rapid, label-free, excellent, selective, and sensitive electrochemical aptasensor was developed for the detection of *A. baumannii*.

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Preparation and Application of Ni-Co LDH/$ZnCu_2O_4$ nanoparticles as a High-Performance Electrode Material for Supercapacitors

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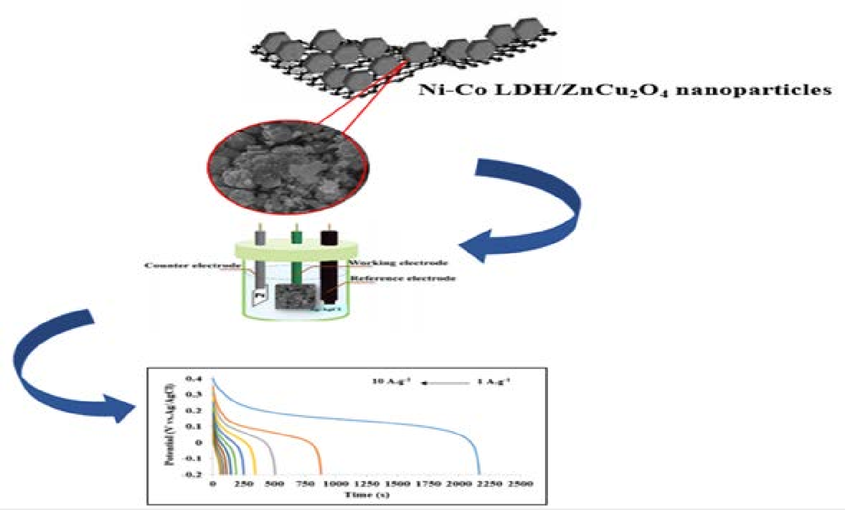
Graphical Abstract



Abstract: Supercapacitors (SCs) as a type of prospective energy storage systems, have been aroused extensive attentions, due to their rapid charge/ discharge process, high power density, long cycling lifetime, wide operation voltage and low maintenance cost [1]. In this work, a nanoarray electrode composed of $ZnCu_2O_4$ nanoparticles and Ni-Co layered double hydroxide (Ni-Co LDH) nanoparticles supported on Ni foam (NF) is successfully constructed by an in-situ growth route. The physicochemical characteristics of the material were investigated by various techniques such as energy dispersive X-ray analysis (XRD) ,field-emission scanning electron microscopy (FESEM), and transmission electron microscopy (TEM). The electrochemical performance of the prepared Ni-Co LDH/$ZnCu_2O_4$/NF electrode as binder-free electrode for supercapacitor was examined by cyclic voltammetry (CV), and galvanostatic charge-discharge analysis (GCD) in 2 mol L^{-1} KOH electrolyte and compared with $ZnCu_2O_4$/NF electrode. The proposed SC illustrated a high gravimetric specific capacitance (3511.77 F g^{-1} at 1 A g^{-1} and a capacity of 1114.51 F g^{-1} at 10 A g^{-1}, good cycle stability (99.84% capability retention after 5000 cycles, together with ~100% coulomb efficiency). This work demonstrates that Ni-Co LDH/$ZnCu_2O_4$ nanoparticles could be a promising electrode for high-performance energy storage devices. The increased pseudocapacitive behaviors are related to its unique core/shell structure which provides facilitated ion diffusion, rapid electron transport kinetics, and good strain accommodation that makes it a potential choice for electrochemical energy storage owing to the their considerable electrochemical properties [2].

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Spherical Molecularly Imprinted Polymers Using Spherical Magnetic Nanomaterials via Electo-polymerization for Sensing of Levodopa

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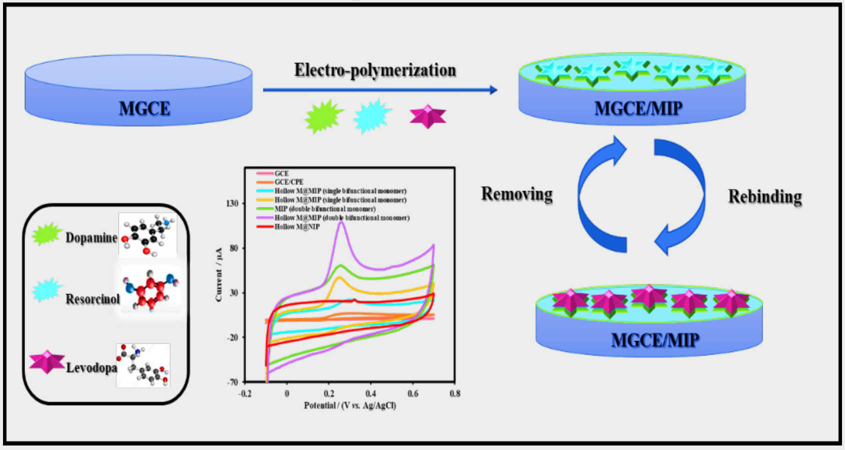
Graphical Abstract



Abstract: Levodopa (L-dopa) is an amino acid precursor of dopamine that is often prescribed to patients suffering from Parkinson’s disease. Levodopa usage comes with various side effects, including nausea, insomnia, headaches, and elevated heart rate, and is dangerous in high doses [1].

Molecularly imprinted polymer (MIP) is a kind of synthetic material with artificially generated recognition sites which can selectively interact with a target molecule to form closely-related compounds. Recently, MIP due to prominent selectivity and strong anti-interference ability has been widely used as molecular recognition material in various fields, such as solid phase extraction, drug delivery and biosensors [2]. In particular, both selectivity and adsorption capacity of MIPs can be elevated to certain extent with carefully selected dual monomers, as the result of increase in polymer-template interaction categories and diversity of functional groups. In addition to the hydrogen bond between the carboxyl group and the pyridyl group, the difunctional monomer can interact with the template polymer through π-π. These monomers can effectively reflect the precise generation of templates and structures. It reminds us that employment of dual functional monomers could increase diversity of the types of blotting sites in MIP architecture and therefore enhance affinity by synergistic effects of different monomers. As a result, the obtained dual-monomer MIP can effectively enrich targets and eliminate interference, thus improving selectivity of MIP to imprinted molecules [3].This work describes a hybrid electrochemical sensor for highly sensitive detection of L-dopa. Firstly, magnetic spheres were produced by solvothermal method. Then, a spherical molecularly imprinted polymer (SMIP) was in situ fabricated on magnetic glassy carbon electrode by electro-polymerization, with dopamine and resorcinol as dual functional monomers and L-dopa as template. Morphological features and electrochemical properties of SMIP were investigated in detail. Compared with typical one-monomer involving MIP, the MIP prepared with dual functional monomers showed higher response and better selectivity. Under the optimal conditions, a calibration curve of current versus concentration of L-dopa was obtained (0.5–1000 µM with low detection limit of 0.05 µM). Determination of L-dopa in real samples was conducted quickly and accurately with the proposed sensor. The SMIP electrochemical sensor proposed in this work has great potential for drug residue determination in biological fluids.

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Design of mixed metal sulfide modified TiO_2-NTAs nanocomposite as photoelectrochemical sensor

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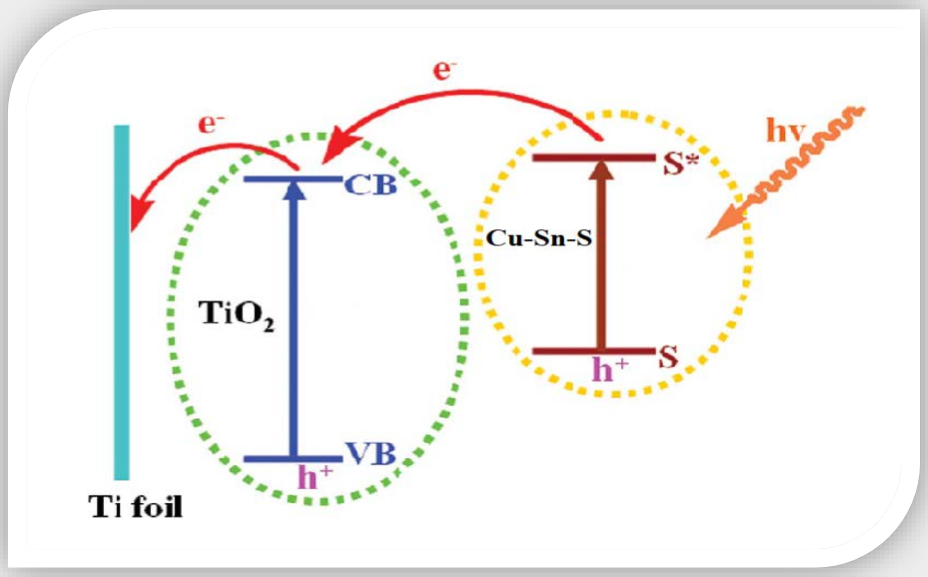
Graphical Abstract



Abstract: Over the past decades, nanoscale TiO_2 has been widely investigated for photoelectrochemical sensor, photocatalysis and dye-sensitized solar cell applications due to its low cost, nontoxic, photo-stable properties and strong photo-oxidation ability. Recently, highly ordered TiO_2 nanotube arrays (NTAs) fabricated by anodization method have attracted much attention for their chemical and mechanical stability, large specific area and good charge-transfer properties [1]. Ti-NTAs were grown on titanium foils by electrochemical anodization in water-ethylene glycol solution containing ammonium fluoride. The anodization was conducted in the constant 25 V for 2 h, and then the as-prepared Ti-NTAs electrode was calcinated at 450 °C for 2h [2]. Cu-Sn-S alloy was deposited on the TiO_2-NTAs by electrodeposition for 90 s under an applied potential of –1.1 V. The results showed remarkable enhanced UV light photoelectrochemical response of the Cu-Sn-S modified TiO_2-NTAs in comparison with TiO_2-NTAs in buffer solution. The photoelectrochemical behaviour of rutile (under the UV irradiation) was studied in the present work using the hydrodynamic photoamperometric method. The optimum conditions for rutile determination were studied and citrate buffer solution (pH=5.5) and E=0.0 V (*vs.* Ag/AgCl reference electrode) were selected as an optimum supporting electrolyte and applied bias potential. The results showed that in the presence of rutile, the photocurrent response of Cu-Sn-S modified TiO_2-NTAs electrode was reduced due to the interaction of rutile with Cu-Sn-S modified TiO_2-NTAs electrode which might impede the electron transfer and accelerate the recombination of photo-induced charge carriers, thus decreasing the photocurrent.

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Uniform Growth of Mn-Sn-Co Ternary Oxide Nanostructure as a High-Performance Electrode Material for Electrochemical Capacitor Applications

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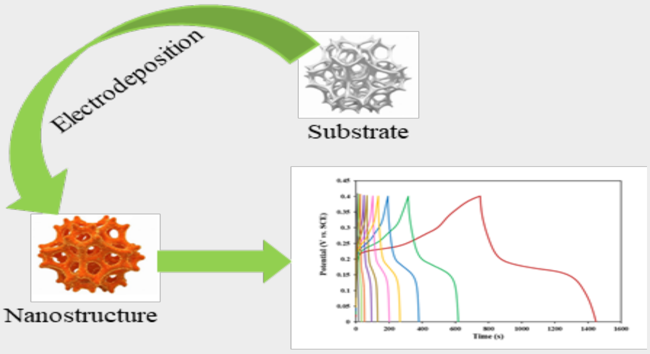

Abstract: In recent years, population growth and economic-social development have increased dramatically the energy consumption, promoting the depletion of non-renewable resources while pushing environmental pollution to alarming levels [1]. Thus, the electrochemical energy storage devices such as fuel cells, batteries, and supercapacitors play a vital role in fabrication of renewable, clean, more abundant and environmentally friendly energy systems. Amongst the electrical energy storage devices, supercapacitors have attracted significant attention due to their fast charge-discharge ability, excellent rate capability, high power density, and long life stability. These outstanding properties of existing supercapacitors put them somewhere between batteries and regular capacitors [2,3]. In this work, we have developed a facile strategy to prepare Mn-Sn-Co oxide ultrathin nanostructure on Ni foam substrate through a fast electrochemical approach. The physicochemical characteristics of the material were investigated by various techniques such as energy dispersive X-ray analysis, field-emission scanning electron microscopy, and transmission electron microscopy. The prepared nanostructure was examined using various analytical methods and electrochemical activities such as cyclic voltammetry, galvanostatic charge and discharge analysis, electrochemical impedance spectroscopy, and cycle life measurements in 3 M KOH electrolyte. Impressively, the as-prepared Mn-Sn-Co oxide nanostructure delivers a high specific capacity (1730 F g^{-1} at 1 A g^{-1}), good rate capability, and excellent cyclic stability with 92.9% capacitance retention after 3000 cycles, suggesting the great application potential of this material in renewable energy storage.

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Preparation, Identification and Performance of CoS_2/PPy Nanocomposite in Supercapacitors

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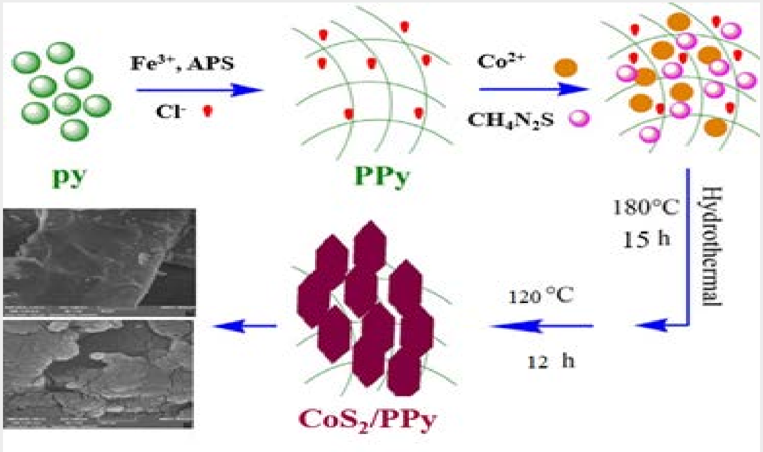
Graphical Abstract



Abstract: Today, one of the most important concerns of energy researchers is to hybridize the sources of electrical energy production with sources of electrical energy storage and select the type of sources in the desired applications with a view to technical and economic categories [1,2]. In recent years, the use of supercapacitors has been seriously discussed due to the main advantages such as longer life, faster charge and discharge time and much higher power density, very high efficiency, etc. [3]. This study uses a single-step hydrothermal method to synthesize polypyrrole/cobalt disulfide (CoS_2/PPy) nanocomposites on a Ni foam substrate, then the electrochemical behavior of the electrode is tested for use in electrochemical supercapacitors. Ni foam has the advantages of high electrical conductivity and surface area (due to its three-dimensional structure), low weight and so on. Also in order to compare the efficiency and capacity of the synthesized supercapacitor; CoS_2 and polypyrrole were synthesized separately on the Ni foam. Field emission scanning electron microscopy (FESEM) was used for the morphology and structural study of the synthesized electrodes. Also, X-ray diffraction pattern (XRD), energy dispersive X-ray (EDX), and elemental mapping, were used to identify the composition and phase of the produced nanostructures. The electrochemical efficiency of the modified electrodes was investigated in 2 M KOH as the electrolyte, by several electrochemical methods such as cyclic voltammetry (CV), galvanostatic charge-discharge (GCD) and electrochemical impedance spectroscopy (EIS). CoS_2/PPy/Ni Foam electrode prepared in this study, showed high specific capacitance (1441 F g^{-1} at a current density of 1 A g^{-1}) and excellent cycle stability (94.97% capacity stability after 3000 cycles). Also, the maximum energy and power density of this supercapacitor electrode was 35.32 Wh kg^{-1} and 6.30 kW kg^{-1}, respectively.

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Electrochemical oxidation of acetaminophen in the presence of Amino acids

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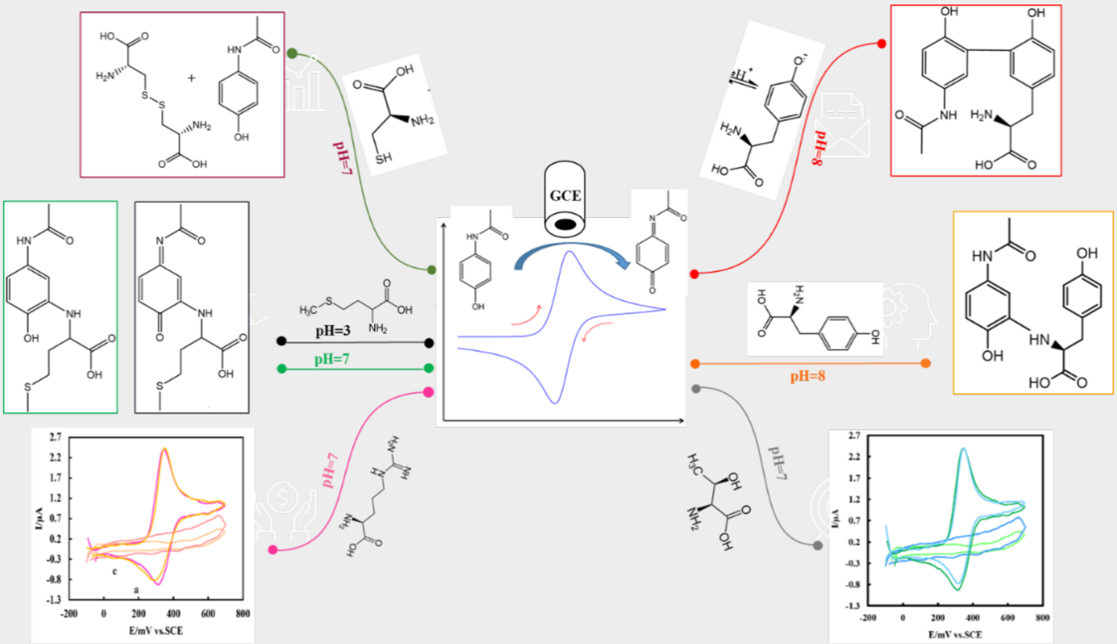
Graphical Abstract



Abstract: The in vitro studies of the electrochemical oxidation of drugs in aqueous medium are important. In fact, the results provide the criterion for investigating of drugs behavior in the body biological systems [1]. The present work aims to report, the effect of amino acids on the electrochemical oxidation of acetaminophen by using cyclic voltammetry and coulometry techniques. The results show that N-acetyl-P-benzoquinone (NAPQI) induced by acetaminophen oxidation, [2] reacts with amino acids (i.e. L-Cysteine, L-Methionine, L-Tyrosine, L-Serine, L-Arginine, L-Threonine, D-Galactose). NAPQI participate in via EC´ reaction mechanism with L-Cysteine, ECE and EC reaction mechanism with L-Methionin and EC reaction mechanism with L-Tyrosine. The final product was synthesized using coulometry in aqueous solution through a catalyst-free environmentally friendly method at room temperature under green conditions. The product was then separated and purified. It was characterized by NMR, FT-IR and MS techniques. Electrochemical oxidation of acetaminophen was performed in the presence of different concentrations of amino acids at different pHs using cyclic voltammetry (CV) [3]. The voltammetric results show that the ratio of cathode peak current to anodic peak for acetaminophen in the presence of the amino acids L-cysteine, oxidized L-tyrosine, L-arginine, L-threonine, L-methionine and not oxidized L-tyrosine had the most reductions; that indicate the reactivity of these amino acids with acetaminophen. Therefore, the studied amino acids can be used as an antidote in overdoses of acetaminophen.

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Electrocatalytic Oxidation of Methanol at the Surface of Carbon Paste Electrode Modified with Graphene Oxide and Cu-Al Layered Double Hydroxide Composite

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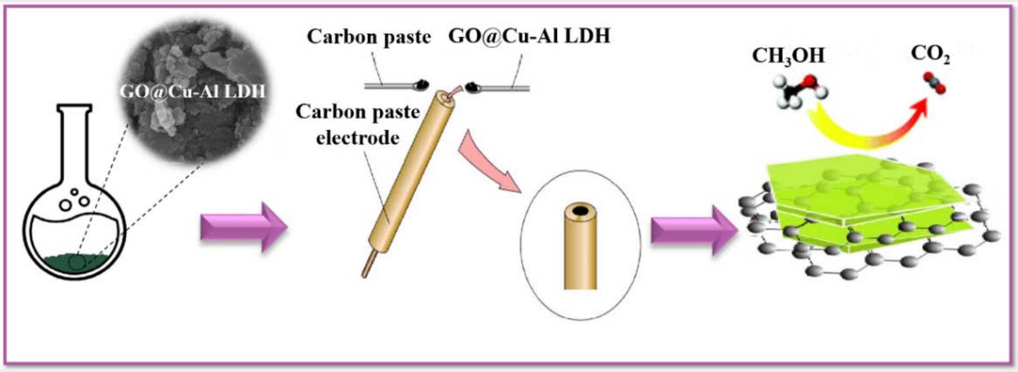
Graphical Abstract



Abstract: In recent years, excessive consumption of non-renewable energy sources caused human being concern. Fuel cells technology is recognized as one of the best alternatives for future energy sources. In this direction, a direct methanol fuel cell (DMFC) is one of these systems that is a suitable choice to solve the challenges of energy crisis and environmental issues. One of the challenges in this field is the preparation of efficient electrocatalysts for the electrooxidation of methanol as a fuel [1-3]. In this work, GO@Cu-Al LDH composite was prepared and characterized by using XRD, FT-IR, FE-SEM, EDS and BET techniques. Therefore, GO@Cu-Al LDH composite modified carbon paste electrode was used as anode for studying the electrooxidation of methanol in alkaline media. The electrocatalytic performance of the modified electrode was also evaluated for methanol electrooxidation in alkaline media by using various electrochemical methods such as cyclic voltammetry, chronoamperometry, and electrochemical impedance spectroscopy. Comparing the activity of prepared GO@Cu-Al LDH and Cu-Al LDH electrocatalysts modified carbon paste electrodes have shown respectively the current densities of 2.1 mA/cm^2 and 0.6 mA/cm^2, in 0.5 M methanol and 0.1 M NaOH solution by using CV method with a scan rate of 50 mV s^{-1}. The results proved that GO@Cu-Al LDH composite with suitable properties related to peresent of both GO and LDH, has higher current density and more stability than Cu-Al LDH. The GO@Cu-Al LDH electrocatalyst is considered cost-effective due to its simple method of synthesis and lack of precious and noble metals [4-6].

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Polydopamine imprinted polymer-based tunable electrochemical synthesis of Cu-BTC MOF film as a hybrid dual recognition element for ultra-trace sensing of Pregabalin (Lyrica)

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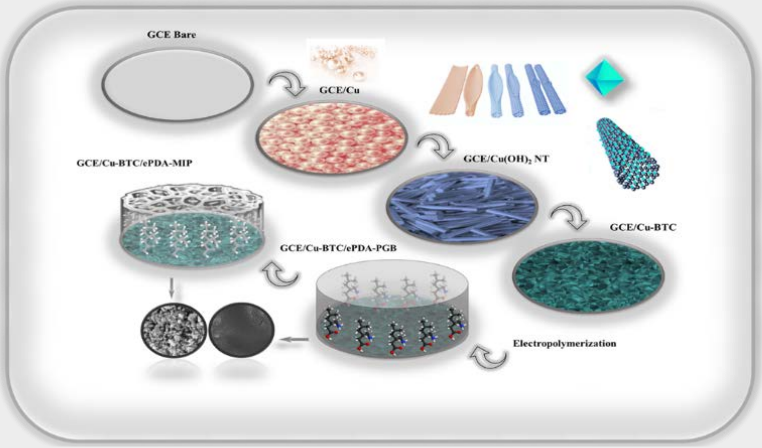
Graphical Abstract



Abstract: The present study propounds an innovative diagnostic hybrid dual recognition strategy for detecting trace amounts of Pregabalin (PGB) using the combination of two powerful metal-organic framework (MOF) structures and the molecularly imprinted polymer (MIP) method [1]. To improve the electrode analytical performance, copper-benzene-1,3,5-tricarboxylic acid frameworks (Cu-BTC MOFs) were synthesized directly on the electrode through three stages by electrodeposition of Cu nanoparticles on GCE and direct chemical conversion of them to copper hydroxide nanotubes ($Cu(OH)_2$ NTs) intermediate and then swiftly chemically convert NTs to Cu-BTC MOF layer[2]. Then MIP film was synthesized via the in-situ dopamine (DA) electropolymerization [3]. Surface morphology was characterized by field emission scanning electron microscopy (FESEM) images at all stages. To explore the effect of independent variables on the MIP construction, the Box-Behnken design was considered to determine response surface methodology[4]. In this sense, the predicted optimum combination variables were DA=3 mM, pH = 7.4, cycle number = 16, KOH 4 mM, KOH: Et-OH ratio of 5:1 and 18 min elution time. Incubation time was obtained at 12 min. Electrochemical impedance spectroscopy (EIS) was used to determine the PGB analyte in experimental stages. In addition, in optimal research conditions, analytical parameters such as linear range, accuracy and precision, were explored. The linear dynamic range and LOD were 0.05- 800 pM and 2.9 fM respectively. The imprinting calculated factor was 24.60, showed good interaction between the PGB and DA monomer. Inter-day and intra-day reproducibility measurements showed 2.08% and 1.58% and, 98% of the initial current response was maintained. Coefficients of selectivity against the PGB, for tapentadol, gabapentin, levetiracetam, L-Carnitine, and L-Histidine were 4.20, 0.76, 0.48, 1.50, 0.64 and 0.64. CV scanning at various scan rates verified that adsorption kinetics follows a diffusion control mechanism. Finally, the GCE/Cu-BTC/ePDA-MIP sensor's efficiency in urine and human serum samples was evaluated, and satisfactory results were achieved.

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Synthesis and Application of MOFs Derived Nanoporous Carbon as an Electrocatalyst in Oxygen Evolution Reaction

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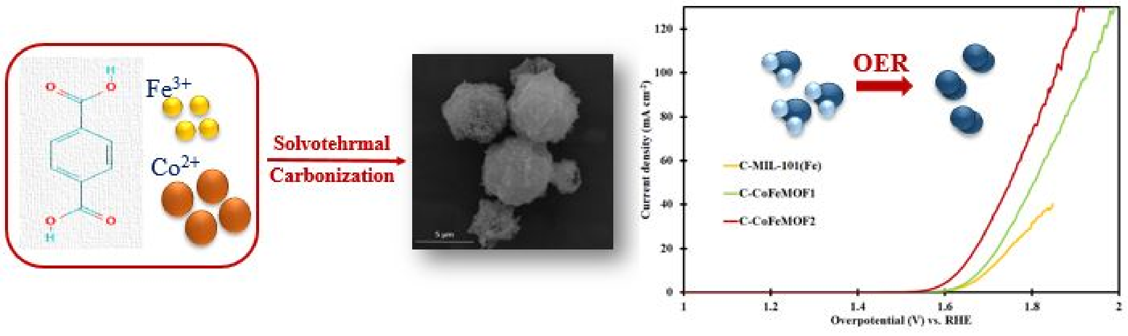
Graphical Abstract



Abstract: To meet the world's rising energy demand and environmental problems, developing sustainable clean energy is unavoidable [1]. Hydrogen is being more widely used. Splitting water into hydrogen and oxygen through an electrochemical process is accepted especially, the anodic reaction of oxygen evolution reaction (OER). This reaction is facing problems due to the needed high energy and slow kinetic. So the overpotential of the OER can be reduced by efficient electrocatalysts [2]. Metal-organic frameworks (MOFs) can be good choices as an electrocatalyst [3]. In this study, C-MIL-101(Fe), C-CoFeMOF1, and C-CoFeMOF2 were synthesized by solvothermal method and then carbonized. MOFs were characterized by Scanning Electron Microscopy (SEM). Also, electrocatalytic performance of these materials was investigated for OER in 1.0M KOH. The prepared C-CoFeMOF2 has shown excellent electrocatalytic activity toward OER in the alkaline condition. The SEM image of C-CoFeMOF2 showed that the C-CoFeMOF2 appeared as a spherical morphology. Also, the OER performance of materials was evaluated by LSV curve, Tafel plot and EIS. The overpotential of C-MIL-101(Fe), C-CoFeMOF1 and C-CoFeMOF2 at 10 mA cm^{-2} was 470 mV, 450 mV and 390 mV, respectively. The Tafel Slop of C-CoFeMOF2 was equal to 65.2 mV dec^{-1} which was much smaller than C-MIL-101(Fe) (100.2 mV dec^{-1}) and C-CoFeMOF1 (96.4 mV dec^{-1}). The Nyquist plots of C-MIL-101(Fe), C-CoFeMOF1 and C-CoFeMOF2 were prepared also. The R_{ct} amount was calculated, and it was equal to 690.7 Ω, 590.6 Ω and 80.5 Ω for the C-MIL-101(Fe), C-CoFeMOF1 and C-CoFeMOF2, respectively. In summary, materials were synthesized by solvothermal method and then the carbonization process was done. By using LSV polarization curves, Tafel plots, and EIS, the electrocatalytic performance of materials was investigated. The carbonization process improves the OER performance. So, C-CoFeMOF2 was investigated better electrocatalyst than others.

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A Non-enzymatic Textile-based Urea Sensor Using Ag Doped NiO

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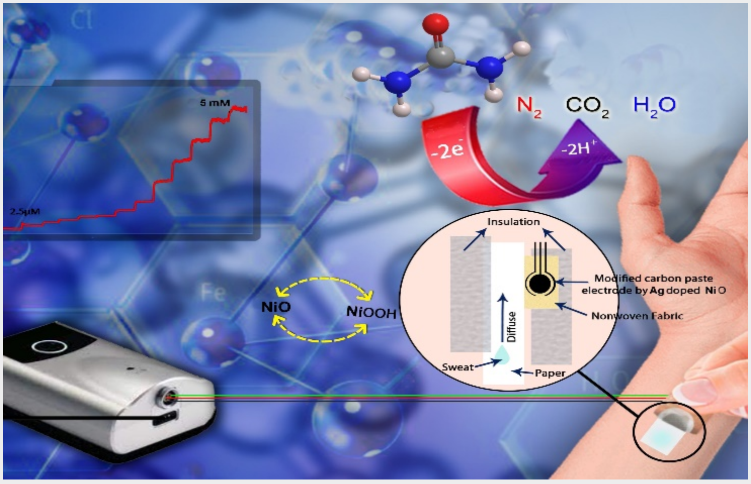
Graphical Abstract



Abstract: Among the various compounds, many metals and their oxides have been used as modifiers for the preparation of non-enzymatic electrochemical sensors. Different fabric bases have been used to make flexible sensors [1]. In the present work, we have designed a modified carbon paste electrode based on a suitable fabric using Ag-doped NiO (Ag@NiO) as a non-enzymatic electrochemical urea sensor. Various techniques for studying the structure of synthesized Ag@NiO include scanning electron microscopy (SEM), energy scattered X-ray spectroscopy (EDX), electrochemical impedance spectroscopy (EIS), diffuse reflectance spectroscopy (DRS), and X-ray diffraction (XRD) were used. Electrochemical impedance spectroscopy and cyclic voltammetric methods have been used to study the electrochemical properties of the prepared electrode. The amperometric method was used to quantitative determination of urea using the prepared electrode. This electrode is very suitable for measuring urea in alkaline solutions. Important features of the electrode are high sensitivity, wide linear range (2.5×10^{-3} to 5.0 mmol L^{-1}), low detection limit ((8×10^{-4} mmol L^{-1}), long-term stability, and very good selectivity. Finally, the prepared electrode was used to determine the urea concentration in human sweat samples.

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Determination of Gallic Acid by Multilayer Titanium Metal Organic Framework/Multi-walled Carbon Nanotubes Modified Electrode

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Abstract: Gallic acid (GA) is an omnipresent compound found in green tea, fruits and vegetables. GA possesses properties such as antioxidant [1], anti-viral, antibacterial, anti-diabetic, anti-cancer, anti-aging, and anti-inflammatory properties [2]. GA shows cytotoxic effects against cancer cells without harming normal cells [3]. Recently, in order to increase the catalytic performance, nanocomposites including MOFs and MWCNTs have been developed [4]. In this work, we have successfully developed a sensor for the detection of GA based on electrodeposition of multilayer films of titanium based metal organic framework and multi-walled carbon nanotubes (Ti-MOF/MWCNTs). Scanning electron microscopy and energy dispersion spectroscopy were used to investigate the surface of the modified electrode. The electrochemical characterization experiments showed that the Ti-MOF/MWCNTs modified glassy carbon electrode had excellent electronic conductivity and high stability. Several parameters such as pH, concentration of modifiers, scan rate and the amount of nanocomposite controlling the performance of the sensor were examined and optimized. Differential pulse voltammetry experiments indicated a linear concentration range of GA from 1.0×10^{-7} M to 1.0×10^{-3} M, the detection limit was estimated to be 3.5×10^{-7} M. The repeatability, reproducibility and stability of the developed sensor were examined and the results showed that the developed sensor was in superior in condition (RSD=1.71%). The proposed electrochemical sensor exhibited high specific electrochemical response to GA and it was used for voltammetric determination of GA in real samples of green tea and black tea.

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Preparation of polyaniline/Vanadium oxide hydrogel for high performance binder-free supercapacitor electrodes

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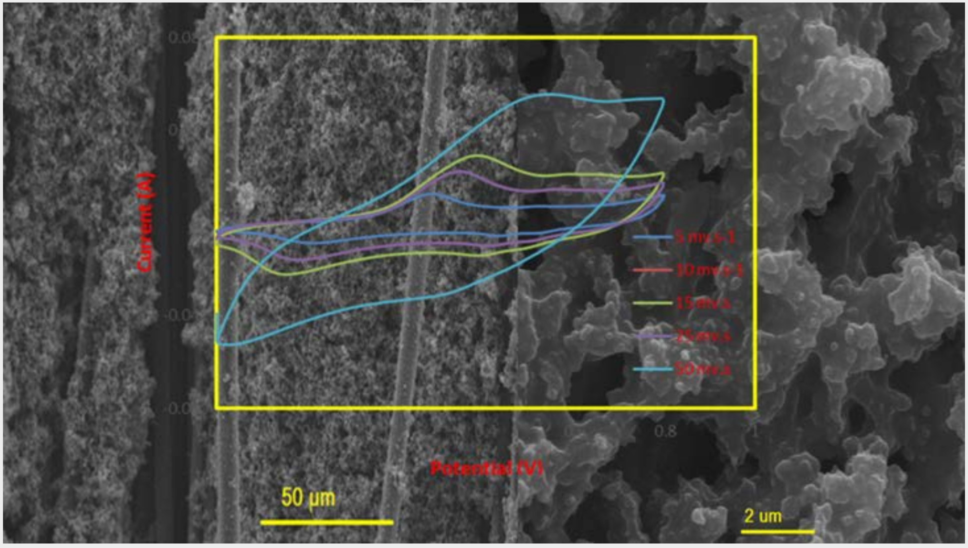
Graphical Abstract



Abstract: In recent years, there has been a tremendous increase in demand for high-performance flexible and portable electronics with great power/energy density. Researchers in energy storage, particularly supercapacitors and batteries, are currently interested in flexible technologies. A ternary nanostructured polyaniline/Vanadium oxide decorated graphene composite hydrogel was developed as a promising electrode material for flexible supercapacitors. In a 1 M H_2SO_4, the ternary composite hydrogel on carbon cloth has a high specific capacitance of 982.8 Fg^{-1} when the current density is 2.7 Ag^{-1}. Because of the well-designed cross-linked hydrogel structure, high surface area, and synergistic effects among all three components, ternary composites hydrogel shows superior electrochemical performance. Next-generation flexible supercapacitors can benefit greatly from this remarkable performance.

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Preparation of A New Electrochemical Sensor for Individual and Simultaneous Determination of Ascorbic acid, Dopamine and Uric acid using Zirconium Oxide-Porous Carbon Nanocomposite Derived from Metal-Organic Frameworks

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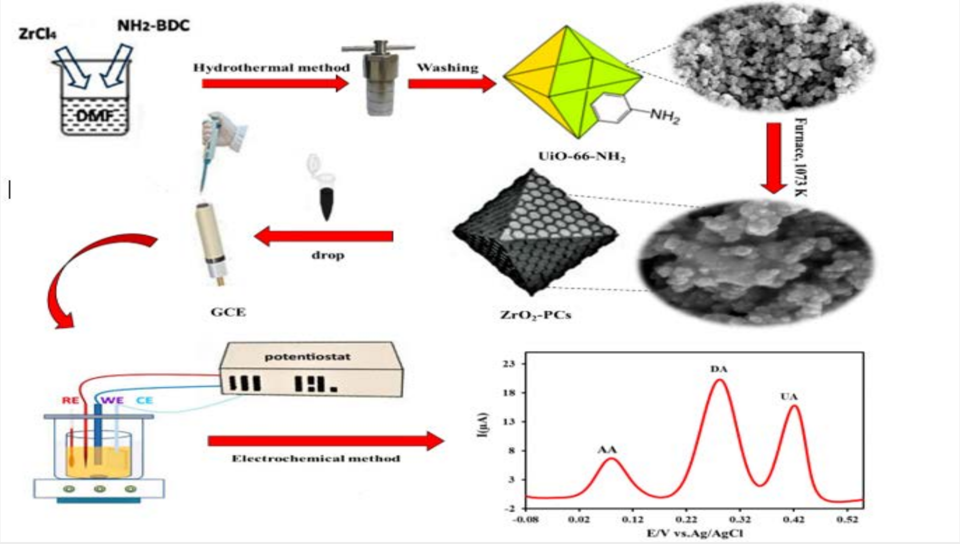
Graphical Abstract



Abstract: Ascorbic acid (AA), dopamine (DA) and uric acid (UA) are among biomolecules that have very important physiological functions in the metabolic system of living organisms. In the present study, a porous carbonaceous platform containing zirconium oxide (ZrO_2-PCs) was synthesized by calcination of a metal-organic framework for the simultaneous determination of AA, DA and UA. The morphology and structure of ZrO_2-PCs was characterized by XRD, FT-IR, SEM, TEM, EDX and nitrogen adsorption/desorption methods. In addition, CV, DPV and EIS were used to investigate the electrochemical properties of the fabricated electrode. Considering the influence of pH on the results, PBS (0.1 M, pH 7.0) was selected for the simultaneous analysis of AA, DA and UA. Three well-separated DPV peaks were observed on ZrO_2-PCs/GCE for the three bio-molecules. At the optimized conditions, wide linear ranges, i.e., 1-5998.08, 0.5-220.87 and 0.5-270.88 µM for AA, DA, and UA in ternary mixture were obtained, respectively. The respective detection limits were 0.33, 0.16 and 0.16 µM. Repeatability and reproducibility of the method were calculated as relative standard deviation (RSD%) with maximum values of 2.0, 2.22, 4.71 and 2.59, 4.48, 1.37 for AA, DA, UA respectively. The method was applied to the simultaneous detection of AA, DA and UA in human serum samples by DPV using standard addition method, successfully. These performance improvements can be attributed to the catalytic effect of ZrO_2 nanoparticles and increase in active surface area of the electrode surface. In addition, defect structures (oxygen vacancies) of ZrO_2 play an important role in improving catalytic activity and can significantly reduce the oxidation potential of analytes [1, 2]. On the other hand, excellent electrical conductivity of porous carbon accelerates electron transfer during electrochemical reactions [2, 3].

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Synthesis of Core-shell nanostructures for high-performance supercapacitor

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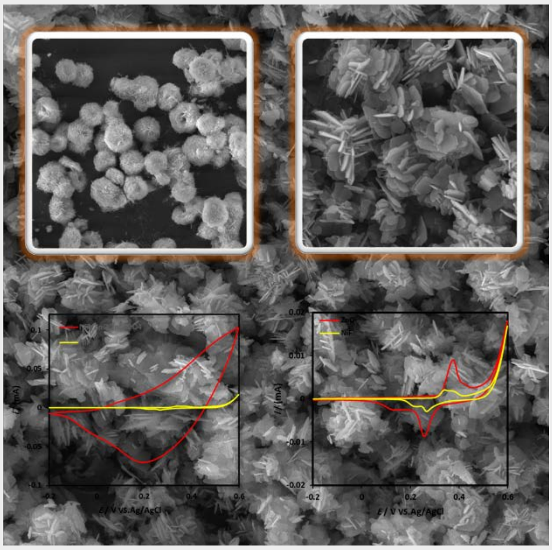
Graphical Abstract



Abstract: Recently, owing to increasing concern about the depletion of fossil fuels and environmental pollution, there is an urgent need for clean, efficient, and sustainable energy resources to maintain a sustained, healthy and rapid economic development. Among the promising electrical energy storage devices, lithium-ion batteries (LIBs) and supercapacitors (SCs) have attracted worldwide attentions. Owing to their fast recharge ability, high power performance, long cycle life, and low maintenance cost, supercapacitors have gained enormous interests swiftly [1]. Recently, nanocomposite materials have drawn considerable attention because they can provide better characteristics than the single materials [2]. Core–shell nanoparticles (CSNs) are a class of nanostructured materials recently have received increased attention because of their exciting properties and broad range of applications in catalysis, biology, materials chemistry and sensors. It is possible to fabricate a range of core–shell nanoparticles with tailorable properties that can play important roles in various catalytic processes and offer sustainable solutions to current energy problems. Traditionally, composite nanomaterials constructed with cores and shells, both at nanoscale, are broadly defined as CSNs (core–shell nanoparticles) [3].In the present work, the core-shell of $ZnO@ZnCo_2O_4$ were directly grown on nickel foam substrate through a facile hydrothermal method. These nanomaterials were analyzed using X-ray diffraction spectroscopy (XRD), scanning electron and transmission electron microscopies (SEM and TEM). Also, $ZnO@ZnCo_2O_4$ electrode was investigated using cyclic voltammetry (CV), galvanostatic charge–discharge (GCD) and electrochemical impedance spectroscopy (EIS) to check the supercapacitive behavior. The $ZnO@ZnCo_2O_4$/NF exhibited a high specific capacitance of 1655.76 F g^{-1} at 1.25 A g^{-1} in 2 M KOH solution. Our findings confirm that $ZnO@ZnCo_2O_4$/NF electrode can be considered as an outstanding candidate for high performance energy storage devices.

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A Pencil Graphite Electrode Modified by Carbon Nano-Onions and Graphene Nanoplate for Trace Determination of Olanzapine

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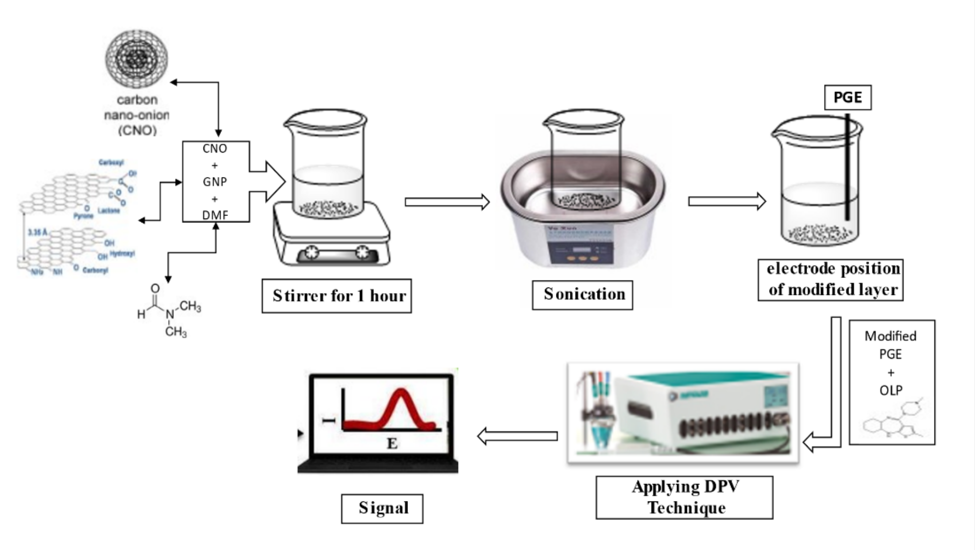
Graphical Abstract



Abstract: Stress is the body's way of responding to any kind of demand or threat which can cause a variety of disorders, especially, when it is followed by danger or fear. Several types of drugs have been used for the therapy of stress. Olanzapine (OLP), 2-methyl-4-(4-methyl-1- 41 piperazinyl)-10H-thieno-[2,3-b] benzodiazepine, is a thienobenzodiazepine derivative which has the most application for the stress treatment. Olanzapine has been widely used as antimigraine and antidepressant. Some side effects such as drowsiness, dizziness, lightheadedness, stomach upset, dry mouth and constipation can be serious through the high prescription of OLP. Thus, the monitoring of OLP concentration in biological fluid for therapeutic drug monitoring purposes is very important. In this study, a modified pencil graphite electrode (PGE) modified with the carbon nano-onions (CNOs) and graphene nanoplate (GNP) was used for the measurement of OLP by differential pulse voltammetry (DPV) technique. The electrochemical studies revealed the modified electrode is capable of the voltametric determination of traces of OLP in the phosphate buffer at a working potential of 0.2 V (*vs.* Ag/AgCl). The surface morphology and electrical properties of the modified electrode were characterized by scanning electron microscopy (SEM) and electrochemical impedance spectroscopy (EIS). To obtain the great efficiency of the sensor some experimental factors such as time, the potential of accumulation and pH value of the electrolyte were optimized. The modified PGE under optimized conditions represented a linear range of 1.0×10^{-10}_ 4.5×10^{-8} M and a detection limit of 5.4×10^{-11} M. These satisfactory results can be attributed to utilizing the CNOs and GNP in the electrode modification process due to some of its admirable characterizations of this nanostructure including high surface area, excellent electrical conductivity and good electrocatalytic activity. Consequently, the developed sensor was applied for the determination of OLP in real samples such as pharmaceutical tablet blood serum samples.

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Synthesis and Application of Bimetallic MOFs as Electrocatalyst in Water Splitting

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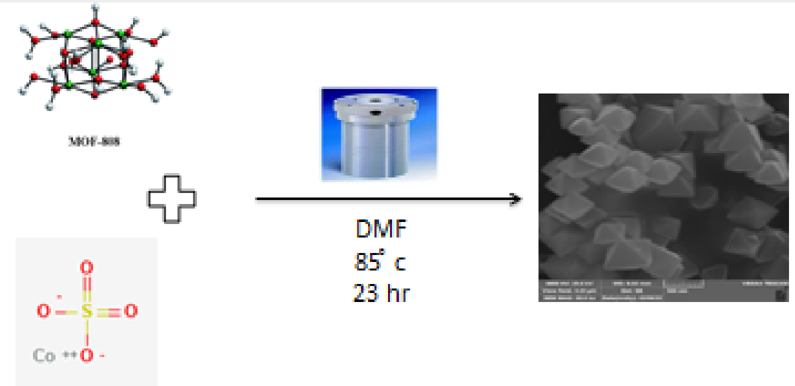
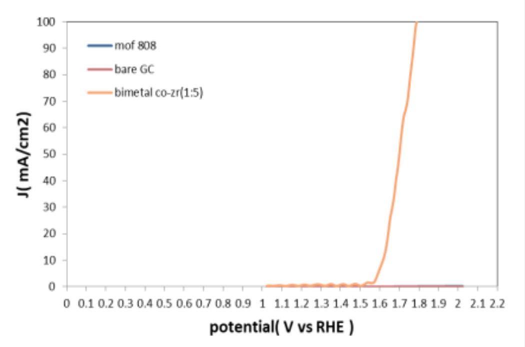
Graphical Abstract





Abstract: Weak electrical conduction impedes the application of mono metallic-organic frameworks (MOFs) in electrochemistry. In this study, Zr-Co based MOFs were prepared via a simple solvothermal method. Hydrogen, regarded as the most promising green energy source,is abundant, recycable with high energy density ideal fuel. Water pyrolyses is a carbon-free method to producing H_2 [1]. Transition based-bimetallic MOFs could contribute to accelerate the sluggish kinetics for obtaining excellent electrocatalytic performance. Here, we synthesised Zr-Co based MOFs via a simple method with good performance in watersplitting. To synthesis of Zr-Co MOF, 25 mg of as-prepared MOF808 [2] and 125 mg $CoSO_4$ were added to 15 ml DMF and stirred for 2 hr. After that the solution was sealed into an autoclave and heated to 85 ̊C for 23 hr. Then, the pink mixture was cooled down to room temperature and centrifuged with DMF and dried in oven. Samples with nafiuon, dropp casted on glassy carbon as working electrode and then put in three-electrode system with Pt wire and Ag/AgCl as counter and refrence electrodes, respectively. In the graphical abstract, the LSV curves of bare GC, MOF 808 and Zr-Co bimetallic MOF in 1M KOH electrolyt have been shown.

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Development of carbon paste sensor for low level and selective determination of ondansetron in pharmaceutical and biological samples

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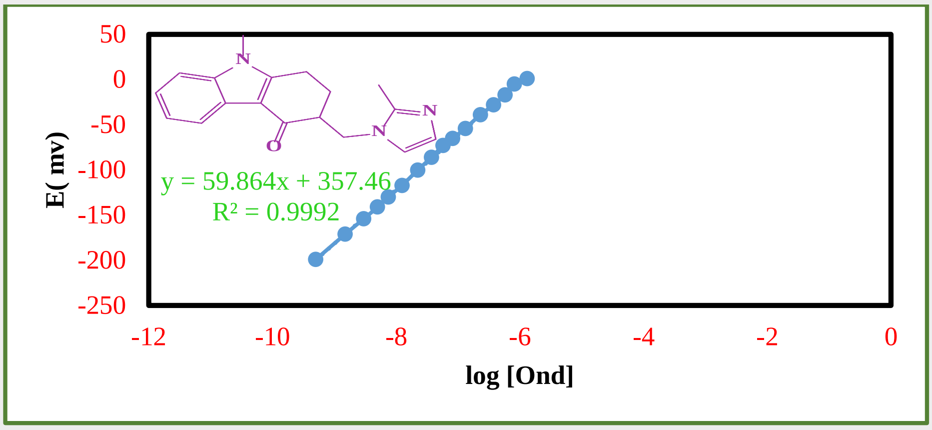
Graphical Abstract



Abstract: Ondansetron (or Zofran, or Demitron), (RS)-9-methyl-3-[(2-methyl-1H-imidazol-1-yl) methyl] 2,3-dihydro-1H-carbazol-4(9H)-one, is a highly selective and potent 5-hydroxytryptamine type 3 (5-HT3) receptor antagonist. It is effective in the treatment of nausea and vomiting during cancer chemotherapy and radiotherapy, and has reported anxiolytic and neuroleptic properties. 5HT released by chemotherapy, radiotherapy and surgery also activates the 5HT3 receptors that are found in the CTZ in the brain, causing further messages to be sent to the vomiting center. Ondansetron works by blocking the 5HT3 receptors that are found in the brain and gut [1]. A few methods have been used for quantitative determination of ondansetron including HPLC [2], voltammetry [3], spectrophotometry [4] and potentiometry [5]. The reported potentiometric sensor was the liquid membrane electrode, while, no carbon paste potentiometric sensor for this drug has been reported so far. In this study, performance characteristics of a new potentiometric carbon paste electrode (CPE) for the fast, easy and selective determination of ondansetron was described. The constructed potentiometric sensor was based on the use of the 2-hydroxypropyl-β-cyclodextrin as a good ionophore in the carbon paste matrix. The resulted modified CPE exhibited Nernstian slope of 59.8 mV/decade over a wide concentration range of 1.5×10^{-9} – 1.3×10^{-6} M with low detection limit of 8.4×10^{-9} M. The potentiometric response of the electrode was studied in buffered solution. The proposed sensor manifested advantages of high stability, fast response, long life time and, most importantly, good selectivities for ondansetron relative to a wide variety of common foreign inorganic and biological species. The influence of temperature on the potential response of the sensor was tested at different temperature and the temperature coefficient of the sensor was calculated. The sensor was successfully applied as an indicator electrode in potentiometric determination of ondansetron in ondansetron tablet and blood serum samples.

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Determination of Trifluralin Using Adsorptive Differential Pulse Voltammetric Method

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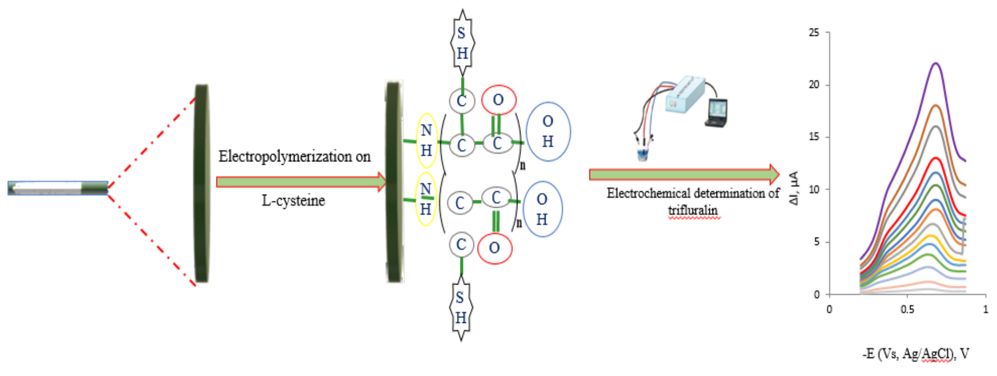
Graphical Abstract



Abstract: Herbicides are classified as carbamates, amides, diphenylethers, amino phosphates, and dinitroanilines. Trifluralin (2,6-dinitro-N,N-dipropyl-4-trifluoromethyl-amine) is a benzene derivative belonging to the dinitroaniline family [1]. This toxin increases abortion, kidney and liver damage, allergies and decreases T lymphocyte count. Trifluralin can also affect the function of the endocrine glands. In addition, due to its low solubility in water, it is one of the soil contaminants and causes several changes in the soil. In some reports, trifluralin has also caused air pollution, so it can be said that the identification of trifluralin is important [2]. Trifluralin is detected mostly by chromatographic technology such as gas chromatography (GC) [3]. The electrochemical techniques, which are often simple and less expensive, have been also used to determine trifluralin [4]. In this project, an electrochemical method is used to measure this herbiside. To prepare the modified electrode, the pencil graphite electrode (PGE) was firstly placed into a solution containing 2.4 mM L-cysteine and phosphate buffer solution (pH=7) and then poly L-cysteine/pencil graphite electrode (PLC/PGE) was fabricated using cyclic voltammetric technique with applying of 3 cycles over the potential range -0.4 to 1.6 V. After electrode preparation, the reduction current of trifluralin was measured in the presence of sodium dodecyl sulfate (SDS) with applying adsorptive potential of -0.2 V during 200 sec. In order to achieve the best voltammetric response the electrode components and the other effective parameters were optimized and then trifluralin was determined using adsorptive differential puls voltammetric method. The linear dynamic range was investigated on the determination of trifluralin obtained as 0.25-100 µM. Also, the effect of some cations, anions and environmental pollutions was investigated. Finally, this method was successfully used for the determination of trifluralin in some real samples such as soil, water, tomatoes and oranges.

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Coated wire thiopental ion-selective electrode based on imprinted polypyrrole as recognition element

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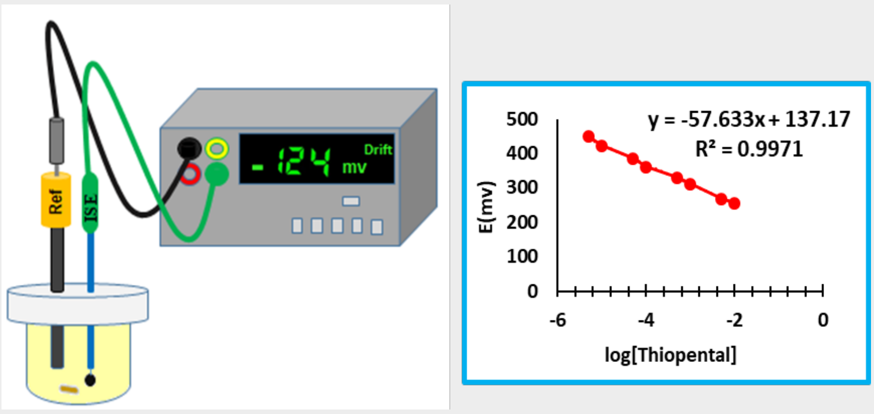
Graphical Abstract



Abstract: Thiopental is a drug widely used as an intravenous agent for the rapid induction of general anesthesia. [1] It is also used as a supplement to other anesthetics and to induce sleep during anesthesia in balance with other medications. Thiopental drugs can also be deadly, and taking this drug in high doses can cause euphoria, rape, and even suicide. It is important to diagnose brain death before organ donation, so it is necessary to measure this drug before it leaves the body's biological fluids [2, 3]. In this work, In order to detect thiopental anion in an aqueous solution, a coated wire solid-state ion-selective electrode (ISE) based on doped polypyrrole with thiopental anion was prepared by electrochemical methods. The electrochemical polymerization was carried out with a three electrodes electrochemical system by using a potentiostat/ galvanostat instrument. A spherical head (1.0 mm diameter) platinum electrode was used as working electrode. The polymeric film as a recognition element was prepared with different electrochemical conditions in the presence of thiopental anion. Then, the response behavior of fabricated sensors for detection of thiopental anion with different concentrations (10^{-7} – 10^{-2} M) was tested at various pH levels in aqueous solution. According to the calibration curve, the Nernstian slope of about -57 mV/decade and correlation coefficient (R^2) of 0.99 were obtained in optimum conditions. Based on the results, the fabricated solid-state ISE based on doped polypyrrole with thiopental anion is a promising candidate for detection and measurement of thiopental anion in pharmaceutical preparation and water samples.

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Electrochemical Study of Phenylhydrazine and its Application in the Electrosynthesis of New Phenylhydrazine Derivatives

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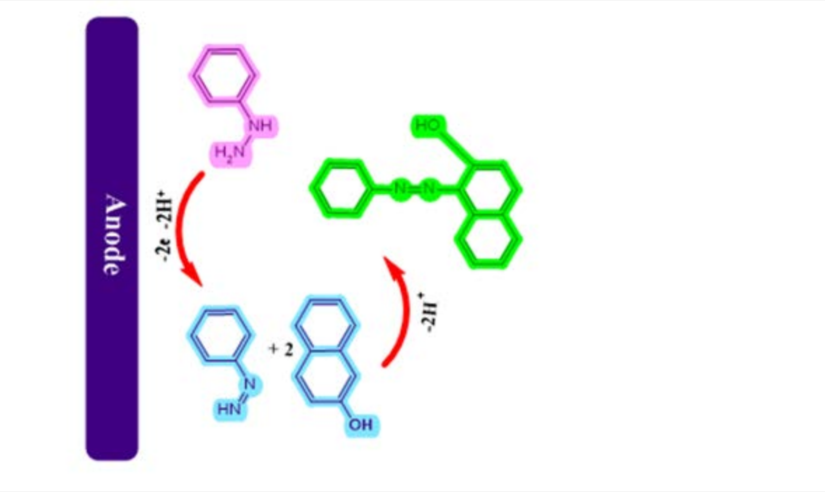
Graphical Abstract



Abstract: Hydrazine (HZ) and its derivatives are widely used in agricultural chemicals (pesticides), chemical blowing agents, pharmaceutical intermediates, photography chemicals, and boiler water treatment in hot-water heating systems for corrosion control [1]. Moreover, HZ salts and its derivatives are used as rocket fuel, gas generators, and explosives [2]. Phenylhydrazine is the first hydrazine derivative to be used to prepare indoles by the synthesis of indole fisher, which is used as an intermediate in the synthesis of various dyes and drugs. This molecule is also used to induce acute hemolytic anemia in animal models. In this work, the electrochemical oxidation of phenylhydrazine in the presence of ß-Naphthol as a nucleophile has been investigated. Based on the results which obtained from the voltammetric data, the electrolysis was performed under the constant current method in order in an aqueous buffered solution (pH = 8.0, C = 0.2 M H_3PO_4) to synthesis of new product. Product formation was confirmed by using IR, MS and NMR spectra.

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Application of Carbon Quantum Dots in Inhibiting Copper Corrosion: The Effect of Different Layer Deposition Techniques

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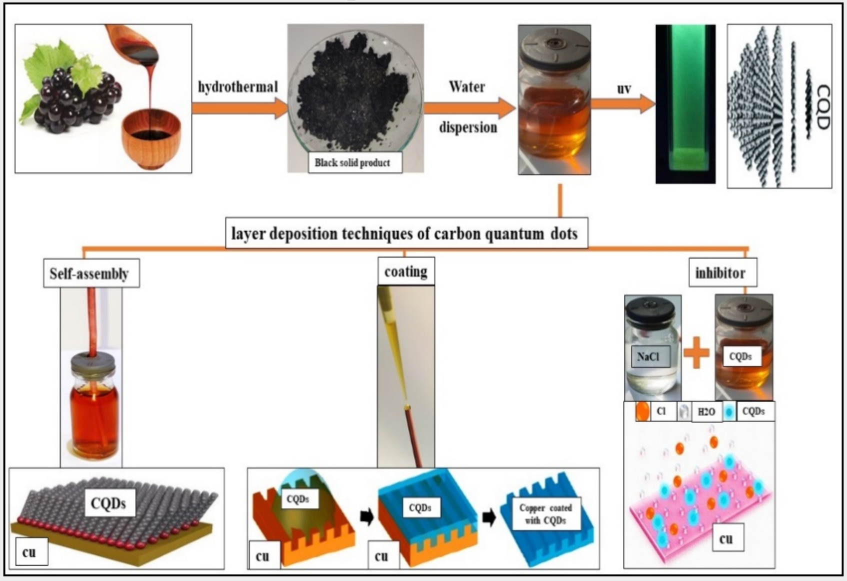

Abstract: Corrosion is an attack on metallic material by reaction with its environment [1]. Corrosion is a spontaneous phenomenon which causes huge damage to industries worldwide [2]. According to the reports, the current global cost of corrosion is estimated to be US$2.5 trillion, which is equivalent to 3.4% of the global GDP (gross domestic product) (2013). By using available practical corrosion control, it is estimated that savings of between 15% and 35% of the cost of corrosion could be realized, between US$375 and $875 billion annually on a global basis [3]. The use of inhibitors is one of the best methods of corrosion control, which is discussed in this article, due to its advantages such as cost-effectiveness and ease of use in industry. Most corrosion inhibitors are expensive and environmentally hazardous chemicals, so the research on environmentally friendly inhibitors is a new field of interest. Carbon quantum dots (CQDs) are new class of fluorescent carbon nanoparticles family that are usually less than 10 nanometers long [4]. These materials are a new class of zero-dimensional nanomaterials that have many functional groups and good solubility in water [5]. Herein, novel CQDs have been prepared from Pekmez (Doshab) using a facile hydrothermal method. CQDs adsorption film was formed on copper substrate as a barrier that could prevent transportation of corrosive particles. For this purpose, three different concentrations of CQDs were made to investigate (1%, 2%, 5%). To investigate the inhibitory effect of quantum dots, three different methods as self-assembling, coating and directly adding inhibitor to the corrosive media of NaCl 3.5% were used. In each method, CQDs deposit on the surface on coper metal automatically. The inhibition performance of purposed methods was compared through electrochemical impedance spectroscopy (EIS), linear sweep voltammetry (Tafel plots) and FT-IR methods. The results showed that the coating and self-decoration methods are more effective than the direct addition of the inhibitor to the corrosive environment. Also, coating method showed even better efficiency in lower concentrations of CQDs than self-assemble method for long periods of time.

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Study of different magnetic nanocomposites as additives in azolla modified copper electrode for improving the sensitivity of sensor in determination of phthalic acid esters via surface blocking effect

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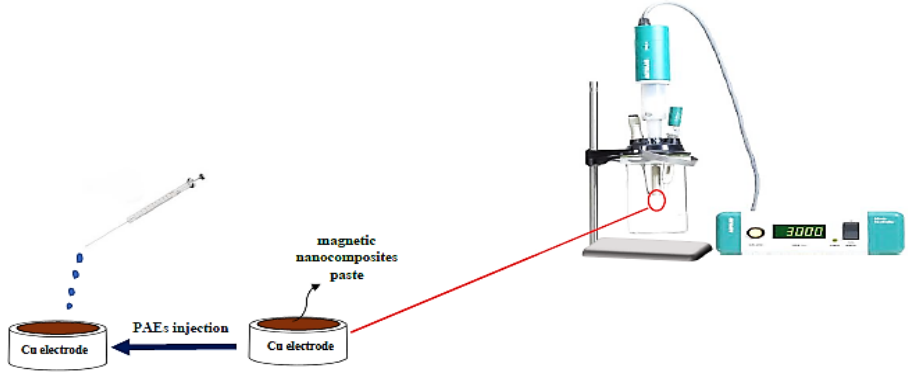
Graphical Abstract



Abstract: Nanoscale materials and devices have been widely explored in the last 20 years [1]. Nano magnetite (Fe_3O_4) is one of the magnetic materials with applications in the electrochemical methods due to its strong super paramagnetic property, low toxicity and easy preparation [2]. At this work, it was tried to design a sensitive and efficient electrochemical sensor for determination of some phthalic acid esters (PAEs) including dibutyl phthalate (DBP), dimethyl phthalate (DMP), di(2-ethylhexyl) phthalate (DEHP) and dicyclohexyl phthalate (DCHP) in aqueous solutions with differential pulse voltammetry (DPV) via PAEs blocking effects on the surface of azolla modified copper electrode in reduction of electrochemical responses of Fe2+/Fe3+. Herein, different magnetic and mesoporous nanocomposites were used for modification of electrode surface to detect trace amounts of PAEs in aqueous solutions. For this purpose, various magnetic nanocomposites including Azolla+Fe_3O_4@Ag, Fe_3O_4@Azolla, Azolla+Fe_3O_4@CMK, Azolla+Fe3O4 MNPs, Azolla+Fe3O4@SiO2@SBA-3, Azolla+Fe3O4@SiO2@SBA-15, Azolla+Fe3O4@SiO2@Kit-6, Azolla+Fe3O4@SiO2, Azolla+NiFe2O4, Azolla+Kit-6-NH2, Azolla+Fe3O4@SiO2@TiO2, Azolla+SBA-15, Azolla+Kit-6, Azolla+Cu NP, Azolla+SBA-3 and Azolla+GO NP were added as surface electrode modifier and were investigated. According to the DPV responses, Fe3O4@Azolla modified electrode showed the best responses and was selected for further studies. Therefore, the surface of the copper electrode was modified with Fe3O4@Azolla powder and electroencephalography gel. The modified electrode was characterized by field emission scanning electron microscopy (FESEM). In the presence of PAEs as electrode blocker, the DPV responses of redox pairs of Fe2+ and Fe3+ was decreased proportional to increase in PAEs concentration. A comparison was made between DPV responses of Azolla modified electrode and Fe_3O_4@Azolla modified electrode. After PAEs injection on the surface of Fe_3O_4@Azolla modified electrode, anodic peak current was decreased more than Azolla modified electrode and the DPV responses were improved between 39.3%-72.5%. Linear relationships were found between the DPV responses and the log C_{PAEs} with LOD = 0.11 $\mu g\ L^{-1}$ and LOQ = 0.36 $\mu g\ L^{-1}$. Good recovery percentages ranging 91.7-101.0% with RSD < 2.2 % suggested the proposed method for sensitive, accurate and quick determination of PAEs in real water samples.

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Electrochemical Study of 1,3-Dinitrobenzene and its Application in Electrochemical Synthesis

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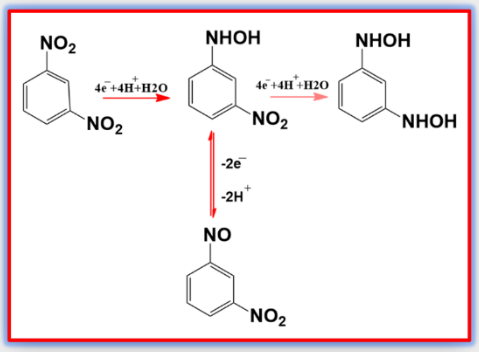
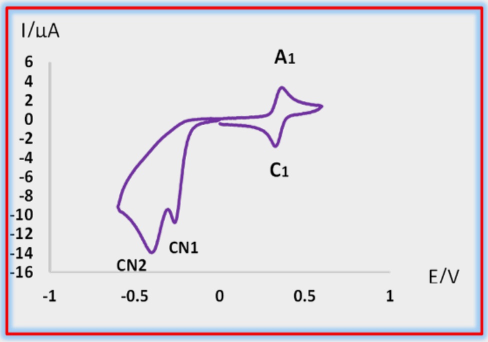
Graphical Abstract





Abstract : The synthetic nitro-aromatic compound 1,3-dinitrobenzene (1,3-DNB) is utilized in explosives, dyes, pesticides, and insecticides, and as an intermediate in the synthesis of chemicals.[1] 1,3-DNB contamination in the environment may be hazardous to animals and humans, and since it is easily absorbed through the skin and auditory therapy, it is continually causing damage to the central nervous system, visual adaption, and overall health.[1] Electrochemical studies are primarily used to track the chemical and electrochemical reactions that occur at the electrode surface.[2] Various types of reactions can be explored using electrochemical methods at varied time intervals ranging from microseconds to hours. By electrochemical reactions, the desired species can be synthesized, identified, and decomposed near the electrode surface. The electrochemical reduction of the nitro group in 1,3-DNB represents a powerful and widely used transformation that allows for the introduction of an amine or hydroxylamine group in the molecule.[3] This study set out to investigate the electrochemical properties of 1,3-DNB in various aqueous solutions (with pH=1-13). The electrochemical behavior of 1,3-DNB was investigated by cyclic voltammetry (CV), chronoamperometry, and chronopotentiometry on a glassy carbon (GC) electrode. Differential pulse voltammetry (DPV) measurements were additionally performed to determine the number of transferred electrons in the reduction process of the nitro group. The cyclic voltammograms of 1,3-DNB showed two cathodic peaks (CN1 and CN2) corresponding to two nitro groups, whereas no anodic peaks were observed in the sweeping area.[4] The first irreversible cathodic peak (CN1) corresponds to the reduction (by transferring $4e^-/4H^+$) of one of the nitro groups of 1,3-DNB to the hydroxylamine group. The second irreversible cathodic peak (CN2) also corresponds to the reduction (by transferring $4e^-/4H^+$) of the other nitro group to hydroxylamine. The anodic (A1) peak and its cathodic counterpart (C1) correspond to the reversible reduction/oxidation of hydroxylamine to nitroso. We also synthesized a new derivative of 1,3-DNB in the presence of benzene sulfinic acid (BSA) in a green condition (acetic acid buffer 0.25 mM, pH = 4). The purified product was then characterized by FTIR, ^{13}C-NMR and ^{1}H-NMR.

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New and Facile synthesis of Zn-Mn-Co LDH/PolyPyrrole Nanocomposite as an Excellent Catalyst for Electro-oxidation of Ethanol in Alkaline Medium

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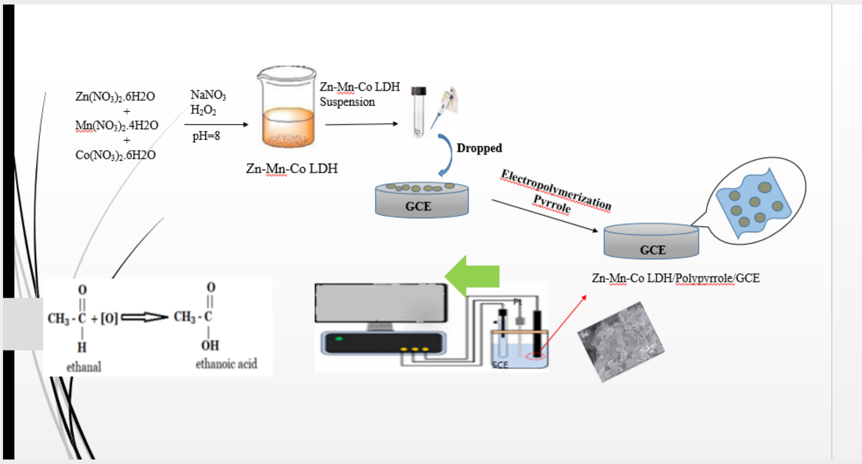
Graphical Abstract



Abstract: In recent decades, fuel cells have received much attention due to their efficiency and biocompatibility in energy conversions. These systems are highly efficient due to convenient transportation and storage, simplicity, high energy density, etc. Direct alcohol fuel cells (DAFCs) have received a great deal of attention due to their efficiency and environmental friendliness in portable electrical appliances. Therefore, the use of alcohols such as methanol, ethanol, and ethylene glycol has many advantages. These fuels are liquid, easy to store, and have a high energy density [1]. Proposed sensor for ethanol oxidation, layered double hydroxides are with the general formula $[M^{II}_{1-x}\ M^{III}_{x}(OH)_2][X^{q-}_{x/q}.\ nH_2O]$. The types of cations participating in their structure are bivalent and trivalent and X^{q-} is an interlayer anion, which is theoretically a limitation for the type of anion between the two the layer is placed, does not exist [2]. And electrochemical polymerization of conducting polymers is simple and most convenient method for preparation of a film on the metal surface [3]. In this work, introduce a new Zn-Mn-Co LDH/ PolyPyrrole nanocomposite as an excellent catalyst for ethanol electrooxidation in alkaline media. The above nanocomposite was synthesized by combining chemical and electrochemical methods. Zn-Mn-Co LDH, a new synthesis, was synthesized through a novel chemical route. Polypyrrole was also synthesized by electrochemical method. The synthesized product was characterized by High resolution TEM, field emission scanning electron microscopy (FESEM), X-ray diffraction (XRD), energy dispersive X-ray spectroscopy (EDX), Raman, FT-IR spectroscopy, and cyclic voltammetry techniques. This product was then used for electrocatalytic oxidation of ethanol in alkaline medium. Zn-Mn-Co LDH/ PolyPyrrole/GCE showed higher electrocatalytic activity than ethanol oxidation. To evaluate the performance of the above catalyst, electrochemical measurements such as cyclic voltammetry and electrochemical impedance spectroscopy were performed using a three-electrode system. The stability of Zn-Mn-Co LDH/PolyPyrrole/GCE catalyst was investigated using chronoamperometry technique, which showed higher stability by retention 92% of the initial current density after 6000 seconds in the long-run current-time curve. This newly developed Zn-Mn-Co LDH/ PolyPyrrole nanocomposite could be a promising anodic catalyst for the use of direct ethanol fuel cells (DEFCs).

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Application of Molybdenum Schiff base Complex and MWCNTs in Preparation of Modified Electrode for Detection of Acetaminophen and Ascorbic Acid

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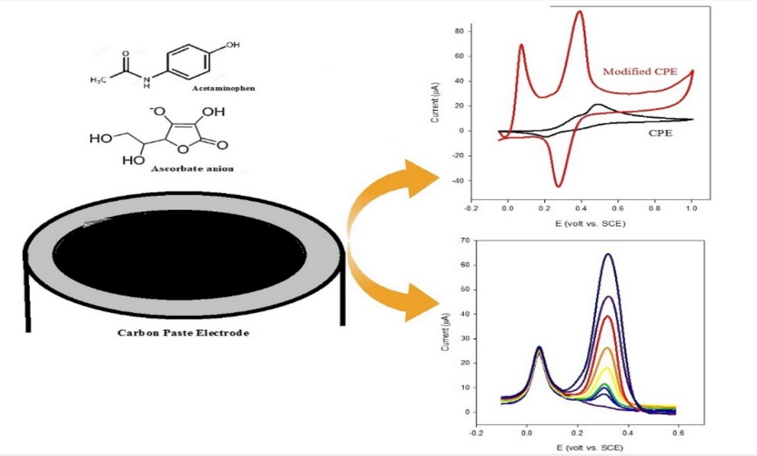

Abstract: The present work reports on a carbon-paste electrode (CPE) modified by incorporating Schiff-base complex of Molybdenum and multi-walled carbon nanotubes (MWCNT). The resulted modified electrode is used as a sensitive voltammetric sensor for determination of acetaminophen (AC) in the presence of ascorbic acid (AA) The electrode showed efficient electrocatalytic activity in lowering the anodic overpotentials and enhancement of the anodic currents. This electrode is able to completely resolve the voltammetric responses of AA and AC. The effects of potential sweep rate and pH of the buffer solution on the response of the electrode, toward AA and AC, and the peak resolution is thoroughly investigated by cyclic and differential pulse voltammetry (CV and DPV). The best peak resolution for these compounds (240 mV) using the modified electrode is obtained in solutions with pH 5.0. Peak potential separation is resulted for two compounds. The modified electrode showed good sensitivity and selectivity. To the electrode has very good reproducibility (R.S.D.≤3%), high stability in its voltammetric response (more than two months without any considerable change in response) and low detection limit (sub micromolar) for AC in the presence of AA. The prepared electrode is successfully applied for the voltammetric detection of AC in pharmaceutical preparations.

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Determination of Acetaminophen and Dopamine in the Presence of Ascorbic Acid Using the Silver Nanoparticles/MWCNTS -Modified Carbon Paste Electrode

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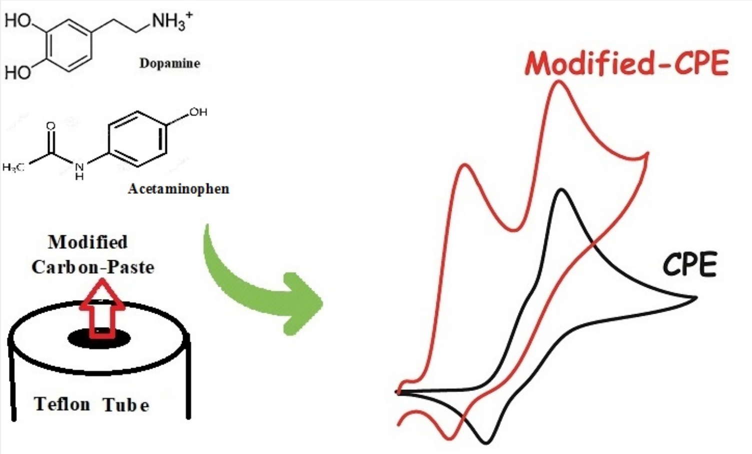
Graphical Abstract



Abstract: In this work, a carbon-paste electrode (CPE) is modified by incorporating silver nanoparticles/MWCNTs composite. The resulted modified electrode is used as a sensitive voltammetric sensor for simultaneous determination acetaminophen (AC) and dopamine (DA). The electrode showed efficient electrocatalytic activity in lowering the anodic overpotentials and enhancement of the anodic currents. This electrode is able to completely resolve the voltammetric responses of AC and DA. The effects of potential sweep rate and pH of the buffer solution on the response of the electrode, toward AC and DA, and the peak resolution is thoroughly investigated by cyclic and differential pulse voltammetry (CV and DPV). The best peak resolution for these compounds using the modified electrode is obtained in solutions with pH 4.0. Peak potential separation is resulted for two compounds. The modified electrode showed good sensitivity and selectivity to the electrode has very good reproducibility (R.S.D.≤3%), low detection limits for AC and DA was 0.1 and 0.6 micromolar respectively. The prepared electrode is successfully applied for the voltammetric detection of AC and DA in pharmaceutical preparations.

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Rapid Detection of SARS-CoV-2 Antigen Using Electrochemical Aptasensor Based on In-Situ two-step Preparation of $Co_3(BTC)_2$/SPCE

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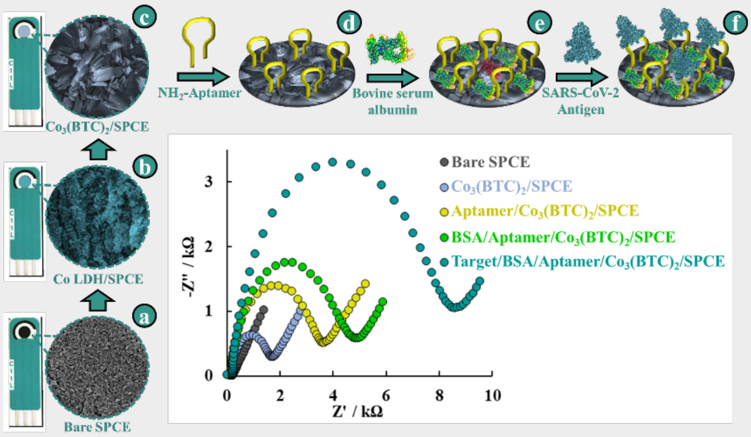
Graphical Abstract



Abstract: In global diseases such as SARS-CoV-2 pandemic, a rapid, accurate, selective, inexpensive, and portable test is needed to diagnostic test at home and preventing the spread of the disease [1, 2]. Aptamer-sensors (aptasensors) as novel diagnostic tools have attracted sufficient research interest in biomedical sciences. Thus, a label free and ultra-sensitive electrochemical aptasensor for SARS-CoV-2 spike glycoprotein detection using $Co_3(BTC)_2$ MOF, synthesized by in situ growth method, were presented. For this purpose, Co-LDH was first synthesized on the disposable screen-printed carbon electrode (SPCE) surface by electrodeposition method, then, by pouring the H3BTC on the surface, the Co-LDH became directly Co-BTC MOF [3]. $Co_3(BTC)_2$ as a high-performance surface substrate leads to more loading of aptamer strings, as biological receptor elements, and increases electrochemical sensitivity. The presence of SARS-CoV-2 spike glycoprotein and the formation of the target-aptamer complex leads to a change in charge transfer resistance (Rct) between $[Fe(CN)_6]^{3-/4-}$ as the redox probe and the electrode surface. The designed aptasensor showed a wide linear sensing range between Rct and increasing SARS-CoV-2 spike glycoprotein concentration in the range of 0.5 fg mL^{-1} to 1.0 µg mL^{-1} with a low detection limit of 0.1 fg mL^{-1} and response time 15 min. In addition, desirable repeatability and reproducibility, selectivity and possibility of application in real samples were found from the provided aptasensor. Also, the aptasensor efficiency was evaluated by real samples of sick and healthy individuals and compared with the standard polymerase chain reaction (PCR) method and acceptable results were observed. Then, the combination of intrinsic properties of electrochemical technology and the advantages of aptamers can promise promising sensors with fast, sensitive and stable performance.

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NiCo hydroxide nano-boxes as an electrode material for reduction of hydrogen peroxide

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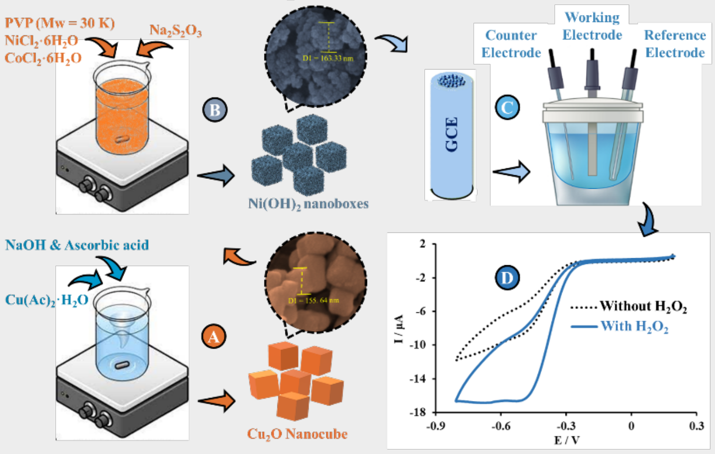
Graphical Abstract



Abstract: The detection of H_2O_2 has attracted considerable attention as H_2O_2 plays an important role in medicine, food, pharmaceutical and environmental pollution, H_2O_2 is a catalytic by-product of a large number of oxidases, and the excessive of H_2O_2 could damage DNA or proteins [1]. So, the analytical determination of H_2O_2 is of utmost importance in many different fields. Transition metal oxide nanostructures are highly regarded by researchers in various fields of electrochemistry due to their attractive properties such as excellent electrochemical activity, ease of preparation, high physical stability, natural abundance and cost-effectiveness [2]. The hollow transition metal oxides nanostructures, as an important family of functional materials, in various applications such as energy storage systems and catalysis has been highly regarded by researchers due to their unique physical and chemical properties, i.e. high surface area, short diffusion pathways for electrons and ions, high electrochemical performance, high porosity etc [3]. Here, uniform amorphous NiCo hydroxide nano-boxes with intact shell structure and nano-sizes were synthesized, while $S_2O_3^{2-}$ was deliberately chosen as the coordinating etchant to the Cu_2O nanocubes templates under optimal reaction conditions. Then, it was used to coat the surface of a glassy carbon electrode as an effective electrocatalyst for the reduction of hydrogen peroxide. This porous and three-dimensional structure provided abundant electrochemical active sites, high electrolyte/analyte availability, and high conductivity due to the synergistic effect of the bimetallic. As an amperometric H_2O_2 sensor, it shown a high sensitivity of 60 µA mM^{-1} cm^{-2} and wide linear range from 0.5 µM to 3 mM with a low detection limit of 0.16 µM. Moreover, the developed sensor shown a great potential in determining H_2O_2 in biological real samples.

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A selective and sensitive sensor based on a glassy carbon electrode modified with nanodiamond-derived carbon nano-onions and silver nanodendrites for methamphetamine detection

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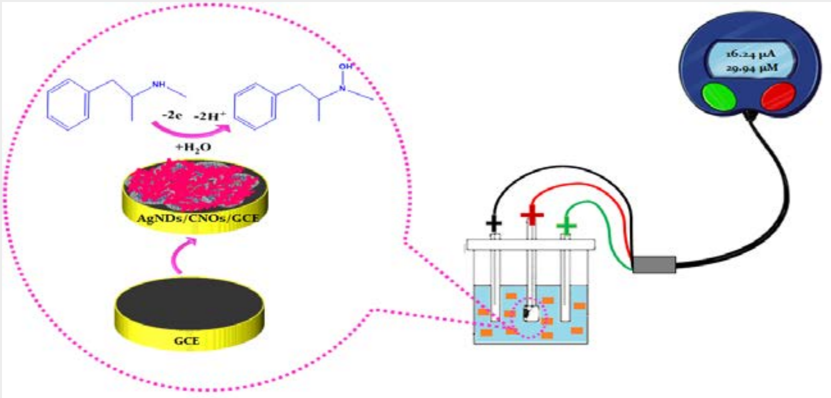
Graphical Abstract



Abstract: Since amphetamine-type stimulants (ATS) drugs have been approximately abused by 24.7 million people [1], there is a growing global demand for the selective trace amount measuring them in the biological fluids. Methamphetamine (MAMP) is one of the famous industrial opiates. Its excessive consumption disrupts the function of the brain neurotransmitters and increases heart rate, respiratory problems and even worse, may lead to stroke and death [2]. So, sensitive MAMP detection in the biological and street samples is vital for several organizations such as forensic medicine, anti-drug headquarters and diagnostic clinics. We fabricated a glassy carbon electrode (GCE) modified with nanodiamond-derived carbon nanoonions (CNOs) and silver nanodendrites (AgNDs), denoted as the AgNDs/CNOs/GCE, as the sensor for MAMP detection. The electron dispersive X–ray analyzer (EDX) and X-ray diffraction patterns (XRD) techniques confirmed the presence of the applied elements as the modifier on the surface. SEM images of the embedded sensing interface revealed a unique distribution of the dendritic structures similar to tree foliage on the GCE surface. The sensing interface presented a high surface area, more conductivity and excellent capability for MAMP catalysis compared to the bare GCE. MAMP was quantitatively detected under two broad concentration ranges from 99 nM to 7.48 µM and 7.48 µM to 59.88 µM with a limit of detection (LOD) of 30 nM. The sensor feasibility in MAMP monitoring was satisfactorily evaluated by analyzing some human blood serum and urine specimens.

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A high sensitive methamphetamine detection by the aptasensor based on hollow carbon spheres and silver nanocubes

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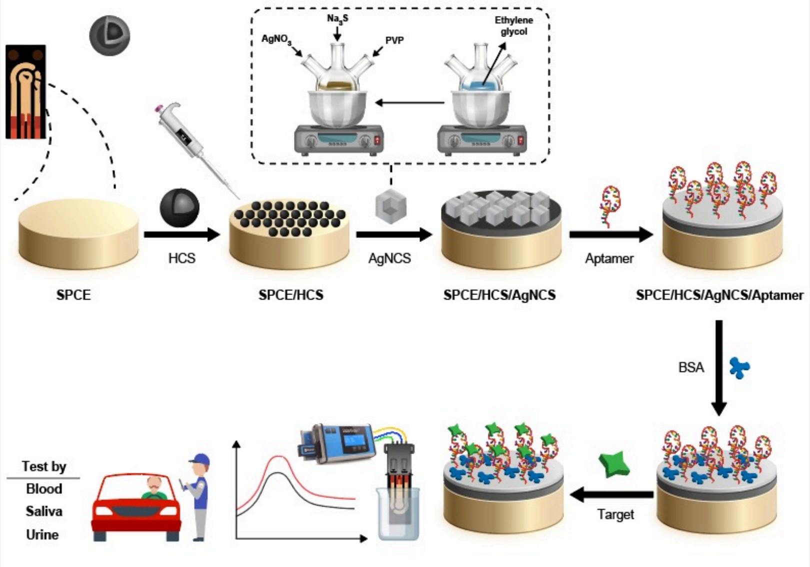
Graphical Abstract



Abstract: Methamphetamine (MAMP), N-methyl-1-phenylpropane-2-amine, has been warned as one of the most popular psychostimulants in the world [1]. The abuse of this industrial opiate causes unconventional mental behaviors with severe heart and respiratory problems, stroke and death [2]. Although several studies have focused on MAMP sensing, a highly sensitive and selective method for accurate tracking of trace amounts of MAMP in the streets samples with complex matrices is still necessary for clinical and judicial organizations to overcome the global addiction problem. Since the aptamer as a highly selective bioreceptor can trap MAMP among different molecules with similar structures or properties, we propose a novel aptasensor for accurate and sensitive MAMP measurement in human serum, urine and saliva samples. The aptasensor works based on the aptamer immobilized on a screen-printed carbon electrode (SPCE) modified by hollow carbon spheres (HCS) and silver nanocubes (Ag NCs). In the MAMP presence, the aptamer arm wraps around MAMP and the electrochemical signal of ferro/ferri cyanide as the redox probe is correspondingly changed by the space barrier increasing. The aptamer can detect MAMP in a broad range of 1 pM to 50 mM with a limit of detection (LOD) **of** 0.02 pM. Since the practical efficiency of MAMP aptasensor has been evaluated appropriately in human serum, urine and saliva samples, the proposed method is promising for MAMP measuring under a non-invasive strategy.

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A New Cu-MOF/Bentonite Hybrid as High-Performance Sensor for Electrochemical Determination of Trazodone

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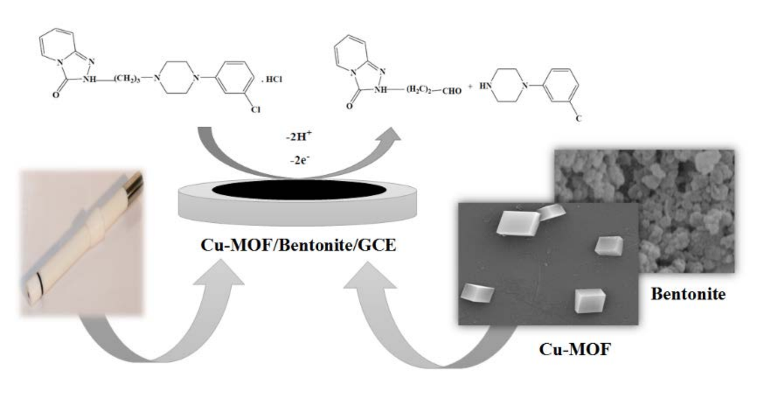
Graphical Abstract



Abstract: Trazodone (TZD) is an antidepressant drug that belongs to the category of serotonin antagonist and reuptake inhibitor [1-4]. It is a triazolopyridine derivate and presents antidepressant, anxiolytic, and hypnotic effects, but produces lower anticholinergic side effects than other antidepressants [5]. TZD was first authorized in the 1970s for the treatment of depression. However, due to its heterogeneous mechanism, other therapeutic uses have been reported, namely, sleep disturbance, anxiety disorders, dementia psychosis, parkinsonism, trembling states, emotional disorders, or dyskinesia. Some side effects are detailed for this antidepressant drug, including nausea, insomnia, agitation, dry mouth, constipation, headache, hypotension, blurred vision, and confusion. Hepatotoxicity is another adverse effect reported for TZD, mainly by oxidative stress and by dysfunction in intracellular organelles. Additionally, interactions with other therapeutic drugs are reported, namely for TZD and warfarin, and TZD and tandospirone. In this research, a novel, reliable and low-cost electrochemical sensor based on Cu-MOF/bentonite hybrid was designed and used for determination of trazodone (TZD). The morphology and chemical characterization of the modified electrode was studied by SEM, TEM, UV-vis, FT-IR, XRD. The electrochemical behavior of trazodone was investigated in detail. The cyclic voltammetric results show that modified glassy carbon electrode by Cu-MOF/bentonite hybrid can remarkably enhance electrocatalytic activity towards the oxidation of trazodone. Under the optimal conditions, the linear dynamic ranges (LDRs) and limit of detections (LODs) were obtained for trazodone 0.1–100 µM and 0.015 µM. The proposed electrode was successfully applied for the analysis of trazodone in pharmaceutical samples.

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Captopril Electrochemical Oxidation using CPE-MIL-53 as a novel electrochemical sensor and its quantitation in Pharmaceutical and human plasma

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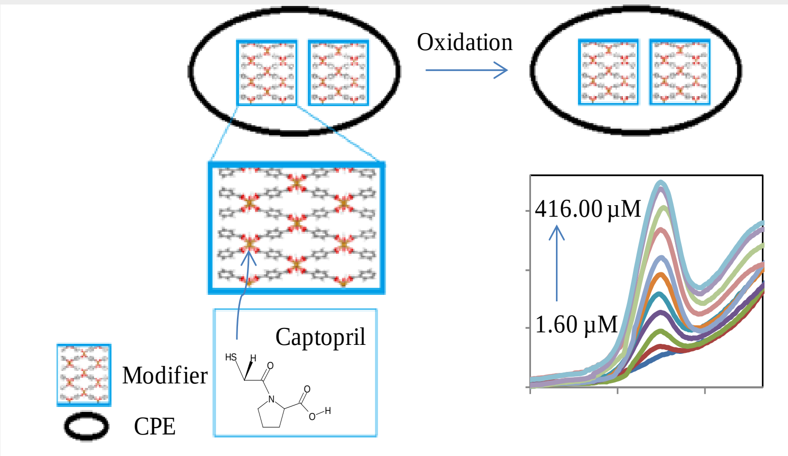
Graphical Abstract



Abstract: A metal-organic framework MIL-53(Cu), is suggested as a favorable novel analytical sensor for the quantitation of captopril in various samples. The electrode was made by modifying a carbon paste electrode with MIL-53(Cu). The electro-oxidation process of CAP has been studied by cyclic voltammetric (CV) and square wave voltammetric (SWV) in phosphate buffer solution (PBS) from pH 3.00–6.00 at a MIL-53-CPE. The prepared electrode shows irreversible current responses for CAP at the potential 1.23 V, while at the surface of the CPE electrode, a current response for CAP cannot be observed at the potential 1.23 V. nanoparticles, MIL-53(Cu), were prepared via microwave procedure in this work. Morphological characterization illustrated that MIL-53(Cu) was well-defined crystallinity, with porous structures and a specific surface area of 1.33 cm^3/g and 5.80 m^2/g, respectively. The consequences displayed that the active sites in the appearance of carbon paste cause the electrocatalytic activity of the MIL-53-CPE electrode into CAP. The MIL-53(Cu) displayed the analysis of CAP with a detection limit of 0.85 µM and a linear range of 1.60 – 416.00 µM. E^0 was calculated to be 1.22, and the electron transfer rate constants (k^0) were obtained to be 0.86 s^{-1}. Recovery evaluations were performed using Pharmaceutical and spiked human plasma samples. The results show recoveries from 95.11% to 104.74% and 95.37% to 106.30% for analyses of drug tablets and human plasma, respectively.

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Electrochemical Oxidation of Acetaminophen and 4- Amino phenol in the Presence of 2-methyl indole

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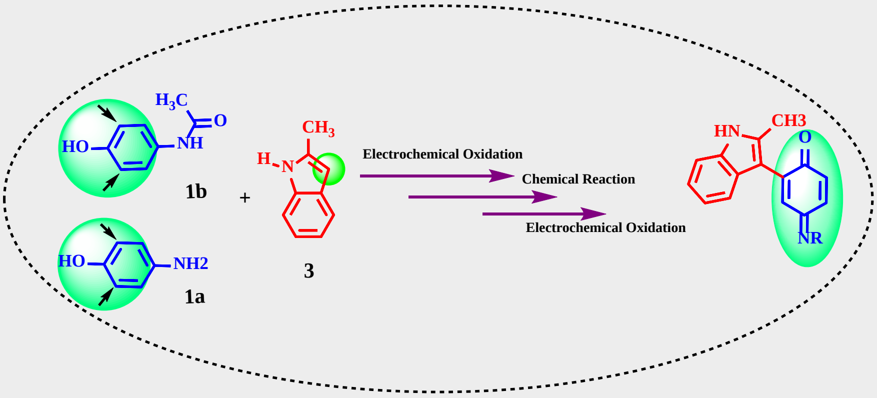
Graphical Abstract



Abstract Electrochemistry has emerged as a powerful tool for the synthesis of complex organic molecules [1]. Unique selectivity because of in situ formation of an active species at the interface, inversion in polarity by transfer of electron and variability in product formation by control of potential are some of advantage of electrosynthesis. On the other hand, synthesis of molecules with desirable functional groups in the absence of catalysts and drastic conditions continues to be of great interest [1, 2]. Our previous studies show that the electrochemically generated *p*-quinone is a reactive intermediate and as a Michael acceptor, participates in different types of reactions [3]. In this work electrochemical oxidation of acetaminophen (**1a**) and 4- amino phenol (**1b**) has been studied in the presence of 2-methyl indole (**3**) as nucleophile (graphical abstract). Some electrochemical techniques such as: cyclic voltammetry using diagnostic criteria derived by Nicholson and Shain for various electrode mechanisms and controlled-potential coulometry were used. Our results indicate the participation of electrochemically generated *p*-quinones in Michael-type addition reaction with 2-methyl indole (**3**) to form new indole derivatives. Diagnostic criteria of cyclic voltammetry, the consumption of four electrons per molecule of **1a**, **b**, and the spectroscopic data of the isolated products, indicated that the reaction mechanism of electrooxidation of **1a**, **b** in the presence of **3** is *ECE* mechanism. Nature stability of *p*-quinones in comparison with *o*-quinones in one hand and stability of final products arising from intramolecular hydrogen bonding, on the other hand, are responsible for remaining the final products in quinone form.

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Preparation of electrochemical sensor based on 3-thiophene acetic acid molecularly imprinted polymer for detection of *Streptococcus pneumoniae*

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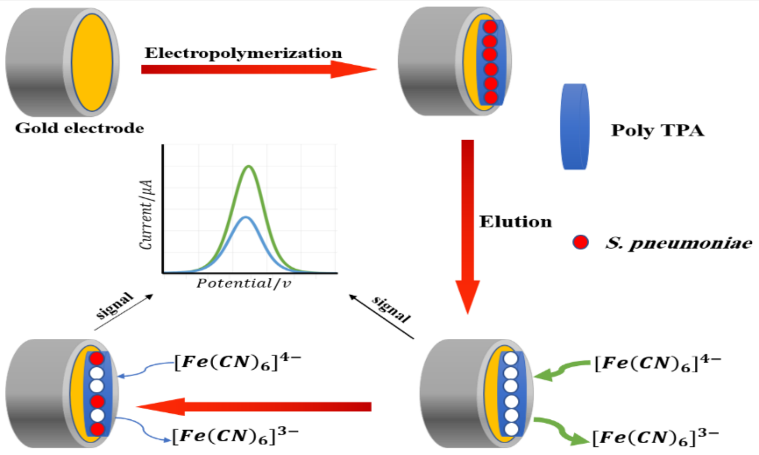

Abstract: *Streptococcus pneumoniae (SP)* is one of the most common gram-positive bacteria human pathogens and a cause of including pink eye, meningitis and pneumonia, sinusitis, and otitis media. Sometimes it infects tissues in other parts of the body Therefore, the rapid and sensitive detection and measurement of this bacteria is vital. Among various methods, electrochemical analyses, with label-free, are a promising technique because of their merits of being rapid, highly sensitive, and is widely used [1, 2]. *S. pneumoniae* -imprinted polymer-based sensor was prepared to detect *S. pneumoniae* by electropolymerization of 3-thiophene acetic acid on the gold electrode surface using the cyclic voltammetry method in the presence of *S. pneumoniae* as the template and 3-thiophene acetic acid as the functional monomer. After the removal of the bacterial template from the electrode surface, a series of cavities will be formed in the shape of the desired bacteria in the polymer structure [3]. To study the morphology of the desired bacterium and poly TPA, SEM images of different polymer films modified electrodes were used during the process of bacterial imprinting and recognition. CV and EIS techniques were used to characterize the imprinting process and DPV technique was introduced to draw a calibration graph under optimal conditions and for all of the electrochemical measurements. The selectivity of the proposed sensor was investigated by interfering bacteria. The high selectivity of the sensor against the template bacteria could be ascribed to its sensitivity to the chemical conformation of bacterial outer cell structures. The imprinted electrochemical sensor based on a surface-imprinted polymer-modified electrode was successfully applied to detect *S. pneumoniae* in human serum.

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Electrochemical Production of Biodiesel Using Carbon Quantum Dots and Mixture of Homo-Heterogeneous Catalysts

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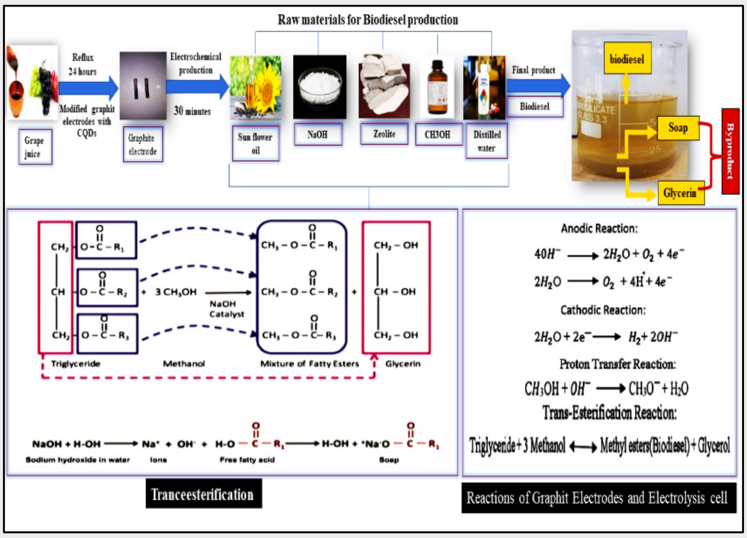

Abstract: The scarcity of known petroleum reserves will make renewable energy resources more attractive. The most feasible way to meet this growing demand is by utilizing alternative fuels. Biodiesel is defined as the monoalkyl esters of vegetable oils or animal fats. Biodiesel is the best candidate for diesel fuels in diesel engines. The biggest advantage that biodiesel has over gasoline and petroleum diesel is its environmental friendliness. Biodiesel burns similar to petroleum diesel as it concerns regulated pollutants. On the other hand, biodiesel probably has better efficiency than gasoline. The major economic factor to consider for input costs of biodiesel production is the feedstock, which is about 80% of the total operating cost. The high price of biodiesel is in large part due to the high price of the feedstock [1]. Problem of high price of biodiesel production solved by electrochemical methods that reduces the cost of heat required for the reaction and the cost of washing due to the high amounts of sodium hydroxide in the conventional methods [2]. This research introduces inexpensive way of production biodiesel using the electrolysis methods with graphite electrodes modified with carbon quantum dots (CQDs) in the presence of zeolite and in alkaline medium. Low applied potential (4V) with less time of production have been used for the synthesis of biodiesel from cooking oil as a sustainable feedstock throw out the transesterification method. The effects of methanol/cooking oil ratio and the mixture of zeolite clinoptilolite and sodium hydroxide concentration as a catalyst on biodiesel conversion were carefully investigated. The optimum condition for the appropriate transesterification conditions were: sodium hydroxide as homogeneous catalyst (1 wt%) and zeolite clinoptilolite as heterogeneous catalyst (1 wt%) with optimum solvent (methanol) to oil volume ratios (1:6) and water (1.8 wt%). The obtained product was studied by UV-visible spectroscopy in the range of 200-400 nm wavelength to ensure the synthesis of biodiesel. The results showed that due to the characteristics of the graphite electrode, there was no voltage drop or reduction in reaction rate and the temperature from the beginning to the end of the reaction, which ultimately leads to the production of biodiesel with 99.36% efficiency during 30 min with the minimum amount of glycerol and soap as by-products.

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Non-enzymatic sensing of hydrogen peroxide using a glassy carbon electrode modified with nanolayers of manganese-calcium oxide

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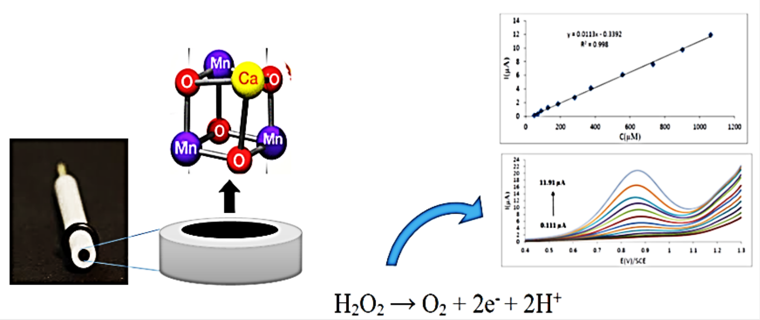

Abstract: Manganese-calcium oxide has the chemical formula ($MnCaO_2$). The appearance of this compound is dark brown powder. This compound is an effective catalyst for the oxidation of organic and inorganic compounds. In this research, glassy carbon (GC) electrode modified with manganese-calcium oxide nanolayers was used to study the electrochemical behavior and measurement of hydrogen peroxide. For this purpose, the surface of the electrode was modified by drop casting of nanolayers of manganese-calcium oxide. Optimization of some parameters affecting the efficiency of the modified electrode decompositions such suspension, volume of drop, solution pH, buffer type, buffer concentration, electrolyte type and electrolyte concentration were performed using differential pulse voltammetry and cyclic voltammetry techniques [1,2]. MnCaO2 nanolayers were also examined by scanning electron microscopy (SEM), X-ray energy diffraction (EDX), X-ray diffraction (XRD) spectroscopy and Fourier-transform infrared (FTIR) spectroscopy. Increasing the current and reducing the excess potential of hydrogen peroxide on the glassy carbon electrode modified with manganese-calcium oxide nanolayers is the main advantage of the designed sensor. This modified electrode has an excellent linear range and a lower detection limit than the bare glassy carbon electrode. The modified $MnCaO_2$ / GC electrode has sensitivity, selectivity, stability, good reproducibility, larger active surface area than bare glassy carbon electrode and fast response. Also, using differential pulse voltammetry technique, a calibration graph was drawn in concentration range of 49.75 to 1063.4 μM. Finally, after obtaining a linear range with a detection limit of 47.84 μM, the analytical efficiency of this modified electrode against measurement of hydrogen peroxide in mouth wash solution was evaluated.

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Fabrication of Electrochemical Aptasensor Modified by NanoParticles to Ultrasensitive Detection of Tetracycline in Milk

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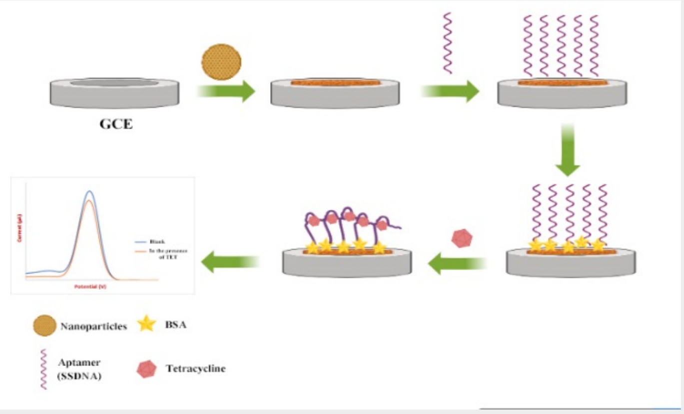

Abstract: Using antibacterial drugs plays an important role in the treatment of human diseases and veterinary medicine for more than a hundred years. Nowadays, excessive use of antibiotics resulted in antimicrobial resistance (AMR), and antibiotic resistance has become a serious public health problem.[1, 2] Among all antibacterial drugs, tetracycline (TET) has been commonly used in the treatment of animal and human diseases due to its good bactericidal properties and low cost [3]. Therefore, development of a selective, and sensitive technique is urgent to detect tetracycline in food samples [4] In this study, an ultrasensitive and selective label-free electrochemical aptasensor based on a glassy carbon electrode (GCE), modified by nano particles and aptamer single strand was fabricated for detection of tetracycline (TET) trace values in food samples.

The stepwise modification process of the aptasensor fabrication was characterized by CV, DPV and EIS techniques. The optimization of the instrumental parameters and experimental conditions (pH, incubation time and concentration of nanoparticles, aptamer and TET) was performed. Under optimized conditions, the developed aptasensor was used to determine the tetracycline concentration using Differential Pulse Voltammetry (DPV). This biosensor was achieved a good dynamic linear range (10^{-16}–10^{-10} M) and a low detection limit (1.079×10^{-17} M). Fabricated aptasensor showed excellent stability, selectivity, repeatability (RSD = 1.58%), reproducibility (RSD = 3.40%) and accuracy. Finally, this aptasensor was applied to the determination of tetracycline in milk.

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Investigation of electrochemical behavior of mesalazine in the presence of amino acids

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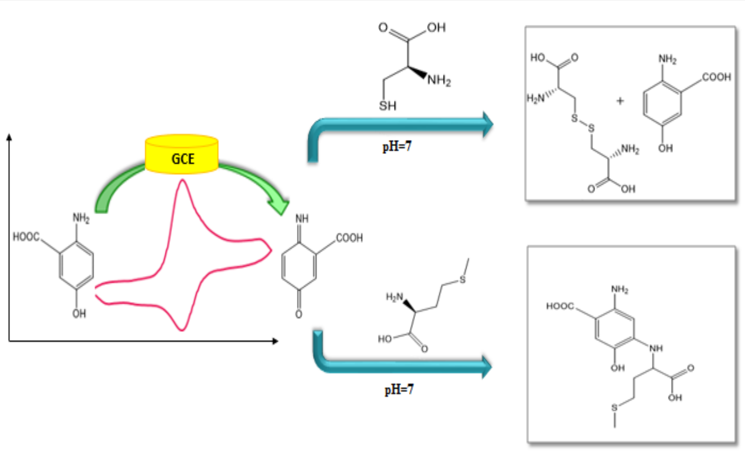

Abstract: Electrochemical oxidation of mesalazine in the presence of L-cysteine, and L-methionine as essential and semi-essential amino acids in aqueous solution was performed using cyclic voltammetry and potential colometric techniques on the surface of a glassy carbon electrode [1]. The results showed that quinone imine induced by mesalazine oxidation reacts with amino acids. Quinone imine reacts with L-cysteine via catalytic (EC '), and with L-methionine via EC mechanism [2]. In this work, the final products were synthesized using potential and current controlled condition in aqueous solution through a catalyst-free environmentally friendly method, without toxic reagents and solvents at a carbon electrode in a single cell, at room temperature under green conditions. The final products after separation and purification, were studied and by NMR, IR and MS spectroscopic methods [3].

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Preparation of Silver Ion Selective Electrode Using a New Synthesized Ligand as Ionophore

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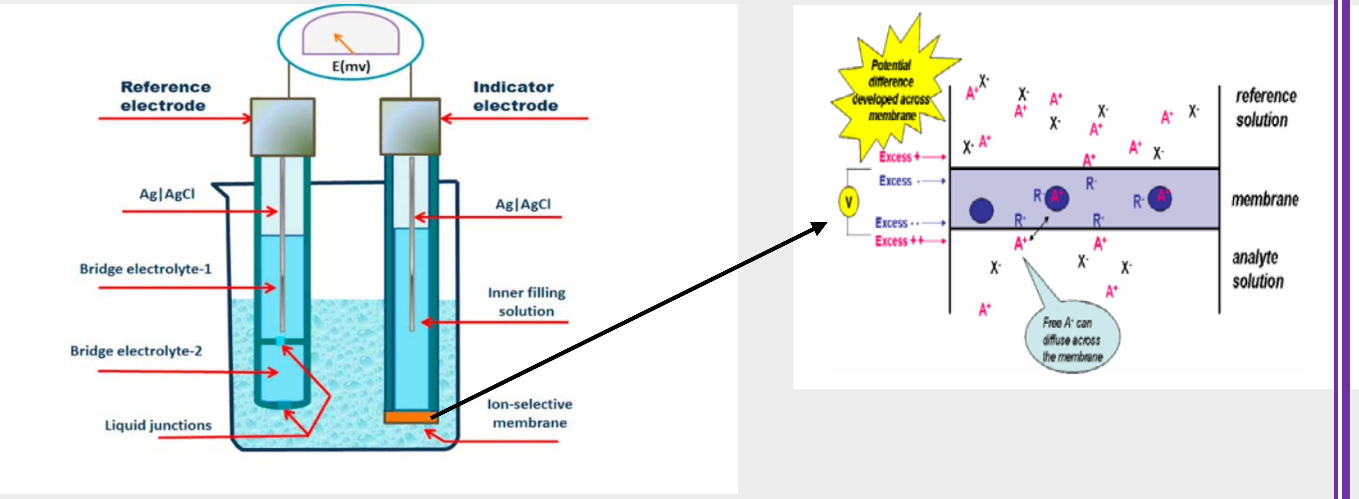

Abstract: Identification, measurement and separation of silver ion is important due to the many applications of this metal in various industries and medicine. Another issue that makes silver important is the poisoning caused by silver in the body. In this study, we are looking for a cheap, portable and high-precision method to measure silver ions. The chosen method is the method of ion-selective electrodes (ISE). The main point in the manufacture of ion-selective electrodes is the construction of the membrane. The constructed membrane includes an ionophore, ion additive, PVC and lubricant. The ligand used is with N'-N'-N'-S (5-Bermo-2-Hydroxybenzylin) -2-2-Dimethylpropane-3. Benzyl acetate is used as a lubricant and sodium Tetra phenyl borate (NaTPB) is used as an ionic additive. In addition to the type of membrane components, its amount is also important for making an ion-selective electrode. PVC, ionic additive, ligand, and lubricant are: 39, 3, 5, and 53 percent, respectively. The (ISE) showed linear Nernst responses to Ag + ions in the range of 1.0×10^{-2} to 1.0×10^{-7} M The built ion selective electrode gives a correct linear response in the shortest possible time in the presence of interfering ions and also in the range of pH range.

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Construction of novel and nonenzymatic electrochemical biosensor with modification of carbon paste electrode with Cr_2O_3 /PSY nanocomposite for determination of PA in tablet sample

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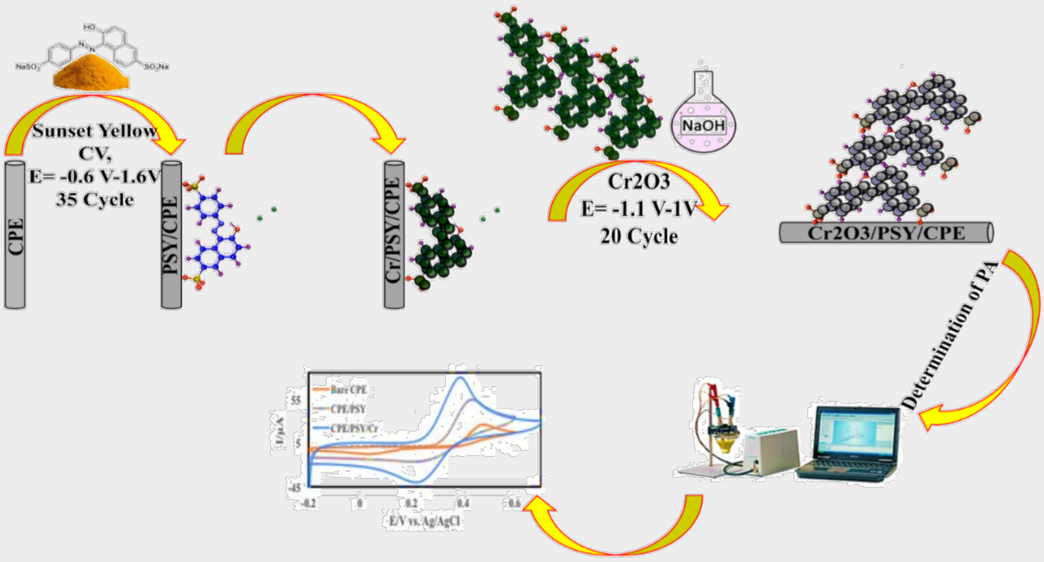

Abstract: Paracetamol (PA) plays an important role in the pharmaceutical field due to its wide use as an antipyretic and pain reliever. Limited use of PA has no harmful side effects, but excessive and addictive use of this compound can cause injury and death in some cases. Monitoring the amount of PA in pharmaceutical formulations is essential for quality control as well as public health perspectives. Recently, electrochemical techniques based on modified electrodes (sensors) have been widely used for the measurement of pharmaceutical compounds [1-3]. Various composites can be used to modify the surface of the working electrode. The materials that can be used in the preparation of these composites are basically nanocomposites, including polymers, carbon nanomaterials, metal nanoparticles, etc. (Cr_2O_3) nanoparticles were successfully fabricated and used for PA measurement with high sensitivity. The morphology and structure of nanocomposite were investigated by FE-SEM, EDS, Mapping, ATR-IR and XRD techniques. Compared with CPE and PSY/CPE, modified Cr_2O_3/PSY/CPE showed better electrocatalytic activity for PA oxidation. Cyclic voltammetry and differential pulse voltammetry techniques were used for the measurements. The amount of surface conductivity was investigated by impedance technique and the results showed an increase in surface conductivity. The limit of detection (LOD) obtained was 99.4 nm and the linear dynamic range (LDR) from 0.5 µM to 650.0 µM (S/N = 3) was observed. The fabricated sensor has good features such as high sensitivity, high reproducibility and reproducibility, low detection limit and fast response time. The results obtained from the real sample were quite satisfactory.

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Determination of prostate Cancer Biomarker, Citrate Ion, by Electrochemiluminescence sensor based on NanoPIM/N-CQDs Luminophore

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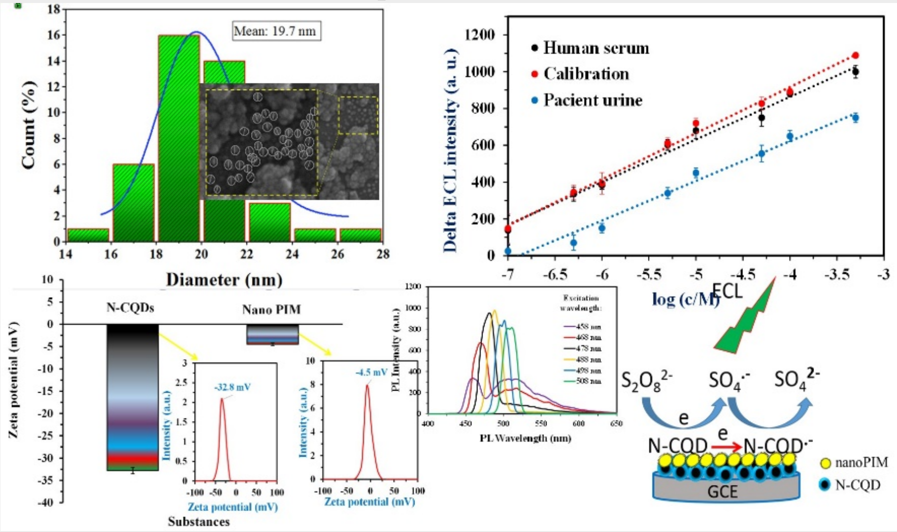

Abstract: Prostate cancer is one of the most important diseases among men around the world. Like other types of cancer, diagnosis of this disease in early stages is crucial for treatment. Studies showed that citrate ion level in men body fluids, especially in urine, could be one of the important signs of this cancer in early stages [1]. Citrate has special functions in prostate gland and during the cancer disease its secretion is reduced to lower levels than normal (0.06-0.14 mM) [2]. Therefore, determination of citrate as an important biomarker of prostate cancer could help to diagnosis the cancer in early stages. Although different methods have been used to determination of citrate, electrochemiluminescence (ECL) has not been studied yet [1, 3]. ECL has become one of the important analytical methods for determination of various analytes because of its high sensitivity and selectivity. Classical ECL Luminophores like Tris(bipyridine)ruthenium(II), are toxic and expensive. Therefore, it is essential to find the new luminophores for preparation of green and cheap ECL sensors. In this study, an ECL sensor for determination of citrate ion was prepared using nanostructure polymer of intrinsic microporosity (nanoPIM) as ECL enhancer of N-CQDs/$S_2O_8^{-2}$ system. Based on the results of our previous study, ECL of N-CQDs was quenched by the citrate ion [4] and this quenching later was observed to be more sensitive and linear using nanoPIM/N-CQDs as luminophore. Therefore, it was proposed to develop the mentioned ECL sensor for determination of citrate in human serum and urine. NanoPIM was synthesized form the bulk PIM and characterized by FT-IR, FESEM, DLS, Zeta potential and PL methods. The linear range and LOD of determination were 5×10^{-4} to 10^{-7} M and 2.23×10^{-8} M respectively. Prepared sensor also was successfully applied for determination of citrate in human serum and patient urine with the recoveries of 97.7% and 86.0% respectively. Considering the broad linear range, high sensitivity and selectivity of the sensor in real samples, comparing with previously used methods for determination of citrate, it showed that prepared sensor could be applied for screening of citrate in human biological fluids for diagnosing of prostate cancer in early stages.

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PVC/Mg-Al LDH Electrospun Nanofibers as a New Sorbent for Simultaneous Micro-SPE and Determination of DA and UA in Human Blood Plasma Samples Followed by Sensitive Voltametric Biosensor (GCE/Ppy/DEA MIP) Detection

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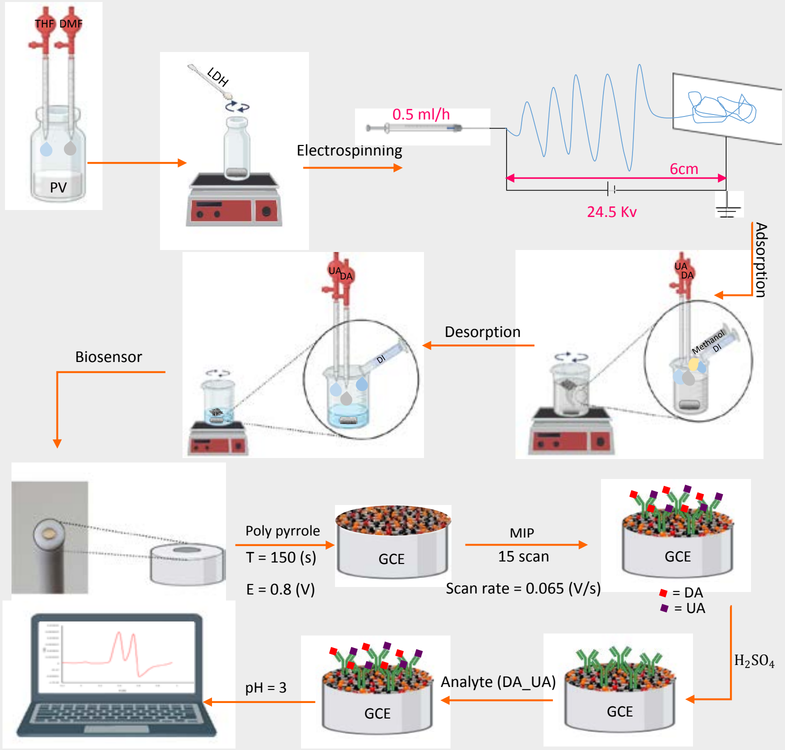
Graphical Abstract



Abstract: Dopamine (DA) and uric acid (UA) always coexisted in the extracellular fluids of the central nervous system and serum in mammals, which play critical roles in human metabolism [1-3]. New electrospinning nanofibers with polyvinyl chloride (PVC)/a layer of double hydroxide (Mg-Al-LDH) were prepared. Since measuring trace concentration of DA and UA in complex matrix is essential, we used electrospinning method for preparing nanofibers and then used nanofibers as a new sorbent in micro extraction method for simultaneous extraction of DA and UA prior detection with glassy carbon electrode (GCE)/ poly pyrrole (Ppy) /DA, UA, and Diethyl amine (DEA) as molecularly imprinted polymer (MIP). Effective factors on adsorption process; including pH of adsorption solution, adsorption time, Percentage of salt added to the adsorption solution, stirring rate, and the size of sorbent, were optimized by the one-at-time (OAT) method. Effective factors on constructed biosensors such as time and potential and effective factors on desorption process; including desorption time, eluent type, and the volume of eluent, were optimized by design-expert-12.0 software. The nanofibers and biosensor were characterized by FE-SEM, XRD, and FT-IR. Finally, under optimal conditions, the electrochemical biosensor was put into human blood plasma samples, and the standard addition method was applied with the spike of DA and UA to them. The wide linear range and lower detection limit were obtained.

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Innovative hydrothermal MgFeAl-LDH synthesis as an effective nickel foam modification agent for catalyzing water oxidation

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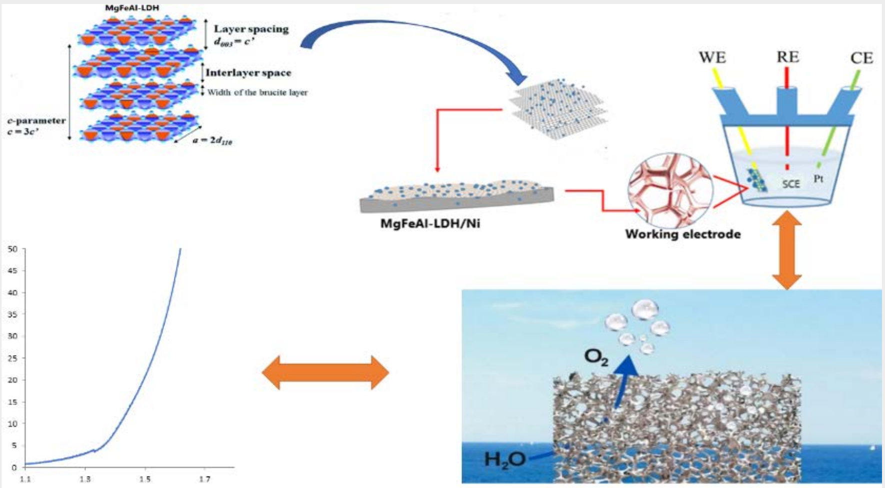

Abstract: Due to the increasing energy needs and global environmental concerns, extensive research is underway to find clean energy sources at a low cost. Hydrogen has been proposed as a clean fuel and renewable energy source. One of the most methods of producing hydrogen fuel is water splitting. However, the oxidation of water is kinetically slow and limits the rate of the water splitting reaction. Therefore, it is necessary to develop materials that can oxidize water effectively [1]. Various catalysts for water oxidation have been proposed. The proposed catalyst is Mg-Fe-Al LDH. Double-layer hydroxides (LDHs) are a large class of natural and synthetic materials that have received much attention in recent years due to their promising applications in many fields, such as catalysts [2]. After preparing nickel foam, which increases the surface area of the electrode and rate of electron transfer, was used to study water oxidation. Techniques such as X-ray diffraction (XRD), energy scattered X-ray (EDX), transmission electron microscopy (TEM), and field emission scanning electron microscopy (FE-SEM) were used to study the structure and morphology of the synthesized sample. The proposed sensor has good stability, which makes it a very active, stable, low-cost and promising catalytic system in the field of energy analysis, conversion and storage. The catalytic performance of oxygen evolution reaction (OER) was studied by chronoamperometry (CA), cyclic voltammetry (CV), and linear sweep voltammetry (LSV), techniques. Electrolysis of water using Mg-Fe-Al LDH /NF with a current density of 5 mA cm^{-2} can be achieved with a cell voltage of 1.41 (vs. RHE) volts in 1 M KOH solution. The catalyst synthesized in the oxygen evolution reaction to reach 5 mA cm^{-2} has a overpotential of only 180 mV.

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Design of an electrochemical sensor for non-enzymatic detection of glucose using a mixed Ag/NiO-Ag/NiS as a modifier

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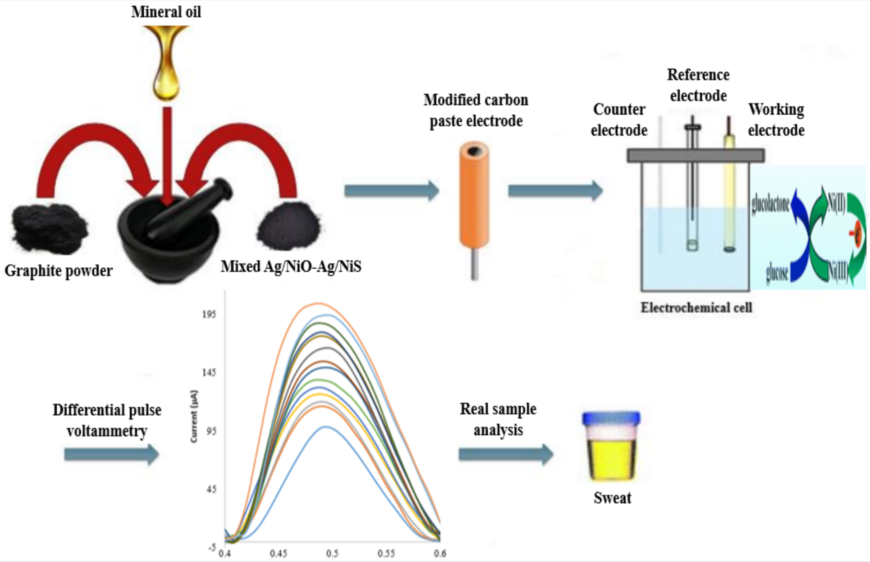
Graphical Abstract



Abstract: Diabetes is a group of metabolic diseases caused by high glucose levels [1]. This disease, over time, causes significant damage to many of the body's systems, particularly the nerves and blood vessels. Current glucose sensors in the market use enzymes as an active material and modifier. These sensors have intrinsic chemical and thermal sensitivity. A wide variety of enzyme-free sensors, including transition metals, have attracted attention among the various modifications of glucose electrochemical sensors due to their low cost and ubiquity on the earth [2]. Metal oxide sensors can be a suitable alternative to enzymatic sensors with a longer lifetime [3]. Monitoring glucose through sweat can simplify the work and provide data for more accurate diagnosis and treatment [4]. This work reports a non-enzymatic glucose sensor based on doping Ag in NiO and NiS (Ag/NiO-Ag/NiS) nanocomposite compounds. Modifier was synthesized by hydrothermal method, the carbon paste electrode was modified with it and used as working electrode. The platinum wire, Ag/AgCl electrode were used as counter and reference electrode in this work. Ag/NiO-Ag/NiS was characterized using scanning electron microscopy (SEM), Energy-dispersive X-ray spectroscopy (EDX), electrochemical impedance spectroscopic (EIS), and X-ray diffraction (XRD). The electrocatalytic activity of the modifier was investigated by cyclic voltammetry, and the differential pulse voltammetry technique was applied for monitoring glucose in alkaline media. The proposed sensor has a linear range from 1 μM to 4 mM with a detection limit of 0.23 μM, and also displays excellent selectivity, stability, and reproducibility. Finally, the sensor was successfully applied to detect glucose in human sweat as a real sample.

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Design a new electrochemical sensor based on Co-Fe layered double hydroxide for simultaneous determination of Hydroquinone, Catechol, and Resorcinol

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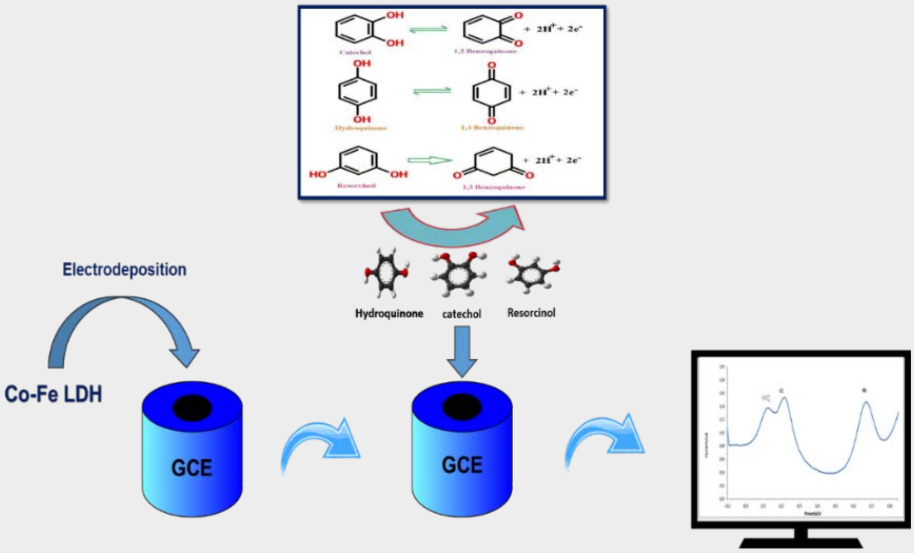

Abstract: Hydroquinone (HQ), catechol (CC) and resorcinol (RS), isomers of dihydroxybenzene, are important organic intermediates for the products of industry and agriculture, considered highly toxic to both the environment and humans even at very low concentrations [1]. Layered double hydroxides (LDH), also known as hydrotalcite like anionic clay, are a class of ionic lamellar compounds consisting of positively charged brucite-like layers and charge compensating anions and solvation molecules in the interlayer region [2]. In this work, an electrochemical sensor for dihydroxybenzene isomers was developed using a glassy carbon electrode modified electrochemically with Fe-Co layered double hydroxides (Fe-Co LDH). The modifier morphology and characteristics were determined by electrochemical impedance spectroscopy (EIS) and field emission scanning electron microscopy (FESEM). Using electrochemical techniques, it was shown that the electrode's specific surface was significantly enhanced. The results obtained show that this novel sensor displays excellent electro-catalytic activity towards the oxidation of these isomers and that these CV peaks are well separated. The sensor was applied to the simultaneous determination of each of the three isomers using DPV with a linear response in the concentration range of 2.5×10^{-4} - 1.7 mM , 2.5×10^{-4} - 1.7 mM, and 1.2 $\times10^{-3}$ - 0.42 mM for HQ, CC, and RS, respectively. Excellent reproducibility, selectivity, stability, and sensitivity results are achieved for the determination of HQ, CC, and RS.

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Electrochemical Carbon Dioxide Reduction in Tandem with Electro-Oxidative Synthesis of Sulfa Drugs

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Graphical Abstract

Abstract: The continuous increase in oil-based sources consumption has led to accumulation of carbon dioxide in the atmosphere, causing serious harmful ecological and environmental effects over the last decades. CO_2 is considered the main gas responsible for climate change. At the same time, CO_2 is also a potentially useful carbon resource [1]. Converting CO_2 to fuel provides a unique advantage that can be a dual-purpose way to remove CO_2 as well as produce value-added products. A broad spectrum of strategies is exerted to convert CO_2. Among these, electrochemical approach has emerged as the greenest known methods providing a green, modern, and safe technique for achieving a diverse range of chemical conversions, as well as minimizing energy consumption [2]. However, CO_2 is a thermodynamically stable molecule, which makes it difficult to be reduced. Therefore, a CO_2 conversion reaction requires high input energy and efficient catalysts to achieve greater efficiency. A more valuable study is to introduce a catalyst that is highly selective and leads to a significant reduction in energy demand for CO_2RR. On the other hand, the strategy of paired electrochemical synthesis, in which the reactions at both the anode and cathode simultaneously contribute to the formation of the final product(s), could result in as much as a 50% reduction in energy consumption as compared to conventional electro-organic syntheses [3]. Herein, we describe a paired electrochemical synthesis for simultaneous CO_2RR with cobalt phthalocyanine@nitrogen-doped ordered mesoporous carbon (CoPc@N-doped OMC) modified electrode in the cathodic part and producing of sulfa drugs in the anodic part. N-doped OMC exhibits superior chemical and electrochemical performance as a result of their unique properties derived from the nitrogen lone-pair electrons [4, 5]. In this direction, N-doped OMC was fabricated by the carbonization of ionic liquid (1-methyl-3-phenethyl-1H-imidazolium hydrogen sulfate) and guanine using hard templating with ordered mesoporous silica SBA-15. The synergistic effect of CoPc and N-doped OMC substances has increased the electrocatalytic activity of CO_2 reduction and the presence of nitrogen groups has resulted in a completely uniform distribution of the complex centers. The use of molecular catalysts with high activity and selectivity, in an appropriate cell configuration, leads to CO_2RR in tandem with electrochemical synthesis of sulfa drugs.

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A Novel Electrode for a High Capacitance Supercapacitor by using Nickel Sulfide/Copper Sulfide Composite Derived from Metal Organic Frameworks

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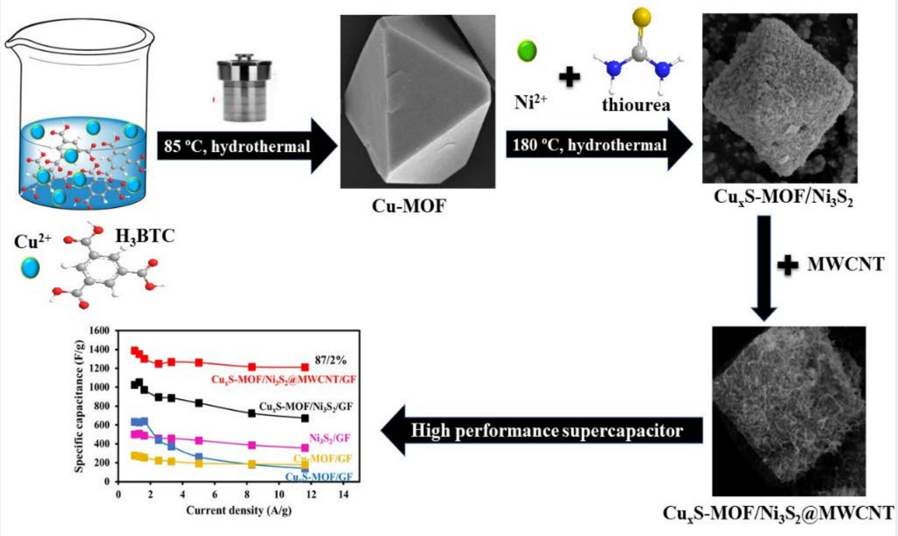
Graphical Abstract



Abstract: In view of the importance of rational design of novel nanocomposites for high performance supercapacitors to achieve high energy and power density and high rate capability, we have presented a promising and facile strategy for fabrication of a mixed-metal sulfide composed of Ni_3S_2 and Cu-MOF-derived Cu_xS. Cu_xS/Ni_3S_2 has been synthesized by using in situ conversion of host Cu-MOF during deposition of Ni_3S_2 on its surface through a simple hydrothermal method. Then, the prepared Cu_xS-MOF/Ni_3S_2 was encapsulated with MWCNT using ultrasonication. Placement of the nanocomposite hybrid material on graphite foil (GF) resulted in superior capacitive properties and stability for use in electrochemical supercapacitors. The synergistic effect of the components in Cu_xS/Ni_3S_2@MWCNT/GF showed excellent supercapacitor performance [1-5]. The composite electrode was characterized by several methods (XRD, XPS, FT-IR, SEM, TEM, EDX and BET). In addition, electrochemical methods such as CV, GCD and EIS were used to investigate the electrochemical properties of the fabricated electrode. Electrochemical performance of Cu_xS/Ni_3S_2@MWCNT/GF exhibited a great improvement in capacitive properties. It showed an extremely large electrochemical surface area (ECSA = 1779 cm2), which is of primary importance in supercapacitors. CuxS/Ni3S2@MWCNT/GF electrode achieved an outstanding capacitance of 1388.7 F g^{-1} at 1 A g^{-1}, superior rate capability of 1211.2 F g^{-1} at 11.6 A g^{-1} (87.2 %), and excellent cycling stability (108% after 10,000 GCD cycles at 33.3 A g^{-1}). Due to the easy synthetic route, this paves the way for a new perspective for high energy and power storage applications in the energy storage sectors.

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Electrofenton process based on iron electrodes for decoloration of Malachite green from aqueous solutions

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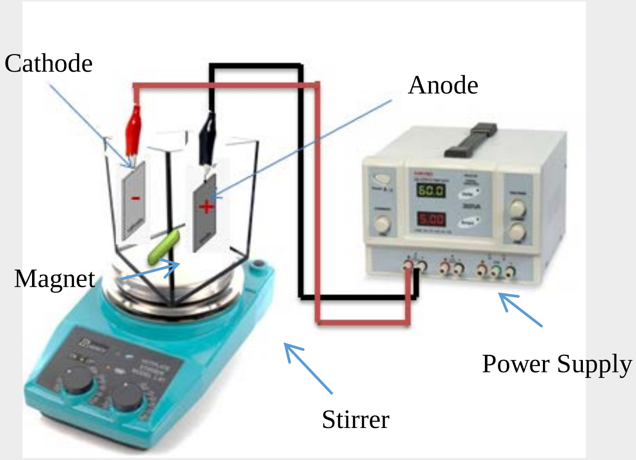
Graphical Abstract



Abstract*:* The electrofenton process has been widely used in dye removal due to its reduced energy consumption, high oxidation strength and production of environmentally friendly by-products. Malachite green (MG) as a cationic dark green dye (λ_{max} = 617 nm) is available in the form of the chloride salt $[C_6H_5C(C_6H_4N(CH_3)_2)_2]Cl$. It was most widely used for coloring purposes in industries of fibers and as an antifungal and antiparasitic agent in aquaculture [1, 2]. The aim of the present work was to degrade the MG content in aqueous samples using electrofenton process. All experiments were performed in a glass cubic reactor with a total volume of 900 mL with two iron electrodes (63 × 80 × 63 mm) connected to a direct current power supply. The effect of five effective parameters such as applied voltage (5, 10, 15, 20 V), ionic strength (0, 0.05, 0.1, 0.2 M), dye concentration (50, 100, 200, 250 mg L^{-1}), pH of solution (2, 3, 4, 6) and percentage of H_2O_2 (10, 20, 25, 30 %) were investigated in four levels and optimized by Taguchi orthogonal array design method (OA_{16}). The residual concentrations of MG after electrofenton process were spectrophotometrically determined in the time range of 0-90 min with time intervals of 10 min and the removal efficiencies were calculated. Based on the results, the optimum conditions were obtained as pH: 2, voltage: 10 V, H_2O_2: 10%, salt concentration (Na_2SO_4): 0.2 M and the initial concentration of MG: 50 mg L^{-1}. Under the optimum conditions, more than 98 % of MG was removed during 10 min. The results obtained in this work, demonstrate MG can be effectively decolorized by electrofenton process with maximum efficiency at a short time.

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Fabrication of a Molecularly Imprinted Polymer Modified Glassy Carbon Electrode for the Electrochemical Determination of Naproxen

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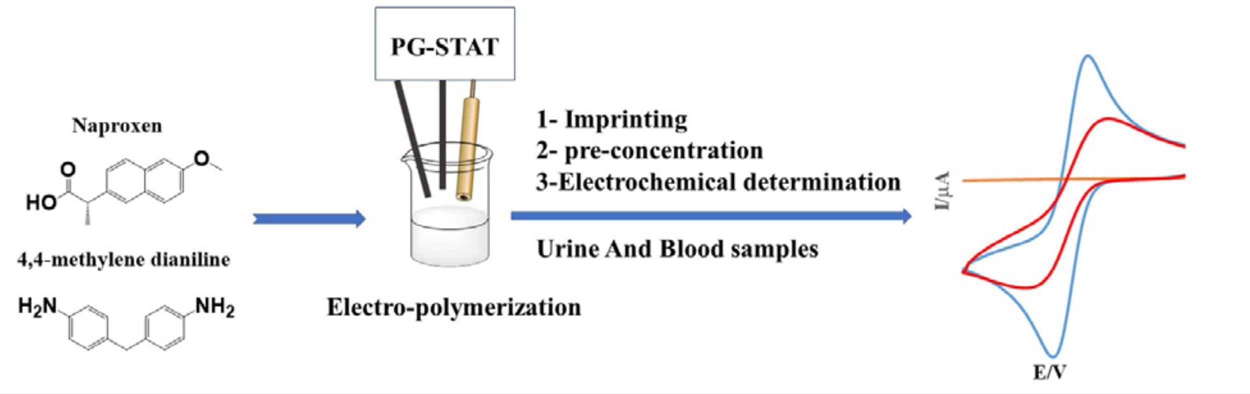
Graphical Abstract



Abstract: An electrochemical sensor based on the electro-polymerization of 4,4-methylene dianiline on a glassy carbon electrode has been proposed for the measurement of naproxen. The morphology of the uncoated glassy carbon electrode and the glassy carbon electrode modified with the imprinting polymer was examined by scanning electron microscopy. Determination of naproxen has been made based on decreasing of current for pair $[Fe(CN)_6]^{3-}$ / $[Fe(CN)_6]^{4-}$ due to the occupation of naproxen complexing sites [1]. Parameters affecting the signal including 4,4-methylene dianiline monomer concentration, number of electro-polymerization cycles, potential scan rate in electro-polymerization process, pH of Electro-polymerization solution, effect of time (pre-concentration step), number of cycles to removing the pattern molecule from the surface of the modified electrode and pH of the measuring medium were optimized. Also, the electrochemical parameters related to differential pulse voltammetry technique including pulse amplitude value, pulse life, and potential scan rate were optimized. Under the optimum condition, the current change was linear relative to the concentration of naproxen. The detection limit, the inter- and intra-day standard deviations of the sensor were found. Finally, the developed sensor was used to measure naproxen in real samples (urine and blood) and the results were satisfactory.

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Microwave assisted synthesis of a nickel-copper composite; promotive effect of melamine on the electrocatalytic activity towards methanol oxidation

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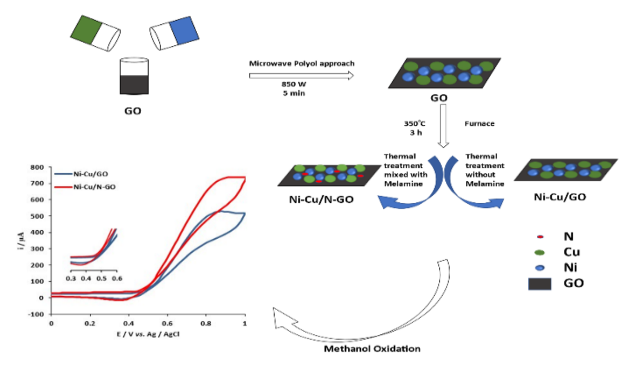

Abstract: Direct methanol fuel cells (DMFCs) are promising power sources for various applications due to their low operating temperatures, relatively quick start-up, and high power density. Methanol oxidation reactions (MOR) are the key anodic reactions in DMFCs [1]. They receive significant attention due to various benefits, including their superior specific energy, economical price, and liquid physical state that unambiguously determine the system's operational safety [2]. Platinum-based nanomaterials have been used primarily as anodic electrocatalysts for methanol oxidation. However, the high cost, slow kinetics, and low stability of platinum-based electrocatalysts severely hinder the further commercialization of fuel cells [3]. Melamine is a nitrogen source for N doping in the Graphene oxide (GO) structure. As an electrocatalyst, the Ni-Cu/N-GO hybrid displays excellent catalytic activities for the MOR with good stability. The synergic effects of metallic Ni and Cu, Nickel and Copper oxides and Ni-Cu/N-doped GO may enhance the performance of the multi-functional hybrid catalyst. In this work, we developed a new pot synthesis strategy for preparing Ni-Cu / N-GO nanocomposites by microwave-assisted process and studied their electrochemical performance. The synthesis method is efficient and fast because microwave heating provides enough heat needed for the reaction in a short time [4]. Literature reports show that nickel-based catalysts are good choices for this reaction due to their high conductivity and excellent electrocatalytic activity, and copper due to their high electrochemical stability and high resistance to poisoning [3, 5]. Microwave irradiation heating became more attractive for forming catalysts for fuel cells. There are many advantages of using microwave heating instead of traditional heating, such as shorter reaction time, high efficiency, product purity, low cost, reduced hazardous substances, nanostructures with smaller sizes and narrower size distributions which is very important nowadays [2]. The effect of melamine on the electrocatalytic activity of a bimetallic catalyst supported on a graphene oxide is synthesized using a rapid microwave-polyol combined with a thermal treatment as a convenient and controllable technique. The synthesized catalyst is studied by cyclic voltammetry (CV), chronoamperometric (CA), and electrochemical impedance spectroscopy (EIS) methods. The promotive effect of melamine is proved by a 203 µA increase in the current and a 0.03 V decrease in onset potential during MOR. Electrochemical studies for fuel cell applications show that the prepared electrode materials show a good performance, which can be attributed to the excellent dispersion of metal nanoparticles or metal oxides on GO and their synergistic interaction with GO support.

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Nickel-based one-pot hydrothermal synthesis with organic-metal framework for OER / HER bi-functional system

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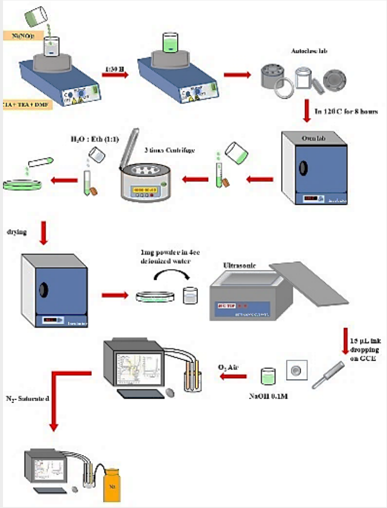
Graphical Abstract



Abstract: Many researches have recently been reported on metal-organic frameworks in various fields. Metal-organic frameworks (MOFs) are composed of central metal ions and ligands. They have easily adjustable structural chemistry and selective metal ions as distributed active sites. Recent studies have shown that MOF can provide a desirable improvement in catalytic performance as a promising precursor to the synthetic process of non-noble metal catalysts. In addition, recent research on nickel-based MOFs as electrocatalysts has been widely discussed, suggesting that Ni-based MOFs can react with oxygen. However, finding a higher activity for a MOF catalyst that could be better than a Pt / C catalyst is still challenging [1-3]. Currently, most fuel cells use pure oxygen or air oxygen as the oxidizing agent. As an example of a natural fuel cell, consider a hydrogen-oxygen fuel cell with an aqueous alkaline or acid electrolyte and its specific properties. The reactants are gaseous in a hydrogen-oxygen fuel cell with a liquid electrolyte. Under these conditions, porous gas-emitting composites are used in cells, where metal-organic template composites use suitable porosity to absorb gases. In the present work, nickel nitrate in nitrogen-doped mesoporous carbon (NMPC) [4], obtained from citric acid (CA) and triethylamine (TEA), in a liquid fuel cell (0.1M NaOH as a liquid electrolyte) in the anodic area for oxygen evolution reaction (OER) and cathodic area for hydrogen evolution reaction (HER) in linear sweep voltammetry (LSV) for an excellent HER / OER bi-functional system [5] have been reported , which have shown perfect performance. The starting potential point for OER 1.436V and HER 0.0066V has been observed that the optimum performance of this bi-functional system is comparable to that of synthesized electrocatalysts so far. Furthermore, for characterization, FT-IR and XRD analysis were used to better recognize the composite and FESEM for observed the morphology and size of the synthesized particles.

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Metal-Organic Framework Derived Co_3O_4 as a an Efficient Bifunctional Electrocatalyst for the Overall Water Splitting Reaction

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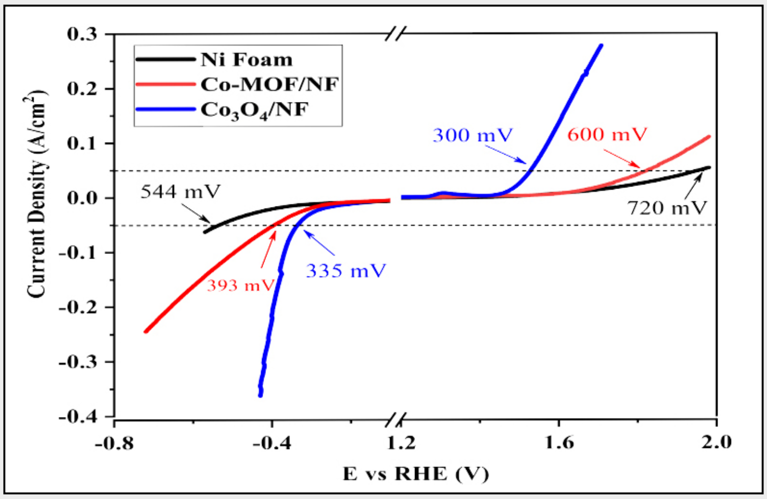
Graphical Abstract



Abstract: With the gradual depletion of conventional energy resources, including petroleum and natural gas, exploring renewable energy sources has become the spotlight of attention over the past few decades [1]. Among the renewable energy sources, molecular hydrogen is a promising candidate due to its high heat value and zero-carbon emission [1]. Electrolysis of water has been considered a sustainable and environmentally friendly technique for the green production of hydrogen fuel [1]. However, it is hindered by the sluggish kinetics and high overpotentials of the corresponding half-reactions, namely, the hydrogen evolution reaction (HER) and the oxygen evolution reaction (OER). Therefore, developing low-cost and high-efficiency electrocatalysts to boost water splitting performance is a priority. In recent years, low-cost Co-based materials have been extensively investigated as alternatives to the conventional noble metal-based catalysts for electrochemical water splitting [2]. Metal-organic frameworks (MOFs), a comprehensive novel class of crystalline and nanoporous materials, are constructed utilizing metal ions and multifunctional organic ligands. MOFs have been widely used as ideal templates or precursors to make metal oxides with various morphologies [3]. In the present study, we report a facile and convenient method to synthesize MOF-derived Co_3O_4 as an efficient electrocatalyst for the overall water splitting reaction. The electrocatalytic performance of Co_3O_4 on Ni foam (Co_3O_4/NF) was far superior to the Co-MOF/NF. Co_3O_4/NF exhibited low overpotentials of 300 and 335 mV to deliver the current density of 50 mA/cm^2 in OER and HER, respectively, along with excellent long-term electrochemical stability. Moreover, it displayed small Tafel slopes of 77 and 67 mV/decade in OER and HER, respectively, indicating the fast reaction kinetics. In summary, this work provides new insights for designing efficient MOF-based bifunctional electrocatalysts for the overall water splitting reaction.

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Electrocatalytic oxidation of ethanol on Pt-Co structure designed bySilica template as a morphology−controlling agent

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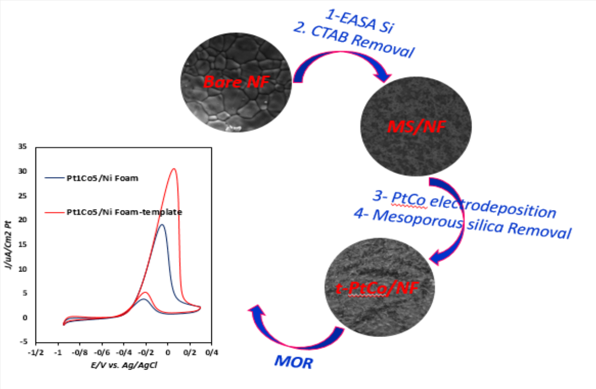

Abstract: Among various types of fuel cells, direct methanol fuel cells (DMFCs) have attracted much attentions due to their interesting properties such as low operating temperature, high power density and relatively quick start-up. Pt is recognized as the best monometallic electrocatalysts for methanol electrooxidation. However scarcity, too pricey, and serious poisoning of Pt make demands for improvement of Pt efficiency [1]. In this regard, incorporation of a second element and examination of the morphology (size and shape of Pt) are identified as the best approaches. The synthesis of nanostructures through templating method presents porous structure with high surface area which will undoubtedly have highly beneficial for catalyzing of methanol oxidation reaction (MOR). Since the exploration of porous silica materials and their effects on the production of mesoporous structures with monotonous distribution, and high surface area, more attentions have been devoted to the using of the mesoporous silica as the sacrificial templates. The synthesis of many nanostructures with different morphology such as nanowires, nanotubes, and so on have been reported [2]. Besides, bimetallic Pt-based catalysts possess good tolerance to CO poisoning as compared with pure Pt. As the excellent effect of Co on electrocatalytic performance of Pt has been discovered, the using of Co as the contributor transition metal not only promotes the MOR activity but also decreases high cost of anode materials [3]. Considering the superb effects of composition and morphology, we prepared a templated structure of Pt-Co supported on Ni foam for electrocatalytic oxidation of methanol in alkaline media. The electrochemical studies including cyclic voltammetry (CV), chronoamperometry (CA), and electrochemical impedance spectroscopy (EIS) confirmed the utility of templating method and elemental composition for electrocatalytic activity towards MOR.

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Electrodeposition of PtNi nanoparticles on a polymer substrate to investigate the electrocatalytic reduction of oxygen in the alkaline media

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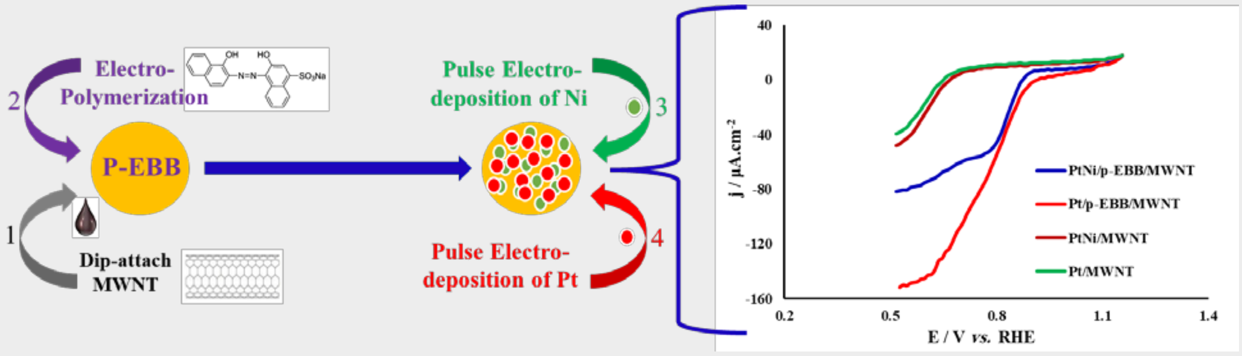
Graphical Abstract



Abstract: Polymeric backing materials for catalysts in oxygen reduction reaction (ORR) are very important. In this work, the glassy carbon electrode was coated with a film of polymeric Eriochrome black B after modification with carbon nanotubes [1]. Metallic nanoparticles including nickel and platinum were electrodeposited on the polymeric substrate by applying a constant potential through the chronoamperometry and pulses methods. Next, the electrocatalytic reductions of oxygen in alkaline medium on the surface of the modified electrodes were investigated [2]. Electrochemical studies including cyclic voltammetry and linear scanning voltammetry showed that the pulse method dramatically improves the performance of Pt nanoparticles for the oxygen reduction reaction. This improvement was confirmed by examining the onset potential and current density during oxygen reduction [3]. In order to investigate the charge transfer resistance at the modified electrode surface, the electrochemical impedance spectroscopy study was performed to compare the behavior of the catalysts in terms of charge transfer resistance.

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Electrochemical determination of dopamine based on derived CuMOF and reduced graphene oxide nancomposite

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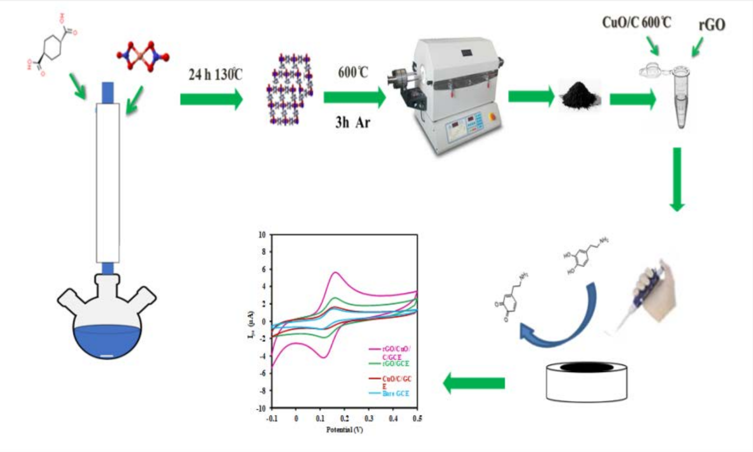
Graphical Abstract



Abstract: Dopamine (DA) is a neurotransmitter that exists in the central nervous system of mammalians and regulates human cognition and emotions. In addition, it plays a significant role in cardiovascular, memory and hormonal systems. The normal level of dopamine in human serum and blood is 1-0.001 µM. The abnormal level of dopamine concentration in the human body is responsible for neurological diseases, such as Parkinson’s disease, schizophrenia, and Alzheimer’s disease. Therefore, the design and fabrication of diagnostic tools for the determination of dopamine is important inside biological systems for proper diagnosis and treatment of associated disorders [1]. At present, there are several methods for determination of DA, such as chemiluminescence, colorimetry, fluorescence and electrochemical methods. The electrochemical detection methods offer high sensitivity, low cost, and excellent selectivity. In recent years, metal─organic frameworks (MOFs) and their derived compounds as inorganic organic hybrid multifunctional materials with fantastic features, such as a changeable skeleton structure, adjustable pore sizes, and exposed metal activity sites, attracted many attentions in the chemical sensor field [2, 3]. However, the poor electron conductivity of MOFs will seriously suppress their electrochemical activity. Up to now, the most common strategy to address the poor conductivity of MOFs is to couple MOFs with conductive materials such as carbon nanotubes, graphene oxide, reduced graphene oxide carbon nanofibers, and so on. Reduced graphene oxide (rGO) has received a great deal of attention due to its high load-bearing mobility, high mechanical stiffness, environmental friendliness and large surface area [4]. In this study, a nanocomposite of CuO/C@RGO was designed as electrochemical sensing platform for the detection of dopamine. The CuO/C nanocomposite was synthesized by a facile approach via the calcination of Cu-based metal organic framework (Cu-MOF). The as-prepared CuO/C/rGO nanocomposite was characterized using scanning electron microscopy (SEM), X-ray diffraction spectroscopy (XRD), Fourier transform infrared spectroscopy (FTIR), energy-dispersive X-ray spectrum (EDS). The Effect of several parameters such as pH, scan rate were investigated. Under optimal conditions, an excellent linearity range was obtained from 0.1 to 200 µM. Finally, the CuO/C/rGO Nanocomposite reveals an excellent DA recovery rate through real samples (urine, serum samples) analyses.

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Glassy carbon electrode modification with a molecularly imprinted polymer by electropolymerization method for preparation of a valsartan sensor

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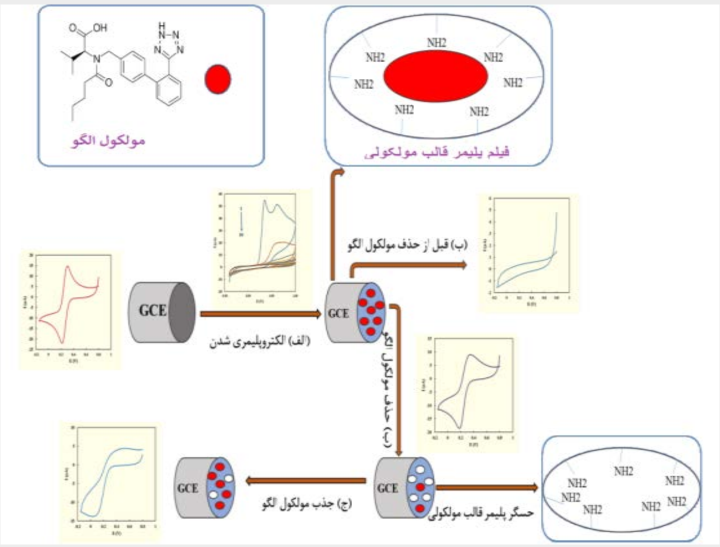
Graphical Abstract



Abstract: In this study, an electrochemical sensor based on the electropolymerization of orthophenylene diamine (O-PD) on a glassy carbon electrode (GCE) for measuring valsartan was proposed. The measurement is based on the reduction of the oxidation-reduction pair $[Fe(CN)_6]^{3-}$ /$[Fe(CN)_6]^{4-}$ due to the occupation of the Valsartan complexing sites [1]. Under optimal conditions (buffer solution with pH 5, extraction time 10 minutes, valsartan concentration 1 mM, monomer concentration 5 mM, scan speed 50mV/s, number of polymerization 10, number of engraved scans 50 and duration of drug application is 20 minutes). Using cyclic voltammetry and differential pulse voltammetry, the change in current was obtained linearly with a concentration of valsartan in the range of 0.1 to 10 mg/L and a detection limit of 0.001 mg/L. The effect of disturbance for the proposed sensor was studied by determining valsartan in solutions containing valsartan / amylodepine, valsartan / ascorbic acid, valsartan / urea, valsartan / glucose. The construction of this sensor is very simple and cost-effective, and the detection limit obtained is much lower than other methods.

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Fabrication of high-performance supercapacitor based on Co_3O_4 nanostructures

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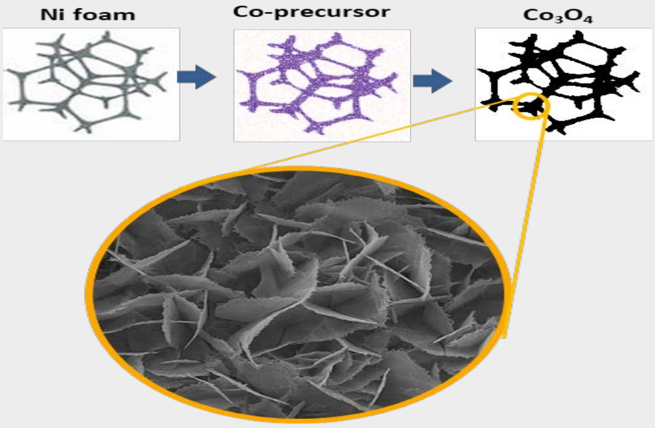
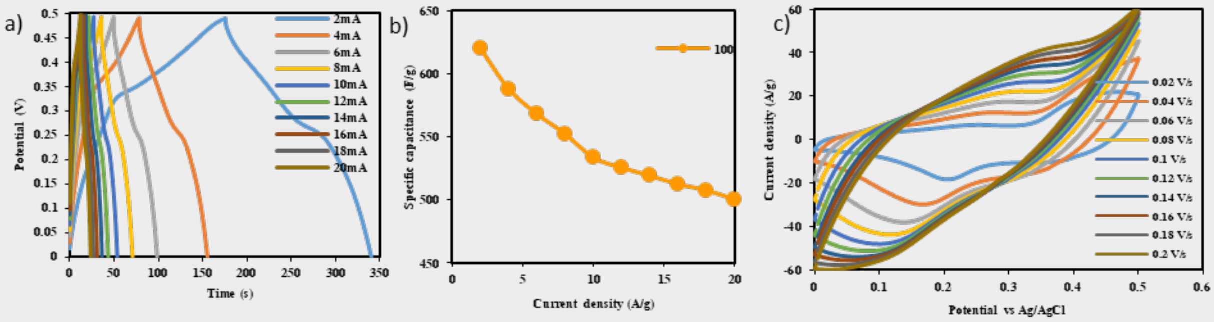
Graphical Abstract





Abstract: In this work, Co_3O_4 nanostructures were hydrothermally grown on Ni foam for the fabrication of high-performance cathode materials of supercapacitor. Co_3O_4 nanostructures with different morphologies were grown by adjusting the amounts of urea, NH_4F at various temperatures. The specific capacitance of the prepared Co_3O_4 electrode reached 620 Fg^{-1} at the applied current density of 2 Ag^{-1} in 1 M KOH aqueous electrolyte. Also, good rate performance of 500 Fg^{-1} at the current density of 10 Ag^{-1} with the retention rate of 80.6%. Electrochemical performance measurements in three-electrode system: (a) GCD curves of the Co_3O_4/Ni, (b) specific capacitances at different current densities from 2 to 10 Ag^{-1}, (c) CV curves of the Co_3O_4/Ni electrode at different scan rates of 20 to 200 mVs^{-1}.

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Electrochemical investigation of drug interaction of mesalazine with captopril in aqueous solution

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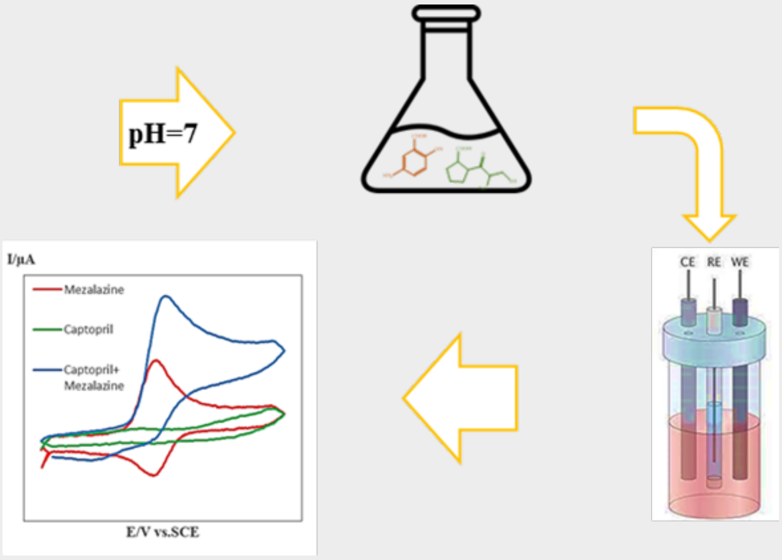
Graphical Abstract



Abstract: Simultaneous oral intake of herbs, supplements, foods and drugs with other drug(s) may result in pharmacokinetic or pharmacodynamic interactions. Drugs that are taken simultaneously by a patient can already cause drug-drug interactions. Drug-drug interactions is one of the most common causes of adverse drug reactions, but these interactions may also be responsible for an increase in clinical efficacy. Mesalazine is an anti-inflammatory drug used to treat chronic inflammatory bowel disease. The control and treatment of chronic inflammatory bowel disease are long-term and may last for years so it may be used concomitantly with other such as blood pressure and blood sugar medications [1]. Given that captopril is used to treat high blood pressure, and inflammatory diseases and to regulate the immune system. It is also an affordable drug for all age groups, including the elderly. It is possible for mesalazine to be taken concomitantly with captopril [2]. In this study, the electrochemical behavior of mesalazine in the presence of captopril was investigated using cyclic voltammetry and controlled-potential coulometry techniques in an aqueous buffer solution (pH~7). The results indicated that the peak structures and currents have changed and captopril has increased the mesalazine current value in cyclic voltammetry. Corresponding products were electrochemically synthesized in aqueous solutions using a carbon electrode in a divided cell. These products were identified by spectroscopic methods (FT-IR, ^{1}HNMR, and^{13}CNMR).

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Electrochemical investigation of drug interaction of epinephrine with captopril in aqueous solution

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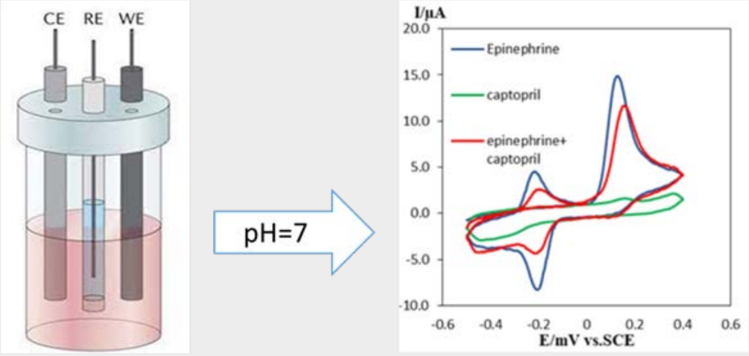
Graphical Abstract



Abstract: Drug-drug interaction are the most common type of drug interaction. These types of drug interaction can increase minor or serious unexpected side effects, may decrease performance of medications, or the most important one is increasing the blood level and possible toxicity of a certain drug. In recent years, studies in this field by using different techniques have become more important. Epinephrine is a hormone that is naturally released when a person experiences high excitement and stress, and also it is a medication which is used in emergency medical treatment for increasing blood pressure [1]. Due to that the disease of hypertension and being in a stressful situation is very common in today societies. Captopril is a drug that is now used in all age groups to treat high blood pressure as an available and affordable drug [2]. In this study, the electrochemical behavior of epinephrine in the presence of captopril was investigated using cyclic voltammetry and controlled-potential coulometry techniques in an aqueous buffer solution (pH~7). The results indicated that the peak structures and currents have changed and captopril has decreased the Epinephrine current value in cyclic voltammetry. Corresponding products were electrochemically synthesized in aqueous solutions using a carbon electrode in a divided cell. These products were identified by spectroscopic methods (FT-IR, ^{1}HNMR, and^{13}CNMR).

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Electrochemical Investigation of the Interactions of Hydroxychloroquine and Captopril in the Treatment of Patients with Covid-19

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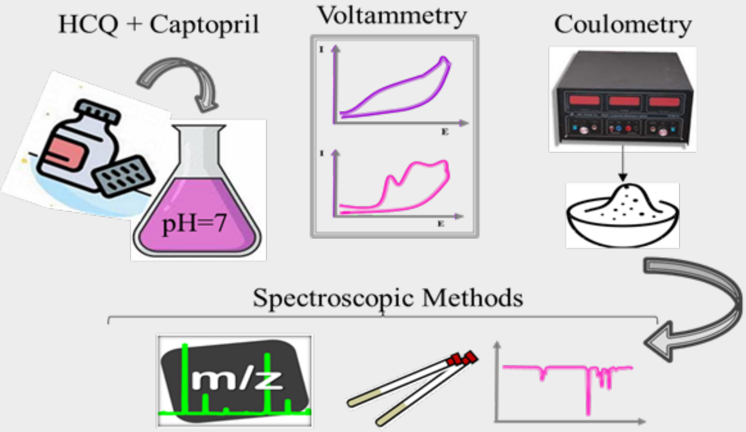
Graphical Abstract



Abstract: Drug interactions occur when a drug's mechanism of action is disturbed by the concomitant administration substances such as foods, beverages or other drugs. Despite the diminishing developments of COVID-19, there is no drug still approved to have significant effects with no side effect on the treatment for COVID-19 patients. Based on the evidence, many antiviral and anti-inflammatory drugs have been authorized by the Food and Drug Administration (FDA) to treat the COVID-19 patients even though not knowing the possible drug-drug interactions [1]. In coronavirus disease, hypertension and cardiovascular diseases are major risk factors for critical disease progression [2]. Nowadays Concomitant use of chloroquine (CQ) and hydroxychloroquine (HCQ) derivative (used for inflammatory diseases as a potential treatment in patients with Quid 19) and captopril (for the treatment of hypertension) is very common. In this research the electrochemical behavior of HCQ in the presence of captopril was investigated using cyclic voltammetry and controlled-potential coulometry techniques in aqueous buffer solution (pH~7). The results indicated that the peak structures and currents have changed and captopril has reduced the HCQ current value in cyclic voltammetry. The type of interaction, possible reactions and proposed mechanisms were determined using the coulometric method. In order to ensure drug interaction, the synthesized products after separation identified by spectroscopic methods (FT-IR, 1HNMR, 13CNMR and MS). The observed homogeneous rate constants (kobs) were estimated by comparison of the experimental cyclic voltammetric responses with the digital simulated results.

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Electrochemical Determination of Amlodipine Using $GdTb(WO_4)_3$ Nanoparticles Modified Carbon-Paste Electrode

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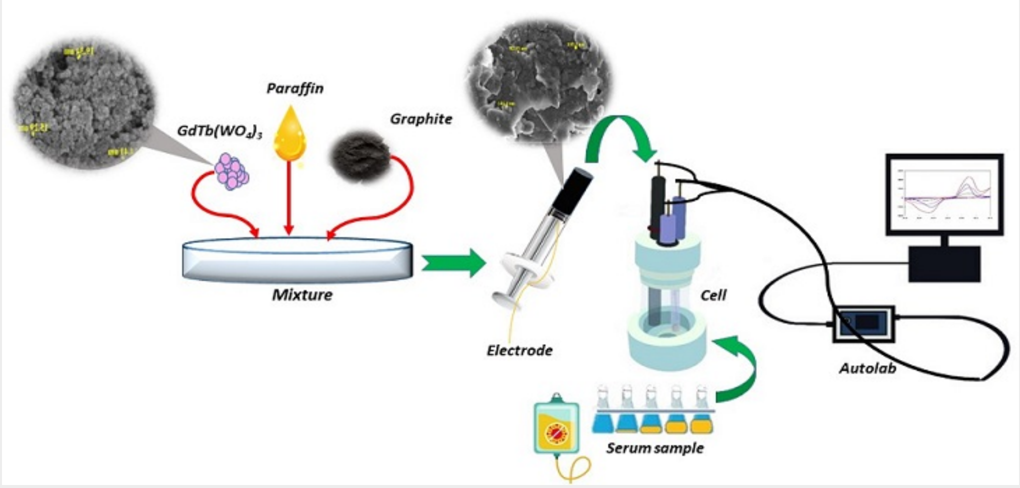
Graphical Abstract



Abstract: Amlodipine, as a calcium channel blocker, is used to treat coronary artery illnesses, high blood pressure, angina, and chest pain [1]. However, overdosages of amlodipine can lead to shock, hypotension, dyspepsia, kidney and liver damage, and increase the risk of heart attack [2]. Therefore, a sensitive platform is required for monitoring amlodipine in biological and pharmacological matrixes. In this regard, the main objective of the present investigation is development of a novel, sensitive, and simple electrochemical method for the online monitoring of amlodipine in human plasma and urine samples. For this purpose a carbon paste electrode modified with $GdTb(WO_4)_3$ nanoparticles and differential pulse voltammetry (DPV) and cyclic voltammetry (CV) were applied. Based on the obtained electrochemical results from, the designed electrode showed an excellent sensitivity and enhanced oxidation peak current of amlodipine. In comparison with other techniques, electrochemical methods show some important advantages such as short response-time, cost-effectiveness and easy operation, which make them suitable analytical methods for detecting trace levels of a target molecule [3]. The proposed nanomaterials, synthesized through the precipitation method, and the modification of electrode were characterized morphologically and structurally by Field Emission Scanning Electron Microscopy (FESEM), High Resolution Transmission Electron Microscopy (HR-TEM), zeta potential analysis, and Fourier Transform Infrared spectroscopy (FTIR). Analytical variables such as $GdTb(WO_4)_3$ volumes in electrode, scan rates, and pH values were also optimized. Under optimal conditions, the designed electrochemical method was applied for the determination of different concentrations of amlodipine in plasma and urine samples. A linear range covering from 0.2 to 3.5 μM along with a detection limit of 10 nM and the relative standard deviation (RSD) of 4.12% was successfully obtained.

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Superior Electrocatalyst Based on Mesoporous Silica Nanoparticles/Carbon Nanotubes Modified by Platinum-Copper Bimetallic Nanoparticles for Amperometric Detection of Hydrazine

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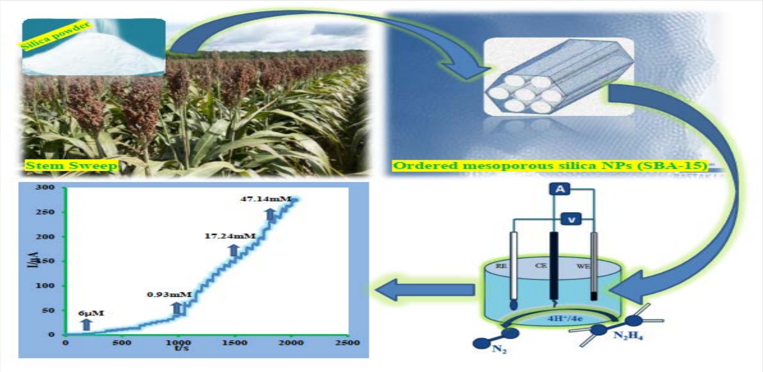
Graphical Abstract



Abstract: Hydrazine is a volatile, water-soluble molecule with strong reducing ability that is widely used in various industrial and pharmaceutical fields. It is known as a combination of carcinogens and neurotoxins that have harmful effects on the liver, brain, nervous system and DNA and also cause blood disorders. Therefore, developing a sensitive, accurate, simple and fast way to measure hydrazine concentration is very important. Here, a novel and efficient sensor for the determination of hydrazine at trace level is introduced by modifying mesoporous silica nanoparticles/multi-walled carbon nanotubes dispersed in carbon paste electrode with PtCu nanoparticles (PtCu NPs/MSNPs/CNTs/CPE). SBA-15 NPs as one of the MSNPs are stable matrix for bimetallic NPs which prevent their accumulation and provide conditions for low loading of catalyst due to its unique structure. For this purpose, MSNPs was firstly synthesized from stem sweep ash (SSA) as raw material. The suggested sensor indicated favorable analytical performance in 0.1 M phosphate buffer solution (PBS, pH = 7.4) with a low limit of detection of 0.09 mM in the wide linear range of 6 mM17.24 mM, high sensitivity of 47.18 mA mM1, rapid response time of ~3 s and high repeatability/reproducibility. The analytical performance of PtCu NPs/MSNPs/CNTs/CPE originates from the presence of more available bimetallic active sites and the synergistic effect of MWCNTs with high electrical conductivity. In the presence of some usual interference, the sensor indicated high selectivity for hydrazine detection. The practical application of PtCu NPs/MSNPs/CNTs/CPE in real samples was also evaluated. The results showed that the MSNPs with large porosity and high surface area are efficient substrate for catalyst formation and cause to fabrication of sensor with excellent accuracy, precision, and recovery for the determination of hydrazine.

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PdCu Bimetallic Nanoparticles Decorated on Ordered Mesoporous Silica (SBA-15) /MWCNTs as Superior Electrocatalyst for Hydrogen Evolution Reaction

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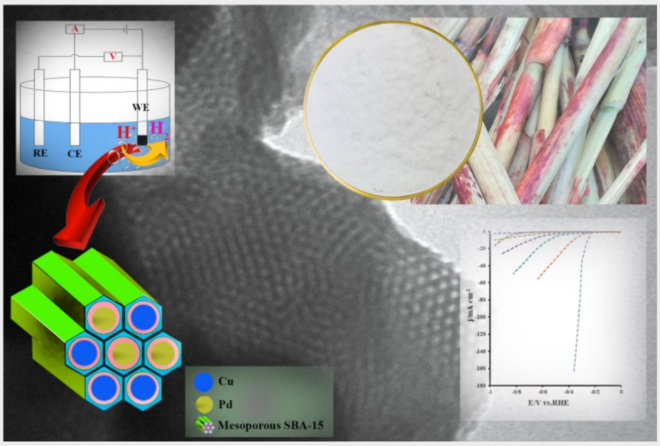
Graphcal Abstract



Abstract: In the present study, a novel electrocatalyst with excellent catalytic performance based on PdCu bimetallic nanoparticles (NPs) supported on ordered mesoporous silica and multiwalled carbon nanotubes (PdCu NPs/SBA-15-MWCNT) was prepared for electrochemical hydrogen evolution reaction (HER). For this purpose, low-cost mesoporous SBA-15 was synthesized using silica extracted from Stem Sweep Ash (SSA) as an economically attractive silica source. Mesoporous SBA-15 with unparalleled porous structure is a stable support for PdCu bimetallic NPs which prevents the accumulation of PdCu bimetallic NPs and improves its efficiency in the catalytic process. The main advantage of this strategy is low loading of bimetallic catalyst with high catalytic activity. The presence of both mesoporous SBA-15 and MWCNTs materials in PdCu/SBA15-MWCNTs/carbon paste electrode (CPE) increases the metallic active sites and the electrical conductivity of electrode which provides great performance for HER. PdCu/SBA15-MWCNTs-CPE provided small Tafel slope (45 mV dec1), low onset potential (~-150 mV), high current density (165.24 mA cm2at -360 mV) and exchange current density (2.51 mA cm2) with great durability for HER in H2SO4 solution. Analysis of kinetic data suggests that the electrocatalyst controls HER by the Volmer-Heyrovsky mechanism. In addition, studies showed that the presence of sodium dodecyl sulfate (SDS) in electrolyte can decrease the potential of HER and increase the current density.

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Electrophoretic Deposition of Graphene Oxide on screen-imprinted Carbon Electrode and its Modified using Tl^{3+}-imprinted polymer as Ionophere for Determination of Thallium (III) ions by Potentiometric Sensor

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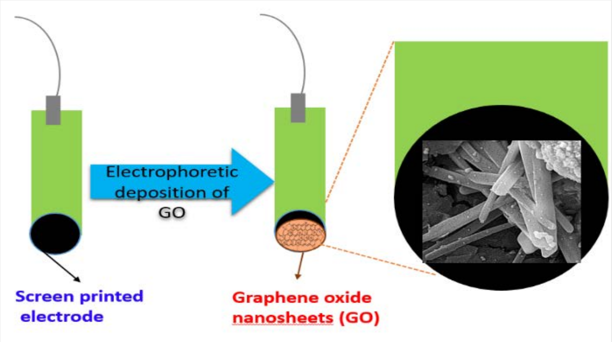
Graphical Abstract



Abstract: A heavy and metallic element in the environment is Thallium. The most dangerous features of Thallium are damage to testicular tissue, liver, peripheral nerves, kidney intestine, hair loss, and even it's suspected for a human carcinogen. Thallium is present in nature as Tl (I) and Tl (III) ions, and each Thallium oxidation state has its bioavailability and toxicity property. Universally, the concentration of Thallium in the aqueous solution is very low, so the analytical methods are required to determine it in the environment [1]. In the electrochemical methods, the construction of effective electrode is principle challenge. The success of an electrochemical method depending on the successful choice of electrodes. Recently, screen-printed electrode (SPE) was applied for determining metal ions. This electrodes have been miniaturize and low cost [2]. The SPE surface was modified with different materials such as nanoparticles and surfactant for enhance the electrochemical performance. Graphene oxide is an important nanoparticle among the new nanoparticles. GO has suitable properties such as conductivity and great surface area. A screen-printed carbon electrode system for the determination of Thallium (III) was introduced using screen-printing technology. An electrophoretic deposition (EPD) method has been used for the deposition graphen oxide nanosheets onto the surface of SPE. This research study to determining Thallium (III) ions have described a novel ion-selective electrode according to Thallium (III)-imprinted polymer particles dispersed in 2-nitrophenyloctyl ether (NPOE) and embedded in a polyvinylchloride (PVC) matrix. The Tl (III)-ion selective electrode showed a nernstian response for thallium (III) ions over the dynamic concentration range of 0.49 to 20.5 µM, with a slope of 19.6 mV per decade. The response time was determined to be about 20 seconds. One of the significant points is the selectivity coefficient of the prepared ion-selective electrode. The selectivity of this electrode for other ions is less than 1.1× 10-3 and this indicates the selectivity of the electrode to thallium (III) ions and other ions are less disturbing.The desired electrode showed good selectivity over a wide range of ions. The accuracy of the suggested electrode was investigated in spiked tap water.

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Enhanced electrocatalytic behavior of melamine by its co-polymerization for oxidation of methanol

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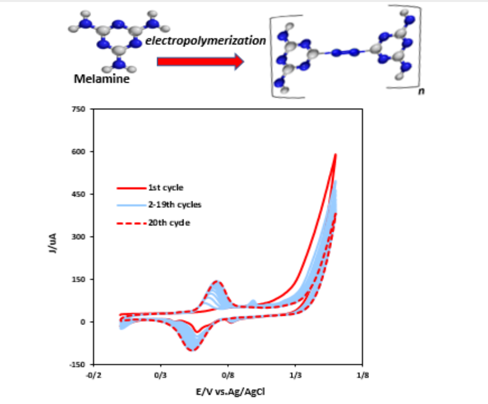
Graphical Abstract



Abstract: Among various types of fuel cells, direct methanol fuel cells (DMFCs) have attracted much attentions due to their interesting properties such as low operating temperature, high power density and relatively quick start-up. Pt is recognized as the best monometallic electrocatalysts for methanol electrooxidation. However, scarcity, too pricey, and serious poisoning of Pt make demands for improvement of Pt efficiency. In this regard, supporting materials not only promote the charge transfer rate and improve the efficiency of the catalyst but also decrease the loss of catalyst owing to the strong interaction with the catalyst nanoparticles [1]. Recently, the use of conducting polymers (CPs) as the supporting materials due to high conductivity, stability, and activity remarkably improve the activity, speed, and over potential of reactions catalyzed on the polymeric supported metallic nanoparticles [2]. Different factors can be affected the efficiency of polymeric substrate. For example, the conductive polymers based on melamine (1,3,5-triazine-2,4,6-triamine) possessing the amine groups significantly promote the immobilization and activity of catalytic nanoparticles. In addition, copolymerization technique that simultaneously polymerizes different monomers and achieves intended properties can enhance the recognition ability and stability of polymer films [3]. If the monomers can be electropolymerized under similar condition, copolymers can also be prepared by facile electrochemical methods. In these realities, we use the catalytic nanoparticles doped on a co-polymer based on melamine for electrocatalytic oxidation of methanol in alkaline media. The formation of co-polymer film on the surface of a glassy carbon electrode is performed by consecutive cyclic voltammetry (CV) in an acidic solution of two monomer. The effective parameters on the electropolymerization process are studied. The metallic nanoparticles supported on the optimized co-polymer exhibits the excellent activity of methanol oxidation reaction (MOR) in alkaline media.

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Design and fabrication of electrochemical sensor for determination of thyroxine

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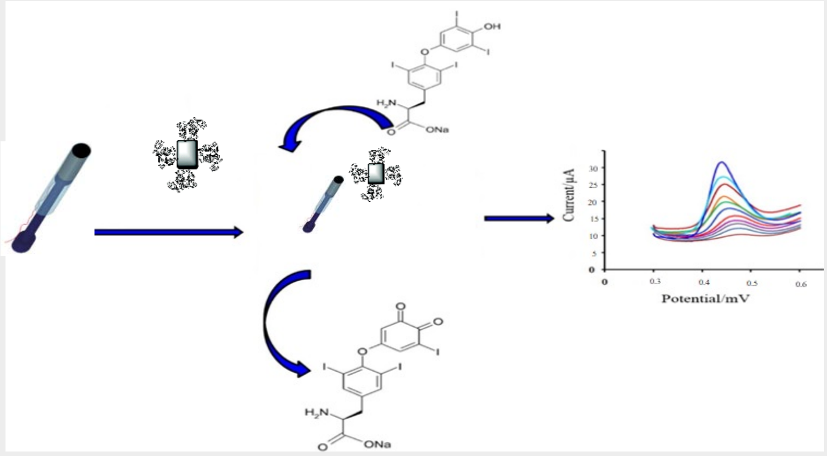
Graphical Abstract



Abstract: Thyroid hormones including thyroxine (T_4) and triiodothyronine (T_3) are hormones derived from the amino acid tyrosine that are produced and secreted by thyroid gland. The main form of thyroid hormone (about 95%) secreted in the blood is T_4. Despite the conversion of T_4 into T_3 in the surrounding tissues, the amount of T_4 in the blood is still much higher than T_3 [1]. Generally measuring T_3 is not vital. At the beginning of hypothyroidism, T_4 decreases, but because T_4 is converted to T_3 in the surrounding tissues, the amount of T_3 does not decrease and most of T_4 molecules are converted to T_3. Therefore, despite the fact that there is an inverse relationship between T_4 and TSH in these conditions, there is not much change in the amount of T_3. Consequently, its measurement does not help in diagnosis. Considering the biological importance of T_4 hormone, it is very important to determine it to diagnose hyperthyroidism or hypothyroidism [2-4].

Since T_4 is an electroactive material, its electrochemical behavior at an AgGO-Chit modified carbon paste electrode was investigated in this work. Thyroxine underwent totally irreversible oxidation in this system and a well-defined peak at 0.42V was observed. The influence of various nanoparticles on the oxidation of thyroxine was examined through cyclic voltammetry. In the range of 10^{-9} to 10^{-4} mol/L, with the oxidation peak current was linear with the thyroxine concentration.

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Multivariate Curve Resolution-Alternating Least Squares (MCR-ALS) and Central Composite Experimental Design For Monitoring and Optimization of Simultaneous Removal Of Some Organic Dyes By Synthesized Composite Active Carbon@$CoFe_2O_4$

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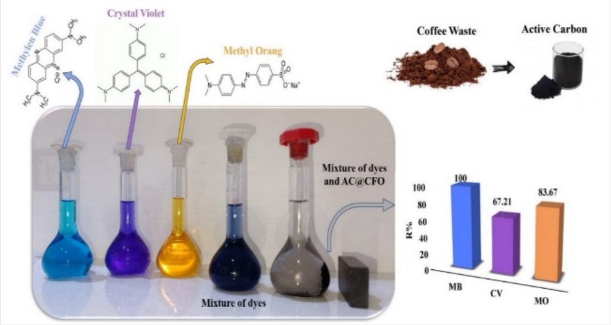
Graphical Abstract



Abstract: Water pollution by pollutants released from various industries is one of the environmental concerns, However, adsorption of pollutants is a promising option for wastewater treatment [1]. The type of adsorbent is the main parameter in this process and also the actual wastewater is usually a mixture of different pollutants [2]. Therefore, the overall purpose of this work is to synthesize activated carbon@cobalt ferrite (AC@CFO) nano adsorbent from coffee waste in two pyrolysis and co-precipitation steps and evaluate its performance in simultaneous removal of a mixture of anionic and cationic dyes including Methylen Blue (MB), Crystal Violet (CV), and Methyl Orang (MO). AC@CFO Characterized by Brunauer Emmett Teller (BET), Vibrating Sample Magnetometer (VSM), and X-Ray Diffraction Pattern (XRD). BET showed that the synthesized adsorbent has a higher adsorption capacity than commercial AC adsorben, VSM showed good magnetic strength to separate from aqueous solution, and XRD showed that CFO nanoparticles were crystallized in the spinel structure. The effect of three parameters (dose adsorb, concentration dyes, and initial pH) and their possible interaction in the simultaneous removal of mentioned dyes were studied and optimized using experimental design and response surface method [3]. The values of the regression coefficient for MB, CV, and MO were determined as 98.48, 97.39 and 98.67 percent, respectively, revealing the reliability of the obtained polynomial models to predict removal efficiencies. In this work, multivariate curve resolutionalternating least squares (MCR-ALS) has been applied to resolve and study the simultaneous removal of three organic dyes using adsorbtion reaction. Maximum dyes removal efficiencies were 100%, 67.21% and 83.11% for MB, CV, and MO respectively at optimum conditions. According to the results, the AC@CFO has great potential for the removal of various dyes.

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Genetic algorithm using DD-SIMCA one class through FT-IR spectroscopy to classification of Listeria samples

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Graphical Abstract

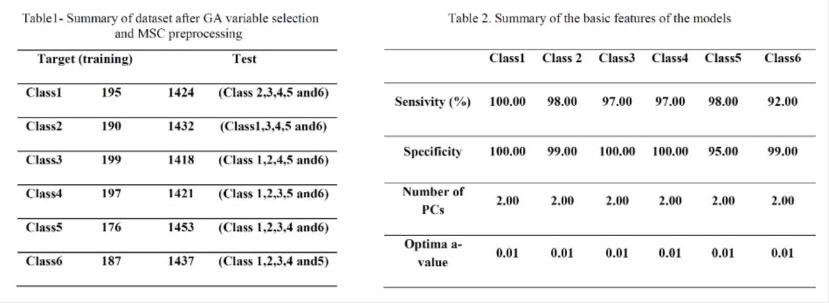

Table1- Summary of dataset after GA variable selection and MSC preprocessing

Target (training)		Test	
Class1	195	1424	(Class 2,3,4,5 and6)
Class2	190	1432	(Class1,3,4,5 and6)
Class3	199	1418	(Class 1,2,4,5 and6)
Class4	197	1421	(Class 1,2,3,5 and6)
Class5	176	1453	(Class 1,2,3,4 and6)
Class6	187	1437	(Class 1,2,3,4 and5)

Table 2. Summary of the basic features of the models

	Class1	Class 2	Class3	Class4	Class5	Class6
Sensivity (%)	100.00	98.00	97.00	97.00	98.00	92.00
Specificity	100.00	99.00	100.00	100.00	95.00	99.00
Number of PCs	2.00	2.00	2.00	2.00	2.00	2.00
Optima a-value	0.01	0.01	0.01	0.01	0.01	0.01

Abstract: Nutrition-caused listeriosis is one of the most serious and most severe nutrition-transferable diseases that can be considered a threat to human health. Owing to the high resistance of listeria monocytogenes against environmental conditions, existence of this bacterium in raw or processed foods is also possible and the appropriate evaluation and monitoring is necessary actions and regulations from the beginning of the production cycle to the consumption of the nutrients. The diagnosis of pathogens in food safety is essential [1,2].

This study investigated the classification of Listeria samples using IR spectroscopy. The Mid-FTIR spectra were pretreated by baseline corrected, multiplicative scatter correction (MSC) transformation to eliminate the baseline shift deletion and multiplicative effect of scattering and orthogonal signal correction (OSC) which removes unrelated or orthogonal systematic variation from the spectral data. In the next step, from 792 wavelengths, 202 wavelengths were selected by the genetic algorithm (GA) algorithm as a feature selection procedure for DD-SIMCA. The dataset of 1717 samples was split into two subsets as calibration and validation sets through randomly. Table 1 (graphical abstract) is shown summary of dataset after outlier detection, GA variable selection and MSC preprocessing. The results are summarized in Table 2 (graphicalabstract). Therefore, the aim of this research is to introduce an easy, fast, and low cost method to identify listeria based on spectroscopy studies and using chemometrics method. Table 2- Summary of the basic features of the models.

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ATR-FTIR spectroscopy based on PLS-R multivariate calibration technique for assessing transformer oils samples to predict of dielectric dissipation factor (DDF) in power industry

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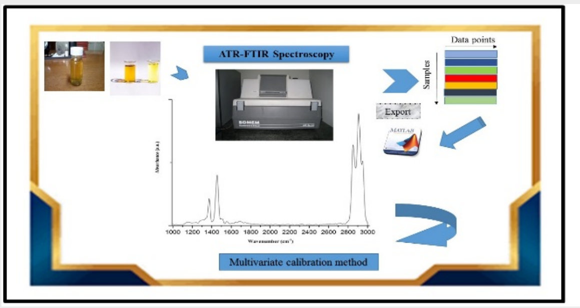
Graphical Abstract



Abstract: Transformer is one of the most applied equipment's in electrical circuit. Transformers change the voltage from one level to another one by using magnetic field effect. It is cooled down by using oil in power industry for reducing current for transforming electrical energy. Increasing the temperature of oil transformer leads to damage and aging of this oil. The most important parameters of transformer oil are about the water content, breakdown voltage, furfural concentration, color, viscosity, surface tension, sulfur content and solid content. Nowadays, the quality of a power transformer is determined by measuring the dielectric dissipation factor (tan-delta) as a primary parameter for evaluating the deterioration of an oil transformer. The dielectric dissipation factor (DDF) of oil samples was determined under the IEC 60247:2004 standard testing method [1].

In this research, attenuated total reflection Fourier-transform infrared (ATR-FTIR) spectroscopy is employed as an analytical technique based on multivariate calibration techniques to determine the DDF for oil samples used in the power industry. The partial least squares regression (PLS-R) as a linear multivariate calibration technique was used to forecast the DDF in oil samples using successive projection algorithm (SPA) variable selection as suitable models. As a result, in the SPA-PLS-R chemometric method, the correlation coefficient (R^2pred) and root mean square prediction error (RMSEP) were 0.9773 and 0.0045, respectively. Thus, using mid-IR spectroscopy, PLS-R combined with SPA can be a practical method for reliable, rapid, and simple quantitative analysis of DDF in the power products industry.

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Robust Regression Method / a Comparison Study

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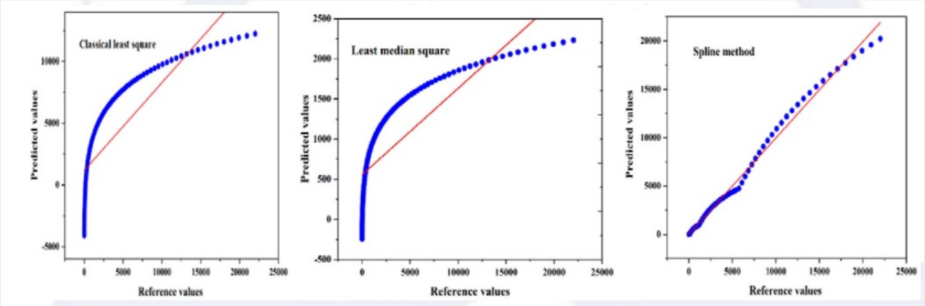
Graphical Abstract



Abstract: The least squares method (LS) is one of the most common methods for estimating the coefficients of linear regression models. However, it is not robust model against the existence of non-linear data. In this study, Classical least squares (CLS), least median squares (LMS) and Spline model (SM) are studied. The CLS regression, which consists of minimizing the sum of the squared residuals assumes among others a normal error distribution. The LMS method as a median-based robust regression method is based on the minimization of the median of the squared residuals.[1] In SM, splines are function estimates obtained by fitting piecewise polynomials and the x range is split into fixed intervals. The intervals are separated by so-called knot locations [2]. In each interval a polynomial is fit with the constraint that at the knot locations the function be continuous. A spline is defined by its degree, by the number of knot locations, by the position of knots and by the coefficient of polynomial fitted at each interval. CLS, LMS and SM are among the studied methods that have R-square 0.706, 0.709 and 0.994 in the data generated according to $y = \exp(\sqrt{x} + e)$ equation, respectively. Graphical abstract shows the Predicted values versus reference values by CLS, LMS and SM methods. It was concluded that the SM algorithm seems to have a better result of data due to the R-square.

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Design and Construction of Cerium Selective Electrode Based on QSPR Model and Experimental Evaluation

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Abstract: The increased use of ion sensors in different fields leads to many intensive studies to introduce sensing materials in the construction of ion-selective electrodes, particularly lanthanide sensors. Since the selectivity of ion-selective sensors, directly depends on the stability constants of ion-ionophore complexes, we predicted the stability constants of cerium ions with different ionophores by the QSPR model, previously [1]. Although the stability constant of the complex between the ion and the ionophore is required to be high enough to produce a noticeable selectivity, this constant must not be so large that ions are tightly bound, making the complexation process kinetically irreversible, so the sensor exhibits long response time [2]. Based on previous studies, the useful range of ionophore complex formation constants in an ISE is usually about 104 –107.

In this work, we designed and synthesized a new ligand as the selectophore, 3-(2, 3-dihydro-2-methylbenzo[d]thiazol-2-yl)-7-hydroxy-2H-chromen-2-one for cerium ion based on the predictive GA-MLR model and compared the theoretical and practical results. At the first, the stability constant of the cerium-selectophore complex was calculated using the proposed MLR model. Then the stability constant of the complex was determined by the conductometric method in acetonitrile solvent and the KINFIT program. The obtained results, 3/91 and 4/26 by theoretically and experimentally method, respectively, were satisfactory. Potential responses of cerium selective electrode based on the ligand as a selectophore was evaluated by varying the amount of ionophore, plasticizer, and ionic additive. The electrode prepared with 4% ionophore, 61% nitrobenzene, 2% NaTPB as an additive, and 33% PVC has shown a Nernstian slope of 19.18 mV per decade to activities of Ce^{3+} ions with a short response time of 10 s. The sensor has shown an appreciable selectivity for Ce^{3+} ions in the presence of other lanthanide ions.

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Analysis of Volatile Components of Ziziphus Spina Christi Leaves Using a Combination of HS-SPME / GC-MS and Multivariate Curve Resolution Methods

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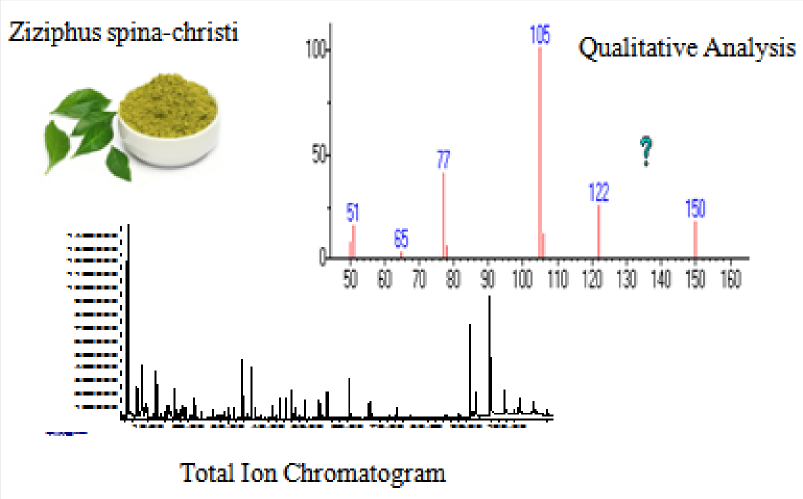
Graphical Abstract



Abstract: The main aim of this work was identification of the volatile components of Ziziphus spina-christi. The leaves of this plant were supplied from Khuzestan province and were dried for preparation of Ziziphus spina-christi extract. Aqueous and ethanolic extracts of this plant were taken and dried. Then, 40 g of aqueous extract, 40g of ethanolic extract and 20 g of the Ziziphus spina-christi powder were mixed and soaked in water to get a sample for volatile extraction. The volatile components of Ziziphus spina-christi were extracted by head space-solid phase micro extraction (HS-SPME) technique. The extracted components of Ziziphus spina-christi were analyzed by using GC-MS method under appropriate condition. The obtained chromatogram was divided into 133 peak clusters for further investigation by the multivariate curve resolution (MCR) method. The MCR was used for more accurate analysis of the chromatogram. After background correction and denoising [1, 2], the number of components of each peak cluster was determined by calculating the morphological score. The peak clusters were resolved using multivariate curve resolution-alternating least square (MCR-ALS) [3, 4] method by imposing proper constraints. The resolved mass spectra were exported to NIST software for qualitative analysis. Using the MCR method, the numbers of identified components in the Ziziphus spina-christi sample are extended from 81 in direct analysis to 155 compounds by GC-MS/MCR method. It is concluded that the combination of hyphenated chromatographic techniques with chemometric resolution methods can provide a complementary method, or in several cases an alternative method for the quick and accurate analysis of real multi-component sample.

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Design of Novel Quinazolines as Potent Anti-Cancer Compounds by Coupling QSAR and Docking Methods

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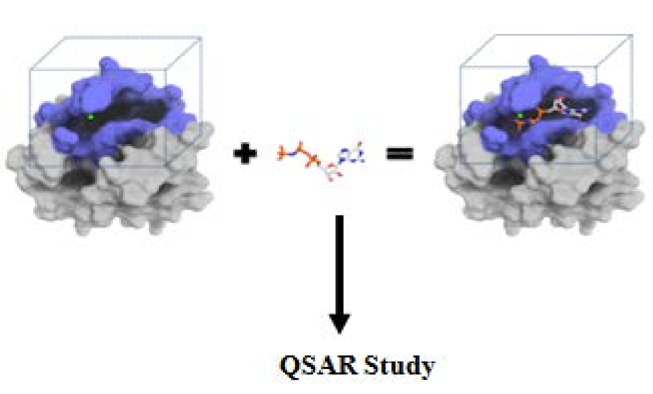

Abstract: The main aim of this work is designing new derivatives of quinazolines that have desirable anti-cancer properties by coupling quantitative structure_activity relationship (QSAR) and molecular docking methodes. Quinazolines are a class of heterocyclic compounds that have unique properties in the field of medicinal chemistry, especially in treatment of cancer. In this study, a data series containing 48 molecules of quinazoline derivatives was selected from several valid sources [1-4]. The interaction of the molecules with the 4WKQ protein was investigated during the molecular docking process and the best conformer of the data set molecules was used for QSAR study. DRAGON software was used to calculate 1497 descriptors for each data set molecules. After evaluation of the calculated descriptors, 366 descriptors were remained for further investigation. The compounds were divided into training (39 molecules) and test (9 molecules) sets, where the training set was used for modeling process and the test set was used to evaluate the models. QSAR models were developed by the stepwise-MLR, GA-ANN, GSA-MLR and GSA-ANN methods. The stepwise-MLR and GA-ANN models were identified as invalid models, because their evaluation results were not satisfactory. The nonlinear GSA-ANN model was recognized as the best obtained model, which includes eight molecular descriptors, GGl6, MATS5p, GATS2p, G(N...Cl), RDF030p, Mor23e, ISH and H5u. The QSAR model exhibited good statistical values for training (R^2_{train}=0.874) and test (R^2_{test}=0.866) sets. Hence, it can claim that the gravitational search algorithm (GSA) is a more powerful descriptor selection method than others [5, 6]. Finally, the model obtained by this method was used to predict the anticancer activity of new candidates of quinazoline compounds.

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Design of New Candidates of Pyrazole Pyrimidinone as Inhibitors of Phosphodiesterase 5 (PDE5) Effective in the Treatment of Alzheimers by Combining QSAR and Docking Methods

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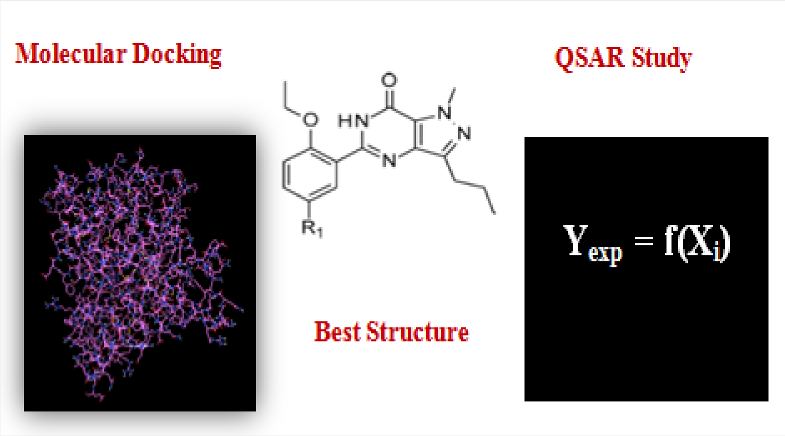
Graphical Abstract



Abstract: Alzheimer's disease (AD), characterized by a progressive impairment of memory and recognition, is a major health problem in both developing and developed countries. Currently, no drugs can reverse the progression of AD. Phosphodiesterase 5 (PDE5) is a critical component of cyclic guanosine monophosphate/protein kinase G(cGMP/PKG) signaling pathway in neurons, the inhibition of which has produced neuroprotective effects, and PDE5 inhibitors have recently been thought to be potential therapeutic agents for AD [1]. In this study, a novel quantitative structure_activity relationship (QSAR) for inhibition activity against PDE5 was performed based on molecularly docked structures of 43 pyrazole pyrimidinone derivatives [2]. Molecular docking study was carried out for finding the best conformer of compounds which have suitable interaction with 1TBF protein. This structure was used in QSAR study. QSAR method can provide adequate information for understanding the role of pyrazole pyrimidinone derivatives for Alzheimer disease. QSAR models were developed by the stepwise-MLR, Stepwise-ANN, GA-ANN, GSA-MLR and GSA-ANN methods. The nonlinear GSA-ANN model was selected as the best model, which includes MATS5e, MATS8p, Mor06e, G3p, ISH and PSA descriptors. In validation process [3, 4], the model exhibited satisfactory statistical parameters (R^2_{train}=0.914, R^2_{test}=0.898, R^2_{LOO}=0.771, R^2_{L5O}=0.772 and R^2_{rand}=0.239). In the next step, a series of pyrazol pyrimidinone derivatives were proposed as effective drugs for Alzheimer disease. Using the GSA-ANN selected model, the activity (IC50) of newly designed compounds were predicted. Among the proposed compounds, five compounds were selected as active compounds and introduced as candidates for new anti-Alzheimers drugs.

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Investigating the production process of dairy products by infrared spectroscopy and chemometrics

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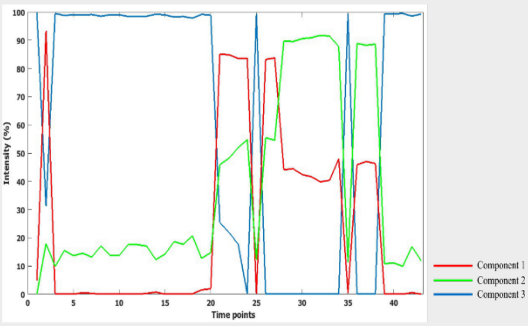
Graphical Abstract



Abstract: The increasing growth and development of the dairy industry requires quality control over the production process of these products. Therefore, improving the quality of these products will lead to savings for manufacturers and customer satisfaction. As a result, using optimal methods of quality evaluation of dairy products with the quality of the final products is recommended. Infrared spectroscopy has been extensively used in dairy products quality [1, 2]. In this study, the main and effective components of the fermentation process during the formation and production of cheese in the initial coagulation of milk and its maturity for 90 days using infrared spectroscopy in near and infrared regions to detect tissue factors Cheeses were examined. Also, infrared spectroscopy has the ability to detect several compounds in a sample and does not require any reaction and is a non-destructive and non-invasive method. The sample tested in this study was prepared during the cheese formation process and in the next stage, spectroscopy was performed. 42 samples were selected by first 30 samples in the initial coagulation of milk and 12 other samples on days 1, 7, 30 and 90 during the cheese ripening period with three repetitions for each sample for spectroscopy. Infrared spectroscopy relies on multivariate analysis methods to quantify and identify the desired components in a compound. Multivariate analysis can simplify complex data and reveal hidden information, and also analysis can be done in both quantitative and qualitative forms, which is based on the science of chemistry. MCR-ALS chemometric technique was used to obtain spectral and concentration profiles.

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Random Subspace Ensemble-Multivariate Curve Resolution-Alternating Least Squares (RSE-MCR-ALS) as a new technique for first-order calibration

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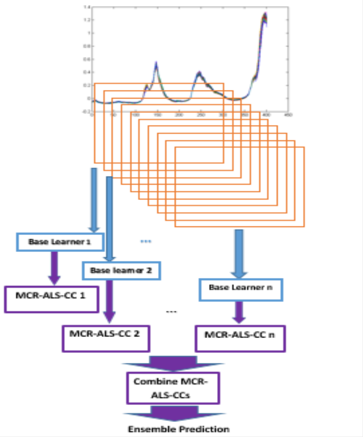
Graphical Abstract



Abstract: One of the most important aspects of any analytical method is quantitation especially using first-order multivariate calibration techniques which allows the simultaneous determination of multiple components in the mixed sample. Multivariate curve resolution-alternating least squares with correlation constraint (MCR-ALS-CC) has played an important role in this area, provided both the qualitative and quantitative determination and prediction of components in complex mixtures [1-3]. Due to this importance, in this study a novel strategy called random subspace ensemble multivariate curve resolution-alternating least square (RSE-MCR-ALS) was proposed in order to improve the proficiency of the conventional MCR-ALS with correlation constraint for calibration purposes. Ensemble regression with purpose of combining several models to make a prediction has been the object of many researches in recent years, considerably improved both the robustness and prediction accuracy of the models [4]. In this regard, three different near infrared (NIR) data sets (including meat (fat), milk (water adulteration), and gasoline (octane numbers)) were chosen to check the feasibility of the proposed method. Two parameters/factors namely the number of base learners, and the number of subspaces, which are the two vital parameters of RSE-MCR-ALS were optimized using central composite design (CCD) and simplex optimization algorithm. The weighted average of root mean square error of prediction (RMSEP) for MCR-ALS-CC was considered as response of the experimental design. Due to the results obtained by analysis of variance (ANOVA) of three NIR data sets, the number of subspaces was not a significant parameter, but the number of base learners was significant. Finally, by considering the optimum number of base learners and subspaces (contains the segments of features in order of their arrival) and applying them on MCR-ALS-CC model and weighted averaging over the RMSEP values of base learners, considerably better prediction results based on RMSEP were obtained (0.07, 0.20, and 0.02 for prediction of fat, water adulterant, and octane number respectively) in comparison to conventional MCR-ALS-CC model (with RMSEP values of 7.74, 9.14, and 9.14 respectively). Moreover, by considering the same procedure on PLSR as the most used first order multivariate calibration technique in analytical chemistry, better prediction results were obtained for RSE-PLSR (0.30, 0.56, and 0.05 respectively). However, these results were still poor in comparison to RSE-MCR-ALS.

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Investigating the Effect of Preprocessing on the Authentication of Extra Virgin Olive Oil using Raman and DD-SIMCA One Class Modeling

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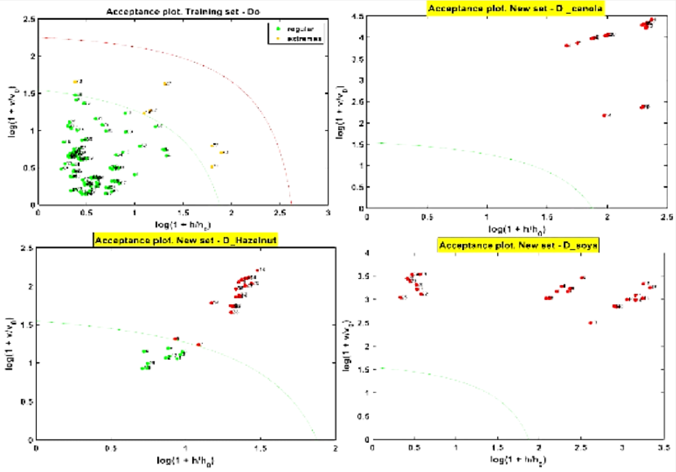
Graphical Abstract



Abstract: Data preprocessing, a component of data preparation, describes any type of processing performed on raw data to prepare it for another data processing procedure. In addition to solving data problems, such as corrupt data or irrelevant or missing attributes in the data sets, one may be interested in learning more about the nature of the data, or changing the structure of data in order to prepare the data for a more efficient analysis. One form of preprocessing the calibration data which is performed in virtually all situations is mean centering [1]. In discriminant analysis, mean centering can reduce the effect of differences in signal intensities. In fact, mean centering closes together the scale of signals in different variables and it can effect on class modeling efficiency. PCA is a linear modeling method which has been commonly used to explore the data sets. By performing PCA, the experimental data matrix D is decomposed into two matrices: scores, containing the information related to objects, and loadings, containing the information related to variables (spectral information). PCA, through feature reduction and visual display, allows us to observe the sources of variation in complex data sets. It is, however, possible to extract much more information from a PCA. The principal components (PCs) are called latent variables. The purest variables can identify by convex hull of the principal component scores. It was shown by removing all other data points; the data set can be reduced to a very sparse set of essential data points [2]. ESPs can be used as feature selection techniques without losing important information of the data set. In this regard, the effect of preprocessing (mean centering, normalization) was investigated on efficiency the data driven soft independent modeling of class analogy (DD-SIMCA) results for authentication extra virgin olive oil samples before and after data reduction with ESPs. In this study by using Raman spectra of pure samples and samples adulterated with hazelnut, sunflower, soybean, and canola oils one class models were developed to evaluate the authenticity and adulteration of extra virgin olive oil before and after preprocessing. Graphical Abstract shows the preliminary results related to the acceptance plots for authenticity and adulterations models.

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Modeling the Optimized Ratio of Fluorophores: A Step Towards Enhancing the Sensitivity of Ratiometric Probes

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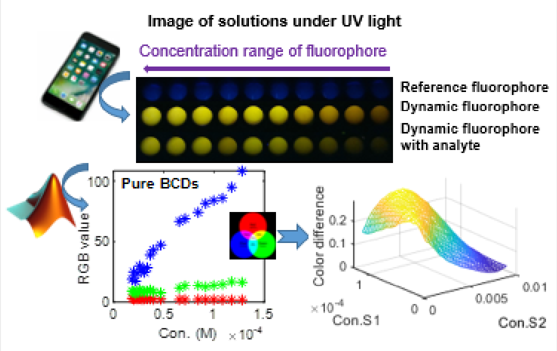
Graphical Abstract



Abstract: In recent decades, ratiometric fluorescent probes have attracted considerable interest due to their potential for visual qualitative/quantitative detection in a range of applications [1]. In ratiometric strategy, mixing two or more fluorophore provides an intermediate emission color. In some designs, some fluorophores are introduced as internal standards, while in others they are analyte-sensitive. Following the interaction of the target with one or even several fluorophores in the probe, the fluorescence emission of the probes changes due to the quenching or enhancing phenomena, altering the emission color of the probe. In order to provide maximum variation in emission color of the probe in the presence of the target, the ratio of the fluorophores is required to be optimized. A number of experiments are required though [2]. Despite a lot of efforts in optimization of fluorophores, it is still remains an elusive task to estimate an accurate and low-cost method to obtain a real optimized condition of mixing fluorophores. The visual monitoring of the targets in ratiometric probes relies on acquiring photos using smartphones, cameras and scanners. The emission intensity and tonality of the probe is concentration-dependent. Thus monitoring the color space of the systems is an appropriate signal enabling quantitative detection of the targets. It seems that modeling the response profile of two-component or multi-component ratiometric probes is a way toward achieving the real-optimal ratio of fluorophores. Herein, in a two-component ratiometric nanoprobe, emission color of the nanoprobe is recorded in the presence of methyl parathion (MP), an organophosphate pesticide, using yellow emissive TGA-capped CdTe QDs (YQDs) and blue emissive carbon dots (CDs). In this regard, the emission profile of the internal standard, herein CDs, in the absence of MP, also the emission color of the dynamic fluorophore, YQDs, in the absence/presence of MP were primarily recorded and modeled using mathematical polynomial equations. Based on the additive color theory, the entire range of emission colors in the color gamut can be predicted by control of mixing ratios of the fluorescent probes [3]. Hence, using fitted equations and additive color theory, the color of binary mixtures of CDs and YQDs in the absence and presence of MP were calculated. Finally, the distance between the color of the fluorophores in the absence and presence of the analyte in the CIE xyY color space was calculated using Euclidean distance method. The maximum distance can be find the real-optimal concentration of the fluorophores.

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Class Modeling Using Multivariate Curve Resolution Methods

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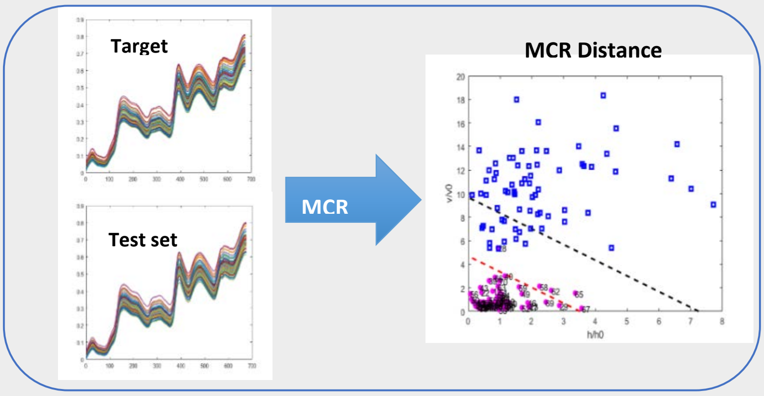
Graphical Abstract



Abstract: Classification or supervised pattern recognition is a name given to a set of numerical techniques developed to solve the class membership problem. An empirical relationship or classification rule is developed from a set of samples for which the property of interest and the measurements are known. The classification rule is then used to predict this property in samples that are not part of the original training set. Multivariate Curve Resolution (MCR) strategies are powerful tools allowing the description, species identification and system understanding, of totally or partly unknown chemical processes and reactions where species cannot be easily isolated and where unknown intermediate species may be present. These methods are also powerful techniques for quantification of complex mixtures. Recently MCR-ALS was used enable discrimination between the constituents of two benchmark and two high-dimensional data sets and was compared with other commonly used techniques [1]. In this work, MCR methods, which already enjoy broad use in various fields, were applied for supervised learning. MCR is a bilinear decomposition approach that supports inclusion of additional system information within the form of numerical constraints. In order to develop an MCR algorithm for supervised learning, the target class of the samples use MCR subspace for two distance measures. They are the score distance (SD) and the orthogonal distance (OD) [2]. MCR method was tested to enable classification between the constituents of data sets. Several real experimental complex data have been used for evaluating the power of MCR methods for classification. For example, Spectra of eighty corn samples (700 wavelengths) with reference value percent moisture, oil, protein, and starch were measured from 1100 to 2498 nm at 2-nm intervals on three NIR instruments designated as M5, Mp5, and Mp6 [3]. The results were compared with the output of the application of different data classification methods like DD-SIMCA and have reliable result. The proposed MCR approach, in comparison with other commonly used supervised techniques, has the advantages of improved accuracy from the inclusion of additional system information in the form of numerical constraints, MCR profiles and subspaces are excellent compressed information that represents adequately the initial information of the data set and, in difference with analogous representations coming from PCA, the information enclosed in the MCR profiles is compound-specific and chemically meaningful, and the ability to resolve pure components signal weights.

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Ag-based nanoparticle as a colorimetric sensor array for identification and determination of mixture of halides and sulfide

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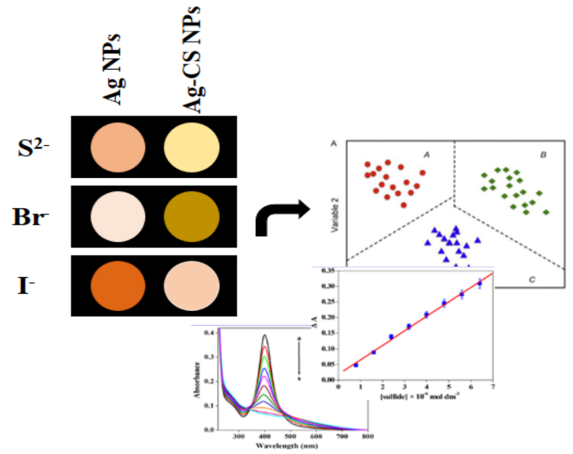
Graphical Abstract



Abstract: Inorganic anions such as halide ions and sulfide in excessive or insufficient dosage could lead to environmental risk and human health problems. Given the pressing demand for inorganic anions detection, considerable progress in analytical methodology development has been made in the past decade. The plasmonic metal sensors have recently received much attention in colorimetric assaying of various chemical species because of size-dependent visual changes, simplicity, selectivity, and high sensitivity in multidisciplinary research areas. Colorimetric sensor arrays comprised of metallic nanoparticles combining with chemometric techniques are suitable analytical tools for the detection and classification of different compounds in the mixture [1, 2]. In this study, a very simple and efficient color sensor array based on the cumulative behavior of silver nanoparticles, which is a combination of two different nanoparticles including Ag NPs and Ag-chitosan NPs, to identify and classify inorganic anions including Br^-, I^- and S^{2-} will be developed. The characterization study of silver embedded chitosan were studied by Fourier transform infrared spectroscopy (FTIR), X-ray diffraction spectroscopy (XRD) and scanning electron microscope (SEM). The interaction of nanoparticles and anions will be measured by a spectrophotometer and the amount of change in their observable spectra. The obtained results were shown that the collective behavior of nanoparticles was changed by introducing anions into array, resulting in providing different colorimetric responses and fingerprint-like patterns for each anion. The principal component analysis (PCA) and hierarchical clustering analysis (HCA) were used to analyze the obtained patterns and generate a clustering map for classifying Br^-, I^- and S^{2-}. In addition, the quantitative detection performance of the sensor array on simultaneous determination of studied anions in mixtures was examined.

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Hard modelling analysis of image based kinetic data

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Abstract: Image analysis involves image processing for extracting meaningful information, which can include noise cancellation, improved image quality and statistical calculation, and can be done both automatically and semi-automatically Computer image analysis is when a computer or electrical device automatically reads an image to obtain useful information. It is usually a computer, but it may be an electrical circuit or a digital camera or even a cell phone. Applications of image analysis include identification of new plant and animal species, astronomy, metallurgy, cancer diagnosis, forensics and error level analysis, and noise cancellation. . Image analysis allows quantitative image evaluation .Various techniques are used for automatic image analysis. Each technique is possible. It is useful for a small range of tasks. Therefore, various software in the field of photo analysis has been designed. [1-2]

In this study two colorless reactants (N- naghtyl amine ans sulfanilic acid) were reacted to form a red diazoniuom product. The reaction was recorded for 10 mines under visible light and its move was recorded with smartphone. In the next step the movie was converted to frames in MATLAB and was fitted to first order kinetic model. The reaction was monitored in different initial concentrations of reactants. The reaction rate constant was also obtained as 3.599×10^{-5}. The error of estimated parameter was estimated 1.0513×10^{-7}. The applied method is very simple, and can be used in analysis of reactions involving a change in color without need to spectrophotometer. The reaction process is very simple and easy to do.

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Classification of Iranian honey according to their geographical origin based on the chemical composition and pattern recognition methods

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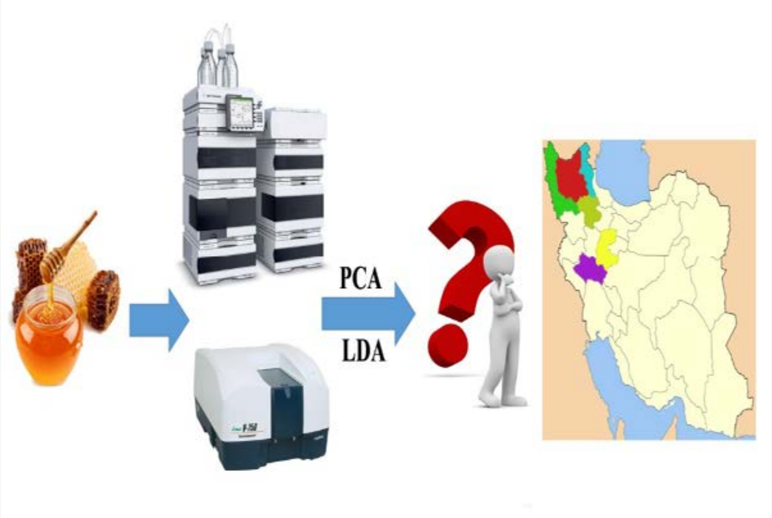
Graphical Abstract



Abstract: Honey is the natural sweet substance produced by honey bees which is composed mainly of sugars and a complex mixture of minor components such as amino and organic acids, minerals, flavonoids, and other phytochemicals [1]. The composition and sensory attributes of honey vary considerably depending on its botanical and geographical origin [2, 3]. Therefore, it is studied in different countries and zones because of the interest of consumers in origin and quality of food. Iran produces and exports honey from different zones; therefore, the main aim of this work was to study the chemical parameters of honeys (from Ghalikouh, Jasb and North West of Iran) using pattern recognition methods in order to classify honeys according to their geographical origin. Because honey samples contain a number of fluorophores, it is not enough to measure only a single variable spectrum to collect sufficient data. Therefore, multivariate measurements are required. Amino acid contents were obtained using HPLC with fluorescence detection after derivatization and spectrophotometric data (UV-Vis spectra) were obtained after liquid-liquid extraction. Then, the results were analyzed by principal component analysis (PCA) and linear discriminant analysis (LDA) for classification purposes. The results showed that the chemometric analysis allowed classification of samples according to their geographical origin by using spectrophotometric data. A high percentage of correct classifications for the training data demonstrated the strong relationship between the spectral data and the geographical origin, while a high percentage for the prediction set showed the ability to indicate the origin of an unknown honey sample based on its chemical composition spectrophotometric data.

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A Combined Multivariate Classification and Calibration Approach for Gas Chromatographic Fingerprint Analysis and Antioxidant Activity Modelling of Secondary Metabolites of Saffron

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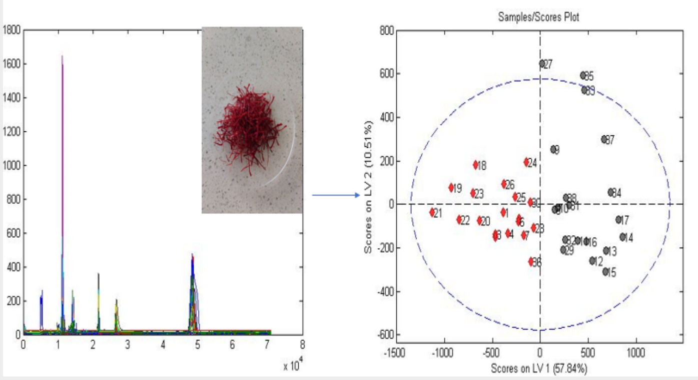
Graphical Abstract



Abstract: Chromatographic fingerprinting is a common method for authentication and quality control of natural complex samples such as plat extracts. In this regard, gas chromatography (GC) is the best option for fingerprinting and identification of chemical constituents of such samples, and it can also provide reliable quantitative and quantitative information about these samples. On the other hand, due to complexity of natural sample matrices and lack of selectivity in analytical instruments, multivariate chemometric methods have been extensively used to exploit useful information from chromatographic fingerprints. Saffron, called as red gold, has some bioactive compounds including crocetin, crocin, safranal and picrocrocin and it can have antioxidant effects, which can be measured by the 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging activity of the saffron samples. In the present contribution, a chemometrics based-strategy is proposed for GC fingerprints analysis of saffron in order to control its quality and its correlation with antioxidant activity of saffron. On this matter, an optimized ultrasonic-assisted extraction-dispersive liquid-liquid microextraction (UAE-DLLME) was used for extraction of chemical components of thirty-eight saffron samples. The GC fingerprints of saffron samples were obtained in optimum extraction conditions and they were arranged in a data matrix and this data matrix was mean centered and Pareto scaled before multivariate classification. The data was then analyzed using principal component analysis (PCA), hierarchical cluster analysis (HCA) to find similarities and dissimilarities among samples. In general, two clear-cut clusters were determined using PCA score plot and HCA dendrogram. Then, partial least squares-discriminant analysis (PLS-DA) was used for supervised classification of the two classes and it properly could classify samples with 94.7% sensitivity and 89.5% specificity. Finally, the DPPH radical scavenging activity of the saffron samples was measured by the fact that the absorption of visible light by DPPH declines when DPPH is reduced by an antioxidant. It was attempted to correlate the GC fingerprints of saffron samples to their DPPH radical scavenging activity using partial least squares regression (PLSR). It is concluded that the proposed strategy in this work can be successfully applied for comprehensive analysis of chromatographic fingerprints of complex natural samples.

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^{1}HNMR Based Metabolomics Study of Altered Metabolic Pathways in Patients with Covid-19

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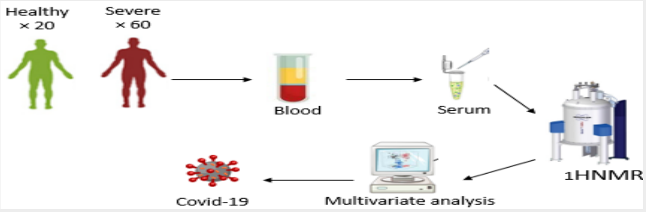
Graphical Abstract



Abstract: Coronavirus disease (COVID-19) is an infectious respiratory that was first reported in December 2019 in Wuhun, China. it is quickly spread around the world by severe acute respiratory syndrome coronavirus 2(SARS-COV-2). There is limited information about pathogenesis and targeted treatment strategies. The severity of the disease in patients with COVID-19 is divided into three states: mild, moderate and severe, due to differences in the immune response of individuals against the coronavirus [1]. Metabolomics is a newly developed technique in the field of systematic biology following genomics and proteomics, and The study of metabolomics refers to the wide and quantitative analysis of all metabolites in biological samples [2]. Metabolomics has become one of the most important methods in biomedical research using advanced methods such as Nuclear magnetic resonance (NMR) and high resolution mass spectrometry (MS). Various metabolic studies have been performed around the world to study Covid-19, which show metabolic disorders during disease progression [3-4]. NMR metabolomics can identify the differences in low molecular weight molecules that exist in states of health or disease.In this study, serum samples of 61 covid-19 patients and 20 healthy people as the control were examined and the potential spectral markers were identified by multivariate and statistical analysis. Analysis show that the following metabolic pathways including Glysine,serine and threonine, Pirimidine metabolism, Histidine metabolism, Aminoacyl-tRNA biosynthesis, Pantothenate and CoA biosynthesis, beta-Alanine metabolism, Amino sugar and nucleotide sugar metabolism, Ascorbate and aldarate metabolism, Amino sugar and nucleotide sugar metabolism, Valine, leucine and isoleucine biosynthesis, Synthesis and degradation of ketone bodies are changed during illness. Our results provide valuable information related to COVID-19 serum markers, on the other hand selecting a proper data by pretreatment method is an essential step in the analysis of metabolomics data. In this study, autoscaling and range scaling performed better than the other pretreatment methods.

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Determination of Ametryn and Chlorpyrifos Pesticides in Saffron Using Dispersive Liquid–Liquid Microextraction Followed by Gas Chromatography with the Aid of Chemometrics

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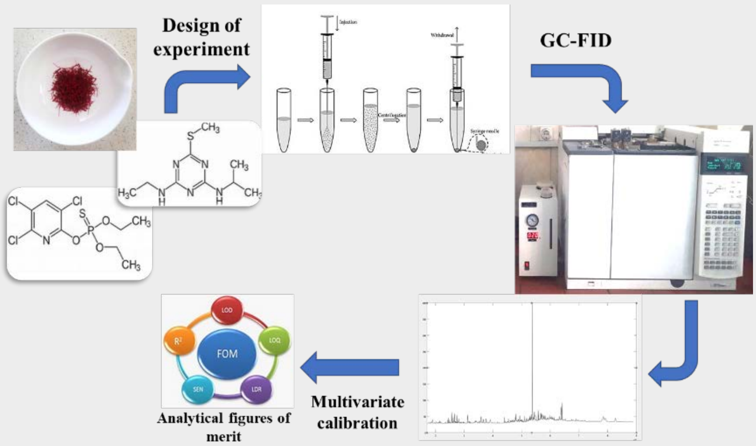
Graphical Abstract



Abstract: Saffron, the world's most expensive spice, is valuable for its color, taste and aroma properties, which are influenced by the postharvest treatments [1]. Pesticides and herbicides are among the most priority pollutants to be monitored in a wide variety of matrices because their incorporation into water, soil, and crops may represent a serious hazard to human health and wild life. As a result, the quality control of saffron to achieve the healthiest production is incredibly considerable. Plenty of efforts have been developed to determinate the amount of pesticides in complex matrices [2]. In the present study, dispersive liquid-liquid microextraction (DLLME) is developed for extraction of a triazine herbicide, ametryn and an organophosphate pesticide, chlorpyrifos from saffron and simultaneous quantitative determination of them with gas chromatography-flame ionization detector (GC-FID). It should be noted that central composite design (CCD), multiple linear regression (MLR), and Nelder-Mead simplex optimization method were used in order to design, model, and optimize all of the effective factors of the extraction. Accordingly, global optimum conditions were gained which were 40 μL of acetonitrile (disperser solvent), 40 μL of chloroform (extraction solvent), 20 minutes of sonication time and 3.0 % (w/v) NaCl [3]. A multivariate calibration model using partial least squares regression (PLSR) was built in concentration range of 10-100 ngmL^{-1} of calibration samples and multivariate analytical figures of merit (AFOMs) were calculated for two pesticides. In the next section, these two pesticides were determined in saffron samples under optimized DLLME-GC-FID and using PLSR. It is concluded that the proposed method can reliably determine ametryn and chlorpyrifos in saffron close to their maximum residue limit (MRL).

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Chemometrics-Assisted Liquid Phase Microextraction Based on Deep Eutectic Solvent Followed by Gas Chromatography for Determination of Polycyclic Aromatic Hydrocarbons in Aqueous and Juice Samples

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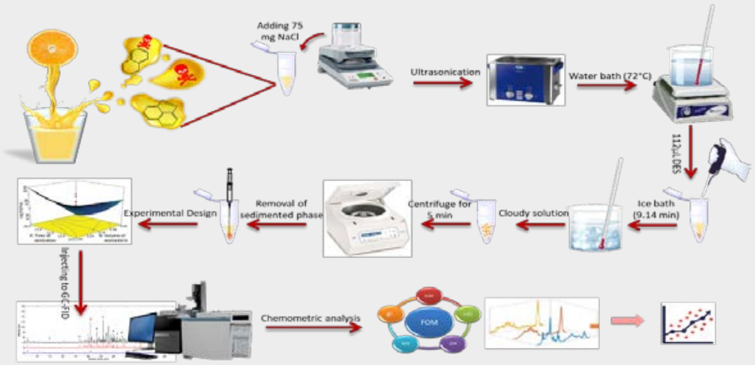
Graphical Abstract



Abstract: Nowadays, deep eutectic solvents (DESs), a new generation of solvents, are widely used in extraction procedures because of their low cost, low toxicity, and biodegradability characteristics. One of the main features of DESs is their ability to be used to extract a wide range of organic pollutants such as polycyclic aromatic hydrocarbons (PAHs), known as one of the contaminants of emerging concerns (CECs), from aqueous matrices. Extraction from complex matrices requires the simultaneous study of different extraction variables. In this regard, chemometric methods have been widely used to maximize extraction efficiency [1-3]. In the present study, DES-based liquid phase microextraction (DES-LPME) methodology followed by GC-FID in combination with chemometrics was studied in order to determine 13 PAHs in aqueous samples. In the first step, different DESs were synthesized based on previous studies, and a mixture of 4-chlorophenol and choline chloride in a 2:1 molar ratio was selected as the best extracting solvent. After that, important LPME parameters were optimized using the central composite design (CCD) and the simplex optimization method. On this matter, the optimal DES volume, extraction time, temperature, and salt amount were 112µl, 9.14 min, 72°C, and 75 mg, respectively. In the next step, artifacts in GC chromatograms such as baseline and elution time shift were corrected using asymmetric least squares (AsLS) and correlation optimized warping (COW) algorithms [4]. Next, both univariate and multivariate calibrations were performed in seven concentration levels (0.2-70 ppb) with partial least squares regression (PLS-R) and artificial neural network (ANN). For PLS-R as the model with better performance, sensitivity (SEN), analytical sensitivity (γ), limit of detection (LOD), limit of quantitation (LOQ) and root mean square error of prediction (RMSEP) were calculated for each PAH. For all PAHs, except for fluorene with $R^2=0.87$, the calculated R^2 was obtained at about 99%. Finally, to test the applicability of the proposed methods in real samples, sea waters, as well as orange and apple juices, were analyzed at 10 and 30 ppb concentration levels. The relative recoveries were obtained in the range of 83.4%-109% for 10 ppb and 78.4%-103% for 30 ppb. In conclusion, the developed method based on DES-LPME combined with GC-FID and chemometric techniques can be used for determining PAHs in real matrices.

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Soft Classification Models Combined with Hyperspectral Imaging for Diagnosis of Breast Cancer

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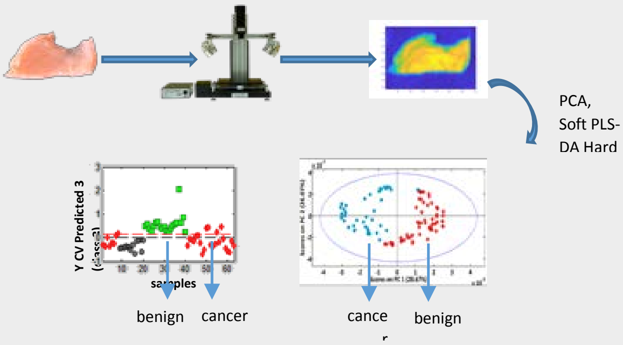
Graphical Abstract



Abstract: Breast cancer is one of the known cancers. Among every 8 females, one will suffer from malignant breast tissue growth, during their lifespan [1]. Diagnosis of breast cancer in the early stages is an important matter and can lead to full recovery. Therefore, finding a solution for diagnosis in the early stages, with a high sensitivity is a great concern. Currently biopsy is the standard method for breast cancer sampling [2]. There are some drawbacks for this method, for instance the breast tissue has to be fully removed in order to determine the margin of the tumor and as a result some parts of the healthy tissue will be in the biopsy sample. Hyperspectral imaging (HSI) is a novel method in medical field and it has shown promising results in the diagnosis of cancer (benign and malignant) [3]. HSI offers information about spatial and spectral properties of the sample that determines the distribution map and the identity of the present components in the sample [4]. In the present contribution, Vis-NIR HSI combined with different hard and soft classification methods is proposed for diagnosis of breast cancer. In this regard, 56 breast cancer biopsy samples from healthy and cancerous tissues were provided. It should be pointed out that the cancerous biopsy samples were from grade 2 and grade 3. Vis-NIR HSI images of the samples were acquired in the spectral range of 400-950 nm. To explore the similarities and dissimilarities among samples, initially, principal component analysis (PCA) was used which showed a distinction between healthy and cancerous samples. The data was then analyzed with soft models of partial least squares-discriminant analysis (soft PLS-DA) and soft independent modelling of class analogy (SIMCA). The goal was to explore the ability of these methods in comparison with hard classification methods to model different classes. The classification figures of merit in terms of sensitivity and specificity were promising for both algorithms. As an instance, the sensitivity values for healthy, grade 2 and grade 3 samples were respectively 89.5%, 84.4%, 92.5%. Additionally, the specificity values were 87.0%, 72.1% and 89.2% for healthy, grade 2 and grade 3 samples, respectively. Comparison of the results for soft classification models with conventional PLS-DA (hard PLS-DA) showed some benefits including better classification figures of merit.

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Multivariate Curve Resolution-Alternating Least Squares for comprehensive resolution and quantification of Polycyclic Aromatic Hydrocarbons in Oil Fractions by Means of GC×GC-TOFMS

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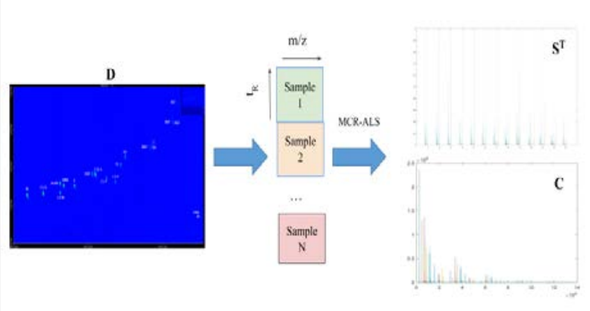
Graphical Abstract



Abstract: PAHs are a group of organic pollutants with adverse effects on both humans and the environment. They are mainly a fraction of petroleum hydrocarbons and various types of PAHs are discharged into the environment by both natural and anthropogenic activities, therefore the determination and quantification of PAHs are of prime importance [1]. When comprehensive two-dimensional chromatographic techniques are coupled with a high acquisition rate detector, like a time-of-flight mass spectrometer (TOFMS), the result is an extremely powerful instrument (e.g., GC × GC−MS and LC × LC−MS) for the analysis of very complex natural samples. However, one of the main challenges in multidimensional chromatography is related to the difficulty of the analysis and interpretation of the enormous amount of data obtained in these cases. Additionally, complete separation of all detectable components still cannot be achieved because of the extremely high complexity of natural samples and limitations in experimental and instrumental Conditions [2]. Chemometric methods can be used to solve this issue and exploit pure analyte signal from a complex mixture (second-order advantage). In this regard, multivariate curve resolution-alternating least squares (MCR-ALS) is frequently used for GC × GC data [3]. Up to now, GC × GC-TOFMS combined to MCR-ALS has been proposed for the resolution and quantification of very complex mixtures of compounds such as PAHs in heavy fuel oil (HFO) [4]. This approach is based on the analysis of the chromatographic segments. However, segmented analysis is very time consuming and complex, therefore, in this study a new MCR strategy is proposed for full chromatogram analysis for the determination of 13 PAHs. GC × GC-TOFMS data arranged in a column-wise augmented data matrix and then the time direction was reduced using discrete wavelet transform which was allowed reducing the size of the data. Then, compressed augmented data matrix was analyzed by MCR-ALS using spectral normalization and non-negativity constraint during the ALS optimization procedure. Also, orthogonal projection approach (OPA) was used to calculate initial spectral estimates to start ALS optimization. Then, calibration curves based on MCR-ALS built up with standard samples of PAHs and analytical figures of merits (AFOMs) were calculated. Finally, determination of target PAHs in aromatic fraction of different oil fractions such as North Sea Oil was performed using proposed strategy and proper quantitative results were also obtained.

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Application of Image Processing in the Analysis of pH-Sensitive Optical Sensors

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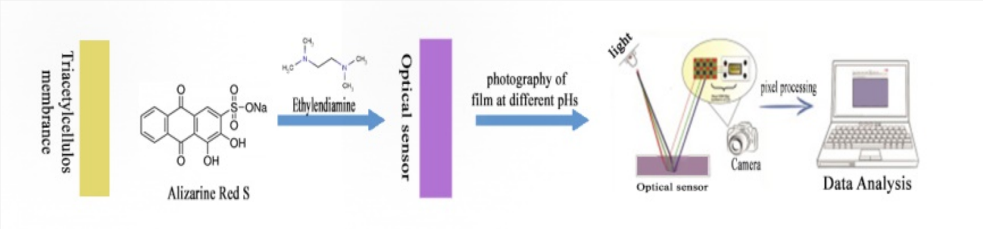
Graphical Abstract



Abstract: pH is an important factor in many fields of chemical, biological, and environmental sciences. Importantly, the pH level is useful as a qualitative, diagnostic, or surrogate parameter in industrial, physiological, or clinical practice [1]. Many methods have been developed for determination of pH but Optical sensors (optodes) have several advantages such as simplicity, rapidity, low cost, capability for incorporation into easy-to-use kits for remote and continuous monitoring and the selectivity and sensitivity for trace analysis [2]. Chemometric methods have been widely employed for simultaneous determination of pH. Optodes and chemometric methods are well-established research areas in analytical chemistry for metal ion and pH determination. However, there are little efforts to employ both tools together [3-5].in this study a novel optical sensor has been proposed for sensitive determination of pH based on immobilization of alizarin red s on a triacetylcellulose membrance. A change in pH of solution caused a color change in the sensor containing the ligand, which was also visible in spectroscopic absorption and digital images that obtains by a smartphone. pH values of solutions were studied by calibration modeling and using application of pixel selection to predict the pH and comparison with experimental data based on absorption spectra and digital images of 26 sensors in solutions with different pH . In other words, pixel of images was employed as input data for the construction of chemometric models, exploiting multivariate tools, and then validated using independent measurements. The studied model showed valuable result and satisfactory output validation parameters, which made the model acceptable that can help researchers to design of new sensors for determination of various parameters such as pH, drugs and metals in aqueous solutions.

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Classification of Two Dimensional GC-MS Based Data Applying Kohenen Self Organizing Maps: Classification Based on Geographical Origin of Saffron Samples

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Graphical Abstract

class	sensitivity	specificity
Class1	0.05	0.93
Class2	0.93	0.05

Abstract: Crocus sativus or saffron is one of the most valuable indigenous herbs in Iran and is known as the most expensive spice in red gold. Classification of different saffron samples based on their geographical distribution using gas chromatography-Mass spectroscopy was used to determine the metabolites [1].

In this study, 86 saffron samples were collected from 7 regions in Khorasan Razavi province including Kashmar, Taybad, Torbat-e Jam, Torbat Heydariyeh, Zaveh, Neishabour and Rashtkhar cities to show if they can be discriminated based on their volatile metabolites. After coding saffron samples, these samples were placed in a sealed container in a dark place. The selection of samples in this experiment is completely random and the number of samples varies from city to city. For example, the number of samples was Kashmar 9, Taybad 17, Torbat Jam 15, Torbat Heydariyeh 10, Zaveh, 13 Neishabour 10 and Rashtkhar 12 samples. Saffron extract will be extracted using diethyl ether and injected into GC-MS. The data is then analyzed in the MATLAB environment. Area data were analyzed applying different preprocessing methods on data in MATLAB environment and Kohenen toolbox [2]. Findings show that saffron in different cities of Iran, in spite of their many similarities ,have differences, and these differences cause the separation of saffron samples of different classes from each other. This means that in these samples the level of volatile metabolites in different geographical regions are different. However, saffron of some regions were similar but some of them were different. Metabolites which they are responsible for discrimination of these saffron were determined. These differences can be achieved using chemometrics methods and the relationship between data [3].

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Error propagation in self-modeling curve resolution of size reduced data

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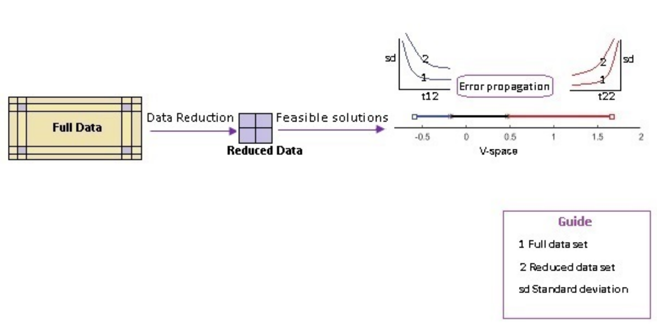
Graphical Abstract



Abstract: Self-modeling curve resolution (SMCR) analysis decomposes bilinear multivariate measurement matrices into more physically interpretable forms, such as spectral profiles, concentration profiles, elution profiles, and so on using a minimum amount of prior information about the process under investigation. By applying different constraints, SMCR techniques don't produce necessary a unique solution for every component in a system and a range of feasible solutions is possible [1]. Evaluation of the error estimation and propagation of experimental errors is a key issue in the quality assessment of the results obtained by any chemometric methods and in particular for the MCR methods. Different statistical procedures have been proposed to evaluate the error propagation for those cases where formulae for analytical evaluation of errors are not available owing to the strong non-linear behavior of the proposed model. Among these procedures, Monte Carlo and numerical resampling methods have been proposed and become popular owing to their relatively easy implementation on today's computers [2]. Despite recent advances both in the field of self-modeling curve resolution (SMCR) and on the practical side, (bio) chemical data sets and images are difficult to analyze because they are big and spatial spectral information is highly mixed. Hence, there is a need for data compression and reduction techniques for which the preservation of essential information is guaranteed by low-level data transformation and minimum user involvement [3]. As the full and reduced data share the same vector space, MCR analysis can be equivalently performed on each data set [4].This work aims to investigate how the reduction of the data set dimensions affects the reliability of the feasible solutions. To study the effect of the reduction of the data set dimensions, several simulated data sets and their reduced sizes have been systematically investigated. The obtained results by simulated data sets showed that the reduction of the data sets dimensions increases the uncertainties of the feasible solutions.

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Classification and authentication of Iranian tea according to their geographical origin based on UV-Vis spectral fingerprinting using pattern recognition methods

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Abstract: The chemical composition of tea, as one of the most important quality characteristics, depends on several parameters including the genetic nature of the plant, climatic conditions (temperature, sun exposure time, rainfall), growth altitude, soil and horticultural practices, the plucking time, sorting of the leaves, the processing steps and the storage conditions [1].

The reputation of particular countries and farms in producing high quality tea allowed these producers to offer higher prices for their specific product. This may tempt some manufacturers to use a fraudulent label in order to introduce their tea product as the product of these famous areas, so that they can sell their product at a higher price [2]. This has made consumers sensitive and precise to know the exact geographical origin of the tea crop.

Classification of tea samples based on the geographical origin was investigated using the UV-Vis spectra. Tea samples from five regions in North of Iran were collected during the harvesting period 2018–2019. Mean centering (MC), multiplicative scatter correction (MSC), standard normal variate (SNV) and their combinations, were employed as the spectral pre-processing methods in order to improve the quality of the spectra. Principal component analysis (PCA), principal component analysis-linear discriminant analysis (PCA-LDA) and partial least square-linear discriminant analysis (PLS-LDA) were employed in order to assess the feasibility of discrimination of tea samples. The results showed that the tea samples can be identified based on their geographical regions using UV-Vis spectral fingerprints. A high percentage of correct classifications for the training set using cross validation shows the strong relationship between the UV-Vis spectral fingerprinting and the geographical region, while the satisfactory results for the prediction set demonstrates the ability to identify the geographical origin of an unknown tea sample based on its UV-Vis spectral data.

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Spectroscopic Methods

isac27-1113	Faeze Mohammad hosseini sharifabad - Alireza Bazmandegan-Shamili - Mohammad Anary-Abasinejad - Masoud Rohani-Moghadam - Maryam Oudi	A Novel Fluorescent Chemosensor Based On Phosphorus Ylides For Sensitive Detection Of Copper Ions	328
isac27-1117	Parisa Mousareza - Mahsa Amiri - Naader Alizadeh	Determination of Phosphate Anion in Food Samples by Near IR Spectrophotometry	329
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isac27-1275	Hanieh Khorramdareh - Siavash Nourozi	Carbon Quantum Dots as a Fluorescent Sensing Platform for Amoxicillin Detection	338

Quantification of Rhodamine B and Fluorescein in microliter samples using multivariate fluorescence data from paper surface

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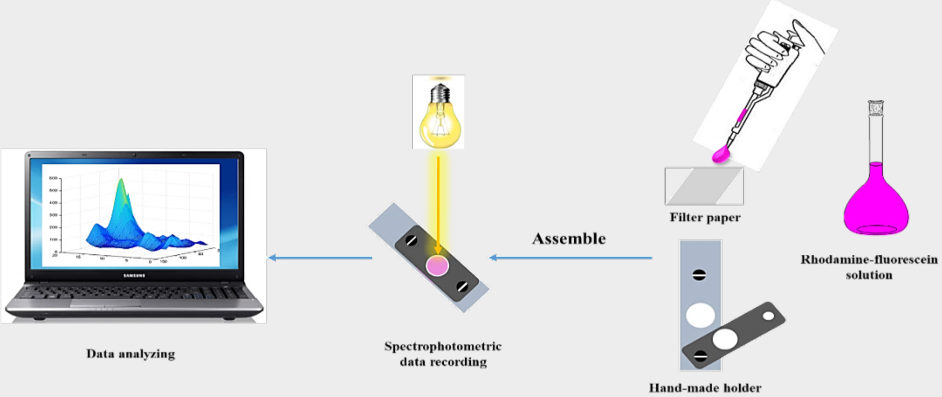
Graphical Abstract



Abstract: A simple, facile, and cost-effective method is described for quantification of two commonly used fluorophores, Rhodamine B (RhB), and Fluorescein (FLU), using solid surface fluorescence data from the filter paper surface. Spectrofluorometers are common for measurement of fluorescence from liquid samples however, this work is a simple method for recording fluorescence data from solid RhB and FLU samples applying any common spectrofluoremeter assisted with a simple device. The hand-made device is a holder cuvette for measurement of the three-dimensional excitation emission matrix (3D-EEM) of fluorescent data from a paper strip. Applied wavelength ranges were 300-600 nm for excitation and 400-700 nm for emission. For RhB-paper strip the fluorescence intensity remarkably was increased compared to RhB in solution, and FLU-paper showed acceptable fluorescence intensity although it is not emissive in the solid-state. Considered concentration ranges of FLU and RhB were 10.0 to 100.0 μM and 2.0 to 25.0 μM, respectively. Moreover, in the solid mixture (RhB-FLU-paper strip) the Forster resonance fluorescence transfer (FRET) was observed from FLU to RhB, the same as the observed behavior in the liquid form. Finally, analytes were successfully quantified using powerful multivariate methods such as partial least squares (PLS) and parallel factor analysis (PARAFAC) and coefficient of determination (R^2) >0.90 was obtain. Main advantages of the presented solid surface fluorescence technique is the microliter scale of required samples, and lower fluorescence self-absorption compared to liquid samples.

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Nanolitmus Paper: A Wide Range Colorimetric pH Sensor Based on Formation of Gold@Silver Nanorods

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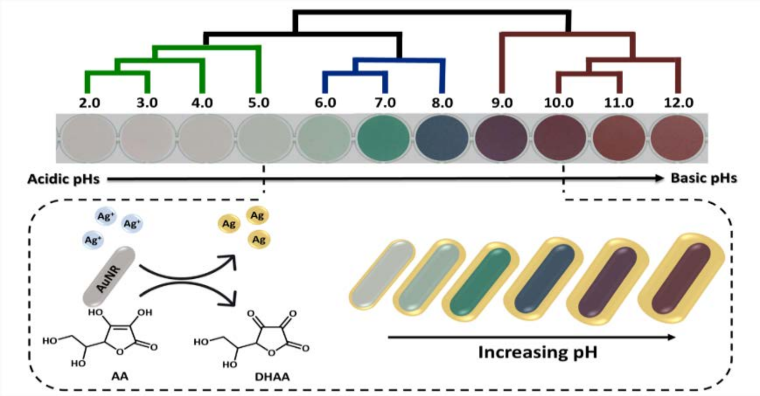
Graphical Abstract



Abstract: The potential of hydrogen (pH) has been defined as a significant parameter that provides crucial information on local chemical conditions. In this regard, the measurement of pH has received great attention in diverse fields such as clinical diagnostics, agriculture, food/beverage industry, and environmental/biomedical applications [1]. Due to the widespread applications of pH in diverse fields, the development of rapid and simple yet reliable probes for the determination of pH has attracted significant interest. In this regard, the appearance of nanoparticles (NPs) can be considered to be a milestone in the design of optical pH sensors. Among the diverse types of NPs, noble metal NPs with unique localized surface plasmon resonance (LSPR) properties, associated with their composition, size, and morphology have been considered the exciting colorimetric reporters [2].

In the present work, a multicolor colorimetric probe has been developed for pH monitoring by exploiting the silver metallization on the surface of AuNRs. The reduction of silver ions by ascorbic acid is strongly influenced by pH, resulting in silver nanoshell deposition on the surface of AuNRs. The formation of plasmon-tunable Au@Ag core-shells caused a blue shift in the longitudinal plasmonic peak of AuNRs in different pHs ranging from 2.0 to 12.0 at optimum conditions. As a result of spectral shifting, distinct color variations were observed from brownish pink/light green color (acidic media) to dark green/blue color (neutral media) and eventually purple/brown color (basic media). Furthermore, a paper-based analytical device (PAD) was introduced for on-site pH screening. The pH-sensitive PAD was simply fabricated by immobilizing AuNRs on the surface of filter paper. Finally, the developed plasmonic probe was validated to be capable of accurate pH monitoring in urine, spoiled meat, drinking water, and seawater samples.

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Preparation and Characterization of Sulfobutyl Ether Beta-Cyclodextrin Binary Inclusion Complexes of Bosentan with Enhanced Bioavailability

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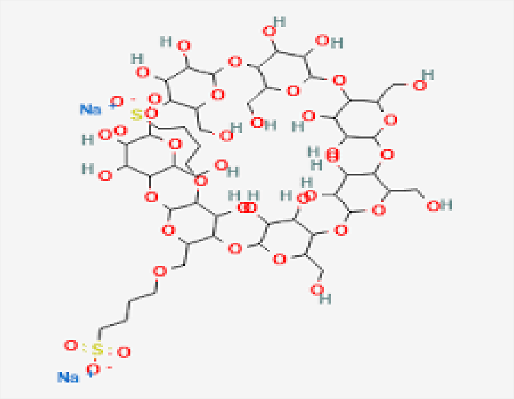
Graphical Abstract



(SBE-β-CD)

Abstract: Bosentan (BOS), an endothelin receptor antagonist, presents poor solubility in water, low bioavailability and side effects [1]. The aim of this study is to enhance the solubility and bioavailability of BOS and its antioxidating activity through complexation using sulfobutylether-β-cyclodextrin (SBE-β-CD), which is used as a delivery material. The formation of this novel inclusion complex of BOS and SBE-β-CD was synthesized and investigated in two states. Solid state of complex was obtained through freeze drying and assessed by the formation of inclusion complexes was characterized by fourier transform spectroscopy (FTIR), ultraviolet-visible (UV-Vis) and nuclear magnetic resonance (NMR) spectroscopies [2]. The Job's method was performed to illustrate that BOS was intended to form a 1:1 complex with ligand. The phase solubility study conducted in distilled water was carried out to acquire the stability constant. Phase solubility analysis showed AL-type diagram, according to the Higuchi and Connors method [3]. The solubility and dissolution of complex was significantly improved in the contrast with the BOS alone as the pure drug. In addition, the results of this study confirm the formation of inclusion complex in solution. Furthermore, the antioxidant activity was investigated by using DPPH (2,2-diphenyl-1-picrylhydrazyl) indicator with its violet color. The antioxidant activity of BOS and the inclusion complex indicated that the inclusion complex was more reactive than its free form into antioxidant activity [4]. The interactions of complex were investigated in the presence and absence of chitosan as a carrier for delivery of drug.

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Dual-emissive Carbon Dots as "On-Off-On" Fluorescent Probe for Cu^{2+} Ions and Aspartic Acid Detection

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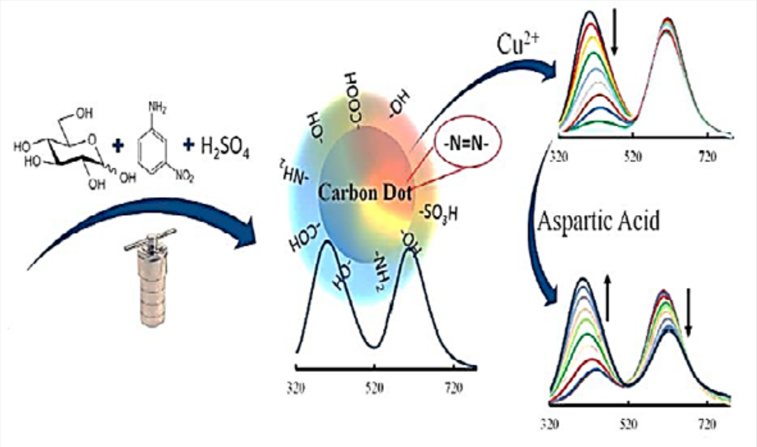
Graphical Abstract



Abstract: Carbon dots (CDs) as a new class of metal-free fluorescent nanomaterials have attracted increasing attention, because of unique advantages such as easy and non-expensive preparation, low cytotoxicity, excellent water solubility, high chemo-/photo-stability, and ease of surface functionalization [1, 2]. Up to now, various ratiometric fluorescent nanoprobes have been fabricated based on fluorescent nanoparticles such as CDs [3-5].

Herein, novel intrinsic dual-emitting carbon dots (CDs) are prepared through a one-step hydrothermal treatment of glucose and 3-nitroaniline in sulfuric acid solution and utilized for ratiometric determination of Cu^{2+} and aspartic acid (Asp). The CDs exhibited an interesting pH-switchable emission behavior displaying an intrinsic dual-emitting peak with emission maxima at 400 and 610 nm at pH 4.0-5.0. The presence of Cu^{2+} intensively quenched the first emission peak at 400 nm, but it had a negligible effect on the second emission peak. The ratiometric signal displayed a high selectively for Cu^{2+} over other metal ions and provided a linear response over the concentration range of 0.01-1.00 μM with a detection limit of 7.0 nM. Moreover, at pH 4.0, Asp was able to restore the quenched fluorescence of the CDs-Cu^{2+} system with a much more successful performance than other amino acids. This on-off-on fluorescence behavior provided a selective ratiometric fluorescence method for the determination of Asp in the concentration range of 0.2 to 15 μM. The acceptable detection results for Cu^{2+} in a river water sample and for Asp in human serum samples confirmed the potential application of this ratiometric nanoprobe for sensing in real samples.

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Colorimetric Sensor Array Based on Au Nanoparticles for Detection and Discrimination of Antibiotics in Environmental water

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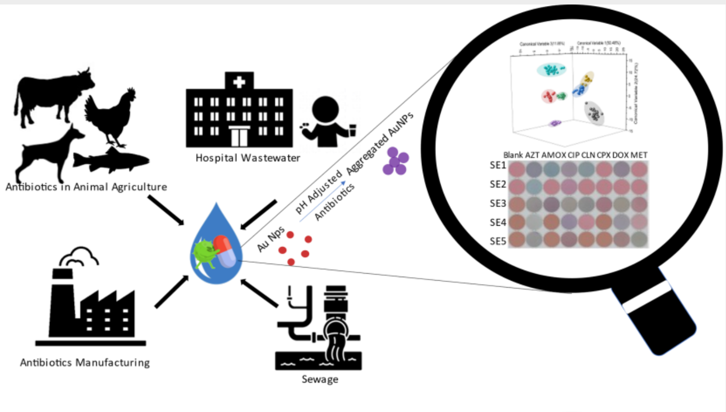

Abstract: Antibiotics (ABs) have been regarded as powerful medications to treat or prevent bacterial infections. The antibiotic resistance though, is one of daunting challenges of our time which has been attributed to the overuse of these drugs and usage of antibiotic-contaminated food resources. Developing rapid and visual detection methods capable of simultaneously monitoring ABs is highly desired due to their harmful effects on human health. In the present study, we have designed a colorimetric sensor array consisting of two types of gold nanoparticles (i.e., Citrate-capped gold nanoparticles (Cit-AuNPs) and Borohydride-capped gold nanoparticles (BH_4-AuNPs) for the discrimination and determination of critical ABs belong to different antibiotic classes (i.e., azithromycin (AZT), amoxicillin (AMOX), Ciprofloxacin (CIP), clindamycin (CLN), cefixime (CFX), Doxycycline (DOX) and metronidazole (MET)). The design principle of the probe was based on the aggregation of Cit-AuNPs and BH_4-AuNPs in the presence of ABs. Unique aggregation profiles of sensor elements in the presence of the targets at various pH conditions, using citrate and phosphate buffers, were visually assessed and statistically analyzed by various data visualization and pattern recognition methods. Namely, linear discriminant analysis (LDA) and partial least squares (PLS) regression were employed for the qualitative and quantitative determination of ABs. The excellent statistical parameters for classification (sensitivity 100% and specificity 100%) and for multivariate calibration ($R^2_{cal}>0.99$ and $R^2_{cv}>0.99$) show the ability of the designed array to discriminate and detect ABs. Also, the color variation of the sensor array in presence of ABs indicates rapidly visual discrimination of ABs by the developed sensor array. Finally, the practicality of the sensor array in real samples was investigated by the determination of ABs in the river and underground water by which the potential of the probe for on-site determination of antibiotics was successfully verified.

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Monitoring Meat Freshness with a Ratiometric Fluorescence Nanoprobe and a Combinational Logic Gate

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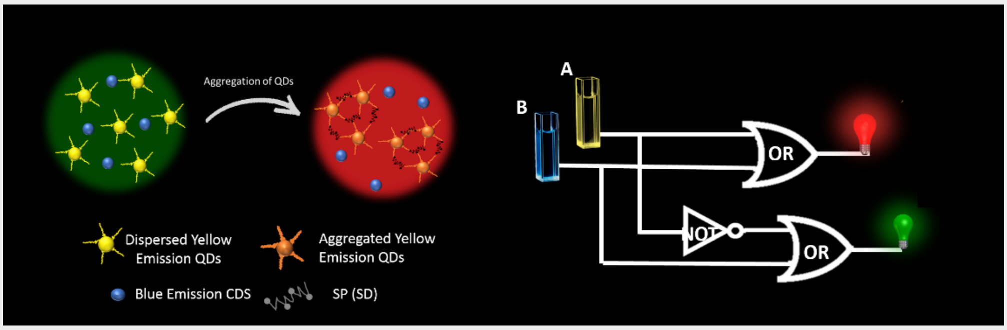

Abstract: Monitoring meat freshness has always been an important topic in food safety and control. Although significant advances have been made, there are still some challenges to meet [1]. Probing markers of meat spoilage, particularly spermine (SP) and spermidine (SD), become even more challenging when rapid, sensitive and portable detections are desired [2]. Herein, we have developed a ratiometric fluorescent nano probe with a wide color variation for visual determination of SP and SD in meat samples. The green emission of the ratiometric probe which is provided from the combination of the yellow emission of MPA-capped CdTe quantum dots (YQDs) and the blue emission of carbon dots (BCDs), turns into pink when SP or SD are present. The higher sensitivity of the probe towards SP which eventually causes earlier color alterations, is manipulated for the quantification of their mixtures. The results show that the developed sensor has good linearity in the range of 0.5- 10 μM and 0.5-80 μM for SP and SD, respectively and suitable detection limits including 0.2 μM for SP and 2.1 μM for SD. The ratiometric probe also revealed high selectivity towards SP and SD in the presence of amino acids and other biogenic amines. Moreover, the RGB indices of the fluorescence response were extracted to build a two input - two output combinational logic gate for visual and simultaneous detection of SP and SD. The dual functional logic gate was easy to design and convenient to operate. Finally, a portable sensor was fabricated for visual, rapid and on-site monitoring of meat spoilage.

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A Nanoprobe for Furazolidone Determination Based on the N, P graphene quantum dots and Copper Ions

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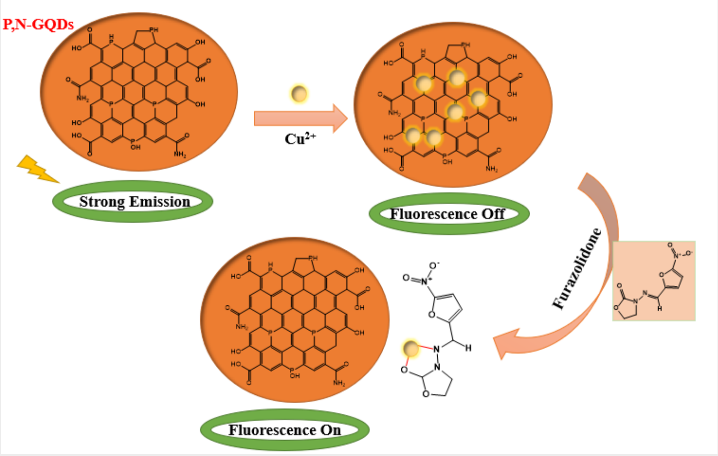

Abstract: Furazolidone (FZD) is one of the nitrofuran antibiotics that was widely used as a growth stimulant in the livestock and poultry industry. It was also used for the prevention and treatment of gastrointestinal infections caused by Escherichia coli and Salmonella in birds, especially chickens and roosters. Despite the strong antibacterial properties of furazolidone, its residues are recognized to be mutagenic and carcinogenic. So, since 1991, the Food and Drug Administration has refused the prescription of furazolidone and nitrofurans, and in 1995 the European Union banned the use of furazolidone in food-producing animals. However, because of its low price, availability, and strong effectiveness, it is still illegally used in many livestock, poultry, and aquaculture farms. Thus, the development of sensitive and selective methods for monitoring furazolidone residues in biological tissues and food products is a necessary task [1]. Graphene quantum dots are a new type of nanomaterials belonging to the graphene family which gained noticeable interest in the design of sensors for the determination of drugs [2, 3].

In this study, a sensitive and selective fluorimetric method is designed for the direct determination of furazolidone based on the nitrogen and phosphorus graphene quantum dots (P, N-GQDs) and copper ions. Thus, the addition of copper ions to the P, N-GQDs, resulted in a decrease in its fluorescence intensity at 447 nm due to the electrostatic interaction between copper ions and the charged surface of quantum dots. However, in the presence of furazolidone, the fluorescence emission of P, N-GQDs is retrieved as the result of the formation of the complex between the drug and copper ions and the release of the P, N-GQDs surface. Under optimal conditions, the calibration graph was linear in the concentration range of 0.05-20.0 µM with a limit of detection of 0.009 µM. The intra- and inter-day relative standard deviations at the 2.0 µM level of furazolidone (n = 5) were 1.8 and 3.7%, respectively. The method was successfully employed for the determination of furazolidone in urine and serum samples.

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Fluorometric Determination of Lamotrigine Using Folic Acid Functionalized Nitrogen and Sulfur Graphene Quantum Dots and Silver Nanoparticles Coated with Glycine and Cysteine

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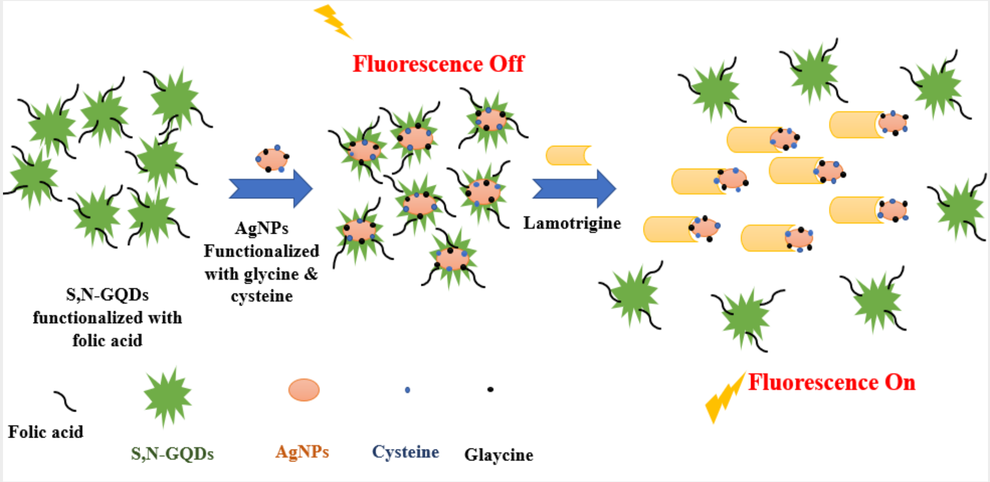

Abstract: Lamotrigine (LTG) is an antiepileptic drug that has been successfully applied in the treatment of epilepsy in adults and children over two years old as well as bipolar disorder. However, over-dose consumption of lamotrigine may result in the side effects such as the enhancement of suicide risk, a decrease in red blood cells, and an increase in allergic reactions. Thus, significant demand has been raised for the development of new and rapid methods for the quantification of LTG in biological samples [1].

For monitoring drugs, metal nanoparticles and graphene quantum dots have been recognized as effective fluorescent nanoprobe [2, 3]. Thus, in this study, a sensitive fluorometric method for the determination of lamotrigine has been developed using folic acid functionalized with sulfur and nitrogen graphene quantum dots (FA-S, N-GQDs) and silver nanoparticles coated with cysteine and glycine. The fluorescence emission of FA-S, N-GQDs at 493 nm decreases in the presence of the silver nanoparticles coated with cysteine and glycine due to Forrester's resonant energy transfer mechanism (FRET), however, the addition of lamotrigine to this system resulted in the retrieval of the fluorescence emission. This phenomenon can be related to the formation of the hydrogen bond between the functional group of drug and glycine and cysteine groups of silver nanoparticles followed by the release of FA- S, N-GQDs surface. Under optimal conditions, a linear relationship was obtained between the analytical signal and the concentration of lamotrigine in the range of 0.01-3.0 μM with a detection limit of 0.002 μM for the lamotrigine. The intra- and inter-day relative standard deviations at the 0.5 μM level of lamotrigine (n = 5) were 2.1 and 3.4%, respectively. The method was successfully utilized for the determination of lamotrigine in urine and serum samples.

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Preparation and invitro Characterization of Cholestrol loweringdrug incorated Captisol® inclusion complex

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Abstract: In present study, a novel formulation was developed to enhance solubility, stability, and bioavailability ofAtorvastatin. Atorvastatin as an anti-cholesterol drug has poorr water solubility of 14% which reduces its therapeutic benefit [1] The ability of captisol®(sulphobutylether-b-cyclodextrin, SBE-b-CD), to from inclusion complexes, both in solution and in thesolidstate, haz been tested in order to improve some unfavorable chemical-physical characteristics [2]

The aim of present study was to explore the impact of Atorvastatin (ATR) Sulfobutylether beta-cyclodextrin complex (ATR-SBE-B-CD) on ATR dissollotion rate and oral bioavailability. Preliminary comparative phase solubility study indicated ATR exhibited maximum solubility in SBE-β-CD solution.The job 's method performed to illustrate the ATR was intended to from a 1:1 complex with captisol. Solid state of complex was obtained through freeze drying and assessed by The formation of inclusion complex was charactenrized by fourier transform spectroscopy (FTIR) , ultraviolet –visible (UV-Vis) and nuclear magnetic resonance (NMR) spectrscopies [3].The phase solubility study conducted in distilled water was carried out to acquire the stability constant. The estimated apparent stability constant (K1:1) according to the Higuchi and Connors method. The results of this study confirm. The formation of inclusion complex in solution and suggest that the complexes formation between ATR and SBE-β-CDs could improve the bioavailability of the drug due to the enhancing absorption expected from increased drug solubility. Furthermore, the antioxidant activity of ATR and SBE-β-CDs inclusion complexes were determined by the 1,1-diphenyl-2-picrylhydrazyl (DPPH) method. The experimental results confirmed the forming of ATR complexes with SBE-β-CDs also these indicated that the ATR/SBE-β-CDs inclusion complexes were the most reactive than its free from into antioxidant activity [4].

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Measurement of copper and iron in Sarcheshmeh copper mine soil by XRF technique

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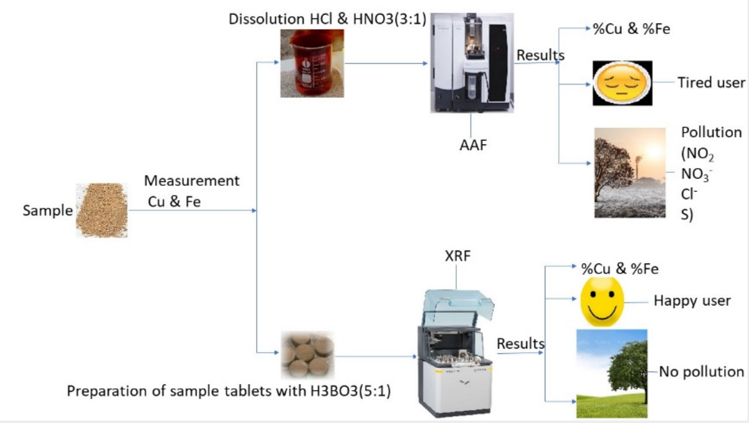
Graphical Abstract



Abstract: Measurement of copper and iron is important in copper ore soil samples. Chemical dissolution method is used to measure copper and iron in these samples [1-2]. One of the disadvantages of using these methods is the use of chemicals, which leads to climate pollution. Therefore, a suitable instrumental method can be a good alternative to the old methods. Due to the similarity of the sample matrix, the use of X-ray machine was studied. First, 19 samples of mine soil were selected and ground. The basis for selecting the samples was that their copper concentration was in the range of 004 to 2.0%. First, the amount of copper and iron in the samples was analyzed by chemical dissolution and reading by atomic flame absorption apparatus [3]. Then, the sample size of 250 micrometers was selected by sieving [4]. Then the tablets of these samples were prepared and the X-ray machine was calibrated. Calibration of copper is in the range of 0.04 to 1.87% and iron is in the range of 1.70 to 14.29%. Finally, the amount of copper and iron in one hundred samples of mine soil was analyzed by this program. The error rates of copper and iron results with this method compared to the wet method were 0.52 and 1.16%, respectively, which is satisfactory. This method leads to much less climate pollution. For annual dissolution of Sarcheshmeh mine soil samples, the consumption of drinking water, hydrochloric acid and nitric acid are 30,000, 1,500 and 300 liters, respectively, which leads to the production of 55,415 liters of NO_2 gas (air pollution), 140 kg nitrate (water pollution), 637 kg chloride. (Water pollution) and 10.1 kg of sulfur (water pollution). While in X-ray method, the contaminants were removed. And the results are satisfactory.

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Multivariate experimental design for construction of a new selective optical sensor for determination of cobalt in water samples

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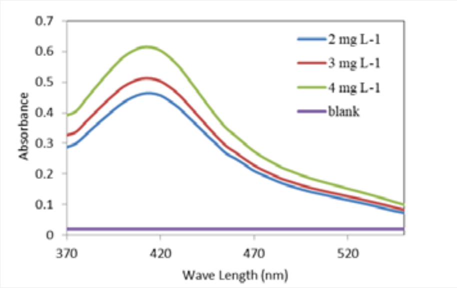

Abstract: Cobalt is a rare metal that has a variety of commercial, industrial and military applications, and it is an important essential element in the performance of many biological processes, such as in the molecular structure of vitamin B12 [1, 2]. This research study is focused on the fabrication of a new, highly selective and simple optical membrane for the determination of cobalt (II) ions in aqueous solutions. To eliminate the influence of the concomitants species and increase the selectivity in the determination of cobalt (II) in real samples, it was first oxidized using a hydrogen peroxide (H_2O_2) solution. The obtained cobalt (III) ions were then determined by recording the absorbance intensity of the new selective and sensitive thin optically transparent sensor. In the optical sensor, 1-nitroso-2-naphthol (1N2N) was used as the selective ligand. 1N2N is used as a sensitive, selective and stable complexing ligand that changes the absorbance of the transparent membrane in the presence of cobalt (III). The ready-made optical sensor displayed good reproducibility and relatively long lifetime and stability with appropriate linear response from 0.1 to 5.0 mg L^{-1}. Its detection limit was obtained 0.03 mg L^{-1}. Further investigations were evaluated intra-day and inter-day precision (RSD %) 2.76-4.46% and 0.89-5.39%, respectively and the mean percentage relative error (RE %), were in the 1.89 to 3.41 range. The results indicated that the presented method could be successfully used to determine cobalt (II) in different water samples including tap water, seawater and spring water, without any pretreatment.

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Spectrophotometric Determination of Trace amounts of Sulfate impurity in Lithium Carbonate used as cathode material for Lithium Ion Batteries

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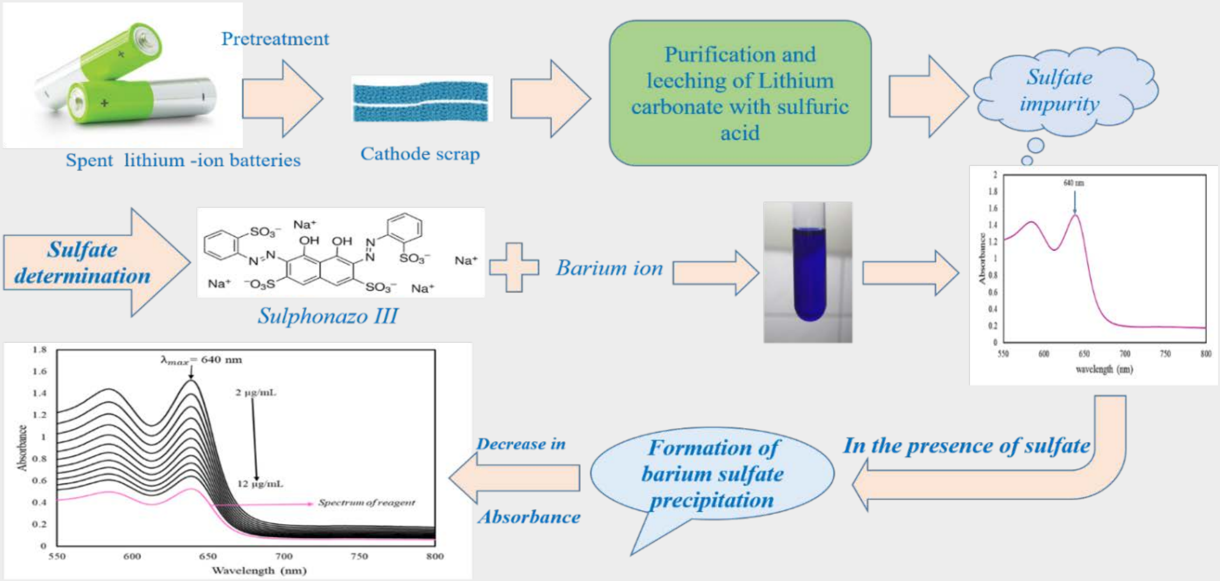

Abstract Lithium carbonate (Li_2CO_3) is a key raw material that is used to prepare cathode materials in a Li-ion battery. Also, any impurities present in the raw materials used to make the battery can affect its performance and lifespan. Therefore, impurity analysis of battery-materials is vital for suppliers of the raw materials, battery manufacturers, and emerging industries such as the electric vehicle power battery sector. One of the impurities is sulfate ion. In this work, we developed a simple and selective method for the determination of sulfate ion impurity in Lithium carbonate. The method is based on reaction of sulphonazo III with barium ion, that the absorbance of barium - Sulphonazo I11 chelate was decreased in the presence of sulfate because of formation of barium sulfate complex. The decrease in absorbance is proportional to the concentration of sulfate ion. Under optimal conditions, at λ_{max}= 640 nm, the linear range of determination of sulfate was 2.0-12.0 µg mL^{-1} with detection limits of 1.0 µg mL^{-1}. The validity of the method was evaluated by means of the data statistical analysis. For this purpose, the method was applied to the determination of sulfate in Lithium carbonate and the results were statistically compared with those obtained by the by HPLC method using t- and F-tests. There was no significant difference between the mean values and the precisions of the two methods at the 95% confidence level. The proposed method was successfully applied for the determination of sulfate in Lithium carbonate with good recoveries ranged from 99-104%, and RSD less than 2.0 %. The results showed that the proposed method offers an accuracy and reliable approach for the determination of sulfate in Lithium carbonate raw materials, and can be suggested as a routine method in battery quality control laboratories.

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A Novel Fluorescent Chemosensor Based on Phosphorus Ylides for Sensitive Detection of Copper Ions

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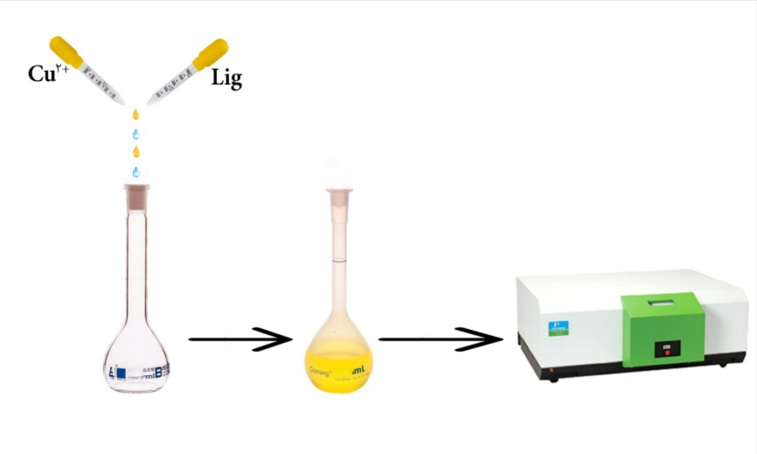

Abstract: Copper is an abundant transition metal with very high flexibility and thermal-electrical conductivity. This element is one of the few metals that exist in metallic form in nature. Cu^{2+} ions play a crucial role in the metabolism of carbohydrates and lipids. So, this ion is essential for human, animals and plants. But high concentration of copper in the environment leads to water and food contamination. Thus, development of a simple and sensitive method for determination of Cu^{2+} is of great attention. Various techniques including spectrophotometry, spectrofluorometry, electrochemistry, chromatography and radioactive analysis, have been used for the determination of Cu^{2+} ions. Among these methods, spectrofluorometry has the advantages of simplicity, speed, sensitivity and cost-effectiveness. Therefore, in the present study, a novel fluorescent chemosensor based on phosphorus ylides coupled to spectrofluorimetric analysis was used to detection of copper ions. The intensity of emission was increased by addition of copper ions. To enhance the sensitivity, the factor affecting the emission such as pH, reaction time and ligand concentration were optimized. Under the optimum conditions the calibration curve was linear over the range of 0.1 – 5.0 µM and limit of detection was 0.03 µM.

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Determination of Phosphate Anion in Food Samples by Near IR Spectrophotometry

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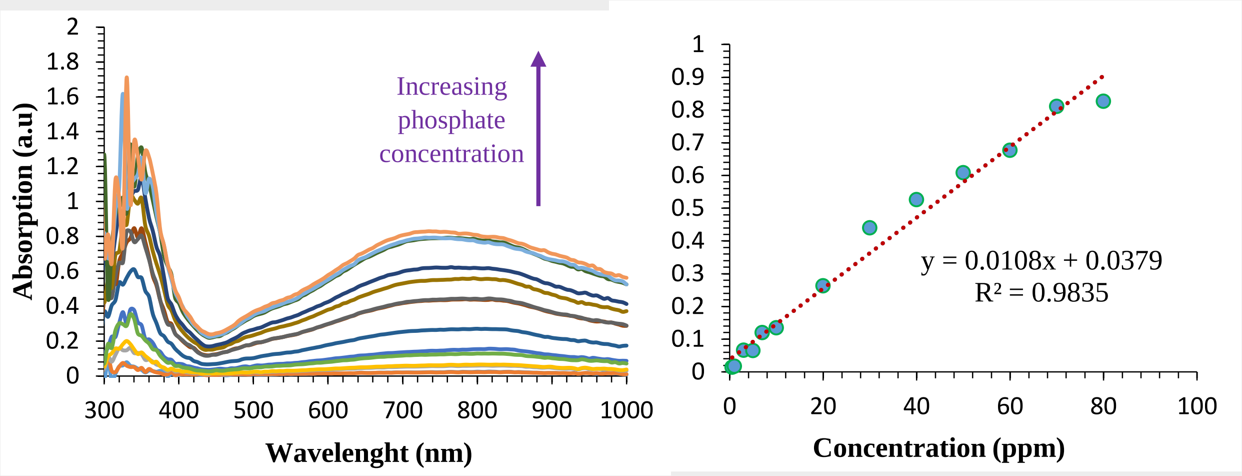

Abstract: Phosphorus (P) is a required nutrient for life, it plays a main role in the metabolism of both plants and animals. It is a growth-limiting nutrient, which makes it an important parameter to control in the environment. Phosphate exists in three forms: orthophosphates (mainly), condensed phosphates, and organic phosphates [1]. Several analytical methods have been reported for the determination of orthophosphates such as chromatography, electrochemical, fluorescence, and molybdenum blue (MB) spectrophotometric method [2]. In this work, the amount of phosphate anion in food samples was measured by NIR-spectrometry method. The measurement was based on the formation of molybdophosphoric acid by adding the defined amount (48 mM) of ammonium molybdate to beverage samples (soda). Then, the reduction of molybdophosphoric acid complex (Mo(V)) was done by the addition of ascorbic acid to form molybdenum blue complex (Mo(VI)) in strong acidic conditions (pH: 1-2). The reaction conditions as well as the various experimental parameters affecting the development and stability of the prepared coloured complex were investigated and optimized for the quantitative determination of phosphate existing in various samples. This complex has a maximum absorption at 815 nm, which can be used to determine the amount of phosphate in beverage samples. So, different concentrations of phosphate (0.5-80 ppm) were prepared to form the molybdenum blue complexes; then, the absorbance of each sample at 815 nm was measured with three repetitions (RSD = 5%). The calibration curve showed a good correlation coefficient (R^2) of 0.98. To examine whether light scattering is interfering or not, the absorption of the molybdenum blue complex was measured by the dual-wavelength method. The results show that the NIR spectrometry method based on the formation of the molybdenum blue complex is a good candidate to measure the phosphate anion in food samples as it provides fast, easy, and reliable measurement.

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Green Synthesis of Carbon Dots from Pumpkin Seeds for Fluorometric Determination of L-Cysteine

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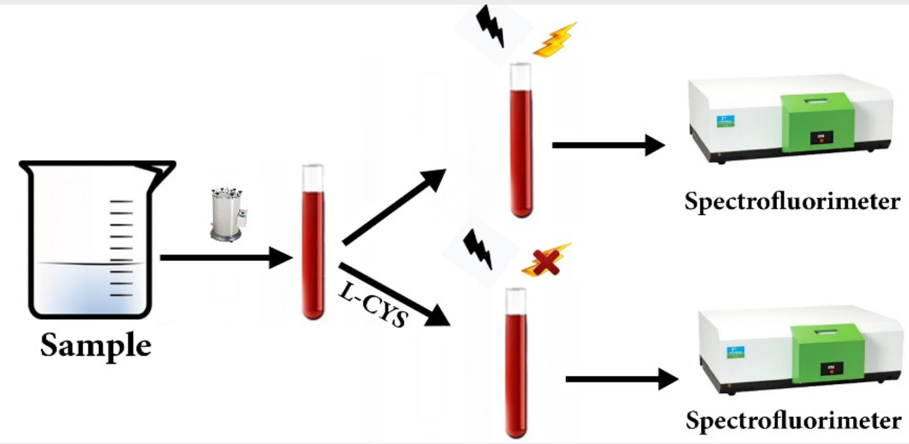

Abstract: L-Cysteine (L-Cys) is an important sulfur-containing compound which is extensively used in the food industry, environmental research, drugs formulation and cosmetics. An abnormal level of L-Cys can cause heart diseases, liver problems, L-Cystinuria, and Acquired Immune Deficiency Syndrome. Accordingly, detection of low concentrations of L-Cys using a sensitive, rapid, selective and cost effective analytical method in the environmental, industrial and biological matrixes is of great interest. Various techniques including spectrophotometry, chromatography, capillary electrophoresis, flow injection analysis, electrochemistry and spectrofluorometry were used for determination of L-Cys. In recent years, fluorescence carbon dots (CDs) have been widely applied as fluorescent probes to monitor analytes due to their excellent optical properties. Thus, in this work, a sensitive and selective biosensors based on carbon dots prepared from pumpkin seeds coupled to fluorometric technique was design and successfully used for determination of trace amount of L-Cys. After addition of L-Cys to the carbon dots solution, the fluorescence intensity was decreased. Experimental factors affecting the fluorescence intensity such as pH, amount of carbon dots, reaction time and salt concentration were investigated and optimized. Under the optimum conditions, the calibration curve was obtained over the range of 5 – 500 nM and limit of detection was 1.5 nM.

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pH-Triggered Cross-Reactivity for Visual Pattern Recognition of Antidepressant Drugs

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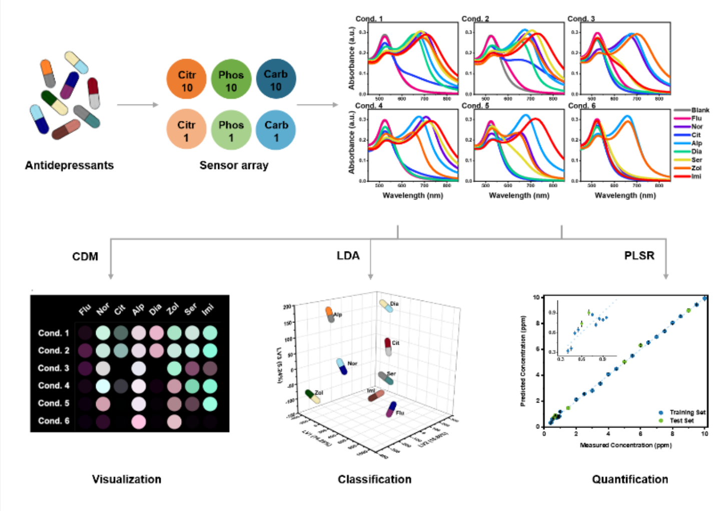

Abstract: Pattern-based sensing with multi-component sensor arrays, despite its merits, may be laborious and time-consuming. As an alternative approach, herein, a condition-based single component sensor array has been provided which represents an elegantly simple, low cost and minimally instrumented format for the quantification and classification of antidepressant drugs. Tuning the pH and ionic strength enabled the single component probe to interact with the target analytes through different binding modes, providing the required cross-reactivity. Gold NPs are promising candidates for the design of colorimetric sensor arrays and have been widely applied in this field due to their facile surface functionalization, tunable surface plasmon band and ease of control over their size, shape, morphology and aggregation state during the synthesis step or post modifications [1]. To date, a high number of AuNP-based colorimetric sensor arrays have been developed with the aim of addressing important analytical challenges [2-3]. None of them have yet reported the array-based sensing of antidepressant drugs. To generate the array, a single AuNP was embedded into six experimental conditions: three different buffers each at two concentration levels. The analytical figures of merit of the sensor array exhibited excellent sensitivity and selectivity results towards antidepressants and low limit of detections (as low as 0.009 µg mL^{-1}). The prediction accuracy (R^2_{pred}) for the identification and determination of the antidepressants over their wide concentration ranges were all above 0.99. These metrices verified that the condition-based multiplex sensing approach is precise and accurate for both the discrimination and quantification of the anti-depressant drugs. The results of multivariate calibration ($R^2_{cal}> 99\%$ and $R^2_{cv}> 99\%$) and discrimination ($R^2_{cv}> 98\%$) of antidepressants in human urine ensured the practicability of the array in complex biological fluids. Furthermore, the wide-ranging colorimetric responses that appeared due to different aggregation patterns allowed visual detection.

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Indirect determination of cyanide by Vortex-assisted liquid-liquid microextraction prior to UV-Vis spectrophotometry using methylene blue method

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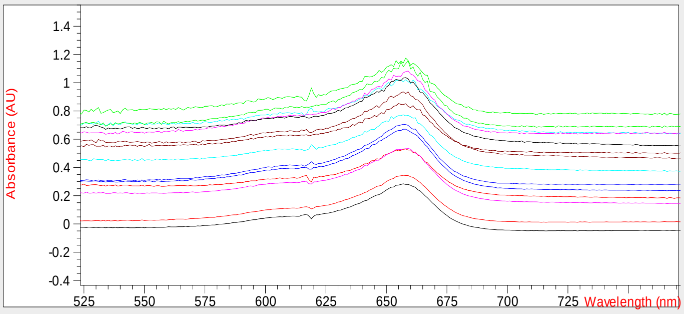

Abstract: Cyanide is extremely toxic and hazardous for human beings. The natural generation or accidental release of cyanide from the wastewater of industrial processes such as electroplating, metallurgy, Nylon and acrylic plastics production, ore leaching and gold mining, lead to contamination of aquatic environments. The World Health Organization (WHO) and the European Union have set the maximum permissible level of cyanide in drinking water to 70 and 50 μg L^{-1} [1, 2], respectively. Electrochemical and chromatographic methods have been reported for the accurate and sensitive measurement of cyanide in water samples. In this research, the FeS particles are added to cyanide solution then is mixed with stirrer for 10 min. Ferrocyanide complex is formed and the result, sulfide ions are produced in solution. The leaching solution was separated through a filter paper from FeS particles. Sulfide ions reacts with N, N-dimethyl-p-phenylenediamine in the presence of ferric chloride to produce methylene blue. To determine trace amount of sulfide, a pre-concentration method is used. A vortex-assisted liquid-liquid microextraction method along with UV-Vis spectrophotometric method based on methylene blue formation reaction for selective and sensitive determination of sulfide ions. Methylene blue is extracted into 1,2-dichloroethane organic solvent and measured at the wavelength maximum of 658 nm. The peak height of transient signal was proportional to cyanide concentration and was monitored for quantitative analysis. The influence of various chemical and microextraction relevant parameters has been investigated and the performance of the method optimized. At the optimized conditions, the proposed method successfully detected cyanide, which the detection limit obtained as 0.025 ng mL^{-1}. The Effects of several potential interferences have studied. The method was successfully applied to determine cyanide in wastewater (electroplating) and various types of water samples.

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Magnetic Dispersive Solid Phase Extraction of Silver ions

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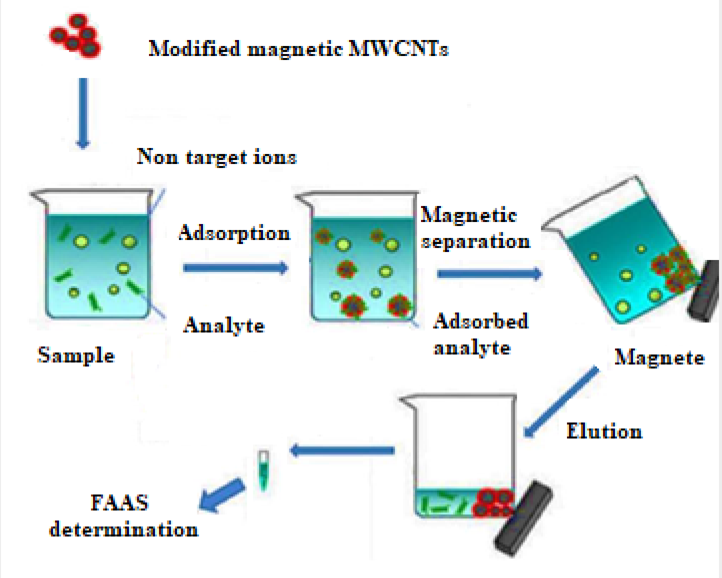

Abstract: Common sorbents applied in SPE suffer from some disadvantages including low selectivity toward the analyte, low stability as well as low sorption capacity. To solve these problems, nanostructured materials are promising due to their high surface area, enhanced active sites, and abundant functional groups on their surfaces. Among these materials, carbon nanotubes are interesting sorbents owing to their unique properties of extremely high surface-to-volume ratio, as well as good electrical and thermal conductivities [1]. However, they have some limitations such as hydrophilicity, small size, and high dispersibility. Moreover, they have low selectivity toward metal ions. The problem of separation can be overcome by the combination of carbon nanotubes with magnetic nanoparticles [2] and the selectivity toward metal ions can be achieved through the modification of carbon nanotubes surface with suitable chelating agents.

In this study, the magnetic multi-walled carbon nanotube (MWCNT) coated with diphenylthiocarbazone was synthesized and characterized by FT-IR, XRD, FESEM, and VSM. The prepared sorbent was applied for dispersive magnetic solid-phase extraction of trace silver ions from aqueous solutions before flame atomic absorption spectrometric determination. Important factors affecting the extraction of the analyte were investigated and optimized. Under the optimized conditions, the limit of detection based on 3sb/m was 0.15 µg L^{-1} and the maximum sorption capacity of the sorbent was 42.7 mg g^{-1} of silver ions. The relative standard deviation at 10 µg L^{-1} of silver (n = 5) was 2.8%. The method was successfully applied to the determination of silver ions in different water samples.

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Production of Solid Lipstick with Domestic Raw Materials, Comparison with Foreign Products and Measurement of Heavy Metals in Them by Atomic Absorption Spectrometry

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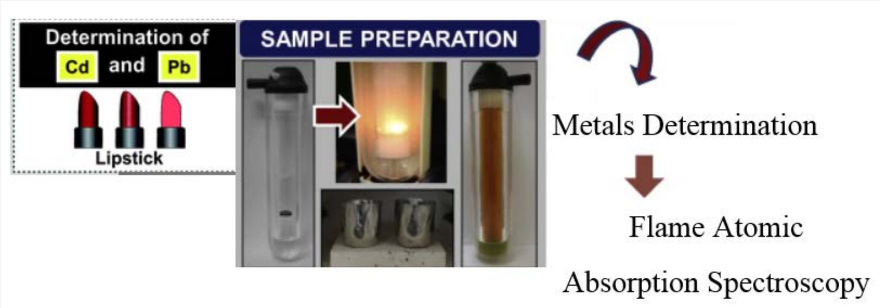

Abstract: The purpose of this research is to produce a solid lipstick product from domestic raw materials and to prevent the export and support of production prosperity. On cosmetics, especially liquid lipsticks, excessive use of which can have many side effects has always been and will be important. On the other hand, production with domestic raw materials has made the business prosperous and it is possible to control heavy metals such as lead and cadmium from the source of raw material production. In addition, the methods of analyzing these products and especially measuring heavy metals, such as Cd^{2+} and Pb^{2+} ions, are of special importance. In addition of Cd^{2+} and Pb^{2+} ions determination using atomic absorption spectrometer after dry ashing sample preparation, we also use other tests such as the softening point in the dry matter and microbial tests to ensure the quality and health of the product. The amounts of Cd^{2+} and Pb^{2+} ions in the sample produced by domestic raw materials was determined 4.75 and 4.72 ppm respectively, which is lower than the amount of these ions in the foreign lipstick goods and the samples that produced with foreign raw materials. Therefore, it was concluded that the lipstick produced with domestic raw materials, in addition to having good physical quality, contains much less heavy metals than the foreign goods available in the Iranian market and the goods made with foreign raw materials.

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Cu(II) Modified Magnetic Chitosan Nanocomposite for SPE-trace Detection of Alizarin Yellow GG

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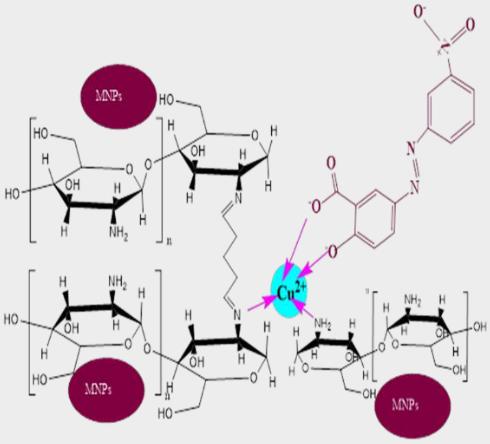

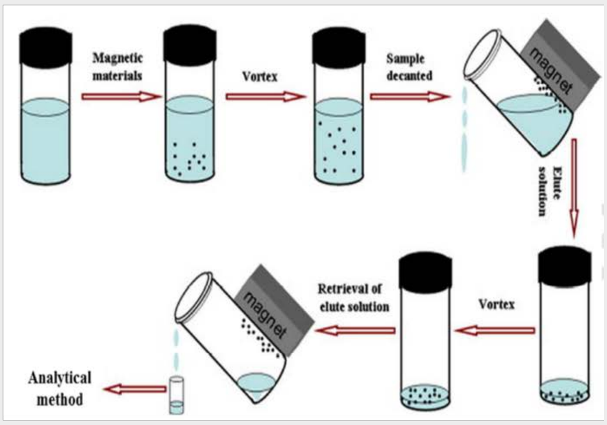

Abstract: The rapidly growing world population and rapid industrialization has caused the dyes to be widely used in some main domains such as textile related industries. Many of the dyes are generally harmful and toxic to living creatures and mammals. One of the groups of the dyes are the azo based salicylic dyes. Alizarin yellow GG (AYGG) is one of the dyes with carcinogenic effects which causes serious problems to aquatic vegetal life and the environment. The effects are raised mainly due to its structural stability affording weak thermal and biological degradation [1].

Magnetic solid phase extraction (MSPE) has been developed based on the use of magnetic nanoparticles (MNPs). MNPs usually introduce a high surface area to volume ratio preparing the great extraction capacity and efficiency. Furthermore, MNPs are suitably dispersed and have superparamagnetic properties for easy magnetic collection and re-dispersion in the solutions [2].

A cross-linked magnetic chitosan nanocomposite was prepared and its structure and morphology were characterized by different spectroscopic and microscopic techniques, respectively. The preliminary experiments showed that the adsorbent was efficient for the adsorption of AYGG when Cu(II) was present in the suspension. Different parameters related to the adsorption and elution steps were considered during the optimization. The studies showed that a solution containing ammonia and sodium hydroxide was efficient on the both elution of AYGG (from the adsorbent) and colorimetric-detection of AYGG at 452 nm. The method introduced the linearity of absorbence against the AYGG concentration in the ranges of 2-50 (R^2 = 0.9979), 50-200 (R^2 = 0.9933) and 200-2000 (R^2 = 0.9965) nmol L^{-1} for AYGG. Also, a limit of detection equal to 0.7 nmol L^{-1} was found for AYGG. Further experiments showed that the accuracy, reproducibility, repeatability and selectivity of the method were excellent. Different soil and water samples were successfully analyzed according to the appropriate standard addition methods.

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Adsorption of Sulfasalazine by Using a Cu(II) Modified Magnetic Chitosan

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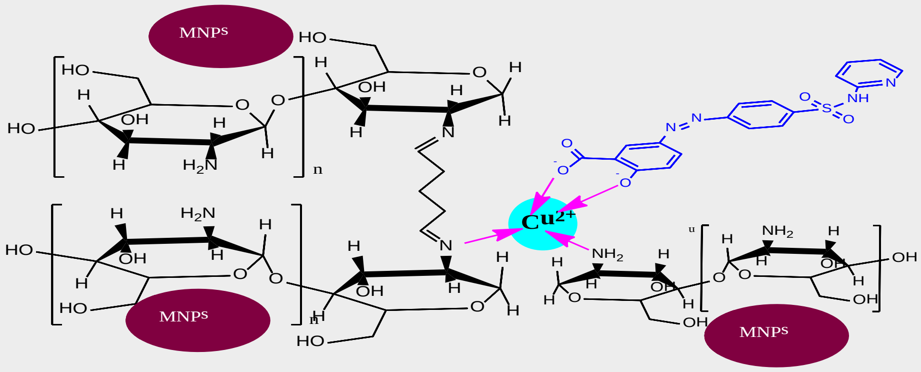
Graphical abstract



Abstract: Groundwater contamination by pharmaceutical ingredients such as antidepressants, analgesics, contraceptives, antibiotics has become an environmental problem of widespread concern. Antibiotics are probably the most successful family of drugs so far developed to improve human health. Sulfasalazine (SSZ) is a synthetic drug and a combination of antibiotic (sulfapyridine) and an anti-inflammatory agent (5-aminosalysilic acid). Many methods have been attempted for the removal of antibiotic drugs from different water sources [1]. Magnetic nanoparticles (MNPs) have been extensively studied as an efficient adsorbent with large surface area causing small diffusion resistance for the separation of pollutant chemicals such as dyes, metals and pharmaceuticals [2]. Moreover, MNPs cause advantages on ease of separation and fast recovery by employing an external magnet due to prevention of the additional centrifugation or filtration processes [3].

In this work a magnetic cross-linked chitosan nanomaterial was prepared through the reaction of glutaraldehyde with chitosan in the presence of magnetite nanoparticles. The characteristics of the nanocomposite were obtained by using appropriate spectroscopic and microscopic techniques. Then, the magnetic nanoadsorbent was used to study the adsorption and extraction of SSZ from the effluents in the presence of Cu(II). The isotherms, kinetics and thermodynamics of the SSZ adsorption on the bio-nanosorbent were investigated. The obtained results revealed that the adsorption of sulfasalazine on the sorbent occurs on a homogenous monolayer and is an endothermic process. The applicability of the nanoadsorbent on the SSZ separation from lake, well and river water samples was considered. A certain amount of the magnetic nanoadsorbent (20 mg) was employed for 6 consecutive adsorption and elution (with ammonia) steps. The results showed that the adsorption and elution yields were about 97% and 86-90%, respectively.

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Measurement of Nitrate and Nitrite by Griess Method with Aromatic Amines and the Effect of Suitable Reducing Agents

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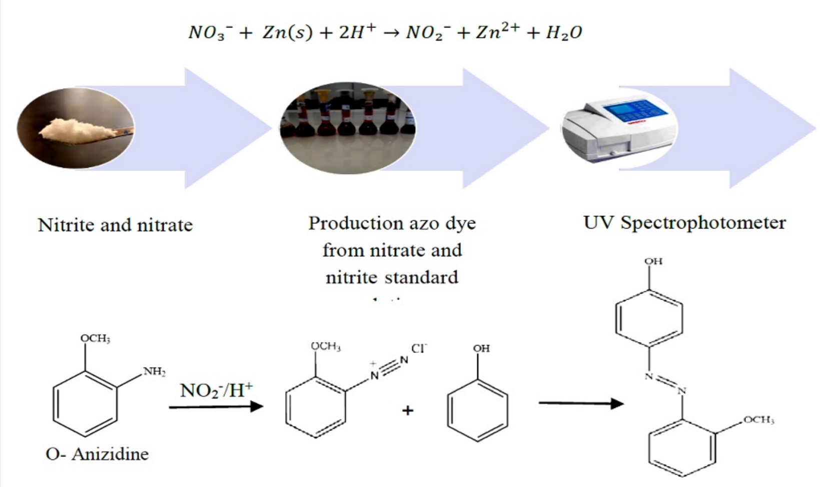
Graphical Abstract



Abstract: Nitrate and nitrite are important in environmental samples such as water, soil, feed and agricultural products. These ions are repeatedly analyzed to detect contamination levels. These ions are used in the preparation of chemical fertilizers, food additives, etc [1]. High nitrate concentrations lead to accused globin in aquatic animals and baby blue syndrome in human infants. Nitrite produces N-nitroseamine carcinogen by reacting to secondary amines in the human body. Nitrite plays an important role in various aquaculture diseases and is harmful to humans if available in drinking water. Therefore, determination of nitrite is essential for environmental protection and public health [2]. Among the method based on nitrate and nitrite measurement, spectroscopic method due to its easy availability and high sensitivity, low detection limitation cost effectiveness and easy procedure are discussed in this paper. UV spectroscopy due to its versatility, simplicity and feasibility is an extensive method for determining nitrate in water samples. Griess assay is based on the production of azo dye from a suitable aromatic amine in acidic media nitrite followed by the coupling reaction. Therefore, a highly colored azochromophore of nitrate is produced whose concentration is determined in the region of 500-600 nm to evaluate nitrite concentration. In order to measure nitrite, since nitrate is unable to produce diazonium salt, it must be revived by various chemical methods such as the nitrite use of transition metals such as zinc, cadmium, ascorbic acid and hydroxyl amine as reducing agents [3].Various modifications have been made to this basic methods, such as using different reducing agents and phenolic and aromatic amines.

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Carbon Quantum Dots as a Fluorescent Sensing Platform for Amoxicillin Detection.

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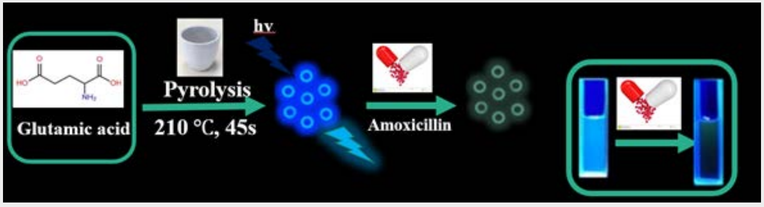
Graphical Abstract



Abstract: Quantum dots (QDs) are semiconductor nanoparticles with unique optical and electrical properties that have been extensively studied and used for analytical purposes in the last two decades due to their strong fluorescence emission and tunable nanoparticle sizes [1]. However, many QDs are toxic even at relatively low concentration. One of the most recently discovered QDs are carbon dots (CDs) or carbon quantum dots (CQDs) [2]. Due to ease of synthesis methods, low cost, low toxicity, strong fluorescence emission, environmental compatibility and abundant synthesis sources, applications of CQDs has expanded in the fields of bioimaging, drug delivery and chemical sensors [3]. Amoxicillin as an antibacterial drug, strengthens the immune system and is used to treat bacterial infections [4]. In this study, carbon quantum dots were synthesized using hydrothermal method and used for measuring amoxicillin based on fluorescence quenching technique. Preliminary studies have shown that the intensity of CQDs fluorescence (λex=350 nm and λem=440 nm) is related to the concentration of amoxicillin, in which increasing its concentration reduces the fluorescence intensity of CQDs. Based on these observations, the effect of various factors such as pH, temperature, time and ionic strength of the analytical solution on the fluorescence of CQDs were studied and optimized. According to the experimental data, the linear dynamic range of 2.0-30.0 μM amoxicillin, the method detection limit of 2.0 μM amoxicillin and the relative standard deviation of 0.2 % in repeated measurements of 10 μM amoxicillin were obtained. The effect of many ionic species and different chemical compounds on the analytical signals (fluorescence intensity of CQDs) were also investigated. The proposed method for measuring the amount of amoxicillin in pasteurized milk samples was also used and evaluated.

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Multi-emission Fluorescent Sensor Array Based on Carbon Dots for Distinguish of Seven Metal Ions

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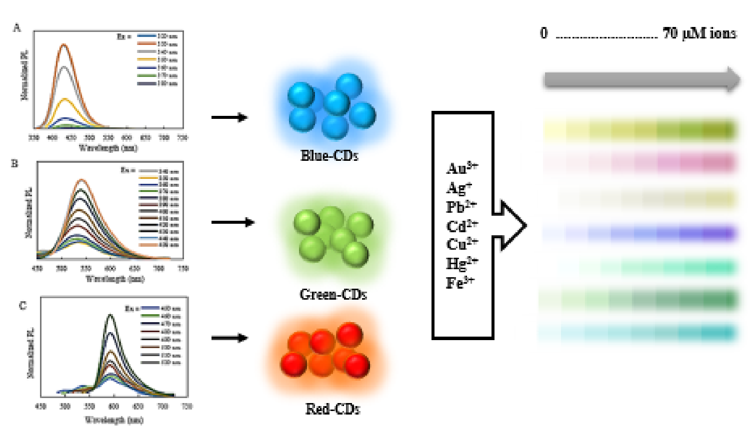
Graphical abstract



Abstract: The development of a convenient method for detection and identification of toxic metal ions is of great interest [1]. The use of carbon dots (CDs), as a new class of photoluminescent nanomaterials is rapidly expanding. Their many advantages, including water solubility, high stability, low toxicity, ease of functionalization, and cost-efficient and simple synthetic routes have introduced them as potential alternatives to conventional semiconductor-based quantum dots [2, 3]. Up to now, various fluorescence sensor arrays have been fabricated based on fluorescent carbon dots [4-6]. In this work, we have developed a facile fluorescent sensor array based on luminescence carbon dots for detecting and differentiating a set of metal ions.

Herein, we have designed three hydrothermal methods to synthesis three CDs with red, green, and blue emission for distinguishing seven metal ions (Au^{3+}, Ag^{+}, Pb^{2+}, Cd^{2+}, Fe^{3+}, Cu^{2+}, and Hg^{2+}), at pH 6. The CDs displayed various fluorescence quenching behaviors to ions, due to the presence of different functional groups on their surface. Seven metal ions were distinguished in the concentration range of 0.20 to 70 µM. In this method, we were able to design three CDs with different fluorescence emissions, and quenching responses to metal ions. Also, the PCA proved that the seven metals ions with different concentration could be discriminated from each other. The outcomes indicated that the number of sensor elements can be easily increased by changing the synthetic conditions of CDs. The use of RGB CDs with tunable optical properties will broaden an innovative way of developing efficient sensor array systems.

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An "on-off" Sensor Based on Silver Nanoprisms for the Determination of Phenytoin Sodium in Exhaled Breath Condensate

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Abstract: In the current study, a fluorescence-based detection system that uses silver nanoprisms is proposed for determination of phenytoin sodium. This probe is based on turning off and turning on the fluorescence emission related to luminol, which is performed by nanoprism and phenytoin sodium, respectively. Energy transfer from luminol to silver nanoprism reduced its fluorescence emission intensity. Phenytoin sodium adding cause the silver nanoprism aggregation and inhibits the fluorescence quenching of luminol and fluorescence intensity is increased in proportional with phenytoin sodium concentration. The experimental condition are optimized using one-a-time method and full validated in exhaled breath condensate as a chosen biological sample. The validated method is capable of technically detecting the phenytoin sodium in the range of 0.01 to 2.0 mg.mL-1 with limit of detection of 0.005 mg.mL-1 [1].

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Visible and Fluorescent Detection of Melamine with One-Step Synthesized Silver Nanoparticles Using Carbon Dots

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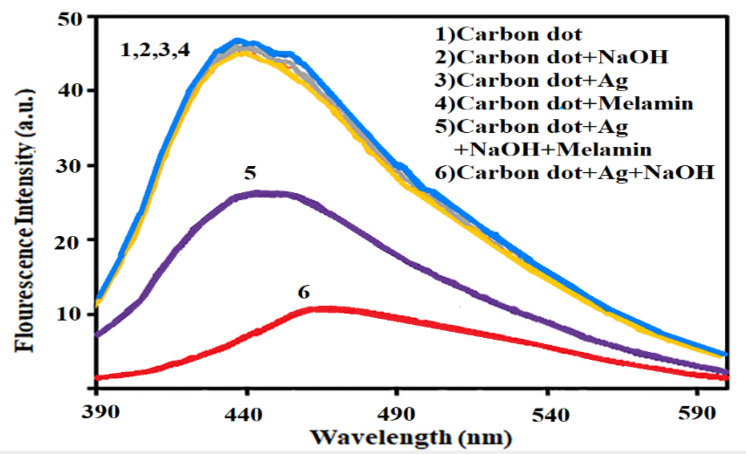

Abstract: Melamine (1,3,5-triazine-2,4,6-triamine) is an organic compound that serves as raw material for the production of melamine resins. As melamine contains a high level of nitrogen (66% by mass), the illegal addition of melamine to dairy products can lead to significant enhancement of protein content that is measured by using the Kjeldahl method. In this assay, one-step synthesized silver nanoparticles with C-dots as the reducing and stabilizing reagent were employed for visible and fluorescent detection of melamine in raw milk. C-dots played an important role in reducing silver ions and stabilizing the nanoparticles. When C-dots were added to the silver ions in alkaline solution, monodispersed AgNPs would be obtained, resulting in remarkably reduced fluorescence emission. Aggregated AgNPs were observed when melamine was present. Visual color changes corresponding to the UV−Vis absorption spectra were observed. Moreover, the fluorescence signals would be increased with the further increase of melamine. As a result, a dual optical platform combined UV−Vis absorption and fluorescence signals could be achieved through the addition of melamine into the system. However, in the presence of melamine, silver ions could interact with the nitrogen atoms in amine and triazine groups of melamine. With 0-2 µM melamine, aggregated AgNPs were found after the reduction by C-dots, resulting in color and absorbance changes. With further increase of melamine (2-20 µM), both formation and aggregation of AgNPs were inhibited, and the fluorescence was gradually increased. The results for melamine assay based on visible and fluorescent method showed the requisite sensitivity with a low comparable detection limit, as well as high selectivity.

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Selective Optical Detection of Citalopram using a Fluorescent Probe Based on Carbon Quantum Dots Embedded in Silica Molecularly Imprinted Polymer

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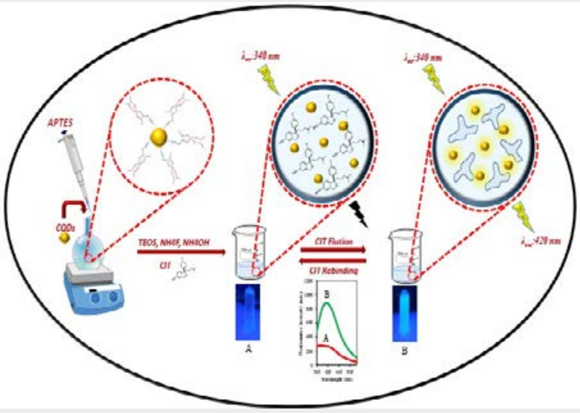
Graphical Abstract



Abstract: Citalopram is a selective serotonin reuptake inhibitor used to treat major depressive disorder, obsessive compulsive disorder, panic disorder, and social phobia [1]. Aggregation of citalopram in the body leads to dangerous side effects, including diarrhea, drowsiness, headaches, insomnia, nausea, and many sexual side effects in about 70% of patients taking this drug [2]. Regarding the wide use of citalopram, quantification of this medicine and its metabolites has great importance -. This study introduces a sensitive optical nano-sensor based on carbon quantum dots (CQDs) embedded in silica molecularly imprinted polymer (SMIP) for the detection of citalopram in biological samples. This fluorescent nanosensor was made by the polymerization of silica polymer in the presence of CQD nanoparticles as the fluorescent materials and citalopram as the template molecule [3]. After polymerization, template molecules were removed, and cavities, which were similar to citalopram in shape and size, were maintained in the polymeric matrix. The synthesized nanocomposite of CQDs embedded in SMIP (CQDs-SMIP), showed a strong fluorescence emission at 420 nm in the absence of the template molecule. When citalopram was added to the nanostructure, the fluorescence intensity was quenched. The correlation between the extent of quenching in fluorescence emission and the concentration of the added citalopram provided a highly sensitive and selective nano-sensor for the detection of citalopram. The synthesized materials were characterized using Fourier Transform Infrared (FTIR), UV-Visible (UV/Vis) and Fluorescence Spectrophotometry, High-Resolution Transmission Electron Microscopy (HR-TEM), and Field Emission Scanning Electron Microscopy (FESEM). The prepared nano-sensor responded to the different concentrations of citalopram with a low detection limit of 3.10 µg.L^{-1} over a linear range of 100 to 700 µg.L^{-1}, and the relative standard deviation (RSD) was 3.15% (n=3). This nano-sensor was applied to analyze pharmaceutical formulations of citalopram in plasma and human urine samples with remarkable results.

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Application Development and Performance Optimization of IMS Detector through an Alternating Ion Shutter and Rapidly Heated Drift Tube

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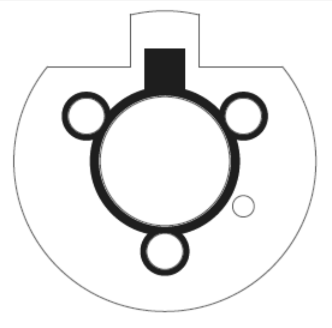
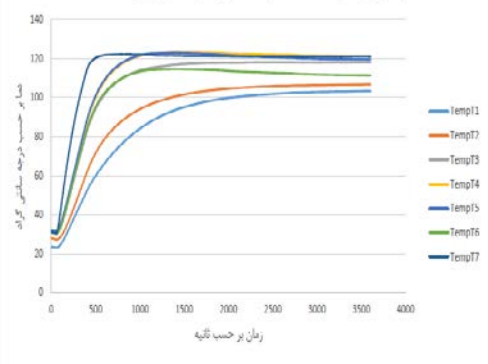
Graphical Abstract





Abstract: Ion mobility spectrometry (IMS) is an analytical method used to detect the presence of materials based on the mobility of product ions within an electric field. In this method, first the sample evaporates and then the gas molecules are ionized. The resulting ions are directed to the drift zone through an ion shutter and move towards the detector under the influence of an electric field. Qualitative and quantitative analysis and measurement of these molecules is done on the basis of analytical separation resulting from different ionic mobility of different molecules. IMS is one of the paramount methods for detecting and identifying traces of explosives, narcotics, and chemical warfare agents. This device is made in both stationary and portable forms, which has been highly utilized in the fields of military, security and environmental measurements [1]. Institute of Materials and Energy, Iranian Space Research Center has several decades of experience in the field of designing and manufacturing detector devices based on IMS technology, which has successfully developed it in various fields including device miniaturization, software upgrade, device performance optimization, and the ability to quickly switch between positive and negative modes, as well as the design and manufacture of portable instruments. The detection of some important and widely used substances by IMS technology has been faced with challenges. One of these substances is ammonium nitrate which due to the production of a peak very close to the air peak, practically overlaps with air peak and it is not possible to separate and detect it by IMS. The proposed methods to detect ammonium nitrate generally include the use of some other technologies in addition to IMS and the implementation of some hardware changes, which generally add to the complexity of the device [2-3]. In the present study an attempt was made to detect ammonium nitrate without applying these complex changes and only by using a software method. Indeed, through alternating ion shutter pulses we could control the concentration of ammonium nitrate introduced to the drift tube and detect this strategic explosive without scarifying sensitivity. The accuracy of the method was confirmed in field applications of the device. In addition, IMS drift tubes generally work at high temperature, which will cause a relatively long initial warm-up time (about 30 minutes in typical commercial devices). In order to obviate this problem, a drift tube was designed and constructed through which the warm-up time of the device was reduced to less than 15 minutes, and practically a big step was taken in the direction of better operational use of this device.

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Design and Construction of a Pulsed Corona Ionization Source for IMS Detector

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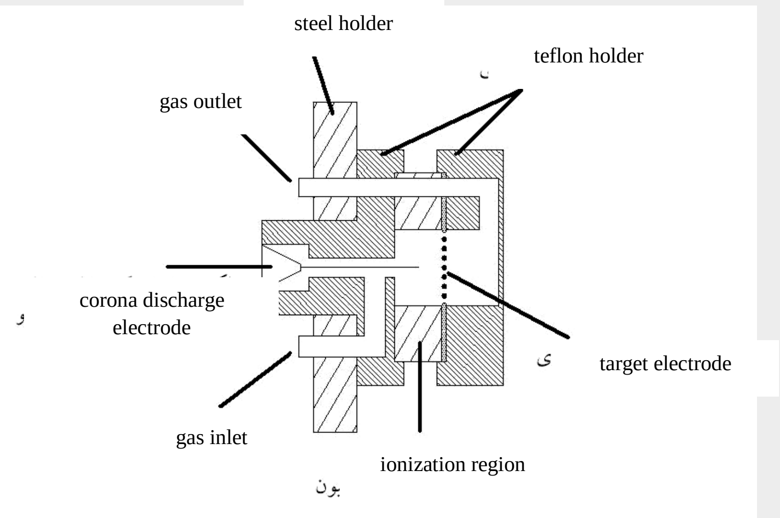
Graphical Abstract



Abstract: Ion mobility spectrometry (IMS) is a technique for qualitative and semi-quantitative measurement of organic chemical compounds. This system includes ionizing the molecules and materials resulting from decomposition and drifting these particles in an electric field. Qualitative and quantitative analysis and measurement of these molecules are based on the different ionic mobility of various molecules. Stationary and portable IMS applications in military, security, and environmental measurements have attracted the attention of many researchers to study and work in this field [1]. For a long time, radioactive radiation (such as ^{63}Ni) was used as a source of ionization in these systems. Due to the specific issues of these sources, many researchers have been looking for suitable non-radioactive alternatives to be used as an ionization source in IMS. As a suitable replacement for the old radioactive systems, the phenomenon of corona discharge was considered. The research on IMS ionization sources in the air has revealed that the reactants in the positive corona discharge mode are the same as those in the old radioactive ionization source. Recently, the use of pulsed corona discharge as a source of ionization was considered. In this method, electric pulses are used to create corona discharge. These pulses are created by applying a voltage jump (from several hundreds of volts to several kilovolts) in pulse form on two electrodes that are biased with an initial DC voltage (from zero to several kilovolts). The energy needed in pulse corona discharge will be used for the production of electron-ion pairs and not to drive the ions towards the cathode electrode. Minimizing this effect which is dominant in continuous corona, will greatly improve the energy for producing ions. Another advantage of pulse corona source is the possibility of removing the injection grid in the IMS cell [2]. There are two major advantages of using gridless IMS cells: Firstly, the number of ions entering the drift region is greatly increased. This increase is due to the elimination of ion losses on the grids, which will cause more effective use of produced ions and increase the signal-to-noise ratio in IMS. Secondly, removing the electronic, mechanical, and software parts related to the injection grid will reduce the peripheral equipment used in IMS. The two mentioned factors will play a very important role in miniaturization and optimization of IMS systems [3]. Accordingly, this study is dedicated to the structural development and replacement of the IMS ionization source.

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Production of Solid Lipstick with Domestic Raw Materials, Comparison with Foreign Products and Measurement of Heavy Metals in Them by Atomic Absorption Spectrometry

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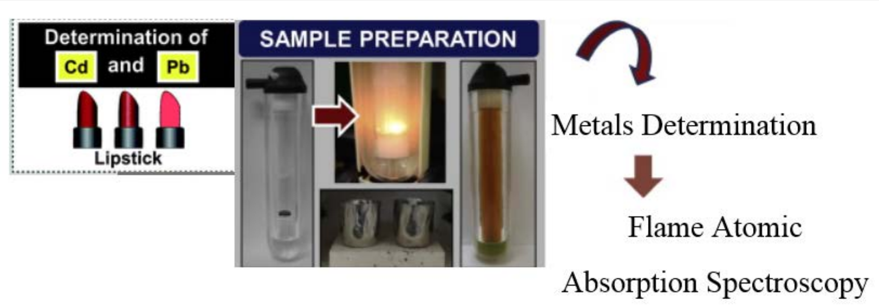
Graphical abstract



Abstract: The purpose of this research is to produce a solid lipstick product from domestic raw materials and to prevent the export and support of production prosperity. On cosmetics, especially liquid lipsticks, excessive use of which can have many side effects has always been and will be important. On the other hand, production with domestic raw materials has made the business prosperous and it is possible to control heavy metals such as lead and cadmium from the source of raw material production. In addition, the methods of analyzing these products and especially measuring heavy metals, such as Cd^{2+} and Pb^{2+} ions, are of special importance. In addition of Cd^{2+} and Pb^{2+} ions determination using atomic absorption spectrometer after dry ashing sample preparation, we also use other tests such as the softening point in the dry matter and microbial tests to ensure the quality and health of the product. The amounts of Cd^{2+} and Pb^{2+} ions in the sample produced by domestic raw materials was determined 4.75 and 4.72 ppm respectively, which is lower than the amount of these ions in the foreign lipstick goods and the samples that produced with foreign raw materials. Therefore, it was concluded that the lipstick produced with domestic raw materials, in addition to having good physical quality, contains much less heavy metals than the foreign goods available in the Iranian market and the goods made with foreign raw materials.

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Tracing Dopaminergic Agents in Human Urine by a Smartphone-Based Fluorescent Electronic Tongue

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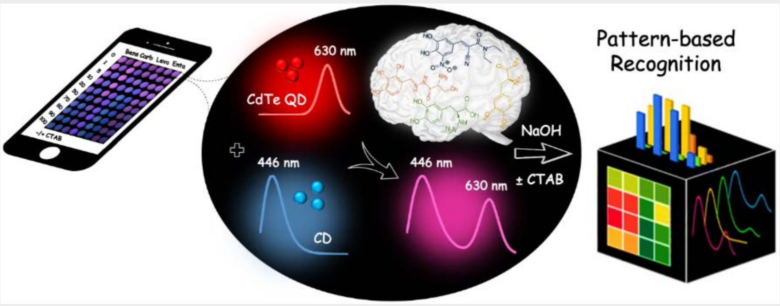
Graphical abstract



Abstract: Parkinson's disease (PD) is a progressive neurological disorder that has affected more than 10 million people worldwide. Dopaminergic agents such as levodopa (Levo), carbidopa (Carb), benserazide (Bens) and entacapone (Enta) are often prescribed to treat PD by facilitating the function of the neurotransmitter dopamine. Despite the therapeutic efficacy of the dopaminergic drugs, these agents' dysfunction contributes to PD progression and disrupts the patient’s quality of life [1]. The importance their dosage control in human biological fluids has boosted the demand for fast, sensitive and real-time multi-analyte detection. Herein, a simple and efficient electronic tongue based on fingerprint fluorimetric patterns has been developed for rapid, visual and simultaneous detection and discrimination of these agents by a single fluorometric test. The optical assay principle mainly relies on the different fluorescence quenching of the dual emissive nanoprobe in the absence and presence of cetyltrimethylammonium bromide (CTAB) at alkaline conditions, upon the addition of target analytes. The distinct assembly dynamics of the nanoprobes and the oxidized targets in the absence and presence of CTAB led to diverse aggregation levels under electron transfer (ET) mechanisms. A smartphone camera was used to take photos from the solutions in the wells and extract RGB values on which data analysis was performed. Distinct changes in the spectral profiles along with vivid and concentration-dependent color variations led to visual discrimination of dopaminergic agents in a broad concentration range (1 µM to 100 µM). In addition, color difference maps (CDMs) were used as distinct visual fingerprints for discrimination between the targets. The Linear discriminant analysis (LDA) model successfully discriminated among different concentration of targets having similar structures. In addition, excellent results of heat maps and radar plots of the data matrix were visual evidence of the remarkable classification ability of the constructed array. Furthermore, the feasibility of the array for quantification and prediction of different concentrations of target analytes in human urine, as a complicated biological media, was satisfactorily appraised by the array under an LDA model with discrimination accuracy of 100% [2]. Since simultaneous monitoring of agents is essential in evaluating the disease process, the strategy of the designed electronic tongue as a model may be a vital remedy for practical point-of-care diagnostics by any user soon.

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Environmental Analytical Chemistry

Analysis of heavy metals in vegetables cultivated in the Kashafrud's riverside, Razavi Khorasan Province, Iran

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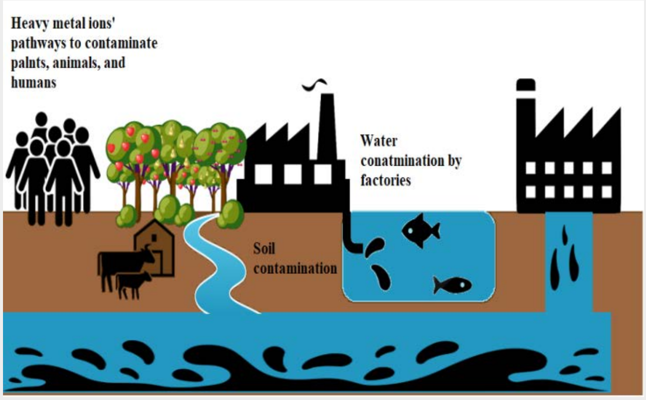
Graphical Abstract



Abstract: Nowadays, due to the risk related to eating vegetables contaminated with heavy metal ions, the safety of vegetables for consumption is becoming an increasing concern to consumers. Industrialization, unrestrained human population, urbanization, and over-usage of pesticide can be within the major reasons' category of heavy metals (HMs) contamination in the food chain. The present study was conducted to quantify some heavy metal ions and to evaluate their sources and health risks in various samples. These samples included alfalfa, barley leaves, beet, and beet leaves. In addition, these samples' agricultural soil, irrigation water, and treated and untreated wastewater of Charmshahr Industrial Town of Mashhad was analyzed for heavy metal ions including Fe, Co, Cd, Zn, Cu, Cr, Pb, and Ni by inductively coupled plasma – optical emission spectrometry (ICP-OES). Results showed that among eight heavy metal ions, five HMs including Fe, Co, Zn, Cu, and Cr were more than the safety limits given by the food and agriculture organization (FAO) and the world health organization (WHO) for human consumption. The rest of HMs showed lower levels than those of permissible amounts. Overall, the number of heavy metal ions that were exceeding the permissible values set by FAO/WHO, were beet leaves > alfalfa> barley and beet > all water samples except the beet irrigation water, respectively. The leaf of vegetables contained more heavy metal ions and were exceeding the permissible levels given by FAO/WHO as compared with the vegetable itself. To be more precise, the concentration of heavy metal ions including Fe, Co, and Cr, in the beet's leaves was more than the root of the beet. However, the concentrations of chromium in most samples were higher than the allowable level, which indicates the existence of several tanneries around the river.

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Loading of Metal Species in SBA-15 for Elimination of Organic Pollutants

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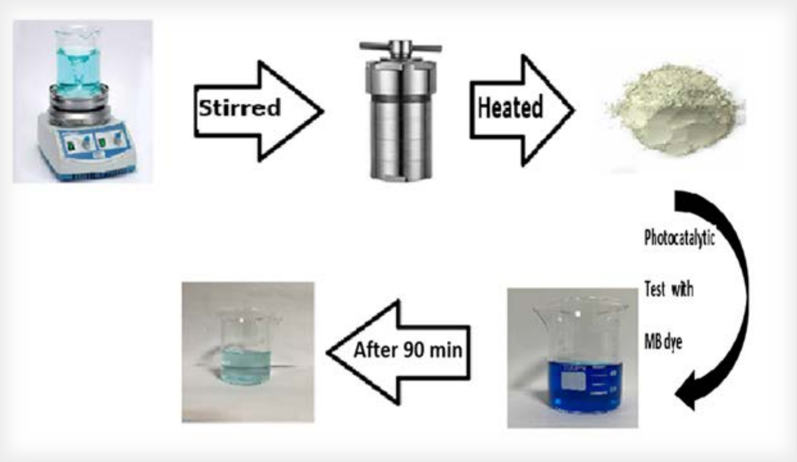
Graphical Abstract



Abstract: Today, humans are exposed to various dangerous and toxic pollutants throughout their lives. Organic compounds such as organic dyes and drugs as water pollutants are necessary to be removed [1]. Photocatalysts are widely used in the fields of water purification, pollutant reduction, air purification and degradation of organic contaminants [2]. Mesoporous silicate metals are suitable substrate for depositing metal species into their structures [3]. In this study, SBA- 15 as a silicate-based mesoporous material were loaded with some transitional metals such as Fe_2O_3 and ZnO or species including SeO_2, or its mixed oxide CuO-SeO_2 with different mass ratios. Mesoporous SBA-15 was synthesized by hydrothermal method using P123 as structure directing template. The prepared metal –inserted mesoporous materials were studied by XRD, BET, SEM, TEM, FT-IR and EDS [4]. The synthesized photocatalyst with 5-5 W% of CuO-SeO_2 showed the best photocatalytic efficiency in the degradation of methyl green and methylene blue dyes in an optimal time of 90 minutes under visible light and in the presence of the optimal concentration of H_2O_2.

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Synthesis of Magnetic Polyvinylchloride/Polypyrrole Nanocomposite; Investigating of Adsorptive Removal of Cationic Dye of Crystal Violet from Aqueous Solutions

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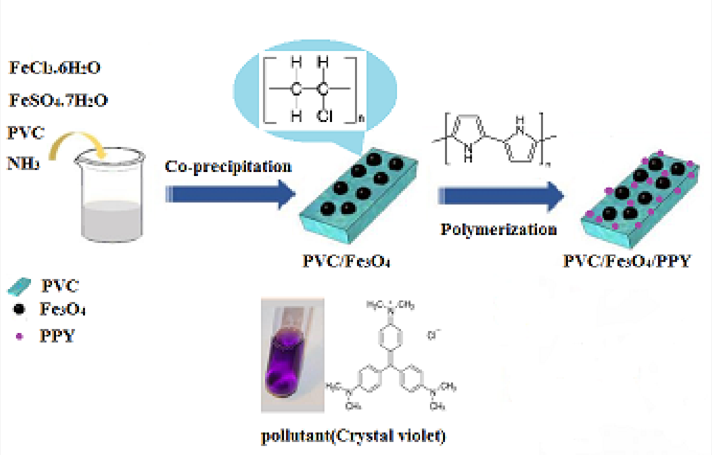
Graphical Abstract



Abstract: One of the most important directions for environmental remediation is the effective removal of dyes and toxic heavy metal ions from water sources using newly fabricated nanoadsorbents [1]. Polypyrrole-based (PPY) composites are promising candidates for water and wastewater treatment [2]. The most suitable method among pollutant removal methods is adsorption due to process simplicity, high efficiency and low cost [3]. Herein, nanocomposite PVC/Fe_3O_4/PPY in order to removal of dye from aqueous solutions by a simple and low cost method was synthesized. First PVC/Fe_3O_4 was synthesized via co-precipitation method and then the monomer of pyrrole was polymerized onto it [4]. The properties of the prepared nanocomposite were characterized by the X-Ray Diffraction (XRD), Diffuse Reflectance Spectroscopy (DRS), Vibrating Sample Magnetometer (VSM), Fourier Transform Infrared (FT-IR) and Field Emission scanning electron microscopy (FE-SEM) techniques. In order to obtaining the optimum conditions for achieving maximum adsorption capacity, the effect of various parameters such as pH, contact time, pollutant concentration, adsorbent dosage, temperature, etc. were studied. The results obtained discovered that percentage removal of dye increases with pH increasing up to pH=10. It was found that the percentage of crystal violet (CV) dye removal at pH of 10 with 50.0 mg of adsorbent, initial dye concentration of 50.0 mg L^{-1}, in contact time of 40 minutes was above 93%. According to the carried out kinetics and isotherm investigations, it was found that the adsorption process are well described by the pseudo-second-order synthetic model ($R^2 = 0.998$) and the Langmuir isotherm model ($R^2=0.992$). Based on Dubinin-Radushkevich model, the adsorption of crystal violet on PVC/Fe_3O_4/PPY was temperature dependent, spontaneous and endothermic (ΔS and ΔH are 260.0 J $mol^{-1}K^{-1}$ and 72.0 KJ mol^{-1}, respectively). The reusability of the adsorption-desorption cycle of PVC/Fe_3O_4/PPY was studied to 3 cycles, and it was found that CV dye removal efficiency was unchanged after at least 3 cycles.

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Rapid determination of nitrate by colorimetric thin film based on sol-gel method

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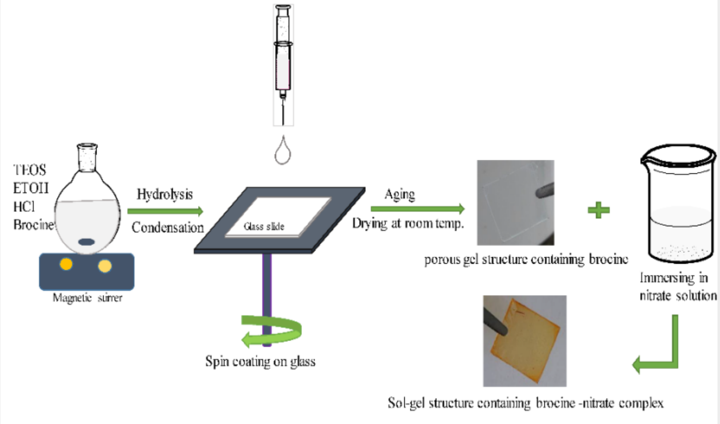
Graphical Abstract



Abstract: Nitrate has been added intentionally at the curing process of certain meat products, due to their ability to inhibit the outgrowth spores of clostridium botulinum and to impart characteristic color and flavor to this kind of foodstuffs. The significance of nitrate to human health is related to the fact that nitrate, after being metabolized or reduced to nitrite, can react with secondary or tertiary amines to form N-nitroso compounds, which are potent carcinogens. We herein describe the fabrication of a brucine incorporated sol-gel glass with nanoporous structure as an optical sensor to measure nitrate. Tetraethyl orthosilicate was used as precursor to form the porous polymeric network for entrapment of brucine in porous silica gel matrix. In order to modify pore size, quality and leaching behavior of the sol-gel network, several sols were prepared with different sol-gel pHs and water: alkoxide ratios. Our thorough experimental-based results revealed that the porous feature of the matrix as well as its average pore size could play significant roles in almost all critical parameters, affecting the sensor performance, including sensitivity, selectivity and response time of sensor. The FE-SEM technique equipped with EDAX was also employed to evaluate the surface morphology, porosity growth and elemental identification of the thin films. The constructed sensors showed optimum performance under the working conditions, including water: alkoxide ratio of 4:1, water acidity of 1 M. Under optimum conditions, a linear calibration curve over the range 2.0 to 8.8 $\mu g\ mL^{-1}$ of nitrate was obtained, along with a detection limit value of 0.5 $\mu g\ mL^{-1}$. Meanwhile, the sensor exhibited excellent RSDs for both intra-day and inter-day precision.

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Smartphone-Based Colorimetric Method for Determination of Water Solutions pH

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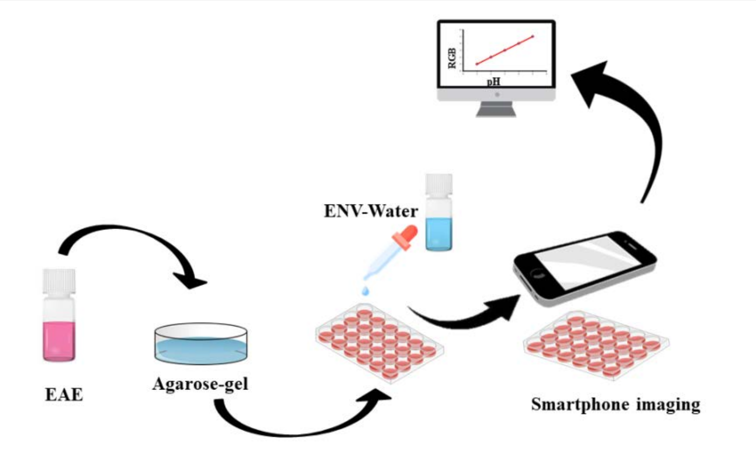
Graphical Abstract



Abstract: Due to the importance of pH measurement in different aqueous environments and its effects on living creatures [1-3], the aim of this study is to present a new, simple, sensitive, and inexpensive analytical method, smartphone-based colorimetry, for pH measurement in water samples. In other hand, smartphone-based analysis has recently emerged as useful tool, and it has been found to be promising in several fields including point-of-care analysis [4-6]. In this work, an Android application for measurement of pH determination is presented. An optical sensor, based on the natural pigments (*Echium amoenum* anthocyanin's) doped on the agarose-gel substrate, was designed and used to measure pH in different aqueous samples. The extracted anthocyanin from *E.amoenum* (EAE) solution exhibited good color variations when exposed to pH 2 to pH 12. The effective variables, which affect the sensor response, include pigment concentration, temperature, shaking rate and stirring time in the extraction of the pigments, the amount of doped pigment, and the casting and molding type of the monolith are studied and optimized. The response times of the EAE doped sensor obtained in less than 20 seconds and linear ranges are obtained in the pH range of 2.00 to 12.00 respectively. The relative standard deviation of the sensor with seven repetitions is less than 1%. The designed sensor's performance has been evaluated by comparing the experimental data with the commercial-grade pH-sensing tool. The results confirm good use of a mobile phone as an analytical instrument in pH determination.

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Ion mobility spectrometry for evaluation of bovine serum albumin nanoparticles ability to adsorb nalidixic acid

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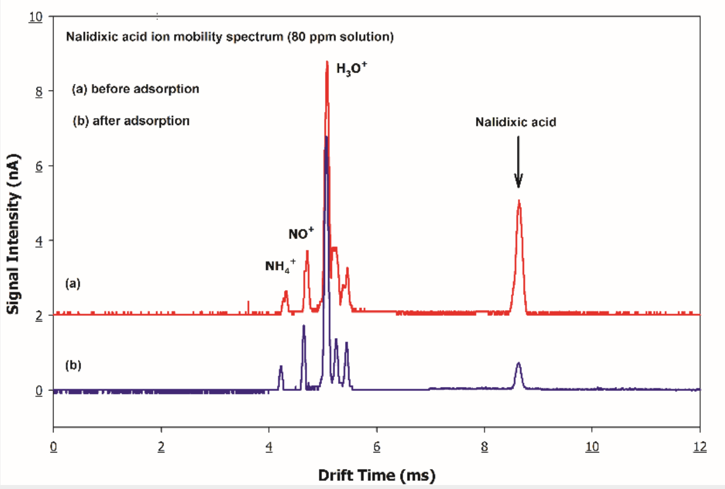
Graphical Abstract



Abstract: Antibiotic pollutants in water and sewage can cause severe damage to the environment and human health. Therefore, removing them from the water is very important [1]. Ion mobility spectrometry (IMS) is a separation method that works at atmospheric pressure. The advantages of ion mobility spectrometer are high sensitivity, low detection limit, simple operation, and low cost [2]. In the present study, an ion mobility spectrometer was used to measure the remaining nalidixic acid (NA) in an aqueous solution. The adsorption of NA on bovine serum albumin (BSA) nanoparticles was investigated aimed at the removal of NA from a water environment. The BSA adsorbent was synthesized by the desolvation technique. Transmission electron microscope (TEM) and FT-IR were used to identify the characteristics of synthesized BSA nanoparticles. The adsorption experiments were performed to examine the effects of solution pH (3-10), contact time (0.5-5) hours, and initial NA concentration 30-80 ppm. Moreover, adsorption kinetics and isotherms were studied. The results showed that the removal efficiency of NA in optimal conditions pH = 10, temperature 25 °C, NA concentration 80 mg /L, contact time 5 hours, and adsorbent dose 0.5 mg was 75%. The NA adsorption isotherms and kinetics were in agreement with the Freundlich isotherm and pseudo-second-order model, respectively. Regarding the results of this study, BSA nanoparticles can be used as appropriate adsorbents for antibiotic removal from aqueous solutions.

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Evaluation of acetaminophen removal from aqueous solution using bovine serum albumin nanoparticles and determination of removal efficiency by ion mobility spectrometry

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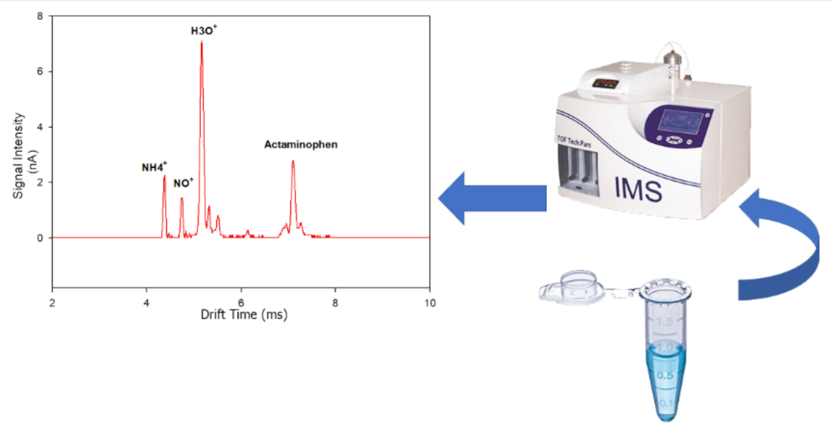

Abstract: Pharmaceutical compounds are considered emerging environmental pollutants that have a potentially harmful impact on the environment and human health. Due to the enormous production and high consumption worldwide, acetaminophen (ACT) has become one of the most frequently detected pharmaceuticals in the aquatic environment [1]. Ion mobility spectroscopy (IMS) is a separation method that works at atmospheric pressure. The advantages of ion mobility spectrometer are high sensitivity, low detection limit, simple operation, and low cost [2]. The aim of this study was to investigate the potential of bovine serum albumin (BSA) nanoparticles as sorbent for the removal of ACT from an aqueous solution. The initial concentration of ACT and the concentration of ACT remaining in the aqueous solution were determined by IMS. The effect of various parameters such as pH (2-10), initial ACT concentration (100-400 ppm), and contact time (0.5-5 hours) on ACT removal has been investigated. Furthermore, adsorption kinetics, as well as adsorption isotherms, were studied. The results showed that the maximum adsorption capacity of BSA nanoparticles for ACT adsorption is 360 mg/g. The optimal conditions for ACT removal using BSA nanoparticles were found to be: contact time 300 min, sorbent dose 0.5 mg, pH=7, and initial concentration ACT 100 mg/L. Under these conditions, the removal efficiency was 90%. The kinetics and isotherm data were compatible with the pseudo-second-order and Freundlich models. These results demonstrate that BSA has an excellent potential to adsorb ACT from an aqueous solution.

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Determination of polycyclic aromatic hydrocarbon (PAHs) in soil and plant samples by method of ultrasonic-coupled QuEChERS and Gas chromatography-Mass Spectroscopy

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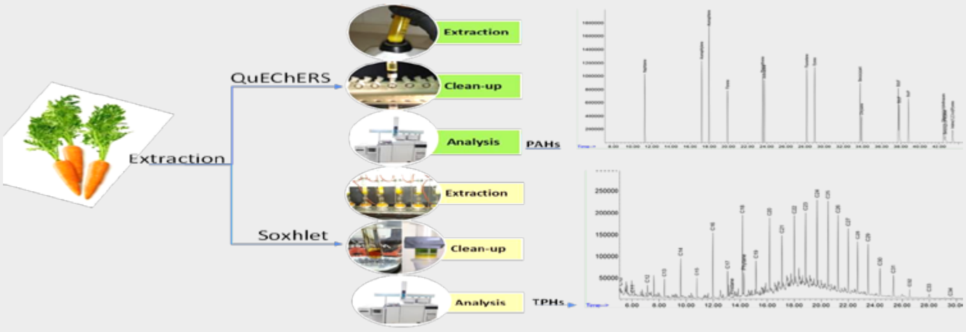

Abstract: Environmental pollution from hazardous and toxic chemicals is one of the major problems facing the world today. Persistent organic pollutants (pops) are a group of toxic substances among environmental pollutants. Polycyclic aromatic hydrocarbons (PAHs) are included in group of persistent organic pollutants due to chemical structure persistent and intrinsic resistance against biological and non-biological decomposition and In case of existence in food chain, polycyclic aromatic hydrocarbons are one of the most worrying indices and threats for living things. The use of kerosene as a herbicide in some vegetable fields in Khuzestan province, including Dezful city, leads to contamination of soil and vegetables grown in these fields, especially carrots. The current research aims to measure petroleum pollutant in soil and plant samples. Increasing the concentration of kerosene significantly increases the polycyclic aromatic hydrocarbons in the soil and plants. In order to extract PAHs and TPHs in soil and plant samples, Soxhlet extraction method and ultrasonic coupled extractors were used. Gas chromatography - Mass Spectroscopy (GC-MS) was used to measure the amount of PAHs. Determination of heavy metals in the soil sample was performed using dissolution method with hydrofluoric acid and perchloric acid according to the standard with induced coupled plasma mass spectrometry. Lead and cadmium were measured in plant samples by spectroscopic method of atomic absorption of graphite furnace after microwave digestion. In order to the Clean-up of extracts and separate the analyte from the complex matrix, SPE solid phase extraction was used using C_{18} cartridge in succulent extraction, as well as C_{18} cartridge and DMLLE diffusion fluid microextraction in QuEChERS method. Considering the excessive absorption of PAHs in carrots, it can be concluded that the use of kerosene in vegetable fields has increased the concentration of these compounds in vegetables. The results of this study show that the proposed method of ultrasonic-coupled QuEChERS followed by DLLME purification and GC-MS analysis has a very good ability to determine very small amounts of PAHs in carrot and soil samples. According to the competency figures comparable to other methods, its good performance can be mentioned.

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Using of Fe_3O_4-graphene oxide- modified chitosan with melamine magnetic nanocomposite in magnetic dispersive solid phase microextraction of Cr(VI) ions in aquatic samples

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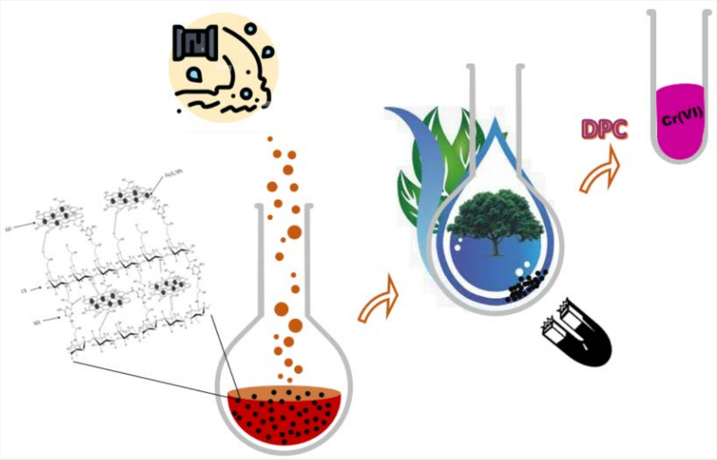

Abstract: Chromium as a heavy metal enters to the environment with human activities from various industries such as leather tanning, wood treatments or metallurgy industries. Hexavalent chromium is a very toxic species with high risks to human health and the environment. The presence of such harmful pollutants demands proper separation and enrichment methods to increase the detection power of the analytical instruments [1]. In recent years, magnetic solid-phase extraction (MSPE) based on iron oxide nanoparticles have received special attention as adsorbents to high-efficiently extract heavy metals such as chromium (VI). In the present study, biocompatible, effective and magnetic graphene oxide modified chitosan with melamine nanocomposites (GO-Fe_3O_4-CS-MA) were synthesized [2]. The structure and morphology of synthesized nanocomposites was characterized using FT-IR, SEM, EDX, XRD, DLS, Zeta-Potential, VSM and TGA. The application of synthesized nanocomposites for magnetic dispersive solid phase extraction of chromium (VI) from aqueous samples was investigated. The chromium determination is based on the complex formation reaction of Cr(VI) with 1,5-diphenylcarbazide. In order to achieve high enrichment factors and reduce the detection limit of the method, various parameters were studied and optimized. Optimal values for the adsorbent amount, adsorption and desorption time, salt percentage and pH were 20 mg, 5 minute, 0% and 4, respectively. Also, 1 ml of sodium hydroxide 0.01 M was selected as the optimal desorption solvent. The suggested method was linear (R^2 = 0.9993) over a range of 0.05-0.5 mg L^{-1}. The limit of detection was 0.015 mg L^{-1}. Finally, the applicability of the proposed method was evaluated by analysis of the hexavalent chromium in the industrial effluent of Charmshahr Tabriz as well as some surface waters. The percentage of recoveries was in the range of 101-91% and the percentage of relative deviation was 2-6%.

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Evaluation of microplastic contamination in the Persian Gulf

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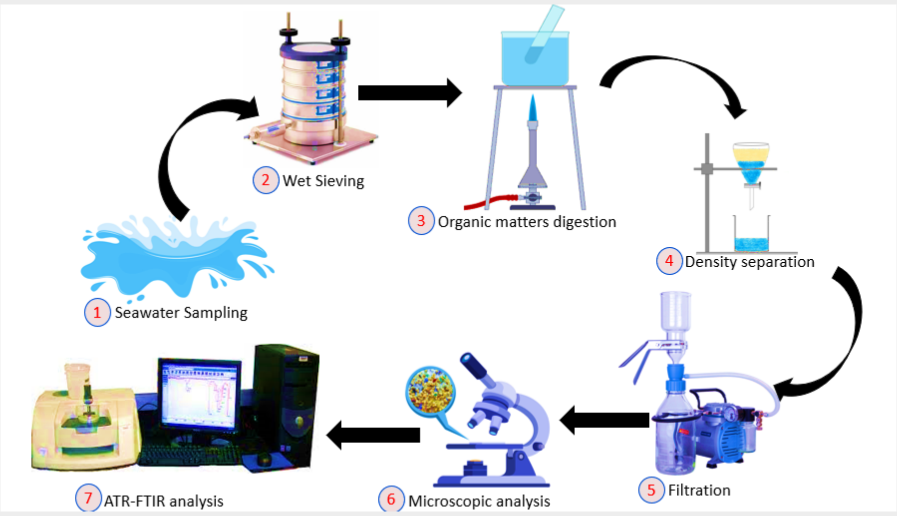

Abstract: Micro-plastics are plastics with a diameter between 1 micrometer to 5 millimeters. These plastics are worrying contaminants that found in more aquatic environments in estuaries, lakes, coastal waters and densely populated areas [1-4]. Due to the properties of micro-plastics in the adsorption of toxic substances and their adverse effects on living organisms and food chains [1-3], the purpose of this study was to evaluate microplastics and their impact in the Persian Gulf. The scope of this study is to further identify and eliminate them in this ecosystem. In this study in the Persian Gulf basin, sediment sampling of 22 stations on the coast of Bushehr province, 59 stations on the coast of Hormozgan in 2018 and 2019 were performed. In this evaluation, after sediments sampling, plastic materials were identified by Fourier transform infrared spectroscopy (FTIR). The results confirm many of them were fiber type. Based on the studies, the plastic samples were observed in terms of color in black, blue, red and white, respectively, with sizes in range 100 to 2000 micrometers. The abundance of these particles in the study was estimated at an average of 30.29 Particle/Kg. Therefore, various factors such as population, incoming sewage, shipping and fishing activities and industrial activities on the coast are very effective on microplastic contamination [3]. As a result, considering the importance of the Persian Gulf as a large marine habitat and the increasing number of microplastics, this study can provide useful information for further research and management decisions to understand the potential effects on marine life in the Persian Gulf.

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Synthesis of CuO/TiO_2 and investigation of photocatalytic properties for removal of Direct Red 16 pollutant under visible and ultraviolet light

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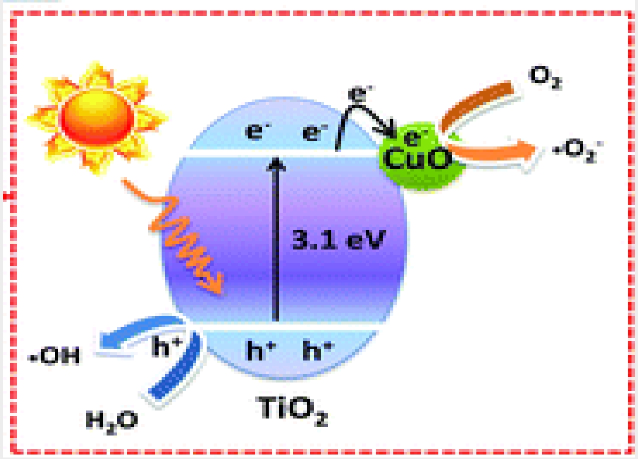
Graphical Abstract



Abstract: Nowadays, with the development of science and technology and the production of thousands of chemicals and their entrance into the environment, many problems have created in human life. Contamination of sources with pollutants have required the use of new methods of controlling and purification. Photocatalytic oxidation process due to high efficiency and effective removal of pollutants has attracted a lot of attention. During the photocatalytic oxidation process, with light radiation in the presence of semiconductor catalysts, contaminants are decomposed into carbon dioxide, water, or other harmless substances. In the present study, copper oxide with titanium dioxide was coupled by chemical reduction method and obtained CuO/TiO_2 composite was investigated for direct red photocatalytic removal from aqueous solution under visible and ultraviolet radiation. The physical and chemical properties of CuO/TiO_2 composite was studied by X-ray diffraction, microscope electron scanning, photoluminescence and diffuse reflectance spectroscopy. The X-ray diffraction analysis and images of electron scanning microscopy confirmed the presence of CuO and the formation of CuO/TiO_2 composite. The diffuse reflection spectroscopy and photoluminescence analysis also showed that due to the coupling of CuO with TiO_2, the absorption in the visible region increases and also the recombination rate of the photoelectron and the hole significantly reduces. According to the results of photocatalytic tests, the CuO/TiO_2 composite removed 68% of Direct Red 16 in ultraviolet light and 64% in visible light.

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Removal of toluene from air with photocatalytic oxidation by GO/ZnO composites

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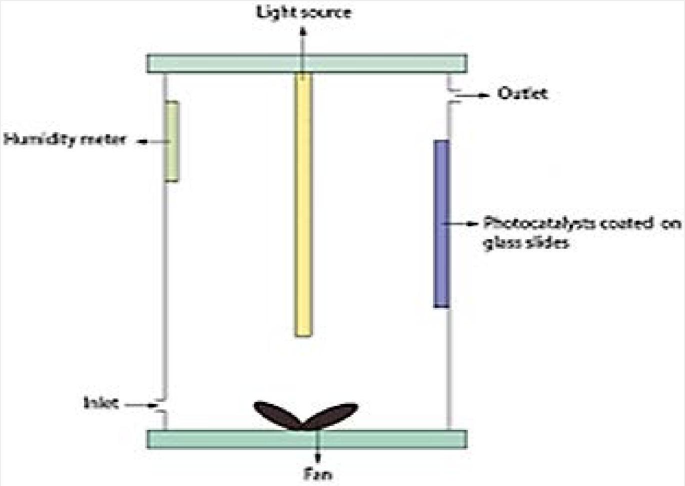
Graphical Abstract



Abstract: One of the most important issues facing human being is the availability of clean air. Volatile organic compounds (VOCs) are hydrocarbons that in usual temperature and pressure, their normal vapor pressure is more than 10.3 kPa, and they easily evaporate. The release of these compounds into the air causes respiratory problems. Toluene is one of the most common volatile organic compounds and is used in the military industry to make tri nitro toluene and in the chemical industry is used as a solvent in the manufacture of paints, paint polishes, nail polishes, printing materials, pharmaceuticals and tobacco. Due to the extensive use of toluene in various industries and to prevent damages to workers' health (Possibility of carcinogenesis), appropriate methods must be used to remove or reduce toluene vapors in the air. The photocatalytic oxidation process has attracted a lot of attention, due to high efficiency and effective in removing pollutants. During the photocatalytic oxidation process, with light radiation and in the presence of semiconductor catalysts, pollutants decompose into carbon dioxide and water. In this research, graphene oxide (GO) with different percentages was loaded on zinc oxide and the resulting composites (GO/ZnO) were investigated in the photocatalytic removal of toluene from air under visible light radiation. The results showed that GO/ZnO composites had high photocatalytic activity and finally, GO (40%)/ZnO composite as an optimal photocatalyst degraded 76% of toluene under visible light radiation. Diffuse Reflectance Spectroscopy analysis showed that GO (40%)/ZnO composite has high absorption in visible light and photoluminescence analysis also showed that due to GO loading, recombination of photoelectrons and holes is greatly reduced. The GO acts as an electron acceptor and transmitter and causes better separation of charge carriers and consequently, photocatalytic performance is enhanced.

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Synthesis, Characterization and Application of Montmorillonite/$MgFe_2O_4$ Nanocomposite for Removal of Environmental Pollutants

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Abstract: In this research Montmorillonite (MMT) modified with magnesium ferrite (termed as MMT/Mg Fe_2O_4) was prepared and then was examined for its dye removal properties. MMT-based adsorbents have relatively complex structural and adsorption properties. MMT has a good cation exchange and expansion capacity [1]. MMT is mineral clay and its derivatives are cheap, abundant and environmentally friendly, which distinguish them as an adsorbent. They can be used directly as a high-performance adsorbent for various cationic contaminants. The purpose of using MMT is to increase the efficiency of wastewater treatment and lower the cost of removing color contaminants. Congo red dye (CR) is a benzidine based anionic dye. It is seriously hazard to aquatic living organisms and can cause human carcinogen [2]. In this study, MMT/$MgFe_2O_4$ nanocomposite as a novel adsorbent was synthesized by co-precipitation method. The properties of the prepared nanocomposite were investigated by using Fourier-transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM) and X-ray diffraction (XRD) techniques. In order to achieve optimal removal conditions and isotherm, kinetics and thermodynamic studies, the effects of many empirical factors including pH, initial dye concentration, contact time, temperature, the amount of adsorbent, ionic strength and regeneration were investigated. In thermodynamic investigation, $\Delta G°$, $\Delta H°$ and $\Delta S°$, were determined and adsorption mechanism was discussed [3]. It was found that, removal efficiency was higher at pH value of 4.0. The results revealed that 50.0 mg of MMT/Mg Fe_2O_4 removal was able to remove more than 93% of CR dye from 50.0 mL of 250 mg/L of initial CR dye aqueous solution. The results obtained have shown that the introduced adsorbent reveals excellent dye removal properties and can be used as an efficient adsorbent for dye removal from aqueous solutions. In order to test the reusability of the exhausted adsorbent regeneration study was also carried out.

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Sono-Fenton Degradation of Ciprofloxacin in Aqueous Solution using $FeMnO_3$/GQDs

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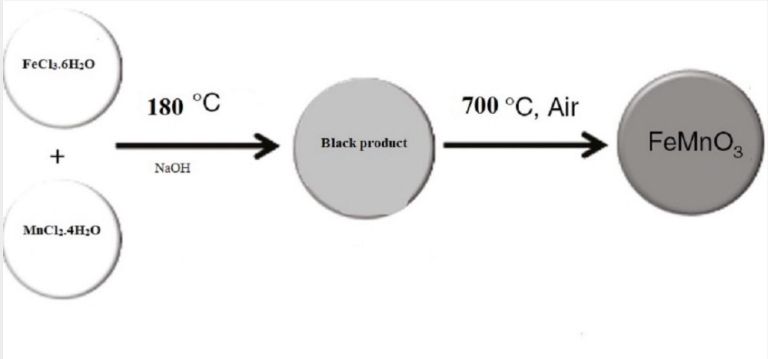
Graphical Abstract



Abstract: Consumption of pharmaceuticals has dramatically risen over the past few years. Antibiotics are considered as the commonest type pharmaceuticals which causes inhabitation in microorganism functions. Antibiotics are not completely absorbed in human body and 30-90% of them find their ways into environment through urinary and defecation systems [1-2]. The irreversible effects of drug compounds on the environment, such as the development of antibiotic-resistant bacteria as one of the environmental challenges, have led to their elimination. Emerging methods based on nanotechnology such as nanocatalysts have been used successfully to remove drug compounds due to their many advantages. Conventional methods used are not effective in removing drug contaminants and the main reason for this is the low rate of biodegradation of these substances [3]. Therefore, it’s necessary to find a suitable method to remove antibiotics from environment. In this study, heterogeneous Fenton- like catalytic processes was evaluated for removal of antibiotic Ciprofloxacin from a synthetic wastewater. So this nanocomposite was synthesized by hydrothermal method. The synthesized catalyst was identified using methods such as Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), energy dispersive X-ray spectroscopy (EDS), Field emission scanning electron microscopy (FESEM), Transmission Electron Microscopy (TEM), and visible-ultraviolet spectroscopy, and the results confirmed the catalyst structure. The effect of the various factors on the degradation of the Ciprofloxacin, such as the amount of the synthesized catalyst, time of irradiation, hydrogen peroxide concentration and pH of the initial solution, were also studied. Based on the results after optimizing the parameters, the maximum degradation efficiency of Ciprofloxacin by the synthetic catalyst was 83%. Hence using this method and process was recommended for degradation antibiotics from environment.

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Photocatalytic Degradation of Naphthalene Using Copper Oxide Nanoparticles Synthesized from the *Nigella Sativa*

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Graphical Abstract

Abstract: By using advanced oxidation processes (AOPs), it is possible to decompose toxic organic compounds soluble in water under UV radiation and in the presence of metal oxides. In this research, CuO nanoparticles were synthesized using the extract of a native plant called *Nigella Sativa*. The samples were then characterized by SEM, XRD, and EDX techniques. The results showed that the synthesized CuO nanoparticles are spherical and have an average size of about 100 nm. Then, the removal percentage of naphthalene, as an organic compound in textile wastewater, was investigated by photocatalytic processes in the presence of copper oxide nanoparticles. According to the GC-MS analysis, the toxic organic compounds such as naphthalene, decane, undecane, dodecane, decahydro-2,3-dimethyl, methylmethylenecyclohexane, decahydro-1,5-dimethyl, tridecane, tetradecane, and hexadecane constitute the main pollutants in textile wastewater. In this work, about 95% of 2-methylmethylenecyclohexane, naphthalene, decahydro-1,5-dimethyl, hexadecane, and decahydro-2,3-dimethyl can be eliminated by CuO nanoparticles under UV radiation. The highest and lowest removal percentages in the photocatalytic degradation process were 88% and 48%, respectively, for naphthalene and decahydro-2,3-dimethyl.

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Removal of Reactive Dyes from Real Textile Wastewater Using Zinc Oxide Nanoparticles under Ultrasonic Irradiation

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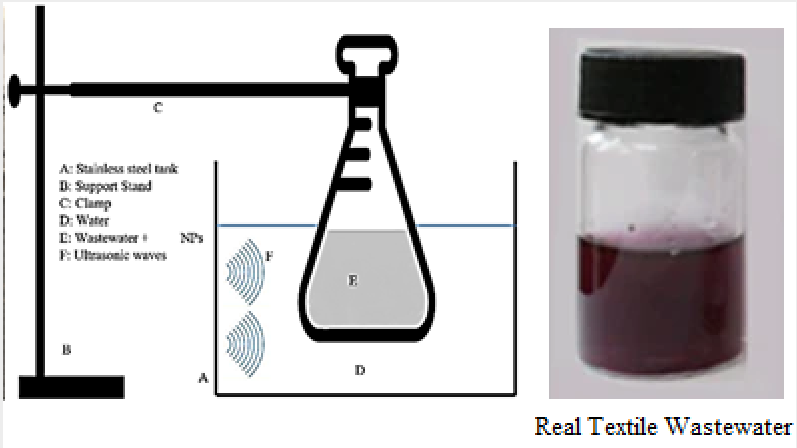
Graphical Abstract



Abstract: Wastewater from the textile industry is one of the most important and serious environmental concerns for the safety of land and aquatic ecosystems. In this study, ZnO nanoparticles (NPs) were synthesized using sol-gel method. Then, the synthesized NPs were identified by TEM, FE-SEM, XRD, FTIR, and EDX techniques. The results showed that the synthesized ZnO nanoparticles are spherical and have an average size of about 40 nm. The synthesized ZnO NPs were used to remove reactive dyes in textile wastewater such as yellow 145, and blue 5. Then, the effect of operational parameters such as the pH of the solution, dose of ZnO NPs, ultrasonic irradiation time and power were investigated. It was found that the degradation ratio of organic dye pollutants in the textile wastewater in the presence of ZnO NPs was much higher than that only with ultrasonic radiation. According to the GC-MS analysis, the toxic organic compounds such as reactive blue 5 and reactive yellow 145 dyes composed about 28% of the initial wastewater sample. All experiments showed that the sonocatalytic degradation method in the presence of zinc oxide nanoparticles is a suitable choice for the treatment of non-transparent organic wastewater in the textile industry.

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Measurement of heavy metals (Pb^{+2}, Cd^{+2} and Ni^{+2}) in real samples of Anzali wetland by atomic absorption spectrometry technique

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Graphical Abstract

Abstract: For environmental protection, public health, and pollution control, we must have precise information about the extent of pollutants, especially, heavy metals in water environments. Having high stability, heavy metals can produce danger in living organisms. Therefore, the present research was conducted to assess the Concentration of heavy metals and compare the findings with national and international standards. In this study, the concentration of metals (Pb^{2+}, Cd^{2+} and Ni^{2+}) in water, surface sediments and tissue of a species of aquatic plants (serratophyllum) in Anzali wetland was determined. In this study, four areas of Anzali wetland were selected for sampling. Sediment and texture samples of the studied plants after drying and pulverization by wet digestion and acid mixing method ($HClO_4$, HNO_3 and H_2SO_4) using a multi-house heater Chemical digestion of the samples took place [1,2]. Extraction and separation of the studied heavy cations in the aqueous samples were performed using organic solvent (MIBK) and complexing agent (APDC) [3]. The average accumulation of lead, cadmium and nickel in surface sediments was 3.74, 1.49 and 8.29 µg/g dry weight, respectively. The highest accumulation of heavy metals in the aquatic plant tissue of Serratophyllum belonged to nickel metal at 17.34 µg/g and the lowest concentration belonging to cadmium metal was 0.309 µg/g dry weight. The maximum concentrations of nickel and lead in water were 141 and 89 µg/L, respectively, at the east wetland area. In this study, the concentrations of metals obtained in water, sediment and plant tissue samples were compared with NOAA, ISQG, WHO and EPA standards .The results showed that the concentration of studied metals at the east wetland area was higher than other areas due to the entry of industrial wastewater.

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Facile Synthesis of g-C_3N_4/NH_2-Fe-MILs for Effective Degradation of Imidacloprid under Visible Light Irradiation

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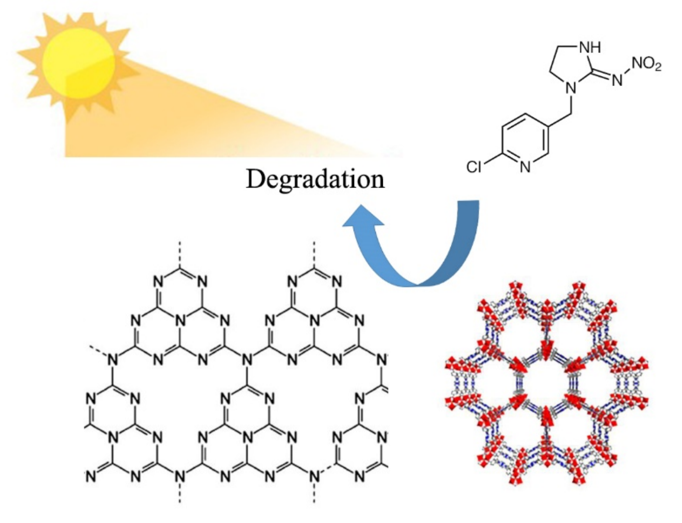
Graphical Abstract



Abstract: Mesoporous g-C_3N_4/NH_2-MIL-101(Fe) composites were successfully made by the solvothermal method. The g-C_3N_4/NH_2-MIL-101(Fe) composite indicates higher photocatalytic activities than bare g-C_3N_4 and NH_2-MIL-101(Fe) for imidacloprid degradation. The optimum heterojunction photocatalyst, g-C_3N_4/NH_2-MIL-101(Fe) (250 mg of mass ratio gC_3N_4), reveals the highest photocatalytic efficiency up to 80% within 1 h. The response surface methodology (RSM) was used in the design of IMC photodegradation experiments to assess the interaction effects of the independent variables and to specify the optimal conditions. Eventually, a possible reaction mechanism had also been investigated in detail.

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Microplastic Pollution Evaluation in Sediments of South Caspian Sea (Mazandaran Province, Iran)

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Graphical Abstract

Abstract: Microplastics have been identified as an emerging pollutant in marine environments and can have serious impacts on the marine environment and coastal ecosystems. An important aspect of microplastics is that they threaten aquatic life and, on the other hand, are eaten by aquatic animals and then enter the food chain. In this study, marine sediments were evaluated as a sample of microplastic contamination on the south coast, sea and Caspian.Microplastics were observed in various colors of blue, white, red, black, etc .Dimensions of microplastics collected during this study; it varied from 275.760 µm to 81171.063 µm. To determine the polymer composition of the microplastics removed from the sediment, a Fourier transform infrared spectroscopy equipped with a generalized attenuation system or Raman spectrometer was used. Also, SPSS software was used to analyze all data.

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Cadmium Removal from Aqueous Solution with Nano γ-Alumina/β-Cyclodextrin as Nanobiosorbent and Determination by FAAS

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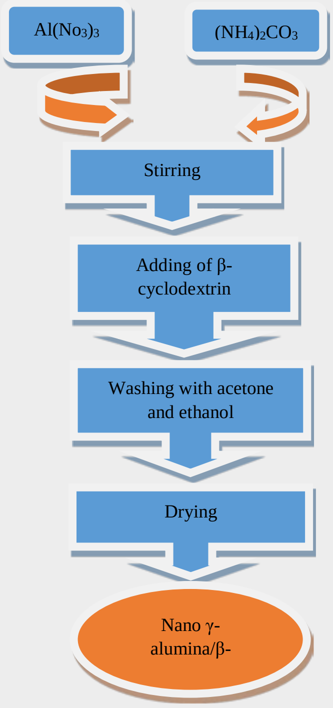
Graphical Abstract



Abstract: *A* novel extraction method for selective extraction and determination of trace amounts of Cadmium (II) ions in water samples has been investigated. This method is based on the selective sorption of the Cd(II) ions used nano γ-alumina coated with β-cyclodextrin as nano adsorbents and determination by atomic absorption spectrometry. The effects of various parameters such as pH, the Cd(II) concentration, amount of sorbent, and type and concentration of the eluting agents were determined on the removal efficiency. The limit of detection (LOD) and repeatability (RSD%) values (0.389) obtained were found to be in the ranges of 6.77-6.81 µg/L. The results showed adsorbed cadmium ions are recovered on the nano γ- alumina/β-cyclodextrin surface with an optimum amount of 16 ml of 0.3 M nitric acid as eluting agent at pH 7.

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Analytical Chemistry and Industries

Adsorption and Solid-Phase Extraction UV-Vis Spectrophotometric Determination of Rhodamine 6G in Food Sample Using Marble Powder Waste

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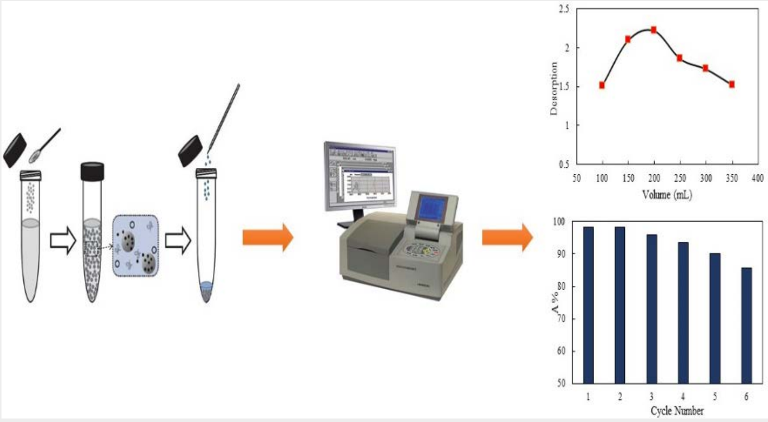
Graphical Abstract



Abstract: The solid-phase extraction (SPE) as a routine technique of sample preparation is used in the sorbent extraction method, and this technique has diverse applications such as food analysis [1]. Nowadays, natural sorbents have been highly regarded by researchers in various industries, especially in the food industry due to their non-toxicity, cost-effectiveness, lack of need for complex production processes, and abundantly available [2]. These sorbents are usually prepared from agricultural, mineral, and construction wastes. Natural adsorbents can be a lucrative alternative to synthetic and commercial adsorbents in SPE methods. In this study, we provided marble powder wastes (MPW) from Doroud Stonework, Lorestan, Iran, and we washed and calcined it. We employed MPW to adsorb and detect trace amounts of Rhodamine 6G in food samples using the solid-phase extraction UV-Vis spectrophotometry method. The structure and morphology of MPW were characterized by FT-IR, XRD, and FE-SEM analysis. A three-factor, three levels of Box-Behnken design was utilized to explore the optimum condition of the sorbent mass, pH, and uptake time. The sorption percent of Rhodamine 6G at 544 nm was considered as the dependent variable against these parameters. According to information obtained from ANOVA analysis, the model's F-values and P-values were calculated at 193.57 and 0.103, which illustrate the factors in the models have a substantial effect on the response. Under optimum conditions: adsorption percent, linear range, the limit of detection, and limit of quantification were obtained at 97.60±1.04, 50-20000 µg.L^{-1}, 8.50 µg.L^{-1}, and 28.35 µg.L^{-1}, respectively. The enrichment factor was determined at 66.66 and relative standard deviations for 6 repetitions are achieved at 3.68 %. This SPE method by MPW adsorbent has been successfully feasible for the determination of rhodamine 6G content in food samples. This operation was carried out by adding 50, 100, and 200 µg.L^{-1} of rhodamine 6G in 5 mg/L of jelly, orange juice, saffron ice cream, and tap water in the analytical laboratory of Lorestan University. Finally, the reusable, good LOD, notable RSD %, short-time extraction, selective, sensitive, low-cost, and high enrichment factor are benefits of this SPE method.

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Effect of Ultrasonic Pre-treatment on the Oil Yield Obtained from *Linum usitatissimum* Seeds

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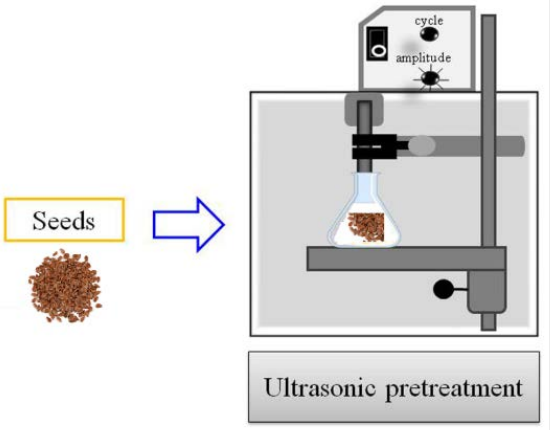

Abstract: *Linum usitatissimum* seed is considered to be an important oil seed crop that is especially rich in essential fatty acids. The seed contains essential omega-6 and omega-3 fatty acids that our bodies require to maintain optimal health [1]. Omega-3 fatty acids are essential for growth and development, and have been associated with health and the prevention and treatment of arthritis, inflammatory, cancer, and heart and autoimmune diseases [2]. In this research, we aimed to study the effect of ultrasonic pre-treatment prior to oil extraction by mechanical expellers on the oil yield obtained from *Linum usitatissimum* seeds. Optimization of three parameters including ultrasonic temperature, ultrasonic duration, and ultrasonic power to obtain oil with high yield was performed. According to the results, the maximum yield of oil was obtained with the ultrasonic temperature of 40°C, duration of 15 min, and power of 100 W. The results showed that the ultrasonic pre-treatment prior to oil extraction produced the highest yield compared with conventional method (mechanical expellers). The higher extraction of oil by ultrasonic pre-treatment might be possible due to increase in mass transfer by rupturing the cell wall and formation of micro cavities thus enhances the product yield. The obtained oils were analyzed using gas chromatography (GC) and Gas chromatography–mass spectrometry (GC-MS). *Linum usitatissimum* oil was found to contain high amounts of linolenic followed by oleic, and linoleic, while the dominant saturated acids were palmitic and stearic. So, ultrasonic pre-treatment prior to oil extraction is obviously an effective method to improve the oil yield, then to reduce the extraction time and energy consumption, comparing to conventional method (mechanical expellers).

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Effect of Ultrasonic Pre-treatment on the Biological Activities of Oil Extracted from *Linum usitatissimum* Seeds

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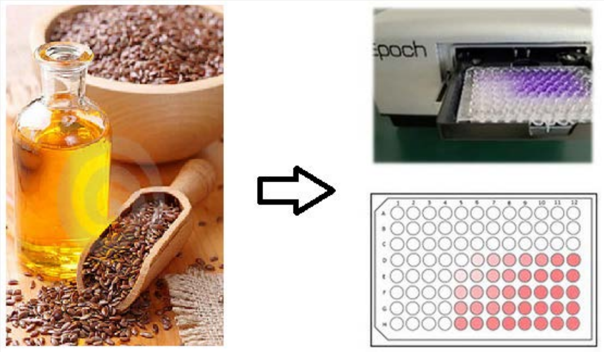

Abstract: *Linum usitatissimum* seed is considered to be an important oil seed crop that is especially rich in essential fatty acids with biological activities. [1]. The quantity and quality of the oil is associated with its content and chemical constitutes. Thusly, to select an adequate extraction technique is of high significance. The hired technique should avoid the decomposition of interest compounds, and maintain the quality and biological activities of the extract [2]. Up to now, the extraction of oil from seeds was done using conversational methods such as mechanical expellers or nonpolar solvents [3]. In this research, we aimed to study the effect of ultrasonic pre-treatment prior to oil extraction on the biological activities of oil obtained from *Linum usitatissimum* seeds. After optimization of three parameters including ultrasonic temperature, ultrasonic duration, and ultrasonic power, the antioxidant and cytotoxicity activities of the oils were evaluated. DPPH (2,2-diphenyl-1-picryl-hydrazyl-hydrate) free radical was used to test the antioxidant property of the oils. Also, the cytotoxic activity of the oils against BH-E adenocarcinoma and QU-DB cell lines was determined using the 2,5-diphenyl-2H-tetrazolium bromide (MTT) assay. The results indicated that the oils inhibitory activities depend upon concentration and the increase in the concentration increased the inhibitory percentage. In the concentration range, the synthetic antioxidant manifested further antioxidant activity than the oils. Moreover, from obtained results it can be seen that the oil extracted after ultrasonic pre-treatment had significantly higher antioxidant activity compared with conventional method (mechanical expellers). Also, the cytotoxicity results exposed that the oil extracted after ultrasonic pre-treatment had higher cytotoxic properties against the studied cell lines. So, ultrasonic pre-treatment prior to oil extraction is obviously an effective method to improve the oil biological activities, comparing to conventional method (mechanical expellers).

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Comparison of flumethrin and fluvalinate residues in honey and beeswax

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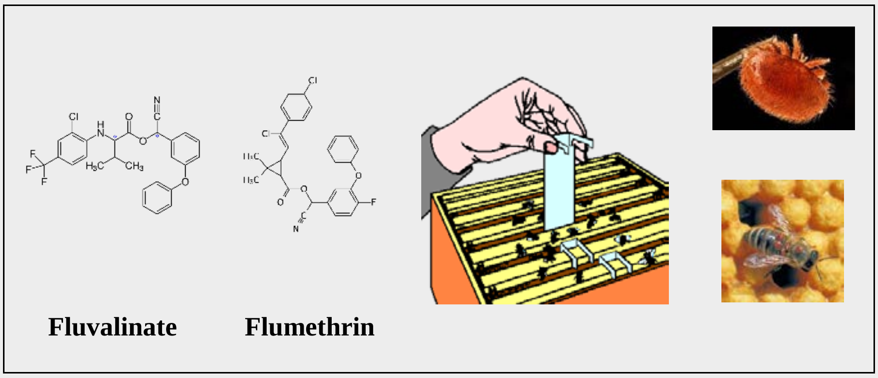
Graphical Abstract



Abstract: Production of sound foods is a major concern. Like other foods, honey product is also subjected to contaminations. The most important contributor to the contamination is chemicals used to face to pests and illnesses. Varroa mite is one of significant pests in honey bee production and, therefore, anti-mite chemicals are of important contaminators of the hives. A total number of 12 hives with comparable situations of population were selected. The study was conducted for five weeks in a completely randomized design with two treatment groups, including Apistan (active ingredient, fluvalinate) and bayvarol (active ingredient, flumethrin) with 6 replicates each. Residues of flumethrin and fluvalinate in honey and beeswax were identified using high performance liquid chromatography (HPLC). Sample preparation was performed using the QuEChERS method. The QuEChERS (quick, easy, cheap, effective, rugged, and safe) method uses a single-step buffered acetonitrile (MeCN) extraction and salting out liquid–liquid partitioning from the water in the sample with $MgSO_4$.

Examination of the results shows that the residue of Apistan in both honey and wax products was significantly higher than that of bayvarol. In our study, fluvalinate had an average of 64 ppm remaining in honey, and in the wax was calculated to be 17 ppm, which is higher than previous reports. Unlike fluvalinate, residue is not a major problem with flumethrin and is probably due to the amount of active ingredient in each strip (flumethrin 3.6 mg per strip and fluvalinate 880 mg per strip).

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Carcinogenic and Non-Carcinogenic Risk Assessment Induced by Pesticide Residues in Commercially Available Ready-To-Eat Raisins of Iran Based on Monte Carlo Simulation

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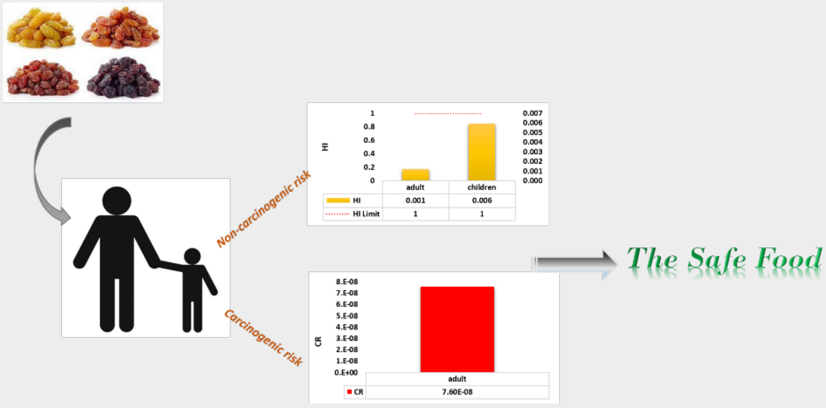
Graphical Abstract



Abstract: Pesticide residues in fruits lead to serious public health and environmental problems. This study was undertaken to analyze 57 pesticides residues in 60 commercially available ready-to-eat packed raisins samples from Iran's markets using Quick, Easy, Cheap, Effective, Rugged, and Safe (QuEChERS) approach along with acetonitrile for the extraction, surface adsorbents for clean-up procedure, following with a ultra-high performance liquid chromatography-tandem mass spectrometry (UHPLC-MS/MS) [1]. Moreover, the probabilistic health risk assessment includes non-carcinogenic and carcinogenic risk were estimated by hazard quotient (HQ), Hazard Index (HI) and cancer risk (CR) using the Monte Carlo Simulation (MCS) method [2]. The respective numbers attributed to LOD and LOQ were 0.001–0.03 mg kg^{-1} and 0.005–0.05 mg kg^{-1}. Results showed that 23% of samples were contaminated by at least one pesticide according to national MRLs. The rank order of pesticides based on HQ was carbendazim > acetamiprid > thiodicarb > iprodione for raisins. The rank order of pesticides based on CR was thiodicarb > iprodion. But total CR was lower than 1E-6 value so consumers were not at considerable carcinogenic risk in these commodity. Acetamiprid and carbendazim does not pose a cancer risk to humans. Therefore, a dietary exposure assessment for the purpose of assessing cancer risk is unnecessary. The hazard quotient values varied among the samples and was greatly affected by the reference dose of pesticides. The estimated health risk assessment of the compounds in all type of pesticides via Monte Carlo approach showed that adults and children are not at considerable non-carcinogenic and carcinogenic risk of the mix exposure to pesticides by raisins and there is no serious adverse health effect for adults and children. The implementation of good agricultural practice in farms, the promotion of knowledge and awareness of pesticide user, application of alternative safe method to control pests such as biotechnology-based, application of rational pesticide use program, continuous monitoring of pesticide in crops and the implementation of strict governmental regulation towards pesticides residues in food are recommended.

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Acetyl and Butyryl Cholinesterase Inhibitory Activity and GC-MS Analysis of Rashingari and Khuzestani Savory Extracts

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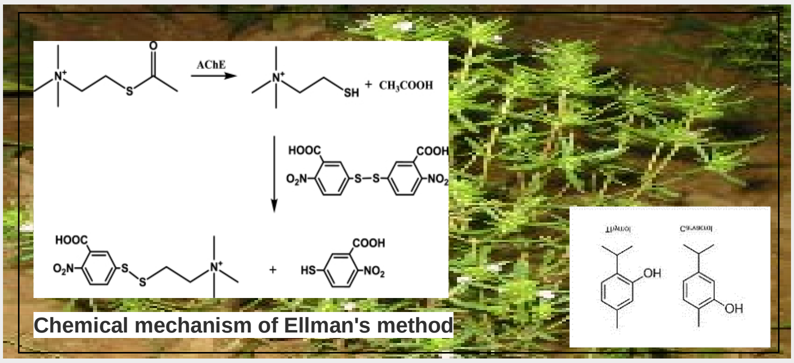
Graphical Abstract



Abstract: Medicinal plants around the world play an essential role in the treatment and prevention of human diseases. These plants are the source of traditional medicine, and many modern medicines are made indirectly from them. More than 65% of the world's population, especially in many developing countries, use traditional medicine to treat human diseases. In the last decade, plants have been recognized as various sources of chemicals, and many of them have strong antioxidant activity. Phenolic compounds such as flavonoids, phenolic acids and phenolic diterpenes are primarily responsible for the antioxidant effect of plant products. Saturia Khuzestanika, belonging to the family lamiaceae, contains 200 species of aromatic plants and shrubs that are commonly found in the Mediterranean regions. It has been reported that different species of Saturia have excellent antimicrobial, antiviral, anti-disease, anti-cell proliferative properties. In the regions of Iran, 14 species of this genus are distributed mainly in the mountains. Saturia Khuzestanika and Saturia Rashingari are two species of the genus Saturia, native to the southern regions of Iran. In this study, Rashingari and Khuzestani savory extract was extracted with ethanol and then its main components were examined and compared by gas chromatography. Chemical analysis of the extract of these medicinal plants with GC-MS showed that the main components of Khuzestani savory extract are carvacrol (68.7%), para-cement (62.4%), terpinol (3.16%), and Rashingari savory extract including thymol (51.28%), carvacrol (22.08%), linalool (4.37%) and cis-beta-Bisabolene (3.08%). The results of enzyme experiments with Elman method showed that Khuzestani safflower extract had the highest inhibition against acetyl- and butyryl-cholinesterase (AChE and BChE) (377±2.36 µg/ml and 251±1.88 µg/ml, respectively). Carvacrol is the main component of the extract of these herbal medicines and the inhibitory activity of this compound has been shown to be high (57±0.25 µg/ml for AChE and 181±0.95 µg/ml for BChE).

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Electrochemical Oxidation and Assessment of Antixidant Activity of Etanolic Leaf *Papaver Rhoeas L.* Extract

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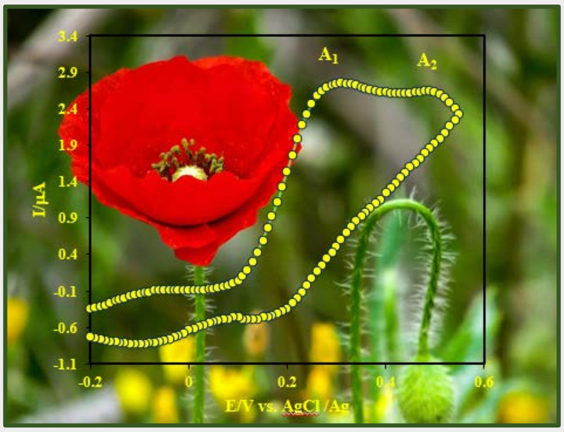
Graphical Abstract



Abstract: Medicinal plants and their extracts deserve special attention because of the vital influence they have on human health. For the majority of the world population, medicinal plants represent the primary source of health care [1]. On the other hand, electrochemical evaluations are useful for the determination of antioxidant activity for example, their application as a rapid evidence of the antioxidant capacity of many organic compounds. The oxidation potentials measured by cyclic voltammetry (CV) used to compare the antioxidant strength of phenolic acids, flavonoids, cinnamic acids as natural compounds in medicinal plants. [2] *Papaver rhoeas L.* (family: *Papaveraceae*) is commonly known as corn poppy and found wild in various parts of the world. Papaver rhoeas L. has a long history of medicinal usage. Extracts derived from this plant have been used for the treatment of a wide range of diseases including inflammation, diarrhea, sleep disorders, cough, analgesia and also the reduction of withdrawal signs of the opioid addiction [3]. In the present work the electrochemical oxidation of etanolic leaf *papaver rhoeas L.* extract has been investigated by cyclic voltammetry and differential pulse voltammetry techniques in the biological pH at the surface of glassy carbon electrode at various concentrations and scan rates. The results revealed that the extract had a *quasi*-reversible redox reaction. Our results show that *papaver rhoeas L.* extract oxidize at low potentials in comparison of quercetin, gallic acid and salicylic acid as standard antioxidants. Antioxidant activities of this extract was evaluated using the 2,2-diphenyl-2-picrylhydrazyl (DPPH) radical-scavenging method and it is directly related to its redox potential value. The results show the good antioxidant activity of the *papaver rhoeas L.* extract and the electrochemical behavior of the constituents in it, at the surface of glassy carbon electrode.

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Determination of “Glycolipid” as Biosurfactant in Hair Baby Shampoo with Lectin Base Affinity Chromatography

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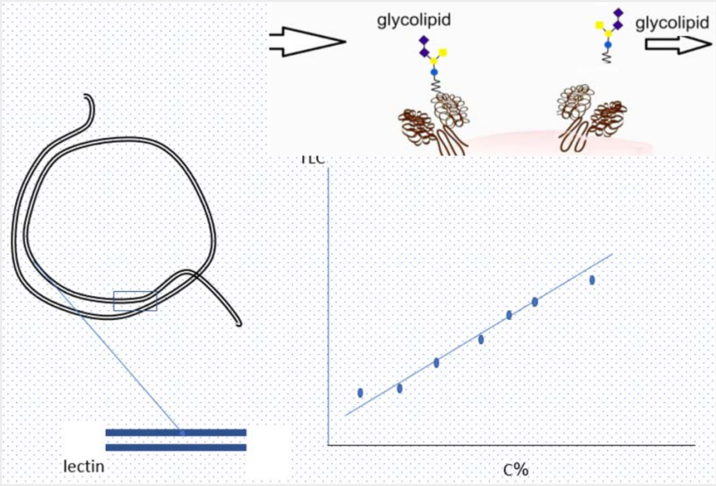

Abstract: Biosurfactants are surface-active molcules produced by microorganisms that have been highlighted as an environmentally-friendly alternative to their synthetic counterpart, chemical surfactants in hair and skin products. The emulsifier function is probably the most important property of biosurfactants in the formulation of cosmetics, because emulsions have considerable advantages over other types of materials. Recently more solvent systems have been described for the separation of glycolipids, quantitative analysis with Chromatography methods has been recognized to be difficult even with post-column detection by ultraviolet (UV), refractive index (RI), and fluorescence detectors. In this work, we reviewing several extraction and analysis methods for biosurfactants in cosmetic products [1-4], finally we examined the amounts of biosurfactants “Glycolipid “used in baby shampoo. Due to the appropriate interaction of this type of biosurfactant with lectin extracted in the plants, we succeeded in using the optimized affinity chromatography method in the capillary column to separate and determine the exact amount of this biosorfactant with using TLC. Results showed that an approximate error of 0.2% with achieve a linear concentration range of 2-13% in the formulation of various baby shampoo products. This method used for determining the amount of several baby shampoos containing glycolipid biosurfactant were analyzed and compared with previous methods, that demonstrate no significant difference was observed in the results. The present method provides easy and usability for other cosmetic products with an acceptable linear range.

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Fe_3O_4 Nanoparticles Modified with Chitosan Functionalized Ionic Liquid as an Adsorbent for The Analysis of Twenty-five Pesticides in Vegetables by Ultrahigh-performance Liquid Chromatography-tandem Mass Spectrometry

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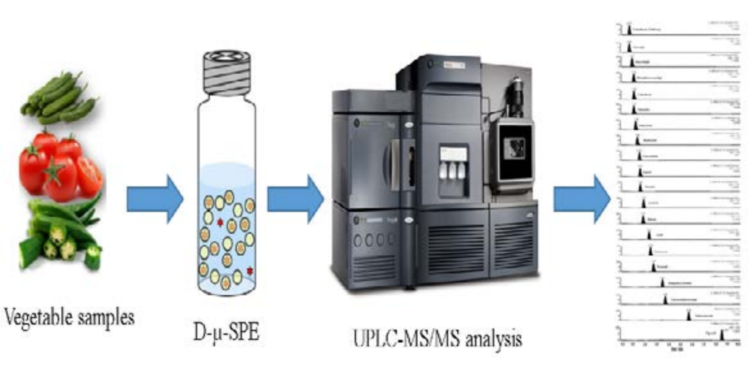

Abstract: This study assessed the presence of 25 pesticide residues in vegetables sold in Tehran's supermarkets and farmer's markets and determined the risk to human health. The estimated daily intake (EDI) and the acceptable daily intake (ADI) were used to calculate the potential health risk assessment of pesticides. Fe_3O_4 nanoparticles modified with chitosan functionalized ionic liquid (Fe_3O_4@CS)-IL NPs) was used as dispersive micro-solid phase extraction (D-μ-SPE) materials for simultaneous extraction of pesticide residues. To monitor these residues reliably, a selective and sensitive, multi-residue system has been developed by ultra-performance liquid chromatography tandem mass spectrometry (UPLC–MS/MS). Fe_3O_4@CS-IL NPs was characterized by SEM, TEM, XRD, FTIR, VSM and TGA. Various condition parameters, such as sample pH, sorbent dosage, extraction time, solvent, volume of the solvent, and desorption time were optimized using central composite design. Under the optimum experimental conditions, limits of detection for 25 pesticides were obtained within the range of 0.031–4.052 $ng.mL^{-1}$. Recoveries ranged from 89.1 to 101.7%. The relative standard deviation values were calculated in the range of 3.6 %–5.9 % (intra-day) and 6.4 %–7.8 % (inter-day). The developed method was validated using matrices consisting of five vegetables (lettuce, cucumber, tomato, pepper, and cabbage) spiked with 25 pesticides at different concentrations. The proposed approach was used to assess 94 samples gathered between 2020 and 2021. In vegetables, eleven pesticide residues were measured. Six pesticides exceeded the European Union's maximum residue limits. However, the sum of the EDI did not exceed ADI. As a result of the findings of this investigation, the identified pesticide cannot be regarded a severe public health concern when consumed in vegetables. To increase food safety and improve public health in Tehran, ongoing monitoring programs and stronger controls for pesticide residues in vegetables are required.

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Removal of Co, Ni and Cd at single-stage purification of zinc solution by Sb_2O_3

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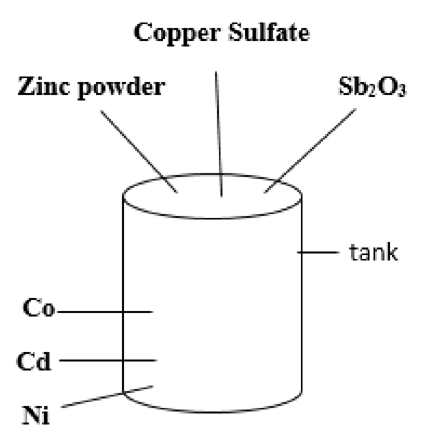

Abstract: Leaching means the selective dissolution of a metal (or several metals) in a concentrate by a suitable solvent to separate and recover the metal. Usually some of the elements in the concentrate will dissolve with the metal, these annoying elements are called impurities. The most important impurities studied in this industry are Co, Ni, Cd, Fe and Pb [1].

The industry uses several tanks to remove these impurities, each of which has a specific temperature and specific materials. In most factories, to remove Co, they use $KMnO_4$ which is considered an expensive substance, and then in another tank, it removes Cd and Ni, but in this method, we remove Co, Cd and Ni in a tank by Sb_2O_3, Copper Sulfate and Zinc powder, which saves time, money and energy. Here we study the effect of pH, Temperature and consuming materials on the removal of impurities, the best temperature and pH with repeated tests measured at different concentrations of Zinc is 85 °C and pH 5.5. The results were examined by a Varian, AA240 atomic absorption instrument and titration method, which has significantly increased the purity of the Zinc ingot [2, 3].

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A Ratiometric Fluorescent Probe Based on Modified Sulfur Quantum Dots for Sensitive Detection of Tetracycline in Chicken Samples

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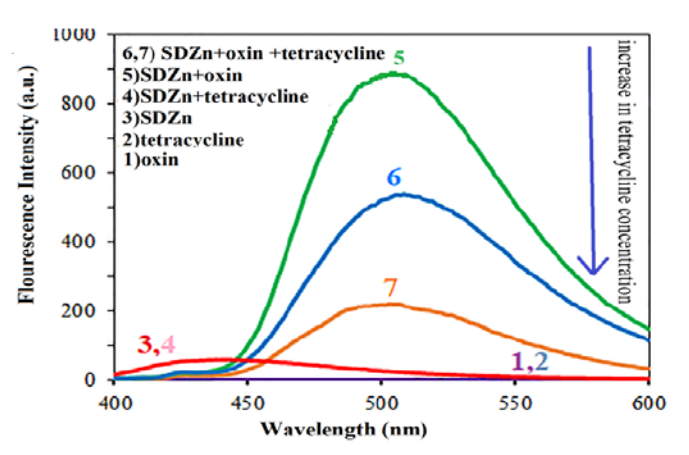

Abstract: Antibiotics residues in animal derived food products are a great concern for public health. The major risk associated with this problem is drug resistance in pathogenic bacteria in consumers. One of the mostly used antibiotics for chicken before slaughter is tetracycline. Herein, a ratiometric fluorescent probe was designed based on new kind of quantum dots which is synthesized on the base of sulfur and Zn under the alkaline situation and reflux, called SDZn Quantum dot. The synthesized quantum dot proved the higher fluorescence intensity in comparison with lone sulfur quantum dot. Tetracycline quenched the enhanced fluorescence intensity at 505 nm which is caused by the interaction of 8-Hydroxyquinoline and SDZn. This fluorescence intensity is higher than the synthesized SDZn with a red shift to the higher wavelength. The fluorescence intensity of SDZn at 450 nm was not influenced by tetracycline, hence, the indirect method as ratiometric measurements were applied for the quantification. The fluorescence color of SDZn changed from blue to light green with increasing tetracycline concentration, which was applied for visual semi-quantitative detection of tetracycline. The suggested method was linear in the range from 1-1000 ppb with correlation coefficient of 0.99. Moreover, the method was used to detect tetracycline in chicken breast and thigh, chicken liver and egg and the recoveries were about 95.0%-103%. The method was accurate, sensitive, and showed good application potential in on-site detection of tetracycline in food samples. The trend exhibited that the eggs contained the greatest tetracycline concentration, followed by the liver, then the breast and lastly, the thigh. It results that not all muscle tissues incorporate antibiotics at the same concentrations in which the breast hold more antibiotics than the thigh. Since, the liver and the kidney are the major storage and excretory organs for tetracycline and are parenchymatous in nature, the higher amount of tetracycline were found in this kind of chicken meat.

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Pharmaceutical and Medical Analysis

isac27-1280	Fereshte Farajian - Payman Hashemi	Silica-Coated Magnetic Tragacanth Gum Nanoparticles Modified with Citric Acid for the Encapsulation and Delivery of Ranitidine	396
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Synthesis, characterization and photothermal therapy performance of Cu-doped NIR-CQDs

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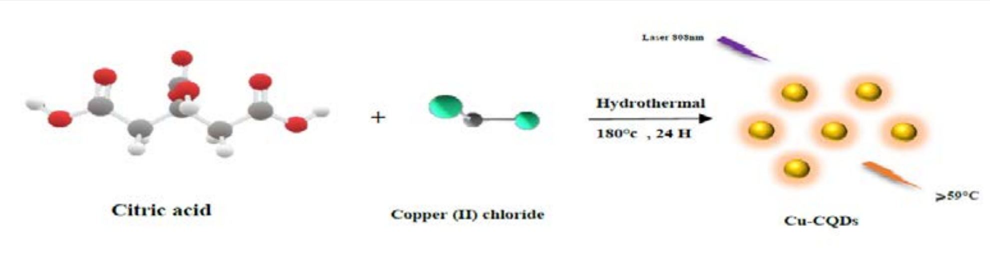

Abstract: Carbon quantum dots (CQDs) are a new class of carbon nanomaterials that have emerged recently and have garnered much interest as potential competitors to conventional semiconductor quantum dots [1]. The accidental discovery of CQDs during the separation and purification of single-walled carbon nanotubes (SWCNTs) by Xu et al. in 2004 triggered subsequent studies to exploit the fluorescence properties of CQDs and create a new class of viable fluorescent nanomaterials [2]. In addition to their comparable optical properties, CQDs have the desired advantages of low toxicity, environmental friendliness low cost and simple synthesis process [1] and potential photoacoustic, photothermal and photodynamic functions. Based on these eye-catching properties, CQDs are considered to be favorable candidates for construction of multifunctional cancer photo theranostic platforms. In order to exploit CQDs in photothermal therapy applications, tuning their absorption in NIR regions are necessary for both deep tissue fluorescence imaging and phototherapeutic functions [3]. The Cu-CQDs was prepared by hydrothermal method using $CuCl_2$ and citric acid in this work, photothermal properties of CQDs were studied and applied for in-vitro cancer cell therapy. An aqueous suspension of 3 mL of 7.5×10^{-4} M Cu-CQDs showed a rapid temperature increase of more than 59 °C after 10 minutes' irradiation with an 808-nm laser. The photothermal effect was studied on MCF-7 cell and acceptable results were obtained. For there we can use it for targeted and effective cancer therapy without side effects.

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Insight into Zeolite/Histidine Nanocomposite as an Advanced Carrier for Metronidazole

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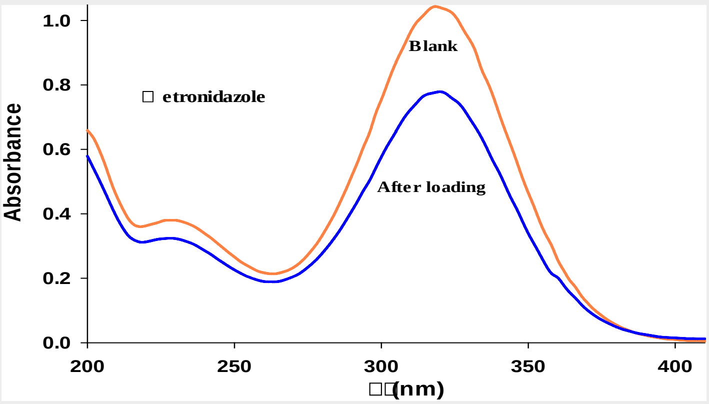

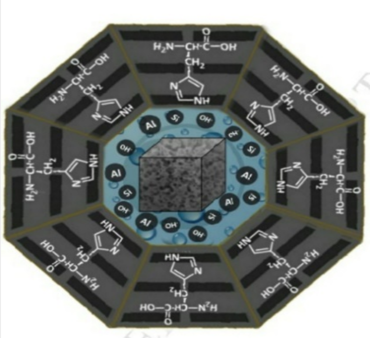

Abstract: The use of porous materials as host systems for medical applications has been considered in recent years. The aim of this work is to construct an efficient carrier for the adsorption and delivering of Metronidazole (Met). For this purpose, pretreated natural nano-sized clinoptilolite (Z) was modified by Histidine (His) and the obtained modified zeolite nanoparticles (Z-His) were used to design a system for loading and release of Metronidazole. The adsorbed and released extents of the drug onto/from the modified zeolite were determined by UV-Vis spectroscopy. The results showed that both decreasing the particle size of clinoptilolite and modifying its surface can significantly increase the adsorbed drug. All the compounds were characterized by SEM, FTIR, and XRD. FT-IR results showed sufficient loading of Histidine and Metronidazole onto the raw and modified zeolite, respectively. IR spectroscopy studies indicated loading of Histidine on the zeolite surface and Metronidazole by the modified zeolite. Due to the presence of hydroxyl and nitro groups in the Metronidazole structure, pH plays an important role on the adsorbed Met.

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Effect of a Nano-sized Zeolite Modified by the Methionine on Betahistine Dihydrochloride

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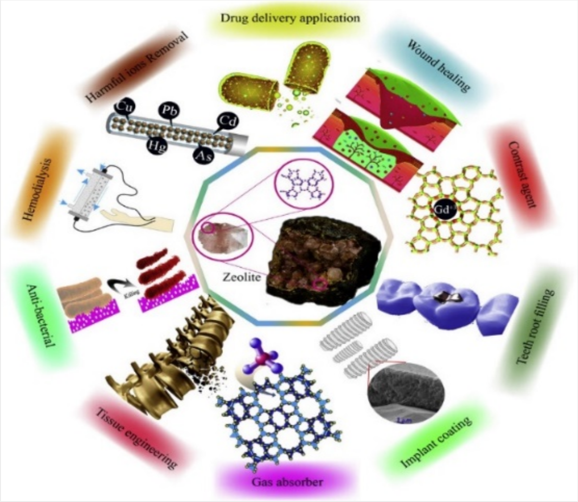

Abstract: Betahistine dihydrochloride (BH.2HCl), an anti-vertigo histamine analog used in the treatment of Ménière’s disease, undergoes extensive first-pass metabolism and suffers from short biological half-life. The aim of the present work was to develop and estimate controlled load and release of BH.2HCl from Zeolite/Methionine nanocomposite as a carrier to overcome this encumbrance. For this purpose, pretreated natural clinoptilolite (Z) was modified by Methionine (Met) and the obtained modified zeolite nanoparticles (Z-Met) were used to design a system for the storage and release of BH. The adsorbed and released extents of the drug onto/from the modified zeolite were determined by UV-Vis spectroscopy. SEM, FT-IR, and XRD were used to characterize the samples. FT-IR results showed sufficient BH was loaded onto the raw zeolite, as well as loading of Met onto the modified zeolite.

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Colorimetric Determination of Captopril Using Mercaptopropionic Acid Functionalized Silver Nanoparticles

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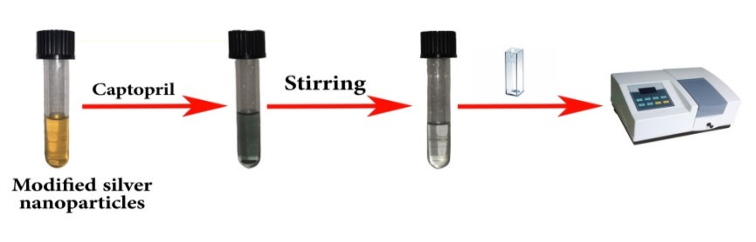

Abstract: Captopril (CPT) as an angiotensin-converting enzyme widely used in the treatment of high blood pressure, coronary artery disease, congestive heart failure. This drug is considered as a drug of choice in the treatment of hypertension due to its effectiveness and low toxicity. It is useful for patients who are chronically ill and need long-term medication. Thus, it is necessary to develop a facile and sensitive method for monitoring of CPT. Up to now, some analytical methods including high performance liquid chromatography, voltammetry, surface-enhanced Raman scattering and chemiluminescence were used for the determination of CPT. In recent years, silver nanoparticles (AgNPs) as colorimetric sensors have drawn extensive attention due to unique optical, low cost, simplicity, sensitivity and physico-chemical properties. The mechanism of measurement is based on change in surface plasmon resonance spectra of AgNPs. Thus, in the present work, a simple, rapid, sensitive and cost-effectiveness colorimetric method based on mercaptopropionic acid functionalized silver nanoparticles was developed for detection of CPT. After addition of CPT, the absorbance intensity was reduced probably due to the aggregation of silver nanoparticles. To increase the sensitivity, the experimental factor affecting the absorbance such as pH, reaction time, temperature and salt addition were optimized. Under the optimal conditions, the calibration curve was linear in the range of 0.1 - 1 mM of CPT and limit of detection (LOD) was 0.03 mM.

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Synthesis of pH-sensitive prodrug micelle for site-specifically release of anticancer drugs

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Abstract: A novel copolymers based on methyl methacrylate polymer (MMA) and methoxy polyethylene glycol (mPEG) with (mPEG)n- b- (PMMA)m architecture were synthesized via atom transfer radical polymerization (ATRP). Subsequent hydrazine reaction with the ester functional group in the copolymer leads to polymers with amine functionalities, which were conjugated with doxorubicin to generate polymer-doxorubicin conjugates. Due to the hydrophobicity of the drug conjugated block, the copolymers took on amphiphilic character leading to micelle formation in aqueous solution with an average diameter of 299.6 nm. In vitro release studies demonstrated that this platinum drug delivery system is relatively stable at physiologic conditions but susceptible to mild acidic environments which would trigger the release of conjugated drugs. As an environmentally sensitive drug delivery vehicle, these conjugates can potentially minimize the drug loss during circulation in the blood, where the pH value is neutral, and after reaching the target cell environment, which has a lower pH. This characteristic drug release profile holds the promise to suppress cancer cell chemoresistance by rapidly releasing a high dose of chemotherapy drugs, thereby improving the therapeutic efficacy of the drug payload.

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Identification of volatile compounds in Tanacetum balsamita by headspace microextraction with deep eutectic solvents

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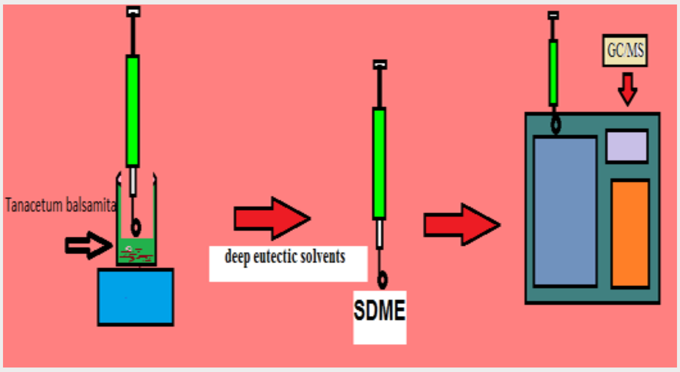

Abstract: deep-eutectic-solvents (DESs) seem to be a cost-effective and alternative for the usage of ionic solvents in biotransformation. DESs are usually developed through gentle warming and stirring of two (low-cost and bio-based) salts. DESs have several superiority over ionic liquids such as their ease of preparation, low production cost and permits large scale applications. Headspace-solvent microextraction (HS-SDME) was made in the current research in order to extract volatile compounds by the use of deep eutectic solvents (DESs) serving as extraction solvents. HS-SDME was constructed as a solvent-minimized extraction method [1]. However, there are a rare number of studies investigating the deep eutectic solvents (DESs) applications to the HS-SDME of bioactive compounds. Deep eutectic solvents, created by mixing choline chloride (ChCl) and p-Chlorophenol at varying ratios, were utilized for extracting essential oils from Tanacetum balsamita via HS-SDME in the present study [2]. Afterward, headspace single-drop micro-extraction (HS–SDME) was conducted, being connected to gas chromatography. HS-SDME is a quick and simple method in comparison with heat reflux extraction. In addition, it is possible to use DESs in HS-SDME for extracting various volatile compounds.

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Evaluation of Extraction of Sulfonamide Antibiotics by Fabrication of α-, β- and γ-Cyclodextrin-Mediated Inclusion Complex using Molecular Docking Studies

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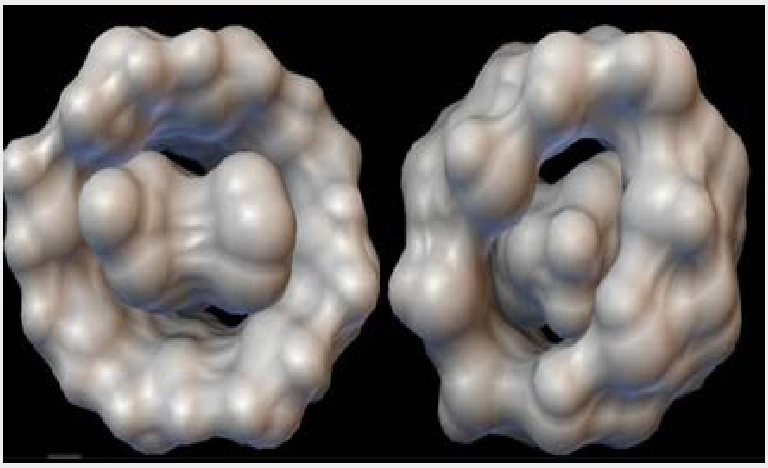

Abstract: The overuse of antibiotics by humans and/or their presence in the environment is considered a serious threat to humans. The entry of these substances into water sources has caused pollution of plants, soil, and animals and has created problems for public health [1-2]. In addition, antibiotics are not completely eliminated in the treatment process and can eventually be released into open water, making microorganisms resistant in the environment. These compounds in nanograms and micrograms can also be stored in the body of animals, poultry, and plants and cause disease for humans and animals [3]. Therefore, it is necessary to control the use of antibiotics carefully and thoroughly controlled. In this study, an attempt was made to evaluate the ability of α, β, and γ-cyclodextrin (CD) in extracting 5 sulfonamides by using host-guest inclusion complexation. According to previous studies, the extraction and pre-concentration of these compounds in aqueous media by cyclodextrin-mediated inclusion complex has not been done so far. Therefore, the computational molecular docking method was used to evaluate guest-host interactions to achieve the ability of CD in the extraction of these compounds and to avoid wasting time and cost. Docking analysis revealed that conventional hydrogen bond and hydrophobic interactions play important roles in the inclusion complex. The modeling experiments helped find the best relative orientation of molecules into the cyclodextrin cavity. From the proposed conformations, the model with the lowest level of connection energy was selected and compared. The results showed the cavity of cyclodextrin type β was the best, and type α was the worst host to create an inclusion complex.

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Designing of Progestrone Drug Nanoemulsion Based on Organic Oils and Optimization of Its Production Conditions Using Chemometrics Methods and Study of Drug Release

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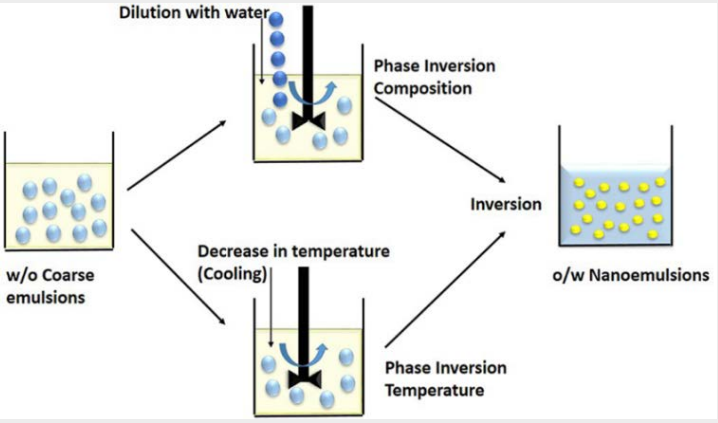

Abstract: Nowadays, the use of organic substances in the preparation of medicines has spread due to less toxicity and side effects. Among these, the use of these materials in the preparation of nanomedicines in the form of tablets, injectable products or topical products is of particular importance. Because the main and ultimate goal of preparing nanodrugs is to create less drug toxicity and increase the efficiency and effectiveness of the drug in the target organ. Accordingly, in this study, Progestrone nanomedicine is initially prepared as a nanoemulsion using an organic drug carrier for targeted drug delivery. Then, using nanomaterial identification methods and analytical techniques, the dimensions of the nanoparticle are prepared and its properties are investigated. In the next step, using statistical analysis techniques, the optimal conditions for the preparation of the resulting nanomedicine will be analyzed. Finally, the release of the drug in an environment similar to the target organ is evaluated using analytical techniques. Nanoemulsion is prepared based on the phase change process. First, the most suitable oil, surfactant and cosurfactant are selected according to the solubility of the active ingredient in it using the solubility of the active ingredient in the same conditions. The organic phase consists of an organic oil, a mixture with a certain ratio of surfactant and cosurfactant, and an active ingredient such as progesterone at 75 °C, and homogenized by a stirrer at a rate of 600 rpm. The aqueous phase consisted of double distilled water and, if necessary, a preservative such as methyl paraben or propyl paraben was prepared separately at 75 °C.

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Preparation of Organic Based Nano Carrier and Optimization of Experimental Conditions

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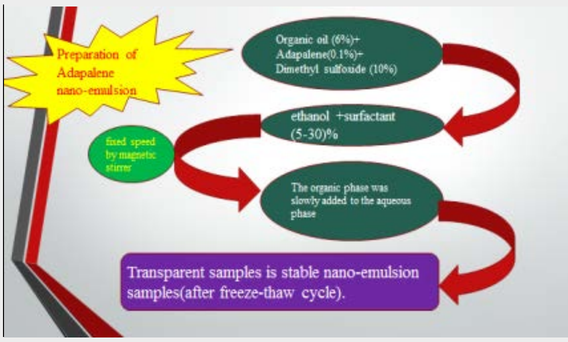

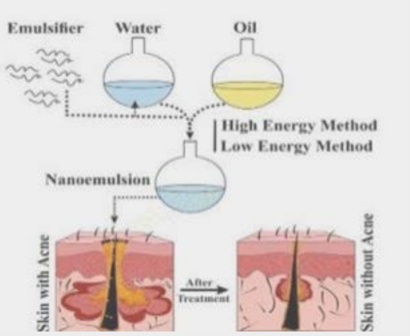

Abstract: This study aims to evaluate the effect of type of organic oil, different surfactant, and cosurfactant such as Cremophor RH 40, Cetomacrogol 1000, and polysorbate 80 (tween 80) and polysorbate 60 (tween 60) on the mean particle size of nano-emulsion and their preparation (by TEM and zeta potential and other analytical techniques). Different effects of nano-emulsion size were studied by type of organic oils and surfactants and concentration of cosurfactants. According to solubility, particle size, poly-dispersity index, morphology, viscosity, and pH, a suitable formulation is chosen. Formulations were developed based on a multilevel factorial design (three factors, two levels). Some properties of nanoparticles like process yield, drug–surfactant interaction, particle morphology, and in vitro diffusion profile, which could affect biological performance also be evaluated. Dermal and transdermal drugs and oral vitamin syrups were chosen for this study. First, the designed formulations were evaluated by considering the PYs (process yields), which can be defined as the weight ratio of the amount of NPs to the total amount of organic oil and active ingredient used. A multilevel factorial design (2^3) was used to study the effect of each factor, as well as the effects of interactions between factors, on the variable response. The studied factors were the amounts of surfactant (Cremophor RH40, Cetomacrogol 1000, and Tween 80) (X1), organic oil (X2), and solvent (X3). The response variable was the LAPI (Y), expressed in percent (related to the amount of active ingredient loaded on nano-emulsion). All experiments were carried out three times. The order of the experiments was fully randomized. The LAPI (Loaded Active Pharmaceutical Ingredient (%)) was determined by an HPLC method. First, the calibration curve was obtained (for Adapalene at 235 nm). The amount of Adapalene loaded into NPs was estimated directly by the dissolution of 10 mg of NPs in 10 mL of mobile phase at room temperature. After the NPs had dissolved, HPLC measurements were done. Then, Adapalene concentrations were calculated from the equation of the standard curve. All LAPI were calculated in triplicate (n = 3) by using equation LAPI values are expressed as mean ± standard deviations.

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A Brief Look at Machine Learning Methods in Chemical Toxicity Prediction

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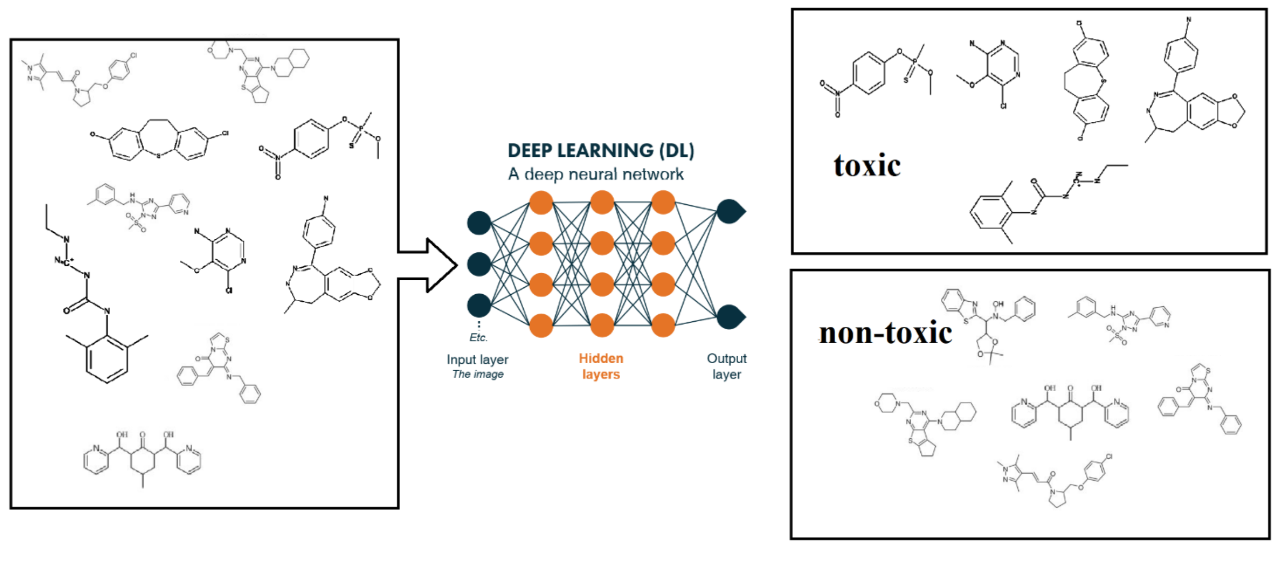
Graphical Abstract



Abstract: The most important aim of modern toxicology efforts is based on the detection of chemical compounds which have the toxic potential. By introduction of newly synthesized chemical compounds, it is necessary to evaluate the toxicity behavior of these newly emerged compounds very quickly [1, 2]. Usually, conformal prediction strategy is used for this purpose but the most challenge of this area is the uncertainty associated with predicted models in conformal predictor [3] Therefore development and introduction of techniques with the ability of toxicity prediction is among the most challenges in scientific communities [3, 4]. Nowadays, deep learning is used in order to overcome this problem [3]. Deep forward neural networks and graph neural networks are two major deep learning techniques usually applied for computational toxicology. In the present paper challenges and recent advanced based on deep learning in the field of computational toxicology is discussed.

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Silica-Coated Magnetic Tragacanth Gum Nanoparticles Modified with Citric Acid for the Encapsulation and Delivery of Ranitidine

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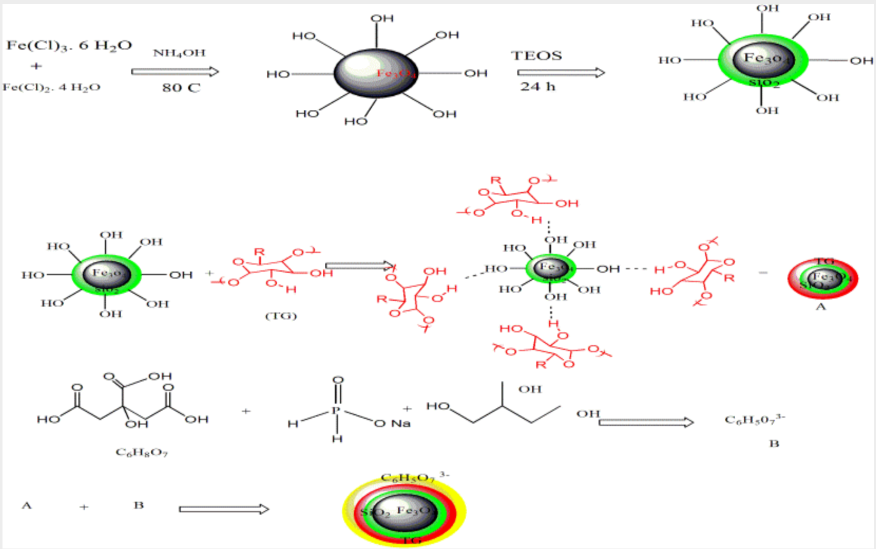
Graphical Abstract



Abstract: A new magnetic nanocomposite was prepared by synthesizing Fe_3O_4@SiO_2 nanoparticles and then coating them with a shell of tragacanth gum (TG) as a natural product modified by citric acid (CA). Fe_3O_4@SiO_2@TG@CA nanoparticles obtained were identified by scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDX) and Fourier transform infrared spectroscopy (FT-IR). Magnetic nanoparticles prepared were used for loading and delivery of ranitidine, an oral drug. Conditions for drug loading were optimized by the central composite design optimization method. The maximum adsorbent loading efficiency for ranitidine was 76.3% that was obtained at pH 11 and its release in vitro was gained at pH 1.6 in a phosphate buffer medium. The loading capacity of the adsorbent was dependent on the initial concentration of ranitidine and exceeded 5.2 mg g^{-1} in a solution of 100 mg L^{-1}. The study of adsorption isotherms to describe the interaction of ranitidine with adsorbent showed the best fit with Freundlich isotherm [1]. The results showed that the prepared adsorbent Fe_3O_4@SiO_2@TG@CA, as a non-toxic and low-cost adsorbent, is quite suitable for drug delivery applications [2].

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Tuberculosis Infection Prediction Based on Raman and Surface Enhanced Raman Spectroscopy

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Abstract: Identifying the possible infections and diseases for patients hastily of time with reasonable accuracy is one of the main challenges that can be solved using machine learning [1-3]. Different approaches have been discussed today, such as based on images to diagnose cancer [4]. This paper proposes a method of tuberculosis prediction based on Raman and surface-enhanced Raman spectroscopy. We used deep learning-based architecture models to solve the problem presented by Kaewseekhao et al. [5]. The base model contains dense layers for matrix multiplication of input and predicting the outputs, the Batch Normalization layer that normalizes the output of the dense layer, and, Dropout layers to stop the model from overfitting over train data. Adam optimizer with a learning rate of 0.0001 and Sparse Categorical Cross entropy were chosen for optimizer and loss function for the model, respectively. We train the models for 60 epochs with call back based on validation data's accuracy to save the best model throughout the training process. Two approaches were used to analyze the models. The first one was a Kfold Classification using ten folds, and the other separated the train and test data randomly, with the training data containing 70% of the data and the test data comprising 30%. Based on the outcomes, the Kfold analysis of the models shows an accuracy of 77%, and for the random-based model, the accuracy is 73%. To better understand the performance f the model, we calculate the recall too. For the Kfold model, the recall was 75% and for the random based was 72%. All the codes, models, and data are available free of charge on this GitHub repository.

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Analytical Chemistry in Forensic Science

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A new disposable glass cuvette for visible spectrophotometry of carboxyhemoglobin detection in postmortem blood

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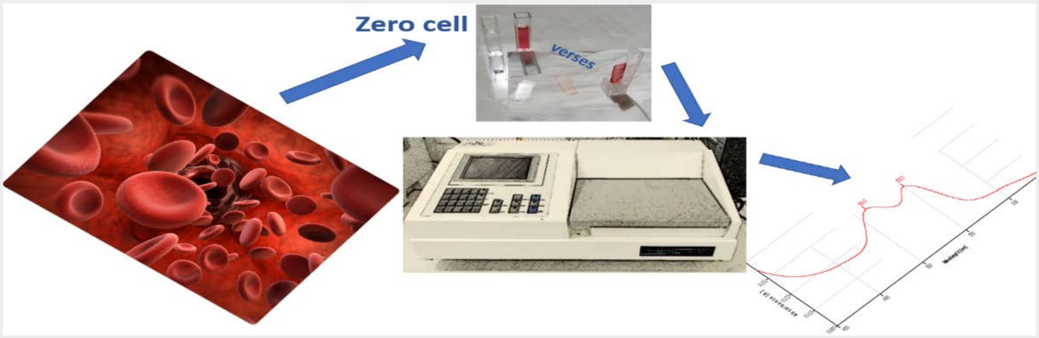
Graphical Abstract



Abstract: Carbon monoxide (CO) is a colorless, odorless, and highly toxic gas that results from incomplete combustion of fossil fuels or contact with dichloromethane vapors [1]. Due to the tendency of CO to hemoglobin (Hb), which is 250 times more than oxygen, its poisoning is one of the major causes of death in developed countries. One of the methods for CO diagnosis is qualitative and quantitative detection of carboxyhemoglobin (COHb) in blood samples using the UV-Vis spectrophotometry. The basis of this method is based on the difference between the absorption peak of the visible spectra of oxygenated forms of hemoglobin (HbO_2) and methemoglobin in the blood after reduction (deoxyhemoglobin) with the pattern related to COHb. In this study, a new Zero Cell (ZC) for visible (Vis) spectrophotometric measurements was designed and the visible spectra at 450 to 650 nm was compared with conventional 1 cm glass cells before and after addition of the reducing agent. To make zero cells (ZCs), a lamel (cover glass) with dimensions of 0.15 × 50 × 24 mm was used. Each lamel was cut into six smaller lumens (ZCs) with a diamond pen, and for each sample, two ZCs were used (blank form also consisted of two empty ZCs). To examine the blood sample, 6 µL of blood sample was placed by the dispenser on two sheets of ZC, and then 0.01 g sodium dithionite was added on the second sheet. As soon as the second slide was placed, the blood sample was automatically spread on the slide. Two cells with a uniform layer of blood were prepared for the detection of pre- and post-regenerated hemoglobin for every single examination. The resulting spectra were recorded at the mentioned wavelengths. The oxyhemoglobin and methemoglobin related peaks appear in the form of a double-wave spectra, and after addition of sodium dithionite to the blood, hemoglobin is reduced. Thus, in negative cases, the twin peaks in the regions of 535-545 and 570-585 nm are changed to a single peak after reduction, and in positive cases, it remains unchanged with two peaks because of stronger binding of CO to hemoglobin. The advantages of the proposed method are the disposable use of Vis-cell, prevention of carryovers and measuring the carboxyhemoglobin without dilution of the blood sample.

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Targeted sampling effect, on the percentage of positive responses in a forensic laboratory, in three one-year, periods of ten years

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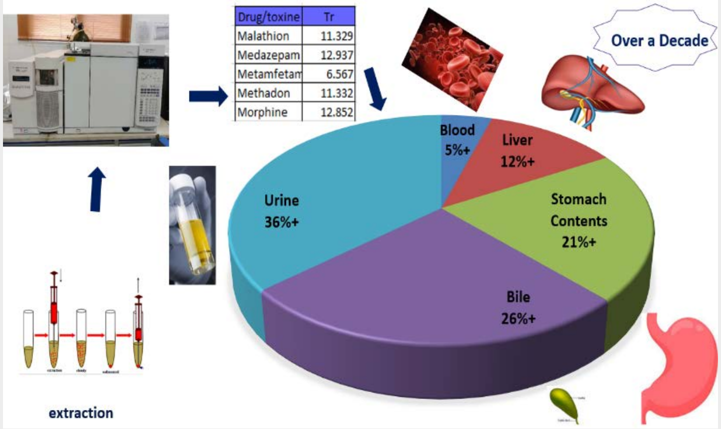
Graphical Abstract



Drug/toxine	Tr
Malathion	11.329
Medazepam	12.937
Metamfetam	6.567
Methadon	11.332
Morphine	12.852

Abstract: The forensic toxicological analysis involves a variety of samples for drug detection and with technological advancements, the need for targeted sampling increases. Various samples are analyzed after the sample preparation, often by gas chromatography-mass spectrometry (GC-MS). Depending on the drugs (pharmacodynamics and kinetics), there are general scientific predictions about the distribution of drugs in the body [1], but in practice, what will be the positive results in the same extraction method?

The objective of this article was to provide another level of biological matrix classification that can affect results in the same sample preparation techniques. This study was performed on about three thousand postmortems of the toxicology laboratory of forensic medicine (Guilan), in three one-year periods (1390, 1396, and 1400), during a decade. Urine, liver, stomach, blood, and bile after liquid-liquid extraction were examined by GC-MS in a general method (HP_5 MS column with initial, final, inlet, and interface temperatures and the rate of temperature, were respectively, 60 ºC, 280ºC, 250 ºC, 280, ºC and 20 ºC min^{-1}). The positive responses were determined in each group of samples by studying for drugs, toxins, and their metabolites in the list of about 650 organic compounds (such as medicines, drugs, toxins, etc.) which were already registered in the local software library with a determined retention time and a high match quality (more than 75%). Positive and negative results were recorded and compared by sample type. The results by years were determined and the statistical percentage of positive (desire) results, were as follows; urine, bile, stomach content, liver, and blood in 1390, were respectively 0, 36, 5,54,88, 1n 1390, 11, 25, 50,72, 1n 1396 and 10, 15, 44,49 77 1n 1400 (a total average can be seen in the pie chart, with 36% urine, 26% bile, 21% stomach content, 12% liver, and blood, 5%). In all three research periods, the results showed that the positive answers were related to the following samples: urine, stomach contents, and bile, which were referred to as the "golden triangle of diagnosis”. Each sample has its characteristic, but in the case of some others, this characteristic is more obvious e.g., in urine samples, drugs can be concentrated up to 100 times and the possibility of finding drug metabolites in urine is high. Targeted sampling is not only useful in sample selection in toxicology laboratories but also, spends fewer resources on detection, is cost-effective, and is in line with environmental considerations.

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Unambiguous Identification of Chemicals related to the Chemical Weapons Convention (CWC) in Environmental Samples in the 50th International Official OPCW Proficiency Test

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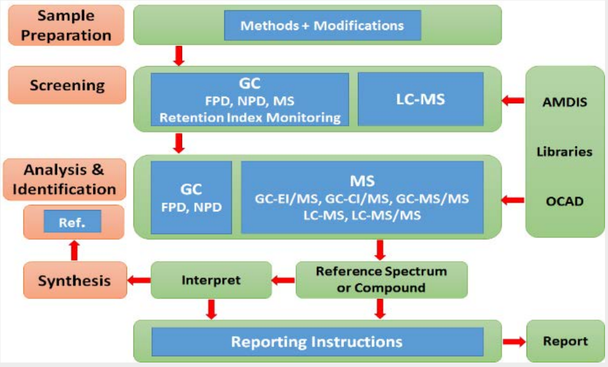
Graphical Abstract



Abstract: The OPCW (Organization for the Prohibition of Chemical Weapons) has a worldwide network of designated laboratories for environmental and biomedical sample analysis related to chemical weapon convention (CWC). These laboratories are required to have international accreditation like ISO/IEC 17025 and participate in annual proficiency tests to keep their designation [1]. The samples are collected by the OPCW inspection team according to the special scenario and analyzed at the OPCW's designated laboratories in each proficiency test. Currently (2022), OPCW has 24 laboratories designated for environmental samples [2]. The 50th Official OPCW Proficiency Test was conducted by OPCW in October and November of 2021, in which 6 liquid samples including 3 solvent waste samples and 3 aqueous waste samples were sent by OPCW in the volume of about 5 and 7 mL, respectively. DCRL received the samples on November 14, 2021. The samples were analyzed for reporting scheduled chemicals related to CWC and/or their degradation/reaction products during 15 days. Liquid-liquid and solid-phase extractions, direct evaporation, different clean-up and various derivatization procedures were used for sample preparation. For unambiguous identification of chemicals, different analytical techniques such as GC-MS (EI), GC-MS (CI), GC-FPD and LC-MS/MS (ESI) were utilized. According to the analysis rules of OPCW, the identification is considered unambiguous if two different spectrometric techniques giving consistent results confirm the presence of the same chemical [3]. In order to support the identification of the reported test chemicals, it is essential to search the measured data against spectral libraries such as the National Institute of Standards and Technology (NIST) MS library or OCAD (OPCW Central Analytical Database). In most cases, the identification is verified by comparison to a reference chemical analyzed under identical conditions [1, 3]. In the 50th Proficiency Test, six spiking chemicals related to CWC were unambiguously identified. All of them were compared with reference standard chemicals which were synthesized at DCRL during the test period. Sufficient data were presented to support the identification of spiking chemicals in accordance with OPCW rules [3]. According to final report of OPCW, DCRL could acquire "A" (maximum) score in the 50th Official OPCW Proficiency Test and keep its designation.

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Verification of Nerve Agents Exposure by Identification of Free Metabolites and Protein Adduct Biomarkers in Human Plasma Samples in the 7th International Official OPCW Biomedical Proficiency Test

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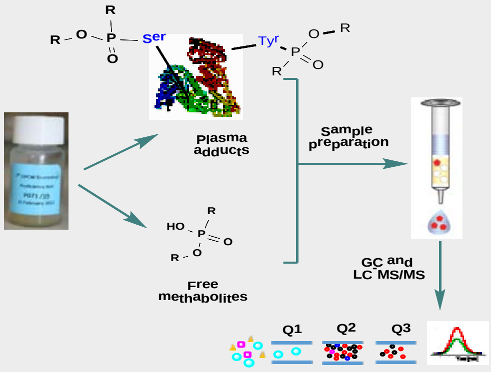
Graphical Abstract



Abstract: The purpose of this study was analysis of human plasma samples for protein adducts and free metabolites with different analytical techniques in order to investigate the presences or absence of nerve agents. Organophosphorus Nerve Agents (OPNAs) target the enzyme Butyrylcholinesterase (BuChE) and albumin to cause acute toxicity [1-2]. In this Biomedical Proficiency Test, plasma samples (7×5 mL vials with codes of P071-P077) were received (1 March, 2022) from OPCW Laboratory (Rijiswijk, The Netherlands) and analyzed for verification of human exposure to nerve agents. BuChE activity was measured spectrophotometrically using an Ellman assay as a preliminary test to diagnose exposure to OPNAs. Fluoride regeneration method and clean-up with HLB solid phase extraction applied to release the agents again. BuChE from plasma purified by Sepharose Q fast flow gel (anion exchange)-procainamide affinity gel chromatography, digested with pepsin, cleaned-up by C18 solid phase extraction and the modified nonapeptide adducts were identified by LC-MS/MS. Totally protein precipitated, digested with pronase E enzyme, cleaned-up by C18 solid phase extraction, then the tyrosine adducts were identified using LC-MS/MS. Cleaning up by LC-Si cartridge, derivatization with Pentafluorobenzyl bromide followed by GC-MS/MS analysis was applied to identify the free metabolites of agents in plasma samples. All reported chemicals were in accordance with preliminary report of the seventh Official OPCW Biomedical Proficiency Test. Cyclosarin was identified in plasma samples with codes of P073 and P074 with real concentration of 20 ng/mL according to OPCW reports. VX and CVX with concentrations of 10 and 20 ng/mL were identified in the plasma samples with codes of P075 and P077, respectively. Reference chemicals (own synthesis) were used for comparison of the obtained data with the test samples. The samples with codes of P071, P072 and P076 were blank samples. The Defense chemical research laboratory (DCRL) correctly identified all the spiked chemicals in the concentration range of 10-20 ng/mL in plasma samples with sufficient analytical data. In conclusion, presented methods could constitute valuable techniques for the forensic verification of exposure to nerve agents.

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Determination of Chlorine Gas Candidate Biomarkers 3-chlorotyrosine and 3,5-dichlorotyrosine in Plasma Samples

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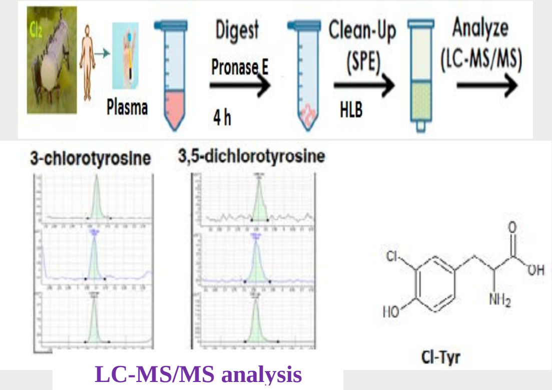

LC-MS/MS analysis

Abstract: Chlorine gas (Cl_2) is a public health concern and potential threat due to its high reactivity, ease and scale of production, widespread industrial use, bulk transportation, massive stockpiles and history as a chemical weapon. After utilizing the chlorine, e.g. in chemical warfare, it is challenging to detect it in the environment, since it rapidly decomposes. Toxicity is due to the formation of hydrochloric acid (HCl) and hypochlorous acid (HOCl) through hydrolysis in the moist linings of the airways. Chlorine or its decomposition products are known to form stable adducts with tyrosine (Tyr) residues in proteins. This work describes a new and sensitive method for the retrospective detection and quantitation of two chlorotyrosine adduct biomarkers, 3-chlorotyrosine (Cl-Tyr) and 3,5-dichlorotyrosine (Cl_2-Tyr), which were isolated after protein precipitation and pronase digestion of plasma sample spiked with sodium hypochlorite (NaOCl) solution. Cl_2-Tyr adduct is distinct representative of exposure to chlorine gas and it is observed in the concentration of more than 50 ppm. Digested samples were clean-up by solid-phase extraction (HLB) and identified by liquid chromatography tandem mass spectrometry (LC-MS/MS) [1-2]. Analysis was performed in the positive ionization mode using multiple reaction monitoring (MRM) of the ion transitions at m/z 216.1 / 135, 170, 199 for Cl-Tyr [1]. Afterwards, matrix effects, LOD, LOQ, precision, calibration range and recovery range were evaluated for the proposed method. The calibration range was estimated at 0.1–50 mg/L ($R^2 \geq 0.995$) with LOD and LOQ of 0.05 mg/L and 0.1 mg/L for Cl-Tyr, respectively. RSD for Cl-Tyr was obtained at 2.9%. Matrix effects were also evaluated at 24.75%. SPE recoveries for Cl-Tyr was 116%. For the animal phase study, 8 male rats (two groups each containing four rats) were intramuscularly treated with 5 and 50 mg/kg sodium hypochlorite (NaOCl) solution, respectively. The rats' plasma were analyzed for the presence of Cl-Tyr through above mentioned method. The presence of Cl-Tyr in rats' plasma was confirmed in all the intramuscularly treated rats by LC-MS/MS. In conclusion, pronase digestion combined with LC-MS/MS (MRM) analysis of chlorinated tyrosine adduct could constitute a valuable technique for the forensic verification of exposure to chlorine gas.

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THE END