



The Eleventh

Iranian Biennial

Electrochemistry Seminar

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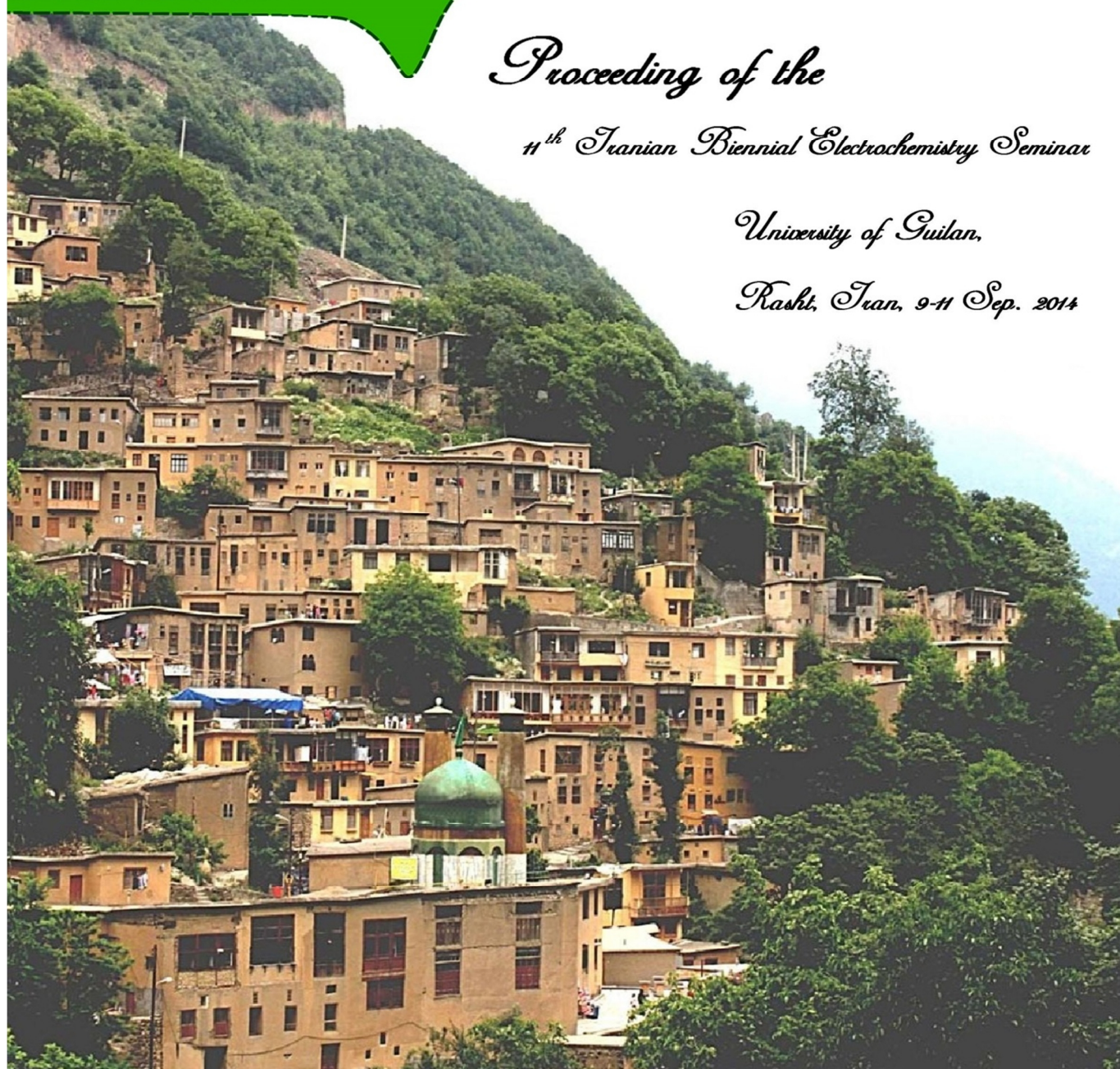
9-11 September 2014

Proceeding of the

11th Iranian Biennial Electrochemistry Seminar

University of Guilan,

Rasht, Iran, 9-11 Sep. 2014





In the name of God, the Merciful, the Compassionate

Proceeding of the

The 11th Biennial Electrochemistry Seminar

September 9-11, 2014

Department of Chemistry

University of Guilan

Rasht, Iran

With Collaboration of

Iranian Chemical Society

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University of Guilan, Rasht, September 9-11, 2014



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Keynote Speakers



ELECTROCHEMISTRY FOR GREEN ORGANIC SYNTHESIS

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Abstract

The twelve principles of green chemistry, include, prevention of the waste, high atom economy, less hazardous chemical synthesis, designing safer chemicals, safer solvents and auxiliaries, using renewable feedstocks, reduce derivatives, catalysis, design for degradation, real-time analysis for pollution prevention, inherently safer chemistry for prevention of accidents. On the other hand, there are several features of electrosynthesis that are often cited as being environmentally favorable. Electrons are considered to be clean reagents to effect on oxidation and reduction reactions; that, under usual circumstances, require potentially polluting metal-based reagents. Such procedures have considerable waste streams which require downstream remediation. In addition, electrosynthesis is characterized by high selectivity, good atom economy, readily available starting materials, low- temperature and energy consumption, low costs for reagents and material failure. Furthermore, the electrodes may be regarded as heterogeneous catalysts that are easily separated from the products. So, it can be concluded that, electrosynthesis is a green tool for organic synthesis. This special issue focuses on using green electrochemical methods for organic synthesis.

Keywords: Electrosynthesis, Green chemistry, Atom economy, Clean reagents, Heterogeneous catalyst.



New advances in design and manufacture of Iranian electrochemical measurement systems: Application of advance of electronic and Mathematical methods in Modern electrochemistry

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Abstract

This seminar is an illustration of the new developments in design and manufacturing of electrochemical instrumentation, in Iran (or in Center of Excellence in Electrochemistry, CEE). The new devices include the interfacing of instrumentation to computer for the purpose of creating new electrochemical techniques. The in-house construction has a professional computer program, which offers various new voltammetry techniques based on FFT AC, FFT-EIS, FFT- square wave and cyclic voltammetry. Also, on-line data acquisition data-processing options using combination various mathematical procedures (such as Fast Fourier transform) that were not practical before in electrochemistry. Even more noteworthy, there is the possibility of performing data analysis for decomposing complex signals into their component parts for extracting more information from the available data and displayed them in two and three dimensional plots. Also, in our system, there are many of computer-based electrochemical simulation for cyclic, AC, Square wave voltammetry and EIS. These instruments can often make difficult measurements very easier; in what ways the new electrochemical system comes to be superior to the classical methods.

In Situ Spectroelectrochemistry of Conducting Polymers, Copolymers and Conducting Polymer Nano Composite Materials: Development and Applications

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Abstract

Functionalized conducting polymers (CPs), copolymers and conducting polymer nano composite materials have been in the focus of considerable attention due to their interesting electrical and optical properties and conceivable applications in the last three decades [1-3]. CPs aroused a great interest due to its large field of applications such as energy storage devices sensors and biosensors, batteries, and others [2]. Incorporation of nanostructures inorganic compounds into the conducting polymer will facilitate the combination of different features of organic and inorganic species. The inorganic fillers in the nano form are expected to modify the properties of the compound leading to the development of multifunctional devices. The synthesis of polymer/inorganic nanocomposites materials has the goal of obtaining a new nanocomposite material having synergetic or complementary behaviors between the polymer and inorganic material [3]. Results of in situ spectroelectrochemistry of CPs [4, 5] (UV-Visible, Raman spectroscopy, conductivity and etc.), in situ characterization and copolymers related to the nano materials, in situ electropolymerization, growth rates of CPs on modified electrodes as a function of cycle number of monomer concentrations and some applications of CPs were presented.

Keywords: Conducting polymers, Nano composites, Applications

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Physielectrochemical aspects in electroactive semiconducting polymeric and relevant nanocomposite films

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Abstract

Conducting polymer (CPs) such as polypyrrol, polythiophen or poly ortho aminophenol represent a group of conjugated π -electron materials which process a combination of various electrical, optical and other semiconductor properties. To improve the performances or extend the functions, CPs usually have to be nanostructured. Electrosynthesis provides an effective and convenient one-step approach to CP nanomaterials. The resulting materials are usually oriented on the electrode surfaces and their properties are easy to be controlled. This review focuses on the syntheses of CPs and relevant nanocomposites by electrochemical polymerization. The applications of the nanomaterials as electrocatalyst and supercapacitors also will be discussed [1-3]. It is well-known that without redox species in solution the charge-transport process of an electrode modified by an electroactive polymer film is affected by ionic transfer at the polymer|electrolyte interface, ionic and electronic charge carrier transport inside the polymer and an electronic transfer at the metal|polymer interface. The other main of the work is to present more insight into the processes appearing during oxidation and reduction of a CPs film electrode in different aqueous electrolytes, role of the electrosynthesis parameters on the kinetics, semiconducting properties, morphology of the CPs, anomalous diffusion behavior of anions, relationship between the fractal dimension of CPs film and diffusion coefficients using electrochemical methods. Ordinary and anomalous diffusion of the ions in the electrolyte and restricted area of the film have been discussed in detail by electrochemical impedance spectroscopy (EIS).

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Electrosynthesis of Pyrano[2,3-*d*]Pyrimidinones and their voltammetric behaviour

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Abstract

There is a continuous widespread interest in the synthesis of pyrano-pyrimidinones because of the diverse biological properties associated with this system. Compounds with such annulated uracils have antitumor, antibacterial, antihypertensive, hepatoprotective, cardiogenic, vasodilator, bronchodilators and antiallergic activities and some of them exhibit antimalarial, antifungal, analgesics and herbicidal properties [1-3]. Electrocatalytic transformation of barbituric acid, aldehyde and malononitrile in ethanol in an undivided cell in the presence of potassium bromide as an electrolyte results in the formation of substituted pyrano[2,3-*d*] pyrimidinones at room temperature in 65-85% yields. The progress and completeness of electrolysis were monitored by cyclic voltammetry (CV). The voltammograms were recorded with μ Autolab FRA2 Potentiostat-Galvanostat. A three-electrode system was used with a platinum rod as working electrode, a GC electrode as the counter electrode and an Ag/AgCl as the reference electrode. The structure of products was characterized by FT-IR, ¹H NMR and ¹³C NMR spectroscopy. In this electrosynthesis, the first step is electrochemical that follows by chemical reaction. Therefore the proposed mechanism is EC. The cyclic voltammetric investigation of some products showed that these compounds were electroactive in appropriate potentials at the Pt rod-working electrode.

Keywords: Pyrano[2,3-*d*]pyrimidinones, Electrosynthesis, Cyclic voltammetry, Electrochemical behaviour

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Evaluation of Pitzer ion interaction parameters of some electrolyte system by using potentiometric method at T=298.15 K

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Abstract

The properties of electrolyte in water and mixed water-organic solvents are of particular interest in many industrial and environmental applications, particularly, for separation processes. To measure the thermodynamic properties of electrolytes, the most common methods are the isopiestic vapor pressure [1] and potentiometric techniques [2]. During the last decades, the series of ion-interaction models for electrolyte solution have been proposed to predict the activity coefficient of each solute and osmotic coefficient of aqueous systems. Among them, the Pitzer ion-interaction model is one of the most famous and useful models. Pitzer equations, with much accuracy, correlate the thermodynamic properties of mixed aqueous electrolyte systems. The Pitzer model extended the Debye–Hückel method with a virial expansion to account for the ionic strength dependence of the binary repulsive short-range forces, and higher order interactions [3]. In this study, Pitzer ion interaction parameters of several electrolyte systems involving cobalt, nickel and magnesium chloride in water and water-organic solvent were determined and compared with each other. The mean activity coefficient of salts in water or mixed solvent system were determined by using the potentiometric technique at T= 298.15 K. The measurements were carried out on the galvanic cell without liquid junction. The PVC based ion selective electrode (M-ISE) and Ag-AgCl electrode used in these works were prepared in our laboratory and had a reasonably good Nernst response. The Pitzer ion-interaction parameters (β^0 , β^1 , C^ϕ , θ and Ψ) for the series system were estimated by fitting experimental results with the Pitzer model. Then, the parameters obtained with the Pitzer model were used to calculate the values of the osmotic coefficients (ϕ) and the excess Gibbs free energies (G^E), which is one of the most important properties, for the whole series of the studied mixed solvent electrolyte systems.

Keywords: Activity coefficient, Pitzer model, CoCl_2 , MgCl_2 , NiCl_2 , Emf method

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Use of nanoelectrodes and ultramicroelectrodes for fabrication of electrochemical DNA (E-DNA) nanosensors and detection of reactive oxygen species (ROS) in prostate cancer cells

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Abstract

We report the use of gold-modified recessed platinum (Pt) nanoelectrodes in the fabrication of linear and stem-loop probe-based electrochemical DNA (E-DNA) sensors. Pt nanoelectrodes with a radius less than 10 nm were reproducibly fabricated using an optimized laser pulling technique. Prior to sensor fabrication, the nanoelectrode was electrochemically etched to create a recessed nanopore, followed by electrodeposition of gold into the nanopore using either cyclic voltammetry or constant potential amperometry. In addition, we systematically determined the optimal experimental condition for DNA probe immobilization and target interrogation. The electron transfer rate constants of methylene blue, as determined using alternating current voltammetry, were found to be much higher than those obtained from E-DNA sensors fabricated on conventional macroscale electrodes. While this unique phenomenon requires further investigation, our results clearly show that these gold-modified nanoelectrodes can be used as substrates for this class of electrochemical biosensors. In the second part, release of ROS from prostate cancer (PC3) cells was studied using scanning electrochemical microscopy (SECM) and fluorescence microscopy. One-directional lateral scan SECM was used as a rapid and reproducible tool for simultaneous mapping of cell topography and reactive oxygen species (ROS) release. Fluorescence microscopy was used in tandem to monitor the tip position, in addition to providing information on intracellular ROS content via the use of ROS-reactive fluorescent dyes. A unique tip current (i_T) vs lateral distance profile was observed when the tip potential (ET) was set at -0.65 V. This profile reflects the combined effects of topographical change and ROS release at the PC3 cell surfaces. Differentiation between topographical-related and ROS-induced current change was achieved by comparing the scans collected at -0.65 and -0.85 V. The effects of other parameters such as tip to cell distance, solvent oxygen content, and scan direction on the profile of the scan were systematically evaluated.



Seminar Abstracts



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Electrochemical preparation of polyproline nanofilm containing copper and nickel nanoparticles on glassy carbon electrodes and evaluation of their electrocatalytic abilities

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Abstract

Recently, developing alternative energy sources such as fuel cells have attracted much attention. So in this article we have tried to introduce a new type of electrodes which can be used in oxidation of alcohols in fuel cells. The important aim in fuel cells has been focused on use of electrodes with low over voltage in electrooxidation process. Modified electrodes are of great groups for this purpose. In the present work, possible polymerization of L-proline using cyclic voltammetry on glassy carbon electrode was studied [1]. The obtained results showed that L-proline is easily polymerized on the electrode surface. In the next step, the possibility deposition of copper and nickel nanoparticles on glassy carbon electrode modified with polyproline film using constant potential coulometry technique was investigated. After entering nanoparticles of copper and nickel on nanopolymer films, the electrocatalytic ability of the modified glassy carbon electrodes towards electrooxidation of three kinds of alcohols (ethanol, methanol and ethylene glycol) in alkaline solutions was evaluated. The characteristic of modified electrodes in various conditions was determined.

Keywords: Polyproline, Glassy carbon electrode, Copper and nickel nanoparticles, Electrocatalysis, Alcohols

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Nickel-poly (1,8-diaminonaphthalene)/multi-walled carbon nanotubes modified glassy carbon electrode; an electrocatalyst for methanol oxidation

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Abstract

Direct Methanol fuel cells have recently received a lot of attention because of their high power density, high vehicle efficiency, relatively quick startup, rapid response to variations in loading, long life, simple system design, and low operating temperature when compared to conventional rechargeable power sources, such as Li ion batteries. The present study reports on the use of a thin film of Nickel-poly (1,8-diaminonaphthalene) on multi-walled carbon nanotubes (MWNTs) modified glassy carbon electrode as anode for electrooxidation of methanol in an alkaline medium [1,2]. The electrochemical behavior and electrocatalytic activity of the electrode were characterized by using cyclic voltammetry. The modified glassy carbon electrode consisting of a thin film of poly (1,8-diaminonaphthalene) which was formed by electropolymerization process. The effects of various parameters such as the number of electropolymerization cycles, potential scan rate, nickel concentration, number of CVs for pretreatment of the modified electrode in an alkaline solution and NaOH concentration effect on the electrooxidation of methanol have been investigated and the optimum values of each factors were suggested.

Keywords: Electrooxidation, Electropolymerization, Direct methanol fuel cells

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Simultaneous Determination of Ascorbic Acid and Dopamine With Nano Composite of Cu by Sol-Gel Method

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Abstract

Dopamine (DA) and ascorbic acid (AA) are compounds of great biomedical and neurochemical interest, playing a potential role in human metabolism. DA is one of the most significant catecholamines, plays a very important role in the functioning of the central nervous system, cardiovascular, renal and hormonal systems as well as in drug addiction and Parkinson's disease [1, 2]. Similarly, AA (vitamin C) has been used for the prevention and treatment of common cold, mental illness, infertility, cancer and AIDS [3]. The modified electrode exhibited excellent characteristics for simultaneous determination of ascorbic acid and dopamine owing to its combined features of sol-gel composite and copper catalysis. The peaks for the oxidation of ascorbic acid and dopamine were well resolved at the reduced potentials with good sensitivity. A linear response to ascorbic acid and dopamine was observed in the concentration range from 250×10^{-6} to 6.0×10^{-3} M and 500×10^{-6} to 8.0×10^{-3} M with a correlation coefficient of 0.9987 and 0.9979, respectively. The proposed modified electrode has promising features such as, ease of fabrication, good reproducibility, high stability and the easy surface renewal by simple polishing. The transfer coefficient (α) for electro catalytic oxidation of ascorbic acid and dopamine and the diffusion coefficient (D) of this substance under the experimental conditions were also investigated.

Keywords: New complex of copper, Cyclic voltammetry, Ascorbic acid, Dopamine, Sol-gel method

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Proton-Coupled Oxygen Reduction at Liquid-Liquid Interfaces Catalyzed by oxovanadium(IV)-4-methyl salophen ([VO(SB)]) and gold nanoparticles

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Abstract

Electrocatalysis at the liquid/liquid interface, or the so called interface between two immiscible electrolyte solutions (ITIES), has recently attracted a great deal of research interest for studying the oxygen reduction reaction (ORR), hydrogen evolution and carbon dioxide reduction. Such a system represents a unique example of combining the advantages of molecular catalysis and electrocatalysis, showing dependence on both catalyst properties and the Galvani potential difference between two phases. Moreover, the liquid/liquid phase boundary offers the possibility to physically separate reactants and to control the flux of either ion (e.g., proton) or electron transfer, which is particularly interesting for studying the ORR considering that it is a proton-coupled electron-transfer (PCET) reaction [1,2]. The present study demonstrates oxygen reduction at a polarized water /1,2-dichloroethane, (DCE), interface, involving aqueous protons, ferrocene, (Fc), in DCE and oxovanadium(IV)-4-methyl salophen complex catalysts adsorbed at the interface. The reaction can be driven by protons supplied by the aqueous phase and electrons provided by electron donor Fc in DCE. The presence of Gold nano-particle in the aqueous phase can increase the area of the interface and the current signals. Cyclic voltammogram measurements at the polarized interface were used for monitoring of the nanoparticles adsorption. The increased current of the reversible Fc waves that can be attributed to a corrugation of the interface accompanied by associated to the charge carried by the metallic nanoparticles [3].

Keywords: Electrocatalysis, Liquid-liquid interface, Gold nano-particle, Proton-coupled electron-transfer

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Fe₃O₄@Ag core/shell magnetic nanoparticles as a sensing platform for adsorptive anodic differential pulse voltammetric detection of olanzapine and uric acid

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Abstract

A novel, simple and selective electrochemical method was investigated for the simultaneous determination of olanzapine (OLZ) and uric acid (UA) in aqueous media and biological samples on Fe₃O₄@Ag modified carbon paste electrode (Fe₃O₄@Ag/CPE) using differential pulse voltammetry (DPV). At first, magnetic nanoparticles of Fe₃O₄ as a core were synthesized by co-precipitation of Fe(II) and Fe(III) ions in deionized water under N₂ gas protection. Then silver nanoparticles were synthesized, as shell, by adding solution of AgNO₃ (0.064 M) and NaBH₄ which was utilized as a reductant agent [1]. The synthesized Fe₃O₄@Ag core/shell MNPs were characterized by X-ray powder diffraction (XRD), transmission electron microscope (TEM) and scanning electron microscope (SEM). The Fe₃O₄@Ag/CPE displayed high effective surface area, more reactive sites and excellent electrochemical catalytic activity toward the oxidation of OLZ and UA. The effect of some experimental parameters like pH of solution, scan rate, amount of modifier, accumulation time and potential were studied [2, 3]. The peak current of differential pulse voltammograms of OLZ and UA increased linearly with their concentration in the ranges of 0.06–20 μM OLZ and 0.4–80 μM UA. The proposed sensor was successfully applied for the determination of OLZ and UA in human blood serum and plasma.

Keywords: Fe₃O₄@Ag core/shell magnetic nanoparticles, Uric Acid, Olanzapine, Differential pulse voltammetry

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A cerium (III) selective polyvinyl chloride membrane sensor based on a diaza-18-crown-6

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Abstract

A new ion-selective PVC membrane sensor for cerium (III) ions based on diaza-18-crown-6 as a good lipophilic additive for highly selective determination of Ce^{3+} ion was fabricated. The best performance electrode was obtained with a membrane composition of 50 mg polyvinyl chloride, 4mg diaza-18-crown-6, 50 mg dibutyl phthalate, 2 mg sodium tetraphenylborate. The electrode shows a linear dynamic response in the concentration range of $1 \times 10^{-1} - 1 \times 10^{-5}$ M with a Nernstian slope of 19.5 ± 0.5 mV per decade and a detection limit of 5×10^{-5} M and has a good response time of ~ 15 s and life time of at least 1 months and good reproducibility. The potentiometric response of the proposed electrode is independent of the pH the solution in the range of $6 \geq \text{pH} \geq 2$. The proposed electrode revealed excellent selectivity for Ce^{3+} over a wide variety of alkali, alkaline earth and heavy metal ions.

Keywords: PVC-membrane, Ce (III) ion-selective electrode, Diaza-18-crown-6

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Application of a nickel hydroxide nanoparticles/multi walled carbon nanotubes modified electrode as a new sensor for sensitive simultaneous determination of piroxicam and dopamine

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Abstract

The application of nanomaterials in various fields of science and technology has been extensively developed due to the unique properties of these materials [1]. Many electrodes have been modified by Ni, NiO₂, Ni(OH)₂ particles and nanoparticles on traditional electrode surfaces. In contrast to Ni nanomaterials which are unstable and easily oxidized in air and solution, hydroxide (or oxide) of these materials are relatively stable. Dopamine (DA) is an important neurotransmitter of the catecholamine group and is well characterized by its electrochemical activity [2]. Piroxicam (PRX) is a nonsteroidal anti inflammatory drug (NSAID) that also possesses analgesic and antipyretic properties. This drug has been widely used in the treatment of rheumatoid arthritis and other inflammatory disorders. It has been found that PRX produced dual effects on dopamine-related behaviors in rats [3]. Therefore it would be useful to study simultaneous determination of DA and PRX. In this work the synthesized nickel hydroxide nanoparticles (NHNPs)/ multi walled CNTs (MWCNTs) composite film-modified glassy carbon (GC) electrode was fabricated as a new modified electrode. The electro-oxidation of dopamine (DA) and piroxicam (PRX) has been investigated by application of the modified electrode using cyclic voltammetry (CV), differential pulse voltammetry (DPV) and chronoamperometry (CA) methods. The modified electrode showed excellent electrochemical responses for DA and PRX determinations. Under the optimum conditions the electrode provides a linear response versus DA and PRX concentrations in the range of 1.0 to 100.0 μM and 1.0 to 110.0 μM and with a detection limit of 0.72 and 0.53 μM respectively, using the DPV method. The interfering study of some species showed no significant interference with determination of DA and PRX could be observed at the modified electrode. The modified electrode was used for determination of DA and PRX in human urine and blood serum with satisfactory results. The simple fabrication procedure, wide linear range, low detection limit and high stability suggest that this electrode could be a reliable and attractive candidate for practical applications.

Keywords: Dopamine, Piroxicam, Multi walled carbon nanotubes, Nickel hydroxide nano-particles

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Voltammetric determination of lead (II) ion by polypyrrole modified electrode

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Abstract

Conducting polymers (CPs), owing to their mixed ionic/electronic conductivity, can be used as sensing material for different chemical sensors or biosensors [1, 2]. In this research a polypyrrole modified electrode was prepared by electropolymerization of pyrrole in a pyrrole/chromotropic acid (CTA) solution and used for the determination of lead (II) ion by preconcentration/differential pulse anodic stripping voltammetry (DPASV). In the voltammetric measurements, optimum values of significant parameters such as polymerization time (7s) and composition of the electropolymerization solution (0.05M CTA, 0.5M pyrrole), preconcentration time (300 s) and important instrumental variables were determined. Selectivity of this modified electrode was enhanced by application of specific potential program. The performance characteristics of this electrode including reproducibility, linear dynamic range and detection limit were specified. The results of this research show that this modified electrode reveals excellent selectivity and sensitivity toward pb (II) ion. This modified electrode exhibited good performance in the analysis of several types of real samples and accurate values were obtained.

Keyword: Polypyrrole, Conducting polymers, Modified electrode

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Synthesis and characterization of a graphitic nanoporous carbon for electrochemical Sensing

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Abstract

Porous carbon structures with outstanding properties, such as tunable pore channels, high surface area, uniform structure, chemical and mechanical stability has already been of considerable interest for gas separation, water purification, catalyst support, energy storage, electrodes of electrochemical double layer capacitors and fuel cells [1–3]. In the present work, a graphitic nanoporous carbon was synthesized via a nanocasting approach by using nano calcium carbonate as hard template; resorcinol/melamine/formaldehyde resin as carbon precursor and dilute HCl as template removing agent. This one-pot strategy involves the formation and then carbonization of organic polymer in the presence of nano template. The prepared material was characterized using nitrogen adsorption/desorption, small angle X-ray diffraction, raman spectroscopy, transmission electron microscopy and Fourier transform infrared techniques. The obtained results demonstrated that the obtained nanostructure carbon has excellent textural properties such as ordered pore structure, high surface area and uniform pore size. The electroanalytical properties of the synthesized material were examined by fabrication of surface renewable graphitic nanoporous carbon paste electrodes (GMC-CPE). This carbon paste electrode was prepared by mechanical mixing of graphitic mesoporous carbon (GMC) and paraffin oil. Electrochemical behavior of the nanocomposite electrode was evaluated and compared with the conventional graphite paste (GPE) and carbon nanotubes paste (CNT-PE) electrodes. The GMC-CPE provided improved electron transfer kinetics and catalytic capabilities in connection with oxidation and/or reduction of different redox systems, such as ferricyanide and some biological species, e. g. ascorbic acid (AA), uric acid (UA), dopamine (DA), paracetamol (PA) and hydrogen peroxide. The excellent experimental results implicate that the new developed paste electrode holds great promise in the design of electrochemical devices, such as sensors and biosensors.

Keywords: Nanoporous carbon, Resorcinol–melamine–formaldehyde resin, Synthesis, Characterization, Biosensor

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A novel zinc sensor based on the ion imprinted polymers and multiwalled carbon nanotubes composite modified glassy carbon electrode

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Abstract

A new selective and sensitive electrochemical sensor for detection of Zn²⁺ ions was developed by modified glassy carbon electrode (GCE) using a synthesized Zn-ion imprinted polymers (Zn-IIPs) and multi-walled carbon nanotubes (MWCNTs) film [1,2]. IIPs were prepared by the copolymerization of styrene as a functional vinyl monomer in the presence of Zinc-8-hydroxyquinoline complex. This polymer has been characterized on the basis of FTIR. In this work, it was shown that IIPs can act as selective recognition zinc ions and due to the high electrical conductivity and high surface area of MWCNTs, electron transfer is accelerated. The potential was scanned from -0.3 V to +0.3 V versus Ag/AgCl and variable affecting parameters such as pH, deposition duration time, deposition potential and stirring speed were optimized for SWASV detection. The resulting calibration curve exhibited a linear response over a concentration range of 5×10^{-12} to 15×10^{-8} mol L⁻¹ with excellent detection limit 5×10^{-12} M. The method was used successfully for Zinc detection in blood serum sample and human hair.

Keywords: Ion imprinted polymer; Multi-walled carbon nanotubes; Square-wave Anodic stripping voltammetry; Glassy carbon electrode.

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Electrochemical study of ascorbic acid and dopamine on carbon paste electrode modified by graphene/silver nanoparticles

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Abstract

Ascorbic acid (AA) and dopamine (DA) are biologically important compounds in human metabolism. So, various electrochemical techniques have been introduced for their determination. Electrochemically deposited metal nanoparticles such as silver nanoparticles (Ag NP) exhibit excellent electrocatalytic activities on the electrode surfaces. Due to the high electron transfer rate, graphene introduced as an efficient electrocatalyst for the modification of the electrode surfaces. In this study, carbon paste electrode (CPE) was first modified by reduced graphene oxide (RGO) via electrophoretic deposition. For this, the cathodic potential of -2.0 V was applied on CPE in two-electrode cell containing 10 mg/mL of MgSO_4 . Then, Ag NP were deposited electrochemically by applying two-pulsed constant potential on RGO nanosheets to prepare CPE/RGO/Ag NP modified electrode. First pulsed potential was 0.228 V for 1s to synthesize Ag NP seeds and the second pulse was 0.238 V for 5s for Ag NP growth on CPE/RGO. On the proposed modified electrode, the electrochemical behaviors of DA and AA were studied using cyclic voltammetry and differential pulse voltammetry. The results showed that Ag NP decorated on RGO nanosheets have significantly increased the electron transfer rates and decreased the overpotential of AA and DA oxidation. Also, in the determination of DA and AA, the sensitivity and detection limits were significantly improved on CPE/RGO/Ag NP. The oxidation peaks corresponding to AA and DA were occurred at 110 mV and 350 mV, respectively. The detection limits for AA and DA were obtained to be 1.0×10^{-6} M and 1.0×10^{-5} M, respectively. The linear range for AA and DA determination were obtained to be 1.0×10^{-6} M to 5.0×10^{-2} M and 1.0×10^{-6} M to 5.0×10^{-3} M, respectively. The morphology of the graphene, and the surface of graphene/Ag NP modified CPE were studied by scanning electron microscopy (SEM).

Keywords: Dopamine, Ascorbic acid, Geraphene, Cyclic voltammetry

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Study of electrochemical behavior of Diclofenac sodium using electrochemically pretreated pencil graphite electrode and application of this electrode for real sample analysis

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Abstract

With increasing regulatory pressures on the pharmaceutical industry, there is a growing need for robust sensor systems that allow rapid and reliable determinations, particularly in quality control analysis. Diclofenac [o-[(2, 6- dichloro phenyl) amino] phenyl] acetic acid, is an extensively used, non-steroidal, anti inflammatory drug with analgesic and antipyretic properties. It is used to relieve the symptoms of many diseases such as rheumatoid arthritis, osteoarthritis, non-articular rheumatism and sport injuries [1]. Several analytical methods have been developed for the quantitative determination of this drug both in pharmaceutical and biological samples [2]. These methodologies include spectrophotometry, Fluorometry, gas chromatography [2] or high performance liquid chromatography and nuclear magnetic resonance spectroscopy. Most of these methods require either sophisticated instruments or expensive reagents or involve several manipulation and derivatization steps [3]. In this work, development of a novel sensor for electrochemical determination of Diclofenac Sodium was described using electrochemically pretreated pencil graphite electrode (PPGE). Potentiostatic protocol was used for pretreatment of PGE. The electrochemical behavior of Diclofenac Sodium at the PPGE was investigated using cyclic and differential pulse voltammetric and chronoamperometry techniques. Several parameters affecting electrochemical pretreatment of PGE were investigated and optimum conditions were suggested. Accordingly, the electrochemical pretreatment of the PGE at -0.5 V/SCE for 5 min in 0.1 M phosphate buffer solution of pH 3 is proposed as the optimum pretreatment procedure. The PPGE was used for determination of low concentrations of Diclofenac Sodium, The described procedure allows the quantification of Diclofenac Sodium with adequate linearity, and reproducibility and accuracy in the concentration range 10-100 μ M with detection limit of 2 μ M. The applicability of the newly developed method was examined by analyzing several urine and human serum samples.

Keywords: Diclofenac Sodium, Electrochemistry, Pencil Graphite Electrode (PGE), Differential pulse voltammetry, Cyclic voltammetry

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Preparation of quantum dot modified carbon paste electrodes and their applications for simultaneous determination of Acetaminophen and Tryptophan

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Abstract

Tryptophan (Trp) is an essential amino acid for humans and also is a precursor of serotonin (a neurotransmitter), melatonin (a neurohormone), and niacin [1]. Acetaminophen (N-acetyl-P-aminophenol or paracetamol, AC) is an aminophenol drug that commonly used for antipyretic and analgesic illness [2]. Therefore, detecting and determining of AC and Trp in samples are of great importance for pharmaceutical and clinical analysis [2]. In this paper, a carbon paste electrode modified with CdS quantum dot was prepared for simultaneous determination of acetaminophen and tryptophan. CdS quantum dots were prepared according to the method described in the literatures [3]. The preparation of quantum dot modified carbon paste electrode was done by hand mixing of 90 Wt% graphite power and 10 Wt% of each quantum dot in an agate mortar for 45 min to get homogeneous paste. The carbon paste was incorporated into 2 mm diameter of glass tube and electrical contact was made via a copper wire. The electrode was smoothed on a piece of soft filter paper for obtaining smooth surface before the measurements. Cyclic voltammetry was used to investigate the modified electrode for the electrocatalytic oxidation of Trp and AC in aqueous solutions. Electrochemical measurements were performed with EG & G instrument. A conventional three electrode cell was used at $(25 \pm 1)^\circ\text{C}$. An Ag/AgCl/KCl (3.5 mol/L) electrode, platinum wire, and carbon paste electrode (CdS/CPE) were used as the reference, auxiliary, and working electrodes, respectively. pH was monitored with a pH/ion meter. All experiments were done in Phosphate buffer (PB) solution (0.10 M) at pH=7.0. The separation of the oxidation peak potentials for Trp-AC was 270 mV and the modified electrode shows an enhancement of electric current in one order of magnitude in comparison with the bare carbon paste electrode. The peak current is found to depend linearly on acetaminophen and tryptophan concentrations in the ranges of 2-200 and 5.0 – 300 $\mu\text{mol L}^{-1}$, respectively. The detection limits for acetaminophen and tryptophan were 1 and 4.5 $\mu\text{mol L}^{-1}$, respectively. As a result, the chemically modified electrode was successfully used for simultaneous determination of acetaminophen and Tryptophan.

Keywords: Quantum dot modified carbon paste electrode, Acetaminophen, Tryptophan, Simultaneous determination

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Using double strand DNA immobilized Au electrode for construction a novel morphine electrochemical biosensor

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Abstract

Morphine is the most abundant alkaloid found in opium. Morphine is used to treat severe pain. Short-acting formulations are taken as needed for pain. To date, many analytical methods have been developed to determine Morphine concentrations. In recent years, the development of electrochemical sensor and DNA biosensors has received great attention due to its extreme importance in many fields, such as clinical diagnostics, gene therapy, environmental monitoring, food safety and biological research [1-3]. The intercalative and electrostatic interaction of Morphine with double-stranded DNA (dsDNA) which was immobilized onto mercapto-benzaldehyde modified. Au electrode, was employed for designing a sensitive biosensor. The interaction of morphine with ds-DNA immobilized onto electrode surface has been studied by differential pulse voltammetry (DPV). Under the optimum conditions, a linear dependence of the Morphine oxidation signals was observed in the range of 0.05-500 μM with a detection limit of 0.01 μM at ds-DNA modified Au electrode. The reproducibility and applicability of the analysis of real samples were also investigated and results showed that this DNA biosensor could be used for the sensitive, rapid, simple and cost effective determination of morphine in urine and blood plasma samples without additional steps of extraction, clean-up, and derivatization. The promising performance of the developed DNA electrochemical biosensor makes this methodological study and application attractive in Morphine analysis.

Keywords: Morphine, DNA biosensor, Au electrode, Diffrential pulse voltammetry, Urine, Mercaptobenzaldehyde

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Preparation and characterization of electrochemical sensor based on molecularly imprinted polymers for detection of anticancer drug tamoxifen

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Abstract

Tamoxifen, [Z]-2-[4-(1,2-diphenyl-1-butenyl)-phenoxy]-N,N-dimethylethylamine, a nonsteroidal anti-estrogen, is the most important hormonal agent for treatment of breast cancer. In this work a molecularly imprinted polymer (MIP) polypyrrole based sensor was fabricated for the determination of tamoxifen. All measurements carried out in three electrode system; a pencil graphite electrode as the working electrode, a Pt wire as auxiliary electrode and an Ag/AgCl as reference electrode. The MIP was obtained by electrodeposition on the surface of the pencil graphite electrode (PGE) using cyclic voltammetry (CV) in the potential range between -0.60 and +0.80 V (scan rate: 100 mV/s) in aqueous solution of 0.1 M LiClO₄, 0.025 M pyrrole and 0.4 M tamoxifen. After the electropolymerization process, the embedded tamoxifen were then extracted to give a surface complementary in shape and functionality to the original template. The extraction was carried out by immersing the MIP film-coated electrode into 0.05 M phosphate buffer solution with agitation provided by a magnetic stirrer. A control electrode (non-imprinted polymer modified electrode, NIP) was prepared in every case under the same experimental conditions but without adding the tamoxifen, to check the reliability of the measurements. The performance of the imprinted and non-imprinted films was evaluated by differential pulse voltammetry (DPV), in the potential range between 0.8 and 1.1 V vs Ag/AgCl. The effect of pH, monomer and template concentrations and electropolymerization cycles on the performance of the MIP electrode was investigated and optimized. The DPV peak current showed a linear dependence on the tamoxifen concentration in the range of 0.2×10^{-6} to 1.5×10^{-6} M of tamoxifen with $R^2 = 0.9946$. The detection limit was determined as 3×10^{-7} M. The PPy electrodes have a low response time and good mechanical stability without any influence of interferents commonly existing in pharmaceutical samples. The proposed method is simple and quick.

Keywords: Tamoxifen, Polypyrrole, Electropolymerization, Molecularly imprinted polymer

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Preparation of liquid membrane potentiometric sensor based on thiodiazolo benzamide derivative

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Abstract

Chromium (III) is an essential nutrient required by the human body to promote the action of insulin in body tissues so that sugar, protein, and fat can be used by the body. One can be exposed to chromium by breathing air, drinking water and eating food containing chromium or through skin contact with chromium or chromium compounds [1, 2]. Ion selective electrodes (ISE) have an important role in modern analytical techniques. Due to simple design a high accuracy of ion selective electrodes, they offer many advantages over other analytical methods. In this study, construction, performance characteristics, and application of chromium (III) ion-selective electrode based on *N*-(2H-[1,2,4]thiadiazolo[2,3-*a*]pyridine-2-ylidene) benzamide as an ionophore or ion carrier in a poly vinyl chloride matrix is described. The best Nernstian response for the electrode was in the membrane composition percentage of PVC (28%), NB (56%), NTPYB (14%), NaTPB (2%). This electrode exhibited a fast and stable Nernst response for chromium ions over the concentration range (1×10^{-1} - 1×10^{-5}) and pH range of (3.5-8). The response time of the sensor is fast (4-5S) and the electrode can be used for 8 weeks with good reproducibility. This sensor has a good selectivity for Cr (III) over a wide variety of metal ions. The proposed electrode has been used as an indicator electrode in the potentiometric titration of Cr (III) with EDTA. In addition, the content of Cr (III) in food sample, e.g tea leaves and cacao powder has been assayed by this electrode and atomic absorption spectrophotometer (AAS). The result was in agreement.

Keywords: Chromium, Selective electrode, Potentiometric

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Investigation of cathodic electrochemiluminescence of luminol at cobalt nanoparticles decorated multi-walled carbon nanotubes

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Abstract

Electrogenerated chemiluminescence (ECL) as one of the most empowerment analytical techniques is a combination of the electrochemical and luminescent techniques and more has been applied in comparison to the conventional electrochemical methods and luminescent techniques because of its high sensitivity, wide dynamic concentration response range and low background signal [1]. Luminol (2, 3-aminophthalhydrazide) is the most popular organic luminescent species that can be oxidized on the electrode surface. The ECL signal can be produced by subsequent chemical reaction between the oxidized luminol and H_2O_2 or O_2 . Since the first report of emission of luminol at electrode surface in 1954 in aqueous alkaline solution [2], several attempts have been performed to find effective and sensitive pathways for the enhancement of ECL signal of luminol [3]. ECL of luminol could be more efficient through the modification of the electrodes surface with nano-materials. In the present study, the electrogenerated chemiluminescence behavior of luminol on a MWCNTs-nanoCo modified glassy carbon electrode (GCE/MWCNTs-nanoCo) was studied. Multi-walled carbon nanotubes (MWCNTs) decorated with cobalt nanoparticles was synthesized by a simple thermal decomposition method and then a sensitive cathodic electrochemiluminescent sensor for luminol was constructed. During the cathodic potential scanning one ECL peak was found at the GCE, GCE/MWCNTs and GCE/MWCNTs-nanoCo which related to the emission of the excited state 3-aminophthalate anions at about 425 nm. The ECL response at the GCE/MWCNTs-nanoCo intensifies relative to other surfaces because of the increase of surface area due to presence of MWCNTs and catalyzed effect of Co nanoparticles on the ECL reaction of luminol. The possible mechanism for the generation of ECL peak has been proposed. The reproducibility of the ECL intensity on GCE/MWCNTs-nanoCo was good, with relative standard deviations (RSD) of 3% for repetitive measurements ($n = 10$) of 20 μM luminol. The ECL generated at the GCE/MWCNTs-nanoCo surface developed to determination of luminol, and a detection limit of 0.11 μM was achieved. The ECL signal intensity of luminol system was linear in the range between 5 μM and 770 μM ($r = 0.9984$).

Keywords: Cathodic Electrochemiluminescence, Multi-walled carbon nanotubes, Co nanoparticles, Luminol

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Stainless steel bipolar electrode for visual sensing of analytes using electrochromics signaling

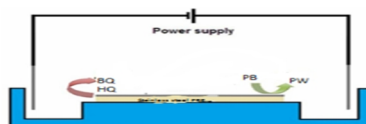
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Abstract

In this work bipolar electrochemistry was used to prepare a visual sensing platform on stainless steel substrate as bipolar electrode and usefulness of this method was proved by sensing different concentrations of hydroquinone and ferricyanide as sample analytes. In this research bipolar electrochemistry was used for the first time to signal both oxidation and reduction reactions. For this purpose Prussian blue was used as signaling agent for reduction reactions and Prussian white was used to signal oxidation reactions. Each of these electrochromic materials were electrochemically deposited on one pole of the bipolar electrode and then the modified electrode was employed for sensing application. By applying a sufficiently large driving voltage through the electrolyte solution containing bipolar electrode and appropriate electroactive species, both reduction and oxidation reactions occur simultaneously on the poles of the electrode [1, 2]; as a result, oxidation (reduction) of the analyte on the anodic (cathodic) pole causes reduction (oxidation) of Prussian blue (Prussian white) on cathodic (anodic) pole of the stainless steel bipolar electrode. These reactions cause a color change from blue to white (white to blue) on the surface of the electrode which can be visually detected [3]. Analytes with higher concentrations cause a faster color change; consequently, the extent of color change is dependent on concentration; therefore, by performing experiment in a fixed time the concentration of analyte can be obtained. Results of this study can be applied for construction of low cost and disposable sensing and biosensing devices based on bipolar electrochemistry.



Keywords: Bipolar electrochemistry, Visual sensing, Stainless steel bipolar electrode.

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Nanoelectrochemical mechanistics aspects of Acesulfame-K

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Abstract

Aspartame, Cyclamate, Caccharin and Acesulfame-K are synthetic and non-caloric sweeteners that have been widely used by the food industries to replace sugars in foods such as soft drinks, juices, jams, candies, and many others [1]. These so-called dietic foods are indicated for people that desire or need to reduce caloric intake as well as for individuals with diabetics for whom sugar restriction is recommended. Because of lack of calorie this material doesn't increase weight. Without License we can't use it because In terms of safety it must get required standards and this license is very important because of kids and pregnant women. In this essay we search for nano electrochemical mechanistic aspects of Acesulfame potassium. To the best of our knowledge, electrochemical mechanistic aspects of this compound have not previously been studied [2, 3]. The voltammograms were recorded with μ Autolab FRA2 Potentiostat-Galvanostat. A three-electrode system was used with platinum rod (2mm/ 76mm) or nano-Pt modified electrode as working, a glassy carbon electrode as the counter and an Ag/AgCl as the reference electrode (Autolab comp.). All tests were carried out under room temperature. A digital pH meter (Jenway 370) was applied for pH adjustment. In this method at first step we used Platinum rod and next nano-Pt modified electrode in two ways including cyclic voltammetry and Differential pulse voltammetry to examine Acesulfame potassium. The effect of pH, supporting electrolyte, scan rate and concentration of Acesulfame-K was evaluated. Because of voltammetric evidences, Acesulfame-K showed different mechanistic behaviors in the Platinum rod and nano-Pt modified electrode surfaces.

Keywords: *Acesulfame-K, Cyclic voltammetry, Differential pulse voltammetry, Mechanistic aspects*

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Non-enzymatic hydrogen peroxide sensor based on copper oxidenano-particles on activated pencil graphite electrode

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Abstract

Rapid and accurate determination of hydrogen peroxide (H_2O_2) is of practical importance in various fields such as food, clinical and environmental analyses. Though good selectivity and high sensitivity can be obtained with enzymatic sensors, their inevitable drawbacks such as the chemical and thermal instabilities originated from the intrinsic nature of enzymes have turned recent efforts to direct determination of hydrogen peroxide at enzyme-free electrodes. It is of great interest to develop an enzyme-less electrochemical sensor with high sensitivity, fast response time, and long-term stability. Nanomaterials have been extensively studied for non-enzymatic electrochemical sensors, because they have large surface-to-volume ratio and increased catalytic activities [1]. Copper- and copper oxide-based materials were of great interest for the electro-oxidation catalysis effect [2]. Although most of metallic Cu (0) nanomaterials are not stable enough to be used for electroanalysis because they are easily oxidized in air or aqueous solution, chemically more stable CuO nanomaterials have been studied for various applications including electrochemical H_2O_2 sensors [3]. Herein, the electrocatalytic activity of a CuO nanostructured modified electrode was investigated in terms of its application to H_2O_2 sensors. For this purpose at first step, the pencil graphite electrode chemically was activated in an ethanolic solution of HCl 0.1 M for 15 minutes then the CuO nano-structures was directly formed by electrochemical deposition of copper under electrochemical deposition condition of 20 mM $CuSO_4$ under -0.6 V vs. Ag/AgCl for 240 seconds. This modified electrode was used as an electrochemical sensor for H_2O_2 detection under the alkaline conditions. The sensitivity of the sensor electrode was $0.055 \mu A/\mu M$ with a linear response in the wide range from 8.9×10^{-8} to 7.9×10^{-2} M and a detection limit of 1.8×10^{-8} M (S/N= 3). Excellent electrocatalytic activity, large surface-to-volume ratio and efficient electron transport property of CuO electrode have enabled stable and highly sensitive performance for the non-enzymatic H_2O_2 sensor.

Keywords: Hydrogen peroxide sensor, Pencil graphite electrode, Non-enzymatic sensor, Copper oxide nanoparticles

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Easy activation of pencil graphite electrode for sensitive determination of environmental hormone bisphenol A

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Abstract

Bisphenol A [BPA, 2,2-bis(4-hydroxyphenyl)propane] is an important organic chemical as an intermediate in the manufacture of polycarbonate plastic, epoxy resin, flame retardants and other special products. These plastics are widely used for the production of baby bottles, water containers, food can lining, sports equipment, medical and dental devices, dental fillings and sealants. Upon heating BPA can leach from these containers and migrate into food and drinking water, due to the breakdown of the polymer [1]. The potential risks of BPA include reproduction and development effects, neurochemical effects and behavioral effects [2]. Therefore, an efficient monitoring system is required for detecting the levels of BPA. The attractive properties of pencil graphite electrode (PGE), which include the ability to easily renew the electrode surface, low background current, favorable anodic potential range, and cost-effective preparation have made them especially attractive for the electrochemical detection of environmental pollutants. The better intrinsic characteristics of the pencil graphite electrode are related to its composite structure and higher level of porosity compared to glassy carbon electrode [3]. In this study, a vast investigations were done to optimize the electrochemical activation of PGE. To the best of our knowledge, this is the first report of this kind for electrochemical PGE activation. The main factors for this activation are electrolyte pH, activation voltage and duration of the activations. The parameters were monitored during the optimization by checking the electrochemical behavior and peak currents of $K_3Fe(CN)_6$ as an electrochemical probe. The optimum conditions consist of an ethanoic acid 0.5 M with a Potential of 1.4 V, (vs. Ag/AgCl), for 5 minutes duration. The pretreated PGE, (PPGE), displayed at least 2 orders of magnitude more sensitive as it compared with the non-pretreated PGE (NPGE). Finally the PPGE was used for BPA determination. The calibration graph was linear in the range of 0.4–40 μM with a detection limit of 0.1 μM .

Keywords: Bisphenol A, Pencil graphite electrode, Modified electrode

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Fabrication of microband electrode from gold compact disk using drilling method and simulation of its voltammetric behavior using COMSOL Multiphysics software

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Abstract

Microelectrodes are attractive tools in electro-analysis because of their high level of sensitivity. They are also invaluable devices for electrochemical mechanistic studies involving ultrafast kinetics [1]. The basic differences observed between the voltammetric response behavior of macro- and microelectrodes can be explained by the specific and radial diffusion geometry, respectively [2]. The commonly used configurations include spherical, hemispherical, disk-shaped tubular and linear microelectrodes. The methods of manufacturing microelectrodes including laser micromachining, sonoelectrochemistry and photolithography techniques, none were considered to offer the simplicity of the drilling method. Tubular microband electrodes were produced by drilling a hole perpendicularly through the thin gold film that has been between two dielectric layers. Gold CD-R is a suitable raw material which has exactly this property and tubular microband electrodes can be readily fabricated from gold CD-Rs using a simple and cheap drilling technique. As a powerful electrochemical method, cyclic voltammetry is often the first choice to characterize a new electrochemical system [3]. Characterization of the microband electrodes using potassium ferro/ferri cyanide with cyclic voltammetry, showed that steady state behavior was displayed which is indicative of microelectrode behavior at low potential scan rates. At scan rates faster than 50mV/s the voltammograms showed a peak due to planar diffusion governing mass transport to the microelectrodes. COMSOL Multiphysics is an excellent and flexible tool for simulation of complex electrochemical systems which can easily help to explore of real systems. The voltammetric response of microelectrode was simulated by using the commercial finite element software COMSOL Multiphysics version 4.0 and simulated result was compared with experimental cyclic voltammograms.

Keywords: Microband electrode, COMSOL multiphysics, Drilling method

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Examine the role of alkyl amide derivatives as inhibitors of γ -Secretase in Alzheimer's disease

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Abstract

Alzheimer's disease is a progressive neurological disorder which is associated with memory impairment and cognitive impairment. With the increasing figures of people with Alzheimer's disease and the emergence of the neurological disorders, new studies are needed to deal with Alzheimer's. Alkylamides gamma secretase act as a deterrent. For attachment methods, quantitative structure-activity (QSAR) their usefulness in the development of drugs proved. In these studies, a mathematical relationship between structure and activity or a combination of features to be found. With the help of a mathematical relationship between the parameter scan can be used for other derivatives of these compounds, the synthesis of derivatives that are not even predicted. This relationship also provides the mathematical possibility that compounds with the desired activity or property advertised. So, alkyl amide derivatives such as "N-Benzyl-N-phenyl sulfon amide" can act as an inhibitor of gamma secretase.

Keywords: Alkyl amide, Alzheimer, Descriptors, γ -Secretase

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QSAR methods for the prediction and design of new anti-Alzheimer drugs using alkyl amides derivatives

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Abstract

Many studies have shown that markers of QSAR models based on descriptions along with information derived from structural bio informatic tools can be very useful for basic research and develop insights into drug to provide it. Necessitates the use of QSAR in the study is important because the QSAR in drug design and as the provider of the dependence between structure and function and understanding of the impact of the activities offered the prediction of drug activity. Such as alkyl amides derivatives show activity against Alzheimer's. The QSAR methods for the prediction and design of new anti-Alzheimer's drugs on the alkyl amide derivatives was used. This method can be time consuming and without the high costs and no practical tests before proceeding to the Synthesis of alkyl amides is possible to measure anti Alzheimer's. This method can also review all of the hypothetical compounds of this class of drug compounds and measure the activity of these compounds against Alzheimer's disease, active compounds against Alzheimer's disease who are very determined, and then the synthesis of these compounds.

Keywords: Alkyl amide, Anti Alzheimer, Descriptors, QSAR methods

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Fabrication of all solid state ion selective electrode based on electropolymerization of aminophenols.

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Abstract

Aminophenols are interesting materials from electrochemical point of view due to containing two oxidizable functional groups (-NH₂, -OH) [1]. Therefore these compounds show electrochemical behavior of both aniline and phenol compounds [2]. It has been reported that electrochemical behavior of aminophenols is highly dependent on the position of amin & hydroxyl groups in the aromatic ring. Para aminophenol (PAP) could be oxidized electrochemically in neutral or weakly alkaline media into complex oligomeric species which indicated strongly tendency toward Ni²⁺ ions for complexation. In this research PAP was oxidized electrochemically by control potential of electrode (three electrode system). Sodium acetate was used as electrolyte cell solution. The electrode product was characterized using different techniques such as CV, UV-Vis, FT-IR, ¹H-NMR & DCS. It was suggested that the product is a polymeric material with OH & NH₂ groups in ortho positions. A mechanism for electropolymerization of PAP into poly PAP has been suggested too. It was found that the polymeric material prepared via electrochemical rout can be used as a solid state ion selective electrode for determination of Ni²⁺ ions. In order to determine the optimized preparation conditions of Ni sensor electrode, various parameter affecting electrode response were investigated. The electrode showed linear response to Ni²⁺ ions in concentration range of 10⁻² – 10⁻⁸ M. The electrode slope was 27 mV per decade and limit of detection was 5 × 10⁻⁸ M.

Keywords: Poly para aminophenol, Electropolymerization, Solid state ion selective electrode, Ni²⁺.

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Bicyclic aziridine-based electrochemical tramadol nanosensor

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Abstract

Tramadol (TRA) is a centrally acting analgesic that was first introduced in Germany in 1977. Today, it has become the most prescribed opioid world wide. It is generally said to be devoid of many of the serious adverse effects of traditional opioid receptor agonists such as the risk for respiratory depression and drug dependence. Tramadol works in two ways to relieve pain. It works on the brain in a similar way to codeine and morphine. It also affects some other chemicals in the brain called noradrenaline and serotonin. These have many functions, some of which include easing pain [1-2]. A variety of quantitative analytical methods have been reported for the determination of TRA including HPLC, LC-MS and GC-MS. Electrochemical analytic technique is an attractive method due to simplicity, rapidity, low expense, high sensitivity and possibility of miniaturization. Construction of electrochemical sensors continues to be an area of great interest [3]. The electrochemical behavior of TRA in aqueous media has been examined by cyclic and differential pulse voltammetry. The voltammograms were recorded with μ Autolab FRA2 potentiostat-galvanostat. A three-electrode system was used with a bicycloaziridin-nanographene modified electrode as working, a platinum rod as the counter electrode and an Ag/AgCl as the reference electrode. All tests were carried out under room temperature and N₂ atmosphere. The pH strongly affects the peak potential of TRA. The best analytical response was obtained at pH 9.0. The cathodic peak currents were proportional to TRA concentrations. The effect of potential scan rate on the peak potential and peak current of TRA was investigated. The correlation of the peak currents against $v^{1/2}$ (v is the scan rate) is linear, which is very similar to a diffusion-controlled process. Proposed method applied to analysis of commercial tablets, successfully.

Keywords: Tramadol, Bicyclic aziridine, Differential pulse voltammetry, Nanographene

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Direct and indirect electrochemical activation of CO₂ by a copper Schiff base complex as a bi-functional electrocatalyst

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Abstract

The electrochemical behaviours of two copper Schiff base complexes [N,N'-bis(2-R-phenyl- salicylidenaminato) Cu(II)] complex, (CuL₂-Me for R = Me, and CuL₂-Cl for R= Cl) and [N,N'-bis(salicylaldimine) propylenediimino Cu(II)] (CuL') and the stability of their reduced forms in a N,N-dimethylformamide (DMF) solution were studied using cyclic voltammetry. The results exhibited that the reduced form of CuL' was stable in experimental conditions. The electrocatalytic activities of the CuL' for CO₂ reduction as well as O₂ reduction in the presence of CO₂ show that CuL' complex activates CO₂ in two ways: direct electrocatalytic reduction of CO₂ to CO₂^{•-} and indirect CO₂ activation by the product of O₂ electrocatalytic reduction to yield peroxydicarbonate anions, C₂O₆²⁻ [1]. Both electrocatalytic reduction of CO₂ and that of O₂ are ligand-based. However, the complex promotes the electrocatalytic reduction of CO₂ better as compared to free ligand.

Keywords: Carbon dioxide, Oxygen, Bifunctional electrocatalyst, Copper (II) complex

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The effect of electronic structure on electrocatalytic behaviors of cobalt Schiff base complexes: electrosynthesis of 2-phenylacetic acid using carbon dioxide

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Abstract

Electrochemical fixation of carbon dioxide was used as an efficient method to synthesize 2-phenylacetic acid [1]. The electrochemical carboxylation of benzyl halides by two cobalt Schiff base complexes of [N, N'-bis(salicylaldehyde)-1,2-phenylenediimino cobalt(II)], [Co(II) salophen], and [N, N'-bis(benzoylacetone) ethylenediimino cobalt(II)], [Co(II) bzaen], in N,N-Dimethylformamide and in the presence of carbon dioxide was investigated using cyclic voltammetry method [2]. The electrocatalytic reduction mechanism of benzyl halides is dependent on the electronic structure of the complexes. Moreover, the presence of CO₂ improves the catalytic efficiency and stability of the complexes. Finally, controlled potential coulometry (CPC) and constant current coulometry (CCC) were carried out in the absence and the presence of CO₂. The electrosynthesis yields of 2-phenylacetic acid from benzyl bromide are obtained by 50% and 55% using CPC and 60% and 70% using CCC under mild conditions using [Co(II) salophen] and [Co(II) bzaen] complexes respectively. DFT calculations along with Mulliken/NBO studies have been carried out in order to support the experimental results [3].

Keywords: Benzyl bromide, 2-Phenylacetic acid, CO₂ fixation, Cobalt complexes, DFT

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Green synthesis of palladium nanoparticles using honey and its application for electrochemical detection of Hg^{2+} in aqueous solution using Cu electrode

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Abstract

Mercury toxicity in environmental pollution is a major concern because of increased usage of fossil fuels and agricultural products, both of which contain mercury which can cause long-term damage to biological systems [1]. Metal nanoparticles have shown superior or advantageous properties for a wide range of technological applications, and they provide three important functions for electroanalysis: improved mass transport, high effective surface area, and catalytic properties [2]. In this work, a rapid and simple biological fabrication of Pd nanoparticles is reported. Spherical Pd nanoparticles with size of 30 nm are synthesized using the honey samples as a reducing material. The morphology of nanoparticles are characterized by EDX, scanning electron microscopy (SEM) and UV-vis techniques. The obtained Pd nanoparticles were coated on copper electrode. The ability of modified copper electrode was studied for the determination of mercury (II) ions in aqueous solution using various electrochemical modes such as cyclic voltammetry and differential pulse voltammetry. The proposed voltammetric method showed a reliable and sensitive method for mercury ions in the concentration range of $2 \times 10^{-7} - 4.7 \times 10^{-5}$ M with a limit of detection 2×10^{-8} M. The proposed method has been successfully applied for the determination of Hg^{2+} in various water samples.

Keywords: Mercury, Cu electrode, Palladium nanoparticles, Honey

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Simultaneous electrochemical determination of tyrosine and tryptophan by mesoporous silica nanoparticles modified carbon paste electrode using H-point standard addition method

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Abstract

Mesoporous silica nanoparticle, a typical inorganic nanomaterial, has been considered as interesting materials for electrode modification due to its numerous convenient properties including good biocompatibility, nontoxicity, thermal stability and excellent electrical properties [1-3]. A simple, selective and sensitive sensor based on mesoporous silica nanoparticales modified carbon paste electrode (MSNs/CPE) was introduced for electrochemical determination of tyrosine (Tyr) and tryptophan (Trp). Compared with the unmodified electrode and commercial SiO₂ modified electrode (SiO₂/CPE), the peak current significantly improved for both species. Under optimized conditions a linear relationship between the peak currents and concentration was obtained in the range of 5.0×10^{-8} to 4.0×10^{-4} M and 5.0×10^{-7} to 6.0×10^{-4} M for Trp and Tyr, respectively. In order to simultaneously determine Trp and Tyr, H-point standard addition method was applied for resolving the overlapping in differential pulse voltammetric peaks corresponding to the oxidation of Trp and Tyr.

Keywords: Mesoporous silica nanoparticle, H-point standard addition method, Tyrosine, Tryptophan

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Fabrication of a novel casein phosphopeptides/multi-walled carbon nanotubes/micro hybrid resin as mixed matrix membrane-junction reference electrode

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Abstract

Reference electrodes are generally adopted in the electrochemical systems for various purposes such as estimation of the electroactive behavior as well as the redox strength of redox half reactions, during measuring the electrode potential of an indicator electrode in potentiometry, in the controlled potential electrolysis, and also during applying a fixed electrical potential to the working electrode in voltammetry [ref]. Stable composition, fixed electrode potential, ideally non-polarizability, simplicity, low cost, and ease of use are considered as the main characteristics of an ideal reference electrode. In this study, a novel reference electrode was fabricated via modification of a Pt electrode with solid-state mixed matrix membrane generated via mixing casein phosphopeptides as Ca^{2+} and Cl^- ionophores [1, 2], multi-walled carbon nanotubes (MWCNTs) as an electrical support (inorganic phase) and micro hybrid resin as binder (organic phase) with the weight ratios of 25:25:50 (wt.%), respectively. The selectivity of casein towards Ca^{2+} during formation of casein phosphopeptides- Ca^{2+} nanocomplex (Average diameter: ~250 nm) was evidenced using UV-Vis spectrometry. Potentiometric technique revealed the strong adsorptivity of the nanocomposite to the chloride ion. Longmuir behavior was also estimated for the fabricated nanocomposite during the linear adsorption of Cl^- . The fabricated reference electrode showed adequate stability in various ionic strengths generated in the presence of ions such as Na^+ , K^+ , Mg^{2+} , Cl^- , I^- , I_3^- , SO_4^{2-} , PO_4^{3-} , ClO_4^- , etc. The non-polarization of the fabricated reference electrode was confirmed during passing at least 10.0 mA electrical current thorough the electrode. The fabricated half-cell as reference electrode with notation of "Pt | KCl (satd.) | Ca^{2+} (1.0 μM) | nanocomposite membrane" had a fixed reduction potential equal to -0.070 ± 0.002 V vs. standard hydrogen electrode. The electrical stability of the electrode was originated from irreversible adsorption behavior of the nanocomposite to the Cl^- as well as from the stability of casein phosphopeptides- Ca^{2+} nanocomplex during the conditioning steps. The reliability of fabricated reference electrode was evaluated during testing its stability in a three-electrode system including Pt and glassy carbon as working and counter electrodes during evaluating the nerstian behavior of cyclic voltammetry of 1.0 mM $\text{Fe}(\text{CN})_6^{4-/3-}$ solution with anodic and cathode peak potential difference (ΔV) of 63.0 mV. The lifetime of the fabricated reference electrode was estimated to at least 12 months.

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Differential Pulse Voltammetric Atropin Nanosensor Utilizing β Zeolite

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Abstract

Atropin (AT) has wide medical applications, e.g. for dilating the pupils in the ophthalmic operations, as an antispasmodic and as an antidote for poisoning of opium, eserine and muscarine. Till now, the commonly employed techniques for the determination of the drug in bulk form, pharmaceutical formulation, and biological fluids are based on HPLC, LC/MS, spectroscopy, and microbiological assays [1]. Since these techniques have expensive instrumentation and running costs, the use of simpler, faster, and cheaper, yet sensitive, electrochemical techniques can be interesting alternatives, especially those based on electroanalytical techniques. Modification of electrodes with suitable materials facilitates the electrochemistry of the redox biological compounds, which generally results in increased selectivity and sensitivity of the determinations. Among these methods, chemically modified electrodes (CMEs) have been applied for the determination of drugs in real samples. Zeolite-modified electrodes (ZMEs), the important type of the CMEs, have been largely developed during the past two decades because they enable to combine the intrinsic properties of the zeolites with electron transfer reactions. Zeolite-modified carbon paste electrodes (ZMCPEs) have been largely and successfully applied for electroanalytical purposes [2]. In these work, the nano Cesium- β Zeolite modified electrode was prepared by ion exchange process for rapid determination of Atropin. The effect of the composition of Agar-based electrode on its voltammograms was evaluated in acidic solution with 5.9×10^{-3} M AT. It was found that addition of nano Cesium- β Zeolite to the Agar would generate the peak current of AT because of its catalytic effect on redox process. The pH affects the peak potential of AT. The best voltammogram was obtained at pH 2.0. The linear dynamic range was $1.0 \times 10^{-3} - 1.0 \times 10^{-2}$ M under the optimized experimental conditions. The detection limit was 6.2×10^{-4} M. The effect of potential scan rate on the voltammetric parameters of AT was investigated. The proposed nanosensor was applied to the determination of AT in commercial samples, successfully.

Keywords: Atropin, Nanographene, Organometalic Complex, Voltammetry

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Acorus calamus as eco-friendly corrosion inhibitors for copper in HCl media

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Abstract

Use of inhibitors is one of the most practical methods for protection against corrosion especially in acid solutions to prevent unexpected metal dissolution and acid consumption [1]. Different organic and inorganic compounds have been studied as inhibitors to protect metals from corrosion attack. These natural organic compounds are either synthesized or extracted from aromatic herbs, spices and medicinal plants. Plant extracts are viewed as an incredibly rich sources of naturally synthesized chemical compounds that can be extracted by simple procedures with low cost and are biodegradable in nature. The use of these natural products such as extracted compounds from leaves or seeds as corrosion inhibitors have been widely reported by several authors [2]. The inhibition effect of Acorus calamus crude extract was investigated for copper corrosion in 0.5M HCl medium. Potentiodynamic polarization and electrochemical impedance were employed for this study. Results showed that crude extracts reduced the corrosion rate significantly at all concentrations. Polarization measurements indicated that these green inhibitors acted through mixed type inhibition. The inhibition efficiency of copper in 0.5 M HCl increases by increasing the concentration of Acorus calamus. Inhibition efficiency up to 95% has been achieved for 1.6 g/l Acorus calamus at 25°C. Acorus calamus is effective in reducing corrosion of copper in the temperature range of 25-65°C. Polarization studies showed that Acorus calamus acts as a mixed-type inhibitor without modifying the mechanism of hydrogen evolution.

Keyword: Inhibitor, Copper, Acidic media

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Electrochemically imprinted molecular recognition sites on carboxyl functionalized MWCNTs surface for detection of hydroquinone

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Abstract

Hydroquinone (HQ) is used as a depigmenting agent and widely used in cosmetics, medicines, environment, and human diet. It can be metabolized to benzoquinones which are potentially haematotoxic, genotoxic and carcinogenic compounds [1]. In this work an electrochemical sensor combining a molecular imprinted technique and an electropolymerization method was developed. A molecular imprinted polymer (MIP) film was fabricated by electro polymerizing pyrrole using cyclic voltammetry in the presence of HQ after electrodepositing carboxyl functionalized multi-walled carbon nanotubes (fMWCNTs) on to a glassy carbon electrode surface (MIP-fMWCNTs/GCE). The HQ template molecule was successfully trapped in the pyrrole film where it created artificial recognition sites. After extraction of the template, the pyrrole film acted as a molecularly imprinted polymer for the specific and selective recognition of HQ whereas the non-imprinted polymer (NIP) film did not exhibit any oxidation peak which demonstrates that the imprinted pyrrole film are specific towards HQ [2]. The effects of pH, the monomer concentration, the number of cycles for the electropolymerization and the scan rate for the sensor preparation were optimized. The MIP-fMWCNTs/GCE displayed an excellent recognition capacity toward HQ compared with other structurally similar molecules. This sensor was used successfully for HQ determination in HQ topical cream and coffee samples.

Keywords: Hydroquinone, Molecularly imprinted polymer, MWCNTs, Electrochemical polymerization

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Electrochemiluminescence Genotyping of Single Nucleotide Polymorphisms using Monobase/luminol functionalized Platinum Nanoparticle

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Abstract

Analysis of DNA sequences shows considerable variability between individuals, and most of the variations in the human genome results from single nucleotide polymorphisms (SNPs) [1]. A major challenge in the area of DNA detection is the development of simpler and inexpensive assays. Electrochemiluminescence, or electrogenerated chemiluminescence (ECL), can be described as chemiluminescence (CL) produced directly or indirectly as a result of electrochemical reaction or a reaction sequence [2]. Recently, our group has developed an electrochemical assay for the genotyping of SNPs using monobase-conjugated modified Au and Ag NPs [3]. Herein, a sensitive, selective, simple and rapid approach based on ECL, for the genotyping of SNPs (A-C and G-T mismatches) is introduced. To monitor the genotyping of different SNP targets, an ultrasensitive ECL sensor was designed which employed Monobase/luminol functionalized Pt-NPs (M/luminol/Pt-NPs) that are complementary with mismatch bases in the targets. To this end, the mismatch targets are hybridized by complementary M/luminol/Pt-NP labels in the presence of DNA polymerase I (Klenow fragment). The polymerase enzyme, can induce coupling of the M/luminol/Pt-NPs to the mutant site of duplex DNA. Upon hybridization, when a sufficiently high electric field is applied the electrode, the ECL reaction resulting from the label catalyzes oxidation of luminol and hydrogen peroxide. It provided a platform for quantitative detection using photo multiplier tube (PMT) and was further used to detect the level of SNPs. This biosensor can genotype thermodynamically stable SNP (G-T mismatches) in the range of 1×10^{-4} - 1×10^{-9} M with detection limit of 0.5 nM and with quite reliable reproducibility (%RSD=5.1) and stability (RSD = 4.2%).

Keywords: Electrochemiluminescence (ECL), luminol, Platinum Nanoparticles, Genotyping, Single Nucleotide Polymorphisms

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Determination of mean activity coefficients of MgCl_2 in (MgCl_2 + lactose + water) system by potentiometric method at 298.15 K

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Abstract

Thermodynamic properties of electrolytes in saccharide solutions are important not only for studying the nature of interactions of saccharides with ions, but also for providing significant information on the stereo-structure of saccharide molecules and the action mechanism of their biological activity. To measure the thermodynamic properties of electrolytes in mixed solvent, the most common methods are the isopiestic vapor pressure [1] and potentiometric techniques [2]. In the previous decades, the series of ion-interaction models for electrolyte solution have been proposed to predict the activity coefficient of each solute and osmotic coefficient of aqueous systems. Among them, the Pitzer ion-interaction model is one of the most famous and useful models. Pitzer equations, with much accuracy, correlate the thermodynamic properties of mixed aqueous electrolyte systems. The Pitzer model extended the Debye–Hückel method with a virial expansion to account for the ionic strength dependence of the binary repulsive short-range forces, and higher order interactions [3]. In this article, the mean activity coefficient of MgCl_2 in lactose-water mixed solvent system were measured by using the potentiometric technique at $T=298.15$ K. The measurements were carried out on the galvanic cell without liquid junction of the type: $\text{Ag}/\text{AgCl}|\text{MgCl}_2(\text{m}), \text{Lactose}(\text{w}\%), \text{H}_2\text{O}(100-\text{w}\%) | \text{Mg-ISE}$ over total ionic-strengths from 0.001 to $6.00 \text{ mol} \cdot \text{kg}^{-1}$, the mass fractions of lactose in solution were 0.025, 0.050, 0.075, 0.100 and 0.125. The cell potentials were analyzed by using the Pitzer equation and the Pitzer ion-interaction parameters (β^0 , β^1 and C) for the series system were estimated by fitting experimental results with the Pitzer model. Then these parameters were used to calculate of osmotic coefficient of water and Gibbs free energy of solution. The results showed that the Pitzer model could satisfactorily describe the investigated system.

Keywords: Mean activity coefficient, Gibbs free energy, Interaction parameter, MgCl_2 , Lactose

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Chemical sensor for copper (II) determination using disyclo hexano18-crown-6 as ionophore in poly(vinyl chloride) matrix

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Abstract

A polyvinyl chloride (PVC) based membrane sensor for copper ions was prepared by employing *disyclo hexano 18-crown-6* an ionophore, sodium tetraphenyl borate as anion excluder and bis (2-ethyl(hexyl))phthalate as plasticizer. The plasticized membrane sensor exhibits a Nernstian response for Cu (II) ions over a wide concentration range (1.0×10^{-6} to 1.0×10^{-2} M) with a limit of detection as low as 8.91×10^{-7} M. It has a fast response time (<15 s) and can be used for 1 months. The sensor revealed a very good selectivity with respect to common alkali, alkaline earth and heavy metal ions. The response of the proposed sensor is independent of pH between 3.0 and 8.0. It was used as an indicator electrode in potentiometric titration of fluoride, carbonate and oxalate anions and determination of copper in simulated mixtures.

Keywords: Copper selective electrode; Polyvinyl chloride; Membrane electrodes; Schiff base; Potentiometric sensors

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Fabrication of Immobilized Liquid Membrane Electrode and Coated Graphite Electrode

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Abstract

In this research, two new lead ion selective membranepotentiometric sensors based on Benzo-18-Crown-6 as neutral carrier in poly vinyl chloride[1] (PVC) matrix was fabricated by two immobilized liquid membrane and coated graphite forms. The electrodes shows a linear dynamic response in the concentration range of 1.0×10^{-3} - 1.0×10^{-7} M with a Nernstian slope of 28.2 ± 0.5 and 30.0 ± 0.5 mV per decade and a detection limit of 1.6×10^{-7} M for immobilized liquid Membrane electrode and 1.1×10^{-7} M for graphite coated electrode. The electrodes show a good reproducibility and have a good response time of 15 and 5 s and a life time of 9 and 15 days for immobilized liquid Membrane and graphite coated Electrode respectively. The best performance was obtained with a membrane composition of 26.6% PVC, 68% DOP, 4.1% Benzo 18 crown-6 and 0.91% sodium tetraphenylborate for liquid membrane and a membrane composition of 27% PVC, 70% DOP and 2.7% benzo 18-crown-6 and 0.1% sodium tetraphenylborate for graphite coated electrode. The potentiometric response of the proposed electrodes is independent of the pH of solution in the range of $3 > \text{pH} > 12$ and $4 > \text{pH} > 12$ for liquid Membrane and coated electrode respectively. The both of Electrode shows good selectivity (based on match potential method [2, 3]) towards lead against most of the transition and common alkali, alkaline earth metal.

Keywords: Poly vinyl chloride, Potentiometric sensor, Benzo-18-Crown-6, Ion selective electrode, Liquid membrane, Graphite coated, Lead ion

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Organometallic Complex-Nanographene based Voltammetric Methadone-Sensor

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Abstract

Methadone hydrochloride (MHC) is a powerful narcotic analgesic resembling morphine in its action and use. It is a potent analgesic sedative and treatment of heroin addiction and is often encountered in forensic specimens. Because methadone kills sensitive leukemia cells and breaks chemo resistance and apoptosis resistance in leukemia's, it has been proposed as a promising agent in leukemia therapy, especially when conventional therapies are not effective. Due to its use in clinical trials several analytical methods have been developed such as: Gas Chromatography, High Performance Liquid Chromatography, Potentiometry and voltammetry [1-3]. Simplicity, short analysis time and low cost are the main advantages of voltammetric sensor. For the first time, Organometallic complex-nanographene based voltammetric Methadone-sensor was developed. The voltammograms were recorded with μ Autolab FRA2 Potentiostat-Galvanostat. A three-electrode system was used with a modified electrode as working, a platinum rod as the counter and an Ag/AgCl as the reference electrode. All tests were carried out under room temperature and N₂ atmosphere. The pH strongly affects the peak potential of MHC. The best analytical response was obtained at pH 2.21. The cathodic peak currents were proportional to MHC concentrations in the range of 1.15 mM– 0.74 nM under the optimized experimental conditions. The detection limit was 0.15 nM. The effect of potential scan rate on the peak potential and peak current of MHC was investigated. The correlation of the peak currents against $v^{1/2}$ (v is the scan rate) is linear, which is very similar to a diffusion-controlled process. Proposed method applied to analysis of commercial tablets, successfully.

Keywords: Methadone, Nanographene, Organometallic complex, Voltammetry

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Adsorptive stripping voltammetric detection of thorium on the MWCNT modified glassy carbon electrode

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Abstract

Glassy Carbon electrodes (GCE) modified with multiwalled carbon nanotubes present an interesting alternative in the determination of thorium using square wave adsorptive stripping voltammetry. In comparison with the bare GCE, the modifications of MWCNTs demonstrated more sensitive responses. Voltammograms were performed with a three electrode system consisting of a modified GCE as the working electrode, an Ag/AgCl reference electrode, a platinum wire auxiliary electrode. The pH adjusted at 5.0 and a scan rate of 5 mV/s, and also adsorptive voltammetric peak corresponding to the adsorption of oxine in the complex is occurred at -0.2V (vs. Ag/AgCl). The peak current of the complex was linearly dependent upon the concentration of Th (IV) over the range of 1.0×10^{-8} - 3.0×10^{-7} M, the limit of detection (LOD) 0.01 nM, accumulation time 200s without the need of preconcentration. The method has been successfully applied to the determination of trace Th (IV) in different water samples.

Keywords: Thorium, Oxine complex, Glassy carbon electrode, Multiwalled carbon nanotube, Stripping voltammetry

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Adsorptive stripping voltammetric detection of thorium on the MWCNT modified screen printed electrode

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Abstract

Screen-printed electrodes (SPEs) modified with multiwalled carbon nanotubes (MWCNTs) present an interesting alternative in the determination of thorium (IV). This method can be achieved by square wave adsorptive stripping voltammetry of the thorium complex with oxine at a screen printed electrode (SPE). In comparison with the bare SPE, the modifications of MWCNTs demonstrated more sensitive responses. The pH adjusted at 5.0, and also adsorptive voltammetric peak corresponding to the adsorption of oxine in the complex is occurred at -0.3V without the need of preconcentration. The electrode displayed excellent linear behaviour in the concentration range examined (0.5×10^{-9} - 22×10^{-8} M) with a limit of detection of 0.5 nM. The method has been successfully applied to the determination of trace Th (IV) in different water samples.

Keyword: Thorium; Oxine complex; Screen Printed electrode; Multiwalled carbon nanotubes; Stripping voltammetry.

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Fabrication of a potentiometric membrane Coated electrode sensitive to cobalt (Co^{2+}) cation based on N-phenylaza-15-crown-5 as neutral carrier in PVC matrix

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Abstract

A new PVC membrane coated graphite electrode for cobalt ion based on N-phenylaza-15-crown-5 as ionophore was prepared. The electrode shows a near Nernstian response of 28.3 mv decade⁻¹ over a wide activity range of 1.0×10^{-5} - 1.0×10^{-1} M with a limit of detection 3.9×10^{-6} M. The proposed electrode is suitable for use in aqueous solution in the pH range of 2.0–7.0. It has a fast response time of 10 s and can be used for at least 20 day without any considerable divergence in potential. This electrode has been used as an indicator electrode in potentiometric titration of cobalt (Co^{2+}) ion and in direct determination of it in spiked water samples. The increasing use of ion sensors in the fields of environmental, agricultural and medicinal analysis is putting more and more pressure on analytical chemists to develop new sensors for the fast, accurate, reproducible and selective determination of various species. During the past few decades, many intensive studies on the design and synthesis of highly selective ionophores as sensory molecules for ion-selective electrodes have been reported. The need of selective determination of heavy metal ions has increased immensely during the last few decades due to the growing environmental problems. Among heavy metals, the pollution caused by cobalt is of considerable concern [1]. The general procedure to prepare the PVC membrane was to mix thoroughly 34.2 Wt% of powdered PVC, 59.8 Wt% of plasticizer dibutyl phthalate and 2.6 Wt% of additive sodium tetraphenylborate in 3 ml of THF. To this mixture was added 3.4 Wt% of ionophore N-phenylaza-15-crown-5 as and the solution was mixed well. The solvent was evaporated slowly until an oily concentrated mixture was obtained. Then the graphite was coated by dipping into the membrane solution in THF. A membrane was formed on the graphite surface, which was allowed to dry overnight. On the basis of the above mentioned results, the Co (II) ion-selective electrode which was designed in this work has many advantages including: easy preparation, low cost, wide dynamic range, low detection limit, suitable pH range and good reproducibility.

Keywords: Cobalt, N-phenylaza-15-crown-5, PVC matrix, Membrane Coated electrode potentiometry

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Voltammetric sensor for glutathione determination based on electrochemical oxidation of 4, 4'-biphenol as a mediator onto glassy carbon electrode

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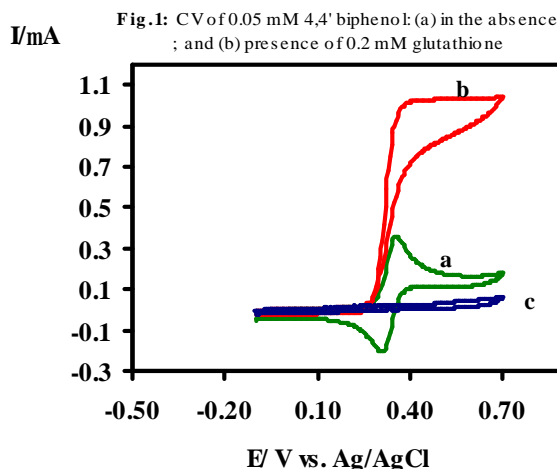
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Abstract

The electro-catalytic oxidation of glutathione (GSH) has been studied in presence 4,4'-biphenol. Cyclic voltammetry (CV), double potential step chronoamperometry and differential pulse voltammetry (DPV) techniques were used to investigate the suitability of incorporation of 4,4'-biphenol / 4,4'-diphenylquinone redox as a mediator for the electro-catalytic oxidation of GSH in buffered aqueous solution. Therefore we described redox couple (4,4'-biphenol and 4,4'-diphenylquinone) can be used as a suitable mediator for determination of glutathione concentration based on the catalytic current (curve b in figure 1). The effect of pH, scan rate and analyte concentration has been examined. Under the optimum conditions, the peak current was linear to the concentration of glutathione in the range 5.0×10^{-6} M - 2.0×10^{-4} M and 1.0×10^{-6} M - 1.0×10^{-4} M in the cyclic voltammetry and differential pulse voltammetry respectively. The detection limits were determined as 3.0×10^{-6} M and 0.6×10^{-6} M by CV and DPV methods, respectively. Finally, the electrocatalytic oxidation of glutathione by 4,4'-diphenylquinone can be employed as a new method for the voltammetric determination of glutathione in real samples such as human blood.



Keywords: Glutathione determination, Catalytic current, 4, 4'-biphenol

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preparation of liquid membrane potentiometric sensore based on imine obtained from triazinamine and isatin

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Abstract

Zn²⁺ ion is an essential metal cation of exceptional biological and public health importance, where it has multiple roles in both extra- and intra-cellular functions. For example Zn²⁺ is known to be indispensable to growth and development and transmission of the genetic message [1]. Ion selective electrodes (ISE) have an important role in modern analytical techniques. Due to simple design and high accuracy of ion selective electrodes, they offer many advantages over other analytical methods [2,3]. In this study, construction, performance characteristics, and application of zinc ion-selective electrode based on imine obtained from triazinamine and isatin ionophore or ion carrier in a poly vinyl chloride matrix is described. The best Nernstian response for the electrode was in the membrane composition percentage of PVC (28%), NB (56%), (14%), NaTPB (2%). This electrode exhibited a fast and stable Nernst response for zinc ions over the concentration range (1×10^{-1} - 1×10^{-5}) and pH range of (4/5-8). The response time of the sensor is fast (4-5S) and the electrode can be used for 10 weeks with good reproducibility. This sensor has a good selectivity for Zn²⁺ over a wide variety of metal ions. The proposed electrode has been used as an indicator electrode in the potentiometric titration of Zn²⁺ with EDTA. In addition, the content of Zn²⁺ in food sample, e.g. bingo leaves and rice powder has been assayed by this electrode and atomic absorption spectrophotometer (AAS). The result was in agreement.

Keywords: Zinc, Selective electrode, Potentiometric

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Fabrication of optical nano composite Sensor for biological species sensing in human blood serum

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Abstract

A nano optical sensor for direct measurement of B9 vitamin was developed. A new B9 vitamin nano optical sensor was designed with (4,7-Diphenyl-1, 10 phenanthroline disulfonic acid, disodium salt) as a indicator with metod sol-gel for B9 vitamin monitoring in human blood serum sample. A liner response curve was observed for the membrane sensor in B9 vitamin concentration range of 1.18×10^{-9} - 7.34×10^{-5} with a R^2 valve of 0.998 at pH 4. Response time was withing 8 min, with detection limit of 1×10^{-10} at 320 nm. The sensor showed a good durability and low detection limit and short response time.

Keywords: Sensor, Optical, Sol-gel, B9 vitamin

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Photoelectrochemical oxidation of hydrazine on TiO₂ modified titanium electrode

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Abstract

In recent years, photoelectrochemical (PEC) sensing have emerged as a novel area of research because of their reliability and high sensitivity [1,2]. Despite extensive investigations on the photoelectrochemical properties of TiO₂ based electrodes, no photoelectrochemical investigation has been done for the case of hydrazine oxidation. In this work, TiO₂ modified titanium (Ti/TiO₂) electrode was fabricated by anodization process under a constant applied potential. The morphological and microstructural characterization of the Ti/TiO₂ electrode was performed by scanning electron microscopy (SEM). The electrochemical and photoelectrochemical behavior of Ti/TiO₂ electrode in the absence and presence of hydrazine was examined by cyclic voltammetry and linear sweep voltammetry. Experimental results indicated that the photocurrent response of photoelectrode was highly increased in the presence of hydrazine especially at lower potentials. It can be due to the hole scavenging effect of hydrazine which decreases the recombination of photogenerated electrons and holes. Based on these perceptions, a simple photoelectrochemical sensor for detection of hydrazine was obtained. The sensor showed high reproducibility, stability and selectivity properties. This photoelectrode was successfully applied for direct determination of hydrazine in tap water.

Keywords: Photoelectrochemical, Ti/TiO₂, Hydrazine, Voltammetry

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Construction of resorcinol voltammetric nanobiosensor

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Abstract

Resorcinol (RS) is one kind of phenolic compounds with high toxicity. It can be easily absorbed through the gastric tract and human skin, which can cause dermatitis, catarrh, convulsion, cyanopathy, and even death. At present, various methods have been employed for RS determination, including spectrophotometric, high-performance liquid chromatography with diode array detection, microchip capillary electrophoresis with end channel amperometric detection, quartz crystal microbalance, flow injection chemiluminescence, surface Plasmon resonance, fluorescence and spectrofluorimetric [1]. Electrochemical methods offer unique characteristics such as low maintenance costs, high accuracy, and excellent sensitivity. Electrochemical processes have been proposed also for the prevention and the remedy of pollution problems such as treatment of wastewater, phenolic compounds pollutants, insecticides and pesticides [2]. Different kinds of solid electrodes such as metal and carbon electrodes have been employed as the transducers for electrochemical DNA biosensors. Among these solid electrodes, carbon paste electrodes (CPEs) have some specific advantages that include a wide potential window, low background current, and ease of fabrication. Most important, CPEs can be easily endowed with specific properties by introduction of functional materials. Moreover, the surface of the electrode is polishable and renewable [3]. A voltammetric study of RS was carried out at the surface of CPE modified with silver nanoparticles (AgNPs) and DNA, using cyclic and differential pulse voltammetry (CV and DPV). The voltammograms were recorded with μ Autolab FRA2 PG stat. All tests were carried out under room temperature and N_2 atmosphere. The composition of nanobiosensor was optimized. The results showed that the best composition was; 10 μ L DNA and 5 mg AgNPs. The effect of pH and scan rate was tested. The DPV method was used for finding linear dynamic range and detection limit of sensor. Under the optimum condition, results demonstrated that the new nanobiosensor could be used for resorcinol determination in real samples, rapidly.

Keywords: Silver nanoparticles, Voltammetry, DNA, Resorcinol

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Selective electrochemical determination of epinephrine in the presence of ascorbic acid using a carbon paste electrode modified with cobalt hexacyanoferrate nanoparticles

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Abstract

Epinephrine (EP) is one of important neurotransmitters in mammalian central nervous systems [1], and it exits in the nervous tissue and body fluid in the form of large organic cations. The changes of its concentration may result in many diseases [2]. Ascorbic acid (AA), which is electro-active and largely coexists with EP in biological system, could be oxidized at near oxidation potential of EP on most solid electrodes. Thus, selective determination of EP in is significant for developing nerve physiology, pharmacological research and life science. In this work, a carbon-paste electrode (CPE) modified with cobalt hexacyanoferrate nanoparticles (CoHCFNPs) was used for the sensitive and selective voltammetric determination of EP in the presence of high concentration AA. CoHCFNPs were prepared via co-precipitation method by addition of appropriate precursors under ultrasonic agitation. The electrochemical behavior of EP on the modified electrode was explored with cyclic voltammetry (CV) and differential pulse voltammetry (DPV). EP gave a pair of redox peaks at $E_{pa} = 0.216$ mV and $E_{pc} = -0.284$ mV (versus SCE), respectively. The modified electrode shows an excellent electrocatalytic activity for the selective oxidation of EP in the presence of AA at a concentration of 50-fold of DA. Under the optimum pH of 4.0 in 0.1M acetate buffer solution, the DPV current with EP concentration shows a linear relation in the range of 2.0×10^{-6} to 7.9×10^{-5} molL⁻¹ with the correlation coefficient of 0.9976. The detection limit is 3.6×10^{-7} molL⁻¹. The modified electrode exhibited a good reproducibility, sensitivity and stability for the determination of EP injection.

Keywords: Cobalt hexacyanoferrate nanoparticles, Epinephrine, Ascorbic acid, Carbon paste electrode

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Selective electrochemical determination of dopamine in the presence of ascorbic acid using a glassy carbon electrode modified with 1, 4-dihydroxyanthraquinone and carbon nanotube

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Abstract

Dopamine is one of the most important catecholamine neurotransmitters in the mammalian central nervous system. Abnormalities in dopamine concentrations have been linked with several neurological disorders such as the debilitating ailment Parkinson's disease and the mental disorder schizophrenia [1]. Dopamine is also believed to play a central role in Huntington's disease, a fatal genetic neurodegenerative movement disorder and has also been associated with drug addiction and attention disorders [2, 3]. Monitoring the concentration of dopamine is particularly challenging using electrochemical methods because dopamine coexists with many interfering compounds in biological samples. These interfering compounds are usually present at concentrations much higher than dopamine and, moreover, they are oxidised at similar potentials to dopamine at most solid electrodes. This is particularly true of ascorbic acid, the main interfering compound in the determination of dopamine [4]. This work reports on the performance of carbon nanotube and 1, 4-dihydroxyanthraquinone modified glassy carbon electrode (GE) for the selective determination of dopamine (DA) in the presence of ascorbic acid (AA) differential pulse voltammetry (DPV). It can inhibit the voltammetric response of ascorbic acid (AA) while the redox reaction of dopamine is promoted. When a differential pulse voltammetric (DPV) technique was used, the peak separation between DA and AA was 236 mV. Based on this, a selective method could be constructed to detect DA in the presence of 250 times higher concentration of AA. The effect of various experimental parameters on the voltammetric response of dopamine was investigated. Under the chosen conditions, the peak currents are correspondent linearly to the concentrations of DA in the range of 1.0×10^{-6} to 8.7×10^{-5} molL⁻¹ with a limit of detection of 8.5×10^{-8} molL⁻¹. The proposed method offers a tremendous advantage for the simple and clean detection of DA free of the interfering AA signal in a real assay.

Keywords: 1, 4-dihydroxyanthraquinone, Carbon nanotube, Dopamine, Ascorbic acid

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Transition Metal Ions Effect on the Electrochromic Properties of Nanostructured Polypyrrole

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Abstract

An electrochromic material is the one that changes color in a persistent but reversible manner by an electrochemical reaction and the phenomenon is called electrochromism. Conducting polymers (CPs) are finding increasing use in electrochromic applications because of their cathodic, anodic and multicolor control, ease in processability, rapid redox switching, high optical contrast, and long-term stability [1]. The physical material properties such as color, conductivity, and surface energy can be controlled by electrochemical doping in a CP. By doping a CP, new energy levels are created within the band gap due to generation of polarons and bipolarons in the polymer bulk [2]. In this paper, we report on the electrochemical preparation of the conducting polypyrrole (PPy) synthesized with Tironas doping anion. The interest in application of Tironas dopants attributed to relatively high charge to mass ratio of Tiron, compared to other aromatic sulfonate dopants. Investigations showed that film conductivity increased with increasing charge to mass ratio of the dopant molecules [3]. Here in, the Tiron-doped PPy was electrochemically synthesized on a fluorine doped tin oxide FTO/glass electrode in aqueous solution. The use of Tiron promoted charge transfer during electropolymerization and allowed reduced electropolymerization potential of PPy. The resultant polymer was characterized via scanning electron microscopy (SEM), electrochemical and spectroelectrochemical methods. The spectroelectrochemical analysis revealed the PPy-Tiron has distinct electrochromic properties in the presence of different transition metal chloride salts (CoCl_2 , NiCl_2 , CuCl_2 , FeCl_2 , MnCl_2 and ZnCl_2) as an electrolyte compared to conventional electrolyte solutions. Tiron belongs to the catecholate family of molecules, exhibiting chelating properties and can form complexes with transition metal ions. Results showed that the electrochromic properties of PPy such as optical contrast, switching time, film stability and coloration efficiency were greatly changed in the presence of transition metal ions. PPy-Tiron exhibited a switching time of less than 1.5 s and the maximum transmittance contrast ($\Delta T\%$) and coloration efficiency calculated 59% and $136.78 \text{ cm}^2 \text{ C}^{-1}$ at 800 nm respectively in optimum condition. In addition, the incorporation of Tiron into the PPy matrix causes formation of nanostructure, uniform, stable and well adhered coating.

Keywords: Polypyrrole, Nanostructured, Transition Metal Ions, Electrochromic.

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Determination of activity coefficients of CaCl_2 in $[\text{CaCl}_2 + \text{Ethanol} + \text{H}_2\text{O}]$ system by potentiometric method at $T=298.15 \text{ K}$

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Abstract:

In the last decades, a series of ion-interaction models for electrolyte solutions have been proposed to predict the activity coefficient of each solute and the osmotic coefficient of aqueous systems. One of the most famous and useful models is that proposed by Pitzer [1]. To measure the thermodynamic properties of electrolytes in mixed solvent, the most common methods are the isopiestic vapor pressure [2] and potentiometric techniques [3]. Compared to the isopiestic method, this technique presents advantages, such as rapidity and relative simplicity to generate experimental electromotive force (emf) data. Thermodynamic properties of the aqueous binary and ternary electrolyte solutions are useful in the prediction of behavior of mixed salt electrolyte systems, development the electrolyte solutions models, estimation of the interactions occurring in these solutions and so on. In this work, the Pitzer ion interaction model was used to illustrate the ternary electrolyte system $\{\text{CaCl}_2 + \text{Ethanol} + \text{H}_2\text{O}\}$. The activity coefficients were determined using a potentiometric method. The PVC based calcium ion-selective electrode (Ca^{2+} -ISE), and the Ag/AgCl electrode used in this work were prepared in our laboratory and showed a reasonably good Nernst response. In this work, the modeling purposes were achieved based on the experimental potentiometric data of a galvanic cell containing Ag/AgCl electrodes. The measurements were achieved over the AgCl electrolyte molality ranging from 0.001 up to about 6 mol kg^{-1} in mixed ethanol (w%) + water (100 - w%) solvent with different solvent mass fractions percent (w% = 10%, 20%, 30%, 40% and 50%), at $298.15 \pm 0.05 \text{ K}$. The experimental results showed that Pitzer model were suitable to be used satisfactorily to describe this ternary system.

Keywords: Thermodynamic properties, CaCl_2 , Ethanol, Pitzer model, Potentiometric method

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Voltammetric sensing of Copper (II) ion with complexing agent doped polyaniline membrane

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Abstract

Polyaniline is one of the most studied conducting polymers that can be used in different sensor applications [1, 2]. This study was aimed at developing conducting polyaniline film electrodes loaded with a complexing ligand, 5-sulfosalicylate, for preconcentration and electroanalytical determination of Copper (II) ion. A solution of 0.2 M 5-sulfosalicylate + 0.5 M aniline was used to develop a layer of polyaniline on the glassy carbon electrode. The electroreduction of copper into the polyaniline layer was investigated using cycling voltammetry and scanning electron microscopy. The resulting modified electrodes were used for the detection of copper (II) ions by means of the electrochemical preconcentration-differential pulse anodic stripping voltammetry. The effects of polyaniline thickness and preparation conditions, composition of electropolymerization and preconcentration solutions, pH and instrumental variables have been investigated. Experimental results revealed that the peak current for accumulated copper species showed good concentration dependence in the range of 1×10^{-7} to 1×10^{-4} M. The detection limit was determined to be 1×10^{-8} M. The most severe interferents were mercury (II) and Ag (I). The method has been confirmed for the determination of copper using spiked river water. The average recovery was high with a good %RSD.

Keywords: Conducting polymers, Polyaniline, Modified electrodes, Preconcentration.

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Electrocatalytic degradation of Eriochrome Blue Black B using anode Pb/PbO₂

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Abstract

Wastewaters from textile industries represent a serious problem all over the world. They contain different types of synthetic dyes which are known to be a major source of environmental pollution in terms of both the volume of dye discharged and the effluent composition [1]. Most of these dyes are toxic, mutagenic and carcinogenic. Moreover, they are very stable to light, temperature and microbial attack, making them recalcitrant compounds. From an environmental point of view, the removal of synthetic dyes is of great concern [2]. Among several chemical and physical methods, electrochemical degradation process is one of the effective techniques that have been successfully employed for color removal from wastewater [3]. In this study, the electrochemical degradation of Eriochrome blue Black B (ECBBB) was studied. All experiments were carried out at 25°C in a conventional three-electrodes including Pb/PbO₂ as working electrode, a saturated calomel electrode (SCE) as a reference and steelwire as a counter electrode. For the electrochemical oxidation to remove ECBBB from aqueous solutions, various factors such as pH, conductive electrolyte type, time of electrolysis, initial concentration of ECBBB were studied and optimized. Two main parameters were measured to evaluate the electrochemical treatment efficiency, the remaining pollutant concentration and the chemical oxygen demand (COD). The obtained results showed that a high concentration of tested dye (20 ppm) was oxidized to CO₂ under electrolysis at acidic pH and at constant potentials (1/5V vs. reference electrode) during 60 minutes.

Keywords: Anodic oxidation, Azo dyes, Eriochrome blue black B, Electrocatalytic oxidation, PbO₂/Pb

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Electrocatalytic activity of CNT/Smamarium hexacyanoferrate modified glassy carbon electrode toward oxidation of dopamine

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Abstract

Metal hexacyanoferrates (MHCFs) are important inorganic compounds that have been used for modification of electrode surfaces [1]. These compounds are able to form stable crystalline structure films showing reversible redox processes without dissolution in solution [2]. The CNT/SmHCF modified glassy carbon electrode was obtained by potential cycling 40 scans the electrode potential between -0.2V and 0.8 V at a scan rate of 100 mV/s [3]. In order to the study of electrocatalytic activity of CNT /SmHCF modified electrode, dopamine was selected as a model compound and the electrochemical behavior was investigated in 0.1 M PBS solution. The results showed the high catalytic activity of a CNT /SmHCF modified electrode toward dopamine and two well defined redox couples with the formal potentials of 0.225V and 0.150 V, vs. Ag/AgCl at a scan rate of 100 mV s⁻¹ was shown (Fig.1).

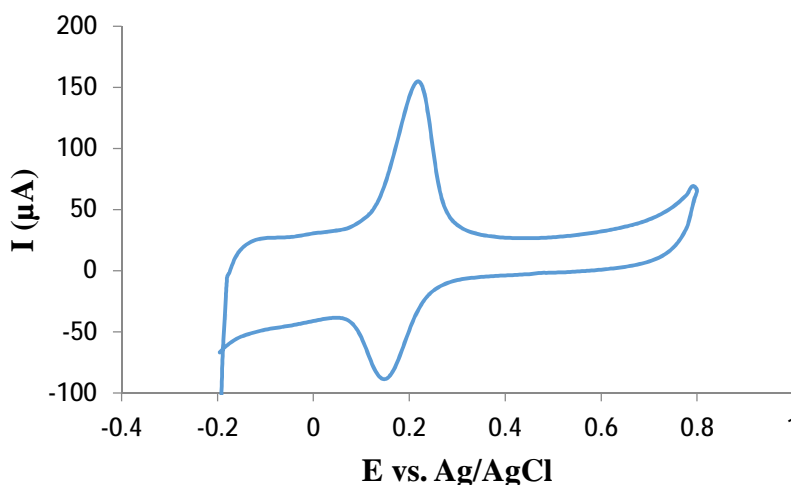


Fig. 1: Results of different scan rate studies of a CNT /SmHCF modified GCE in 0.1 M PBS in presence of 5×10^{-5} M dopamine

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Modification of copper electrode with polyproline – oxalate and its application for electrooxidation of various alcohols

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Abstract

Electrocatalysis is accompanying with decrease potential or increase the current density of the electrode processes during oxidation or reduction of an electroactive species. Usually modified electrodes can be used for the electrocatalysis of chemical species that its redox process on the normal electrode is slow. In the present work, the surface of copper electrode was electrochemically modified with oxalate and polyproline. The electrochemical behavior of modified copper electrode with nanocomposite polyproline-oxalate (PPOxN) was studied and its ability in electrocatalytic oxidation of various alcohols such as methanol, ethanol and ethylene glycol in alkaline solutions was evaluated. The effects of various parameters such as oxalate loading, thickness of polyproline, potential scan rate, number of scan rate and different concentration of tested alcohols in NaOH solution on the electrocatalytic oxidation at the surface of PPOxN modified copper electrode were investigated. Spectrometry techniques such as FT-IR and SEM measurements were performed for surface physical characteristics of the modified electrode, revealing that the polyproline-copper oxalate nanocomposite particles were highly dispersed on the surface copper electrode with a narrow size. The obtained current density for this catalytic oxidation is very high which could come from high surface area caused by the polyproline-copper oxalate nanocomposite modification. An electrocatalytic mechanism was proposed based on the obtained results from cyclic voltammetry and chronoamperometry techniques.

Keywords: Electropolymerization, Electrocatalytic oxidation, Coppermodified electrode, Polyproline, Alcohols

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Preparation of a polypyrrole modified electrode and its application for determination of tin (II) ion

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Abstract

Polypyrrole (PPy) and overoxidized polypyrrole (OPPy) film modified electrodes, were used as voltammetric sensor for determination of some anions and cations in water samples [1]. The OPPy films have been shown to have cation permselectivity, Because of the introduction of oxygen-containing groups on the PPy backbone and the loss of the cationic charge. Further, these oxygen-based groups are hard Lewis bases and favor complexation of hard cations [2]. In this research, Conducting polypyrrole membranes were prepared onto glassy carbon electrodes by electropolymerizing pyrrole in the presence of an anionic complexing ligand, 5-sulfosalicylate anion, and used for voltammetric detection of tin (II). The electrode was oxidized by cyclic voltammetry in NaOH solution. Several important variables that affect the performance characteristics of the prepared sensor, including electropolymerization time (8s), the concentration of doping anion (0.1M), the pH of the preconcentration solution (1.5), and accumulation potential (-700 mV) and time (200 s) were investigated and optimized. The voltammetric response of the electrode was linear within the Sn^{2+} concentration range of 5.0×10^{-7} to 1.0×10^{-3} M, with detection limit of about 10^{-8} M. This overoxidized electrode showed excellent repeatability and stability, also exhibited a good voltammetric selectivity and sensitivity for tin (II). The SSA/OPPy modified electrode used for determination of Sn^{2+} in real samples by preconcentration/differential pulse anodic stripping voltammetry (DPASV). The results were in good agreement with certified values.

Keywords: Polypyrrole, Electropolymerization, 5-sulfosalicylate anion.

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Electrochemical impedance spectroscopy of poly (pyrrole-co-2,4-dinitroaniline) modified composite graphite electrode

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Abstract

Conductive polymers due to their wide applications have attracted the attention of many researchers in recent years [1]. Electrochemical impedance spectroscopy (EIS) is a useful technique for studying the structure and conductive properties at the conductive polymers such as polypyrrole and polyaniline [2]. In this work, electrocopolymerization of pyrrole (py) and 2, 4- dinitroaniline (DNA) on composite graphite electrode is done by cyclic voltammetry (CV) method in potential range of - 0.2 to 1 Volt (vs. Ag/AgCl), at a scan rate 100 mVs^{-1} in 1M phosphoric acid, including equal amounts of molar concentrations of potassium chloride (KCl) and para toluene sulfonic acid (PTSA) as support electrolyte. These modified composite graphite electrode (MCGE) have been characterized using Fourier transform infrared (FTIR), and scanning electron microscopy (SEM) techniques, respectively. Electrochemical behavior of poly(py-co-DNA)/CGE is investigated by electrochemical impedance spectroscopy (EIS) and differential pulse voltammetry (DPV). EIS applied to study the conductive properties modified electrode in 1mM iron solution including 1 M KCl. Semicircle diameter of the Nyquist diagram is equal to the charge transfer resistance (R_{ct}) at the electrode surface. The Nyquist plot shows less than charge transfer resistance (R_{ct}) of poly (py-co- DNA)/CGE compare to bare CGE (Fig. 1). The results showed poly (py-co-DNA)/CGE be applied to the field of electronic and sensor fabrication [3].

Keywords: Electrochemical impedance spectroscopy, Charge transfer resistance, Nyquist plot.

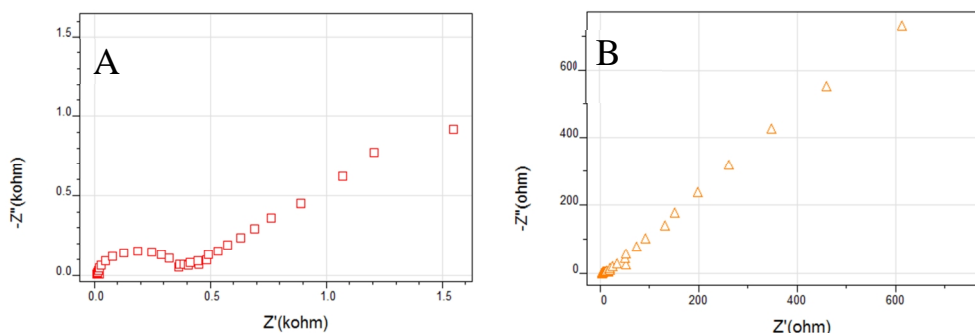


Fig. 1. Nyquist plot of EIS obtained in 1mM $\text{K}_4\text{Fe}(\text{CN})_6 / \text{K}_3\text{Fe}(\text{CN})_6$ and 1M KCl at $E = 0.23 \text{ V}$ for a bare CGE (A) and poly(py-co-DNA)/CGE (B).

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Electrocatalytic Oxidation of Formaldehyde on a Modified GC Electrode with New Complex of Cu

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Abstract

Its scientific name is Metanal, formaldehyde, the gas with a sharp smell. This compound is the simplest aldehyde group, its chemical formula HCHO . Formaldehyde is usually an aqueous solution of 37% formalin to be released. The purpose of this research study electrocatalytic properties of formaldehyde oxidation on the modified electrode with Cu complex was prepared by sol-gel method chronoamperometry and cyclic voltammetry at different scan rates are used to check. A novel chemically modified electrode containing Cu complex was achieved on the surface of GC electrode by sol-gel technique. The electrochemical behavior of modified electrode was characterized by cyclic voltammetry in detail. The film electrode obtained was very stable and exhibited electrocatalytic response for oxidation of formaldehyde. After the preparation of modified glass carbon electrode by sol-gel technique, the electrocatalytic oxidation of formaldehyde in alkaline solution was investigated using the cyclic voltammetry method. Results showed at bare GC electrode, a small oxidation peak current was observed at about 1.55 V and a well-formed sharp catalytic oxidation peak at about 1.14 V was observed at Cu complex modified electrode. The transfer coefficient (α) for electrocatalytic oxidation of formaldehyde and the diffusion coefficient (D) of this substance under the experimental condition were also investigated.

Keywords: Formaldehyde, GC electrode, Cu complex, Cyclic voltammetry, chronoamperometry, electrocatalytic oxidation.

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Ionic liquids functionalized nanoporous silica gel modified carbon paste electrode as a new sensor for potentiometric determination of sulfate ions

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Abstract

Sulfate is a highly widespread anion, and its determination in environmental objects (such as sweet, mineral and seawater; atmospheric precipitation, in particular, acid rain), in various products and raw materials of chemical and pharmaceutical industry, in food, etc., has been an important task [1]. Existing methods for sulfate determination have several drawbacks such as long duration and unacceptability for low concentrations; laboriousness, insufficient selectivity and reproducibility [2]. Here, we report the design and fabrication of a new modified carbon paste electrode, based on nanoporous silica gel that is modified with 1-Methyl, 3-Octyl Imidazolium Chloride ionic liquid for potentiometric determination of Sulfate anions. The sensor displayed rapid and linear response for SO_4^{2-} over a wide range from 10^{-1} M to 10^{-5} M with a Nernstian slope of 27 mV/decade. The sensor worked well in the pH range 2.5-9.0 and its detection limit was 4.9×10^{-5} . Selectivity coefficients were determined for some inorganic anions such SCN^- , NO_3^- , CO_3^{2-} and CrO_4^{2-} . The electrode manifests fast response, good reproducibility and most importantly good selectivities relative to a wide variety of other common inorganic anions. The sensor was successfully tested for monitoring of SO_4^{2-} in tap water.

Keywords: Modified carbon paste electrode, Sulfate ions, Ionic liquids functionalized nanoporous silica gel, Potentiometry.

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Thermodynamic modeling of the KCl + maltose + water system based on potentiometric method at T=298.15 K

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Abstract

Recently, there has been an increasing amount of work about the measurement of thermodynamic properties of electrolytes in mixtures of solvents, because their composition is important in industrial chemistry, biological and other fields [1]. To measure the thermodynamic properties of electrolytes in mixed solvent, there are some methods like isopiestic vapor pressure, vapor pressure lowering and potentiometric method [2]. In the last few decades, a series of ion-interaction models for electrolyte solutions have been proposed to predict the activity coefficient of each solute and the osmotic coefficient of the aqueous systems. One of the most famous and useful models is that proposed by Pitzer [3]. Pitzer equation use with much accuracy, correlate the thermodynamic properties of mixed aqueous electrolyte systems. In this work, the results relating to the mean activity coefficient measurements of KCl in (KCl + maltose + water) system using potentiometric method are reported. The mean activity coefficient of KCl in mixed solvent was determined on a galvanic cell containing a solvent polymeric (PVC) K-ISE and Ag/AgCl electrodes at T=298.15 K. The resulting values of the mean activity coefficients were correlated with Pitzer model and Pitzer ion-interaction parameters (β^0 , β^1 and C^ϕ) were determined. Then these parameters were used for calculation of osmotic coefficient of water and excess Gibbs free energy for the series under the investigated system are reported.

Keywords: Activity coefficient; Pitzer model; KCl; Maltose

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Determination of Naltrexone Hydrochloride by FFT continuous cyclic voltammetry at carbon paste electrode modified with Cerium oxide nanoparticles

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Abstract

Naltrexone is an effective and clinically useful narcotic antagonist [1]. So many methods have been reported for determination of naltrexone such as liquid chromatography (LC) methods with amperometric detection, gas chromatography and high performance liquid chromatography (HPLC) methods [2, 3]. In this work a combination of a new modified carbon paste electrode (CPE) and Fast FFT continuous cyclic voltammetry technique was used for the monitoring of naltrexone hydrochloride in flow-injection analysis. During the measurements, the potential waveform (consist of stripping and potential ramp) was continuously applied on CPE, modified by cerium oxide nanoparticles to improve sensitivity. A special computer-based numerical calculation method (using Fast Fourier Transformation) is used for enhancing the analyte signal and noise reduction, by integrating the current at the range of the redox processes of the analyte. The objective of the performed experiments was to investigate the effects of different parameters on the method sensitivity. The linear range for the concentration was 1-100 nM ($r = 0.991$) with a limit of detection 0.17 nM. Effects of stripping potential, scan rate, and stripping time on the sensitivity and selectivity of the method were investigated.

Keywords: Naltrexone hydrochloride, Flow-injection, FFT continuous cyclic voltammetry, Cerium oxide nanoparticles.

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Mediatory determination of hydrazine on ferrocene functionalized ionic liquid/ carbon nanotube nanocomposite modified carbon-ceramic electrode

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Abstract

Hydrazine (also called diazane) (HZ) is an inorganic compound with the formula N_2H_4 . HZ and its derivatives have been recognized as potential carcinogens. When absorbed through skin they produce caustic-like burn, affect blood production and also cause damages to liver and kidney. Therefore development of a simple and sensitive analytical method for the determination of HZ is necessary for human health inspections and safety assessments [1]. Electrochemical techniques because of their good sensitivity, simplicity, good stability and low cost are the good choice for determination of HZ [1]. Ionic liquids (ILs) have attracted great attention in the field of electroanalytical chemistry due to their excellent properties such as high electronic conductivity, wide electrochemical windows and inherent catalytic effect as not only the solvent but also the modifier [2]. Carbon nanotubes, because of their unique properties such as high electrical conductivity, high surface area, chemical stability, and significant mechanical strength have attracted much attention in electrochemistry. They can be used as electrode materials to promote electron transfer reactions in electrochemical devices. On the other hand, ferrocene and its derivatives due to their good stability in solution, rapid responses to many electroactive substances, independent pH, stability in both oxidized and reduced forms, unreactivity with oxygen, regeneration at low potential and having fast electron transfer are the most successful mediators [3]. In the present work ferrocene functionalized ionic liquid/ carbon nanotube nanocomposite was used for surface modification of carbon-ceramic electrode. This modified electrode was applied for mediatory determination of hydrazine. Under the optimized operational parameters the modified electrode behaved linearly to hydrazine concentration in the range of 3×10^{-8} – 8×10^{-6} M by hydrodynamic amperometry technique. The detection limit was 1.1×10^{-8} M. The applicability of this technique is further tested by analyzing the hydrazine content in boilers' water samples containing different concentrations of hydrazine with appreciable recovery values.

Keywords: Hydrazine, Ionic liquid, Carbon nanotube, Ferrocene

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Synthesis of nano-Ag particles and its application in chemically modified electrode

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Abstract

Olanzapine (OLP) is chemically known as 2-Methyl-10-(4-methyl-piperazin-1-yl)-4H-3-thia-4,9-diaza-benzo[f]azulene, is an atypical antipsychotic drug used in the treatment of schizophrenia and other psychotic syndromes. Three indirect methods for the quality control of olanzapine (OLP) in commercial formulations based on titrimetric and spectrophotometric techniques are described using N-bromosuccinimide (NBS) and two dyes, namely amaranth and janus green B as reagents [1]. Modification of electrodes with suitable materials facilitates the electrochemistry of the redox drugs, which generally results in increased selectivity and sensitivity of the determinations. The electrochemical sensors are inexpensive and possess many advantages such as low background current, wide range of potential windows, rapid surface renewal and easy fabrication [2, 3]. At first, AgNPs were synthesized via reduction process in presence of NaBH₄. XRD, FT-IR, DRS and SEM characterized prepared nanoparticles. After that, a chemically modified electrode using AgNPs for the voltammetric determination of OLP in solution was introduced. The results showed that the modified electrode exhibited excellent electrocatalytic activity to OLP and showed great promise for simple, sensitive and quantitative detection and screening of OLP. The reduction process of OLP was shown to be irreversible over the entire pH range studied (1.0 - 8.0). The study of experimental parameters showed that the maximum current response was obtained at pH = 1.0. OLP was determined by differential pulse voltammetry and the dynamic range was in the range of 3.2×10^{-9} – 3.2×10^{-10} mol L⁻¹. The obtained modified electrode was successfully applied for simple and rapid determination of OLP in pharmaceutical tablets using proposed sensor.

Keywords: Olanzapine, Differential pulse voltammetry, AgNPs, Sensor.

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Fluorescent conjugated polypyrrole derivative nanostructures: synthesis and sensing property

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Abstract

Fluorescent conjugated polymers are highly efficient emitters and have applications as fluorescent sensors [1] or blue emitters in organic displays (OLEDs) [2]. Since polypyrrole (PPy) was first electrochemically synthesized more than two decades ago by Diaz et al. [3], it has been intensively investigated on different electrode surfaces. A new fluorescent polymer, poly (1-pyrrolyl benzene) (PPyB), is synthesized from electrochemical reduction of a water-soluble 1-pyrrolyl benzene monomer. The polymer was characterized by UV-Vis spectrophotometer, scanning electron microscope (SEM), FTIR and fluorescence spectroscopy. Also electrochemical property of PPyB film, synthesis over FTO glass was studied. Optical fluorimetric response in terms of fluorescence enhancing or quenching of PPyB upon exposure to different electron donor and electron acceptor molecules was studied. The change in fluorescence intensity of thin polymer layers in the presence of bromide ion is used as a sensor signal. Sensing tests performed under exposition to calibrated amounts of bromide ion reveal that these simple and inexpensive sensors are able to detect Br⁻ at aqueous solution in a fast reliable way, with a sensitivity linearly related to concentration in the range between 2×10^{-7} and 5×10^{-5} mol L⁻¹. Electro optical PPyB sensor presents low detection limit (8×10^{-8} mol L⁻¹) and good reproducibility in construction (RSDs < 6%). The sensor is selective to bromide ion over diverse anions and cation. The method was applied for determination of bromide in environmental real samples with satisfactory recovery.

Keywords: Fluorescent nanostructure; Electro optical sensor; Poly pyrrole; FTO glass; Bromide sensor

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Amperometric biosensor based on direct electrochemistry of hemoglobin in poly-allylamine@Fe₃O₄ film

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Abstract

Direct electron transfer between an electrode material and aredox-active protein or protein clusters has become an intriguing phenomenon for last three decades [1]. In general, the functions of most proteins depend upon precise three dimensional structures [2, 3], which are affected by a number of noncovalent interactions such as hydrogen bonds, van der Waals interactions, electrostatic interactions, hydrophilic/hydrophobic effects, and soon [4]. In the present work, the novel biocompatible poly-allylamine@Fe₃O₄ nanocomposite (PAA@Fe₃O₄) was synthesized via emulsion polymerization. Hemoglobin (Hb) was immobilized in poly-allylamine@Fe₃O₄ film onto the gold electrode by layer by layer (LBL) method. The modified electrode exhibited a pair of well-defined peaks during cyclic voltammetry, which was attributed from the direct electron transfer of heme proteins. The immobilized Hb showed an excellent electrocatalytical response to the reduction of hydrogen peroxide. The sensor exhibited a fast response and high sensitivity. Through the use of optimized conditions, the linear range for H₂O₂ detection was from 2.5×10^{-6} M to 5×10^{-4} M with detection limit of 0.2 μ M. The proposed biosensor showed long-lasting stability and excellent reproducibility.

Keywords: Amperometric biosensor, Hemoglobin, Poly-allylamine, Nanocomposite.

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A novel sensor for simple and fast determination of tetracyclines based on glassy carbon electrode modified with Ni (II) complex of benzil bithiosemicarbazone

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Abstract

An antibacterial is a compound or substance that kills or slows down the growth of bacteria. The term is often used synonymously with the term antibiotic(s) [1]. Today, antibiotic (s) has come to denote a broader range of antimicrobial compounds, including anti-fungal and other compounds [2]. According to the report of Union of Concerned Scientists, it is known that the estimated 16,000 tons of antimicrobial compounds are used in US, nearly 93 tons of antibiotics used in New Zealand and 14,600 tons of active antimicrobials used in Kenya [3]. A chemically modified electrode constructed by immobilization of functionalized multi-wall carbon nanotubes (fMWCNTs) and Ni (II) complex of benzil bithiosemicarbazone (Ni-BBTC) at the surface of glassy carbon electrode (GCE). The Ni-BBTC-fMWCNT-MGCE was used as a sensitive electrochemical sensor for detection of tetracyclines. There was a good linear relationship between the peak current (I_{pa}) and tetracycline concentration in the range from 8.0×10^{-7} to 1.0×10^{-5} M, and the detection limit was 2.0×10^{-7} M (S/N= 3). The method has been successfully employed to detect tetracycline in pharmaceutical and clinical preparations.

Keywords: Thiosemicarbazone, Tetracycline, Carbon nanotube, Nickel

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Electrochemical determination of ascorbic acid at poly Co(Phen)₃/Carbon -Nanotube-modified Electrodes

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Abstract

The present study demonstrates electro-catalytic oxidation and determination of ascorbic acid on the surface of a glassy carbon electrode modified with carbon-nanotube and tris (1, 10 phenanthroline)cobalt complex, (Co(Phen)₃)/(MWCNTs/GCE). The modified electrode was prepared by electro-polymerization of Co(Phen)₃, (cyclic voltammetry), on the surface of the MWCNTs/GC electrode. The electro-catalytic oxidation peak current of ascorbic acid on the surface of the modified electrode was significantly higher relative to on the surface of a bare glassy carbon electrode at the physiological pH value (pH= 7.4) [1, 2]. The apparent electron transfer rate constant (k_s) and transfer coefficient (α) were also determined by cyclic voltammetry method. The effect of some parameters including electro-polymerization pH, amount of Co(Phen)₃, deposition time, number of cycle electro-polymerization and pH of ascorbic acid determination were studied and the optimum values for each factor is suggested. The anodic oxidation peak potential of ascorbic acid at the poly-Co(Phen)₃/MWCNTs-modified glassy carbon electrode occurred at low over-potential (0.13 V versus Ag/AgCl), and the treatment of the voltammetric data showed that it was a purely diffusion-controlled reaction involving one-electron in the rate-determining step. This modified electrode showed excellent electro-catalytic activity toward ascorbic acid at the physiological pH value and provides an easy sensitive method for the determination of ascorbic acid. The proposed method was also successfully used for the determination of ascorbic acid content in the fruit juices.

Keyword: Glassy carbon electrode modified with carbon-nanotube (MWCNTs/GC), Co(Phen)₃, Ascorbic acid, Cyclic voltammetry

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A sensitive RDE amperometric bromate sensor based on graphene nanosheets-supported palladium nanoparticles modified glassy carbon electrode

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Abstract

Bromate ion (BrO_3^-) is a kind of contaminants that has a potential carcinogen to humans. Bromate is a disinfection byproduct that can be formed during chlorination or ozonation in bromide-containing water treatment. Potassium bromate is recognized as one of the best dough improvers in the bakery industry. It widely used as a flour additive in the bread-making process for the maturation of flour [1]. Because the carcinogenic characters of bromate, the determination of bromate is important for environmentalist and the scientist. Graphene nanosheets (GNs), as a new kind of two-dimensional carbon material, have the excellent electrical conductivity, large specific surface area, high electron mobility and high thermal conductivity. Therefore, GNs has tremendous potential for electrochemical catalysis and biosensing [2]. Metal nanoparticles have the unique properties different from those of the bulk materials such as good conductivities, outstanding electrocatalytic performance, large surface-to-volume ratio and special binding sites available on the surface of the particles [3]. In this manuscript, a new amperometric sensor was fabricated based on graphene nanosheets-supported palladium nanoparticles (PdNPs-GNs) modified glassy carbon (GC) electrode for bromate determination. The surface morphology of modified electrodes was investigated by scanning electron microscopy (SEM). Cyclic voltammetry (CV) and rotating disk electrode (RDE) amperometry methods were used to determinate and investigate electrochemical parameters of bromate reduction. The experimental data showed that this modified electrode has significantly electrocatalytic activity towards bromate reduction in a wide concentration range (1–10 μM and 10–1000 μM) with a detection limit of 1.05×10^{-7} M. This sensor has a short response time (15 s) and the sensitivity is 7.428 A/Mcm^2 . The recovery experiments of bromate were utilized by this method in different flour samples and good accurate results were obtained. Furthermore, this electrode demonstrated good stability, repeatability and selectivity remarkably.

Keywords: Bromate reduction, RDE amperometric sensor, Graphene nanosheets, Palladium nanoparticles

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Design and Construction of a Novel Plasma-Based Electroanalyzer for Sensitive and Ultra Trace Detection of Some Metal Ions

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Abstract

Modern electrochemical theory is based on the properties of electroactive species in liquids or solids. In contrast, the electrochemical property of species in gaseous plasmas has been captured similar attention. In this study, plasma has been considered as suitable media during investigation of the electrochemical properties of electroactive species [1]. For this purpose, doping laminar plasma with ionizable additives can increase the extent of ionization, thus producing an electrolyte suitable for electrochemical studies. Therefore, metals in flames are ionized either by thermal ionization (chemi-ionization) or to a lesser extent by various mechanisms such as charge transfer, collisional ionization, electron impact, etc. [2]. The main purpose of this study is based on measuring the redox potentials, in absence of any contribution of diffusion/junction potential. To achieve to this aim, a three-electrode system has been designed to make the ion current measurements using a potentiostat during nebulizing electrolyte solution to the plasma [3]. Based on the voltammograms under optimized conditions, the generated plasma behaves as suitable an electrolyte media with very small over potential for sensitive or sometimes selective detection and determination of some electroactive species with detection limit at parts per billion (ppb) levels. The prospect of this gas phase electroanalyzer promises access to a plethora of redox reactions, free of any potential limits, in the absence of any solvent. Based on the experimental results, significant advantages of this electrochemical system is i) wide potential window that is probably attributed to the absence of solvent ii) improved detection limit owing to small over potentials, iii) high sensitivity (at ppb level) and iv) very fast response time (less than ~1.0 s).

Keywords: Plasma-Based Electroanalyzer, Metal Ions

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Application of a new nanostructured modified electrode for electrochemical determination of captopril using a redox mediator

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Abstract

Captopril, (2S)-1-[(2S)-3-mercapto-2-methyl propanoyl] pyrrolidine-2-carboxylic acid (CP), is a specific inhibitor of the angiotensine converting enzyme which is widely used in the treatment of hypertension [1]. Para-aminobenzoic acid (PABA) is a non-protein amino acid that is widely distributed in nature. Since a small amount of it is present in Vitamin B-complex, it is included as a member of vitamins. Since it improves the protein used in the body and is also related to red blood cell formation as well as for assisting the manufacture of folic acid in them intestines, it is biologically important [2]. Manganese titanate, pyrophanite MnTiO_3 , is a humidity sensing material with excellent sensitivity, good selectivity, low temperature coefficient near zero and good stability. The pyrophanite MnTiO_3 has also been studied for magnetic and electrochemical properties [3]. Electrochemical determination of CP is difficult using an ordinary carbon paste electrode due to large oxidation overpotential. One promising approach for minimizing overpotential of analytes is the use of electrocatalysis. In the present study, a novel modified carbon paste electrode based on MnTiO_3 nanopowder was prepared and applied for analyzing of CP in the presence of PABA as a mediator. The diffusion coefficient (D), and the kinetic parameters such as electron transfer coefficient (α), for CP were also determined using electrochemical approaches. Under the optimized conditions, the electrocatalytic oxidation peak current of CP showed two linear dynamic ranges with a detection limit of 1.57 nM. The linear calibration range was 1.0×10^{-8} to 1.0×10^{-6} M using differential pulse voltammetry (DPV) technique. Finally, the proposed method was successfully applied for determination of CP in real samples.

Keywords: Captopril, Electrocatalytic, Manganese titanate nano powder, Modified carbon paste electrode.

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Palladized aluminum as a novel solid electrode for anodic stripping voltammetric determination of trace silver

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Abstract

The electrochemical techniques, especially anodic stripping voltammetry (ASV) have obtained considerable attention for trace analysis of heavy metal ions due to their good sensitivity, low cost, rapidity, easy operation, high selectivity, portability and low detection limits [1]. Silver is an important commercial metal for use in photography, dentistry, electronic compounds, etc. Currently, with increasing use of silver compounds and silver-containing preparations in industry and medicine, the content of silver in environment has been increased. Thus, silver (I) ion determination in industrial and environmental samples owing to potential toxicity of silver is very important [2]. Also, because of the extremely positive reduction potential of silver (I), a suitable electrode that can provide sensitive and selective determination of silver is highly desired [3]. In present work, Palladized aluminum electrode has been used as a novel solid electrode for stripping voltammetric determination of silver at micro molar concentration levels. It has been demonstrated that this electrode has advantages such as simple and rapid preparation, low cost, high stability, wide anodic potential range, good sensitivity and high selectivity. The effects of experimental conditions such as electrode preparation, pH, deposition potential, deposition time and etc were investigated. Under the optimum conditions, a linear calibration graph was obtained in the range of 3-30 μM with a correlation coefficient of 0.999.

Keywords: Palladized aluminum electrode, Silver determination, Stripping analysis

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Thermodynamic modeling of the KCl + Lactose + water system based on potentiometric measurements at 298.15 K

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Abstract:

Recently, there has been an increasing amount of work concerning the measurement of thermodynamic properties of electrolytes in mixtures of solvents, because their composition is important in industrial, chemistry, biological and other fields [1, 2]. During the last decades, the series of ion-interaction models for electrolyte solution have been proposed to predict the activity coefficient of each solute and osmotic coefficient of aqueous systems. Among them, the Pitzer ion-interaction model is one of the most famous and useful models [3]. In this work, the results relating to the mean activity coefficient measurements for KCl in (lactose + water) mixtures using the potentiometric method are reported. The mean activity coefficient of KCl in the (KCl + lactose + water) system was determined on a galvanic cell containing a solvent polymeric (PVC) K-ISE and Ag/AgCl electrodes at T=298.15 K. In the present study, the determination of the activity coefficient for KCl in various glucose/water mixed solvent systems containing 2.5, 5, 7.5, 10, 12.5 mass fractions of glucose over ionic strength ranging from 0.0001 to 3 mol kg⁻¹ are reported. The resulting values of the mean activity coefficients, the osmotic coefficients and the excess Gibbs free energy together with Pitzer ion-interaction parameters ($\beta^{(0)}$, $\beta^{(1)}$ and C^ϕ) for the series under investigated system are reported.

Keywords: Activity coefficients; Pitzer model; KCl; Lactose; Potentiometric

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Voltammetric Rizatriptan Biosensor

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Abstract

Rizatriptan benzoate (Riz) belongs to a group of medicines known as serotonin agonists [1]. It is described chemically as: *N, N* dimethyl-5-(1*H*-1, 2, 4-triazol-1-ylmethyl)-1*H*-indole-3-ethanamine mono benzoate. It is used in the treatment of migraines. The literature reveals that various methods for the determination of rizatriptan benzoate are spectrophotometry, gas chromatography, high-performance liquid chromatography, liquid chromatography/tandem mass spectrometry, microemulsion electrokinetic capillary chromatography and reversed-phase liquid chromatographic [2]. The electrochemical biosensors are inexpensive and possess many advantages such as low background current, wide range of potential windows, rapid surface renewal and easy fabrication [3]. There is no voltammetric report using bovine serum albumin (BSA) for construction of Rizatriptan biosensor in the accessible literature. In this work, a chemically modified electrode using bovine serum albumin (BSA) for the voltammetric determination of Riz in solution was introduced. The results showed that the modified electrode exhibited excellent electrocatalytic activity to Riz and showed great promise for simple, sensitive and quantitative detection and screening of Riz. The oxidation process was shown to be irreversible over the entire pH range studied (1.0 - 10.0). The study of experimental parameters showed that the oxidation reaction is totally irreversible and maximum current response was obtained at pH=10 in phosphate buffer. Riz was determined by differential pulse voltammetry and the peak current was found linearly with its concentration in the range of $8.0 \times 10^{-3} - 1.0 \times 10^{-3} \text{ mol L}^{-1}$. The obtained modified electrode was successfully applied for simple and rapid determination of Riz in standard solutions and pharmaceutical tablets using differential pulse voltammetry.

Keywords: Biosensor, Voltammetry, Bovine Serum Albumin, Rizatriptan

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Quantitative Determination of Trace Amount of Lanthanum (III) using Adsorptive Stripping Voltammetry Technique

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Abstract

Trace amount of lanthanum (III) was determined using adsorptive stripping voltammetry methods. In this work an alizarin S and carbon paste electrode were used as as complexing agent and working electrode, respectively. The applied electrode was formed by mixing the carbon powder with paraffin in micropipette tip (diameter, 0.5 cm). The recommended method involves two steps: 1) adsorptive accumulation of metal ion with chelating agent at the surface of the carbon paste electrode. 2) The stripping of the formed complex from the applied electrode into the aqueous solution. In the stripping step the generated current was recorded. The optimized parameters include accumulation potential 550 mV, accumulation time of 100 s, and solution pH= 6. In the present work, a LOD of 1.48×10^{-11} M, a RSD of 6.18% and a correlation coefficient of 0.987 were obtained. Finally, the present work was applied for trace determination of lanthanum in real samples [1, 2]. Table 1 states determination of lanthanum in standard lanthanum samples.

Table 1. Determination of lanthanum in standard lanthanum samples

Sample	Lanthanum present (mg kg ⁻¹)		
	Certified value	Present method*	Relative error (%)
ACV-1 (74/19)	35.0 34	50 ± 0.08	- 1.43
BCR-1 (52/19)	26.0	26.40 ± 0.10	+ 1.54

*Average of four determinations ± S.D.

Keywords: Lanthanum (III), Adsorptive stripping voltammetry, Carbon paste electrode, Chelating agent.

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Trace Determination of Cadmium Using an Efficient Flow Injection Anodic Stripping Voltammetric Technique

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Abstract

In this work, a practical flow injection anodic stripping voltammetric technique was applied for trace determination of cadmium. A modified electrode was formed by coating the bismuth film on glassy carbon and applied as a working electrode. Acetate buffer was used for adjusting the pH of the solutions in all experiments. Afterwards, the deposition step of cadmium was carried out by using a constant potential of -1.40 V versus Ag/AgCl. After this step, square wave waveform method was applied by scanning the potential from -1.40 to 0.45 V and the voltammogram was recorded as the analytical response. In this work, the optimized parameters involved a linear dynamic range of 5 - 65 $\mu\text{g L}^{-1}$, a RSD of 3.4 and a sampling frequency of 13 h^{-1} . Finally, the present work was applied for trace determination of cadmium in ground and surface water samples. Figure 1 states linear scan of ASV of cadmium with different concentrations [1, 2].

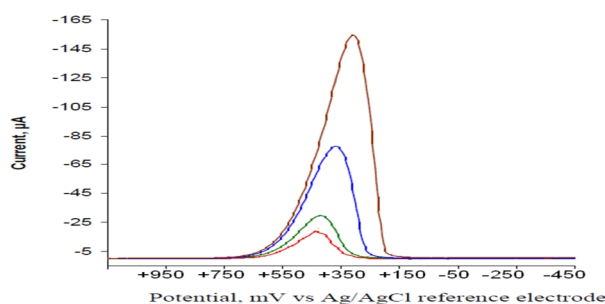


Figure 1: Linear scan of ASV of cadmium with different concentrations

Keywords: Flow injection anodic stripping voltammetric technique, Modified electrode, Cadmium, Trace determination

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Graphene oxide nano sheets paste electrode as a novel electrochemical sensor for epinephrine

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Abstract

Epinephrine (EP) is an important catecholamine neurotransmitter in the mammalian hormonal and central nervous system. Presence of EP in the body affects the regulation of blood pressure and the heart rate, lipolysis, immune system, and glycogen metabolism. It is a drug which has been used as a common emergency healthcare medicine for emergency treatment in severe allergic reaction, cardiac arrest and sepsis. Therefore, the quantitative determination of epinephrine concentration in different human fluids, such as plasma and urine, is important [1]. Several methods have been described for the determination of EP such as spectrofluorimetry, spectrophotometry, chromatography and chemiluminescence methods. While these methods are often time consuming, inherently expensive or complicate, electrochemical methods for the determination of EP present advantages such as simple, rapid, inexpensive and easy to use [1]. Graphene (G) is a flat monolayer of carbon atoms tightly packed into a two-dimensional (2D) honeycomb lattice. It exhibits good electrical conductivity, high electrocatalytic activity, high surface area, and strong mechanical strength. The suitable electronic properties of graphene suggest that it has the ability to promote electron transfer when used as the electrode material, which provides a new way to design novel electrochemical sensors and biosensors. Hence, the modified graphene electrodes have been successfully applied to study or determine some biological and organic molecules [2]. In the present study, carbon paste electrode modified with graphene oxide nano sheets was used for the determination of EP. The cyclic voltammetry and square wave voltammetry investigations showed effective electrocatalytic activity of the modified electrode in lowering the anodic over potential for the oxidation of EP. Compared with the bare electrode, the oxidation current of EP increased greatly and the oxidation peak potential shifted negatively by 160 mV.

Keywords: Epinephrine, Graphene oxide nano sheets, Carbon paste electrode, Electrochemical sensor

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Novel isoproterenol electrochemical sensor using graphene oxide nano sheets paste electrode

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Abstract

Isoproterenol (IP) is widely used for the treatment of primary pulmonary hypertension and allergic emergencies, status asthmaticus, bronchial asthma, ventricular bradycardia, cardiac arrest, and glaucoma. It is also used to bronchitis, cardiac shock and heart attack. Nevertheless, the excess of the drug may cause heart failure and arrhythmias. Several methods have been described for the determination of IP such as spectrofluorimetry, spectrophotometry, chromatography and chemiluminescence methods. While these methods are often time consuming, inherently expensive or complicate, electrochemical methods for the determination of IP present advantages such as simple, rapid, inexpensive and easy to use [1]. Graphene (G) is a flat monolayer of carbon atoms tightly packed into a two-dimensional (2D) honeycomb lattice. It exhibits good electrical conductivity, high electrocatalytic activity, high surface area, and strong mechanical strength. The subtle electronic properties of graphene suggest that it has the ability to promote electron transfer when used as the electrode material, which provides a new way to design novel electrochemical sensors and biosensors. Hence, the modified graphene electrodes have been successfully applied to study or determine some biological and organic molecules [2]. In the present study, carbon-paste electrode modified with graphene oxide nano sheets was used for the determination of IP. The cyclic voltammetry and square wave voltammetry investigations showed effective electrocatalytic activity of the modified electrode in lowering the anodic over potential for the oxidation of IP. Compared with the bare electrode, the oxidation current of IP increased greatly and the oxidation peak potential shifted negatively by 230 mV.

Keywords: Isoproterenol, Graphene oxide nano sheets, Carbon paste electrode, Electrochemical sensor

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Voltammetric determination of ascorbic acid using a 5-(4'-amino-3'-hydroxy-biphenyle-4-yl) acrylic acid-carbon nanotube modified carbon paste electrode

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Abstract

Ascorbic acid (AA) is an antioxidant, which plays significant roles in proper functioning of human metabolism and central nervous. It can prevent or treat of common cold, mental illness and all types of cancer [1]. Various analytical techniques for determination AA have been reported in literature such as fluorometry, chromatography, spectrophotometry and capillary zone electrophoresis. These methods are time consuming and/or expensive and often need the pretreatment step. Also, some of them suffer from low sensitivity and selectivity in the corresponding determinations. To overcome these defects, electrochemical methods are used extensively for the elegant and sensitive properties such as selectivity, reproducibility, low cost and simplicity of this approach. Carbon nanotubes (CNTs) have attracted considerable attention because of their excellent properties such as high electrical conductivity, good chemical stability, and extreme mechanical strength [2]. It has been shown that application of CNTs results in extraordinary advantages over conventional electrodes, including enhanced mass transport, catalysis, highly effective surface areas, high porosity, more adsorption, and reactive sites. In recent years, chemically modified electrodes have attracted large interest due to their potential applications in various analyses. In the present study, carbon-paste electrode modified with carbon nanotube and 5-(4'-amino-3'-hydroxy-biphenyle-4-yl) acrylic acid was used for the determination of ascorbic acid. The cyclic voltammetry and square wave voltammetry investigations showed effective electrocatalytic activity of the modified electrode in lowering the anodic over potential for the oxidation of AA. Compared with the bare electrode, the oxidation current of AA increased greatly and the oxidation peak potential shifted negatively by 270 mV.

Keywords: Ascorbic acid, Carbon nano tube, Carbon paste electrode, Voltammetric determination.

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Study Behaviour of Electrolyte Additives Effect on Lead Acid Battery Performance

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Abstract

Additives should be chemically, thermally and electrochemically stable in H₂SO₄ solutions for prolonged periods of time, and also have low cost. Inorganic compounds, carbons and polymer emulsions can be used as electrolyte additives. Inorganic Compounds include phosphoric acid, boric acid, citric acid and some soluble metal sulfates. It has been found that additions of H₃BO₃ in concentrations up to 0.4% inhibit the formation of hard PbSO₄ and decrease the self-discharge of the PbO₂ electrode. Addition of 0.8 wt% H₃PO₄ to sulfuric acid electrolyte prevents passivation of positive plates with Sb-free grids on deep discharge cycling and facilitates formation of a-PbO₂ modification as well as of fine PbSO₄ crystals during discharge [1]. The presence of citric acid has a beneficial effect on the charge/discharge processes, but with the increase of its concentration, evolution of oxygen and hydrogen increases, too. The optimum effect of citric acid is achieved when added in amounts of about 2 g L⁻¹ of electrolyte [2]. The other additives electrolyte is SnSO₄. Addition of 0.1 g of SnSO₄ per 1L of H₂SO₄ electrolyte improves substantially the re-chargeability of batteries on deep discharge cycling. It has also been established that addition of 0.0225 M Al₂(SO₄)₃ to the sulfuric acid electrolyte increases significantly the rate of PbSO₄ reduction and thus prevents passivation of the negative plates of lead acid batteries [3]. The ultra-fine carbon (UFC) and polyvinyl alcohol (PVA) colloid solution comprises carbon black particles 0.15 mm in size and PVA in a ratio of 10:4 by weight. Addition of 5 vol% of UFC-PVA colloid to the cell electrolyte has been recommended. The UFC-PVA colloid reduces the self-discharge of negative plates and can act as an activator (de-passivator) of the sulfated lead [1]. The use of polyaspartate (PASP) as electrolyte additive has been found to control the crystallization process of lead sulfate by modifying the shape and size of the crystals. Addition of PASP to electrolyte improves the utilization of the negative active material and reduces the internal resistance of the negative plates. A beneficial effect of PASP on the performance of lead acid batteries is observed during high-rate partial state of charge (HRPSoc) operation, when the electrolyte contains 0.1% PASP. Addition of PASP prevents accumulation of 'hard sulfate' on the negative plates of batteries.

Keywords: Electrolyte additives, Lead acid battery-carbons, Polymer emulsions, Phosphoric acid. Boric acid, Citric acid

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Kinetic modeling of photocatalytic degradation of sulfonic acid as a surfactant in UV/TiO₂ process

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Abstract

This investigation focuses on a kinetic modeling of photocatalytic degradation of the surfactant containing sulfonic acid groups. Present modeling, in fact, predicts concentration of surfactant during the photodegradation process at different light intensities and surfactant concentrations [1]. The photocatalytic degradation of the surfactant has been investigated using UV radiation in the presence of TiO₂, as a function of initial concentration of surfactant, TiO₂ amount, light intensity and pH. TiO₂ nanopowders were synthesized by hydrolysis of Ti (OCH(CH₃)₂)₄ in a distilled water. X-Ray powder Diffraction (XRD) outputs revealed that the obtained powders were nano structure and the predominant crystalline phase is anatase [2]. Our findings demonstrate that various parameters exert their individual influence on the photocatalytic degradation of surfactant in wastewaters [3]. The mentioned parameters have been categorized as follow: the initial pH of the solution to be degraded, the impacts of oxidizing agents on photocatalytic degradation, amount of TiO₂, concentration of pollutant and irradiation time. Furthermore, our study has led us to conclude that, this anionic surfactant of sulfonic acid class, can easily be degraded by UV radiation in the presence of TiO₂ as a photocatalyst [1]. In addition, the rate of degradation process is calculated by a kinetic modeling.

Keywords: Kinetic modeling, Photocatalytic degradation, UV/TiO₂

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Synergistic effect of single-walled carbon nanotube decorated with silver nanoparticle for the estimation of glucose in real systems

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Abstract

Glucose detection is of great interest from several points of view ranging from medical applications of blood glucose sensing to ecological approaches, such as in wastewater treatment, in food and textile industries, in environmental monitoring, and so forth [1]. Many attempts have been made to determine glucose without the use of enzymes. The majority of these nonenzymatic electrochemical glucose sensors rely on the current response of glucose oxidation directly at the electrode surface. Early research on this subject have focused on the use of noble metals for developing nonenzymatic sensors [2, 3]. Herein, we report a study on the nonenzymatic glucose detection based on the Ag-single-walled carbon nanotube (Ag-SWNTs) nanostructure catalyst. The high density of Ag nanoparticles was grown on the surface of SWNTs and characterized with transmission electron microscope (TEM) and X-ray diffraction (XRD) techniques. We fabricated highly sensitive, stable, and fast response amperometric glucose sensors operating at physiological conditions (pH 7.4). The modification of a glassy carbon (GC) electrode with the Ag-SWNT nanostructures increases its active area and promotes the electron transfer for the glucose oxidation reaction via the SWNTs. The Ag-SWNT nanostructures not only catalyze glucose oxidation at a remarkably negative potential in enzyme-free solution but also are insensitive to potential interfering agents such as ascorbic acid and uric acid. This study demonstrates that Ag-SWNT nanostructures can be a potential catalyst in fabricating novel nonenzymatic glucose sensors with high sensitivity, selectivity, and stability.

Keywords: Glucose, Nonenzymatic reaction, Ag nanoparticle, Sensors.

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Simultaneous determination of 4-aminohippuric acid and uric acid on a glassy carbon electrode modified with gold–platinum alloy nanoparticles/multiwall carbon nanotubes

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Abstract

4-aminohippuric acid (PAH) is useful for the measurement of renal plasma flow because it is secreted primarily by the renal tubules [1, 2]. Herein, a novel 4-aminohippuric acid (PAH) sensor was constructed, based on a glassy carbon electrode (GCE), which was modified with gold–platinum alloy nanoparticles (Au–PtNPs) by electrodeposition on multiwall carbon nanotubes (CNTs) in chitosan (CS) film (CNTs/CS) [3, 4]. The properties of Au–PtNPs/CNTs/CS were characterized by scan electron microscopy (SEM), X-ray diffraction (XRD), cyclic voltammetry (CV), and electrochemical impedance spectra (EIS). Primary study indicated that Au–PtNPs/CNTs had a better synergistic electrocatalytic effect on the reduction of PAH than did AuNPs/CNTs or PtNPs/CNTs at a low applied potential window. The sensor exhibited excellent performances for PAH at a low applied potential (0.1 V) with a high sensitivity ($6.43 \mu\text{A mM}^{-1}$), a low detection limit (0.2 μM), a wide linear range (0.002–7.0 mM), a fast response time (<5 s), and good reproducibility, stability, and selectivity. In addition, the sensor was applied in the determination of PAH in human blood and urine samples, and satisfied results were obtained.

Keywords: 4-aminohippuric acid, Nanoparticles, Alloy, Sensors

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Development of a new carbon paste Gabapentin sensor and its applications to pharmaceutical and biological analysis

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Abstract

Gabapentin, 2-[1-(aminomethyl) cyclohexyl] acetic acid, is an anticonvulsant that is used for preventing seizures and for treating post herpetic neuralgia [1]. It was originally developed to treat epilepsy, and currently is also used to relieve neuropathic pain. It is recommended as a first line agent for the treatment of neuropathic pain arising from diabetic neuropathy, post-herpetic neuralgia, and central neuropathic pain [2]. The literature survey showed that gabapentin was determined by several sophisticated analytical methods include high-performance liquid chromatography, liquid chromatography coupled with mass spectrometry, capillary electrophoresis, spectrophotometry, and voltammetry [3]. However, despite the importance of a potentiometric carbon paste sensor for the easy and fast determination of gabapentin in pharmaceutical and biological samples, CPE for this drug hasn't been reported. The most advantages of CPEs are ease of preparation, modification and regeneration and especially low cost. In this study a new carbon paste electrode based on the use of ion association complex of gabapentin with phosphotungstate is constructed. The proposed electrode exhibits a Nernstian slope of 59.7 mV/decade for gabapentin over a concentration range of 8.1×10^{-6} to 2.7×10^{-3} M, with a detection limit of 5×10^{-6} M. The electrode has a fast response time (5 s), satisfactory reproducibility and good life time (more than 2 months). The potentiometric response of the electrode was studied in buffered solution. The influence of the temperature on the response of the electrode was investigated and the temperature coefficient of the electrode was calculated by recording the calibration graphs at different temperatures. The electrode was applied in potentiometric titration of gabapentin solution and also determination of gabapentin in tablet and blood serum samples.

Keywords: Gabapentin, Phosphotungstate, Carbon paste electrode, Potentiometry, Sensor

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Electrochemical properties of Mixed-Ligand Ni (II) Complex

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Abstract

Aromatic nitrogen heterocycles such as 2, 2'-bipyridine, 1,10-phenanthroline and di-2-pyridylamine are classical but ever green ligands in transition metal chemistry. Dpa is less rigid than bpy and phen and its metal complexes often show particularly good redox catalytic characteristics due to the conjugation and flexibility properties of that ligand. Moreover, dpa has the N-H amine group as additional function allowing the formation of hydrogen bonds and more interestingly, the deprotonation of this amine in dpa and its analogues has made possible the formation of mononuclear and polynuclear species where the deprotonated bridging amine nitrogen can also be involved in the coordination pattern[1-3]. The Ni(II) mixed – ligand complex of [Ni(phen)₂(dpa)](PF₆)₂, Where dpa is di-2-pyridylamine and phen is 1,10-phenanthroline has been prepared and characterized by cyclic voltammetry , elemental analysis, IR, ¹H NMR and electronic absorption spectroscopies. Cyclic voltammograms were recorded by using a Micro Autolab Type III. Three-electrode system consisting of a GC disk working electrode, a Pt wire auxiliary electrode, and an Ag/AgCl reference electrode. Cyclic voltammetry of [Ni(phen)₂(dpa)](PF₆)₂ was performed on an acetonitrile solution with 0.1 M TBAH as a supporting electrolyte. The Ni(III/II) couple was observed at 1.51 V versus Ag/AgCl. In this complex an irreversible reduction peaks at -1.82 V is assigned to the reduction of dpa ligand. It should be mentioned that phenanthroline is also electroactive (- 1.68V).

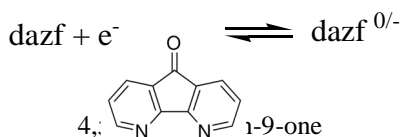
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Cyclic voltammetry of [Ni (phen)₂(dazf)](PF₆)₂Ghobad Mansouri^{a,*} Arezo Noori^a^a Department of Chemistry, Payame Noor University, PO BOX 19395-3697, Tehran, IranE-mail: mansouri.gh@gmail.com

Abstract

The metal chelating properties of polypyridyl ligands have been widely utilized in all aspects of coordination chemistry such as ligand substitution, redox reactions and photochemistry reactions. 4,5-diazafluoren-9-one (scheme 1), as well as the modified 1,10-phenanthroline neutral ligand, have allured interest in electrochemical, DNA intercalation and biological properties. Because of the larger bite distance (2.99 Å) between two coordination nitrogen atoms, 4,5-diazafluoren-9-one provides an uncommon coordination environment, yielding a number of coordination compounds. Metal complexes of this ligand potentially allow for the variation and control of redox properties over a wide range as well as the fine tuning of potentials through pH changes [1-3]. The Ni(II) complex, [Ni(phen)₂(dazf)](PF₆)₂. Where phen is 1,10-phenanthroline and dazf is 4,5-diazafluoren-9-one has been prepared and characterized by elemental analysis, cyclic voltammetry, IR, ¹H NMR and electronic absorption spectroscopies. Cyclic voltammograms were recorded by using a Micro Autolab Type III. Cyclic voltammetry of this complex was performed on an acetonitrile solution with 0.1 M TBAH as a supporting electrolyte. The Ni(III/II) couple was observed at 1.70 V versus SCE. The dazf ligand is electrochemically active due to the presence of the o-quinone moiety. In this complex a reversible reduction couples at -0.73 V and -1.90 are assigned to the reduction of dazf (dazf^{0/-}) and phen ligands, respectively, by analogy to other complexes. The order of ligand reductions is indicative of the relative π-acceptor orbital energies of each ligand. Due to o-quinone moiety of dazf, a shift to more positive potentials is observed than for phenanthroline ligands.



scheme 1.

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Determination of activity coefficient of NiCl_2 in Triton x-100/water mixed solvent by using potentiometric method at $T=298.15\text{ K}$

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Abstract

During the last decades, the series of ion-interaction models for electrolyte solution have been proposed to predict the activity coefficient of each solute and osmotic coefficient of aqueous systems. Among them, the Pitzer ion-interaction model is one of the most famous and useful models. Pitzer equations, with much accuracy, correlate the thermodynamic properties of mixed aqueous electrolyte systems. The Pitzer model extended the Debye-Hückel method with a virial expansion to account for the ionic strength dependence of the binary repulsive short-range forces, and higher order interactions [1, 2]. The properties of electrolyte in mixed water-organic solvents are of particular interest in many industrial and environmental applications, particularly, for separation processes. Apart from laboratory use, Triton X-100 can be found in several types of cleaning compounds, ranging from heavy-duty industrial products to gentle detergents. It is also a popular ingredient in homemade vinyl record cleaning fluids together with distilled water and isopropyl alcohol. It is a good micellar catalyst. To measure the thermodynamic properties of electrolytes in mixed solvent, the most common methods are the isopiestic vapor pressure [3] and potentiometric techniques. In this research, the mean activity coefficient of NiCl_2 in surfactant-water mixed solvent system were determined by using the potentiometric technique at $T=298.15\text{ K}$. The measurements were carried out on the galvanic cell without liquid junction of the type: $\text{Ni-ISE} \mid \text{NiCl}_2 (\text{m}), \text{Triton x-100 (w\%)}, \text{H}_2\text{O (100-w)} \mid \text{Ag-AgCl}$ over total ionic-strengths from 0.001 to 6.00 mol.kg^{-1} for different mass fraction of Triton ($w = 0, 1, 2.5, 5, 7.5$ and 10%). The PVC based nickel ion selective electrode (Ni-ISE) and Ag-AgCl electrode used in this work were prepared in our laboratory and had a reasonably good Nernst response. The Pitzer ion-interaction parameters (β^0 , β^1 and C) for the series system were estimated by fitting experimental results with the Pitzer model. Then, the parameters obtained with the Pitzer model were used to calculate the values of the osmotic coefficients and the excess Gibbs free energies (G^E) for the whole series of the studied mixed solvent electrolyte systems.

Keywords: Activity coefficient; Pitzer model; NiCl_2 ; Triton x-100; emf technique

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Non-enzymatic glucose sensor based on disposable pencil graphite electrode modified by copper nanoparticles

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Abstract

Non-enzymatic glucose sensor based on disposable pencil graphite electrode (PGE) modified by copper nanoparticles (Cu(NP)) was prepared for the first time. The prepared Cu(NP) exhibited an absorption peak centered at ~562 nm by UV-Vis spectrophotometry, and almost homogenous spherical shape by scanning electron microscopy (SEM). Cyclic voltammetry of Cu(NP)-PGE showed an adsorption controlled charge transfer process up to 90.0 mVs⁻¹. The sensor was applied for determination of glucose by amperometry technique with detection limit of [0.44(±0.01) μM] and concentration sensitivity of [1467.5 (±1.3) μA mM⁻¹cm⁻²]. The preparation of Cu(NP)-PGE sensor was reproducible (RSD=2.10%, n=10), very simple, fast and inexpensive, and the Cu(NP)-PGE was meant to be used as disposable glucose sensor.

Keywords: Glucose sensor, Copper nanoparticle, Pencil graphite

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Study of electropolymerization of 4-nitroaniline and its analytical application

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Abstract

Recently much attention has been paid to electrochemical investigations of mono- substituted benzenes and substituted anilines by both electrochemical and spectro-electrochemical techniques [1]. 4-nitroaniline is a member of family of isomeric nitroanilines has attracted much attention because of specific effects of an electron withdrawing nitro group and an electron-donating amino group being in the para position of an aromatic ring system [2]. 4-nitroaniline could be reduced electrochemically in acidic and alkali media [3]. In this work 4-nitroaniline was polymerized in sodium acetate medium on a lead cathode in a separated electrochemical cell under constant current condition. The electrode-product was dark-brown powder, insoluble in water, but soluble in DMSO and Formic acid. The electrode-product was characterized by FT-IR, DSC, CV and Mass spectrometry. A suitable structure and a mechanism for electrode-reaction were suggested. The prepared polymer was used as a sorbent in solid phase extraction for separation and preconcentration of Cu^{2+} , Ni^{2+} , Co^{2+} following determination by Flame Atomic Absorption Spectrometry. The proposed method was applied for determination of above mentioned cations in some lagoons South of Caspian Sea.

Keywords: electropolymerization, 4-nitroaniline, Solid phase extraction.

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The novel carbon paste electrode modified with sulfonated-montmorillonite to measure copper ion in the aqueous solution

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Abstract

Copper is the third most used metal in the world. It is present in all organisms, land and marine, so the determination of copper ion becomes important. A number of methods such as flame atomic absorption spectrometry, anodic stripping voltammetry, ion chromatography, UV-Vis spectrometry are used for copper ion determination at low concentration level. Potentiometric measurements with copper ion-selective electrode provide analytical procedures for aqueous samples as they are fast and convenient [1]. In this work the carbon paste electrode modified with sulfonated-montmorillonite was prepared and used as copper ion-selective electrode. The influence of various parameters on the electrode response such as pH, carbon paste composition, detection limit, response time and selectivity was investigated. Results after optimization, showed almost Nernstian behavior in the 1.0×10^{-9} - 1.0×10^{-1} M concentration range with a slope of 20 ± 1 mV per decade change in concentration. A detection limit of 5.8×10^{-10} was estimated. The response time of the electrode was 10 s. The pH dependence has been tested in the range 2.0-7.0 of 1.0×10^{-3} M Cu (II) and the optimum pH was found to be 4.0. During the experiment selectivity coefficients for various interfering ions were estimated. This chemically modified electrode with low response time, low detection limit, wide working concentration range and high selectivity toward Cu (II) will offer a possibility of developing a sensitive method for the determination of copper cation in solutions [2].

Keywords: Sulfonated-montmorillonite, Chemically modified electrode, Copper ion, Aqueous solution

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Electrochemical Synthesis of New Benzothiazole-benzene-1,2-diol derivatives

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Abstract

Electroorganic synthesis has become recognized as a powerful tool to develop environmentally compatible processes [1]. Benzothiazole derivatives have exhibited interesting biological activities and attracted continuing interest for further molecular exploration as useful anticancer agents [2]. Also, many of catechols such as catecholamines are biologically active compounds [3]. Because of the medicinal properties of benzothiazole and catechol derivatives, we thought that synthesis of new organic compounds with both catechol and benzothiazole moieties may be useful from the point of view of pharmaceutical properties. The electrochemical oxidation of some catechols have been studied in the presence of benzothiazole-2-thiol as a nucleophile in a water/acetonitrile mixture using cyclic voltammetry and controlled-potential coulometry. The voltammetric data show that electrochemically generated *ortho*-benzoquinones participate in Michael addition reactions with benzothiazole-2-thiol to form the corresponding benzothiazole-benzene-1, 2-diol derivatives. In this work, we have proposed an EC mechanism for the electrode process and we report an efficient and one-pot method for the synthesis of benzothiazole-benzene-1, 2-diol derivatives in an undivided cell at a carbon rod electrode.

Keywords: Benzothiazole-2-thiol, Catechol, Cyclic voltammetry, Michael addition reaction.

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A barium (II) selective polyvinyl chloride membrane sensor based on a benzo -15-crown-5

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Abstract

Barium(II)-selective sensors have been fabricated from poly vinyl chloride(PVC) matrix membranes containing neutral carrier benzo-15-crown-5 as electroactive material, sodium tetrphenylborate (NaTPB) as an anion excluder and dibutyl phthalate as plasticizing. The membrane having the composition of crown ether: NaTPB:DBP:PVC in the ratio 5:2:60:43 (w/w%) that could be used for the estimation of Ba²⁺ ions with a Nernstian slope (29.1 mV/decade of activity) and suitable for use in aqueous solution in the pH range of 2.0–6.0. It shows wider working activity range (1.0×10^{-6} to 1.0×10^{-1} M), with a limit of detection 0.6×10^{-5} M, low response time (20 s) and better selectivity.

Keywords: PVC-membrane, Ba (II) ion-selective electrode, diaza-18-crown-6

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Direct Determination of Trace Amounts of Aluminum Ions by A New Polymeric Membrane Electrode

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Abstract

Aluminum is one of most widely used metal at the present time and is present in various environment samples where its determination becomes important in view of its toxic effects. Many studies have focused on the toxicity of aluminum on living systems, showing that aluminum toxicity have a great impact on plants growth in acidic soil¹ and excessive aluminum can result in a great deal of fishes die.² In this work a highly selective PVC membrane electrode for Al^{3+} based on new synthesized macromolecule include Nitrogen atoms as a novel carrier has been prepared and studied. The sensor exhibits a good response for Al^{3+} over a linear range of 2.0×10^{-7} to 1.0×10^{-1} mol/L, with a Nernstian slope of 19.5 ± 0.2 mV/decade and detection limit of 9.5×10^{-8} mol/L. Selectivity coefficients determined by the method of separate solution³ indicate high selectivity for Al^{3+} . It was used as an indicator electrode in potentiometric titration of Al^{3+} with EDTA and in the determination of Al^{3+} in real samples. The electrode has a relatively fast response time, long life time and satisfactory stability.

Keywords: Aluminum, Polymeric membrane electrode, Potentiometry

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A conductometric study of complexation reaction between benzo-15- crown-5 and Al^{3+} cation in some pure and binary mixed non-aqueous solvents

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Abstract

The complexation reaction between the macrocyclic ligand, benzo-15-crown-5 (B15C5) with Al^{3+} cation was studied in pure methanol (MeOH), propanol (Pr), methylacetate (MeOAc), ethylacetate (EtOAc) and their binary mixed solvents with acetonitrile (AN) at different temperatures using electrical conductometry method [1]. The conductance data show that the stoichiometry of complex formed between B15C5 and Al^{3+} in all solvent systems is 1:1 (ML). The value of formation constant of the complex was determined from computer fitting of conductance versus mole ratio data using a GENPLOT computer programme. In most cases, a non-linear relationship was observed between the stability constant ($\log K_f$) of the complex and the composition of the binary mixed solvent solutions [2, 3]. The values of the standard thermodynamic parameters ($\Delta H^\circ, \Delta S^\circ$) for formation of $(\text{B15C5} \cdot \text{Al})^{3+}$ complex obtained from the temperature dependence of the formation constant of the complex using the van't Hoff plots and the data show that the standard thermodynamic quantities change with the composition of the binary solvent solutions.

Keywords: Conductometry, Al^{3+} , B15C5.

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Simultaneous electrochemical determination of lidocaine and prilocaine in pharmaceutical preparations using multivariate calibration methods

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Abstract

In this research a simple, rapid and environment friendly method for simultaneous determination of two anesthetic drugs, Lidocaine Hydrochloride (LH) and Prilocaine Hydrochloride (PL) in pharmaceutical preparations by square wave voltammetry (SWV) using laboratory constructed carbon paste electrode (CPE), has been presented. The linear calibration graphs for LH and PL were in the concentration range of 2-120 $\mu\text{g mL}^{-1}$ and 5-25 $\mu\text{g mL}^{-1}$, respectively. The obtained equations for LH and PL were $(\Delta i) = 0.314 C_{\text{LH}} - 0.748$ ($r = 0.998$) and $(\Delta i) = 0.079 C_{\text{PL}} - 0.262$ ($r = 0.996$), respectively. Partial least squares regression (PLS) was applied to resolve the seriously overlapped voltammograms without any pre-separation step. The five- level partial factorial design was used as calibration design method and the cross-validation method was used to select the number of significant factor for PLS model building. Seven and eleven significant PLS components were obtained for LH and PL, respectively. A set of synthetic sample mixtures were used to validate the proposed method. The root-mean-square error of calibration (RMSEC) and root-mean-square error of prediction (RMSEP) were (1.31 and 0.98) and (1.04 and 0.89) for LH and PL, respectively. The developed method was then applied to the analysis of these two compounds in pharmaceutical formulation samples with satisfactory results.

Keywords: Lidocaine, Prilocaine, Square-wave voltammetry, Partial least squares, Simultaneous determination.

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Square wave voltammetric determination of prilocaine in pharmaceutical preparations using carbon paste electrode

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Abstract

In this work a new voltammetric method procedure for determination of Prilocaine hydrochloride was described. Square wave voltammogram of (PLH) solution using carbon paste electrode showed a well-defined peak between at +0.80 V. In this method instrumental and chemical parameters such as pH and scan rate influencing on voltammetric response were optimized. The results showed that at pH 10.5 and scan rate of 0.25 Vs⁻¹, maximum peak current with normal shape could be obtained. Under optimum experimental conditions the peak current is linear to PLH concentration over the range of 1 – 25 µg mL⁻¹ with a correlation coefficient of 0.9960. The limit of detection (LOD) and limit of quantification (LOQ) for PLH based on three and ten times the standard deviation of the blank (3Sb,10Sb) were 0.3 and 0.8 µg mL⁻¹ (n = 10) for PLH, respectively. RSD% 2.8 obtained for 5 µg mL⁻¹ solution of PLH indicates reasonable reproducibility of the method. In this work, a simple, economic and environmentally friend electrode for determination of PLH in pharmaceutical preparations, was fabricated. The capability of this proposed method was established by application of the method for determination of PLH in injection preparations.

Keywords: Prilocaine Hydrochloride, carbon paste electrode, Square wave voltammetry, Pharmaceutical preparations.

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Simultaneous determination of salicylic acid and gallic acid using a modified electrode based on multiwall carbon nanotube

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Abstract

Salicylic acid (SA) is a beta hydroxy acid used in organic synthesis and functions as a plant hormone. It is derived from the metabolism of salicin. SA is the main metabolite of acetylsalicylic acid, that is, one of the drugs most widely used in the world as a painkiller and anti-inflammatory, but banned from veterinary therapeutic treatment [1]. Gallic acid (GA) acts as a free molecule or as part of a tannin molecule and is found in almost all plants. GA has been shown anti-fungal and anti-viral properties. Also, GA acts as an antioxidant and helps to protect our cells against oxidative damage [2]. Carbon nanotubes (CNTs) have attracted considerable attention due to their extraordinary structural, mechanical, electrical, and electrochemical properties as well as their promise in the field of materials science. Several types of CNT electrodes have been reported, including CNT paste electrodes, CNT film-coated electrodes, CNT powder microelectrodes and CNT paper electrodes [3]. In this paper, a modified carbon paste electrode based on multiwall carbon nanotube (MWCNT/CPE) was designed and applied for the sensitive and simultaneous determination of SA and GA. For surface investigation of the modified electrode, scanning electron microscopy (SEM), electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV) techniques were used. Differential pulse voltammograms of the two analytes showed enhanced redox responses for the MWCNT/CPE with respect to the bare CPE. Also, electrochemical behavior of SA and GA was investigated by various electrochemical techniques such as CV, chronoamperometry, chronocoulometry and linear sweep voltammetry. Under the optimum experimental conditions a broad linear dynamic range with low limit of detection was obtained for the two analytes. Also, for further evolution of the validity of the proposed method, simultaneous determination of SA and GA was performed in biological samples with good recovery.

Keywords: Salicylic acid, Gallic acid, Voltammetry, Carbon nanotubes

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Boron electrodeposition from KCl–KF–KBF₄ melts; a process for electrolytically production

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Abstract

The aim of this work was synthesis of elemental boron with the high purity in a cost effective method. Boron is an element which due to its unique properties such as low density, high melting point and a hardness like that of diamond, has wide applications in many industries, including a medicine, Metal Industry, neutron adsorption,.... The preparation of elemental boron in pure form is somewhat difficult and the properties of the obtained boron are influenced by the preparative technique. The world prices of elemental boron with high purity is very expensive. Therefore, preparation of boron in pure form is very important. One of the ways of producing high purity of boron is the electrochemical reduction of boron-containing compounds in molten salt baths [1]. In this research, Elemental boron was produced through Electrolytic deposition processes from molten salt bath consist of potassium fluoroborate was dissolved in a mixture of molten potassium fluoride and potassium chloride. The experimental apparatus for the boron electrodeposition include of Electric furnace to melt the mixture of salts, a electro-winning cell consisted of external enclosure made out of titanium was equipped with a system for neutralizing the ambient atmosphere, A high density graphite crucible was placed inside this vessel also as an anode, steel rod for cathode and Rectifiers was used to supply DC current. Elemental boron was electrodeposited from a near-saturated solution of KBF₄ in a molten mixture of KCl and KF held at 1060K, by the application of a potential (2 to 4V) for 3 hours under argon atmosphere. The final product was prepared after acid leaching and washing processes. Characteristics of the boron powder were studied with several analysis. The purities of the powders were determined with EDS and ICP. The morphology and particle size of the deposits was examined using scanning electron microscopy (SEM). The results showed that elemental boron powder with a purity of 93–96% can be produced using a high temperature in the molten salt electrolysis process. Also, the particle size of the purified elemental boron was about 1 to 4 micron.

Keywords: Electrodeposition, Molten salt electrolysis, Elemental boron

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Investigation of electro/persulfate process efficiency in removing furfural from aqueous solution by iron electrode

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Abstract

In order to generate sulfate radical ($\text{SO}_4^{\cdot-}$), persulfate ($\text{S}_2\text{O}_8^{2-}$, PS) is often activated by heat, UV irradiation and intermediate metals like iron ions. The objective of this study was to investigate the efficiency of activated $\text{S}_2\text{O}_8^{2-}$ by the electrically produced iron using the iron electrode (modern process of electro/persulfate) for furfural removal. In this study, a lab-scale batch reactor equipped to 4 electrodes and direct power source to was used to remove furfural. The effects of key parameters such as pH, voltage, initial concentration of $\text{S}_2\text{O}_8^{2-}$ and furfural on the process were studied. Furthermore, the amounts of pH changes during the process as well as the effect of the optimized situation on the efficiency were investigated. The findings showed that initial pH, $\text{S}_2\text{O}_8^{2-}$ initial concentration and voltage played an important role in furfural removal. Under acidic situations, an increase in the efficiency was seen, and by raising pH the amount of furfural removal decreased. 98% of furfural was removed in 60 min (pH=3, voltage=10 V, $\text{S}_2\text{O}_8^{2-}$ concentration=7 mM). When the concentration of furfural was raised, the efficiency declined. At pH=5, 7, 9 and 11 after 60 min, the efficiencies were 56, 53, 50 and 10, respectively. By increasing voltage by 10 V, the highest amount of efficiency was seen. By applying $\text{S}_2\text{O}_8^{2-}$ in a separate approach after 60 min the efficiency was 39% while at these conditions, the efficiency of the electrochemical process was only 28%. By contrast, the contrast could remove 98% of furfural. The use of $\text{S}_2\text{O}_8^{2-}$ ion in electrochemical reactors equipped to iron electrode can increase the efficiency of the process. Thus, the application of this novel process can be hopeful for industrial wastewater plants operators.

Keywords: Sulfate radical, Persulfate, Electro/persulfate, Iron electrode, Furfural

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Smart corrosion protection of carbon steel by electrodeposited polyaniline-polypyrrole coating

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Abstract

In this study a smart coating based on polyaniline (PA)-polypyrrole (PPY) composite was electrodeposited by cyclic voltammetry to protect carbon steel oil pipes (API X-52). Because of carbon steel sensitivity to acidic solutions [1], in order to achieve the best coating, effects of various organic and inorganic acids were investigated in electropolymerization of pyrrole and aniline monomers. Other experimental factors such as concentration of substrates, potential window and potential scan rate, were also studied and then optimized. These factors can determine the nature and properties of deposited layer [2]. Due to different characteristics of PA and PPY layers, their deposition order was examined using three coating systems including: PY/PA, PA/PY and simultaneous coating of both monomers. Finally in order to evaluate protection performance of coatings their open circuit potential (OCP) values were monitored during a linear polarization process. Electrochemical impedance spectroscopy (EIS) was also used to evaluate different coatings. All electrochemical tests were carried out in NaCl 3.5% solution. The results showed that simultaneous electrodeposition of pyrrole and aniline in a weak organic acid solution leads to a coating with better performance. That is due to formation of weak acid oxide layer on electrode surface which prevents iron dissolution and improves deposition yield [3]. Optimized values of other experimental factors are -0.5 to 1.6 V for potential window, 20 mVs⁻¹ for scan rate. Concentration of aniline, pyrrole and oxalic acid were also optimized.

Keywords: Corrosion protection, Polyaniline, Polypyrrole, Electrodeposition, Carbon steel

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Sensitive Amperometric TNT Sensor Based on Glassy Carbon Modified Electrode with Graphene Sheets and Polyoxometalate Complex

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Abstract

The detection of nitroaromatic explosive compounds such as 2, 4, 6 trinitrotoluene (TNT) has received considerable attention in connection with various security and environmental needs. Electrochemical measurements of TNT are usually carried out at various carbon electrodes. In this work the sensitive TNT sensor was fabricated based on modified with graphene and Ir-substituted dawson type polyoxometalate complex (GC/Gox/POM). At first electrochemical behavior of GC/Gox/POM was studied at various pHs (1-5). The result indicated that the GC/Gox/POM has good stability in acidic media. The electrocatalytic activity of modified electrode toward TNT was evaluated by cyclic voltammetry. The results imply to excellent electrocatalytic activity of modified electrode in electroreduction of TNT in pH 1 buffer solution and -0.17 V versus Ag/AgCl reference electrode. The electrochemical reduction TNT was investigated by cyclic voltammetry on a glassy electrode in aqueous phosphate buffer solution with different concentrations of TNT. In order archiving the best analytical parameters, the hydrodynamic amperometric techniques was used. The amperometric detection of TNT is carried out at -0.2 versus Ag/AgCl reference electrode in phosphate buffer solution with pH 1. The results show low detection limit, good sensitivity and wide dynamic range in detection of TNT. Based on the our studies the GC/Gox/POM have many advantages such as remarkable catalytic activity, good reproducibility, simple preparation procedure and long term stability of signal response during TNT reduction.

Keywords: Trinitrotoluene- amperometric, polyoxometalate

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Preparation of nickel hydroxide-activated carbon nanocomposite carbon paste electrode and its application for determination of cyanide ion in aqueous solutions

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Abstract

The composite electrode has good electrochemical performance and high charge-discharge properties. To take full advantage of the double-layer capacitance and pseudo- capacitance, many metal oxide/carbon composite electrode has been investigated for electrochemical capacitors (ECs)[1]. Based on electrical double-layer capacitance, activated carbon is very suitable for ECs, and has good conductivity, high surface area, excellent temperature stability and a relatively low cost [2]. In the present work, a nickel hydroxide carbon active (AC) nanocomposite electrode for use in an electrochemical capacitor was prepared by a simple chemical precipitation method. The structure and morphology of nickel hydroxide/(AC) were characterized by XRD and SEM. The development and improvement of cyanide sensors based on voltammetry and especially amperometry has been developed. The obtained result showed a electrocatalytic oxidation of cyanide through a EC' mechanism using a carbon paste electrode modified by nickel hydroxide carbon active nanocomposite. The proposed voltammetric method showed a sensitive response toward cyanide ions in alkaline medium. The obtained calibration curves was linear in the concentration range of 1.8×10^{-7} - 2×10^{-5} M with a limit of detection 5×10^{-8} M. The proposed method has been successfully applied for the determination of cyanide in various water samples.

Keywords: Nickel hydroxide-activated carbon nanocomposite, Cyanide, Carbon paste electrode.

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Determination of activity coefficients for CaCl_2 in methanol/water mixed solvents by potentiometric measurements

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Abstract

In many industrial and environmental processes, modeling the thermodynamic properties of various electrolytes in water–organic solvent mixtures is of particular interest. In this respect, although the potentiometric method, like the isopiestic method, is a useful experimental technique for studying the thermodynamic properties of electrolyte solutions, nevertheless, the former method benefits from further advantages such as rapidity and possibility of measurements in a wider concentration range [1, 2]. In the last decades, a series of ion-interaction models for electrolyte solution have been proposed to predict activity coefficient of each solute and osmotic coefficient of aqueous systems. One of the most famous and useful models is that proposed by Pitzer [3]. This model is relatively simple, and it performs electrolyte calculations with accuracy, especially for concentrations under 6 molal. The mean activity coefficient of calcium chloride were determined on the galvanic cell of the type: $\text{Ag}|\text{AgCl}||\text{CaCl}_2(\text{m}), \text{methanol}(\text{wt.}\%), \text{H}_2\text{O}(1-\text{wt.}\%)||\text{Ca}^{2+}\text{-ISE}$, in various mixed solvent systems containing 0, 10, 20, 30, 40 and 50% mass fractions of methanol over ionic strength ranging from 0.0010 to 6.0000 kgmol^{-1} . The resulting values of the mean activity coefficients were correlated with Pitzer ion interaction model and Pitzer ion-interaction parameters ($\beta^{(0)}$, $\beta^{(1)}$ and C^ϕ) were determined. Then these parameters used for calculating of the osmotic coefficients and the excess Gibbs free energy for the series under investigated system.

Keywords: Activity coefficients, Pitzer model, Potentiometric method, Methanol, CaCl_2

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Plasmonic behavior of bowtie plasmonic nano-structures

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Abstract

The interaction between the incident electric field and conduction band electrons of metal in metallic nanoparticles such as gold and silver nanoparticles leads to strong absorption and scattering cross sections in visible and NIR part of the spectrum [1]. Due to the optical properties of metallic nanostructures, they are very interesting for different applications such as biological sensing [2], solar cell optimization, field enhancement spectroscopy, cancer therapy and biological applications [3]. Localized surface plasmon resonance in metallic nano-structures when light incident to these nano-structures causes concentration of electromagnetic energy, so they can be used as optical nano-antenna. In this paper, we investigate absorption, scattering and extinction cross section of triangular nano-structures, also we investigate the effect of coupling between triangular nano-structures in bowtie nano-antenna. Our simulation results show that different parameters can influence on optical properties (plasmon resonance wavelength, absorption, scattering and extinction cross section) of bowtie nano-antennas such as the distance between different parts. So we investigate the effect of gap distance between two triangular nano-structure, material of metallic nano-structures and surrounded dielectric constant on optical properties of coupled bowtie nano antenna. We obtain optical properties of bowtie nano-antennas using boundary element method (BEM) by solving full Maxwell's equation. BEM approach is a 2-dimension and fast numerical method that use Green's function for solving Helmholtz equations. We validate our BEM approach by comparison of our results with Mie theory for spherical NPs.

Keywords: Plasmon resonance, Metallic nano-structures, Optical properties, Bowtie nano-antenna

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Preparation of coating based on organic-inorganic silanes by sol-gel method on mild steel and study of its corrosion properties

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Abstract

In this work sol-gel film deposited onto St-12 mild steel by dip-coating technique from TEOS (tetraethyl orthosilicate), MTES (methyl triethoxysilane) and organic-inorganic hybrid of their precursor solutions [1]. Heating rate in the sintering step was 3 °C/min [2]. Morphology analysis of the coatings was carried out by scanning electron microscopy (SEM) and corrosion analysis of the coated and uncoated samples in 3.5% NaCl were measured by electrochemical impedance spectroscopy (EIS). Comparison of the corrosion and morphology results indicates that the coatings prepared from MTES and TEOS exhibits better hydrophobicity and smooth film respectively. The hybrid coating properties were a combination of hydrophobicity and corrosion resistivity with respect to the un-hybridized films.

Keywords: TEOS, MTES, Hybrid, SEM, EIS, Sol-gel

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Study of physical properties of TiC nanowires using quantum concepts

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Abstract

We study physical properties of TiC nanowires have been investigated through density functional theory (DFT) [1, 2], the full potential augmented plane wave plus local orbital method (FLAPW + lo), using Wein2k package. Infinite and free standing nanowires in periodic positions were simulated by super cell approach and the optimized structure and bond length were calculated. Young's modulus (E) of TiC nanowire is obtained by the generalized gradient approximation (GGA) and bulk modulus (B) using phonopy package. Heat capacity at constant pressure C_p at temperatures (0-1000K) were calculated by phonopy package. According to our results, Young's modulus and Bulk modulus of TiCNWs are 4.09 and 4.78 times bigger than those of bulk TiC, respectively. The Increase in Young's modulus, Bulk modulus and shear modulus is due to bonding of Ti atoms to each other in relaxed structure. C_p of TiCNW increase with temperature; but at a specific temperature, C_p of TiC nanowire is less than those of bulk TiC.

Keywords: TiC nanowire, DFT, Heat capacity at constant pressure

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Application and characterization of electroless Co-P coating on AZ31 magnesium alloy via an environmentally friendly pretreatment

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Abstract

Magnesium and its alloys are light-weight materials with high strength to weight ratio and have many applications especially in aerospace and automobile industries. Unfortunately, the magnesium alloys have poor corrosion resistance, and then their anticorrosion performance should be strongly improved before any outdoor application. Coating application is one of the most effective methods to protect metals from corrosion. Application of uniform, dense, protective and hard coating on magnesium alloys is so hard due to high affinity of magnesium to aqueous plating solutions and also due to electrochemical heterogeneity of alloy surface. Therefore, it is necessary to treat the alloy surface with appropriate conversion coating before any final plating. In this paper Cerium–Lanthanum–Permanganate (CLP) conversion coating was firstly applied on the AZ31 alloy as environmentally friendly pretreatment and the then final electroless Co–P coating was deposited from a plating solution containing CoSO_4 , NaHPO_2 , H_3BO_3 , HF , $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$, NH_4HF_2 , Thiourea and NaOH . The morphology and cross-section images of coated samples were observed by Scanning Electron Microscopy (SEM). Moreover the microstructure and chemical composition of the deposits were investigated by X-ray diffraction (XRD) and Energy Dispersive X-ray spectroscopy (EDS) respectively. Also, the anticorrosion properties of the applied coating was studied by Electrochemical Impedance Spectroscopy (EIS) and potentiodynamic polarization methods in 3.5 wt.% NaCl corrosive solution. The results showed the formation of uniform, dense and high crystalline Co–P electroless coating with excellent anticorrosion characteristics.

Keywords: Electroless plating, Magnesium alloy, Corrosion, Protection

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Investigation of the activation mechanism of the electroless Co-P plating by Cerium-Lanthanum-Permanganate conversion treatment

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Abstract

One of the most important limitations of magnesium alloys is their high chemical and electrochemical reactivity which make them prone to corrosion in a set of corrosive environments such as humid air and water. Electroless plating is an appropriate procedure to apply uniform and protective coatings on magnesium alloys. Unfortunately, the application of electroless coating on magnesium alloys is difficult because these alloys have the chemical affinity to aqueous solutions. It is well-known that the electroless plating process should be activated in order to produce the metallic nuclei on the alloy surface. Generally, the substrates are activated by acidic solutions of SnCl_2 and PdCl_2 respectively. In the case of the magnesium alloy, the use of these chloride salts containing solutions is impossible due to high corrosion rate of the magnesium in Sn^{+2} and Pd^{2+} containing media. Therefore an especial pretreatment is necessary to activate the plating process and also in order to minimize the chemical affinity of the magnesium substrate before any successful coating application. The aim of this work is to investigate the activation mechanism of electroless Co-P plating process by application of Cerium-Lanthanum-Permanganate conversion coating as a new, cheap and environmentally friendly procedure. The results showed that the electroless Co-P process can be activated by CLP treatment. The activation mechanism has been investigated by Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS). The SEM images showed that the CLP conversion coating is a porous film and then the replacement reaction take place between the magnesium at the bottom of pore and the electrochemical noble Co^{+2} ions in the plating solution in order to the formation of Co nuclei at the bottom of the pores. SEM images and also the EDS analysis after 10 and 30 min of electroless process clearly showed that the Co nuclei form only at the edge of the pores more probably due to evolution of hydrogen gas bubbles (by reduction of dissolved oxygen) at the center of the pores. After the initial nucleation, the nuclei extend in vertical and horizontal directions to completely cover the alloy surface.

Keywords: Electroless, Co-P, Activation, Mechanism, Conversion coating

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Thermodynamic study of electrolyte mixture (NaCl + NaNO₃ + water + urea) by potentiometric measurements at T = 298.2

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Abstract

In this work, the thermodynamic properties of the mixed electrolyte (NaCl + NaNO₃ + water + urea) system were determined using the potentiometric method at 298.2 K. The potentiometric measurements were performed on the galvanic cells without liquid junction of the type: Ag–AgCl|NaCl (m₁), NaNO₃ (m₂), (100-wt%) H₂O + wt% urea|Na-ISE and NO₃-ISE|NaNO₃ (m₁), (100-wt%) H₂O + wt% urea|Na-ISE over total ionic strengths from 0.01 to 3.000 mol kg⁻¹ for different series of salt molal ratios r ($r = m_{\text{NaCl}}/m_{\text{NaNO}_3} = 0.1, 1, 2.5, 5.0, 7.5, 10.0$) and pure NaCl in mixed solvent system containing 10% mass fraction of urea. The PVC based sodium and nitrate ion selective electrodes (Na, NO₃-ISE) and Ag–AgCl electrode used in this work were prepared in our laboratory and had a reasonably Nernst response. The activity coefficients data were interpreted based on the Harned rule and Pitzer model. The Pitzer binary and ternary interaction coefficients (θ_{ClNO_3} , ψ_{NaClNO_3}) were evaluated for the studied mixed electrolyte system according to Pitzer graphical method. Then, the parameters obtained with the Pitzer model were used to calculate the values of the mean activity coefficients of NaNO₃, the osmotic coefficients, and the excess Gibbs free energies for the whole series of the studied mixed electrolyte system.

Keywords: Potentiometry, Activity coefficients, Pitzer model, Ion selective electrode.

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Electrochromic properties of composite thin film incorporation polyaniline and magnesium oxide

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Abstract

Since recent decades, many studies have been carried out on conducting polymers (CPs). This class of novel solid-state materials with extended π -conjugation along the polymer backbone have indicated electrochromic and semiconducting behavior [1]. Much effort is devoted to research on such systems, because of many potential applications in various fields such as anti-static coatings, organic light emitting diodes, batteries, biosensors, touch screens and electrochromic devices. In this study, a simple and economic method is used for the production of a new composite film based on aniline in the presence of magnesium oxide by electrodeposition on ITO-coated glass electrodes. Electrochemical and electrochromic behaviors of thin films were examined by cyclic voltammetry (CV). UV-Vis spectrophotometer was used to study the optical transmittance of thin films. The doped films indicated multiple color changes (yellow-green- bluish green). The optical switches were fast and fully reversible. The spectra also showed that produced layer has high absorption of UV radiation towards pure polyaniline (PANi) films. Because of its good transparency, electrochromic behavior and adhesion, it can be suggested as a promising material for electrochromic devices.

Keywords: Electrochromic, Polyaniline, Magnesium oxide, Thin film

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A survey upon ethanol electrooxidation on Ni with lanthanum oxide as cocatalyst in a DAFC

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Abstract

It has been shown that lanthanum oxides can substantially modify the chemical behaviour of highly dispersed metal catalysts [1]. In this system, several chemical species are present such as La_2O_3 or $\text{La}(\text{OH})_3$, which could be implied in the electro oxidation of alcohols like methanol or ethanol, for example. In the past decades, direct alcohol fuel cells (DAFCs) have received much attention due to their possible applications in transportation and portable electronic devices. Methanol or ethanol can be directly used as fuel in DAFCs without external reformer. Ethanol has higher energy density compared with methanol and it is more attractive as fuel for DAFCs. It is accepted that the coexistence of some metal oxides with Ni can improve the catalytic activity of Ni-based catalysts for this reaction. Electrocatalytic activity toward ethanol electrooxidation of Ni particles in NiLa/C catalysts with different Ni:La ratios has been studied with different electrochemical and spectroscopic techniques, and the results were compared to those of Ni/C catalyst. Significant enhancement in the electrocatalytic activity has been achieved by depositing the Ni particles with lanthanum oxides/hydroxides using an alcohol reduction method. Compared to Ni/C catalyst, NiLa/C materials exhibit a lower onset potential and a higher electron-transfer rate constant for the investigated reaction. These studies illustrate the possibility of utilizing Ni/C with La oxides/hydroxides as electrocatalyst for direct alcohol fuel cells (DAFCs). The combination of electrochemical and spectroscopic techniques has allowed a comparative analysis of the behavior of Ni/C, NiLa/C (40:60) and NiLa/C (20:80) electrocatalysts towards ethanol electrooxidation. A significant increase of performance was observed with the increment of La oxides/hydroxides content, indicating that the addition of La species improves the activity of Ni for this reaction. Also the formation of CO_2 is favoured if the La oxides/hydroxides content is raised to Ni:La 20:80, and, thus, the presence of these compounds as oxygen source can favor both bulk alcohol reactions (acetic acid formation) and adsorbed species oxidation (CO_2 production). The initiation for ethanol oxidation is observed at 0.50 V, that is, in the potential range used for a DAFC. The enhancement of activity towards alcohol electrooxidation in this potential region due to the addition of La oxides/hydroxides to Ni opens a possibility to utilize these materials as electrocatalysts for these devices [1, 2]. However, the detection of representative amounts of acetic acid clearly indicates that the C–C bond is not completely broken and further optimization of the catalysts is needed to improve the energy efficiency of ethanol electrooxidation.

Keywords: Lanthanum Oxide, Cocatalyst, DAFC, Electrooxidation

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Activity coefficient measurements of NaCl in (Urea+Water) mixtures using potentiometric method

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Abstract

This work consists of the results obtained by the activity coefficient measurements of NaCl in the mixed solvent system (water + urea) based on potentiometric method at $T = (303.2 \text{ and } 310.2) \text{ K}$. The potentiometric measurements were performed on the galvanic cell of the type: $\text{Ag}|\text{AgCl}|\text{NaCl (m), Urea (wt\%), H}_2\text{O (1-wt \%)}|\text{Na-ISE}$, in various mixed solvent systems containing 0, 10, 20 and 30% mass fractions of urea over ionic strength ranging from 0.001 to 3.250 kgmol^{-1} . Sodium ion selective electrode was made in laboratory with modified carbon nanotube as electrochemical sensor. The mean activity coefficients of NaCl in the mixed solvent (water + urea) were determined from the potentiometric measurements in according to Nernst equation. The Debye-Hückel and Pitzer ion-interaction model were used for the experimental data correlation and calculation of thermodynamic properties. At first, the Pitzer adjustable parameters ($\beta(0)$, $\beta(1)$ and $C\phi$) and Debye-Hückel parameters (a , c and d) were determined from experimental data. Then, the obtained parameters were used to calculate the values of the osmotic coefficients, solvent activity and the excess Gibbs free energies based on Pitzer model for the system under investigation. The results clearly indicate that a good correlation is obtained with the Pitzer model to describe this system.

Keywords: Activity coefficient, Potentiometric method, Modified carbon nanotube, Pitzer model

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Optical tunability of metallic ellipsoidal and spherical nanoparticles for nanosensing applications

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Abstract

In this work, we study the effect of light polarization, shape and surrounding dielectric constant on optical characteristics of Au-SiO₂ core-shell nanostructures by using boundary element method (BEM) to solve full Maxwell's equations. This method is a fast and suitable method for our analysis. We verify BEM approach with Mie-Gans theory for ellipsoidal nanoparticles (NPs). Also we compare the optical behavior of ellipsoidal and spherical NPs and show that ellipsoidal NPs are fully sensitive to incident light polarization direction unlike the spherical NPs. So ellipsoidal NPs are useful elements for nanosensing applications. In addition, ellipsoidal NPs with anisotropic shape have more tunable optical properties than spherical ones. Nanoscale interaction with incident light between the metal and semiconductor causes enhanced optical spectra and highly optical tunability in metal-semiconductor core-shell nanostructures. In experimental works has been shown that during the fabrication of larger sized NPs the sphericity is usually lost and particles become ellipsoidal; with the introduction of this anisotropy the resonance gets red shifted. The red shift is also observed when the surrounding medium is optically denser. The tunability of this resonance for example can be used to increase the absorption of thin-film Si solar cells in the near infrared domain where Si is a very poor absorber.

Keywords: Plasmonic, Metallic ellipsoidal nanoparticles, Core- shell nanostructures

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Investigation of Some Barbiturate derivatives as corrosion inhibitors for mild steel in acidic media

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Abstract

The use of acid solution during pickling and industrial cleaning leads to corrosive attack on mild steel. The corrosion of mild steel is of fundamental academic and industrial concern that has received a considerable amount of attention. Among efficient corrosion inhibitors use to prevent the deterioration of mild steel are heterocyclic organic compounds consisting of a π -system and/or O, N, or S heteroatoms [1]. Barbituric acid and its derivatives exhibit a wide range of biological activities such as antibacterial and anti-hypertensive agents. The corrosion inhibition efficiency of four Barbiturates (namely 2-((pyridin-2-ylimino)methyl)phenol (S1) and 2-((hexadecylimino)methyl)phenol (S2) and 2-((4-hydroxyphenylimino)methyl)phenol (S3) and 1-(4-(2-hydroxybenzylideneamino)phenyl)ethanone)) of carbon steel in hydrochloric acid have been shown through electrochemical impedance spectroscopy (EIS), Tafel polarization and weight loss, and found that all the examined Barbiturate compounds reduce the corrosion of carbon steel with the order of inhibition efficiency being $S2 > S3 > S1 > S4$ [2]. The effect of the molecular structure on the chemical reactivity has been subject of great interest in several disciplines of chemistry. The quantum chemical calculations have been widely used to study the reaction mechanisms and to interpret the experimental results as well as to solve chemical ambiguities. The correlation between the quantum chemical parameters and inhibition efficiency using quantitative structure activity relationship (QSAR) of these compounds was investigated using DFT/B3LYP and HF calculations. The inhibition efficiency of the inhibitor are closely related to the quantum chemical parameters, the highest occupied molecular orbital (EHOMO), energy of lowest unoccupied molecular orbital (E_{LUMO}), HOMO–LUMO energy gap (ΔE_{H-L}), the hardness (σ), the softness (η) and the fraction of electrons transferred (ΔN) for the neutral inhibitors and no significant relationship was found with parameters, dipole moment (μ) and the total energy (TE).

Keywords: DFT/B3LYP and HF, HOMO–LUMO energy gap (ΔE_{H-L}), The hardness (σ), The softness (η), Dipole moment (μ)

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Sonoelectrochemical synthesis of a new nanolead(II) complex with quinoline-2-carboxylic acid ligand: A precursor to produce pure phase nano-sized lead(II) oxide

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Abstract

A new complex, $[\text{Pb}(\text{Q})_2]$, (Q =quinoline-2-carboxylic acid) was prepared via both conventional electrochemical and sonoelectrochemical methods and compared their particle sizes. The complex was fully characterized based on its ^1H and ^{13}C NMR, IR, and UV spectra and elemental analysis. This new nano-structure was characterized by scanning electron microscopy (SEM), X-ray powder diffraction (XRD), IR spectroscopy and elemental analysis. Thermal stability of single crystalline and nano-size samples of the prepared compound was studied by thermal gravimetric (TG) and differential thermal analysis (DTA). The photoluminescence properties of the nanostructured and crystalline bulk of the prepared complex in the solid state have been investigated at room temperature. The prepared complexes, as bulk and as nanoparticles, were utilized as a precursor for preparation of PbO nanoparticles by direct thermal decomposition at 600 °C in air. The nano-structures of PbO were characterized by scanning electron microscopy (SEM), X-ray powder diffraction and IR spectroscopy. The nanostructure of the prepared complex was obtained by sonoelectrochemical process. In this work, we show that the emission wavelength of the lead complexes can be tuned in the solid-state over a wide range of the wavelength by changing in the size of the complex particles. The absorption and emission peaks of the crystalline bulk of the prepared complex are found to be significantly red shifted in comparison with those of the nanostructured of the prepared complex. Lead oxide nanoparticles were synthesized in a simple one-pot calcination of the nanostructured and crystalline bulk of the prepared complex. The morphology and size of the PbO nanoparticles were determined, and the results showed that particle size and morphology of the PbO nanoparticle depend on the initial particles size of $[\text{Pb}(\text{Q})_2]$. Photoluminescence (PL) properties of the nanostructured and crystalline bulk of the prepared complex and lead oxide nanoparticles core of them have been investigated. The maximum emission peaks showed that a good correlation exists between the size of particles and emission wavelengths.

Keywords: Sonoelectrochemical synthesis, PbO, Photoluminescence

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Electrophoretic deposition of titania/CNTs nanocomposite coatings from different alcohols

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Abstract

The preparation of titania nanostructured coatings and titania/carbon nanotubes (CNT) nanocomposite coatings on 316L stainless steel by electrophoretic deposition (EPD) method has been investigated in this work. The suspensions of titania nanoparticles (20g/L) were prepared in different alcohols (methanol, ethanol, isopropanol and butanol) using triethanolamine (TEA) as dispersant; the suspensions were investigated by different analysis such as sedimentation and zeta potential. Titania nanostructured coatings were prepared by EPD from different alcoholic suspensions at 60V and various times (15, 30, 60, 120, 240 and 360s); the kinetics of deposition and their microstructure were investigated in case of EPD from alcoholic suspensions with different dispersant concentrations and according to obtained results the optimum concentration of dispersants was 16.33, 8, 0.33, 0.33 mL/L for methanol, ethanol, isopropanol and butanol, respectively; In the next step to prepare titania/CNT nanocomposite coatings, CNT was added into alcoholic suspensions containing optimum concentration of dispersant and EPD was performed from them; the effect of CNT addition on the EPD process as well as coatings characterizations was studied.

Keywords: Electrophoretic deposition, Nano composite coatings, Titania, Triethanolamine (TEA), Carbon nanotubes.

Comparison the performance of chromium and chromium oxide coatings toward corrosion protection of carbon steel in seawater

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Abstract

Surface modification by coatings has become an essential step to improve the surface properties such as wear, corrosion and oxidation. Various conventional techniques are utilized for depositing the desired materials on to the substrate to achieve the surface modification. Here, a layer of chromium coating was compared with a layer of the chromium oxide coating in which the layers were produced by electrodeposition technique. The electrodeposition of metallic chromium was carried out using cyclic potential (20 scans between 0 and -1.0V at a scan rate of 50mVs^{-1}) in $0.1\text{ molL}^{-1}\text{H}_2\text{SO}_4$ solution, in a solution containing 0.5 molL^{-1} chromium(III) chloride. The potential was repetitively cycled (30 scans) from 0 to 1.2 V at a scan rate of 100mVs^{-1} in fresh phosphate solution for the passivation of a chromium oxide layer at a carbon steel specimen. In investigating of coating surfaces by scanning electron microscope (SEM), the results showed that the morphology of the coating surface was changed by converting chromium to the chromium oxide. The corrosion behavior of the coatings was assessed by polarization technique in seawater and 3.5% NaCl solution. The results showed that chromium oxide coating reduce corrosion current and corrosion rate more than chromium coating.

Keywords: Chromium and chromium oxide coating, Corrosion, Carbon steel, Polarization

Comparison the performance of nickel and nickel oxide coatings toward corrosion protection of carbon steel in seawater

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Abstract

In industry, the plating of mild steel and iron by metals like nickel, chrome, zinc has been widely used to protect them against corrosion, for many years [1–3] and thin top coat phosphatation and chromatisation on electrodeposited metal coatings have improved the corrosion resistance of the substrates. The nickel plating on mild steel has been generally applied for longer periods, which exceed the car lifetime in automobile industry and for the fabricated parts such as nails and bolts. In this work, a layer of nickel coating was compared with a layer of the nickel oxide coating in which the layers were produced by electrodeposition technique. The electrodeposition of metallic nickel was carried out using cyclic potential (20 scans between 0 and -1.0V at ascan rate of 50mVs^{-1}) in $0.1\text{ molL}^{-1}\text{H}_2\text{SO}_4$ solution, in a solution containing 0.5 molL^{-1} nickel nitrate. The potential was repetitively cycled (30 scans) from 0 to 1.2V at a scan rate of 100mVs^{-1} in fresh phosphate solution for the passivation of a nickel oxide layer at a carbon steel (CS) specimen. Anodic polarization curves were used to evaluate the corrosion performance of Ni coated and NiO coated electrodes in seawater and 3.5% NaCl. It was observed that Ni electroplating reduced drastically the corrosion rate of carbon steel and exhibited an efficient barrier property on CS. However, the porosity of Ni electroplating increased for longer periods. It was found that the porosity of NiO coating was lowered due to the more passivation effect of this layer and NiO coating could provide significant protection efficiency to CS electrode. The results showed that nickel oxide coating reduce corrosion current and corrosion rate more than nickel coating.

Keywords: Nickel and nickel oxide coating, Corrosion, Carbon steel, Polarization

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Electrochemical assessment the performance of amine-based corrosion inhibitors on API 5L X65 carbon steel in fluid produced from a gas reservoir located in southern of Iran

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Abstract

Corrosion of carbon steel is a significant problem in the oil and gas production and transportation systems and causes significant economic loss [1,2]. Rupture as a result of corrosion of the pipe wall frequently causes failure of petroleum and gas pipelines. The breakdowns are followed by large losses of the products, environmental pollution and ecological disasters [3]. The majority of oil and gas pipelines failures result from CO₂ corrosion of carbon and low alloy steels and occurs at all stages of production from downhole to surface equipment and processing facilities. In this work, the performance of two commercial oil soluble amine-based inhibitors used in Tabnak gas field pipelines, were evaluated. Corrosion laboratory tests on API X65 steel grade were performed to study corrosion rate effect of inhibitors added to residual water transported with gas in pipelines. Corrosion rate was measured by electrochemical methods when inhibitor concentration of 10, 25, 50, 75 and 100 ppm were utilized. The results showed that the extent of inhibition was concentration dependent. Potentiodynamic polarization investigations revealed that amine-based acted as a mixed inhibitor showing anodic and cathodic behaviour. Temperature studies revealed an increase in inhibition efficiency with rise in temperature.

Keywords: Inhibitor, Corrosion, Carbon steel, Polarization

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Corrosion protection of carbon steel by epoxy nanocomposite coatings contain Cloisite 30B nanoparticles

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Abstract

Epoxy/clay nanocomposites have become a very interesting topic among researchers in the past two decades because nanoclays have a positive effect on the mechanical, thermal and especially barrier and anticorrosive performances of the polymers [1-3]. In this study, epoxy-clay nanocomposites were synthesized using different weight percentages of cloisite 30B and diglycidyl ether of bisphenol A (DGEBA) type epoxy resin, in order to produce anti-corrosive epoxy coating. Anti-corrosive properties of the nanocomposites were investigated using salt spray and electrochemical impedance spectroscopy (EIS) methods. The results showed an improvement in the barrier and anti-corrosive characteristics of epoxy-based nanocomposite coating and a decrease in water uptake in comparison with pure epoxy coating. Wide-angle X-ray diffraction (WAXD) patterns and transmission electron microscopy (TEM) analysis showed that the interlayer spacing of clays increased after addition of epoxy resin along with applying shear force and ultrasound sonicator.

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Keywords: Nanocomposite, Anticorrosive coating, Epoxy resin

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A study on the anticorrosion performance of epoxy nanocomposite coatings containing epoxy-silane treated nano silica on mild steel substrate

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Abstract

Anticorrosion performance of a clear epoxy coating was enhanced by incorporation of modified nano silica into the polymer matrix. The surface of nano silica was treated with 3-glycidoxypyltrimethoxysilane (GPTMS) in order to achieve proper dispersion of nanoparticles in the polymeric matrix. The surface grafting of GPTMS on the nanoparticles was detected using Fourier Transform Infrared spectroscopy (FTIR) and dispersion stability test. The result revealed that GPTMS molecules were grafted on the nano silica surface. Electrochemical techniques play an important role for corrosion monitoring of the organic coated metals. As a result, corrosion performance of the coated mild steel specimens was investigated employing Electrochemical Impedance Spectroscopy (EIS) and salt spray test. EIS results showed that addition of the nanoparticles in the epoxy matrix enhanced the barrier properties of the coating system. In this way, coating resistance (R_c) increased while the coating capacitance (C_c) decreased showing the less diffusion of the corrosive electrolyte to the polymeric matrix. Nano silica particles could successfully plugging the porosity of the epoxy resin and made a barrier properties for ion diffusion in the coating system. Incorporation of 4-6 wt% SiO_2 nanoparticles possessed the best corrosion performance. The dispersion state of the nanoparticles in the polymeric matrix was studied using scanning electron microscope (SEM). This technique could be extensively utilized for studying of nano composites. Surface morphology of the coatings changed after incorporation of the nano silica particles. This can be stated that addition of more than 6% nanoparticles could make some aggregates in the polymeric matrix which is evident in the SEM results. These aggregates could significantly decrease the barrier properties of the coating system, resulting in the increment of the corrosion phenomena. The electrochemical results are completely in agreement with the morphological results of the surface obtained from scanning electron microscopy.

Keywords: Electrochemical Impedance Spectroscopy, Nanocomposite Coatings, Nano Silica, Surface treatment, Corrosion, SEM

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TiO₂ nanotube arrays on titanium surfaces for delivery of antibiotics

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Abstract

Nanotube arrays on medical titanium implant surfaces were fabricated by electrochemical anodization method in ethylene glycol based electrolytes with different conditions such as water and NH₄F percentage, voltage and time of anodizing. Central composite design and response surface method were used to design the experiments for production of TiO₂ nanotubes and the optimum parameters for storage of gentamicin were chosen. Drug release experiments with the model antibiotic gentamicin showed that the increased surface area of the anodized samples enabled them to be loaded more active agent than the untreated Ti surfaces. It was concluded that nanotube arrays on favored medical implant materials have a high potential for loading with antimicrobial agents and also provide the possibility of more antibiotic content by variation of anodization parameters.

Keywords: TiO₂ nanotubes, Electrochemical anodization method, Drug delivery, Central composite design, Gentamicin

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Inhibition of aluminum corrosion in alkaline solution by aromatic carboxylic acids

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Abstract

Aluminum is an attractive fuel for electrochemical power sources. Its principal advantages as an anode material include high power and energy density, low practice and great abundance[1]. Corrosion of aluminum and its alloys has been subjects of numerous studies due to their wide range of industrial applications especial in aerospace, surface coating and alkaline batteries [2,3]. The corrosion behavior of aluminum in 1 M NaOH solution in the absence and presence of some aromatic carboxylic acids were investigated using potentiodynamic polarization techniques, electrochemical impedance spectroscopy (EIS) and scanning electron microscope (SEM). The results showed aromatic carboxylic acids are effective corrosion inhibitors for aluminum in alkaline medium. Analysis of temperature effect on aluminum corrosion was investigated and revealed increase in temperature accelerated the corrosion rate but at the presence of aromatic carboxylic acid (3-bromobenzoic acid) the rate of corrosion significantly was reduced. The values of different thermodynamic parameters such as adsorption equilibrium constant (K_{ads}), free energy of adsorption ΔG_{ads} were calculated and discussed. The adsorption process of studied inhibitors on aluminum surface obeys the Langmuir adsorption isotherm. The inhibition efficiencies obtained from polarization, EIS and experiment illustrated good agreement.

Keywords: Aluminum corrosion, Aromatic carboxylic acids, Inhibitor, Electrochemical impedance spectroscopy, Potentiodynamic polarization

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Using electrochemical measurement method for investigation of 3-hydroxybenzoic acid and 3-bromobenzoic acid as aluminum corrosion inhibitors in the presence of alkalized Persian Gulf sea water

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Abstract

Aluminum and its alloys are widely used in many engineering applications and scientific technologies, such as in aerospace, advanced nuclear reactor, surface coating, metal/air batteries, etc [1]. They are very good corrosion resistant materials in neutral aqueous solution, due to the formation of passive film however, but it is susceptible to corrosion by acids and alkaline solutions[2]. Several workers have employed organic and inorganic compounds as corrosion inhibitors to control this passive film dissolution and thereby reducing the rate of metal loss in alkaline solutions[3]. The current study was investigated the inhibition efficiency of 3-hydroxybenzoic acid and 3-bromobenzoic acid on aluminum corrosion in Persian Gulf sea water (pH=12) using Potentiodynamic polarization techniques, electrochemical impedance spectroscopy (EIS) and scanning electron microscope (SEM) methods. 3-hydroxybenzoic acid and 3-bromobenzoic acid was shown effective corrosion inhibitors on aluminum in NaOH 1mol/lit. The inhibition efficiency was found to increase with increasing inhibitors concentration and decrease with increasing temperature. The effect of temperature on the corrosion behavior in the presence of 2000 mg/lit of inhibitors was studied in the temperature range of 303-353 K. The values of different thermodynamic parameters such as adsorption equilibrium constant (K_{ads}) and free energy of adsorption ΔG_{ads} were calculated and discussed. The adsorption process of studied inhibitors on aluminum surface obeys the Langmuir adsorption isotherm. The analysis of temperature effect on aluminum corrosion was illustrated in the presence of 3-bromobenzoic acid the rate of corrosion significantly reduced at 353 K.

Keywords: Aluminum, Corrosion, Inhibitor, Persian gulf sea water, Electrochemical method

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Recovery of cadmium from spent Ni-Cd batteries by potentiostatic and galvanostatic electrodeposition methods

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Abstract

Rechargeable Ni–Cd batteries have been used since 1950 in applications that require high energy density, long lifetime, and high discharge rates. Therefore, Ni–Cd batteries are used in many applications such as defense industries including aerospace industry and Non-defense industries such as railways, subways, power plants, refineries and nuclear power plants and the use of cadmium in Industrial plating and coatings, pigments, synthetic products, plastic molding, recovery of valuable metal components [1]. Pyro and hydrometallurgical processes are the basic technologies applied to processing spent Ni–Cd batteries [2]. Recycling by pyrometallurgical process is not appropriate due to the emission of toxic gases, which can contaminate the environment. Recycling batteries by a hydrometallurgical process is an alternative solution for the environmental problem. The hydrometallurgical process involves electrochemical and chemical transformations [3]. In this research, the recovery process for Cd metal is conducted by the potentiostatic and galvanostatic electrodeposition in a leach electrolyte by 4 M HCl and 4M H₂SO₄ with and without a sodium citrate complex. Different techniques such as ICP, XRD, EDX were employed to characterize the chemical composition and morphology of electrode materials. Finally, The recovery of Ni from leach liquor is approximately 90%. The optimum applied potential for Cd recovery is in the range -1100 to -1120 mV. The current efficiency for Cd recovery is 70–90%.

Keywords: Recovery, Ni–Cd batteries, Potentiostatic, Galvanostatic, Electrodeposition

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Electrochemical and morphological investigation of titanium-based nano layer on hot-dip galvanized steel

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Abstract

Improving the corrosion performance of metal substrate using conversion coatings have been studied in the last decades. Cr(VI) based conversion coating is the most common conversion coating which produced superior corrosion resistance on metal bodies. The use of this type of conversion coating is restricted due to toxic effects of Cr(VI) on environment and humans. Hexafluorotitanic acid is one of novel alternatives for chromates [1]. In this study a hexafluorotitanic acid containing solution was developed for treatment of galvanized steel surfaces and some parameters of treatment process which affect the conversion coating resistance are optimized. Solution pH, acid concentration and immersion time are variable parameters which have been considered. Effectiveness of conversion coatings was investigated using polarization tests. Corrosion current density (i_{corr}) was extracted from polarization curves using Tafel extrapolation technique. Considering anticorrosion performance of the layer, the optimum level for each parameter has been obtained. Morphological investigations conducted using atomic force microscopy (AFM) and reveals formation of conversion layer and increase of roughness of the surface. This increase of roughness can also improve adhesion of organic coating on surface. At low pHs the dissolution rate of substrate is greater than the precipitation rate of Ti complexes [2]. At high pHs the precipitation rate of Ti compounds become higher, but formation of non-uniform thick layer on surface containing defects and cracks results in increasing corrosion current density. Results show that increase of Ti concentration cause in reducing coating resistance presumably due to clustering of Ti species on the surface in more concentrated solutions and producing a non-uniform surface. Also, long immersion time of surfaces in solution results in thickening of layer and formation of an uneven coating due to inside stresses and formation of cracks and defects. These defects facilitate the electrolyte permeation toward metal surface and reduce the coating resistance [3]. The effect of solution pH, Ti concentration and immersion time on the anticorrosion performance of a Ti-based conversion coating on hot-dip galvanized steel was examined utilizing polarization test. Considering anticorrosion performance of the layer, the optimum level for each parameter has been obtained. Also morphological investigations was studied using AFM.

Keywords: Conversion coating, Polarization, AFM, Hexafluorotitanic acid

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Application of Mesoporous Silica Nanocontainers as Smart Host of Corrosion Inhibitor in Polymeric Coatings

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Abstract

Mesoporous silica nanocontainer powders were applied as corrosion inhibitor hosts and dispersed in the polypyrrole matrix. Then, the release and corrosion resistance of these composite coatings were studied with and without inhibitor in NaCl solution. Results showed that in higher pHs and more aggressive chloride media, the release content of corrosion inhibitor is higher. OCP, ICP, EIS, and FTIR results showed that the substrates were better protected in the presence of released corrosion inhibitor from mesoporous silica compared to the coatings without inhibitor. Then, the corrosion inhibitor reacted with substrate and made a protective phase during corrosion.

Keywords: Corrosion Inhibitor, Mesoporous silica, Polymeric Coating, Electrochemical Impedance Spectroscopy

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Preparation of modified chitosan nano composite and its application in simultaneous electrochemical determination of ascorbic acid and dopamine by modified GC electrode

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Abstract

(Organic Section): Chitosan (CHT) is a well-known abundant natural polymer with relatively good biodegradability, biocompatibility and bioactivity. But the low solubility in water, the insolubility in common organic solvents has limited its utilization and basic research[1]. In order to overcome these problems, chemoselective N-phthaloylation of chitosan was accomplished for improving solubility. Then, N-phthaloylation of chitosan was modified with 4-cyano, 4-[(phenylcarbothioyl) sulfanyl] pentanoic acid to serve as RAFT agent[2]. The graft copolymerization of acrylic acid was initiated by Chitosan-Raft agent as a macro initiator. Chitosan-g- poly acrylic acid (CHT-g-PAA) was used as stabilizer for preparing colloidal Silver nanoparticles (AgNPs) within the range of (2–10 nm). CHT-g-PAA/Ag has many applications, such as antibacterial agents in organic chemistry and modifier in electrochemistry. (Electrochemical Section): Dopamine (DA) plays an important role in the function of central nervous, renal, hormonal and cardiovascular systems. Low levels of DA may cause neurological disorders such as schizophrenia and Parkinson's disease. Ascorbic acid (AA) is an agent which prevents scurvy and is known to take part in several biological reactions. AA and DA usually coexist together in human metabolism. Therefore, the development of a sensitive and selective method for their simultaneous determination is highly desired for analytical applications and diagnostic researches. However, a major problem encountered is that AA and DA are oxidized at nearly a same potential with poor sensitivity at solid electrodes. The overlap of their voltammetric responses, the pronounced electrode fouling, poor selectivity and reproducibility make their simultaneous determination highly difficult [3]. The modified electrode fabricated by graphene and CHT-g-PAA/Ag can be applied for simultaneous determination of dopamine and ascorbic acid. While the CHT-g-PAA/Ag was the matrix with pH=7.0, the positive-charged DA was repelled by chitosan nano composite, resulting in the potential of DA a positive shift and the peak separations between AA and DA. Therefore, it can realize the simultaneous determination of dopamine and ascorbic acid in their mixture.

Keywords: Chitosan, Acrylic acid, Silver nanoparticle, RAFT polymerization, Ascorbic acid, Dopamine, GC electrode

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Direct electron transfer between cytochrome c and TiO₂/Ag nanocomposite electrode

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Abstract

The direct electron transfer of redox proteins and enzymes is very difficult on many bare solid electrodes. Many methods and materials are used to immobilize the proteins and enzymes in order to enhance the direct electron-transfer rate and provide a suitable microenvironment for them [1]. Cytochrome c (Cyt c) is a very basic redox heme protein, but it is usually difficult for Cyt c to transfer electron to a conventional electrode. To remedy this problem, various promoters have been employed to promote the direct electron transfer between Cyt c and the electrode [2]. In the present study, direct electrochemistry of Cyt c, which was adsorbed on the surface of silver-doped titanium dioxide (TiO₂/Ag), was investigated. The results from cyclic voltammetry (CV) suggested that Cyt c could be tightly adsorbed on the surface of TiO₂/Ag and TiO₂/Ag shows an obvious promotion for the direct electro-transfer between Cyt c and electrode. We fabricated a hybrid bio interface consisting of silver-doped titanium dioxide (TiO₂/Ag) film and cytochrome c (Cyt c) on indium tin oxide (ITO) electrodes. A couple of well-defined and quasi-reversible CV peaks of Cyt c (E_{pa} at 0.09 V and E_{pc} at 0.02 V) can be observed in a phosphate buffer solution (pH 7.0). The TiO₂/Ag film provided a good microenvironment for the immobilization of biomolecules, enhanced the surface coverage of protein, had large surface-to-volume ratio, and high stability. The results showed that TiO₂/Ag promoted the direct electron transfer between Cyt c and the bare electrode.

Keywords: Cytochrome c, Silver-doped titanium dioxide, Cyclic Voltammetry, Direct electrochemistry.

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Detection of a DNA sequence related to hepatitis b virus by a novel gold nano-particles and mercaptobenzaldehyde modified au electrode

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Abstract

In recent years, the interest for DNA-based diagnostic tests has been growing. The development of systems allowing DNA detection is motivated by applications in many fields: DNA diagnostics, gene analysis, fast detection of biological warfare agents, and forensic applications. Detection of genetic mutations at the molecular level opens up the possibility of performing reliable diagnostics even before any symptom of a disease appears. Optical [1], electrochemical [2], and micro-gravimetric [3] DNA transductions have been widely studied. A new procedure for fabricating hepatitis B virus (HBV-DNA) electrochemical biosensor was developed based on covalent immobilization of probe NH₂ labeled HBV-DNA on gold nano-particles modified Au electrode that had been functionalized by Mercapto-benzaldehyde (MB) with self-assembled technology. Firstly, Au electrode modified with gold nano-particles with a simple one-step electro-deposition method. Then, mercapto-benzaldehyde was self-assembled onto the electrode surface through Au-S bond. Mercapto-benzaldehyde was applied as an arm linker to covalently graft the 5'-amino modified HBV-DNA to the gold nano-particles modified Au electrode surface via the facile aldehyde-ammonia condensation reaction. The whole DNA biosensor fabrication process was characterized by cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) using [Fe(CN)₆]^{3-/4-} as an electrochemical redox indicator. The probe DNA immobilization and hybridization on the modified electrode was further studied with differential pulse voltammetry (DPV) using ferricyanide as an indicating probe. A linear detection range of the HBV-DNA was obtained from $5.7 (\pm 0.1) \times 10^{-11}$ to $6.6 (\pm 0.1) \times 10^{-8}$ M with a detection limit of $7.6 (\pm 0.1) \times 10^{-12}$ M.

Keywords: Hepatitis B virus, Gold nano-particles, Au electrode, Mercapto-benzaldehyde, DNA Biosensor

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A Non-enzymatic glucose sensor fabricated by multi-walled carbon nanotubes modified with Cu_xO

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Abstract

Diabetes is a worldwide chronic disease. Nowadays it has been one of the most serious causes of death and disability [1]. Glucose detection is essential in clinical diagnostics, biotechnology and the food industry [2]. The fabrication of non-enzymatic glucose biosensor is essential with the goal of achieving the direct electro-catalytic oxidation of glucose [1]. In this work we present, Cu particles from the solution containing CuSO₄ and H₂SO₄ were deposited onto multi-walled carbon nanotubes (MWCNTs) paste electrode at constant potential and then they were oxidized into Cu_xO by cyclic voltammetry. The electrocatalytic activity of MWCNTs-Cu_xO was investigated for glucose detection under alkaline conditions using cyclic voltammetry and chronoamperometry. In addition, the experimental parameters such as the concentration of CuSO₄ and H₂SO₄ and the amperometric detecting potential were optimized. It was found that the sensor exhibited high electrocatalytic activity, stability and good selectivity against common interfering species such as ascorbic acid, uric acid and dopamine. The applicability of the proposed method to the determination of glucose in human blood serum and human urine samples was detected with satisfactory results.

Keywords: Non-enzymatic sensor, Glucose, MWCNTs, Cu_xO, Chronoamperometry, cyclic voltammetry

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Catalase Immobilization onto Sonicationally-Functionalized Multiwalled Carbon Nanotubes

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Abstract

Catalase (CAT) is an important enzyme of oxidoreductase family which catalysis H_2O_2 degradation. For improvement of its biocatalytic efficiency could use desirable immobilization strategy. In the recent years nanomaterials have been employed for this purpose. Multi walled carbon nanotubes (MWCNTs) are one of the most common nanostructures that has been widely used regards to enzyme immobilization [1]. In this work, firstly to enhancement of efficient interaction between CAT molecules and immobilizing matrix, aqueous MWCNTs suspension were sonicated at 70°C for 20 minutes and were dried under IR lamp. Then 3 μ L of 2mg/ml functionalized MWCNTs suspension in Dimethylformamide (DMF) were dropped on glassy carbon electrode (GCE) surface and was allowed to dry under room temperature. Then MWGNTs/GCE was immersed in CAT solution (0.02 MPBS, pH 7.0) for 5 hours. Finally, the CAT/MWCNT/GCE was investigated for immobilization by Cyclic Voltammetry method. The cyclic voltammogram exhibited a pair of well-defined quasi-reversible peaks corresponding to the Fe(III)/Fe(II) redox couple in the active site of CAT. The formal potential ($E^{\circ'}$) was 0.072 V vs. Ag/AgCl (cathodic and anodic peaks at -0.185 and 0.041 V, respectively) in 0.05 M PBS, pH 7.0. The knowledge gained from this report offer simple way to functionalization and immobilization of enzymes on MWCNTs. CAT/MWCNT/GCE could use for direct electrochemistry studies as well as third generation biosensor to H_2O_2 determination.

Keywords: Direct Electrochemistry, catalase, Multi walled carbon nanotubes, Sonication

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Bio-electrochemical reduction of nitrate from wastewater using carbon cloth cathode modified with multi wall carbon nanotube(MWCNT)

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Abstract

In this study, nitrate reduction from wastewater was investigated in dual-chambers bio-electrochemical reactor (BER). The influence of MWCNT as cathode modifier was studied for bio-electrochemical denitrification efficiency. Also, the impacts of pH, current density, retention time and initial nitrate concentrations on nitrate reduction performance were assessed in this system. Optimum pH, current density and retention time were 7, 15 mA/cm² and 8 h respectively. Scanning electron microscope (SEM) images of the cathode surfaces revealed a homogeneous distribution of the MWCNT on the carbon cloth matrix. The highest nitrate reduction efficiency at optimum condition was 84.32% for MWCNT modified carbon cloth and 68.48% for not modified carbon cloth. These results indicate that MWCNT as a cathode modifier can increase nitrate reduction efficiency in BER.

Keyword: Nitrate reduction, Carbon nanotube, Carbon cloth, Bio-electrochemical denitrification

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Direct electrochemistry of cytochrome c on TiO₂/Au nanocomposite modified electrode

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Abstract

Direct electrochemistry between redox proteins and electrode surface has been extensively studied because of its potential application in the investigation of biomolecules [1]. Cytochrome c (Cyt. c) is an excellent model for studying the electron transfer of typical metalloproteins from a structure point of view. Due to the difficulty of direct electron transfer between Cyt c and a bare electrode, some modified electrodes were used as a tool to investigate the direct electrochemical property. The modifiers of these modified electrodes are organic [2], or inorganic [3] compounds. In the present study, we explored the ability of TiO₂/Au nanocomposite to act as modifier to realize the direct electron transfer (DET) of Cyt c. The investigation on titanium dioxide (TiO₂), a kind of inorganic porous material with unique construction, high surface area and high chemical stability, had shown that it could be used as an electrode material to promote electron-transfer reactions of protein. We investigated the formation of a hybrid biointerface consisting of gold-doped titanium dioxide (TiO₂/Au) film and cytochrome c (Cyt c) on indium tin oxide (ITO) electrodes. The TiO₂/Au film provided a good microenvironment for the immobilization of biomolecules enhanced the surface coverage of protein, had large surface-to-volume ratio, and high stability. Cyclic voltammetry (CV) was performed to evaluate electrochemical properties of the as prepared interface. The ITO/TiO₂/Au /Cyt c electrode display a pair of well-defined redox peak (E_{pa} at 0.06 V and E_{pc} at 0.02 V) at pH 7.0 in phosphate buffer solution. These nanoparticles inhibit the adsorption of Cyt c onto bare electrode and act as a bridge of electron transfer between protein and electrode.

Keywords: Cytochrome c, Gold-doped titanium dioxide, Cyclic voltammetry, Direct electrochemistry

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On/off-switchable molecularly imprinted folic acid “plastic” sensor

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Abstract:

Molecular imprinting of polymers (MIP) is a technique used to create plastic receptors by the formation of a polymer network around a template molecule. MIPs have great potential in bioanalytics due to their ease of preparation, low cost and a high sensitivity, selectivity and stability. The electrosynthetic approach due to create simply and rapidly an adherent and compact polymeric film with controllable thickness and direct communication between the polymer and the surface of the transducer could be very helpful both in improving the molecular imprinting polymerization (MIP) procedure and in extending the application of MIP [1-2]. The combination of smart polymers with molecular imprinting offers a powerful tool to design more effective sensors and medical devices [3]. In this work, a temperature sensitive amine-terminated poly(N-isopropylacrylamide) block with (N,N'-methylenebisacrylamide) cross-linker along with o-phenylenediamine was electropolymerised on a gold electrode in the presence of folic acid (FA) as template to produce an on/off-switchable molecularly imprinted polymer (MIP) affinity sensor for folic acid. The template molecules were removed from the MIP electrode surface by washing with a methanol-acetic acid (9:1, v/v) solution. Differential pulse voltammetry and cyclic voltammetry were used to characterise the FA-imprinted layer. Incubation of the MIP-modified electrode with FA resulted in a suppression of the ferro/ferricyanide redox process. The highest sensitivity of this temperature gated on/off switchable folic acid sensor was achieved at 22 °C. Such switchable affinity materials offer considerable potential for the design of highly selective and controllable biosensors and immunoassays.

Keywords: Molecularly Imprinting Polymer, Smart polymers, On/off switchable folic acid sensor

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Development immobilization of DNA onto a carbon nanotube modified electrode and characterization by electrochemical techniques

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Abstract

In recent years, DNA-based diagnostic tests especially electrochemical DNA biosensors play an important role in clinical genetic analysis because the feasibility of using electrochemical techniques. The process of immobilization of DNA probes on solid surfaces is one of the essential steps toward to DNA sensor development. Up to now, a variety of techniques have been developed for the surface-capturing DNA probes onto transducer surfaces such as electrostatic attraction [1], covalent binding, controlled potential adsorption [2], and via the biotin-avidin interaction [3]. In the present study, we propose a rapid and simple strategy to the immobilization process of single strand DNA (ss-DNA) on modified glassy carbon electrode (GCE) surface. The functionalized multi walled carbon nanotubes (MWCNTs) can be successfully employed for the immobilization of ss-DNA through covalent interaction between this macromolecule and carboxylated MWCNTs (MWCNT-COOH). The both cyclic voltammetry and electrochemical impedance spectroscopy (EIS) provide evidence for the probe immobilization with the use of ferro-ferric cyanide as an electrochemical redox indicator. The peak potential separation ($\Delta E_p = E_{pa} - E_{pc}$) were found 0.087 and 0.174 V for MWCNT-COOH/GCE and ss-DNA/MWCNT-COOH/GCE, respectively. The obtained results shows that ss-DNA sequence have been successfully immobilized on MWCNT-COOH/GCE. The value of $-46.24 \text{ kJ mol}^{-1}$ was calculated for the standard free-energy of adsorption (ΔG_{ads}°) at different concentration of ss-DNA which suggests the formation of covalent bonds. The key parameters for covalent immobilization of DNA probe such as buffer conditions and time has been studied in order to optimize this methodology. The knowledge gained from this report offer new opportunities to future design of novel nucleic acid biosensor.

Keywords: Electrochemical DNA biosensors, Multi walled carbon nanotubes, Cyclic voltammetry, EIS, Covalent bond

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Ethanol/O₂ enzymatic fuel cell composed carbon nanotube composite

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Abstract

Bio fuel cells are important to generate alternative energy. This paper evaluates the electro activity of a carbon felt electrode containing immobilized 1-ethyl-3-methyl imidazolium bis(trifluoro methyl sulfonyl)imide (emimNTf₂)-carboxylated multiwalled carbon nanotubes gel (emimNTf₂-HOOC-MWCNTs), a NAD⁺-dependent alcohol dehydrogenase enzyme (ADH), poly-methyl enegreen (poly-MG), and a supporting agent (poly diallyl dimethyl ammonium chloride, PDDA). The PDDA is an excellent medium to achieve ADH immobilization on the carbon nano structured electrode. We obtained power density and Open circuit voltage (OCV) of 0.250 mW cm⁻² and 0.350 V, respectively for this modified composite of carbon nanotube. This new anode configuration is potentially applicable for small energy devices.

Keywords: Biofuel cell, 1-Ethyl-3-methyl imidazolium bis (trifluoro methyl sulfonyl) imide, Poly diallyl dimethyl ammonium chloride, Carboxylated multiwalled carbon nanotubes, NAD⁺-dependent alcohol dehydrogenase enzyme

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Detection of *rfbE* gene *Escherichia coli* O157:H7 using DNA hybridization by electrochemical impedance spectroscopy

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Abstract

In this study, we have developed a free labels DNA nanobiosensor of low cost, highly selective, simplicity and fast response for detection of *rfbE* gene *Escherichia coli* O157:H7. A convenient electrochemical technique is electrochemical impedance spectroscopy (EIS), widely applied in DNA detection [1]. Recent developments in nanomaterials create many opportunities to advance DNA sensing and gene detection. The Au nanoparticles modified electrode (Au_{nano}ME) was prepared by electrodeposition technique [2]. Then the single-stranded DNA (ssDNA) probes were immobilized through the chemisorptions process on Au_{nano}ME (ssDNA/Au_{nano}ME). Hybridization of ssDNA/Au_{nano}ME was performed with complementary oligonucleotide, mismatched oligonucleotide and noncomplementary oligonucleotide. Electrochemical impedance spectroscopy (EIS) technique has been used to investigation of the hybridization and sensing properties[3]. The results showed that the immobilization and hybridization of DNA on the Au_{nano}ME increases effectively the nanobiosensor response compared with the Au electrode. A nanobiosensor based on hybridization process of DNA/DNA was fabricated. The nanobiosensor showed good selectivity for complementary oligonucleotide. Due to the obtained results, the nanobiosensor is promising for environmental and clinical applications.

Keywords: DNA hybridization, Electrochemical Impedance Spectroscopy, Gold nanoparticles electrode

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Facile Electrochemical Preparation of α -Co(OH)₂ Nanoplates with Excellent Supercapacitive Performance

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Abstract

Co(OH)₂ materials have been attractive in view of their layered structure with large interlayer spacing, their well-defined electrochemical redox activity, and the possibility of enhanced performance through different preparative methods [1]. An emerging application of Co(OH)₂ is an electrode material for electrochemical supercapacitors [2, 3]. In the supercapacitive application, the sheet or plate-like structures are beneficial to improve the electrochemical performance of Co(OH)₂. These structures can provide large inter-sheet spacing for transferring the ions rapidly and increasing the electroactive material electrolyte interface area i.e. electrolyte penetration which resulted high utilization of the electrode materials. In this research, cobalt hydroxide nanoplates were prepared *via* cathodic electrodeposition and their electrochemical performance was investigated using cyclic voltammetry and charge-discharge tests. The deposition experiments were performed in the direct current mode with the applied current density of 0.1 mA cm⁻². The structural analysis (by XRD and IR) and morphological observation by SEM revealed that the prepared hydroxide has pure crystalline α -Co(OH)₂ nanoplates with the sizes in the range of 200–300 nm. A maximum specific capacitance of 2017.3 F g⁻¹ was obtained in aqueous 1 M KOH within the potential range of -0.3– 0.5 V (vs. Ag/AgCl) at the scan rate of 10 mV s⁻¹, suggesting the potential application of the prepared nanoplates in electrochemical supercapacitors. This facile method can be easily scaled up for the large production of uniform α -Co(OH)₂ nanoplates.

Keywords: α -Co(OH)₂ Nanoplates, Cathodic electrodeposition, Electrochemical supercapacitors

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Hydrogen production on reduced graphene oxide/ZnO nanocomposite via photoelectrochemical water splitting

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Abstract

Hydrogen is a clean energy source and maybe a main energy sources in late 21st century [1-3]. The photoelectrochemical (PEC) water splitting is a non-fossil, solar-based method for H₂ production. In this study, TiO₂/Graphene/ZnO nanocomposite was used as an appropriate photoanode to produce photocurrent via UV/Vis irradiation. The produced electron on photoanode will generate H₂. To prepare the photoanode, at first, Ti mesh was heated in the temperature of 500 °C for 24 h to obtain TiO₂ in anatase phase on the surface of Ti mesh. Then, graphene oxide (GRO) nanosheets were electrophoretically deposited on Ti-TiO₂ substrate. During the electrophoretic deposition, reduced graphene oxide (RGO) will be produced from GRO. Then, zinc oxide nanoparticles were deposited on RGO via a hydrothermal method on the substrate. For this, prepared RGO/TiO₂-Ti was placed in an autoclave containing certain amounts of urea and Zn²⁺ under the optimized experimental conditions including the reaction temperature of 180 °C, the reaction time of 24h. The annealing temperature was 350 °C. PEC measurements were carried out using a galvanostat/potentiostat Autolab PGSTAT101 with Ti-TiO₂/RGO/ZnO photoanode as a working electrode, a Pt plate as a counter electrode and an Ag/AgCl (KCl 3.0 M) as a reference electrode. Xe lamp was used as the source of the light irradiation. The photocurrent responses of the photoanode were recorded under the applied potential of 0.5 V with switching-on and -off model. The high amount of photocurrent (about 3 mA) obtained by the proposed photoanode in comparison with TiO₂/ZnO is due to the high electron mobility in graphene. On Pt cathode, however, H₂ is produced corresponding to the produced photocurrent.

Keywords: Graphene, Titanium dioxide, Zinc oxide nanoparticles

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**Photoelectrochemical water splitting and hydrogen production by reduced graphene oxide/
 $\text{Zn}_x\text{Cd}_{1-x}\text{S}$ nanocomposite under visible light irradiation****Neda Tedadi, Omran Moradlou****Department of Chemistry, Faculty of Sciences, Alzahra University, P.O. Box: 1993893973, Tehran, Iran**Corresponding author E-mail: moradlou@alzahra.ac.ir***Abstract**

Photoelectrochemical hydrogen production from water splitting under visible-light irradiation is considered to be an attractive way to solve the increasing global energy crisis and environmental pollution in modern life [1-3]. Photoelectrochemistry (PEC) is the most promising system to sustainably produce hydrogen fuel. In this study, the PECH_2 -production activity over the reduced graphene oxide/ $\text{Zn}_x\text{Cd}_{1-x}\text{S}$ nanocomposites under simulated solar irradiation has been reported. At first, the electrophoretic method was applied to deposit chemically synthesized graphene oxide (GRO) nanosheets onto a titanium mesh as a suitable substrate. During the electrophoretic deposition, GRO is reduced to reduced graphene oxide (RGO). Then, by using a feasible co-precipitation hydrothermal strategy, RGO- $\text{Zn}_x\text{Cd}_{1-x}\text{S}$ photoanode was synthesized. In this way, graphene/titanium mesh was placed in an autoclave included certain amounts of thiourea, $\text{Zn}(\text{NO}_3)_2$ and $\text{Cd}(\text{NO}_3)_2$. For obtaining high efficiency in H_2 production, the experimental conditions such as reagents ratio, reaction temperature and time were examined. Under the optimized conditions (including Zn: Cd ratio of 4:1, the reaction temperature of 180 °C, and the reaction time of 24h) $\text{Zn}_x\text{Cd}_{1-x}\text{S}$ nanocomposite were deposited on RGO/Ti. The photoelectrochemical measurements were carried out using a galvanostat/potentiostat Autolab PGSTAT101 instrument and a three-electrode electrochemical cell with RGO- $\text{Zn}_x\text{Cd}_{1-x}\text{S}$ photoanode as a working electrode, a platinum plate as a counter electrode, and a Ag/AgCl (KCl 3.0 M) as a reference electrode. A high-intensity Xe lamp with UV filter was used as the source of the visible light irradiation. During all measurements, 0.1 M Na_2SO_4 was used as electrolyte. The photocurrent response of the RGO- $\text{Zn}_{0.8}\text{Cd}_{0.2}\text{S}$ nanocomposite photoanode were recorded 1.5 mA under the applied potential of 0.5 V with switching-on and -off model. The photocurrents of $\text{Zn}_x\text{Cd}_{1-x}\text{S}$ were altered by varying the Zn/Cd ratio. It was obtained that under the same experimental conditions, RGO- $\text{Zn}_{0.8}\text{Cd}_{0.2}\text{S}$ nanocomposite with higher photocurrent has higher efficiency in H_2 production.

Keywords: Photoelectrochemical, Graphene oxide/ $\text{Zn}_x\text{Cd}_{1-x}\text{S}$, Hydrogen production

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Preparation and characterization of platinum containing gas diffuse electrode for oxygen reduction reaction in advanced membrane chlor alkali set-up

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Abstract

Chlor-alkali technology is one of the most important industries that operate to produce chlorine gas and caustic soda solution [1]. In this industry, three technologies such as mercury, diaphragm and membrane cells are being operated. However, more than 90% of chlorine and caustic soda are being produced via membrane electrolysis of brine ($\text{NaCl } 300 \text{ g L}^{-1}$) because of its environmental and economical characteristics. Today, many researchers have focused on modification of membrane chlor-alkali to change it to advanced chlor alkali. In advanced chlor-alkali, the hydrogen evolution reaction (HER: $\text{H}_2\text{O} + 2\text{e}^- \leftrightarrow \text{H}_2 + 2\text{OH}^-$) is replaced by oxygen reduction reaction (ORR) [2]. So, in this work, a gas diffuse electrode (Pt/C) was prepared by reduction of Pt (IV) with NaBH_4 [3] and then characterized by SEM, XRD, EDAX and BET. The electrochemical investigations show that the ORR on the Pt/C gas diffuse electrode takes place at positive potential with respect to HER. In the end, the optimized electrode (%Pt>40) was employed as gas diffuse cathode in membrane single cell (10 cm^2) setup and operated more than two weeks. Comparison of this electrode by conventional cathode (to reduce water) shows that the energy consumption reduces more than 25%. It is stated that the conventional cell operated by 3.1–3.3 volt (5.6 kA m^{-2}) whereas this advanced chlor-alkali cell have been working at 2.5–2.6 volt.

Keywords: Gas diffuse electrode, Advanced chlor-alkali, Membrane electrolysis

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CeO₂ Supported Pt Catalyst on the Carbon-Ceramic Electrode for the Oxidation of Formic acid and Formaldehyde

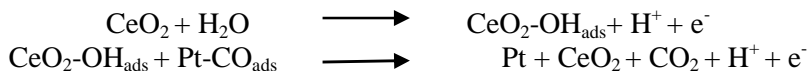
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Abstract

It is well known that platinum (Pt) is an effective electrocatalyst for formic acid and formaldehyde oxidation. In order to improve the electrocatalytic performance of the Pt catalyst, the Pt-based bimetallic catalysts, such as Pt-Ru [1], Pt-Ir, Pt-Sb, Pt-Pd [2], Pt-Fe, Pt-Sn, Pt-Ag [3] and Pt-oxide anode materials like for example Pt-WO₃ have been reported. Metal-oxide (CeO₂, ZrO₂) is widely used as a support for many oxidation catalysts. In this work, the Pt-CeO₂ nanoparticles are deposited on the carbon-ceramic to produce CCE supported Pt-CeO₂. The Pt-CeO₂ nanoparticles were electrodeposited on the CCE surface from an aqueous solution of 0.1M H₂SO₄ containing H₂PtCl₆ · 6H₂O and excess of CeO₂ at (-0.3V) versus the saturated calomel electrode at 25 °C. After, surface, structural, and electrochemical characterizations of the Pt-CeO₂/CCE, the electrocatalytic activity of Pt-CeO₂/CCE towards the formic acid and formaldehyde oxidation in 0.1 M H₂SO₄ solution was evaluated by cyclic voltammometric and chronoamperometric measurements and the obtained results were compared with those obtained on the platinum nanoparticles CCE electrodes. It was found that Pt-CeO₂/CCE was catalytically more active than platinum nanoparticles supported CCE electrodes and had satisfactory stability and reproducibility when stored in ambient conditions or continues cycling. Finally, A mechanism for CeO₂ proposed enhancement of oxidation can also be described by the bifunctional mechanism, according to the bifunctional theory the OH_{ads} species on metal oxide can transform CO-like poisoning species (CO_{ads}) on Pt to CO₂, releasing the active sites on Pt for further electrochemical reaction.



Keywords: Pt nanoparticle, CeO₂, Carbon-ceramic Electrode, Formic acid, Formaldehyde, Fuel cells

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Electrooxidation of formaldehyde on carbon ceramic electrode decorated with Pd/MWCNT electrocatalyst

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Abstract

Palladium (Pd) is one of the most frequently employed electrocatalyst materials for formaldehyde oxidation [1]. Pd nanoparticles catalyze formaldehyde electrooxidation via direct pathway and exhibit great resistance to CO-like poisoning intermediates [2]. To improve the catalytic activity of the Pd nanoparticles, they are deposited on various conductive materials such as carbon supports. Multi wall carbon nanotube (MWCNT) is one of the most widely used supports in fuel cells. MWCNT can enhance electron transfer between electroactive species and electrodes and improve reversibility of electrochemical reaction [3]. In this research, Pd nanoparticles are deposited on MWCNT by chemical reaction via sodium borohydride reducing agent. MWCNT-supported Pd (Pd/MWCNT) was decorated on carbon ceramic electrode (Pd/MWCNT/CCE) and used as electrocatalyst for oxidation of formaldehyde in alkaline media. X-ray diffraction (XRD) and scanning electron microscopy (SEM) analyses were performed to characterize the nanostructure of Pd/MWCNT. Electrochemical studies including cyclic voltammetry and chronoamperometry were used to study electrochemical properties of obtained electrocatalyst. The effects of some experimental factors such as electrocatalyst deposition volume on CCE, formaldehyde and NaOH concentration, scan rate, the upper limit of potential scanning region and long-term stability of electrode were investigated. It was found that Pd/MWCNT/CCE was catalytically more active than Pd nanoparticles decorated on CCE without any support. On the other hand, the Pd/MWCNT/CCE catalyst has satisfactory stability and reproducibility for electrooxidation of formaldehyde when stored in ambient conditions and improve catalytic activity in continues cycling makes it more attractive for fuel cell applications.

Keywords: Electrocatalyst, Pd nanoparticle, MWCNTs, Carbon-ceramic electrode, Formaldehyde fuel cells

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Synthesis and investigation of nanostructured mixed Cobalt-Nickel oxides in silica matrix

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Abstract

Supercapacitors can be a suitable response to accruing demand to the clean energy technology because of their higher energy densities compared to conventional capacitors and their higher power densities and cycle life compared to batteries [1]. Therefore, there are several trends to find appropriate materials for making supercapacitors' electrodes, which should illustrate a reversible behavior and nonzero current. Nanocomposites, a combination of nano-dimensional phases with distinct differences in structure, chemistry and properties, can be the good candidate for this purpose. In this study, we have prepared a nanocomposite containing of nanostructured nickeloxide and cobalt oxide in silica media by sol-gel method [2]. Nickel nitrate, cobalt nitrate and tetraethylorthosilicate in molar ratio of nickel to cobalt salts of 0, 0.35, 0.5, 0.65 and 1 were dissolved in a solutions of acetyl acetone, ethanol and ethylene glycol. One drop of concentrated nitric acid was to the reaction mixture as the catalyst of preparation of nickel oxide and cobalt oxide precursor. Then, sol was prepared by putting the mixture in a 70 °C oven for 10 hours. Then the reaction mixture transferred to an electrical furnace which its temperature was rose to 550 °C for 5 hours. The prepared nanocomposites have different colors from purple to black depends to the ratio of nickel oxide to cobalt oxide ratio. The crystal structure and morphology of the prepared materials were certified by XRD, SEM and TEM. Then, the capacitive behaviour of the prepared nanocomposites was studied by cyclic voltammetry studies using a standard three-electrode consisting of an Ag/AgCl reference electrode, a platinum grid auxiliary electrode and a carbon paste working electrode containing of graphite + the nanocomposites powder in a solution of 1 M KOH and the effect of chemical composition of mixed oxide was investigated.

Keywords: Nanocomposite, Silica matrix, Supercapacitor, Nickel oxide, Cobalt oxide

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Designing and fabrication of vanadium redox flow battery mono-cell using carbon nanotube as the electro-catalyst

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Abstract

Vanadium redox flow battery (VRFB) is a new energy storage system which has various advantages, such as long cycle life, high efficiency, flexible design, high reliability, low maintenance costs and environmental friendship. The following equations show the half-reactions of positive and negative electrode in charging mode:

Positive half cell: $\text{VO}^{2+} + \text{H}_2\text{O} \rightarrow \text{VO}_2^+ + 2\text{H}^+ + 2\text{e}^-$

Negative half cell: $\text{V}^{3+} + \text{e}^- \rightarrow \text{V}^{2+}$

The use of this system along with solar power, wind and thermal power plants, etc. has grown significantly. Due to the actual and potential benefits of this technology, a great future is predicted for this technology. Currently, the materials used in VRBs include polymer-impregnated graphite plates, conductive carbon-polymer composites and polymer-impregnated flexible graphite. The methods that have been developed to modify the graphite felt mainly include heat treatment, chemical treatment, electrochemical oxidation and doping by depositing other metals on carbon fibers. The purpose of this study is designing and fabrication of vanadium battery mono-cell using of carbon nanotube (CNT) as the electro-catalyst because of the high surface area and good electrical conductivity of this material. Carbon nanotube was loaded on the carbon felt electrode by sonicating the electrode floated in the CNT ink for appropriate time. Results indicate that loading of carbon nanotube on carbon felt electrode and then applying heat-acid treatment on CNT-modified felt electrodes can greatly improve the performance of electrode for using in vanadium redox flow battery. All of the effective parameters such as CNT ink composition, floating time and time of thermal treatment were optimized. By using the CNT-modified felt electrode VRFB mono-cell was designed. We used two separable pumps which can pump positive and negative electrolytes with the same electrolyte flow rate of 50 mL/min. The effective area of the cell is defined by the effective area of 5 cm² of graphite felt electrodes with 3.18 mm thickness. The membrane used in the cell is Nafion 117. Membrane treatment, designing and mono-cell assembling has been done in the Nano Technology Laboratory of Isfahan University.

Keywords: Mono-cell, Vanadium redox flow battery, Carbon nanotube, Energy storage

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Effect of hierarchical nanostructures MnO_2 on charge/discharge cyclic of Aluminum/Air batteries

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Abstract

Aluminum air battery is one of the most promising candidates for next-generation energy storage systems because of its high capacity and energy density, and abundance. The performance of air electrode composed of hierarchical nanostructures MnO_2 catalyst for oxygen reduction reaction was examined. hierarchical nanostructures MnO_2 was prepared by pulse electrochemical method and crystal structure and particles size of MnO_2 were examined by XRD and scanning electron microscope (SEM), respectively. Manganese oxide, especially manganese dioxide (MnO_2), is very attractive because it has many advantages including distinctive structures, excellent physicochemical properties, environmental compatibility, and cost effectiveness. The electrochemical performance of Aluminum/Air cells composed of hierarchical nanostructures MnO_2 were examined by using potentiostat (AUTOLAB-30). The results shown that cell contain hierarchical nanostructures MnO_2 have excellent charge/discharge cyclic than micro MnO_2 . The performance of nanostructures MnO_2 as cathode catalyst of air/Al batteries was determined by discharged test at the discharge current of 50 and 0.7 mA/g. it has 80% improvement in capacity by using nanostructures MnO_2 . that is because the active materials for the cathode of air/Aluminum battery, nano manganese dioxide is expected to have large specific surface area and thus provides battery with better capacity performance.

Keywords: Air electrode, Aluminum/Air battery, Hierarchical nanostructures MnO_2

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Preparation and Electrochemical Evaluation of CMK-1/S as Cathode Materials for Li/S Battery

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Abstract

Rechargeable Li-S batteries have received ever-increasing attention recently due to their high theoretical specific energy density, which is 3 to 5 times higher than that of Li-ion batteries based on intercalation reactions. Li-S batteries may represent a next-generation energy storage system. However, it has been reported that Li-S batteries with organic liquid electrolyte have some problems related to low active materials utilization efficiency and poor cycle life, because of the insulating nature of sulfur and the solubility of polysulfides generated during the electrochemical reaction process [1]. In this research, to solve these problems, mesoporous carbon (CMK-1) was synthesized and it was used to absorb the polysulfide which forms during the first discharge step of a Li/S battery. CMK-1 in the electrode not only acts as an adsorbent for the polysulfide, but also acts as an electrically conducting additive [2]. CMK1-S nanocomposite with weight ratios of 70:30, 60:40 and 50:50 were prepared by heating the mixture of sulfur and CMK-1 at 155°C for 4h under N₂ protection [3]. Different technique such as DSC/TGA, ICP, XRD, TEM and BET were employed to characterize the chemical composition, surface area, pore and volume size and morphology of electrode materials. Finally the electrochemical performance of CMK1-S nanocomposite was investigated by charge-discharge and cyclic voltammetry methods. The resulting of these tests representing a promising cathode material for rechargeable lithium batteries with high energy density.

Keywords: Lithium sulfur battery, CMK-1, Sol-gel method, Sulfur electrode, Polysulfide dissolution

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Development of high energy density Li-Sulfur Cells using homogeneous incorporation of nano sulfur in Graphene Oxide/ CuO matrix as a cathode

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Abstract

The energy and power densities of rechargeable lithium batteries require significant improvement in order to power electric vehicles. It is highly desirable to develop and optimize high capacity cathode materials for rechargeable lithium batteries [1]. Sulfur has a theoretical specific capacity of 1672 mAh g⁻¹ about 5 times higher than those of traditional cathode materials based on transition metal oxides or phosphates which makes lithium-sulfur (Li/S) redox couple a promising candidate for high energy density batteries. However the loss of sulfur cathode material as a result of polysulfide dissolution causes significant capacity fading and low coulombic efficiency in rechargeable lithium/sulfur cells [2, 3]. Here, we use a chemical approach to immobilize sulfur and lithium polysulfides via the reactive functional groups on graphene oxide. This approach enabled us to obtain a uniform and thin (around tens of nanometers) sulfur coating on nanocomposite of graphene oxide sheets/CuO by a simple chemical reaction deposition. Strong interaction between carbon and sulfur enabled us to accommodate volume expansion of the coated sulfur particles during discharge, trapping soluble polysulfide intermediates and rendering the sulfur particles electrically conducting. The resulting graphene oxide/CuO/sulfur, representing a promising cathode material for rechargeable lithium batteries with high energy density.

Keywords: Graphene oxide/ CuO nanocomposite, Nano sulfur, Rechargeable Li/ S batter; High cycling stability, Energy storage

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Preparing the zinc-silver oxide bipolar electrode and investigating the electrochemical properties

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Abstract

A battery with bipolar electrodes is known to be advantageous over the conventional monopolar electrodes in terms of power output. In a conventional battery, electrical current is generated by active materials travels to current collector and through an outer circuit to reach the next cell. In bipolar battery, active materials of opposite polarities are placed on two surfaces of a bipolar substrate. Current can thus flow through the substrate to the next cell. Consequently, because of a much shorter electrical path, power loss due to ohmic drop in the circuit is minimized. The volume of the battery is reduced due to elimination of outer circuit material. The proper bipolar battery design minimizes IR losses between adjacent cells in a cell-stack and provides for uniform current and potential distributions over the active surface area [1]. In this study, the silver oxide was chemically prepared by adding aqueous solution of $\text{Na}_2\text{S}_2\text{O}_8$ and NaOH to AgNO_3 . The formed particulate was pasted on Cu substrate and was used as positive side. The thin zinc electrode was prepared by electrodeposition from alkaline bath on another side of Cu substrate [2]. Electrodeposited zinc electrode was used as negative side [3]. The electrochemical performance of these bipolar electrodes was investigated by cyclic voltammetry, charge-discharge methods. The electrodes were discharged over a broad range of current density ($40\text{--}500\text{ mAcm}^{-2}$) in 6.1 M KOH electrolyte. The discharge capacity was greater than 90% of the theoretical value.

Keywords: Bipolar electrode, Bipolar stack design, Zinc-silver oxide battery

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Photochemical deposition of palladium nanoparticles onto TiO₂ nanotube electrode for low overpotential oxidation of formaldehyde

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Abstract

Recent researches have demonstrated that application of TiO₂ nanotubes as a support material for incorporation of metal nanoparticles causes a significant enhancement in catalytic activities of supported catalyst [1-3]. In this work, highly ordered palladium nanoparticle modified TiO₂ nanotube electrode (Pd/TiO₂NT/Ti) was prepared by electrochemical anodization of a Ti sheet, followed by photocatalytic reduction process in palladium chloride solution. The prepared electrodes were characterized by SEM, EDX, and EIS techniques. The electrocatalytic oxidation of formaldehyde at the surface of Pd/TiO₂NT/Ti was studied by cyclic voltammetry and chronoamperometry methods. Oxidation of formaldehyde was well evidenced by appearance of two well defined anodic peaks in the forward and backward scans during CV experiments. Effects of some parameters including formaldehyde concentration, scan rate, and upper potential limit on the electrooxidation performance of electrode were investigated. In comparison with some of the previously reported electrodes, the Pd/Ti/TiO₂NTs anode exhibited superior performance for electrooxidation of formaldehyde with a very low overpotential and high current density. This electrode showed a good stability during experimental and after storage for a long time in dry condition.

Keywords: Electrooxidation, TiO₂ nanotube, Formaldehyde, Voltammetry, Photoreduction

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Supercapacitive behavior of a novel polyaniline-ionic liquid derived ordered mesoporous carbon nanocomposite

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Abstract

A high-performance supercapacitor electrode consisting of novel nanocomposite of polyaniline/ ionic liquid derived ordered mesoporous carbon (PANi/IOMC) was prepared by polymerization of aniline in the presence of IOMC. Then, surface characterization and identification of nanocomposite were carried out using scanning electron microscopy (SEM), Transmission electron microscopy (TEM), fourier transform infrared spectroscopy (FT-IR), and porosimetry [1]. To fabricate the supercapacitor electrode, nanocomposite and activated carbon was sonicated in acetone, and then the resulting dispersion was coated on carbon fiber substrate using a spray-coating technique. It was found that the nanocomposite afforded a relatively high surface area for Faradic redox reactions, leading to a specific capacitance of 600 F g⁻¹ [2]. In addition, high energy and power density were obtained for PANi-IOMC nanocomposite, indicating considerable improvement compared with the pure PANi or IOMC electrodes. Moreover, good rate capability and enhanced electrochemical stability were observed for PANi-IOMC nanocomposite electrode, suggesting it as a good alternative for supercapacitor applications [3].

Keywords: Polyaniline, IOMC, Nanocomposite, Polymerization, Supercapacitor

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Molecular dynamics study of capacitance enhancement of graphene with covalently bonding polypyrrole electrodes in usage of Supercapacitors: effect of chain length of conductive polymer

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Abstract

Supercapacitors (also known as ultracapacitors) are electrochemical structures that are characterized by high power densities and high cycle lifetimes. The simplest supercapacitor is composed of two symmetrical electrodes that are separated by a porous substrate and an electrolyte. Graphene-based electrodes have been widely tested and used in electrochemical double layer capacitors due to their high surface area and electrical conductivity. Recent research efforts have been focused on improving the energy density of supercapacitors by exploring novel electrode materials. Conducting polymers (CPs) can store charges not only in the electrical double layer (EDL) but also through the rapid faradic charge transfer (pseudocapacitance). As a result, the specific capacitance of CP electrodes is higher than that of EDL capacitors based on carbon electrodes. However, one of the drawbacks for CPs as supercapacitor electrodes is their poor cycling stability because CPs is usually brittle and weak in mechanical strengths. The synergistic interaction induced by the growth of p-type polypyrrole on the surface of negatively charged carboxylate functionalized graphene sheets results in higher storage capacity than graphene-only or polymer-only films. The high conductivity of p-doped polypyrrole and high surface area of graphene promotes high charge accumulation in capacitors. In our computational study the microstructure and capacitance of the double layers forming near graphene with covalently bonded polypyrrole electrodes were calculated using classical molecular dynamics. MD simulations were carried out using the Gromacs 4.5 package. In this work, we employed all atoms force field in the frame of OPLS-AA (Optimized Potential for Liquid Simulations/All Atom), and developed it using quantum calculations to parameterize polypyrrole in order to determine the microstructure formed on the graphene electrodes. We employed $34.18 \times 34.53 \times 100 \text{ \AA}^3$ supercells corresponding to 2 graphene sheets of size $34.18 \times 34.53 \text{ \AA}^2$ (which is corresponding to 448 C atoms). We first ran MD at 1000 K for 1.2 ns, followed by 3 ns at 300 K to equilibrate the system. Production runs were carried out for 4 ns with atomic positions recorded every 4 ps. All runs were in the NVT ensemble with the temperature controlled by a Nose-Hoover thermostat with a 100 fs damping parameter. Different chain lengths of polypyrrole were attached to graphene sheets in separate simulations and the respective capacitance were calculated in different concentrations of electrolyte and optimal polymer length is obtained.

Keywords: Supercapacitor, Graphene, Polypyrrole, Molecular dynamics

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Influence of graphite/TiO₂ composite on dye sensitized solar cells efficiency

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Abstract

Dye sensitized solar cell (DSSC) was first invented by Regan and Grätzel in 1991 [1]. Recently, it has attracted considerable attention because of its low cost, convenient fabrication and high efficiency. In the conventional construction of DSSCs, the charge recombination takes place dominantly at two possible interfaces as undesirable reaction: TiO₂/dye/electrolyte and transparent conducting oxide (TCO)/electrolyte. Thus, limiting the backward electrons at these interfaces is one of the most important ways to improve the power conversion efficiency of DSSCs. For reducing backward reaction at TiO₂/dye/electrolyte interface, TiO₂ compact layer can be deposited on the TCO substrate [2]. Various methods have been investigated to achieve efficient electron diffusion and transport by employing TiO₂ nanotube/nanowire arrays, which enable channeled electron transfer so that the loss of photo induced electrons among solid TiO₂ nanoparticles can be significantly reduced. However, controlling the nanostructures of TiO₂ to enhance the electron transfer can also alter various properties of the resulting DSSCs, such as charge separation/recombination and the specific surface area of TiO₂ nanostructure-based photo electrodes. As an alternative approach, carbon material nanocomposites have been suggested to enhance the electron transfer in DSSC photo electrodes [3]. In this work, two subjects were studied to improve the DSSCs efficiency. In one hand, the effect of compact layer on the DSSCs performance and in the other hand, the impact of using composite of graphite/TiO₂ in the photo anode structures to enhance the electron transfer. In the first step, the TiO₂ compact layers were deposited on conducting glasses (FTO) substrate. This TiO₂ compact layer was formed by hydrolysis of TiCl₄ aqueous solution at a special temperature. In the next step the prepared TiO₂/graphite composite were pasted with various %wt to investigate the influence of graphite on DSSCs efficiency. The TiO₂/graphite composite was deposited on TiO₂ compact layer by Dr. Blade method. The prepared electrode was immersed in N719 dye solution for 24 h to obtain the sensitized electrode. A sandwich solar cell was assembled using dye-sensitized solar cell as working electrode and a platinum electrode as counter electrode. The 30 μm-thick thermoplastic film was used to separate the electrodes and sealed by heating. The I⁻/I₃⁻ electrolyte was injected in the gap between the electrodes through the injecting hole that was previously made in the counter electrode. The efficiency, I_{sc}, V_{oc} and fill factor (FF) of fabricated DSSCs were investigated by I-V curves. In addition, the performances of the DSSCs in detail were studied using electrochemical Impedance Spectroscopy (EIS). The obtained results showed that the TiO₂ compact layer with certain thicknesses enhances the DSSCs efficiency due to reduce charge recombination in the TCO/ TiO₂ interfaces. Since graphite have relatively high electrical, it showed an efficient charge transfer and overall increase of DSSCs efficiency as expected.

Keywords: Dye sensitized solar cell, Graphite, TiO₂, Compact layer

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Effect of deposition method and the surfactant on high capacitance of electrochemically deposited MnO_2 on Cu substrate

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Abstract

The supercapacitors have high power and energy density, very long cycle lives, short charge time, high safety and high efficiency. Recently, carbon, metal oxides and conducting polymers are used as supercapacitive materials MnO_2 as active electrode materials are currently attracting a lot of interest due to the relatively low cost, low toxicity, excellent electrochemical performance, environmentally friendly character in comparison with the ruthenium oxides or other transition metal oxides. In this paper MnO_2 has been galvanostatically deposited on Cu substrate from an aqueous acidic solution of manganese sulphate (1 M) in presence of a surface active agent (surfactant), namely, sodium lauryl sulphate (SLS), for supercapacitor applications. Electrochemical characterization was done in a three electrode configuration cell, having 0.1 M aqueous solution of Na_2SO_4 as the electrolyte. The deposits have been developed under different conditions of SLS and their specific capacitance is measured by cyclic voltammetry (CV) and also by galvanostatic charge/discharge cycle. The structure and surface morphology of the deposits have been studied by means of X-ray diffraction (XRD) analysis and Scanning Electron Microscopy (SEM).

Keyword: Supercapacitor, Manganese dioxide, Electrodeposition

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Electrochemical behavior of Electric Double Layer Capacitor (EDLC) with activated carbon from natural sources as an electrode material

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Abstract

Electrochemical double-layer capacitor (EDLC) has high power density and moderate energy density. The energy storage mechanism of EDLC is separation of charges (non-Faradaic process) at the interface between an electrode and electrolytes. In this work, the author describes the activated carbon electrodes for use in a durable EDLC for much long recycle lifetime. EDLC cells were fabricated and tested using composite electrodes of activated carbon with carbon black and PVDF as a binder and 4 M KOH solution was used as aqueous electrolyte. It was found that the proposed model fitted successfully the data of all tested cells and the cells were characterised by cyclic voltammetry, galvanostatic charge-discharge and electrochemical impedance spectroscopy (EIS). Activated Carbons (ACs) are the most commonly used electrode materials for EDLC applications due to their relatively low cost and high surface area was prepared, employing Pistachio hard skin as the raw Material. The measured capacitance of 80-135 F/g was achieved with a composite electrode film containing AC powder with a specific surface area of 1100 m²/g. Variation in the specific capacitance values with the number of cycles shows a good cycling life performance in an alkaline electrolyte.

Keywords: EDLC, Activated carbon, Cyclic voltammetry, Aqueous electrolyte

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Electrodeposition of nickel hydroxide films and its electrochemical performances for supercapacitor

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Abstract

Electrochemical capacitors (ECs), also called supercapacitors or ultracapacitors, have raised considerable attention over the past decades because of their higher power density and longer cycle life than secondary batteries and their higher energy density compared to conventional electrical capacitors. Various materials such as transition metal oxides, metal hydroxides and polymeric materials can be used in ECs for increased of their energy densities; this type of electrical storage devices so called pseudocapacitors. Ni(OH)₂ is an attractive pseudocapacitive material because of its low cost, high specific capacitance and high energy densities, and well-defined electrochemical redox activity. We prepared Ni(OH)₂ by electrodeposition method from an aqueous solution of Ni(NO₃)₂ at 25°C on a copper substrate with geometric area of 0.8 cm². This electrode was used as the working electrode, and too, saturated calomel electrode was used as reference and a Pt disc with area of 5 cm² as a counter. The electrochemical performances are investigated by cyclic voltammetry (CV) and galvanostatic charge-discharge techniques in 1 M KOH solution at 25°C. The structure and morphology of products were characterized using X-ray diffraction (XRD) and scanning electron microscopy (SEM). The results showed that the Ni(OH)₂ with particle-like morphology has excellent electrochemical performances and its specific capacitance value as single electrode.

Keywords: Supercapacitor, Electrodeposition, Nickel hydroxide, Cyclic voltammetry

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Enhancing the capacity of graphene-based supercapacitors by functionalization of graphene with monovalent functional group (-CH₃, -H, -OH, -NH₂ and -COOH): a theoretical study

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Abstract

Supercapacitors is one of the most promising energy storage devices in the field of electrical energy. Graphene and its derivatives are the most commonly used materials in Supercapacitors electrode. However, the major limitation of this type of electrodes is their low energy density. Recently, it has been found that limitation factor in total capacitance of graphene-based Supercapacitors is their finite quantum capacitance at conventional electrolyte stability voltage range. The total capacitance (C_t) at the electrode/ionic-liquid interface is given by

$$\frac{1}{C_t} = \frac{1}{C_Q} + \frac{1}{C_D} \quad (1)$$

Where C_D is the electric double layer capacitance and C_Q is the quantum capacitance, which given by

$$C_Q(V) = e^2 \int_{-\infty}^{+\infty} D(E) F_T(E - \mu) dE \quad (2)$$

Where $D(E)$ is the electron density of state (DOS), $F_T(E)$ is the thermal broadening function, E is the relative energy with respect to the Fermi level E_F , and e is the elementary charge. In this work, we used of density functional theory calculations to explore the effect of the functionalization of graphene with some substituted groups on the electronic structure and consequently on the quantum capacitance of graphene. Substituted groups that were used contain -CH₃, -H, -OH, -NH₂, and -COOH. The full geometry optimizations and density of state calculations were carried out by the ESPRESSO code. We consider the hexagonal 3×3 supercell with one functional group and 25 Å a vacuum gap in the vertical (z) direction to separate the graphene system from its periodic images. Obtained results show that this type of functionalization shows one peak near the Fermi level at the plot of DOS. The impurity DOS that arises around the Fermi level is a direct result of produced local sp^3 hybridization. Inspection of projected DOSs suggests that p_z orbital of carbon atom that substituted group connected to it, contributes to the impurity states near the Fermi level. Our results demonstrate that the quantum capacitance and consequently stored charge in functionalized graphene with each substituted group has increased compared to the pristine graphene. This enhancement in quantum capacitance is directly related to additional availability of states near the Fermi level. In this type of functionalization of graphene, the capacitance is symmetrical around zero voltage. In other word, the use of these materials in both positive and negative electrodes is suggested.

Keywords: Supercapacitors, Quantum capacitance, Functionalized graphene

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Evaluation of the effect of structural defects on the nitrogen-doped graphene electrode on the quantum capacitance**Ehsan Targholi^{*}, S. Morteza M. Khoshdel,^{*} Mohammadsafi Rahmanifar[†] and Hamideh-sadat Dashtmazari[‡]**^{*}*Industrial Electrochemistry Lab, Department of Chemistry,
Iran University of Science and Technology, Tehran, Iran*[†]*Faculty of Basic Sciences, Shahed University, Tehran, Iran*[‡]*Department of Chemistry, Alzahra University, Tehran, Iran**Corresponding author E-mail: mmousavi@iust.ac.ir***Abstract**

Supercapacitors have attracted a great deal of attention as an electrical storage device. The main materials that used in supercapacitor electrodes are carbons, metal oxides, and conducting polymers. Graphene-based material plays an important role in supercapacitor electrode materials thanks to their low production cost, high surface area and excellent conductivity. However, there is a major limitation of low energy density in graphene-based electrode compared to other material such as metal oxides. Recently, scientists have found that this limitation is due to the quantum capacitance. In fact, the total capacity of supercapacitors considered as the sum of two series capacitance of double-layer capacitance and electrode capacitance (quantum capacitance). Thus, Deficiencies in each of them will make a reduction in total device capacitance. In this paper, we explored the effect of nitrogen doping and structural defect as simultaneously on quantum capacitance. The mixed systems that we used are N-doped graphene with some structural defects such as Divacancy (5-8-5), Monovacancy (5-9) and Stone-Wales defect. Density functional theory (DFT) calculations were performed within the plane-wave pseudopotential formalism, as implemented in the Quantum-ESPRESSO code. Integrated quantum capacitance is given with equation 1.

$$C_q^{int}(V) = \frac{1}{V_e} \int_0^V C_q(V') dV' \quad (1)$$

Where, C_q is the differential quantum capacitance and V is the voltage that referenced to the zero-bias Fermi level. Our results show that good enhancement has been reached with doping and making structural defect in graphene sheet as an individually or simultaneously. This Significant enhancement is mainly due to the additional impurity states near the Fermi level. In such a mixed system, each type of mixed systems alter the electronic structure and create unique DOS and subsequently quantum capacitance. Exploring of projected DOS suggest that p_z states near the defect and p orbitals of heteroatom (N) contribute to the induced impurity states. In practice, a fabricated N-doped graphene electrode may have many types of this mixed system, which is outside the range of this work. Our findings suggest that, under charging conditions, these mixed systems will be more effective at positive bias. These results represent design strategies for improving the performance of supercapacitors.

Keywords: Supercapacitors, Nitrogen doped graphene, Quantum capacitance

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Dewatering of Activated Sludge Sewage by Electro-flotation: Optimization of Operating Parameters and Energy Consumption

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Abstract

Common methods for dewatering of wastewater sludge because of the colloidal and jelly property of bacteria cells of sludge need chemicals and high energy consumption and have low efficiency and environmental acceptability. Furthermore, their operating is completely complicated [1]. In this study, the electro-flotation way, as a novel method, was performed to cover these limitations. The dewatering performance via the electro-flotation occur under two mechanisms: first, by employing electrical current density to the system, water electrolyze happens and as a result micro-bulbs of O₂ and H₂ are created. Second, the micro-bulbs are trapped among particles and sludge flocs and then they are floated [2]. The objective of this work was to study the sludge dewatering of the wastewater treatment plant of Kermanshah City through the electro-flotation measure and investigate operating parameters in-detail. In order to carry out the experiments, a batch reactor with the effective volume of 450 ml was used. In this laboratorial research, the effect of pH (2.0-10.0), current density (0.85-3.4 mA/cm²), operating time (15-60) and surface/volume (S/V) (178, 260 and 349 cm²/L) changes on the amount of dewatering and the amount of total solids in both the sludge cake (dewatered sludge) and the water separated from the sludge as well as the content of TSS, TS and COD in the water were investigated. The amount of consumed electricity was calculated and lowered by optimizing the variables. The amount of sludge dewatering was increased by increasing operating time and current density. Of course, optimization of these parameters is vital due to prevention of extra energy consumption and high costs. In one side S/V increase raises the amount of dewatering and on the other side at high S/V amounts in a same distance breed a decrease in applied voltage amount and consequently leads to low energy consumption. The finding showed that the best efficiency (81.6%) of sludge dewatering was achieved at pH = 2, current density = 1.7 mA/cm², operating time = 30 min and surface/volume amount = 178 cm²/L with electrical energy consumption of 0.33-0.726 kWh/m³. As a whole, it can be said that the electro-flotation has more efficiency in comparison with common techniques in sludge dewatering, particularly sludge's which are hardly dewatered. This method can be raised as an alternative in wastewater plants for dewatering and volume reduction of sludge because of simple operation and maintenance, environmental accessibility, lack of adverse by-products in the effluent and economical considerations [2, 3].

Keywords: Dewatering, Electro-flotation, Sludge, Wastewater

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Optimization of operating parameters for azo dye removal from wastewater by monopolar Electro-coagulation

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Abstract

Azo dyes contains the azo group ($-N=N-$) which connects to aromatic rings and forms different groups of the azo dye like monoazo, diazo and polyazo. The discharge of wastewaters containing well-untreated dye results in aesthetic problems and an increase in gases solubility which causes light transmission inhibition into water bodies. Moreover, these compounds are toxic and mostly non-biodegradable [1]. By contrast, the electrochemical processes on account of high versatility, high efficiency and eco-friendly properties are more acceptable [2]. In the current study the removal of azo dye Acid Red 18 (as a model) and chemical oxygen demand (COD) from synthetic wastewater by electro-coagulation (EC) process were investigated. In the EC process, the coagulants (monomeric soluble hydroxides or insoluble metal ions like $Al(OH)_3(s)$) are generated in situ by electrolytic oxidation of an appropriate anode material. In this process, charged ionic species—metals or otherwise—are removed from sewage by allowing it to react (i) with an ion having opposite charge, or (ii) with floc of metallic hydroxides generated within the effluent[2-3]. In order to reach the best conditions of the process, the influence of various operating parameters such as pH (3.0, 5.0, 7.0, 9.0 and 11.0), current density (CD) ($0.2-20 \text{ mA/cm}^2$), operating time (0–120 min) and electrode type (iron and aluminum) on the removal rate of chemical oxygen demand (COD) and color were investigated. EC reactions were done in a batch reactor, made of pyrex glass material, with 0.45 dm^3 of effective volume. 3 anodes and 2 cathodes with dimensions of $10 \times 5 \text{ cm}$ and 2 cm of electrode gap were applied; the electrodes were placed as monopolar in parallel connections. It was found that the process had a very good efficiency in the removal of both COD and color; in the case of iron electrode, the maximum amounts of color and COD removal were 99.5% and 59.0%, respectively. The operating time of 45 min, $\text{pH} = 7$ and $\text{CD} = 11.76 \text{ mA/cm}^2$ were selected as the optimized points. By increasing CD up the optimum amount (11.76 mA/cm^2), the efficiency increased. The optimization of variables is extremely crucial as it results in a decrease in costs, energy and electrode consumption. As a whole the iron electrode used lower energy than the aluminum electrode. Although the amount of corrosion of the iron electrode is higher than that of the aluminum electrode, the electrode of iron is more acceptable owing to economical reasons. The findings of UV/vis spectra illustrated the structures of this dye is removed by the process. In comparison with traditional methods like aerobic and anaerobic systems, the EC process is a suitable alternative for treatment of wastewaters containing dye pollutants.

Keywords: Acid Red 18, Azo dye, COD and color removal, Electro-coagulation, Wastewater treatment

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Continuous electrochemical oxidation of azo dye C.I. Acid Red 18 by using Pb/PbO₂ electrodes: optimization of operating parameters and voltammetry study

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Abstract

Azo dyes constitute 60–70% of synthetic dyes in the textile, food, rubber, color solvents, wool, leather, paints plastic, medicine, and cosmetic industries, have lately become a big concern because of both mutagenic and carcinogenic impacts [1, 2]. Although azo dye-containing wastewaters treatments through physicochemical and biological methods have benefits, such processes produce toxic by-products and huge amounts of sludge which must be disposed safely and requires a lot of oxidant chemicals. These methods are also quite expensive, energy consuming and have hard operating conditions [1-3]. The prime objective of this work was to study the application of anodic oxidation for AR18 degradation. In this study, we tried to use a single system with continuous flow without adding any chemicals. Furthermore, so as to decline the investment cost, inexpensive electrodes were applied. The flow rates of the pilot plant were in the range of 0.42–4.5 cm³/S. Nine monopolar rods of Pb/PbO₂ electrodes were placed per each cathode and anode with dimensions of 2 × 35 cm. The distance between electrodes (1.2 cm) and effective electrode area (0.23 m²) were fixed in all experiments. In order to reach the best conditions of the process, the influence of various operating parameters such as HRT ($t = 20\text{--}200$ min), CDs ($i = 1.7\text{--}15.5$ mA/cm²), initial pH values (2–9), and AR18 concentrations ($C = 0.17\text{--}1.7$ mM) on the removal rate of chemical oxygen demand (COD) and color were investigated as functional of degradation and decolorization of the dye. The findings showed that, respectively, 99.9% and 80.0% of color and COD were removed (at optimized conditions; CD = 8.6 mA/cm², HRT = 120 min) that could be due to the contribution of multiple oxidation mechanisms especially oxidation of organic matters in solution via HO[•]s which is generated from water oxidation. Dye decolorization happens more quickly than COD removal. At the CD lower than the optimum point (8.6 mA/cm²), mineralization current efficiency was high on the condition that HRT was high, but at CDs higher than the optimum point there was not a significant growth in the efficiency and it led to energy consumption increase. By controlling HO[•]/dye concentration ratio via parameters adjustment, particularly HRT and CD, this system can treat AR18 at high concentrations. Furthermore, the voltammetry study indicated that electroactive intermediates which were created during the process were mineralized. Consequently, unlike conventional methods, anodic oxidation does not need other supplementary processes and chemicals. The system does not lead to by-products formation and has a better efficiency at a low detention time.

Keywords: Azo dye Acid Red 18, Anodic oxidation, Constant current electrolysis, Wastewater treatment

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Design and construction of a new electrochemical discharge micromachining system

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Abstract

Electrochemical discharge micromachining is nonconventional method for machining of nonconductive materials. Electrochemical technique is often adapted to machine based on both with the electrical discharge and electrochemical methods. This method involves a two-electrode system that, in this study, cathode electrode is thinner than the anode. The electrochemical discharge therefore occurs between cathode and nonconductive material under optimized condition [1]. The main advantages of the electrochemical method are that firstly, this system is able to machine nonconductive materials such as glass, ceramic and so on, and secondly, this system is considered as a one-step process as compared to the conventional methods [2]. However main problem of this method is that, compared to other methods, dimension of its machining is high owing to i) high temperature of discharge and ii) the length of gas film that formed the around of cathode [3]. To solve these problems hereby in this study, a simple, novel and precise electrochemical discharge micromachining has been designed, which is optimized via controlling some physical and chemical parameter such as kinds and amounts of electrolyte solution, the waveform of the electrical potential applied to the electrode system, the temperature of the electrolyte, size, shape and dimension of a micro-probe as working electrode and finally influence of surfactants.

Keywords: Electrochemical discharge micromachining system, Nonconductive materials

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Comparative study of Chalcopyrite and Chalcocite behaviors in mono and polymetal environment in buffer and pulp solution using cyclic voltammetry

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Abstract

Chalcopyrite and Chalcocite are the main and important copper sulphide minerals. Due to electrochemical characteristic and semiconductive property be able to have electron transfer with others minerals and process environment. A comparative study of electrochemical behavior of chalcopyrite, chalcocite in mono and poly metal environment in buffer and pulp solution .cyclic voltammetry carry out on the mineral electrodes made from mono mineral crystal and poly mineral sample take from Sarcheshme copper mine and mineral processing plant. Voltagrams of chalcopyrite and chalcocite in monomineral environment in pH=12 and Eh= -255mv (by Ag/AgCl/KCl reference electrode) showed the oxidation reaction on the surface of chalcopyrite and chalcocite. By reduce the pH to 11 and Eh to -225 mv, chalcopyrite also active but chalcocite not. By changing in environment condition, in pulp solution, not only in pH=12 and Eh= -275 mv chalcopyrite and chalcocite was active but also in pH=11 and Eh=-225 was active.

Keywords: Cyclic voltammetry, Chalcopyrite, Chalcocite, Electrochemical behavior of chalcopyrite, Electrochemical behavior of Chalcocite

Membrane Chlor-alkali Electrolysis with Oxygen Depolarized Cathode:” Green Technology”

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Abstract

The brine electrolysis of chlorine and caustic soda production is one of the most energy-intensive industrial processes, with electricity accounting for approx. 50% of the variable production costs. It is accepted that the developed-membrane chlor-alkali technology has reached the theoretical end-point on energy consumption. Attempts to reduce manufacturing costs of chlor-alkali technology have recently led to modifications of the conventional membrane electrolyzes for further optimization of the energy savings. Replacing the conventional hydrogen-evolving cathode by an oxygen-depolarized cathode in a chlor-alkali membrane cell reduced the cell voltage and, consequently, lead to a 30% energy consumption reduction at 4 kAm^{-2} . In this study, we have reviewed chlor-alkali technology and its development. The overall cell performance and stability is dominated by the behavior of the cathode, leading to a focus of research effort on cathode development. The impact of electrocatalysts to bring about high activity at lower cell voltages and stable oxygen-depolarized cathode performance over the period of time is of utmost importance. A review of the latest material and preparation method in oxygen depolarized cathode is given. The application of different electrocatalyst cathodes and its performance comparison is studied. Also, we have considered replacement of chlor-alkali cell with oxygen depolarized cathode in energy saving and environmental aspects.

Keywords: Chlor-alkali, Oxygen-depolarized cathode, Saving energy, Electrocatalyst

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Design of Multi-mode Electrochemical Pulse System for Used in Synthesis of Nanostructured Materials Based on PLC Programming

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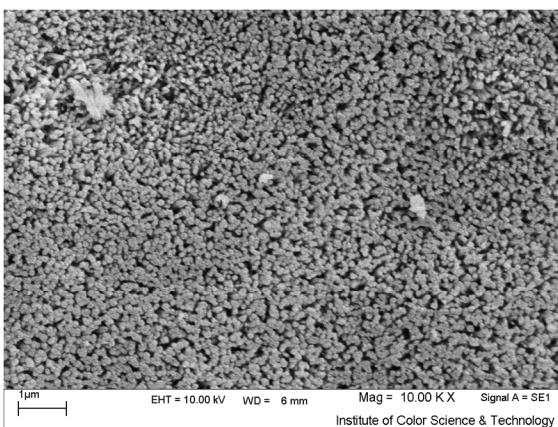
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Abstract

Multi Channel Potentiostats/Galvanostats system was manufactured. In this system, control of potential and current was done using Siemens PLC programming where multi-mode electrochemical deposition including direct, pulse and pulse reverse current/potential modes can be run. Also, ramping the pulse time at constant potential or current modes, ramping both pulse time and potential, and Triangular potential mode are also applicable in this system. HMI programming using PM-Designer software were done for communicate operator and PLC. Using this system, pulse cathodic electrodeposition of cathodic electrodeposition of $\text{La}(\text{OH})_3$ from lanthanum nitrate bath was performed for the first time. The deposition experiments were conducted at the pulse current mode with a typical on-time and off-time ($t_{\text{on}}=5\text{s}$ and $t_{\text{off}}=10\text{s}$) and an average current density of 1 mA cm^{-2} ($I_a=1 \text{ mA cm}^{-2}$) for 50 min. The prepared deposit was then heat treated at 600°C for 3h in dry air atmosphere. The products were characterized by XRD, CHN, FT-IR and SEM techniques. The results revealed that the large scale and vertically aligned one-dimensional (1D) $\text{La}(\text{OH})_3$ and La_2O_3 nanorods have been prepared at the applied conditions. It was concluded that large-scale, uniform and vertically aligned nanorods of $\text{La}(\text{OH})_3$ and La_2O_3 are achievable by pulse electrodeposition followed by heat-treatment.



Potentiostats/Galvanostats system



SEM image of the prepared nanorods

Keywords: Potentiostats/Galvanostats system, PLC programming, Pulse/pulse reverse ramp, Cathodic electrodeposition, $\text{La}(\text{OH})_3$, La_2O_3 nanorods

Preparation of nanostructured manganese dioxide by cathodic electrodeposition and study of its supercapacitive behavior

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Abstract

In this study, different morphologies of nanostructured manganese dioxide was prepared by cathodic electrodeposition from aqueous solution of $\text{Mn}(\text{NO}_3)_2$ on steel substrate and followed by heat treatment. This work were done by suitable selection of synthesis parameters consist of applied current density, electrolyte concentration, PH, bath temperature, heating temperature and time. The morphologies of the as-synthesized products were examined by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). X-ray diffraction (XRD) and Fourier transform infrared (FT-IR) spectroscopy were also employed for characterization of the nanostructures. Analysis results showed that the prepared sample has tetragonal hausmannite crystal structure with rod morphology. The electrochemical performance of the sample was evaluated by cyclic voltammetry (CV) in a three electrode electrochemical system at the constant current. A maximum supercapacitance of 335.4 F g^{-1} was obtained at the scan rate 10 mV s^{-1} . An excellent coulombic efficiency of 92% was calculated during 500 cycles at 10 mV s^{-1} .

Keywords: Nanostructured manganese dioxide, Cathodic electrodeposition, Supercapacitive behavior

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Drug delivery by titanium dioxide nanotubes

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Abstract

Drug delivery is an interdisciplinary and independent field of research that is gaining the attention of pharmaceutical researchers, medical doctors and industry. A safe and targeted drug delivery could improve the performance of some classic medicines already on the market, and moreover, will have implications for the development and success of new therapeutic strategies such as anticancer drug delivery, peptide and protein delivery and gene therapy. Nanotubes (NTs) have been developed as an important strategy to deliver conventional drugs, recombinant proteins, vaccines and more recently, drug-delivery systems modify the kinetics, body distribution and drug release of an associated drug. This research focuses on the potential of nanotechnology in medicine. Titanium dioxide nanotube arrays (TNT) used as drug-carriers was prepared by self-ordering electrochemical anodization [1]. Bacterial infection, extensive inflammation and poor osseointegration have been identified as the major reasons for orthopaedic implant failures based on titanium. Creating implants with drug-eluting properties to locally deliver drugs is an appealing way to address some of these problems. To improve properties of titanium for orthopaedic applications [2]. Nanotube arrays on titanium surfaces were fabricated by anodization methods and their potential for storage and release of drugs was evaluated. The treatment of the Ti surfaces in NH_4F /ethylene glycol electrolyte led to the formation of TiO_2 nanotubes. The prepared TNT-Ti substrates were loaded with drug via a simple method, putting drug solution and drying it [3]. Drug-release experiments with the model antibiotic ciprofloxacin. Significant surface-dependent differences in the release kinetics of ciprofloxacin were observed. The morphological and chemical structures of TiO_2 films were characterized using scanning electron microscopy (SEM). The amount of the drug was determined by uv-vis spectroscopy. There was a linear relation between absorption and concentration of the drug. All the measuring was determined in phosphate buffered saline.

Keywords: Drug delivery, Titanium nanotubes, Ciprofloxacin

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Carbon quantum dots and polyalizarin yellow R modified glassy carbon electrode for nanomolar detection of L-cysteine

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Abstract

A novel electrochemical sensor for the detection of l-cysteine was proposed based on immobilizing poly (alizarin yellow R)/carbon quantum dots on glassy carbon electrode. Hydrothermal treatment was used to prepare carbon quantum dots (CQDs). Transmission electron microscopy (TEM) and FTIR were used for characterization of carbon quantum dots. Electrochemical impedance spectroscopy, cyclic voltammetry (CV) and amperometry were used to confirm the successful stepwise assembly procedure of the sensor. The electrocatalytic behaviors of the sensor were also investigated by cyclic voltammetry and amperometry. Results showed that poly (alizarin yellow R)/carbon dots exhibited a remarkable electrocatalytic activity for the oxidation of l-cysteine under optimal conditions. The electrocatalytic response of the sensor was proportional to the l-cysteine concentration in the range of (0.3 to 3.6 μM) and (3.9 to 7.2 μM) with a limit of detection and sensitivity of 90 nM and 0.482 $\mu\text{A}/\mu\text{M}$, respectively. The modified electrode shows many advantages such as simple preparation, high sensitivity, low detection limit, excellent catalytic activity at physiological pH values, short response time, and remarkable antifouling property toward l-cysteine and its oxidation product.

Keywords: Carbon dots, l-cysteine, Alizarin yellow R, FTIR, Amperometry

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Electrochemical preparation of aligned $\text{La}(\text{OH})_3$ and La_2O_3 nanorods: pulsed cathodic deposition followed by heat-treatment

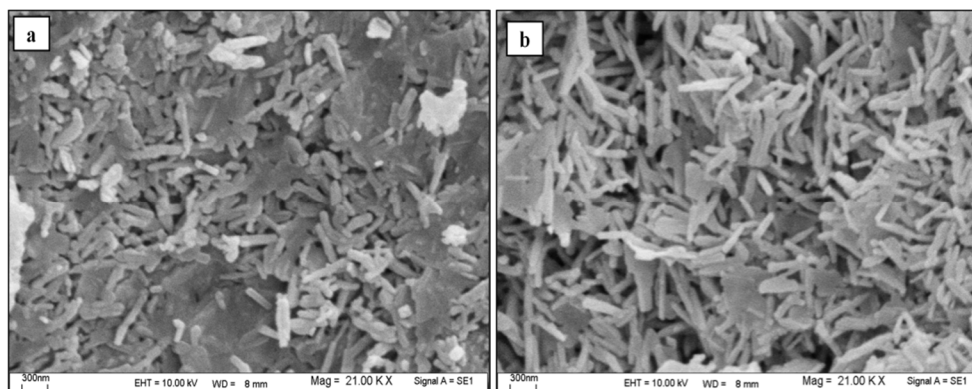
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Abstract

$\text{La}(\text{OH})_3$ was deposited from nitrate bath via pulsed cathodic electrodeposition at a typical on-time and off-time ($t_{\text{on}}=10$ ms and $t_{\text{off}}=40$ ms). The obtained deposit was then heat treated at 600 °C for 3h in dry air atmosphere. The structural and morphological characterizations *via* XRD, CHN, FT-IR and SEM techniques revealed that one-dimensional (1D) $\text{La}(\text{OH})_3$ nanorods have been prepared at the applied pulse conditions. It was also observed that heat-treatment of the prepared $\text{La}(\text{OH})_3$ nanorods promotes the formation of La_2O_3 nanorods without any change in their morphology. Mechanism of the formation and the growth of $\text{La}(\text{OH})_3$ nanorods was proposed on base of the hydrogen bubbles dynamic template. The results showed that horizontally aligned nanorods of $\text{La}(\text{OH})_3$ and La_2O_3 can be easily prepared by the pulse deposition followed by heat-treatment.



SEM images of the prepared (a) $\text{La}(\text{OH})_3$ and (b) La_2O_3 nanorods

Keywords: $\text{La}(\text{OH})_3$, La_2O_3 , Nanorods, Electrodeposition, Heat-treatment

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Fabrication of nano catalyst by coelectrodeposition of Pt-Cu on fluorine doped tin oxide glass (FTO) for methanol sensing.

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Abstract

In this work, we report a technique for fabrication of nano catalyst for methanol sensing. Methanol is very useful organic solvent with wide range of applications and toxicity [1], it made the needs of reliable and selective methanol sensors very crucial. It was observed that the kinetics of methanol oxidation reaction (MOR) was slow; therefore electrodeposition of nano sized noble metals such as Pt on FTO glass as oxidation nano catalyst was investigated. Due to very high price of platinum and the fact that CO and CO₂ which are intermediates of MOR can poison catalyst surface, development a catalyst consisting of Pt-Cu nanoparticle on FTO surface for methanol oxidation reaction [2]. This catalyst was deposited in sulfuric acid solution containing K₂PtCl₆ and CuSO₄.5H₂O at room temperature by cyclic voltammetry [3]. A response surface design (RSD) was employed to optimize experimental factor of voltammetric electrodeposition effecting response of MOR. The limit of detection calculated for sensor was 9.6 ppm.

Keywords: Nobel metals, Coelectrodeposition, FTO, Methanol, Cyclic voltammetry, Sensor

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Electrochemical Modification of glassy carbon electrode with carbon nanotubes and dianix blue as a novel and efficient voltammetric sensor for simultaneous determination of levodopa, uric acid and ascorbic acid

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Abstract

There are high attractions in the development of non-conducting polymer (CP) coatings to improve the electrochemical properties and biocompatibility of electrodes in the area of biosensors [1]. For the first time, simultaneous determination of levodopa (LD), uric acid (UA) and ascorbic acid (AA) has been investigated at a glassy carbon electrode modified with multi-walled carbon nanotubes and dianix blue (GCE-MWCNT/DB). For MWCNT/DB electropolymerization on the GCE, the potential was swept 40 cycles between -0.2 and 1.8 V at scan rate 100 mV s^{-1} . In this work, the electrocatalytic properties of the GC-MWCNT/DB toward the electrocatalytic oxidation of LD, UA and AA were studied using cyclic voltammetry (CV) and differential pulse voltammetry (DPV) [2]. The GC-MWCNT/DB modified electrode offered substantially lower overpotential for electro-oxidation of LD, UA and AA in phosphate buffer solution (pH 3.0) compared with bare GCE. The results of CV and DPV investigations showed a considerable enhancement in the anodic peak current of LD associated with decreasing about 180 mV in the corresponding peak potential. Furthermore, high reproducibility as well as low detection limit of the electrode responses can be considered as significant features of the prepared modified electrode [3].

Keywords: Modified glassy carbon electrode, Multi-walled carbon nanotubes, Simultaneous determination, Levodopa, Ascorbic acid, Uric acid.

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Design and Construction of an Electrochemical Nano-Biosensore to Measure Phenylalanine

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Abstract

Phenylalanine is an essential amino acid. Body cannot synthesize it but needs it for health therefore it has to obtain it from food. Phenylalanine is a precursor for tyrosine, the catecholamine signaling molecules dopamine, norepinephrine and epinephrine and the skin pigment melanin. Symptoms of phenylalanine deficiency include confusion, depression, memory problems. A rare metabolic disorder called phenylketonuria (PKU) occurs in people who are missing an enzyme that the body needs to utilize phenylalanine. This causes high levels of phenylalanine to build up. If it is not treated before 3 weeks of age, PKU can cause severe, irreversible intellectual disability. There are many problems with conventional diagnostic methods used for measurement of phenylalanine in neonatal blood, like low sensitivity and elaborate technique which are time consuming and often associated with false positive or negative results[1]. Biosensors have the ability to detect biological molecules with high sensitivity and low cost. Molecularly imprinted polymers (MIP) have properties such as good selectivity, high sensitivity, long stability and neutrality in chemical solutions. They can also be easily synthesized for most analytes. All these make them suitable for sensor construction. This project is an attempt to use the MIP-based potentiometric electrodes to measure phenylalanine molecule [2]. At optimized conditions the electrode exhibited a Nernstian response (29.528 ± 1.0 mV decade⁻¹) in a concentration range of 1×10^{-8} to 1×10^{-3} M with detection limit of 5×10^{-9} mol L⁻¹. The potential response of the electrode was constant in the pH range of 4.0–7.5. The electrode demonstrated a response time of ~35s. The selectivity coefficient of the sensor towards a number of different Amino acid with molecular similarities and some metal ions was evaluated. In the present work a novel and selective potentiometric sensor, based on a noncovalent imprinted polymer, was developed for determination of phenylalanine.

Keywords: Molecularly imprinted polymers, Phenylalanine

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In-situ preparation of CdS Quantum dots and their application in modified carbon paste electrodes

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Abstract

Nanostructure semiconductors, that are named quantum dots (QDs), are include elements from the periodic groups II-VI, III-V or IV-VI; they are roughly spherical and their size is typically in the range of 1-12 nm diameters. These semiconductor nanocrystals exhibit size-dependent, molecular-like discrete electronic and optical transitions properties due to quantum confinement effects. Because of these properties, QDs have attracted the attention of great number of researchers [1]. Electrochemical methods have been applied in sample analysis and organic and inorganic synthesis because of their low cost, ease of use, and reliability [2]. Recent developments in electroanalytical chemistry have been resulted in advances in sensor design, chemical modification, and enhanced sensitivity and selectivity of electrodes. Recently, chemically modified electrodes have been widely used for sensitive and selective detection of trace amounts of important biological compounds such as ascorbic acid (vitamin C, AA) and amino acids such as Tryptophan [2]. In recent years, electrochemical sensors based on nanoparticle modified electrodes have been widely used for detection of many biological and chemical compounds. In this paper, CdS quantum dot modified carbon paste electrode (CdS/CPE) has been used for determination of aminophenol compounds with good selectivity. The CdS quantum dots were prepared according to a new in-situ method, according to following procedure: 50 ml aqueous solution containing $\text{Na}_2\text{S}_2\text{O}_3$ (0.01 M), thioglycolic acid (0.05 M) and CdSO_4 (0.05 M) at pH=10 (using NaOH) were heated at 348 K for 1 h, under vigorously stirring. As a result, a yellow solution containing CdS QDs was prepared and stored for next investigations. The quantum dots were precipitated with ethanol, centrifuged, washed with ethanol and dried in a vacuum. The prepared QDs powder is highly soluble in water. The modified electrode has been fabricated based on the procedure described elsewhere [3]. The electrodes were smoothed on a piece of soft filter paper for obtaining smooth surface before the measurement. The results demonstrated that the CdS/CPE exhibited a strong electrocatalytic performance and good selectivity for aminophenol compounds in the presence of other phenolic compounds. As a result, the proposed method can be used for determination of aminophenol compounds in pharmaceutical and environmental samples.

Keywords: CdS quantum dots, Modified carbon paste electrode

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Electrochemical behavior and voltammetric determination of efavirenz at glassy carbon electrode modified with carbon nanotubes

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Abstract

Efavirenz (EFV) is a non-nucleoside reverse transcriptase inhibitor (NNRTI) and is used as part of highly active antiretroviral therapy for the treatment of human immunodeficiency virus (HIV) type 1 [1]. Several analytical methods have been developed for the determination of efavirenz, but only two electroanalytical works were reported to date [2, 3]. In this work, the voltammetric behavior of efavirenz was studied at a glassy carbon electrode modified with carbon nanotubes. The cyclic voltammetric results indicated that a significant increase in the voltammetric response was achieved at the modified electrode compared to a bare glassy carbon electrode. The modified electrode presents excellent selectivity and sensitivity toward oxidation of EFV. The measurements were carried out by application of differential pulse voltammetry (DPV) and cyclic voltammetry (CV) methods. The parameters which influence the determination of EFV have been investigated. Application of DPV method showed that under optimized conditions, linear calibration graph was obtained in the range from 15 μ M to 0.75 mM, with a detection limit of 3 μ M. The electrode has been applied to the assay of EFV in human blood serum with good results.

Keywords: Carbon nanotube, Efavirenz, Modified glassy carbon electrode

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Application of a carbon paste electrode modified with electrospun zinc oxide nanofibers for simultaneous voltammetric determination of ascorbic acid, uric acid and folic acid

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Abstract

An electrochemical method for simultaneous determination and direct electro-oxidation behaviors of ascorbic acid (AA), uric acid (UA) and folic acid (FA) was developed at a zinc oxide nanofibers modified carbon paste electrode (ZNFs/CPE). Scanning electron microscopy and transmission electron microscopy techniques were used to examine the structure of the zinc oxide nanofibers [1]. The measurements were carried out using cyclic voltammetry in citrate buffer solution with pH 5.5. This modified electrode exhibits potent and persistent electro-oxidation behavior followed by well-separated oxidation peaks toward AA, UA and FA with increasing the oxidation current. The electrochemical behaviors of AA, UA and FA on the modified electrode were carefully studied by cyclic voltammetry and the electrochemical parameters such as the area of the electrode (A), the electron transfer number (n), the electron transfer coefficient (α), diffusion coefficient (D) and the electrode reaction standard rate constant (k_s) were calculated [2]. Differential pulse voltammetry was proposed for simultaneous determination of these three analytes. Under the selected conditions the oxidation peak currents were proportional to AA, UA and FA concentrations in the range from 0.1 to 80 μM , 0.4 to 100 μM and 0.2 to 90 μM respectively with the detection limits as 0.013, 0.028 and 0.036 μM (3σ), orderly. The proposed method showed good selectivity to the AA, UA and FA detection without the interferences of coexisting substances.

Keywords: Electrospinning, ZnO nanofibers, Uric acid, Ascorbic acid, Folic acid, Voltammetry

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Voltammetric determination of chlorpromazine based on polyPyrrole/graphene oxide nanocomposite modified glassy carbon electrode

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Abstract

In this paper, for the first time, a simple and sensitive electrochemical sensor was developed for voltammetric determination of chlorpromazine based on polypyrrole/graphene oxide (PPy/GO) nanocomposite modified glassy carbon electrode. Polypyrrole has been successfully grown on the surface of graphene oxide nanosheets by using a facile electropolymerization method. The electrochemical response characteristics of the modified electrode toward the oxidation of chlorpromazine were investigated using cyclic voltammetry, differential pulse voltammetry and chronoamperometry. The PPy/GO nanocomposite exhibits high electrical conductivity, large surface area and high electrocatalytic activity for the electrochemical oxidation of chlorpromazine. At the optimum pH of 6.0 in 0.1 M phosphate buffer solution, differential pulse voltammograms increase linearly with chlorpromazine concentration over the concentration ranges of 1 nM – 5 nM. The detection limits for chlorpromazine was found to be 0.6 nM. The proposed method was also successfully applied to the determination of chlorpromazine in real samples.

Keywords: Chlorpromazine, Voltammetric determination, Graphene Oxide, Polypyrrole, Nanocomposite

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Construction of a modified graphene oxide nanao sheets paste electrode for voltammetric determination of methyldopa and folic acid in biological and pharmaceutical formulation

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Abstract

Methyldopa is an antihypertensive agent that is used in the treatment of high blood pressure or hypertension, especially when it is complicated with renal disease. Its hypertensive proprieties are primarily due to its action on the central nervous system. Methyldopa inhibits the enzyme DOPA decarboxylase, which converts L-DOPA into dopamine and is a precursor for norepinephrine and subsequently epinephrine. It is converted to α -methyl norepinephrine in adrenergic nerve terminals, and its antihypertensive action appears to be due to its stimulation of central adrenal-receptors, which reduces sympathetic tone and produces a fall in blood pressure [1]. Folic acid is a water soluble B vitamin. Vitamin B9 (folic acid and folate) is essential for numerous bodily functions. It is vital for red blood cells and for many other cells in the body. The form of folic acid occurring naturally in food is called 'folate'. Lack of these two vital nutrients leads to one variety of anemia called macrocytic anemia [2]. In the present study, carbon-paste electrode modified with graphene oxide nano sheets was used for the determination of methyldopa in the presence of folic acid. The cyclic voltammetry and square wave voltammetry investigations showed effective electrocatalytic activity of the modified electrode in lowering the anodic over potential for the oxidation of methyldopa and complete resolution of its anodic wave from folic acid. This sensor reduced the overpotential for oxidation of methyldopa about 200 mV. A linear dynamic range of 0.09 to 500.0 μ M for methyldopa with a detection limit of 50.0 nM was obtained.

Keywords: Methyldopa, Folic Acid, Graphene oxide nano sheets, Voltammetry

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Voltammetric determination of taxol at ds-DNA modified pencil graphite electrode and its application as a label-free electrochemical biosensor

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Abstract

Taxol, first isolated in 1971 from the stem bark of the western yew tree, is one of the most effective natural anticancer agents usually used in clinical practice for the medical treatment of ovarian, breast and non-small cell lung cancers. As its medical significance, there is an increasing demand of taxol from natural or artificial plants, chemical synthesis and biotechnology. However, the impurities similar to structure of the taxol make it difficult in isolation and identification. Clinical trials have shown that taxol has a narrow therapeutic range, and its elimination half-life varies widely among patients. High-dose taxol treatment may also cause side effects [1]. Using pencil graphite as disposable electrodes in electrochemical DNA sensing make the method easier and more rapid compared with the conventional DNA biosensors [2]. Electrochemical investigation of DNA–drug interactions can provide a rapid and inexpensive method for the determination of drugs. Electrochemical DNA biosensors comprise a nucleic acid recognition layer that is immobilized over an electrochemical transducer. The role of the nucleic acid recognition layer is to detect the changes that occurred in the DNA structure during interaction with DNA-binding molecules. So, in the recent years, growing interest has arisen in electrochemical investigations of interaction between nucleic acid binding molecules and DNA due to their promising functions and biological activities [3]. In the present study, a novel taxol DNA-biosensor was constructed based on the decrease in guanine and adenine signal intensity adsorbed at the surface of a pencil graphite electrode. A linear dynamic range between taxol concentration and ds-DNA signals was obtained in the range of 2.0×10^{-7} to 1.0×10^{-5} M with a detection limit of 8.0×10^{-8} M. Finally this modified electrode was used for determination of taxol in some real samples.

Keywords: Taxol, DNA biosensor, Pencil graphite electrode

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Application of Carbon Ceramic Electrode modified by Polypyrrole/ Reduced Graphene Oxide nanocomposite on sensitive determination of dopamine in real sample

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Abstract

Graphene with honeycomb structure has promising application due to its unique physical and electrical properties and nowadays has been applied as a sensing material in sensors that arises from its high surface area and high mobility of charge carriers [1]. Graphene because of its low solubility and high surface energy is unable to make a stable suspension and wants to be agglomerated and turns into graphitic structure [2]. In recent years, electrically conductive polymer (ECP) like polypyrrole attracted great interest because of its hybrid properties of having metal-like conductivity and polymeric characteristics such as flexibility, low density, and ease of structural modification at the same times [3]. Dopamine (DA) as one of the important neurotransmitter belongs to the family of excitatory chemical neurotransmitters. Low level of DA may cause neurological disorders such as schizophrenia and Parkinson's disease. As DA is an electroactive molecule, electrochemical methods in determination of DA are sensitive, simple, selective, fast and inexpensive. In this work we prepared a carbon ceramic electrode modified with nanocomposite of polypyrrole/electrochemically reduced graphene oxide and applied this electrode to sensitive determination of dopamine in blood serum. To deal with the problem of Graphene low solubility in polar and non-polar solvents, we oxidized graphite with modified Hummer's method and agitated with ultrasonic bath to makes Graphene oxide and increase its solubility in water. By using graphene oxide as counter ion of polypyrrole we can increase physical stability of graphene oxide on surface of electrode and using properties of individual parts simultaneously. The parameters that affect the answer of electrode toward DA, like Graphene and pyrrole concentration, pH potential range, scan rate and number of scans was investigated. Differential Pulse Voltammetry (DPV) was applied to determination of DA with good linear range of 1 to 70 μM and low detection limit of 210 nM. This electrode was successfully applied to determination of DA in blood serum.

Keyword: Graphene oxide, polypyrrole, Nanocomposite, Dopamine, Carbon ceramic electrode

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Metal paste nanoparticles modified electrode for amplified electrochemical sensing of haloperidol

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Abstract

Metal nanoparticles have unique physicochemical properties compared to their bulk counterparts, and they have wide applications in catalysis, electronics, optics, magnetism, and biomedical sciences [1]. Recently, owing to their ability to enhance the electrode conductivity and facilitate the electron transfer, silver nanoparticles, have been utilized increasingly in many electrochemical applications and construction of sensors and biosensors. Haloperidol (HL) is one of the most widely used psychotropic drugs and may be administered for a long period of time. Its major metabolite, reduced HL, may contribute to the effects of the parent drug. Therefore, measuring the concentrations of HL in biological samples is necessary [2]. In this study, a silver paste nanoparticles modified electrode (Ag-NP/ME) was used for the determination of HL. Ag-NP was characterized by scanning electron microscopy and transmission electron microscopy. Simplicity of electrode fabrication, renewability of the electrode surface, together with high electrocatalytic and adsorption effects, increased degree of active area and surface roughness, low cost and low weight are among the advantageous features of this electrode for HL detection [3]. Chronoamperometry, cyclic and differential voltammetry were used for quantitative calculations. The electrochemical parameters such as diffusion coefficient of HL, electrode surface area, electron transfer coefficient and the detection limit were calculated. The proposed sensor was successfully applied for the determination of HL in human blood serum and pharmaceutical samples.

Keywords: Metal paste, Haloperidol, Modified electrode, Voltammetry

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Comparison of corrosion resistance of Ni-W-PTFE nanocomposite coatings with Ni-W coatings in the 3.5% wt NaCl Solution

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Abstract

Nanocomposites are being greatly studied nowadays because of their enhanced magnetic, mechanical, optical and physical properties compared to those of bulk materials[1]. These Composite materials have other various special properties such as dispersion hardening, self-lubricity, high temperature oxidation resistance, excellent wear resistance and corrosion resistance. Electrodeposition is considered to be one of the most important techniques for producing composites, due to precisely controlled near room temperature operation, rapid deposition rates, low cost. Recently, a number of literatures reported SiC, ZrO₂, Al₂O₃, TiO₂ and PTFE particulates, which were added to the plating bath and could be co-deposited with metal or alloy to create composite coatings[2]. Polyfluortetraethylene (PTFE) has superior heat resistance, corrosion resistance and excellent antifriction and self-lubricating properties. Therefore, as an important matrix for preparing composite material for sliding friction part, it has been interested in recent years[3]. The deposition of tungsten with nickel has been of considerable interest due to its singular properties such as high hardness, higher melting point, lower coefficient of linear thermal expansion, high tensile strength, etc[1]. Ni-W alloy was developed as one of the surface treatments to substitute for hard chromium coating for its excellent properties, and the process is according to the consideration of environmental protection[2] and the aim of this work is to produce a coating with properties like corrosion resistant in the 3.5% wt NaCl and self-lubricating and the other great features mentioned above. In the present work, Ni-W-PTFE nanocomposite coatings were electrodeposited in the presence of different concentrations of polytetrafluoroethylene (PTFE) from the tartrate bath on the plates of copper. The effect of the concentration of polytetrafluoroethylene (PTFE) was investigated on the corrosion resistance. Surface morphology and composition of the nanocomposite coating were characterized by scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) measurements. Corrosion resistance of produced coatings were evaluated using open-circuit potential (OCP), electrochemical impedance spectroscopy (EIS) and polarization techniques in 3.5% wt NaCl solution. The results of these studies show a significant shift in the corrosion potential toward noble potentials, decrease in the corrosion current density and increase in the charge transfer resistance whit the incorporating of PTFE particles in the Ni-W matrix. While the highest charge transfers resistance for nanocomposite coatings obtained from the bath containing 8 g/L PTFE (12719 Ω cm²).

Keywords: Nanocomposite, Corrosion resistance, Electrodeposition, polytetrafluoroethylene (PTFE), Ni-W-PTFE nanocomposite, Electrochemical impedance spectroscopy

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Synthesis of silver nanoparticles in presence of pepper extract and its electrocatalytic application

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Abstract

Due to this property being distinctly different from that of the bulk metal, silver nanoparticles have attracted much attention and have found applications in diverse areas, including medicine, catalysis, textile engineering, biotechnology and bioengineering, water treatment electronics and optics [1, 2]. Furthermore, currently silver nanoparticles are widely used as antibacterial/antifungal agents in a diverse range of consumer products. The hydrogen evolution reaction (HER) is an electrochemical process that has received wide attention because of its importance in both fundamental and technological electrochemistry such as fuel cell technology [3]. In this work, we reported a simple and low-cost procedure to synthesize silver nanoparticles (AgNPs) by using pepper extract as reducing agent. The synthesized AgNPs were characterized by a variety of means such as transmission electron microscope (TEM), powder X-ray diffraction (XRD) and energy dispersive X-ray (EDAX). TEM observations and XRD analysis demonstrated that the size of AgNPs is less than 30 nm. Silver nanoparticles modified carbon paste electrode (AgNPs-CPE) displayed excellent electrochemical catalytic activities towards hydrogen evolution reaction (HER). Finally the kinetic of the hydrogen evolution reaction is also discussed on the AgNPs-CPE.

Keywords: Hydrogen evolution reaction, Pepper, Silver nanoparticles, Electrocatalysis

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Functionalization of carbon nanotubes with silver nanoparticles and its electrocatalytic application to detection of hydrogen peroxide

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Abstract

Nanocatalysis has recently been a rapidly growing field which involves the use of nanoparticles as catalysts for a variety of organic and inorganic reactions [1]. Ag nanoparticles show excellent catalytic and electrocatalytic activities [2]. Detection of hydrogen peroxide (H_2O_2) has become extremely important in recent years because of its wide and varied applications [3]. Up to now, many techniques including spectrometry, titrimetry, chemiluminescence, and electrochemistry have been employed for determination of H_2O_2 . Among them, electrochemical technique is a promising tool for the construction of simple and low-cost sensors due to their high sensitivity, good selectivity, and ease of operation [5]. In this work, we reported a simple and low-cost procedure to functionalize of carbon nanotubes with silver nanoparticles (AgNPs/CNT) by using ascorbic acid as reducing agent. The AgNPs/CNTs were characterized by a variety of means such as transmission electron microscope (TEM), powder X-ray diffraction (XRD) and energy dispersive X-ray (EDAX). AgNPs/CNT modified carbon-paste electrode (AgNPs/CNT/CPE) displayed excellent electrochemical catalytic activities towards hydrogen peroxide (H_2O_2). The reduction overpotential of H_2O_2 was decreased significantly compared with those obtained at the bare CPE. The sensor responded linearly to hydrogen peroxide (H_2O_2) with detection limit of μM at 3σ . The studied sensor exhibited good reproducibility and long-term stability.

Keywords: Ascorbic acid, Hydrogen peroxide, Functionalization, Carbon nanotubes, Silver nanoparticles

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Electrosynthesis, Characterization and Electrocatalytic Properties of Pt-Fe Nanoparticles toward the Oxidation of Methanol and Formic Acid

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Abstract

Studies on the fuel cells currently increase because they have great potentials for portable, transport and stationary power supply applications [1]. Due to the increasing demand of energy and the continuously rising oil price the use of renewables as a source for the production of fuel and chemicals has become more and more important during the last few years [1]. Methanol oxidation is of great importance in fuel cell technology, namely in the direct methanol fuel cell (DMFC) aimed to power electric vehicles [2]. On the other hand, formic acid has recently attracted attention as an alternative fuel for direct liquid fuel cells (DLFCs) due to its high theoretical open circuit voltage (1.45 V) and reduced fuel cross-over compared to methanol. In addition, formic acid is a strong electrolyte that facilitates proton transport in catalyst layers [3]. One of the main problems in DLFCs is the development of anodic materials with high electroactivity towards the oxidation of methanol and formic acid. It is well known that platinum (Pt) is an effective electrocatalyst for these oxidations. However, Pt is also highly sensitive to CO poisoning: the catalyst surface is progressively poisoned by the adsorbed CO (CO_{ads}), which is formed as a result of the stepwise dehydrogenation of these oxidants in oxidation reaction. Poisoning CO_{ads} species can be oxidatively removed from the Pt surface through a Langmuir-Hinshelwood-type surface reaction with neighboring OH_{ad} species electroadsorbed from water at more positive potentials. Thus, alloying of Pt with oxophilic metals enables electrochemical dissociation of water on oxophilic metal sites at more negative potentials compared to pure Pt and, therefore, allows electrocatalytic oxidation of CO_{ads} at lower anodic over potentials. In this paper, bimetallic platinum-Iron nanoparticles electrocatalyst was prepared by a two-step procedure: (I) the CCE was produced by using sol-gel technique and (II) platinum-Iron nanoparticles alloy was precipitated electrochemically on the surface of CCE and the resulting electrode was referred as Pt-Fe/CCE. The obtained catalyst (Pt-Fe/CCE) was characterized by scanning electron microscopy, energy-dispersive X-ray spectroscopy, X-ray diffraction and cyclic voltammetry. The electrocatalytic activity of the Pt-Fe/CCE towards the electrooxidation of the methanol and formic acid was evaluated by cyclic voltammetric technique in 0.1 M of H_2SO_4 solution. It was found that the Pt-Fe/CCE was catalytically more active than Pt nanoparticles (alone) supported on the same substrate. The obtained results also showed that the presence of Fe greatly enhances the activity of the Pt toward the electrooxidation of methanol and formic acid. Moreover, it contributes to reduce the amount of the noble metal in the anode of DLFCs, which remains one of the challenges to make the technology of DLFCs economically viable. Finally, the effect of some experimental factors was studied and optimum conditions were suggested. The long-term stability of nanoparticles electrocatalyst has also been studied.

Keywords: Pt-Fe nanoparticles, Electrosynthesis, Methanol, Formic Acid,

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Determination of activity coefficient of the ternary mixed electrolyte (MgCl_2 + glucose + water) system by the potentiometric method at $T = 298.15$

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Abstract:

To measure the thermodynamic properties of electrolytes in mixed solvent, the most common methods are the isopiestic vapor pressure [1] and potentiometric techniques [2]. Recently, there has been an increasing amount of work concerning the measurement of thermodynamic properties of electrolytes in mixtures of solvents, because their composition is important in industrial, chemistry, biological and other fields. In the last decades, a series of ion-interaction models for electrolyte solution have been proposed to predict activity coefficient of each solute and osmotic coefficient of aqueous systems. One of the most famous and useful models is that proposed by Pitzer [3]. This model is relatively simple, and it performs electrolyte calculations with accuracy, especially for concentrations under 6 molal. In this work, the results relating to the thermodynamic properties of (MgCl_2 + glucose + water) system using the potentiometric method are reported. The potentiometric measurements were performed on the galvanic cell of the type: $\text{Ag}|\text{AgCl}|\text{MgCl}_2$ (m), glucose (wt.%), H_2O (1-wt) % | Mg -ISE, in various mixed solvent systems containing 0, 10, 20, 30, and 40% mass fractions of glucose over ionic strength ranging from 0.0010 to 6.0000 kgmol^{-1} . The modeling of this ternary system was made based on the Pitzer ion-interaction model. The resulting values of the mean activity coefficients, the osmotic coefficients and the excess Gibbs free energy together with Pitzer ion-interaction parameters ($\beta^{(0)}$, $\beta^{(1)}$ and C^ϕ) for the series under investigated system are reported.

Keywords: Activity coefficients, Potentiometric method, Glucose, MgCl_2

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Preparation of Carbon Ceramic Electrode Modified with Pd/MWCNT Electrocatalyst for Oxidation of Alcohols in Fuel Cells

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Abstract

Direct alcohol fuel cells (DAFCs) are attractive power sources for portable application due to unique advantages over devices feed with hydrogen. Alcohols such as methanol and ethanol exhibit high energy density and have easy transport properties [1, 2]. Palladium (Pd) is a suitable replacement for platinum in DAFCs. Pd-based electrocatalyst can be highly active for the oxidation of a large variety of substrates in alkaline environments [3]. In order to improve catalytic properties of nanoparticles and minimize the overall cost of fuel cell, Pd nanoparticles are deposited on several materials such as carbon supports. Carbon supports can enhance the electron transfer and improve electrocatalytic activity of Pd nanoparticles. Among carbon supports, multi wall carbon nanotubes (MWCNT) play a key role in the enhancement of Pd catalytic properties. In this study, MWCNT-supported Pd (Pd/MWCNT) catalyst is prepared by chemical reaction and decorated on carbon ceramic electrode (Pd/MWCNT/CCE). X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX) analyses were performed to characterize the nanostructure and composition of the Pd/MWCNT. Pd/MWCNT/CCE is used as anode for oxidation of methanol and ethanol in alkaline media. Electrochemical methods such as cyclic voltammetry and chronoamperometry were used to study electrochemical properties of obtained catalyst. The effects of some experimental factors such as alcohols and electrolyte concentration, scan rate, the upper limit of potential scanning region and long-term stability of electrode were investigated. Pd/MWCNT/CCE was catalytically more active than Pd/CCE and has eligible stability for electrooxidation of ethanol and methanol when stored in ambient conditions. In addition, continues cycling makes Pd/MWCNT/CCE electrocatalyst more active for alcohol fuel cells. The results show that Pd/MWCNT/CCE is a suitable choice for alcohols electrooxidation.

Keywords: Pd nanoparticles, Electrocatalyst, MWCNTs, Carbon-ceramic electrode, Ethanol fuel cell, Methanol fuel cell

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Photochemical degradation of Fluocinolone Acetonidin drug in aqueous solutions using nanophotocatalyst ZnO doped by N-,S-, and C

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Abstract.

In general, studies have shown that the photocatalytic activity of doped semiconductor with some nonmetals is more than pure semiconductor. Hence, with an attempt to achieve higher photocatalytic activity, doped ZnO with C, N, S were prepared by a Sedimentary process, and used for the photocatalytic degradation of drug Fluocinolone Acetonidin in aqueous solutions. X-ray diffraction (XRD), Energy dispersion X-ray (EDX), X-ray photoelectron spectroscopy (XPS), structural equation modeling (SEM) were used to characterize the microstructure and morphology of the precursor and the products obtained at various temperatures. The photocatalytic degradation of drug has been investigated using supported ZnO/C, N, S photocatalyst under UV light irradiation. we have studied the influence of the basic photocatalytic parameters such as the irradiation time, initial concentration of drug, pH of the solution, amount of nanoparticles, addition of oxidant, alcohol on the reaction, and optimum conditions for maximum degradation was determined. In the present work, based on the results, most degeneration of Fluocinolone Acetonidin has been observed at concentration of 20 ppm and amount of photo-catalyst of 7 mg and oxidant K₂S₂O₈ and pH=9. Analysis of the kinetic showed that the amount of Fluocinolone Acetonidin photocatalytic degradation can be fitted with pseudo- first- order model.

Keywords : Photocatalytic degradation, Nanophotocatalyst ZnO doped by N-, S-, C, Fluocinolone acetanidin

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Determination of Diltiazem in the presence of Timolol in human serum samples using nanoFe₃O₄@GO modified glassy carbon electrode

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Abstract

Determination and quantification of drugs in biological fluids and pharmaceutical samples are essential in pharmaceutical, toxicological, doping and clinical chemistry research [1]. The simpler and more rapid methods for simultaneous analysis of several cardiovascular drugs are interesting for therapeutic drug monitoring purposes [2]. Despite their widespread application and use, little works has been reported on the simultaneous determination of cardiovascular drugs by electrochemical and the related sensing devices. Therefore, it's important to point out simultaneous determination of these drugs is necessary for medicines. In the present study, a new chemically modified electrode was constructed based on magnetic graphene oxide modified glassy carbon electrode (NanoFe₃O₄@GO-GC). For preparation of NanoFe₃O₄@GO-GC, GC electrode was transferred to the 1.0M sulfuric acid solution. Potential in the range of -1 to 1.5V in a regime of cyclic voltammetry was applied for 20 cycles. Afterward, the electrode was rinsed thoroughly with distilled water and dried in air. Then, NanoFe₃O₄@GO-GC (50 mg) was dispersed into acetone/double distilled water with the aid of ultrasonic stirring for 6 h. A 10 μ L aliquot of this dispersion (with concentration of 1.0 mgmL⁻¹) was dropped on the GC electrode surface, and the surface was allowed to dry in room temperature (24 h). When not in use, the electrodes were stored at 4 °C. The electrode was evaluated as an electrochemical sensor for simultaneous determination of Diltiazem and Timolol in aqueous solutions. The measurements were carried out by application of the differential pulse voltammetry (DPV) method in phosphate buffer solution with pH 6.00. The results revealed that NanoFe₃O₄@GO-GC promotes the rate of oxidation by increasing the peak current. NanoFe₃O₄ loaded in GO can increase anodic peak currents of Diltiazem and Timolol on electrode surface. The electrostatic interaction between Diltiazem and Timolol cations and the high electron density of hydroxyls group of NanoFe₃O₄@GO-GC would lead to increase in concentration of Diltiazem and Timolol around the surface of the modified electrode and the peak current increased significantly. The prepared electrode shows voltammetric responses with good selectivity for Diltiazem and Timolol in optimal conditions, which makes it very suitable for simultaneous determination of these drugs. The practical analytical utility of the modified electrode was illustrated by simultaneous determination of DT and TM in serum samples.

Keywords: Diltiazem, Timolol, Electrochemical sensor, Magnetic nanoparticle, Pharmaceutical analysis

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A biosensor fabricated by hydrogen bubble evolution and its application to non-enzymatic glucose detection

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Abstract

It is widely thought in electro-biochemical analysis that the sensing interfaces play a key role in the enzymeless detection of biomolecules like glucose, ascorbic acid, dopamine and uric acid[1]. Glucose plays an important role in biosphere. Detection of glucose concentration is important for human health, food safety and biotechnology[2]. On the way to maximize the anti-poisoning sensitivity of nonenzymatic electrochemical glucose sensors as well as achieve favorable selectivity, we propose here a porous interface fabricated by a facile but effective approach for glucose monitoring in alkaline media containing dissolved oxygen. In this study the porous Cu_xO and Ni_xO films were successfully prepared by electrochemical deposition assisted with hydrogen evolution on a Cu electrode. Electro-catalytic oxidation of the Cu_xO and Ni_xO electrode towards glucose was studied by cyclic voltammetry (CV) and chronoamperometry. Chronoamperometric results obtained under optimized conditions reveal that the proposed sensor exhibits desired poison resistance ability in the presence of chloride ions and good selectivity to glucose. The sensor showed good selectivity to conventional intermediates such as AA and UA and long term stability. The application of the Cu-Ni foam based sensor for monitoring glucose in practical samples is also successfully detected.

Keywords: Non-enzymatic sensor, Glucose, Chronoamperometry, Cyclic voltammetry, Cu-Ni foam, Cu electrode

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Carbon nanotubes decorated with magnetic core-shell nanoparticles as a sensing platform for determination of the non-steroidal anti-inflammatory drug

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Abstract

Application of nanomaterials has been greatly expanded in developments of science and engineering such as catalysis, electronics, drug delivery and sensors. Hence, several methods have been tried to obtain excellent nanocomposite containing kinds of nanomaterials, which can combine the advantages of them [1]. Multi-walled carbon nanotubes (MWCNTs) and Fe_3O_4 magnetic nanoparticles have attracted much attention because of their novel physical and chemical properties [2,3]. Considering the above statement, in this study, a nanocomposite was first synthesized based on decoration of MWCNTs by magnetic core-shell $\text{Fe}_3\text{O}_4@\text{SiO}_2$ nanoparticles and used as modifier in carbon paste electrode (CPE). In the next step, the modified carbon paste electrode was successfully used for electrochemical determination of non-steroidal anti-inflammatory drug diclofenac (DCF) under the optimized conditions by square wave voltammetry (SWV). The morphology and properties of the nanocomposite was characterized by TEM (Fig. 1), SEM, EDS and FT-IR. At the optimum conditions, the peak current was linearly dependent on the DCF concentration in the range of 0.5 to 100 μM with detection limit of 0.43 μM . The proposed electrode was successfully applied for the determination of DCF in human blood serum and pharmaceutical samples.

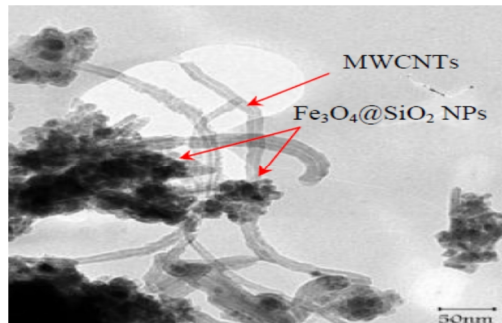


Fig. 1. TEM image of MWCNTs decorated by magnetic core-shell $\text{Fe}_3\text{O}_4@\text{SiO}_2$ nanoparticles.

Keywords: Magnetic Nanocomposite, Core-shell, Multi-walled carbon nanotubes, Diclofenac sodium

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AlMCM-41-NH₂ as an efficient modifier for electro-oxidation of diclofenac in biological samples

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Abstract

Diclofenac, a non-steroidal anti-inflammatory drug, is one of the most commonly used pain killers which largely used as sodium salt. Due to its extensive use, diclofenac residues can nowadays be regularly detected in surface waters throughout the world [1]. Mesoporous materials such as MCM-41 can be used as modifiers to increase the surface area of the electrode and therefore to enhance the electro-oxidation processes. In this work, AlMCM-41 was synthesized using cetyltrimethyl ammonium bromide (CTMABr) as the templating agent [2]. The surfactant template was then removed from the synthesized material by calcination at 600 °C for 5 h [3]. Then, (3-Aminopropyl) silyloxy-AlMCM-41 was prepared by addition of 3-aminopropyl-triethoxy-silane (APTES) to a suspension of AlMCM-41 converting it to AlMCM-41-NH₂. AlMCM-41-NH₂ was characterized by X-ray diffraction, Brunauer-Emmett-Teller (BET) and FT-IR methods. Electrochemical studies suggested that AlMCM-41-NH₂ nanoparticles modified electrode provided a synergistic augmentation on the voltammetric behavior of electrochemical oxidation of diclofenac, which was indicated by the improvement of anodic peak current. Additionally, modified electrodes with MCM-41-NH₂, MCM-41-SH and AlMCM-41-SH were also prepared and their functions towards diclofenac oxidation were checked.

Keywords: Modified electrode, Diclofenac, AlMCM-41-NH₂, Nanoparticles, Voltammetry

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Enhanced electrochemiluminescence sensor for diphenhydramine based on a YbO₂ nanoparticle -modified electrode

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Abstract

An electrochemiluminescence (ECL) detection of diphenhydramine (DPH) at Tris(2,2'-bipyridyl)ruthenium(II) [Ru(bpy)₃]²⁺-YbO₂ nanoparticle-modified carbon paste electrode was developed. The immobilized [Ru(bpy)₃]²⁺-YbO₂ nanoparticle-modified CPE shows good electrochemical and photochemical activities. The modified electrode showed an electro-catalytic respond to the oxidation of DPH. The optimized condition for the highest intensity is pH= 8.5 (0.1M phosphate buffer). In the optimized experimental conditions, the linear ECL response to concentrations of DPH is from 3×10⁻⁹ to 2×10⁻⁶ M with a correlation coefficient of 0.9995 and a detection limit of 8×10⁻¹⁰ M. The relative standard deviation (RSD) was less than 4.6% (n = 5) and the recovery was in the range of 98 - 106% for the determination of DPH in pharmaceutical samples. The electrochemical and electrochemiluminescence characterizations will be made by cyclic voltammetry and electrochemical impedance spectroscopy. The sensor showed good selectivity and long term stability. Its surface could be renewed quickly by a simple polish step.

Keyword: Diphenhydramine, Electrochemiluminescence sensor, YbO₂ nanoparticle

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Study of the effect of barium sulfate nanoparticles sizes as expander in performance of the lead acid batteries

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Abstract

The performance of lead acid batteries decreases prematurely mainly due to the progressive accumulation of lead sulfate on the surface of negative plate. This occurs because of the lead sulfate cannot be converted efficiently to spongy lead after or during charging. Eventually, this layer of lead sulfate develops to such an extent that the effective surface area of the plate is reduced markedly, and the plate can no longer deliver the required current. This significantly reduces the potential life span of the battery [1]. The term expander applies to mixture of barium sulfate, carbon black and lignin, added to the negative active material to maintain performance of the plate during the life of the battery [2].

Barium sulfate is of the expander integrant provides sites for precipitation of lead sulfate during discharge. By providing large number of sites for the precipitation of lead sulfate, the barium sulfate prevents lead sulfate deposition as a tin, impermeable passivating film. Nanoparticle of barium sulfate is a very effective anti passivating agent; its small particle size provides millions of sites for nucleation to take place. This paper proposes a novel method of synthesis of nanoparticle of barium sulfate at various sizes through reaction of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ and NaSO_4 solution. The effect of obtained BaSO_4 nanoparticles as an additive in negative plate with role of expander in performance of the lead acid batteries has been studied. The obtained results showed that barium sulfate in nano size exerted more influence comparing to the larger barium sulfate particles.

Keywords: Lead-acid battery, Barium sulfate, Nanoparticles, Expander

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Electrochemical Study of the MCM-41 Supported NHPI as Catalyst for Green Oxidation of Alcohols

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Abstract

In the last decade several aerobic oxidations of organic compounds were performed using N-hydroxyphthalimide (NHPI) as free-radical promoter in the presence or absence of metal salt initiators [1]. In 1983, Masui et al. carried out a pioneering work on the synthetic application of NHPI as mediator in electrochemical oxidation of secondary alcohols to ketones, and showed that PINO formed by an electrochemical oxidation of NHPI abstracts selectively hydrogen atoms from the alcohols leading to ketones [2]. The primary aims of this study are: voltammetric analysis of the electro-oxidation of various alcohols by NHPI in more details, optimization of reaction condition and study of the effect of pH and solvent on this radicalic reaction and electro catalytic oxidation of alcohols by electrolysis reactions. The final goal of the project is the study of the possibility of the heterogenization of NHPI and making a modified electrode based on ordered mesoporous silica structure bearing NHPI. The desired support is a well-ordered mesoporous silica structure which is obtained by an electrochemical method known as Electro-Assisted Self-Assembly (EASA) [3]. EASA was achieved by applying a suitable cathodic potential to an electrode immersed in a hydrolyzed sol solution containing the cetyltrimethylammonium bromide, NHPI functionalized- triethoxysilane and tetraethoxysilane. Applying cathodic potential causes to generation of OH⁻ species at the electrode/solution interface to induce and catalyze condensation of the silane precursors. Characterization of modified electrodes and films was done by TEM, TGA and elemental analyses. Moreover, voltammetric analysis of the desired modified electrode shows a good reversible electrochemical response due to the electron transfer of anchored NHPI groups on the channels of ordered mesoporous silica structures. The results of both homogeneous and heterogeneous studies showed that the system is capable of oxidation of primary and secondary types of alcohol in good yields with high selectivity, without any chemical oxidizing agent.

Keywords: Ordered mesoporous silica, Oxidation, Electro synthesis, NHPI, PINO, EASA, MCM-41

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Investigation of layered double hydroxide nanocontainers as corrosion inhibitor for carbon steel in acidic medium

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Abstract

Many organic compounds containing heteroatoms like nitrogen, oxygen, phosphorus and sulfur, the unsaturated bonds and aromatic cycles have been presented to be effective inhibitors and decreasing the corrosion rate for the corrosion of steel in acid media by several researcher[1]. Previous studies have inquired the relationship between the structure of the inhibitor compound and its efficiency but less attention has been paid to the relationship between inhibition efficiency on the size of the inhibitor molecule and the electronic distribution in the inhibitor molecule. Recently, layered double hydroxides (LDHs) have been widely studied due to its several unique properties. LDH is a category of synthetic anionic clays with host-guest nanolayer material including positively charged metal hydroxide sheets, embedded anions with negatively charged as guest and water molecules. LDH has been intensively used in various fields such as drug delivery, catalysts, hydrothermal reactor, ion exchange hosts, fire retardant additives and corrosion inhibitor [2]. In this work, the efficiency of novel sodium dodecyl benzenesulfonate (SDBS) modified Mg-AL layered double hydroxide (LDH) as corrosion inhibitors of carbon steel in 1M HCl was investigated on the basis of potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) measurements. Electrochemical experiments were carried out using an Autolab (PGSTAT 12). A three-electrode arrangement was used for electrochemical studies. Potentiodynamic polarization curves were obtained with a scan rate of 0.5 mV s^{-1} in the potential range from -150 to $+150 \text{ mV}$ relative to the corrosion potential. Corrosion current density values were obtained by Tafel extrapolation method. The alternating current frequency range was extended from 100 kHz to 0.1 Hz with a signal amplitude perturbation of 5 mV . Potentiodynamic polarization has been performed at different temperatures (from 35 to $65 \text{ }^{\circ}\text{C}$) in absence and presence of 100 ppm inhibitor after 1 h of immersion time. The results from this study demonstrate that The LDH is a good inhibitor for mild steel in hydrochloric acid solution. The inhibiting protection increases with increasing of the inhibitor concentration. Polarization curves indicated that the LDH act as mixed type inhibitor. The electrochemical impedance diagrams showed mainly a capacitive loop, which can be attributed to the formation of an adsorbed layer on the steel surface. The results obtained from EIS, polarisation curves are in reasonably good agreement. The adsorption of LDH is well described by Langmuir isotherm model. The data indicated that the corrosion current density was increased with rise temperature and they always are lower than blank solution. Thermodynamic adsorption parameters such as E_a , ΔH^* and ΔS^* show that the in situ synthesized inhibitor adsorbed by an endothermic process and its adsorption is physical adsorption. Lead to the formation of an adsorptive film of electrostatic character.

Keywords: Carbon steel, Corrosion inhibitor, Adsorption, EIS, Potentiodynamic polarization

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Development of nano-scale electrochemical biosensor and its application in the stem cell studies

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Abstract

In this study, gold nanoparticle (AuNP) functionalized mesoporous crystalline material [AuNP-MCM-41] was used to engineer a modified glassy carbon electrode for determination of the activity of caspase 3 in cord blood stem cell development. To this end, first, the AuNP-MCM-41 modified electrode was thiolated with the biotinylated Gly-Asp-Gly-Asp-Glu-Val-Asp-Gly-Cys peptide (B-DEVD), which can specifically cleave by caspase 3, and finally combined with horseradish peroxidase (HRP) as signal amplifier. Second, the streptavidin-coated magnetic bead (S-MNB) was conjugated with B-DEVD peptide and also biotinylated with HRP. Afterward, the electrochemical activity of engineered biosensor evaluated by using cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS), chronoamperometry and differential pulse voltammetry (DPV). The electrochemical behavior of the engineered B-DEVD-AuNP-MCM-41 electrode was examined in presence of caspase 3 using phosphate buffer saline (PBS) containing hydroquinone and hydrogen peroxide. Finally, the activity of the caspase 3 in the cell lysates evaluated with the engineered electrode. We found that the activity of caspase 3 can be significantly sensed by the electrode with very low limit of detection. The activity of caspase 3 was different in apoptotic stem cells versus normal cells. Based on these findings, we propose this engineered glassy carbon electrode functionalized with B-DEVD-AuNP-MCM-41 as a highly sensitive and cost-effective biosensor for determination of caspase 3 activity.

Keywords: Electrochemical nanobiosensor, Gold nanoparticles, Mesoporous materials, DEVD peptide, Caspase 3, Apoptosis

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Comparison and investigation the physical and optical properties of Co-polymervinylpyrrolidone/methylmethacrilate with PbS-ZnS/polyvinylpyrrolidone/methylmethacrilate nanocomposite

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Abstract

We report a facile synthesis of PbS-ZnS/ (vinylpyrrolidone -co-methylmethacrylate) Nanocomposite composite via surfactant-free emulsion polymerization. Through the functional groups on the resulting copolymer Pb⁺² and Zn⁺² ions has demonstrated strong affinity to attract. By coordination of Pb⁺² and Zn⁺² ions on polymer matrix and reaction with S²⁻ nanoPbS-ZnS /MMA-NMP composite was synthesized. The structure and morphology of PbS-ZnS nanoparticles have been characterized using x-ray diffraction and Scanning Electron Microscopy (SEM). The XRD measurements suggest the cubic structure for PbS and the particles size was estimated to about 9/4 nm by applying Scherrer's equation. FT-IR spectrum confirmed the polymerization of monomers. Optical properties of nanocomposite evaluated by UV-Vis absorption spectra and photoluminescence (PL) [1]. were explored by thermal gravimetric analysis (TGA). In comparison to the copolymer MMA-NMP showed an improvement on the thermal stability.

Keywords: Nanocomposite, PbS, ZnS nanoparticles, Surfactant-free, Band gap, Optical properties, vinylpyrrolidone, Methylmethacrylate

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Synthesis and application of ZrO₂ nanoparticle carbon paste electrode as a sensor for voltammetric determination of ascorbic acid

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Abstract

Ascorbic acid (AA), as a water soluble vitamin, is an effective reducing agent and a powerful antioxidant in food, preventing color changes and alterations of aroma and flavor as well as extending the storage time of the products [1]. So, determination of AA is very important in food, pharmaceutical and biological samples. Recently, nanostructured materials have also been incorporated into electrochemical sensors for biological and pharmaceutical analyses [2]. In this work, a ZrO₂/nanoparticles (ZrO₂/NPs) modified carbon paste electrode (ZrO₂/NPs/CPE) was fabricated and used to investigate the electrochemical behavior of AA. We study synthesis and characterization of ZrO₂/NPs with X-ray diffraction (XRD) and TEM methods. The cyclic voltammogram showed an irreversible oxidation peak at 0.57 V (vs. Ag/AgCl_{sat}), which corresponded to the oxidation of AA. Compared to common carbon paste electrode, the electrochemical response was greatly improved. Under the optimized conditions, the oxidation peak current of AA showed linear dynamic range 0.09–500 μM with a detection limit of 0.06 μM, using the SWV method. The proposed sensor was successfully applied to the determination of AA in fresh vegetable juice, fruit juices and food supplement samples without previous preparation and was compared with a published electrochemical method.

Keywords: Ascorbic acid, ZrO₂ nanoparticle, voltammetry

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Magnetic core/shell Fe₃O₄@ZnO nanoparticles-modified electrode as an electrochemical sensor for simultaneous voltammetric determination of quercetin and rutin

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Abstract

Quercetin and rutin belong to the flavonoid family that is distributed in fruit and vegetables. Several studies demonstrate that rutin has some physiological properties such as anti-inflammatory, anti-tumor and anti-bacteria. Quercetin is produced by hydrolyzing rutin in aqueous solution and has the similar effects such as anti-tumor, anti-bacteria and anti-allergic. In present work, a rapid and specific electrochemical method was utilized and validated for the simultaneous determination of two biologically active flavonoidal compounds, quercetin and rutin. A carbon-paste electrode modified with Fe₃O₄@ZnO magnetic nanoparticles (MNPs) was used for this propose. Fe₃O₄@ZnO MNPs with core/shell structure have been synthesized by a co-precipitation method [1, 2]. The synthesized MNPs were characterized by X-ray powder diffraction (XRD), transmission electronic microscope (TEM) and scanning electronic microscope (SEM). The XRD results confirmed the coexistence of Fe₃O₄ and ZnO phases in the nanoparticles. SEM and TEM results showed that the prepared Fe₃O₄@ZnO MNPs are made of the spherical shape particles with an average size of about 15 nm. The electrocatalytic properties of Fe₃O₄@ZnO/CP/GCE toward the electrocatalytic oxidation of rutin and quercetin were studied using cyclic voltammetry, chronoamperometry, amperometry and differential pulse voltammetry [3]. Under the optimal conditions electrochemical parameters of quercetin and rutin such as charge transfer coefficient (α), electron transfer rate constant (k_s) and diffusion coefficient (D) were determined. Furthermore, this sensor was employed for the simultaneous determination of rutin and quercetin in some real samples and the obtained results were found to be satisfactory.

Keywords: Magnetic core/shell Fe₃O₄@ZnO nanoparticles, Rutin, Quercetin, Voltammetry

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Investigation of the Intercalation of Graphene Quantum Dots (GQDs) By Tensammetry

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Abstract

In chemistry, intercalation is the reversible inclusion or insertion of a molecule (or ion) into compounds with layered structures. Examples are found in graphite intercalation compounds [1]. Adsorption-based voltammetric methods are often referred to as tensammetric methods [2]. In this study, a novel electrochemical cell was designed using glass and Teflon. A three-electrode system was used to study the electrochemical intercalation behavior of the graphene quantum dot (GQD) using KOH (~1.0 M) as electrolyte. These electrodes include graphene quantum dot -modified Pt disk, platinum wire and Ag/AgCl (3.0 M) as working, counter and reference electrodes, respectively. The experiments were achieved in ambient pressure and temperature. Voltammogram shown in Fig. 1 clearly reveals the effect of various ions during the interaction process on the surface of GQDs for generation of carbon nanodots.

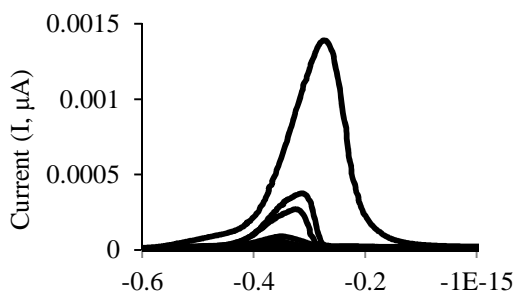


Fig. 1. Voltammogram showing intercalation process of GQD by tensammetry

Keyword: Graphene quantum dots, Tensammetry, Voltammetry

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Novel SiO₂@Au homogeneous core/shell –chitosan nanocomposite based on glucose biosensor

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Abstract

A nano core/shell, consisting of a dielectric core surrounded by a thin noble metal shell, possesses unique optical properties, including high optical absorbance. These features render attractive nano-shells that are used for technology such as conducting polymer devices, bio-sensing, drug delivery and photothermal therapy [1–2]. Nanoparticles have a unique ability to promote fast electron transfer between electrode and the active site of the enzyme. In this context, different types of nanoparticles have been suggested as promising matrixes for enzyme immobilization to improve stability and sensitivity of a biosensor [3]. In the first part of this study, silica nanoparticles (SiO₂) and colloidal gold nanoparticles were prepared by coprecipitation and chemical reduction methods, respectively. Then, the silica / gold nanoparticles in a core / shell structure (SiO₂@Au) were fabricated. Structural and optical properties of nanoparticles were studied by X-ray diffraction (XRD), Scanning electron microscopy (SEM), Transmission electron microscopy (TEM), UV-Vis spectrometry and Fourier Transform infrared spectroscopy (FTIR). The TEM images showed uniform silica nanoparticles with a diameter of 80 nm, colloidal gold nanoparticles with an average diameter of about 8 nm. In the second part, GO_x/CH-SiO₂@Au/FTO bio-sensor was fabricated by the following route: nanocomposite system of the silica / gold (SiO₂@Au) nanoparticles and chitosan were prepared and then deposited on FTO (fabricated by spray pyrolysis technique) substrate using Dip Coating technique. To investigate the biological properties of the sensing electrode, glucose oxidase enzyme was fixed on the electrodes by physical method. Sensing parameters were evaluated by chronoamperometric method. Detection limit of 4 μM, linear range of 1 to 7 mM and Sensitivity of 1 μA/mMcm² were achieved.

Keywords: Core / shell nanoparticles, SiO₂@Au core / shell nanoparticles, Glucose biosensor, FTO

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Biosynthesis of gold nanoparticle and its application for preparation of a voltammetric sensor for morphine determination

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Abstract

Gold nanoparticles have been considered as an important area of research due to their unique and tunable surface plasmon resonance (SPR) and their applications in biomedical science including drug delivery, tissue/tumor imaging, photothermal therapy and immuno-chromatographic identification of pathogens in clinical specimens [1]. Morphine is the most abundant alkaloid found in opium, the dried sap derived from shallowly slicing the unripe seedpods of the *Papaver somniferum* poppy [2]. In this study, the biological synthesis of gold nanoparticles (AuNPs) using extract of *Ficus macrophylla* as reducing agent is reported. The nanoparticles obtained are characterized by UV–Vis spectroscopy, transmission electron microscopy (TEM) and X-ray diffraction (XRD) analysis. In continuous, the electrochemical oxidation of morphine on the new AuNPs ionic liquid carbon paste electrode (AuNPs/IL/CPE) was carefully studied. The sensor exhibits an enhanced effectiveness for the electro-oxidation of morphine in aqueous solution. The electrochemical parameter of morphine on the AuNPs/IL/CPE was calculated. Based on the relationship of the oxidation peak current and the concentration of morphine a sensitive analytical method was established with square wave voltammetry. The linear range for morphine determination was in the range from 0.08 to 600 $\mu\text{mol L}^{-1}$ and the detection limit was calculated as 0.04 $\mu\text{mol L}^{-1}$ (3σ). Finally, the proposed sensor was also examined as a selective, simple and precise electrochemical sensor for the determination of morphine in real samples such as urine and ampoule.

Keywords: Gold nanoparticles, Biosynthesis, Voltammetric determination, Morphine analysis

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Electrospun CuO-NFs/ILnanocomposite modified electrode as a new sensing platform for risperidone determination

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Abstract

Risperidone (RIS) is an atypical antipsychotic drug. Among the neuroleptics, atypical antipsychotics have received great attention in recent years, because of their peculiar pharmacological profile. Due to its wide spread use, rapid, efficient and economical methods of analysis of this compound in pharmaceutical formulations and biological fluids are required, and in this sense electrochemical techniques appear particularly attractive [1]. Recently, great efforts have been made to fabricate one-dimensional (1-D) metal oxide nanomaterials due to their special morphologies, compositions, chemical and physical properties [2]. One of the most important applications of 1-D metal oxide nanomaterials is to develop their potential in chemical sensing or biosensing, profiting from their large surface-to-volume ratios and a Debye length comparable to their dimensions [3]. In this work, a new nanocomposite consisting of copper oxide nanofibers (CuO-NFs) prepared by electrospinning and hydrophobic ionic liquid 1-ethyl-3-methylimidazolium hexafluorophosphate (EMIMPF₆) were demonstrated for the first time for RIS determination. The assay performances to RIS were evaluated by cyclic voltammetry and differential pulse voltammetry. The results indicated that the voltammetric response of RIS was improved distinctly at the surface of CuO-NFs/IL/CPE and the oxidation of RIS at the surface of modified electrode occurs at potentials of about 123 and 62 mV less positive than that of unmodified CPE and CuO-NFs/CPE, respectively. Also, the sensitivity of this sensor was evaluated by comparing it with CuO nanoparticles modified electrode (CuO-NPs/IL/CPE).

Keywords: Electrospun CuO nanofibers, Electrospinning, Risperidone, Voltammetry

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Amperometric sensor based on multi-walled carbon nanotubes/rutin nano-composite for highly sensitive electrocatalytic determination of methimazole

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Abstract

Methimazole (MMI) is one of the most widely used anti-thyroid drugs in the treatment of human hyperthyroidism, which inhibits thyroid hormone biosynthesis by preventing the organification of iodide in the thyroid. MMI may also cause side effects such as nephritis, liver cirrhosis, skin irritation, allergies and pharyngitis with fever as well as impaired taste, olfactory and auditory [1]. Thus, it is necessary to develop a rapid and effective method for the determination of MMI in many areas including clinical chemistry, nutrition, as well as pharmaceutical analysis. In this study, multi-walled carbon nanotubes MWCNTs was evaluated as a transducer, stabilizer and immobilization matrix. The construction of amperometric sensor was based on rutin adsorbed on MWCNTs immobilized on the surface of glassy carbon electrode (R/MWCNTs/GCE). Cyclic voltammogram of the modified electrode indicated a pair of well-defined and nearly reversible redox couple with surface confined characteristics at a wide pH range (2–10). The surface coverage (Γ) and charge transfer rate constant (k_s) of rutin immobilized on MWCNTs were $2.37 \times 10^{-11} \text{ mol cm}^{-2}$ and 14.23 s^{-1} , respectively, indicating high loading ability of MWCNTs for rutin and great facilitation of the electron transfer between rutin and carbon nanotubes immobilized on the electrode surface [2]. Modified electrode exhibited excellent electrocatalytic activity toward oxidation of MMI. The amperometry of modified electrode in a stirred buffer solution (pH 8.0) at constant potential was used for detection of MMI. The detection limit, linear calibration range and sensitivity for MMI were 18 nM, 0.1 μM to 7 μM , and 252.4 nA/ μM , respectively. Excellent electrochemical reversibility of the redox couple, good reproducibility, high stability, low detection limit, long life time, fast amperometric response time, and technical simplicity are great advantages of this sensor. The obtained results showed promising practical application of R/MWCNTs/GCE as an amperometric sensor for MMI detection. The resulted electrode was used for the voltammetric determination of MMI pharmaceutical and clinical (human blood serum) preparations.

Keywords: MWCNTs, Glassy carbon electrode, Electrocatalysis, Methimazole

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Simultaneous determination of epinephrine and uric acid under coexistence of ascorbic acid based on electrodeposition of gold nanoparticles on thioglycolic acid film modified glassy carbon electrode

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Abstract

Epinephrine (EP), is one of the important catecholamine neurotransmitters in the mammalian central nervous system and plays a very important role in the function of the central nervous, hormonal, and cardiovascular systems. Many diseases are related to a change of catecholamine concentration, therefore it is necessary to develop quantitative methods for the determination of catecholamine concentration [1]. Uric acid (UA), a vital biomolecule in biological fluids, is a primary end product of purine metabolism. For a normal healthy person, physiological concentration of UA is within a certain range [2], and abnormal changes in its concentration level are symptoms of some diseases. Thus, it is desirable to develop simple and direct methods for monitoring the level of UA. EP and UA are coexistent in biological fluids of human, so it is meaningful and attractive for biological and chemical researchers to investigate the simultaneous determination of UA and EP in a mixture. A novel biosensor has been constructed by the electrodeposition of Au-nanoparticles (AuNPs) on Thioglycolic acid(TGA) film modified glassy carbon electrode (GCE) and employed for the simultaneous determination of EP and UA in the presence of a large excess of ascorbic acid (AA). SH groups exposed to the TGA layer are helpful for the electrodeposition of AuNPs. The combination of nano-Au and TGA layer endow the biosensor with large surface area, good biological compatibility, electricity and stability, high selectivity and sensitivity and flexible and controllable electrodeposition process. Bare GC electrode fails to resolve the voltammetric signals of EP and UA in a mixture. However, the modified electrode not only separates the voltammetric signals of AA, EP and UA but also shows higher oxidation current for these molecules. Using amperometric method, we achieved the lowest detection of 60 nM EP and 32 nM UA. The modified biosensor was applied to the determination of the concentration of EP in epinephrine injection and EP and UA in human blood serum by using standard adding method with satisfactory results.

Keywords: Simultaneous, Electrocatalytic oxidation, Au nanoparticles

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Graphene oxide-chitosan nanocomposite modified glassy carbon electrode as a voltammetric sensor for the determination of rutin in complex matrices

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Abstract

Chitosan is commonly used to disperse nanomaterials due to its excellent capability of film formation, nontoxicity, mechanical strength and good water permeability [1]. Graphene oxide nanosheets dispersed in a polymer matrix like chitosan have been reported to enhance electrochemical performance of the modified electrode. This study is first time showing application of graphene oxide (GO)-chitosan (CHI) nanocomposite for the determination of rutin in plant materials. Rutin is one of the most important bioactive flavonoid glycosides and has significant antioxidative, antihyperglycemic, antifungal and antihypertensive properties [2]. The GO-CHI/GCE displayed high effective surface area, more reactive sites and excellent electrochemical catalytic activity toward the oxidation of rutin. The experimental parameters such as pH, scan rate and amount of modifier were studied by cyclic voltammetry and the optimized values were chosen. The electrochemical parameters such as electrode surface area and electron transfer coefficient were calculated.

Keywords: Graphene oxide nanosheets, Chitosan, Nanocomposite, Rutin, Voltammetry

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Synthesis of nanophotocatalyst of TiO₂ doped by C,N and its application on degradation of industrial wastewater using uv light by trial and error and tagochi methods.

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Abstract

The photocatalytic degradation of sodium naftalene sulfonate has been investigated in aqueous phase using ultraviolet (UV) and TiO₂ doped C,N nanopowder. The xerogel TiO₂ powder was synthesized by hydrolysis of Ti(OCH(CH₃)₂)₄ in distilled water. The obtained white gels were dried under vacuum at 80°C for 10h and then mixed with urea and calcined at 500°C in air for 3h. The method in this study is very similar to the reported ones[1] except using different precursor and dopant. The catalyst was characterized by X-ray powder diffractometer (XRD) and energy dispersive X-ray spectroscopy (EDAX). EDAX results showed the powder is composed of C,N. XRD outputs revealed that the obtained powderes are nanostructure(15.83 nm) and the predominant crystalline phase is anatase. The photo-degradation with nitrogen and carbon-doped TiO₂ showed superior photo-catalytic activity compared to that for pure TiO₂[2]. TiO₂ mixed with urea in the ratio of 1:2 exhibited the best photo-catalytic efficiency under the different condition. Our experimental arrangement was based on testing five factors, i.e., dosage of KBrO₃, concentration of sodium naftalene sulfonate, amount of TiO₂, irradiation time and initial PH [3].

Keywords: photocatalyst, Degradation, C,N-codoped TiO₂

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Nanomolar simultaneous determination of levodopa and serotonin at a novel carbon ionic liquid electrode modified with Co (OH)₂ nanoparticles and multi-walled carbon nanotubes

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Abstract

A novel modified carbon ionic liquid electrode is prepared as an electrochemical sensor for simultaneous determination of levodopa (L-dopa) and serotonin (5-HT). The fabricated sensor revealed some advantages such as convenient preparation, good stability and high sensitivity toward 5-HT and L-dopa determination. The DPV data showed that the obtained anodic peak currents were linearly dependent on the L-dopa and 5-HT concentrations in the range of 0.25–225 and 0.05–75 μM , respectively. The applicability of the modified electrode was demonstrated by simultaneous determination of 5-HT and L-dopa in human serum. Neurotransmitters play a significant role in the research of Parkinson's disease [1]. L-Dopa is currently the therapeutic drug in the treatment of Parkinson's disease and required by the brain to produce dopamine. Low levels of 5-HT have been associated with several disorders, notably depression, migraine, bipolar disorder and anxiety [2]. As an ion-exchange polymer, Nafion films are highly permeable to cations but almost impermeable to anions [3]. The carbon ionic liquid electrode was prepared by mixing graphite powder and BMIMPF₆ thoroughly in a mortar to form a carbon paste. A portion of the carbon paste was firmly filled into one end of a glass tube and a copper wire was inserted through the opposite end. The response of a sensor is related to its physical morphology. Scanning electron microscopy was performed to the CoHNPs synthesized through the precipitation method. The amount ratio of CoHNPs was increased from 0 to 6%, the response of the electrode improved and when the ratio was more than 6%, the response decreased with larger background current. Therefore 20 μL of 6% CoHNPs nanoscale (94% MWCNTs) was chosen for the fabrication of the sensor. At optimum pH of 7.5, the negatively charged Nafion membrane electrostatically attracts L-dopa and 5-HT cations but rejects AA and UA anions. But on the other hand Nafion has weak electrical conductivity, consequently application of large amount of Nafion would decrease the mass transfer rate. The anodic current could reach the highest value when 10.0 μL of Nafion was used. However by further increasing the amount of Nafion the current decreases. The oxidation peak currents of L-dopa and 5-HT after 60 s accumulation at different potentials from 0.50 to 0.10 V remained almost stable, revealing that potential does not affect the oxidation peak currents of L-dopa and 5-HT. The best peak separation can be seen in pH 6.5. However in the solution with pH 7.5, higher anodic peak currents for both compounds were obtained. In order to investigate the effect of scan rate, cyclic voltammetry of solutions of 50 μM L-dopa and 25 μM 5-HT were obtained in the range 0.01–0.8 V s^{-1} . The results showed that the peak currents vary linearly with the scan rate over the range 10–150 mV s^{-1} for both drugs which confirm the adsorption-controlled process for electro-oxidation of L-dopa and 5-HT on the surface of the electrode. The plot of E_p versus the logarithm of scan rate ($\log(v)$) was not linear for L-dopa, but showed a linear behavior for 5-HT according to Laviron theory. The charge transfer coefficient (α) can be determined by measuring the variation of E_p vs. $\log(v)$. The slope of the E_p vs. $\log(v)$, was about, 0.0541 V. By application of the DPV method two linear ranges were obtained. The first linear dynamic range was from 0.25 to 10 μM and the second linear dynamic range was between 15 and 225 μM . A detection limit of 0.12 μM was obtained. The electrode is practically impermeable to AA and UA and other anionic species and only slightly responsive to neutral metabolites. Thus it becomes selective for the cationic neurotransmitters, L-dopa and 5-HT with high sensitivity in real biological samples. Due to the co-contribution of CILE and modifiers on the electrode surface, the resulting electrode exhibited a good electrocatalytic performance to simultaneous trace determination of L-dopa and 5-HT.

Keywords: Levodopa, Serotonin, Carbon ionic liquid electrode

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Simultaneous determination of levodopa and melatonin at a new cobalt hydroxide nanoparticles and multi-walled carbon nanotubes composite modified carbon ionic liquid electrode

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Abstract

A novel modified carbon ionic liquid electrode (CILE) is prepared as an electrochemical sensor for simultaneous determination of levodopa (L-Dopa) and melatonin (Mel). The experimental results suggest that a carbon ionic liquid electrode modified with multi-walled carbon nanotubes and cobalt hydroxide nanoparticles accelerates the electron transfer reactions of L-Dopa and Mel. The DPV data in 0.1 M phosphate buffer solution (PBS) (pH 7.5) allowed a method to be developed for the determination of L-Dopa and Mel concentrations in the ranges 0.1–300 and 0.01–50 μM , with the detection limits of 0.075 and 0.004 μM , respectively. The proposed method was successfully applied to determinations of these compounds in some pharmaceutical and human urine samples. Melatonin (Mel, N-acetyl-5-methoxytryptamine) is a lipophilic hormone, mainly produced and secreted at night by the pineal gland. The mechanisms that control its synthesis within the pineal gland have been well characterized [1]. Levodopa (L-dopa) is a naturally occurring dietary supplement and psychoactive drug found in certain kinds of herbs and food and is synthesized from the essential amino acids L-phenylalanine and L-tyrosine in the brain and mammalian body [2,3]. Some electrodes such as glassy carbon electrode [4], and carbon paste electrode [5] have been modified by Co and $\text{Co}(\text{OH})_2$ particles and nanoparticles. Cobalt hydroxide nanoparticles (CoHNPs) with a low crystallinity and nano-flake network structure show a high proton diffusion coefficient, giving excellent electrochemical performance and nano-flake network structure show a high proton diffusion coefficient, giving excellent electrochemical performance. The morphology of CHNPs shows a network-like structure which consists of interconnected nano-flakes. The SEM image shows the agglomerated CHNPs with an average size of less than 100 nm. The crystal structure of CHNPs by powder X-ray diffraction (XRD). It can be seen that several diffraction peaks appear at $2\theta = 11^\circ, 23^\circ, 34^\circ, 38^\circ, 45^\circ, 58^\circ$ and 60° , which can be indexed to planes (003), (006), (012), (015), (018), (110) and (113), respectively. The pattern matches well with that assigned to $\alpha\text{-Co}(\text{OH})_2$ (JCPDS: file no. 46-0605). The amount ratio of CHNPs to MWCNTs nanoparticles influences the sensitivity of the sensor. It was found that as the amount ratio of CHNPs was increased from 0% to 6% (w/w), the response of the electrode improved and when the ratio was more than 6%, the response decreased with larger background current, which resulted in poor determinations for L-Dopa and Mel. Therefore 20 μL of 6% CHNPs (94% MWCNTs) in DMF solution was chosen for the fabrication of the modified electrode. The anodic peak currents of L-Dopa and Mel improve with accumulation time. After 60 and 90 s of accumulation time for L-Dopa and Mel, respectively, they remained almost constant. The results showed that the oxidation potentials of L-Dopa and Mel shift to less positive potential with increasing solution pH which is a consequence of the deprotonation involved in the oxidation process that is facilitated at higher pH values. In order to investigate the effect of scan rate, cyclic voltammetry of solutions of 20 μM L-Dopa and 5 μM Mel were obtained in the range 0.01–0.8 V s^{-1} . The results showed that the peak currents vary linearly with the scan rate over the range 0.01–0.15 V s^{-1} for both compounds which confirm the adsorption-controlled process for electro-oxidation of L-Dopa and Mel on the surface of the electrode. By considering two electrons transferred for L-Dopa, cathodic and anodic charge transfer coefficients of 0.59 and 0.41 were obtained. An apparent surface electron transfer rate constant, $k_s = 9.78 \times 10^{-1} \text{ s}^{-1}$. By application of the DPV method two linear ranges were obtained. The first linear dynamic range was from 0.1 μM to 7.5 μM and the second linear dynamic range was between 10 μM and 300 μM . A detection limit of 0.075 μM was obtained.

Keywords: Levodopa, Melatonin, Carbon ionic liquid electrode

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Determination of mean activity coefficient of KCl in (KCl + sucrose + water) system based on potentiometric measurements at 298.15 K

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Abstract

Thermodynamic study of inorganic salts in mixed solvent systems is useful to understand various processes in chemical engineering, geochemistry, atmospheric aerosol chemistry, and biological systems such as proteins, amino acids, and nucleic acids [1, 2]. During the last decades, the series of ion-interaction models for electrolyte solution have been proposed to predict the activity coefficient of each solute and osmotic coefficient of aqueous systems. Among them, the pitzer ion-interaction model is one of the most famous and useful models. Pitzer equations, with much accuracy, correlate thermodynamic properties of mixed aqueous electrolyte systems [3]. In this work, the thermodynamic properties of the electrolyte system (KCl + sucrose + water), using the potentiometric method, are reported. The potentiometric measurements were performed on the galvanic cell of the type: Ag|AgCl|KCl (m), sucrose (wt.%), H₂O (1-wt) %|K-ISE, in various mixed solvent systems containing 0, 10, 20, 30, and 40% mass fractions of sucrose over ionic strength ranging from 0.0010 to 6.0000 kgmol⁻¹ at 298.15 K. The modeling of this ternary system was made based on the Pitzer ion-interaction model. The resulting values of the mean activity coefficients, the osmotic coefficients and the excess Gibbs free energy together with Pitzer ion-interaction parameters ($\beta^{(0)}$, $\beta^{(1)}$ and C^{ϕ}) for the series under investigated system are reported.

Keywords: Activity coefficients, Pitzer model, KCl, Sucrose, Potentiometric method

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Immobilization of hemoglobin onto a glassy carbon electrode modified with cobalt-oxides nanoparticles and collagen for fabricating of third generation nano-biosensor

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Abstract

The study of direct electrochemistry of redox enzymes or proteins can provide a working model to study the mechanism of electron transfer between enzymes in biological systems, and establish a foundation for fabricating new kinds of biosensors, enzymatic bioreactors and biomedical devices that do not use any mediators. For application in the biosensors, the enzymes should be immobilized on the electrode surface to avoid many complications linked to the solution systems. Therefore, suitable electrode materials and immobilization methods of enzymes onto the electrode surface are important for obtaining their direct electrochemical reaction and keeping their bioactivities. Protein film voltammetry (PFV) affords an approach to study the details of electron transfer and the coupled reactions in proteins. Collagens have been utilized for immobilizing redox proteins on electrode surface[1]. In this paper, heme proteins hemoglobin (Hb) was incorporated in collagen films modified with electrodeposited of cobalt-oxides nanoparticles on the surface of GC electrode (Hb/collagen/CoOx/GC electrode). Direct voltammetry and bioelectrocatalytic activity of Hb/collagen/CoOx/GC electrode were investigated by various spectroscopic and electrochemical techniques. The ability of biosensor for electrocatalytic reduction of hydrogen peroxide and oxygen was also evaluated. Modified electrode demonstrated a quasi-reversible CV reduction-oxidation peak pair. Heme proteins in collagen films retained their near-native conformation. The good performance of Hb/collagen/CoOx/GC electrode indicates this film provided a suitable matrix for protein immobilization and well kept the bioactivities of protein. Finally the new matrix is strongly recommended for immobilization of many other enzymes or proteins for fabricating of third generation biosensors and bioelectronics devices [2].

Keywords: Hemoglobin, Nanoparticles

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Electrochemical reduction of ketanserin at a reduced graphene oxide modified glassy carbon electrode

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Abstract

Ketanserin is a selective antagonist of 5-hydroxy tryptamine (5-HT) at 5-HT₂ receptors which also has α ₁-adrenoceptor antagonist activity [1]. Recently, reduced graphene oxide has been widely used as a mediator in electrochemical sensors [2-3]. In this work, a reduced graphene oxide modified glassy carbon electrode (RGMGCE) was developed to investigate of the electroreduction of ketanserin in aqueous solution. Electrochemical behavior of ketanserin was studied at different pH and concentrations using cyclic voltammetry and differential pulse voltammetry techniques. The RGMGCE exhibits electrocatalytic activity for reduction of ketanserin with a detection limited 5.0×10^{-6} M and a linear range of 6.0×10^{-6} to 2.8×10^{-4} M. The proposed method was successfully applied in the determination of ketanserin in water and serum samples.

Keywords: Ketanserin, Reduced graphene oxide, Modified electrode, Voltammetry

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Nickel oxide/multi-walled carbon nanotube-modified electrode for electro oxidation oftartaric acid

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Abstract

Tartaric acid is a white crystalline diprotic acid. This aldaric acid occurs naturally in many plants, particularly grapes, bananas, and tamarinds, is commonly combined with baking soda to function as a leavening agent in recipes. It is added to other foods to give a sour taste, and is used as an antioxidant. Salts of tartaric acid are known as tartrates. It is a dihydroxyl derivative of succinic acid[1].

Recent studies have demonstrated the ability of multiwall carbon nanotubes (MWCNTs) in improving electrocatalytic properties of the nickel oxide modified electrodes [2,3]. The glassy carbon electrode modified with multi-walled carbon nanotubes and nickel (II) oxide (GC/MWCNT/NiO) was examined for electro oxidation of tartaric acid in alkaline aqueous solution. The results were compared with those obtained on a nickel (II) oxide modified glassy carbon electrode. Both electrodes conditioned by potential cycling in a limited potential range (0.2-0.6 V vs. Ag/AgCl) in 0.10 M NaOH solution. The modification of electrode surface by multi-walled carbon nanotubes improves the electrocatalytic activity for tartaric oxidation. Under the optimum conditions, there was a good linear relationship between anodic peak current and tartaric acid concentration in the range of 8.0×10^{-6} M to 1.0×10^{-4} M, and a detection limit of 7.4×10^{-6} M (S/N = 3) was obtained. The prepared electrode showed high stability and reproducibility for determination of tartaric acid.

Keywords: Tartaric acid, Multi-walled carbon nanotubes, Nickel (II) oxide

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Amperometric glucose sensor based on the modified MWCNTs with copper nanoparticles**B. Rezaei*, N. Askarpour, M. Ghiaci, F. Niyazian and A. A. Ensafi***Department of Chemistry, Isfahan University of Technology, Isfahan 84156-83111, I.R.Iran**Corresponding author E-mail: Rezaei@cc.iut.ac.ir***Abstract**

Worldwide, diabetes is a rapidly growing problem that is managed at the individual level by monitoring and controlling blood glucose levels to minimize the negative effects of the disease. Because of limitations in diagnostic methods, significant research efforts are focused on developing improved methods to measure glucose. Nanotechnology has impacted these efforts by increasing the surface area of sensors, improving the catalytic properties of electrodes and providing nanoscale sensors [1]. The functionalization of CNTs with various functional groups is an effective way to prevent them from aggregation allowing for stable and better dispersion of metal nanoparticles on the surface of them. There are several approaches for functionalizing CNTs, which include defect, noncovalent, and covalent functionalization. In the case of covalent functionalization, the functional groups at the surface of CNTs make the strongest type of interfacial bonding with the metal nanoparticles [2]. In these years, carbon nanotubes (CNTs) have more attention for the preparation of metal-CNT hybrid materials with enhanced mechanical and conducting properties. Cu nanoparticles (CuNPs) as an important class of metal nanostructure have been attracted increasingly attention owing to their high electrical conductivity and low cost [3]. New functionalized-multiwall carbon nanotubes (f-MWCNTs) with NH_2 groups were synthesized and then were decorated with redox active copper nanoparticles. A novel amperometric sensor for the detection of glucose was prepared by immobilizing the new nanocomposite (CuNPs/f-MWCNTs) on the surface of GC electrode. The modified electrode was characterized using transmission electron microscopy, X-ray photoelectron spectroscopy and energy dispersive X-ray analysis. The electrochemical performances of the electrode were studied by cyclic voltammetry and electrochemical impedance spectroscopy. The f-MWCNTs decorated with CuNPs immobilized on the surface of electrode exhibited an excellent electrocatalytic response toward oxidation of glucose. The preparation process of the proposed sensor was convenient, and the resulting sensor showed high sensitivity, low detection limit and good stability. The capability of the sensor for analyses in real sample was demonstrated by the detection of glucose level in human serum samples with satisfactory recovery values.

Keywords: Glucose sensor, Amperometric detection, Functionalized-multiwalled carbon nanotubes, Copper nanoparticles

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In situ surfactant modified graphene oxide nanosheetselectrode for electrochemical investigation of clozapine

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Abstract

Clozapine (CLZ) is an atypical antipsychotic drug. This drug is very effective against both the negative and positive symptoms of schizophrenia and has been successfully used in patients who are “non-responder” to the classical neuroleptic drugs. Due to its importance, several analytical methods have been applied for the determination of CLZ in pharmaceutical and biological samples [1]. In comparison, electroanalytical methods have advantages such as high sensitivity, inexpensive equipments, low costs and relative simplicity. In the present study, the electrochemical behavior of CLZ was investigated using in situ surfactant modified graphene oxide nanosheets (ISS-GONs) electrode by voltammetric techniques, and the effects of some experimental variables on the electro-oxidation of this substance were investigated [2]. The GONs modified electrode was prepared in situ on a graphite electrode (GE) by using cyclic voltammetry scanning between 0.0 and 2.0 V (vs. Ag/AgCl) for four cycles [3]. In the next step, the modified electrode was inserted in adodecyltrimethylammonium bromide surfactant solution for 5 min to in situ modification of the electrode surface. Finally, the modified electrode (ISS-GONs/MGE) was successfully used for the electrochemical determination of CLZ under the optimized conditions by cyclic and differential pulse voltammetry. At the optimum conditions, the peak current was linearly dependent on the CLZ concentration in the range of 0.01 to 75 μ M with detection limit of 5.94 nM. The proposed electrode was successfully applied for the determination of CLZ in human blood serum and plasma samples.

Keywords: Clozapine, Surfactant, Graphene oxide nanosheets, Modified electrode, Voltammetry

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Organofunctionalized nanoporous silica gel based on carbon paste electrode for potentiometric determination of copper (II) ions

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Abstract

The application of chemically modified electrodes in analytical chemistry has attracted considerable attention[1–3]. In this work, a novel chemically functionalized nanoporous silica by N¹-(3-(9H-carbazol-9-yl)propyl)-N³-propylpropane-1,3-diamine (CPDNS) was incorporated into carbon paste electrode for the potentiometric detection of Cu(II) ions in aqueous medium. With 9.4 wt% CPDNS proportions, the developed electrode exhibited wide dynamic range of 1.0×10^{-6} to 1.0×10^{-2} M toward Cu(II) with a detection limit of 6.0×10^{-7} M and a Nernstian slope of 27.11 ± 1.0 mVdecade⁻¹. The electrode displayed rapid response (~46 s), long-time stability (more than 6 months), and high sensitivity. Moreover, the potentiometric responses could be carried out with wide pH range of 6.8–11.0. Finally, the proposed electrode was successfully used as an indicator electrode in potentiometric titration of copper ion with EDTA and in direct determination of copper (II) ion in sea water, tabs water and tea leaves samples.

Keywords: Functionalized nanoporous silica gel, Carbon paste electrode, Copper (II) ion

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Direct electrochemistry of Cyt c immobilized onto modified titanium oxide nanoparticles and application to hydrogen peroxide sensor

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Abstract

Recently, direct electron transfer between proteins and electrodes has been widely investigated due to its broad application in many areas. Among these proteins, the study of direct electron transfer between cytochrome c (Cyt c) and electrode has gained increasing attention. Cyt c plays an important role in the biological respiratory chain. Due to the protein denaturation it is difficult for Cyt c to exhibit a voltammetric response at a bare electrode. Therefore different modified electrode widely used to this purpose. In this work an organic-inorganic nanocomposite consisting of modified TiO₂ with 2, 2'-dithio-3, 3'-bis(3-(triethoxysilyl)propyl);(DTP) and graphene was provided for enzyme immobilization and investigate the direct electrochemistry of Cyt c. in the first step DTP was covalently attached to TiO₂ nanoparticles and employed for obtaining a suitable solid surface enzyme attachment. In the second step, cyclic voltammetry used to immobilization of Cyt. c onto TiO₂-DTP. The Cyt c modified electrode known as Cyt c/GO/TiO₂-DTP. The voltamograms displayed a well-known and nearly symmetric redox couple corresponded to Cyt. c with a formal potential of -0.1 in PH 7.0 phosphate buffer solution (PBS). Electrochemical behavior of Cyt c/GO/TiO₂-DTP was studied in different scan rate and different pH solutions. Electrocatalytic activity of modified electrode toward oxygen and hydrogen peroxide evaluated by cyclic voltammetry. The results implies to good catalytic activity of modified electrode. In order to achieve the best catalytic parameters such as detection limit and dynamic linear range hydrodynamic amperometric method was used. The results indicated that Cyt c modified electrode should be used as a good sensor for detection of hydrogen peroxide.

Keywords: Cytochrome, Direct electron transfer, Hydrogen peroxide

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Electrodeposition and Corrosion Resistance Properties of Co-W/PCTFE Nanocomposite Coatings

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Abstract

Recently, a wide range of researches have concerned on the deposition and properties of cobalt alloys attributable to the large-scale of applications in all of the industrials respected to corrosion science [1]. Therefore, the incorporation of second phase of solid particles such as PTFE, SiC, MoS₂, Al₂O₃, etc. Improved the mechanical and chemical properties such as lubrication, wear resistance and corrosion resistance of cobalt alloys [2]. The present work deals with the process of electrochemical deposition of cobalt–tungsten /polychlorotrifluoroethylene (Co-W-PCTFE) nanocomposite coatings in the tartrate bath and electrochemical corrosion behavior in 3.5 wt. % NaCl solutions. The process of reduction of cobalt in cobalt sulfate, sodium tungstenat and sodium potassium tartrate solution with different consternation (0-20 g/L) of PCTFE suspended particles in the 5 different plating baths has been employed for the configuration of the electrochemical Co-W -PCTFE composite coatings. Surface morphology and composition of the composite coatings are characterized by energy dispersive X-ray (EDX), scanning electron microscopy (SEM). Corrosion behavior of coatings is appraised applying open-circuit potential (OCP) measurements, electrochemical impedance spectroscopy (EIS) and polarization techniques in 3.5 wt. % NaCl solution. The study reveals reduce in corrosion current density, important substitute in corrosion potential towards the positive direction with the incorporation of PCTFE particles in the Co-W matrix. The PCTFE particles added to the Ni-P alloy [3] was observed the similar conclusion of the PCTFE particles roles in corrosion resistance. Therefore, the meaningful improvement in corrosion resistance observed for Co-W-PCTFE nanocomposite coatings (7.587kΩ cm²) compared to Co-W (2.325 kΩ cm²). The results of SEM images indicated to decrease of PCTFE particle sizes of micrometric to nanometeric sizes and EDX analyzes confirmed the forming of Co-W-PCTFE nanocomposite coatings.

Keywords: Co-W-PCTFE, Electrodeposition, Nanocomposite, Tartrate bath

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Preparation and electrochemical investigation of nanostructured iron(III) titanate

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Abstract

The direct photoelectrolysis of water was first achieved with a TiO_2 semiconductor photoelectrode in a pioneer work reported by Fujishima and Honda [1]. However, this semiconductor requires a photonic energy of at least 3.2 eV and so only a small fraction of solar spectrum can be used. Significant improvements have been made to find new oxide semiconductors with small and well positioned band gaps, proper to harvest visible light and capable of conducting alone water splitting under solar radiation with attractive efficiencies [2]. The various titanates $\text{M}_x\text{Ti}_y\text{O}_z$, where M is an alkaline earth or transition metal, have received considerable attention recently as possible photoanodes for the electrolysis of water. In this study, we have prepared nanostructured Fe_2TiO_5 with a modified low temperature Sol-Gel method, which we have developed in our laboratory for preparation metal titanates [3]. Briefly, to prepare the sol of Fe_2TiO_5 appropriated amount of $\text{Fe}(\text{NO}_3)_3$ was dissolved in EG, and then the alcoholic solution of tetrapropylorthotitanate gradually was added to the above solution. The obtained gels were kept at 70°C for 5 hours and were calcinated at 650°C for 3 hours. A mechanism was suggested for preparing of the final product in terms of TGA analysis. The crystal structure and morphology of prepared materials were studied by XRD and TEM analysis. The band gap of prepared Fe_2TiO_5 was determined by diffuse reflectance UV-Vis spectroscopy. Finally, the electrochemical behavior of nanostructured iron(III) titanate was studied by making a carbon paste electrode containing the appropriate amounts of graphite, n-eicosan and nanostructured Fe_2TiO_5 in a standard three electrodes cells containing of a platinum grid, Ag/AgCl and carbon paste as auxiliary, reference and working electrodes, respectively, using a Solartron electrochemical work station. The electrolyte was NaOH 1M. The obtained cyclic voltammograms showed a completely different behavior for electrochemical water splitting compared to Fe_2O_3 . The results were interpreted in terms of the electronic structure of the materials.

Keywords: Nanostructure, Iron (iii) titanate, Water splitting

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Determination of activity coefficient of calcium chloride in the ternary electrolyte system $[\text{CaCl}_2 + \text{Ca}(\text{NO}_3)_2 + \text{H}_2\text{O}]$ by the potentiometric method at $T = 298.15$

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Abstract

In the last decades, a series of ion-interaction models for electrolyte solutions have been proposed to predict the activity coefficient of each solute and the osmotic coefficient of aqueous systems. One of the most famous and useful models is that proposed by Pitzer [1, 2]. The potentiometric method using a galvanic cell without liquid junction was found to be an attractive experimental technique for studying the thermodynamic properties of electrolytic solutions [3]. Compared to the isopiestic method, this technique presents advantages, such as rapidity and relative simplicity to generate experimental electromotive force (emf) data. Thermodynamic properties of the aqueous binary and ternary electrolyte solutions are useful in the prediction of behavior of mixed salt electrolyte systems, development the electrolyte solutions models, estimation of the interactions occurring in these solutions and so on. In this work, the Pitzer ion interaction model were used to illustrate the ternary electrolyte system $[\text{CaCl}_2 + \text{Ca}(\text{NO}_3)_2 + \text{H}_2\text{O}]$. The activity coefficients were determined using a potentiometric method. The galvanic cell used had no liquid junction of type: $\text{Ca}^{2+}\text{-ISE}|\text{CaCl}_2 (m_A), \text{Ca}(\text{NO}_3)_2 (m_B), \text{H}_2\text{O}|\text{Ag, AgCl}$. The measurements were performed at $T = 298.15$ K and at total ionic strengths from 0.001 to 5.000 mol.kg^{-1} for different series of salt ratios $r = \frac{m_{\text{CaCl}_2}}{m_{\text{Ca}(\text{NO}_3)_2}}$ pure, 10, 15. The PVC based calcium ion-selective electrode ($\text{Ca}^{2+}\text{-ISE}$), and the Ag/AgCl electrode used in this work were prepared in our laboratory and showed a reasonably good Nernst response. The experimental results showed that Pitzer model were suitable to be used satisfactorily to describe this ternary system. His application of pair electrodes used (ion selective electrode and Ag-AgCl) was confirmed by potentiometric determination of mean activity coefficients of single electrode and comparing them with those calculated by Pitzer ion-interaction model. By potentiometric determination of mean activity coefficient in the mixed electrode system, the Pitzer mixing parameters (θ and ψ) were determined. Then these parameters used for determination of thermodynamic properties of investigated system such as osmotic coefficient and Gibbs free energy.

Keywords: Thermodynamic properties, CaCl_2 , $\text{Ca}(\text{NO}_3)_2$, Pitzer model, Potentiometric method

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Comparative electrochemical studies of Fe₂O₃ and Fe₂O₃-TiO₂ nanocomposite for water splitting**Hossein Farsi^{*}, Sokufeh Moghiminia and Nasrin Salimi***Department of Chemistry, University of Birjand, Birjand, Iran**E-mail: hofarsi@Birjand.ac.ir***Abstract**

The photoelectrolysis of water using n-type semiconducting metal oxide anodes and Pt cathodes is well documented. TiO₂ (E_g=3.0 eV), SrTiO₃ (E_g=3.3 eV) and Fe₂O₃ (E_g=2.2 eV) are particularly well-studied examples. However, while all of these materials show considerable promise, they fall short of being the optimum electrode material. Three primary requirements are needed for such an electrode. It must be inert under any conceivable chemical or photoelectrochemical conditions encountered in the cell. Its band gap should be on the order of 1.5-2.0 eV so as to utilize a large portion of the solar spectrum. Ideally, the system should operate effectively at zero bias so as to obviate the need for an external power supply [1-3]. Against of good chemical stability of TiO₂, it has a large band gap. Although Fe₂O₃ has the right band gap, but it has some stability problems and suffers from poor zero-bias characteristics. A way to overcome these problems can be making their nanocomposites. In this study, we have prepared Fe₂O₃-TiO₂ nanocomposites by impregnation method. Briefly, the appropriate amounts of nanoparticles of TiO₂ were immersed in Fe(NO₃)₃ solutions with different concentrations for 24 hours. Then the solid materials were filtered, washed for several times by de-ionized water and dried at 100°C. Then dried materials were calcined up to 450 °C in oxygen atmosphere. The crystal structure and morphology of the prepared materials were certified by XRD and TEM. The compositions of nanocomposites were determined by atomic adsorption spectroscopy analysis. The band gaps of prepared materials were determined by diffuse reflectance UV-Vis spectroscopy. Finally, the electrochemical studies of nanocomposites and also Fe₂O₃, containing cyclic voltammetry and electrochemical impedance spectroscopy, were carried out by making a carbon paste electrode containing the appropriate amounts of graphite, n-ecosan and nanocomposites or Fe₂O₃ in a standard three electrodes cells containing of a platinum grid, Ag/AgCl and carbon paste as auxiliary, reference and working electrodes, respectively, using a Solartron electrochemical work station. The difference between the electrochemical behaviors of nanocomposites and Fe₂O₃ were interpreted in terms of their electronic structure.

Keywords: Nanocomposite, Fe₂O₃-TiO₂, Electrochemical water splitting

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Direct electrochemistry of adenine on Nafion/GO/Ag-TNFs composite film modified screen printed carbon electrode and its determination in DNA

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Abstract

Adenine and guanine are the building blocks of both deoxyribonucleic acid (DNA) and ribonucleic acid (RNA) that plays a crucial role in protein biosynthesis and the storage of genetic information [1]. Selective and sensitive detection of the purine bases provides valuable insights in fundamental fields such as understanding of DNA sequence, oxidative damage and hybridization and protein metabolism in cells and protein-DNA interactions [2, 3]. Adenine as an important bases of nucleic acids, are often analyzed by separation coupled spectroscopic detection methods. Herein, we are demonstrated a method for rapid sensitive detection of adenine using a composite screen-printed carbon electrode (SPCE) modified with graphite oxide (GO) and silver doped titanium dioxide nanofibers (Ag-TNFs). The synthesized Ag-TNFs were analyzed by XRD and the Ag-TNFs/GO/SPCE was characterized by scanning electron microscopy. The modified electrode showed higher surface area and electrocatalytic activity compared to bare electrode. The proposed electrode showed good electrochemical characteristics for the direct electrochemical oxidation of adenine in buffer phosphate solution (pH 5.0) which led to indicator-free and sensitive detection of adenine concentrations in human DNA.

Keywords: Adenine, Screen printed carbon electrode, Ag doped titanium dioxide nanofibers, Human DNA

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Simultaneous determination of epinephrine-acetaminophen using POM nanoparticles modified carbon paste electrode by differential pulse voltammetry

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Abstract:

A highly selective voltammetric method was developed for the simultaneous determination of Epinephrine (EP) and acetaminophen (AC), using polyoxometalate nanoparticles-modified carbon paste electrode (POMNMCPE). The modified electrode separated the anodic oxidation peaks potential of EP-AC with a well-defined peak separation in the presence of each other to measure EP-AC individually or simultaneously without any intermolecular effect. The calibration curves for EP and AC were obtained in the range of 40-140 and 20-90 μM , respectively. The detection limits were 2.52×10^{-4} and 1.42×10^{-12} M for EP and AC, respectively. The interference of potential interfering substances on the determination of EP and AC were studied, and the results confirm the selectivity of the method. The modified electrode was used for the determination of EP-AC simultaneously in real samples such as drugs and human serum with satisfactory results.

Keywords: Epinephrine, Acetaminophen, polyoxometallate nanoparticles, Modified carbon paste electrode

Optimization electrophotocatalytic removal of nitrate from drinking water by Taguchi model

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Abstract

Profligate utilization of nitrogenous fertilizers, septic tank, municipal sewage treatment system, and decaying plant debris pose the greatest danger to the water sources [1]. Water treatment by electrophotocatalytic (EPC) methods is widely used in the recent years [2]. These methods lead to producing hydroxyl (OH[•]) radical [3]. The goal of this applied-analytical research is to investigate of nitrate removal from drinking water by batch EPC reactor with using zinc oxide (ZnO) nanoparticles immobilized on zinc (Zn) sheet-copper electrode, and lamp emitting dynode (LED) ultraviolet-A (UV-A) lamp. Various operating variables are tested for their effects on nitrate removal; these include current density, distance between lamp and electrode, initial concentration of nitrate, lamp intensity, layering of ZnO nanoparticles, pH, and radiation time. To prepare the ZnO films on the Zn electrode, dry methods are used. The contaminated water sample is prepared by adding 10-100 milligrams of nitrate per ml of drinking water. The studied variables are pH (5.5-8.5), the nitrate concentration (10-100 mg l⁻¹), the lamp intensity (120-360 mW cm⁻²), radiation time (10-40 min), the distance between lamp and electrode (1.5-3.5 cm), layering of zinc oxide nanoparticles (1-3), and current density (1-8 mA cm⁻²). The Nitrate is measured according to procedure detailed in standard method 4500B. Optimal removal (0) is obtained at pH 8, radiation time of 7.5 minutes, 2- layer of ZnO nanoparticles, and current density of 3 mW cm⁻². The nitrate decay follows a first order reaction. The results of nitrate removal efficiency by Taguchi model show that reaction time is the most important variable. The electrochemical (E) process is the least efficiency. The nitrate is slowly removed by OH[•] radical in electrocatalytic (EC) and much more quickly photolyzed by LED UV-A lamp in EPC. Results show that the application of this treatment is preferable at low current densities and high substrate concentrations to obtain the best mineralization current efficiencies. Thus, batch experiments indicate that the EPC reactor can be efficient in removing nitrate from drinking water and may be a promising technology for treating nitrate-polluted drinking water.

Keywords: Drinking water, Electrophotocatalytic, Nitrate, Taguchi model, Zinc oxide

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Electrodeposition of novel MnO_x nanostructures on nafion film: improvement of electrocatalytic activity toward H₂O₂ reduction

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Abstract

H₂O₂ is the product of reactions catalyzed by a large number of oxidases. It is essential in chemical, biological, food production, paper bleaching, sterilization clinical and many other applications. So, sensitive, rapid, and low-cost detection of H₂O₂ is of great practical importance in various fields such as medicine, food control and environmental analysis [1]. Electrochemical have been proved to be an inexpensive and effective way for hydrogen peroxide detection. The direct reduction or oxidation of H₂O₂ at bare electrode is not suited for analytical applications due to slow electrode kinetics and high overpotentials required for redox reactions of hydrogen peroxide. So, different redox mediators immobilized on the electrode surfaces have been used for decreasing of overvoltages and increasing of electron transfer kinetics. Manganese dioxide (MnO₂) is an important functional oxide with distinctive properties and has wide applications in catalysis, molecular adsorption, biosensor and energy storage. Recently, MnO₂ has appeared as one of the most promising electrode materials due to its low cost, high electrochemical activity, and environmentally friendly nature[2, 3]. In the present study the MnO_x nanostructures electrodeposited on the glassy carbon electrode modified with nafion film, using cyclic voltammetry as electrodeposition technique. The proposed procedure appears to be a highly efficient method for the development of a new class of MnO_x nanostructures with Peroxidase-like activity, which can be used in fabrication of electrochemical sensor. The electrochemical activity of modified glassy carbon electrode (GCE) with MnO_x /nafion film for detection of H₂O₂ is investigated by CV and differential puls voltammetry techniques. This modified electrode shows excellent electrocatalytic activity toward H₂O₂ reduction at unusual reduced overvoltage. The reduction potential of H₂O₂ is carried out at 0.576 V versus Ag/AgCl reference electrode in acetate buffer solution with pH 5.5. The detection limit of the proposed sensor was 0.5 nM at dynamic range 5-500 nM, using DPV as measuring technique. The application of the modified electrode toward hydrogen peroxide detection in real sample was also investigated.

Keywords: Manganese Oxide, Nafion, Hydrogen peroxide, differential puls voltammetry

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Electrocatalytic determination of traces of hydrazine by a glassy carbon electrode modified with palladium-gold Nanoparticles

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Abstract

It efficiently oxidizes hydrazine at a low overpotential of -0.26 V versus SCE. The Pd-AuNPs with an average size of 50-80 nm uniformly dispersed at the GCE. The Pd-AuNPs/GCE was used for determination of hydrazine in phosphate buffer solution of pH 7.0. The amperometric current response of the electrode was increased linearly over a hydrazine concentration of 0.1- 500 μM with a limit of detection of 0.07 μM . The prepared hydrazine sensor exhibited high sensitivity, good selectivity reproducibility and long term stability.

Keywords: Palladium/gold nanoparticles, Electrocalalytic oxidation of hydrazine, Amperometric sensor

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Electrochemical Determination of Celecoxib on a Graphene Based Carbon Ionic Liquid Electrode Modified with Gold Nanoparticles and Its Application to Pharmaceutical Analysis

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Abstract

Characterized by a scanning electron microscopy technique and electrochemical impedance spectroscopy. The prepared electrode showed excellent electrocatalytic activity in the reduction of celecoxib in a phosphate buffer solution, leading to remarkable enhancements in the corresponding peak currents and lowering of the peak potential. The advantages are related to the unique properties of graphene and gold nanoparticles such as a large surface area and increased electron-transfer abilities. Differential pulse voltammetry was applied to the quantitative determination. The calibration curve was linear in the concentration range of 0.5 to 15 μM and the detection limit was about 0.2 μM ($S_b/N = 3$). A glassy carbon electrode modified with palladium/gold nanoparticles was successfully prepared by an electrodeposition process.

Keywords: Celecoxib, Modified electrode, Graphene, Differential pulse voltammetry

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Preparation of TiO₂-Containing nano-sized mordenite for a photocatalytic system

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Abstract

Titanium oxide in anatase form is a worthwhile photo catalyst. It is possible to fix TiO₂ in a resistant and high surface area support to increase its ability for pollutants degradation [1]. In this work classical mordenite are transformed to high surface area through proper conditions including treatments in alkaline solutions by varying the factors such as treatment duration, alkaline strength and presence of ultrasound waves. Then TiO₂ was introduced into the structure of the treated mordenite using tetraisopropyl orthotitanate. A calcination temperature of 500° C was used to prepare the photocatalysts. Using these photocatalysts enhanced the efficiency of the degradation of the tested pollutants including methylene blue as a dye. A spectrophotometer was used to record the progress of the degradation. The results showed that the prepared nano-sized photocatalysts have different ability for elimination of the pollutant with respect to the type of the treatments.

Keywords: Nano-sized, Treated mordenite, TiO₂, Photocatalysts

Reference

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Electrochemical oxidation of hydrogen peroxide at gold nanosheets composite electrode

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Abstract

Hydrogen peroxide has wide applications in environmental, pharmaceutical, clinical, and industrial research. It is also a by-product of reactions catalyzed by a large number of oxidase enzymes [1]. Many analytical methods have been reported for the determination of hydrogen peroxide, including spectrophotometry, chemiluminescence and electrochemistry [2]. Among these methods, electrochemistry has become a subject of considerable interest because of its low detection limit, high selectivity and sensitivity. Recently, a new metal nanocomposite electrode has been fabricated that uses good properties of precious metals in nano scale (e.g., Ag, Au, and Pd) as conducting phase and IL (namely, OPFP) as the binder [3]. In this work, a selective and sensitive Au nanosheet composite electrode was used for the electrochemical determination of hydrogen peroxide. Cyclic voltammograms of hydrogen peroxide showed well-defined oxidation signal at the potential of 0.5 V vs. Ag/AgCl. Amperometric method, as a fast and simple method, was used for quantification of hydrogen peroxide in micro and milimolar levels. Two linear ranges of 0.7 to 300.0 μM and 300.0 μM to 15.0 mM were obtained.

Keywords: Electrochemical sensors, Nano-composite electrodes, Gold nanosheets, Hydrogen peroxide

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Simultaneous Determination of Ascorbic Acid, Uric Acid, Acetaminophen, and Mefenamic Acid Glassy Carbon Electrode whit Room Modified Temperature Ionic Liquid/Multiwalled Carbon Nanotube/Chitosan composite

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Abstract

A room temperature ionic liquid (RTIL), multiwalled carbon nanotube (MWCNTs) and chitosan (CHIT)-modified glassy carbon electrode (RTIL-MWCNTs-CHIT/GCE) is fabricated and the electrochemical behaviors of ascorbic acid (ASC), uric acid (URI), acetaminophen (ACT), and mefenamic acid (MEF) are investigated using cyclic voltammetry (CV), chrono amperometry (CA), and differential pulse voltammetry (DPV) as diagnostic techniques[1]. Application of the DPV method shows that the linear calibration curves of ASC, URI, ACT, and MEF are obtained in the range of 4.0×10^{-5} to 4.0×10^{-3} mol L⁻¹, 2.0×10^{-6} to 4.5×10^{-4} mol L⁻¹, 1.0×10^{-6} to 4.0×10^{-4} mol L⁻¹, and 2.0×10^{-6} to 6.5×10^{-4} mol L⁻¹, respectively. The electrochemical responses of the RTIL-MWCNTs-CHIT/GCE were characterized by differential pulse voltammograms (DPVs). Fig. 1 showed the DPVs of 1400 μ mol L⁻¹ ASC, 150 μ mol L⁻¹ URI, 150 μ mol L⁻¹ ACT and 200 μ mol L⁻¹ MEF on the bare GCE (Fig. 1a), CHIT/GCE (Fig. 1b), MWCNTs-CHIT/GCE (Fig. 1c) and RTIL-MWCNTs-CHIT/GCE (Fig. 1d) in PBS (pH 7.0). As can be seen from the GCE data a very small oxidation peak is observed for ASC, URI, ACT and MEF oxidations. The DPVs of ASC, URI, ACT and MEF at RTIL-MWCNTs-CHIT/GCE showed excellent improvement in oxidation peak currents for ASC, URI, ACT and MEF oxidations (Fig. 1d). The presence of MWCNTs and RTIL appears to facilitate the electron transfer between electrode and the analytes causing the enhancements in the corresponding electrochemical oxidation peak currents. In addition the presence of RTIL and CHIT lead to the oxidation peak of URI shifts to less positive potentials and leads to more current peak separation between URI and ACT. Moreover, low cost and simplicity, wide linear range, good reproducibility, and stability are the important advantages of this modified electrode. The analytical performance of this sensor is evaluated for the detection of ASC, URI, ACT, and MEF in human serum and human urine with satisfactory results.

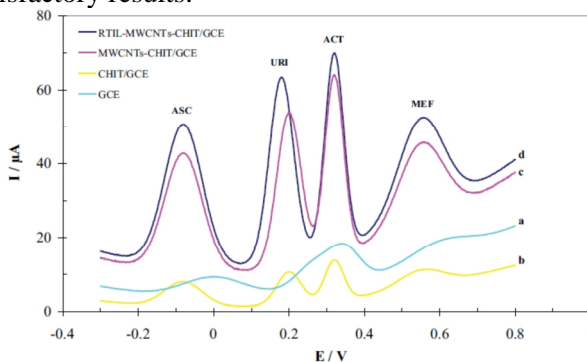


Fig. 1. Differential pulse voltammograms of ASC, URI, ACT and MEF in 0.1 mol L⁻¹ phosphate buffer solution.

Keywords: Ascorbic acid, Uric acid, Acetaminophen, Mefenamic acid, Multi-walled carbon nanotubes

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Electrochemical Determination of Erythrosine in Real Sample using a Carbon Paste Electrode Modified with Multi-Walled Carbon Nanotube

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Abstract

Erythrosine (Fig. 1) is a reddish–pink synthetic dye, which is water soluble and most popularly used as a food coloring agent and a host of other applications such as printing inks, a dental plaque disclosing agent [1]. It is also used in drugs and cosmetics, cocktails, tinned fruits, garlic sausage, scotch eggs and snack foods. It is highly toxic, causing various types of allergies, thyroid activities, anemia, and DNA damage behavior. It is also carcinogenic in nature [2]. In this work, a multi-walled carbon nanotubes modified carbon paste electrode (MWCNT-CPE) was fabricated, and the electrochemical oxidation of Erythrosine was studied in phosphate buffer (PBS) at pH = 10.0 using Cyclic voltammetry (CV), chronocoulometry (CHA) and differential pulse voltammetry (DPV) techniques. The experimental parameters, such as pH, scan rate, pulse height, accumulation time and amount of MWCNT were optimized for determination of Erythrosine. All the voltammetric measurements were carried out using an Autolab potentiostat-galvanostat model PGSTAT30 with a three-electrode system that used a carbon paste working electrode, a Ag/AgCl and a Pt wire as the reference and counter electrode, respectively. After optimizing the experimental conditions, electrochemical parameters including the diffusion coefficient (D), the electron transfer coefficient (α), and the electron transfer number (n) were determined for Erythrosine on the surface of MWCNT-CPE. The peak current increased linearly with the Erythrosine concentration within the concentration range of 0.1 to 10.0 μ M for DPV. The detection limits (S/N > 3) for Erythrosine was found to be 5.0 nM in phosphate buffer solution (PBS) at pH 10.0. The effect of foreign spices on the voltammetric determination of Erythrosine was studied and the tolerance levels were obtained. Finally, this modified electrode was applied to the determination of Erythrosine in drinks and cosmetic products with good recovery. This method possesses many advantages such as a low detection limit, fast response, low cost and simplicity.

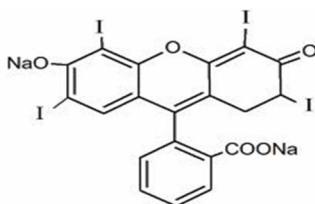


Fig.1. Chemical structure of Erythrosine

Keywords: Voltammetry, Erythrosine, Multi-walled carbon nanotubes

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Ethanol/O₂ enzymatic fuel cell composed carbon nanotube composite

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Abstract

Biofuel cells are important to generate alternative energy. This paper evaluates the electro activity of a carbon felt electrode containing immobilized 1-ethyl-3-methylimidazolium bis (trifluoromethylsulfonyl) imide (emimNTf₂)- carboxylated multiwalled carbon nanotubes gel (emimNTf₂-HOOC-MWCNTs), a NAD⁺-dependent alcohol dehydrogenase enzyme (ADH), poly-methylene green (poly-MG), and a supporting agent (polydiallyldimethylammonium chloride, PDDA). The PDDA is an excellent medium to achieve ADH immobilization on the carbon nanostructure electrode. We obtained power density and open circuit voltage (OCV) of 0.250 mW cm⁻² and 0.350 V, respectively for this modified composite of carbon nanotube. This new anode configuration is potentially applicable for small energy devices.

Keywords: Biofuel cell, 1-ethyl-3-methylimidazolium bis (trifluoromethyl sulfonyl) imide, polydiallyldimethylammonium - chloride, Carboxylated multiwalled carbon nanotubes, NAD⁺ - dependent alcohol dehydrogenase enzyme

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The Verification of Effect of a Nano-sulfur(IV) Thin Layer upon the Electrode Surface for Detecting Biomolecules

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Abstract

In this research, the ECR (electron cyclotron resonance) nano-sulfur(IV) thin layer electrode was studied with respect to morphological and characteristic changes caused by the ECP (electrochemical pretreatment) and also the usage of pretreated ECR nano-sulfur(IV) thin layer electrode for measuring some biologically important analytes, including glutathione (oxidized form, GSSG) and serotonin was investigated [1]. The electron transfer rates of several electroactive species at the ECR nano-sulfur(IV) thin layer electrode are higher than those at the BDD electrodes since the ECR nano-sulfur(IV) thin layer electrode has more sp^2 bonds [1]. Investigation of the ECP of the ECR nano-sulfur(IV) thin layer electrode will be significant in extending its application to various biomolecules. The ECP was done using cyclic voltammetry (CV) between 0 and 2 V for the ECR and the GC or 0 and 2.3 V for the BDD at a scan rate of 0.1 V/s 10 times in a phosphate buffer (PB) solution (pH = 7.0). We studied the basic electrochemical properties of ECR nano-sulfur(IV) thin layers using CV with several redox species. We also studied electrochemical overpotential and stability when measuring biomolecules including GSSG and serotonin using CV in 0.1 M pH = 7.0 PB. The surface properties, structure and electrochemical performance of the ECR nano-sulfur(IV) thin layer electrode were compared with results obtained for other conventional GC and BDD electrodes. Unlike GC, the ECR nano-sulfur(IV) thin layer almost retained its surface flatness after the ECP. This robust surface could be caused by 42 % of the sp^3 sulfur (IV). The electrode activity of ECR nano-sulfur(IV) thin layer was improved due to a sufficient quantity of surface sp^2 bonds remained and the introduction of surface oxygen-containing groups is more efficient than with the BDD electrode. Since ECR nano-sulfur(IV) thin layer maintains its wide potential window and shows high electrode activity with very little increase in C_0 after the ECP, we employed the thin layer electrode to detect biomolecules such as GSSG [2]. The oxidation peak was not observed at the untreated ECR nano-sulfur(IV) thin layer electrode at potentials below 1.7 V. at any rate, the oxidation peak appeared clearly at 1.37 V for the pretreated ECR nano-sulfur(IV) thin layer electrode. This result indicates that the GSSG peak potential for the ECR nano-sulfur(IV) thin layer electrode was reduced by the ECP due to the increase in the surface hydrophilicity with a sufficient quantity of sp^2 bonds on the surface for GSSG oxidation.

Keywords: ECP, BDD, ECR, Nano-sulfur(IV) thin layer

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Electrochemical growth of self-organized hematite nanostructures layers: Effects of applied voltage and time

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Abstract

Formation of nanoporous and nanotubular oxide layer onto different valve metal substrates such as Al, Ti, Hf, Zr, Nb, and W by electrochemical anodization route has been investigated widely [1]. These self ordered nano-oxide structures find a wide spectrum of applications such as templates for nanowire growth, photo-catalysts, photovoltaic electrodes, host for Li-ion intercalation, sensors etc. Among various nanostructure morphologies, nanotubular configuration gives an extra degree of freedom in its wall thickness that can be varied (in addition to diameter and length) for tuning the required properties. Further, nanotubes have a larger surface area than nanowires for a given diameter and length. Anodization route is attractive for the formation of ordered arrays of oxide nanotubes because of its low cost and ability to synthesize large area electrodes. Since the nanotubes/nanopores are grown onto an electrically conducting parent substrate, a separate current collector is not required. Therefore, a costly electrode preparation step is avoided for many applications such as electrodes for photovoltaic cell and battery. Furthermore, this configuration results in ohmic type contact between the semi-conducting material and the substrate [2]. Iron oxide (α -Fe₂O₃, hematite) is one of promising materials for photo-catalytic application due to its narrow band gap at about 2.1 eV, which absorbs light up to 600 nm, collects up to 40% of the solar spectrum energy, and it maybe one of the cheapest semiconductor materials. Several methods have been used for the fabrication of α -Fe₂O₃, including ultrasonic spray pyrolysis, template, chemical vapor deposition, atomic layer deposition, hydrothermal, sol-gel deposition, magnetic sputtering and electrochemical anodization. Among several methods have been used for the fabrication of α -Fe₂O₃, the anodization showed more advantages in easily adjusting the various preparation parameters for controlling the structure, composition, and optimized the photocatalytic activities [3]. In the present work, we show that iron oxide nanostructures can be electrochemically grown with in optimized fluoride-containing ethylene glycol electrolytes. The as-formed layers are amorphous. By annealing the layers can be crystallized. In order to study the effect of voltage and time on the surface morphology of the hematite samples, experiments carried out at different voltage and time.

Keywords: Hematite, Nanostructures, Anodization, Self-organized.

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Enhancing cycle life of lead acid batteries by various methods like PbO₂ nano-particle, multi-walled carbon nano tubes

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Abstract

This paper introduces technologies that considerably enhance cycle life of lead acid batteries. Cycle life of lead acid batteries is improved by adding multi-walled carbon nano tubes (MWCNTs) as an alternate conductive additive in Negative Active Mass (NAM) [1]. MWCNTs added to the negative electrode, exhibits high capacity, excellent cycling performances at 10h rate and high rate partial state of charge cycling and various rates of discharge. It significantly reduces the irreversible lead sulfate on the NAM, increases the active material utilization and improves the electrode performance. The improvement of capacity and cyclic performance of the cell is attributed to the nano scale. The other way is utilization of PbO₂ nano-powder as a positive active material in lead acid batteries [2]. PbO₂ nano-powder was synthesized by the ultrasonic irradiation of an aqueous suspension of dispersed β -PbO, as precursor, in the presence of ammonium peroxy disulfate as an oxidant. The reaction rate increased with an increase in temperature and ammonium peroxy disulfate concentration. In the presence of ammonium peroxy disulfate, the increased concentration of hydroxyl radical facilitated the oxidation of β -PbO to PbO₂ under ultrasonic irradiation. Another way for enhancing cycle life is utilization of 1.0 wt% BaSO₄ and 0.2 wt% Stearic acid (vs. the lead oxide), content in NAM [3]. The optimum BaSO₄ loading level with most beneficial effect on the cycling performance of cells has been determined via the simulated HRPSoc test comprising short (60 s) charge discharge pulses with high currents. Stearic acids are adsorbed onto the lead surface isolating it from contact with atmospheric oxygen. So, they act as a higher potential barrier for the reaction between lead and oxygen. Cycle life performance of lead acid battery improved with use of 0.08M Sodium sulfate in the electrolyte and 0.07 wt% Ca in negative grid alloy. It has been established that, at Ca loading levels higher than the above value yield strength, ultimate tensile strength is reduced. Sodium sulfate and Ca alloys reduced voltage drop and self-discharge processes, therefore enhanced cycle life of lead acid batteries.

Keywords: PbO₂ nano particle, Multi-walled carbon nano tubes, Cycle life, BaSO₄, lead calcium alloys, Lead acid batteries, Stearic acid, Sodium sulfate

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Electrocatalytic oxidation of formaldehyde onto carbon paste electrode modified with synthesized Ni-ZSM-5 nanozeolite

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Abstract

In this research, a novel modified Ni-ZSM-5 nanozeolite was synthesized via an organic template-free hydrothermal route and characterized by X-ray diffraction, scanning electronic microscopy, energy-dispersive X-ray and FTIR techniques. The average particle size of synthesized Ni-ZSM-5 nanozeolite was calculated to be 85 nm by SEM. The development of a novel modified electrode for electrocatalytic oxidation of formaldehyde to decrease overvoltage is of interest [1,2]. The carbon paste electrode (CPE) was modified by Ni-ZSM-5 nanozeolite and Ni²⁺ ions were then incorporated to the nanozeolite by immersion of the modified electrode in a 0.1 M NiCl₂ solution. Electrochemical behavior of this electrode was investigated by cyclic voltammetry that exhibits stable redox behavior of Ni(III)/Ni(II) couple in alkaline medium. The fabricated electrode was used as an anode for the electrocatalytic oxidation of formaldehyde in 0.1 M of NaOH solution. It has been shown that Ni-ZSM-5 nanozeolite at the surface of CPE can improve catalytic efficiency of the dispersed nickel ions toward oxidation of formaldehyde [3]. The values of electron transfer coefficient, charge-transfer rate constant and the electrode surface coverage were obtained to be 0.49, 0.045 s⁻¹ and 4.11×10⁻⁸ mol cm⁻², respectively. Also, the catalytic rate constant for formaldehyde and redox sites of electrode and diffusion coefficient were found to be 9.1×10³ cm³ mol⁻¹ s⁻¹ and 8.58 ×10⁻⁶ cm² s⁻¹, respectively.

Keywords: Ni-ZSM-5 nanozeolite, Template-free synthesis, CPE modified electrode, Formaldehyde Electrocatalysis

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Electrochemical behavior of carbon paste electrode modified with synthesis Ni-SAPO-34 nanozeolite toward electrocatalytic oxidation of methanol

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Abstract

In this paper, a silicoaluminophosphat (SAPO-34) molecular sieve with nano dimension was synthesized and characterized by X-ray diffraction, scanning electronic microscopy, infrared spectroscopy as well as nitrogen adsorption-desorption isotherm. Reducing the particle size of the molecular sieve has a significant impact on the selectivity, activity and stability [1, 2]. Oxidation of methanol leads to large overvoltage at most nonmodified electrode surfaces. The development of a novel modified electrode for electrocatalytic oxidation of methanol to decrease overvoltage is of interest [3]. Carbon paste electrode (CPE) was modified by SAPO-34 molecular sieve and then Ni²⁺ ions incorporated to this electrode by immersion of the modified electrode in a 0.5 M nickel chloride solution. The electrochemical behavior of the modified electrode towards the oxidation of methanol was evaluated by cyclic voltammetry and chronoamperometry methods. It has been observed that SAPO-34 molecular sieve at the surface of CPE can improve catalytic efficiency of the dispersed nickel ions toward oxidation of methanol. It was found that methanol was oxidized by NiOOH generated with further electrochemical oxidation of nickel hydroxide on the surface of this modified electrode during the anodic potential sweep. The effect of some parameters such as scan rate of potential, concentration of methanol, amount of SAPO-34 was investigated on the oxidation of methanol at the surface of modified electrode. The values of electron transfer coefficient and charge-transfer rate constant were found to be 0.75 and 1.62 s⁻¹, respectively. Obtained results from cyclic voltammetry and chronoamperometric techniques indicated that the electrode reaction is a diffusion-controlled process.

Keywords: Nanomaterial, SAPO-34 Molecular sieve, Modified CPE, Methanol, Electrocatalytic oxidation

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Non-enzymatic amperometric glucose sensor based on copper oxide nanoparticles/ nanocellulose/graphene oxide composite paste electrode

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Abstract

Imbalance of blood sugar is a global health care problem that treatable disease affecting about 200 million people around the world [1]. Although there are many different methods for glucose determination, but electrochemical methods have absorption more concentration due to their simplicity, proper selectivity and high sensitivity [2]. In this study, sensitive and stable non-enzymatic glucose sensor based on GO/NC/CuO composite paste electrode was fabricated. Themorphology of nanomaterial and nanocomposit were investigated by scanning electron microscopy(SEM),transmission electron microscopy(TEM)and X-ray diffraction(XRD).Cyclic voltammetry and amperometry were performed to study the electrocatalytic oxidation of glucose in alkaline medium at GO/NC/CuO-CPE. A liner rang of 0.05 to 30mM glucose, limit of detection 2.5 μ M (signal-to-noise 3) were observed.The modified electrodes offer excellent electrocatalytic activity toward the glucose oxidation at low positive potential (0.4 V vs Ag/AgCl). As a result, it could be concluded that GO/NC/CuO-CPE at presence of NC would pave the way for glucose biosensor.The novel glucose sensor also exhibits good reproducibility, long-term stability, as well as no interference from L-ascorbic acid, uric acid, fructose and galactus, so it is reasonable to expect its broad use in non-enzymatic glucose sensor.

Keywords: Non-enzymatic, Nanocellulose, Graphen oxide, CuO nanoparticles, Composite paste electrode

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Synthesis of Iron doped manganese dioxide by means of an electrochemical/chemical method

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Abstract

Since the introduction of Laclanche batteries in the late 1900's, manganese dioxide has attracted intense attention because of its unique properties such as high availability, low toxicity and cost. Manganese dioxide is still a serious rival for other electrode materials, even after the replacement of Laclanche batteries by newer ones[1]. Manganese dioxide has an enormous theoretic storage ability of 1450F/g but the actual storage ability is a complex function of structural parameters. A great number of research works have been conducted to investigate the effect of structural parameters such as crystallinity, particle size, crystalline lattice defect and degree of stoichiometric deviation on electrochemical behavior and storage ability of manganese dioxide [2]. A promising way to improve energy exchange capabilities of manganese dioxide is doping with different substances such as Fe, Ni, Bi, Al, Cr and Mo [3]. Fe is a good candidate when all parameters, including cost effectiveness and environmental impact, are considered together. In this work a versatile method for the Fe doping of MnO₂ via cathodic base generation from a mixed Fe/Mn solution and heat treatment of the resulting mixed oxide has been described. The synthesis procedure comprises galvanostatic generation of hydroxide ion from nitrate baths containing Mn²⁺ (0.01M) and various amounts of Fe³⁺ (Fe/Mn ranging from 0 to 7.5%) to produce a precursor from which the final doped product is prepared by annealing (300°C for 2h). The effect of experimental parameters such as Fe/Mn ratio and current density on morphology, composition, crystallinity and energy storage ability of doped samples have been investigated by means of Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectrometry (EDAX), X-Ray Diffraction (XRD) and Cyclic Voltammetry (CV). Results showed that the amount of dopant has a significant effect on morphology, crystallinity and storage ability of the obtained nanostructures. For example, it was shown that nanorod or multi-walled nano array formation is favored where the amount of dopant is very low. On the other hand, the incorporation of Fe resulted in formation of nanospheres (d<40nm). A mechanism has been proposed for the observed modifications according to the alteration of lattice parameters as well as the anisotropy of manganese dioxide nanocrystals. The calculation of specific capacitance revealed that Fe doping could improve the storage ability of manganese dioxide up to 8.5%.

Keywords: Nanostructure, Fe doped manganese dioxide, Supercapacitor, Electrosynthesis

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Synthesis of manganese dioxide nanosheets and charge storage evaluation

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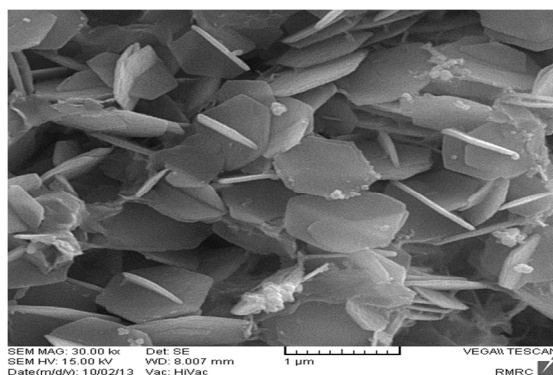
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Abstract

The electrochemical method is a facile and high efficient route for the synthesis of various nanostructures, such as nanorods, nanowires, nanotubes, nanosheets, and composite nanostructures [1]. A great variety of nanostructures can be easily fabricated by cathodic base generation with advantages of low cost, low synthetic temperature, high purity, simplicity, and environmental friendliness [2]. Nanostructured MnO₂ has attracted a lot of interest due to its unique physical and chemical properties. This is because many properties of materials, such as catalytic activity, sensitivity or conductivity, are closely related to surface area and particle size. Manganese dioxide nanostructures hold great potential for supercapacitors and other energy storage systems due to their high surface area and high contact with the electrolyte [3]. In this work, Manganese dioxide nanostructures were synthesized by a chemical-electrochemical process using controlled-current cathodic base generation from nitrate solutions followed by heat-treatment of manganese hydroxide precursor. A variety of characterization methods have been applied to the samples including Fourier Transform Infra-Red spectrometry (FTIR), Energy Dispersive X-Ray Analysis (EDX), X-Ray diffraction (XRD), Scanning Electron Microscopy (SEM). The SEM images show that the morphology of product is greatly affected by experimental parameters especially the current density. Nanosized disc-like hexagonal structures with diameter of approximately 800nm and thickness of 50 nm were prepared at a current density of 0.35 mAcm⁻². The electrochemical properties of MnO₂ nanostructures were investigated by cyclic voltammetry (CV). The maximum specific capacitance was 204 Fg⁻¹ at a scan rate of 2mVs⁻¹.



Keywords: Nanostructure, Nanosheet, Supercapacitor, Electrosynthesis

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First report for simultaneous voltammetric determination of norepinephrine in the presence of tyrosine and nicotine

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Abstract

Norepinephrine (NE) is one of the most important biochemical messengers in mammalian central nervous systems, existing in the nervous tissue and biological body fluid. Many diseases and the caducity process are related to changes of its concentration, thus the quantitative determination of trace NE in biological fluids provides important information on its physiological functions and the diagnosis of some diseases in clinical medicine [1]. Nicotine produces numerous pharmacological effects, many of which are due to its interactions with catecholamine systems. In the periphery, nicotine elicits catecholamine release from postganglionic sympathetic neurons and adrenal medulla. Nicotine also acts centrally to stimulate dopamine release from nigrostriatal and mesocorticolimbic neurons and norepinephrine release from locus coeruleus neurons [2]. L-Tyrosine is the precursor of the catecholamines; alterations in the availability of L-tyrosine to the brain can influence the synthesis of both dopamine and norepinephrine in experimental animals and probably in humans [3]. In this study, a carbon paste electrode modified with ZnO nanoparticles and a new mediator was used to prepare a novel electrochemical sensor for the detection of norepinephrine in the presence of tyrosine and nicotine. Also, this sensor was used for determination of three compounds in real samples.

Keywords: Norepinephrine, Tyrosine, Nicotine, ZnO nanoparticles

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Voltammetric determination of levodopa and carbidopa using a ZnO nanoparticles modified carbon paste electrode

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Abstract

Levodopa [(S)-2-amino-3-(3,4-dihydroxyphenyl)propanoic acid, LVD] is a precursor of the neurotransmitter dopamine, widely used in the clinical treatment of Parkinson's disease. It could be converted to dopamine by DOPA decarboxylase and capable of crossing the protective blood-brain barrier. After administration, LVD is converted into dopamine through enzymatic reaction catalyzed by dopadecarboxylase. Some side effects of systemic DA can appear if LVD is taken at high dosages because of the metabolism of LVD being extracerebral[1]. Administration of levodopa in combination with carbidopa[(-)-1-2-(3,4-dihydroxybenzyl)-2-hydrazinopropionic acid] an inhibitor of the decarboxylase enzyme, which does not cross the blood-brain barrier, helps to control dopamine levels in appropriate manner and reduces side effects. In order to achieve better curative effect and lower toxicity, it is very important to control the content of levodopa and carbidopa in pharmaceutical tablets [2]. Electrochemical methods have also been used and attracted enormous interest due to its advantages of simplicity, rapid response, excellent reproducibility, good stability, low cost and low detection limit, etc. [3]. In this study, a carbon paste electrode modified with ZnO nanoparticles and Ak-3, α Zim was used to prepare a novel electrochemical sensor for the detection of levodopa in the presence of carbidopa. The present method was applied to determination of levodopa and carbidopa in real samples.

Keyword: Levodopa, Carbidopa, ZnO nanoparticles

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Photochemical degradation of rhodamine B in aqueous solution using nanophotocatalyst ZnO doped by N-,S-, and C

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Abstract.

Photocatalytic degradation of Rhodamine B (RB) dye was studied using ZnO/C,N,S catalysts synthesized by sedimentation technique in the laboratory. Zinc sulfate and thiourea was used as the starting material. The photocatalytic degradation of dye has been investigated using supported ZnO/C,N,S photocatalyst by monitoring the change in substance concentration employing UV spectroscopic analysis technique as a function of irradiation time. The structures, morphology, and optical properties of these ZnO/C,N,S nanoparticles fabricated by above-mentioned methods, were also characterized by X-ray diffraction (XRD), Energy dispersion X-ray(EDX), X-ray photoelectron spectroscopy (XPS), Field emission scanning electron microscopy (FE-SEM). The effect of various parameters such as initial dye concentration, amount of nano photocatalyst, PH of the solution, addition of different oxidant such as H₂O₂, K₂S₂O₈, KBrO₃, KIO₃ and inorganic ions, irradiation time, on photo degradation of rhodamine B were investigated. In the present work, based on the results, most degradation of rhodamine B has been observed at concentration of 20 ppm, amount of photo-catalyst of 7 mg, amount of oxidant K₂S₂O₈ 3mM and PH=9. Analysis of the kinetic showed that the amount of rhodamine B photocatalytic degradation can be fitted with pseudo- first- order model.

Keywords: Photocatalytic degradation, Nanophotocatalyst ZnO doped by N-,S-, and C, Rhodamine B

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Structural and magnetic properties of magnetite nanoparticles prepared in the presence of β -cyclodextrine by pulsed sono-electrochemical method

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Abstract

In the last decade, magnetic nanoparticles have attracted great attention because of their special properties which differ significantly from those of the bulk materials. Iron oxide nanoparticles are particularly useful and being successfully employed in many industrial, medical and biological applications. To date, a wide range of different methods has been used to synthesize iron oxide nanoparticles. Electro-crystallization is a less conventional electrochemical synthesis for preparing magnetite nanoparticles. In this method, by applying an appropriate potential difference between two iron plates in an aqueous electrolyte solution containing an stabilizer agent, iron at the anode is oxidized to Fe^{2+} and Fe^{3+} , while water is reduced to hydrogen and hydroxyl anions at the cathode. In the solution, iron ions and OH^- meet to produce an orange-brown iron hydroxide which dehydrates to form magnetite, Fe_3O_4 [1-3]. In this work, we are aiming to analyze the impact of pulsed ultrasound waves on structure and magnetic properties of magnetite nanoparticles produced in the presence of β -cyclodextrine by electrocrystallization method. The crystal structure of the products was analyzed using X-ray diffraction (XRD). A scanning electron microscope (SEM) was employed to examine the morphology, particle size, and dispersion of the Fe_3O_4 nanoparticles. Magnetic measurements were conducted using a vibrating-sample magnetometer (VSM) with a 1.1 T permanent magnet flux source. Mössbauer spectra in transmission geometry were recorded for all samples using a source of Co^{57} in Rh, at room temperature. The obtained results showed that ultrasound has a significant effect on the structure and magnetic properties of magnetite nanoparticles produced by electrocrystallization method. Based on structural characterization, it is possible to improve the structure of samples by ultrasound treatment, depending on the number of applied ultrasound pulses. Also, the particle size and morphology were found to be easily controlled by adjusting the number of applied ultrasound pulses. Based on the electron microscopy and Mössbauer spectroscopy we found that the iron is almost all in the form of 20–70 nm particles of slightly nonstoichiometric $\text{Fe}_{3-\delta}\text{O}_4$, with $\delta \approx 0.1$. Specific magnetization of the magnetite nanoparticles ranges from $18 \text{ Am}^2\text{kg}^{-1}$ to $71 \text{ Am}^2\text{kg}^{-1}$ at room temperature, depending on the particle size and ultrasonic pulses, but most of this variation is due to the mass of attached nonmagnetic organic material. Mössbauer spectra showed that ultrasonic treatment is very effective to remove a strong paramagnetic doublet, associated with organometallic complexes containing Fe^{3+} which were present in the sample prepared without ultrasound. In fact, the formation of a paramagnetic secondary phase is suppressed by ultrasound treatment and the purity of magnetite phase would be improved.

Keywords: Magnetite nanoparticles, Structural properties, Magnetization, Electro-crystallization, Pulsed Sonoelectrochemistry

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Synthesis and characterization of structural and magnetic properties of electro-crystallized magnetite nanoparticles under constant current

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Abstract

It is well known that magnetic nanostructures have had a major impact on the development of modern technologies. Magnetic nanoparticles because of their special properties have found many new industrial, biological and medical applications in areas such as imaging, catalysis and semiconductors, pharmaceutical and cosmetic products, biosensors, cell separation and detection, tissue engineering and regenerative medicine. Magnetite nanoparticles reported as applicable materials owing to their magnetic properties and being successfully employed in magnetic resonance imaging, controlled drug delivery and hyperthermia-based therapy, ferrofluids and removal of heavy metals from waste water. In the last decade, a wide range of various physical and chemical methods have been employed to synthesize iron oxide nanoparticles. Among the less conventional methods for synthesis of Fe₃O₄ nanoparticles electro-crystallization has begun to emerge as one of the more recent methods. With this method, it is possible to produce fine magnetite nanoparticles electrolytically using iron electrodes. With this method, the mean particle size, particle size distribution and morphology can be controlled by adjusting the growth conditions such as temperature and applied potential or current. Also it is possible to avoid the agglomeration of particles by adding a stabilizer agent into the electrolyte solution [1-3]. In this study, the electro-crystallized magnetite nanoparticles were fabricated under constant current in the presence of three different stabilizer agents and then their structural and magnetic properties were characterized. We are especially interested to analyze the relationship between specific magnetization and particle size which is dependent on applying electro-oxidation current. The synthesis was performed in an electrochemical cell containing two iron electrodes and an aqueous solution of sodium sulfate, and either thiourea, sodium butanoate, or β -cyclodextrine as organic stabilizer. All the samples were characterized by XRD, SEM, VSM, and Mössbauer spectrometry. X-ray diffraction patterns, clearly confirmed that all products have the cubic spinel Fe₃O₄ crystal structure. Electron microscope images showed that the mean particle size is in the range 20–80 nm, and depends critically on the applied current and type of the organic additive. Specific magnetization of the samples at room temperature ranges from 60 to 90 Am² kg⁻¹, again depending on the growth conditions. The observed reduction in the magnetization is partly a dilution effect caused by the mass of the associated organic material. Room temperature Mössbauer spectra, which were recorded for all samples using a Co⁵⁷ source, are typical of nonstoichiometric Fe_{3- δ} O₄, with a small excess of Fe³⁺, $0.06 \leq \delta \leq 0.17$.

Keywords: Current, Electro-crystallization, Magnetite nanoparticles, Magnetization, Nonstoichiometric structure

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Study of electrochemical behavior of SWNT- DNA Hybrid self-assembled Nano structure on graphite electrode

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Abstract

In this study, a new effective and conducive hybrid Nano structure SWNT Nano particle and DNA bioconjugate was designed. SWNT-DNA hybrid Nano structure fabricates by region selective covalent combined between DNA and SWNT Nano particles. Amin functional group end of the DNA single strand combined covalently to carboxylic functional group on SWNT. With use self-complementary DNA sequence in combination proses we can fabricate self-assembled DNA Nano structure. This hybrid Nano structure has different electrochemical properties compare of alone SWNT and DNA single strand. Electrochemical behavior of SWNT-DNA hybrid Nano structure is studied by cyclic voltammetry and impedance spectroscopy technique. Also conformation change in DNA structure is studied by circular dichroism technique. Two single-stranded 10 mer oligonucleotide (ssDNA), probe 1 and prob1', was immobilized via an amine linker on side wall of oxidized and shortened SWNT. Impedimetrically and cyclovoltametric analysis of this structure in the presence of the redox system ferri/ferro-cyanide before and after DNA covalent binding to SWNT were studied. Impedance and CV spectra of this structure showed that the resistance was increasing after DNA combination. In addition of 22 mer oligonucleotide as linker DNA to mix of SWNT-DNA hybrid Nano structure, wire shape SWNT-DNA hybrid Nano structure is fabricated. This hybrid Nano structure has favorable electrochemical properties. Electrochemical properties of wire shape SWNT-DNA hybrid Nano structure by stabilized on graphite electrode were studied.

Keywords: Electrochemical behavior, Hybrid nanostructure, Region-selective combination, Functional DNA, SWNT

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A new carbon paste modified electrode based on graphene quantum dots and chitosan for electrochemical investigation of epinephrine

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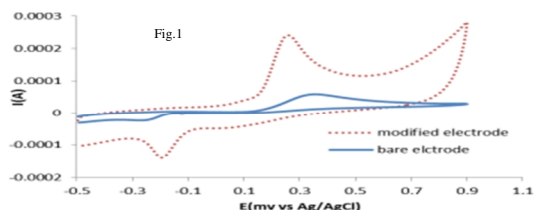
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Abstract

Graphene quantum dots (GQDs), which are emerging luminescent carbon-based nanomaterials, have lately aroused increasing interest in their optical and electronic properties. GQDs are graphene sheets with lateral size smaller than 100 nm in single, double and multiple layers and diameters spanning the range 3–20 nm mainly. Graphene quantum dots (GQDs) are a kind of 0D material with characteristics derived from both graphene and carbon dots (CDs)[1]. These properties confer GQDs a variety of potential uses in photovoltaic devices, bioimaging instruments, sensors and biosensors, among others. The excellent conductivity of the graphene-based materials gives a fine electrochemical response that can be exploited for sensing [2]. Epinephrine (EP), a hormone secreted by the medulla of adrenal glands, is an important catecholamine neurotransmitter in the mammalian hormonal and central nervous system. Presence of EP in the body affects the regulation of blood pressure and the heart rate, lipolysis, immune system, and glycogen metabolism. Therefore, the quantitative determination of epinephrine concentration in different human fluids is important for developing nerve physiology, pharmacological research and life science [3]. In this work, a new carbon paste modified electrode based on GQDs and chitosan is reported. An easy bottom-up method for the preparation of graphene quantum dots (GQDs) has been developed by carbonization and dispersing the carbonized products into alkaline solutions. Different amounts of GQDs and chitosan solution were mixed and used for preparing modified carbon paste electrode and percent of modifiers optimized. In order to investigate of this electrode, electrochemical behavior of epinephrine were studied and in optimum conditions (pH=7 phosphate buffer, percent of modifiers,...), the resulting electrode showed noticeable increasing in electrochemical response of epinephrine. Cyclic voltammogram of epinephrine (0.0003M, pH=7) at bare and modified electrode has been showed in Fig.1. Furthermore, the capabilities of electron transfer on this electrode were also investigated by electrochemical impedance spectroscopy.



Keywords: Graphene quantum dots, Epinephrine, Modified electrode

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Numerical Investigation of Gate-All-Around Nano Wire FETs' Performance in Digital Applications Based on Materials and characteristics

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Abstract

In this paper, SiC and Si nano wire field effect transistors with SiO₂ and HfO₂ gate oxide materials are simulated in various gate oxide thicknesses and channel diameters. In order to study the performance of these transistors in digital applications, the impact of channel material and diameter and also oxide material and thicknesses changes on the important switching parameters like Ion/Ioff, SS, PDP, and τ_d are investigated. Results show that calculated parameters are sensitive to these changes. But, the dependence of the parameters to the type of oxide material is higher in which by changing the oxide material to HfO₂ τ_d decreases considerably. In addition, the optimum feature for the best switching speed is obtained and simulated. This study shows that SiC nano wires FETs are comparable with Si nano wire FETs in digital applications. In this paper, performances of NWFETs in digital applications will be investigated. In this regard, a ballistic model based simulation is applied on NWFETs with two different channel materials including Si and SiC, with different diameters. Then the impact of substituting HfO₂ for SiO₂, and variation of oxide thickness on some important parameters like SS, PDP, and τ_d will be investigated and compared. It should be mentioned that tight binding method is used to calculate effective mass and energy band gap because the energy band diagram of nano wire is dependent to its diameter. The rest of paper includes 4 sections. Section 2 defines device structure and simulation method. Results and analysis are presented in section 3 and finally, conclusions are drawn in section 4. The channel semiconductor material supposed to be Si or SiC and the Gate oxide material is supposed to be SiO₂ or HfO₂. The channel length and V_{DS} are supposed to be 10 nm and 0.4 v, respectively. In this study, the channel diameter (d) supposed to be 0.61, 0.92, 1.23 nm, and the gate oxide thickness (t_{ox}) is supposed to be 0.7 and 1.5 nm for each device.

Keywords: Nano wire field effect transistor, Si nano wire, SiC nano wire, Switching speed.

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Electrochemical determination of hydrazine by glassy carbon electrode modified with nitrogen-doped graphene uniformly decorated with platinum nanoparticles (Pt/N-Gr)

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Abstract

Hydrazine is a reducing agent which has a wide range of applications in many industrial processes. It has also been shown that hydrazine has neurotoxic, carcinogenic, and mutagenic effects. Electrochemical techniques offer the opportunity for portable, cheap, and rapid methodologies for detection of hydrazine. However, electrochemical oxidation of hydrazine is kinetically sluggish and a relatively high overpotential is required for its oxidation at the bare electrode. Therefore, searching for new methodologies to modify the electrode surface in order to reduce the overpotential of the hydrazine oxidation is required [1]. Due to exceptional electronic properties of graphene (G) and nitrogen doped graphene (N-Gr), they are considered as superior supporting platforms for novel metal nanoparticle decorations. Here, we report, a novel one-step electrochemical method for synthesis of nitrogen-doped graphene sheets uniformly decorated with platinum nanoparticles (Pt/N-Gr). For the first time Pt/N-Gr was used for electrode modification and successfully applied for electrooxidation of hydrazine at a reduced potential. The glassy carbon electrode modified with Pt/N-Gr shows excellent electrocatalytic activity toward hydrazine oxidation at reduced overvoltage (-0.4 V versus Ag/AgCl reference electrode in phosphate buffer solution with pH 9). Cyclic voltammetry and hydrodynamic amperometry were used as measuring techniques for hydrazine detection. The detection limit was 50 nM and concentration range was more than 50 μ M.

Keywords: Cyclic voltammetry, N-doped Graphene, Hydrazine, Platinum nanoparticles

Nitrogen doped carbon nanostructures functionalized with non-precious metals, electrocatalytic application in oxygen reduction reaction

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Abstract

Fuel cells are the electrochemical devices that transfer the chemical energy to electrical one. Oxygen Reduction Reaction (ORR) which is performed in cathode electrode plays an important role for industrialization of Fuel cells. Precious metals such as Pt and Pd presented best electrocatalytic activity and the minimum reported over-voltage for ORR but their applications suffer from some limitations. Therefore, it is urgently required to explore inexpensive, non-Pt catalysts for the replacement of Pt-based catalysts. One of the possible solutions for this problem is using non precious metals. Ordered Mesoporous Carbon materials (OMC) are highly attractive for applications in energy conversion devices. Moreover, it has been proved that incorporation of nitrogen heteroatom into carbon framework enhance their electrochemical and catalytic properties [1]. A novel nano-fibrillated mesoporous carbon (IFMC) was successfully prepared via carbonization of the ionic liquid 1-methyl-3-phenethyl-1Himidazolium hydrogen sulfate (MPIHS) in the presence of SBA-15. The material was shown to be an efficient support for the palladium nanoparticle (PdNP) catalyst Pd@IFMC in aerobic oxidation of heterocyclic, benzylic, aliphatic and allylic alcohols and heteroatom containing alcohols on pure water [2]. Continuing our previous studies on catalytic and electrochemical application of nitrogen doped mesoporous carbons, we were encouraged to introduce some non-precious metals, such as Fe, for obtaining nano-structured carbon with "nitrogen-metal sites" as efficient electrocatalyst for ORR. Ordered mesoporous carbon was derived from carbonization of ionic liquid (MPIHS) and guanine as a nitrogen rich organic molecule, as carbon precursor, and ordered mesoporous silica (SBA-15) as template [3]. The structure of obtained OMCs were characterized by transmission electron microscopy (TEM), N₂ adsorption-desorption analysis and thermal gravimetric analysis (TGA). The nitrogen doped OMCs then functionalized by post-treatment with nitrogen containing iron complexes. The resulting porous carbon materials have been used as an electrode modifier and their electrochemical performances have been examined toward ORR. Hydrodynamic and cyclic voltammetry studies of the metal-nitrogen containing carbons has been performed at various pH values and the desired structure have shown very good performance and low over-potential at both acidic (0.1 M HClO₄) and basic (0.1 M NaOH) solutions. The durability of modified electrode based on these structures has been also examined by chronoamperometric studies and the results confirmed very good stability for the obtained modified electrodes.

Keywords: Ordered mesoporous carbon, Metal nanoparticle, Oxygen Reduction Reaction

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Potentiometric determination of trace As(III) in aqueous media with a stainless steel electrode modified by polyaniline/MWCNT

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Abstract

The development of practical sensors for the detection and quantification of metal ions in environmental samples is the subject of considerable research [1]. Arsenic is one of the poisonous substances and widely distributed elements in the earth crust and in the ground water that can exist in many chemical forms such as arsenite and arsenate. The present work is motivated by the need to develop a routine measurement of inorganic arsenic at the trace levels in both the lab and real samples [2]. In this study, we proposed a potentiometric sensor for monitoring and determination of arsenic species in the aqueous media. Novel polyaniline/multiwalled carbon nanotube (PANI/MWCNT) film was fabricated on stainless steel by using cyclic voltammetry (CV) technique. The amount of film components (aniline-MWCNTs-As(III)) was investigated to optimize the electrode materials with better electroanalytical performance. The surface morphological study has been analyzed using scanning electron microscopy (SEM). Calibration plots of potential versus concentration of As(III) were constructed and showed the successful evaluation of arsenic in the linear range of 1 mM to 1 nM. The obtained reproducible limit of detection for this technique was 0.3 nM. The proposed method presents a simple, rapid and reliable detection of the arsenic in drinking water sources [3].

Keywords: As(III), Potentiometry, Polyaniline, Multiwalled carbon nanotube

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Comparison between Fenton and Electro-Fenton regeneration of carbon nanotubes exhausted with natural organic matters

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Abstract

Among the adsorbents, carbon nanotubes (CNTs) are relatively new nano materials which are frequently used for water purification, the spent CNTs should be regenerated for wider application of CNTs adsorption processes. In the present study, the efficiency of Fenton and Electro-Fenton mechanisms for regeneration of CNTs exhausted with natural organic matters (NOMs) were studied. CNTs were produced using chemical vapor deposition (CVD) method. The molar ratios of Fe^{2+} to H_2O_2 for Fenton solution were 0.5:10, 1:10, and 2:10 and for Electro-Fenton methods were 0.03:10, 0.005:10, 0.003:10 and 0.002:10. The saturation cycles were continued up to five stages. Results of Fenton regeneration showed higher regeneration efficiency (65.4%) for 2:10 M ratio comparing to other molar ratios after five cycles of regeneration. The regeneration efficiency after five cycle of regeneration with Electro-Fenton mechanism in optimum $\text{Fe}^{2+}:\text{H}_2\text{O}_2$ molar ratio (0.03:10) decreased to 87%. The results of the present study indicated that Electro-Fenton process has a higher efficiency for the regeneration of CNTs exhausted with NOMs even in very low $\text{Fe}^{2+}:\text{H}_2\text{O}_2$ molar ratios.

Keywords: Fenton, Electro-Fenton, Regeneration, Carbon nanotubes

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Direct growth of highly catalytic platinum 3D nanoflowers onto glassy carbon substrate by a template-free electrochemical route

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Abstract

Among various nano-architectures, very recently nanoflowers have emerged as compelling materials because such structured nanomaterials possess a porous nature, rough surface, providing favorable surface areas and active centers for electro-catalysis [1]. However, the reported synthesis of the flower-like nanomaterials involved complicated preparation processes and costly materials [2,3]. Based on these considerations, in this study, a facile electrochemical approach is developed towards fabricating Pt needle-like nanoflowers (NFs) at the surface of glassy carbon electrode. The morphology and composition of the Pt NFs are characterized by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS), respectively. Taking methanol oxidation as a model reaction in an acid medium, the electrocatalytic performance of as-prepared Pt NFs has been evaluated by cyclic voltammetry (CV), chronoamperometry and electrochemical impedance spectroscopy (EIS) techniques. These three dimensional (3D) NFs exhibit the excellent electrocatalytic activity (2.8 mA cm^{-2}) and high level of poisoning tolerance (I_f/I_b 1.22) to the carbonaceous oxidative intermediates for the electro-oxidation reaction in acidic media. A comparison with Pt nanoparticles (NPs), prepared by electrodeposition in the presence of hydrogen bubbles, shows that the NFs catalyst has higher activity, better long-term stability and lower Pt loading. The simplicity of method and quality of prepared surfaces suggest applications in catalysis where a convenient method to prepare Pt catalysts with high surface area in one step is desirable.

Keywords: Pt nanoflowers, Electrodeposition, Catalysis, Electro-oxidation, Methanol

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A sensitive simultaneous determination of dopamine, acetaminophen and indomethacin on a glassy carbon electrode coated with a new composite of MCM-41/ nickel hydroxide nanoparticles/ multiwalled carbon nanotubes

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Abstract

A novel chemically modified electrode is constructed based on a multiwalled carbon nanotubes (MWCNT), nickel(II) hydroxide nanoparticles (NHNPs) and MCM-41 composite modified glassy carbon electrode. It is demonstrated that the sensor can be used for the simultaneous determination of dopamine (DA), acetaminophen (ACT) and indomethacin (DA). Recent studies have shown that the simultaneous consumption of INDO and AC increases the probability of kidney malfunction in individuals. Similarly the consumption of INDO and DA at the same time cause hypertension which can escalate kidney malfunction (1). The measurements were carried out by the application of differential pulse voltammetry (DPV), cyclic voltammetry (CV) and chronoamperometry (CA) methods. Under the optimum conditions the modified electrode provides a linear response versus DA concentrations in the range of 1.5-45 μM and 70-350 μM , ACT concentrations in the range of 0.2-20 μM and 100-400 μM and INDO concentrations in the range of 0.8-40 μM and 60-160 μM , respectively using the differential pulse voltammetry method. The modified electrode was used for determination of DA, ACT and INDO in real samples with satisfactory results.

Keywords: Dopamine, Acetaminophen, Indomethacin, Multiwalled carbon nanotubes, Nickel(II) hydroxide nanoparticles, MCM-41.

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Electrochemical noise analysis to investigate the inhibition effect of N-salicylidene-aniline Schiff base against AZ91D alloy corrosion in 0.01 M HCl solution

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Abstract

Lightweight materials are required for automotive and aerospace industries. Magnesium alloys are lightweight materials with high strength to weight ratio and then have many potential applications in the mentioned area. However, magnesium and its alloys intrinsically have high chemical reactivity which makes them susceptible to corrosion in various corrosive environments and this limitation restricts their application. Among the different corrosion protection methods, the use of corrosion inhibitors is one of the most practical and convenient ways especially in the corrosive solutions.

In this work, the corrosion inhibition of AZ91D magnesium alloys by N-salicylidene-aniline Schiff base compound has been studied by electrochemical noise (EN) method as a powerful and non-destructive corrosion monitoring method. The studied Schiff base compound was added to the 0.01 HCl solution at different concentrations and the electrochemical noise measurements were carried out by using a electrochemical cell configuration consist of two identical working electrodes (AZ91D alloy samples with surface area of 1cm²) and saturated Ag-AgCl electrode as reference electrode. All EN experiments were carried out at corrosion potential without any external perturbation. Each set of EN records, containing 1024 data points, was recorded by a data-sampling interval of 0.25 s. The EN data were analyzed in both the time and frequency domains in order to calculate noise resistance (R_n) and DC limit of the spectral noise impedance (R_{sn}^0) respectively. The results showed that the studied Schiff base compound effectively inhibits the corrosion process of AZ91D alloy in 0.01 M HCl solution and the inhibition efficiency increases as the inhibitor concentration increases. Electrochemical Impedance Spectroscopy (EIS) tests were carried to approve the EN results. The results obtained with two various electrochemical methods were in good trend agreement. Moreover, the Scanning Electron Microcopy (SEM), X-ray diffraction (XRD) and Energy Dispersive X-ray Spectroscopy (EDS) analysis were performed to study the alloy surface after immersion in blank and inhibited solution. All results confirmed the corrosion inhibition effect of the N-salicylidene-aniline Schiff base.

Keywords: Magnesium alloy, Corrosion, Inhibition, Schiff base, Electrochemical noise

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