

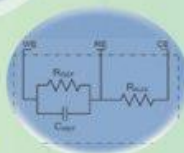
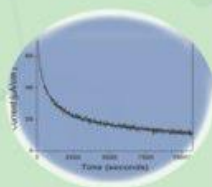
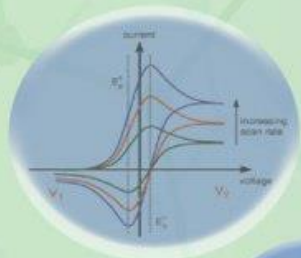


سیزدهمین سمینار سالانه الکتروشیمی ایران

13th Annual Electrochemistry Seminar of Iran



Materials and Energy
Research Center



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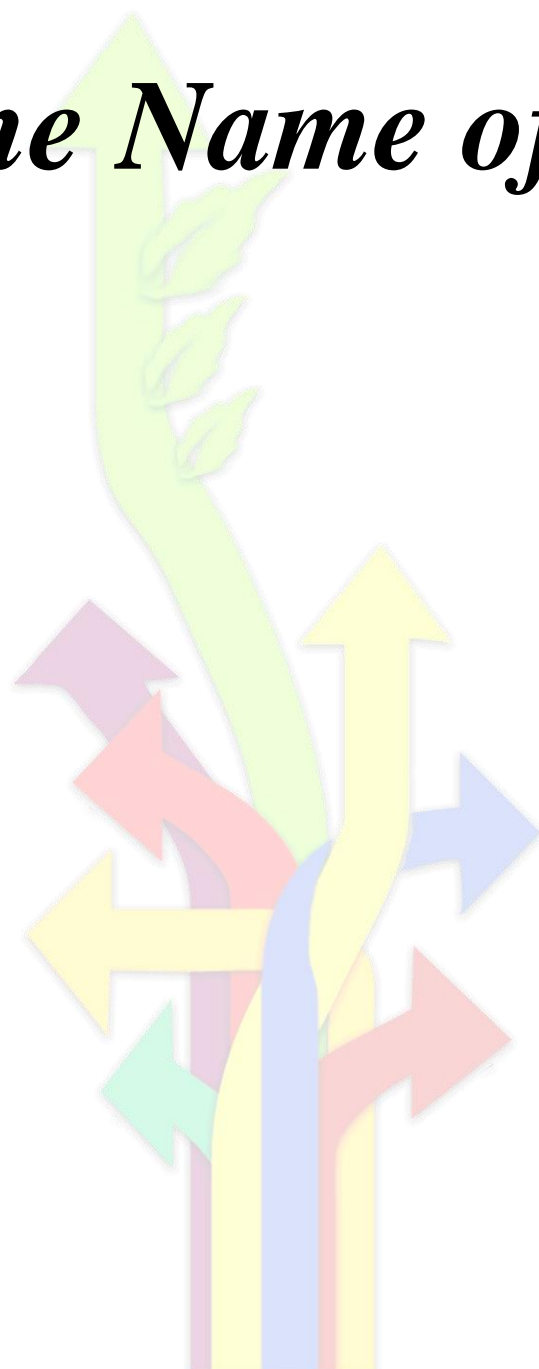


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In The Name of God



13th Annual Electrochemistry Seminar of Iran

Proceeding of
13th Annual Electrochemistry Seminar of Iran
Materials and Energy Research Center (MERC)

22- 23 Nov, 2017

Organized by
Materials and Energy Research Center, Alborz, Iran
Electrochemistry Society of Iran

13th Annual Electrochemistry Seminar of Iran

13th Annual Electrochemistry Seminar of Iran

Held in Materials and Energy Research Center (MERC), Alborz, Iran, 22- 23 Nov, 2017

Seminar Chairman: Prof. Hossein Gharibi

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Materials and Energy Research Center (MERC), Alborz, Islamic Republic of Iran

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13th Annual Electrochemistry Seminar of Iran



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مرکز منطقه‌ای اطلاع رسانی علوم و فناوری

13th Annual Electrochemistry Seminar of Iran

Welcome to the 13th Annual Electrochemistry Seminar of Iran

Dear attendants

On behalf of organizing committee of the 13th Annual Electrochemistry Seminar of Iran, I'm delighted to welcome you all to Materials and energy research center of Iran. Over decades, Iranian researchers have concerned the electrochemical phenomena from both theoretical and applied standpoints mainly the electrochemical aspects of biological, energy storage systems, and corrosion processes. Characterization and analytical considerations have been the most important concerns of researches in this field.

We are pleased to host researchers of electrochemistry field held at Materials and energy research center, Alborz, Iran. It's noteworthy that the seminar includes 10 major fields including Analytical electrochemistry, Physical Electrochemistry (theoretical), Electrochemical Synthesis, corrosion, Bio-Electrochemistry, Nano-Electrochemistry, Computational Electrochemistry, Energy (Conversion & Storage), Electrochemistry – Industry, and Environmental Electrochemistry. We whole heartedly hope that your participation in the 13th Annual Electrochemistry Seminar of Iran in Alborz will be fruitful.

Cordially

Hussein Gharibi

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13th Annual electrochemistry seminar

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Keynote

Name	Title
Dr. Vahid Hosseini	Applications of Chemical Engineering in the Knowledge of Tehran Air Pollution
Prof. Mehran Javanbakht	Lithium- Ion Battery Development & Commercialization
Dr. Jaber Neshati	Application of Electrochemical Techniques in Corrosion science
Ali Shiraojen	Technology, Know-how, Transfer of Technology, Commercialization and Industrialization of the Battery Industry in Iran
Dr. Hasan Karimi Maleh	Electrochemical Sensors and Biosensors for Food Analysis
Dr. Nima Naderi	Photo–electrochemical Etching of Semiconductors



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Research of Area

Electrochemistry

1-Analytical electrochemistry

2-Bio-Electrochemistry

3-Enviromental Electrochemistry

4-Energy (Conversion and Storage)

5-Computational Electrochemistry

6-Nano-Electrochemistry

7-Corrosion

8-physical Electrochemistry

9-Electrochemical Synthesis

10-Electrochemistry-Industry

13th Annual Electrochemistry Seminar of Iran

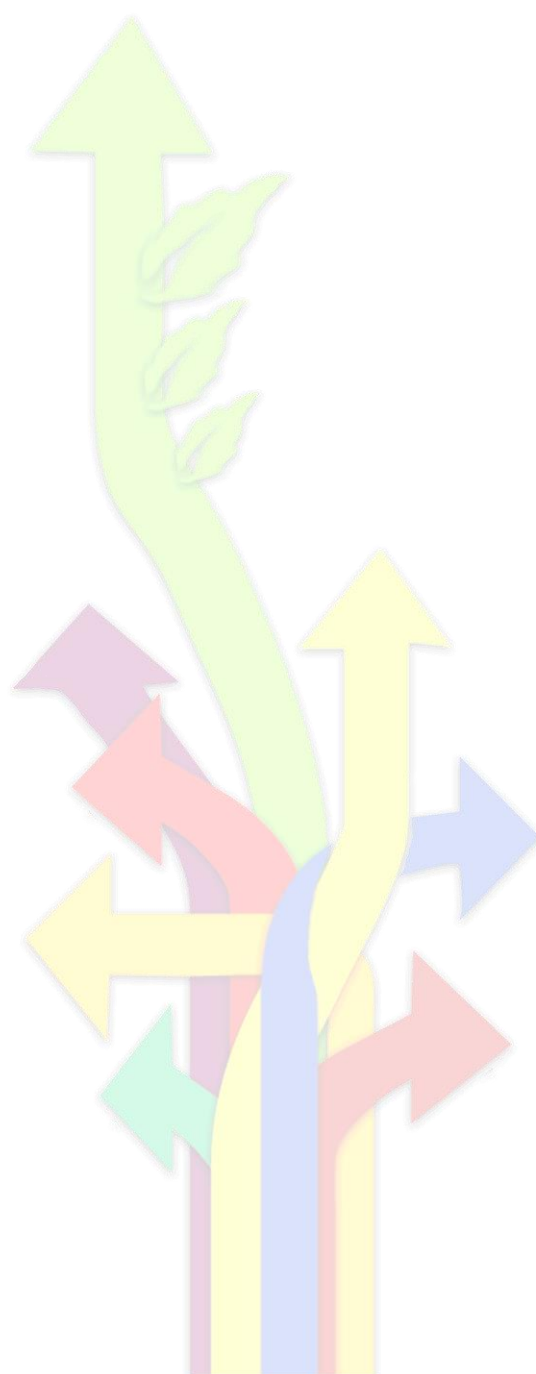


13th Annual Electrochemistry Seminar of Iran



13th Annual Electrochemistry Seminar of Iran

Analytical Electrochemistry



13th Annual Electrochemistry Seminar of Iran

Electrochemical measurement of hydrazine at the surface of glass carbon electrode modified with palladium nanoparticles supported on poly (ethylene glycol) - block poly (citric acid)-functionalized Fe₃O₄ nanoparticles

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Abstract

Chemically modified electrode (CME) is the most important part of electrochemical sciences. Due to the unique properties such as, high electrocatalytic activity and high surface area, noble metal nanoparticles have attracted considerable attention for preparation of modified electrodes [1]. Hydrazine widely used in different industries but it has been known to be very harmful for human life and acute exposure can damage the liver, the kidneys, and the central nervous system [1,2]. In this work, the electrocatalytic oxidation of hydrazine has been studied on glassy carbon modified with palladium nanoparticles supported on poly (ethylene glycol)-block-poly(citric acid)-functionalized Fe₃O₄ magnetic nanoparticles (Pd@PCA-b-PEG-Fe₃O₄). Under optimized conditions, the proposed sensor showed good catalytic activity for the electrooxidation of hydrazine with a substantial decrease in anodic overpotentials and an increase in anodic peak current. Also, a linear relationship was observed between the voltammetric currents and the concentration of hydrazine within the range of 10–300 μ M with the detection limit (S/N = 3) of 3 μ M. Finally the proposed sensor was applied to determine hydrazine in water samples.

Keywords: Magnetic nanoparticles, Palladium nanoparticles, Electrochemical sensor, Hydrazine

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Investigating the pH range of a modified carbon nanotube sensor based on N-quinoline-2-carboxamido-8-aminoquinoline

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Abstract

Potentiometric monitoring of metal ions based on ion selective electrodes (ISEs) offer the advantages of speed and ease of preparation and procedures, relatively fast responses, reasonable selectivity thorough judicious choice of the membrane active materials, wide linear dynamic range, and very low cost. In this work, we wish to introduce a highly selective and sensitive Ho^{3+} ion-selective membrane electrode by incorporation of N-quinoline-2-carboxamido-8-aminoquinoline (QCA) as an ionophore, carbone nano tube and nano silica as modifier, paraffin oil as binder in a carbon paste electrodes (CPEs). The potentiometric response is independent of pH in the range of 3.8 - 9.3 .

Keywords: Potentiometric response, Ion Selective Electrodes (ISEs), pH range, Carbon Paste Electrodes (CPEs)

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Construction of a modified carbon nanotube composite sensor Based on N-quinoline-2-carboxamido-8-aminoquinoline

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Abstract

Potentiometric sensors have shown to be very effective tools for analysis of a wide variety of metal ions. Among them, carbon paste electrodes (CPEs) have attracted interest as a result of their improved renewability, stable response, and low ohmic resistance compared to membrane electrodes. N-quinoline-2 carboxamido-8-aminoquinoline (QCA) seemed to be a suitable ion carrier for metal ions due to the presence of intermediate donating N atoms in its structure. Preliminary solution study shows the selectivity of (QCA) toward Ho^{3+} (III) ions in comparison of other common cations, including alkali, alkaline earth, transition and heavy metal ions. To have a long – term stable electrode for potentiometric uses, (QCA) was applied as ionophore in preparation of a nano-composite carbon paste electrode. The carbon paste electrode composed of 2% MWCNTs, 0.3 % Nano silica, 25% Paraffin oil, 1% QCA, and 71.7% Graphite Powder showed the best response. The proposed sensor exhibits a Nernstian slope of $19.6 \pm 0.4 \text{ mV decade}^{-1}$ toward Ho^{3+} ions in the range of $1.0 \times 10^{-8} \text{ M}$ – $1.0 \times 10^{-2} \text{ M}$ and detection limit of $7.3 \times 10^{-9} \text{ M}$.

Keywords: Potentiometric Sensor, Carbon Paste Electrodes (CPEs) , Holmium, Ionophore, Nernstian slope

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Investigating of Potentiometric Titration of a modified carbon nanotube sensor based on N-quinoline-2-carboxamido-8-aminoquinoline

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Abstract

Potentiometric sensors have shown to be very effective tools for analysis of a wide variety of metal ions. Among them, carbon paste electrodes (CPEs) have attracted interest as a result of their improved renewability, stable response, and low ohmic resistance compared to membrane electrodes. N-quinoline-2 carboxamido-8-aminoquinoline (QCA) seemed to be a suitable ion carrier for metal ions due to the presence of intermediate donating N atoms in its structure. Preliminary solution study shows the selectivity of (QCA) toward Ho^{3+} (III) ions in comparison of other common cations, including alkali, alkaline earth, transition and heavy metal ions. To have a long – term stable electrode for potentiometric uses, (QCA) was applied as ionophore in preparation of a nano-composite carbon paste electrode. This electrode was used as an indicator electrode in the potentiometric titration of Ho^{3+} (III) ions with EDTA.

Keywords: Carbon paste electrodes (CPEs), Selectivity, Indicator electrode, Potentiometric titration

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Investigation of the response time of a modified carbon nanotube sensor based on N-quinoline-2-carboxamido-8-aminoquinoline

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Abstract

In this article a super selectivity potentiometric methodology, using an ion-selective electrode, for determination of Holumium ion (III) in aqueous solution was investigated. a Multi-walled carbon nanotubes (MWCNTs) was used in the composition of the carbon paste to improve conductivity and transduction of chemical signal to electrical signal. Moreover, incorporation of N-quinoline-2-carboxamido-8-aminoquinoline (QCA) as an ionophore to this composition caused to significantly enhanced selectivity toward Ho^{3+} ions. The electrode has a short response time (<10 s) and can be used for at least two weeks without any considerable divergence in potentials.

Keywords: Ion Selective Electrode (ISE), Multi-walled carbon nanotubes (MWCNTs), Response time, Holumium

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Determination of substrate Inhibition concentration of bienzymatic acetylcholinesterase and choline oxidase immobilized biosensor based on carboxylated carbon nanotubes

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Abstract

About 20% of the enzymes are inhibited by increasing the concentration of the substrate, and the acetylcholinesterase enzyme is also in this enzyme group. Substrate inhibition is often considered as a strange biochemical phenomenon. In this study a bienzymatic acetylcholinesterase (AChE) and choline oxidase (ChOx) immobilized biosensor based on carboxylated carbon nanotube (MWNT) modified glass carbon electrode (GCE) was fabricated for acetylcholine detection on the surface of the MWNT-modified GCE in 0.1M PBS. In order to investigate the electrocatalytic activity of biosensor, the enzymes response to a sequential increase in acetylcholin chloride concentration, acetylcholinesterase enzyme substrate, was studied by chronoamperometry method. The detection of inhibition of immobilized enzymes on carboxylic carbon nanotubes by acetylcholine chloride substrate at concentrations of 0.4 to 1.4 mM was investigated and the threshold concentration was obtained to inhibit the enzyme of 1 mM.

Keywords: *Biosensor, Acetylcholinesterase, choline oxidase, carboxylated carbon nanotubes, Acetylcholine chloride*

A novel polymeric ion selective electrode for selective determination of europium (III) in real samples

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Abstract

Ion-selective electrodes (ISEs) based on polymeric membrane with ion carriers have been used in the fields of environmental, medicinal and agricultural analysis. Since the late 1960s, many sensors with high selectivity for specific ions have been applied to measure different environmental samples [1–3]. In this study, a new sensor for europium (III) ion was prepared. Effects of experimental parameters such as membrane composition, amount of plasticizer and amount of additive on the potential response of Eu^{3+} sensor were investigated. The best operation obtained in the membrane including: 4.0 mg benzo-15-crown-5, 2.0 mg sodium tetraphenylborate (NaTPB), 64.0 mg nitrobenzene (NB) and 30.0 mg PVC. The sensor exhibited a Nernstian slope of 20.5 ± 0.3 mV/decade in Eu^{3+} activity. This sensor had a fast static response time of 5s and good reproducibility. It can be used for 32 days without any significant changes in its potential. The proposed electrode was used as an indicator electrode in potentiometric titration of Eu^{3+} with EDTA solutions. The proposed sensor was successfully applied for the recovery of Eu^{3+} ions in real samples.

Keywords: *Europium (III), PVC membrane, ion selective electrode*

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Benzo-15-crown-5 as a neutral ionophore in fabrication of potentiometric sensors

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Abstract

Potentiometric ion selective electrodes (ISEs) are one of the most important types of chemical sensors. A significant number of ionophores, including crown ethers, cryptands, aza-crowns and thiocrowns, have been exploited for the fabrication of polyvinyl chloride (PVC) membrane electrodes for the determination of alkali, alkaline earth, transition and heavy metal ions [1-2]. Potentiometric ionophore-based membrane sensors are preferred measuring tools for a variety of applications. In fact, ion selective electrodes (ISEs) for as many as 60 analysts have been described so far [3-4]. In this project, an europium (III) selective coated graphite electrode based on benzo-15-crown-5 as a neutral carrier has been fabricated. This sensor had a linear concentration range from 1.0×10^{-5} to 1.0×10^{-1} M with a good Nernstian slope in the pH range of 3.0-12.0. The detection limit of the electrode was 4.4×10^{-6} M. This electrode revealed a good selectivity towards Eu^{3+} ion over a wide range of metal cations and a good dynamic response time was observed in the whole concentration range. Also, this sensor was successfully used for direct determination of europium (III).

Keywords: Benzo-15-crown-5, Potentiometry, Coated graphite sensor

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Fabrication of a polymeric sensor using cobalt-nickel-iron nano composite for cerium measurement in different samples

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Abstract

The development of ion selective sensors are the well established in most frequent applications in the fields of environmental, agricultural, industrial and clinical analysis. This is due to their several advantages, such as high speed, ease of preparation, simple instrumentation, relatively fast response, wide working concentration range, adequate selectivity and low cost [1-3].

In this research, a highly selective Ce^{3+} coated graphite electrode was prepared into plasticized polyvinyl chloride (PVC) membrane. The best operation obtained in the membrane including: 4.0 mg benzo-15-crown-5, 2.0 mg nano composite cobalt-nickel-iron, 64.0 mg nitrobenzene (NB) and 30.0 mg PVC. The sensor exhibits a Nernstian slope (17.5 ± 0.5 mV/decade) in a wide linear concentration range of 1.0×10^{-8} - 1.0×10^{-1} M. The detection limit of the electrode is 7.0×10^{-9} M. The electrode has a fast response time of 5 s, with a satisfactory reproducibility and relatively long life time of 35 days without significant drift in potential. The electrode operates in the wide pH range of 2-11. This electrode reveals a good electivity towards Ce^{3+} ion over a wide range of alkali, transition and heavy metal cations. The electrode was used as an indicator electrode for potentiometric titration of Ce^{3+} cation with EDTA solutions with a sharp potential changes occurred at the end points. The proposed electrode was successfully applied for the recovery of Ce^{3+} ions in real samples.

Keywords: Coated geraphite ion selective electrode, Cerium cation, Potentiometry

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Electroanalytical determination of quinine in biological samples using molecularly imprinted polymer-Au nanoparticles/MWCNT-chitosan pencil graphite electrode

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Abstract

Quinine is one of the naturally occurring alkaloids that derives from specified cinchonabarks. Electrochemical method has very advantages such as low detection limit, high precision and sensitivity, low cost, speed and portability. Molecularly imprinted polymers (MIPs) are from important materials that have been used for construction and modification of sensors. A selective and very sensitive sensor was developed to determine quinine based on new molecularly imprinted polymer (MIP). The sensor was constructed by coating of pencil graphite electrode (PGE) surface with a mixture of chitosan and functionalized multiwall carbon nanotube (MWCNT). Then, the electropolymerization of 3-methyl-4-nitrophenol was performed in acidic media and in presence of HAuCl_4 solution to produce Au nanoparticles (AuNPs) together with MIP on the electrode surface. In this way, MIP-AuNPs/chitosan-MWCNT/PGE was constructed and applied to determine quinine. The signal reduction of the Fe^{2+} as a probe in differential pulse voltammetric (DPV) technique versus quinine concentration was linear in the range of 0.1 pM to 1000 pM. This sensor was applied for determination of the quinine in the plasma and urine samples.

Keywords:

molecularly imprinted polymer (MIP), quinine, 3-methyl-4-nitrophenol, quinine, AuNPs, DPV

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An electrochemical sensor based on reduced graphene oxide - hollow platinum nanoparticles modified pencil graphite electrode for ceftazidime determination

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Abstract

In this paper, a pencil graphite electrode (PGE) was modified using electrochemical reduced graphene oxide (rGO) and then hollow platinum nanoparticles (HPtNPs) were generated onto rGO/PGE by electrodeposition of cobalt and after that galvanic displacement reaction of cobalt nanoparticles with Pt (IV) ions (1). Differential pulse voltammetry was used for determination of ceftazidime (CFZ). Ceftazidime (CFZ) is a third-generation cephalosporins with wide spectrum activity against gram-negative and gram-positive bacteria. CFZ is an active agent against *Pseudomonas aeruginosa*, and widely used in the treatment of susceptible infections including respiratory-tract infections such as meningitis, other brain, spinal cord infections, abdominal (stomach area), skin, blood, bone, joint and urinary tract infections. The design experiment as a central composite design (CCD) methodology was developed as experimental strategy for optimization of the influence of variables on the performance of modified electrode. (2) The surface morphology and elemental characterization of the bare PGE, rGO/PGE, CoNPs/rGO/PGE and HPtNPs/rGO/PGE modified electrodes were analyzed performed using field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM), energy dispersive X-ray spectroscopy (EDX) and electrochemical impedance spectroscopy (EIS). CFZ was determined in the linear dynamic range of 5.0×10^{-4} to 1.0 nM using differential pulse voltammetry under optimized conditions.

Keywords: pencil graphite electrode (PGE), hollow platinum nanoparticles (HPtNPs), Ceftazidime (CFZ), Central composite design (CCD)

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Fabrication of a new electrochemical sensor based on gold nanoparticles decorated multiwall carbon nanotubes/graphene oxide nanocomposite for diclofenac determination

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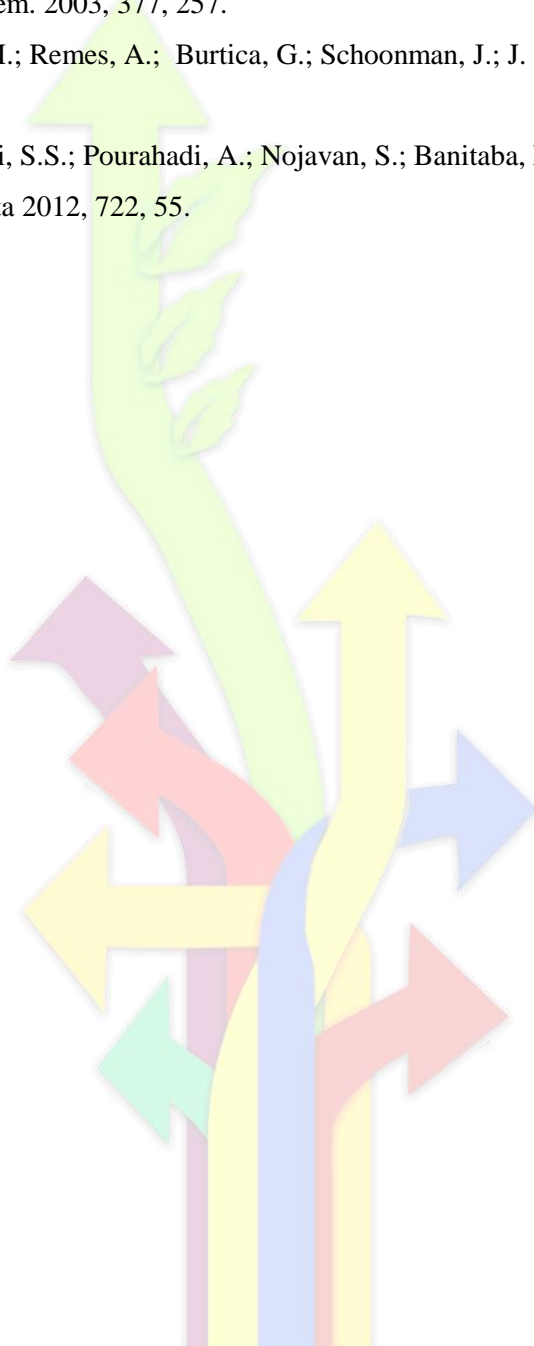
Abstract

Diclofenac sodium (DS) is a synthetic nonsteroidal anti-inflammatory drug (NSAID), with analgesic and antipyretic effects. It is widely used to relieve post-operative pain and treatment of rheumatoid arthritis, osteoarthritis, non-articular rheumatism and musculoskeletal injuries [1,2]. It is also used in veterinary medicine for the treatment of food-producing animals [3]. Therefore, it is important to be able to determine the amounts of diclofenac in biological and pharmaceutical samples. In this study, a new electrochemical sensor was fabricated for determination of diclofenac sodium based on a gold electrode modified with functionalized multiwalled carbon nanotubes (f-MWCNTs)/graphen oxide (GO) nanocomposite and gold nanoparticles. A three-dimensional f-MWCNTs/GO hybrid composite was prepared by homogenous dispersion of f-MWCNTs and GO then the suspension of the composite easily drop cast at the surface of the gold electrode and the electrode was further modified with gold nanoparticles through electrochemical deposition. Electrochemical impedance spectroscopy (EIS), cyclic voltammetry (CV), scanning electron microscopy (SEM) and energy dispersion spectroscopy (EDS) were used to investigate the surface of the modified electrode. Several parameters such as PH, scanning rate and the amount of nanocomposite controlling the performance of the sensor were examined and optimized. The proposed electrochemical sensor exhibited high specific electrochemical response to diclofenac and it was used for voltammetric determination of diclofenac in real sample solutions with promising results.

Keywords: *Diclofenac sodium, Electrochemical sensor, Carbon nanotubes, Graphen oxide, Gold nanoparticles.*

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13th Annual Electrochemistry Seminar of Iran

Development of a pH-assisted homogeneous liquid-liquid microextraction method using a switchable solvent for the electrochemical determination of nitrazepam

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Abstract

Nitrazepam is a hypnotic benzodiazepine drug used in the treatment of moderate to severe insomnia which has sedative and motor impairing properties as well as anxiolytic, amnestic, anticonvulsant, and skeletal muscle relaxant properties. In continuation of previous work [1], we wish to report a simple and efficient kind of homogeneous liquid-liquid microextraction based on an acid–base reaction using switchable-hydrophilicity solvent for the quantification of nitrazepam. Analyte is extracted into the N,N-dipropylamine as an extraction solvent via assistance of HCl and NaOH as pH adjustment reagents. After evaporation of solvent, extracted nitrazepam was analyzed by voltammetric methods. The influences of experimental factors on the extraction efficiency (type of acid, volume of acid, extraction temperature, ratio of the volume of organic solvent/water, pH of solution, volume of NaOH, extraction time, and salt addition) were investigated and optimized using voltammetric methods. Under optimal conditions, the calibration curve was plotted in the range of 0.03–300 ng/mL and the detection limit (at S/N=3) was obtained 9 ng/L. Finally, the proposed method has been applied successfully for the determination of nitrazepam in diluted urine samples.

Keywords: Nitrazepam , Electrochemical determination, liquid-liquid microextraction

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A genosensor based on MNPs/SPE for determination of prostate cancer gene sequence using hemin as an electrochemical indicator

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Abstract

Prostate cancer is a major health burden in all over the world. It is the most common cancer in males and the second leading cause of cancer-related deaths [1]. Genosensors or DNA hybridization biosensors are analytical devices for detection of specific DNA target sequences upon hybridization of the target strand DNA with the complementary probe immobilized on the solid substrate [2]. Electrochemical method can be used for studying of DNA hybridization or its interactions with small molecules and electrochemical transducers are more attractive for monitoring DNA detection at the molecular level and converting the hybridization event into an analytical signal [3]. Electrochemical DNA biosensors rely on the conversion of the DNA base pair recognition events into a useful electrical signal. Recognition of electrochemical DNA hybridization is accomplished via two methods including direct and indirect. Indirect DNA hybridization detection that carry out without any label, the signal induces owing to the inherent oxidation of adenine and guanine bases in DNA strands directly, whereas in indirect method the oxidation or reduction signal of an electroactive indicator causes detection of hybridization event [4]. The aim of this project is to identify prostate cancer gene sequence by using a simple and accurate method. For improving the detection sensitivity, a magnetic nanoparticles (MNPs) is used in the construction of the genosensor. MNPs are a kind of well-known nanomaterials with unique properties, such as: high specific surface area, low toxicity, excellent biocompatibility and ease of functionalization with a variety of ligands [5].

In this work, a new electrochemical genosensor based on MNPs modified graphite screen-printed electrode (MNPs/SPE) as transducer and hemin as a new electroactive label

is described. We used electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV) techniques to investigate the hybridization of DNA at the surfaces of MNPs/SPE. Also, the interaction of hemin and DNA is investigated at the surface of the electrode. The reduction peak current of hemin was increased linearly upon increasing the concentration of the target oligonucleotide sequence in the range of 7.5×10^{-12} to 2.0×10^{-6} M. The limit of detection (LOD) of the genosensor was as low as 1.994×10^{-12} M.

Keywords: *Genosensor; Prostate cancer gene; Magnetic nanoparticles; Hemin*

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An ultrasensitive and selective electrochemical aptasensor based on rGO- MWCNTs/Chitosan/carbon quantum dot for the detection of lysozyme

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Abstract

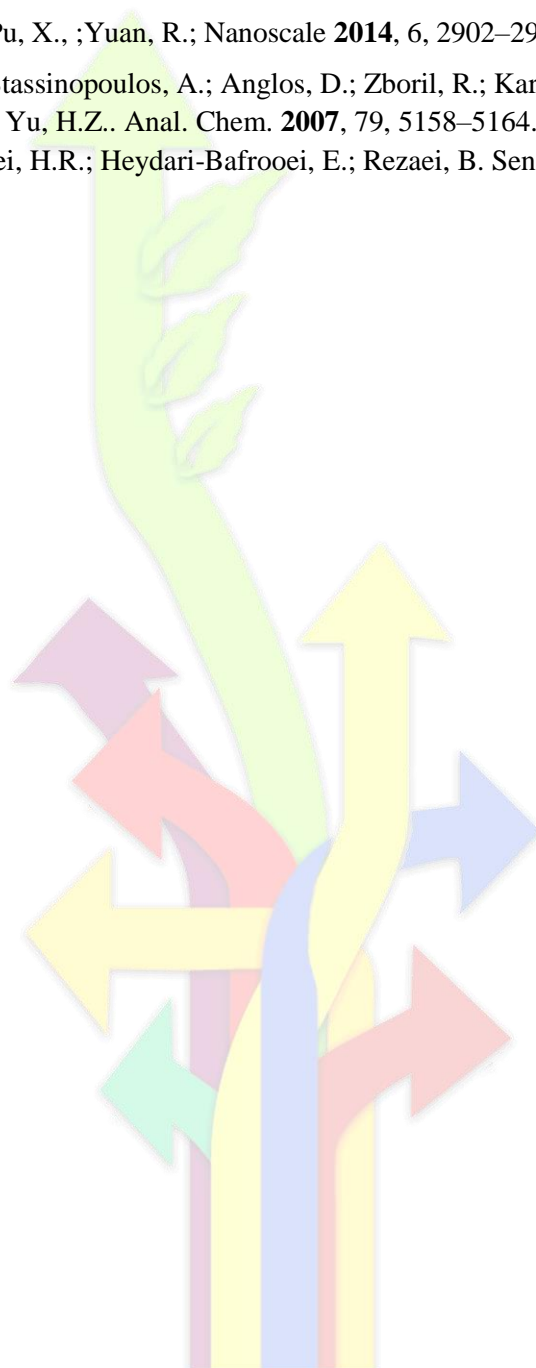
Using a nanocomposite of reduced graphene oxide (rGO), multi-walled carbon nanotubes (MWCNTs), chitosan (CS), and a synthesized carbon quantum dot (CQD) from CS on glassy carbon electrode (GCE) surface, an ultrasensitive and selective electrochemical aptasensor was prepared for lysozyme detection. The proposed nanocomposite (rGO-MWCNTs/CS/CQD) can provide a high surface-to-volume ratio, high conductivity, high stability, great electrocatalytic activity, and a suitable site for better immobilization of aptamers (due to the existence of many amino and carboxyl functional groups, and remaining oxygen-related defects properties in rGO). In addition, this nanocomposite allows considerable enhancement of the electrochemical signal and contributes to improving sensitivity. The amino-linked lysozyme aptamers were easily immobilized onto the nanocomposite through covalent coupling between the amino groups of aptamer and amino groups of the nanocomposite using glutaraldehyde (GLA) linker. For the aptasensor characterization, electrochemical methods including differential pulse voltammetry (DPV), cyclic voltammetry (CV), and electrochemical impedance spectroscopy (EIS) were used. In this electrochemical aptasensor, in the presence of lysozyme, the immobilized aptamer selectively catches the target lysozyme on the electrode interface that leads to a decrease in the DPV peak current and an increase R_{ct} in EIS as an analytical signal. Using the obtained data from DPV and EIS techniques, two calibration curves were drawn. The proposed anti-lysozyme aptasensor has two very low LODs 3.7 and 1.85 fmol L^{-1} within the wide detection ranges of 10 fmol L^{-1} to 50 nmol L^{-1} , and 20 fmol L^{-1} to 50 nmol L^{-1} for EIS and DPV calibration curves, respectively. The aptasensor also shows high reproducibility, sensitivity, rapidity, and specificity for lysozyme that could be used in biomedical fields.

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Keywords: *Aptamer; Lysozyme; Electrochemical aptasensor; Reduced graphene oxide; Multi-walled carbon nanotubes; Chitosan; Carbon quantum dot.*

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13th Annual Electrochemistry Seminar of Iran

Electrochemical Determination of Fenitrothion Organophosphorus Pesticide Using Polyzincon Modified-Glassy Carbon Electrode

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Abstract

In this paper, a glassy carbon electrode (GCE) was modified with polyzincon. The modified electrode was used as a simple, inexpensive and highly sensitive electrochemical sensor for the determination of organophosphorus pesticide fenitrothion. To fabricate the electrochemical sensor, GCE was immersed in 0.10 mmol L⁻¹ zincon solutions at pH 7.0 and then successively scanned between -1.00 to 2.20 V (vs. Ag/AgCl) at a scan rate of 70 mV s⁻¹ for six cycles. The morphology and structure of the polyzincon were studied with atomic force microscopy and scanning electron microscopy. A comparison of the electrochemical behavior of fenitrothion on the unmodified and polyzincon modified-GCE showed that in the modified electrode not only the oxidation peak current increased, but also the overpotential shifted to lower one. The experimental conditions such as sample solution pH, accumulation potential, and time were optimized. The differential pulse voltammetric responses of fenitrothion at potential about -0.60 V was used for the determination of fenitrothion. The peak current increased with increasing the concentration of fenitrothion in the range of 5 to 8600 nmol L⁻¹ with a detection limit of 1.5 nmol L⁻¹. Finally, the electrochemical sensor was used for the analysis of fenitrothion in water and fruit samples.

Keywords: Electrochemical sensor, Organophosphate pesticides, Fenitrothion, Poly-zincon

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An electrochemical aptasensor based on gold nanoparticles-modified graphene oxide for the ultrasensitive detection of breast cancer biomarker

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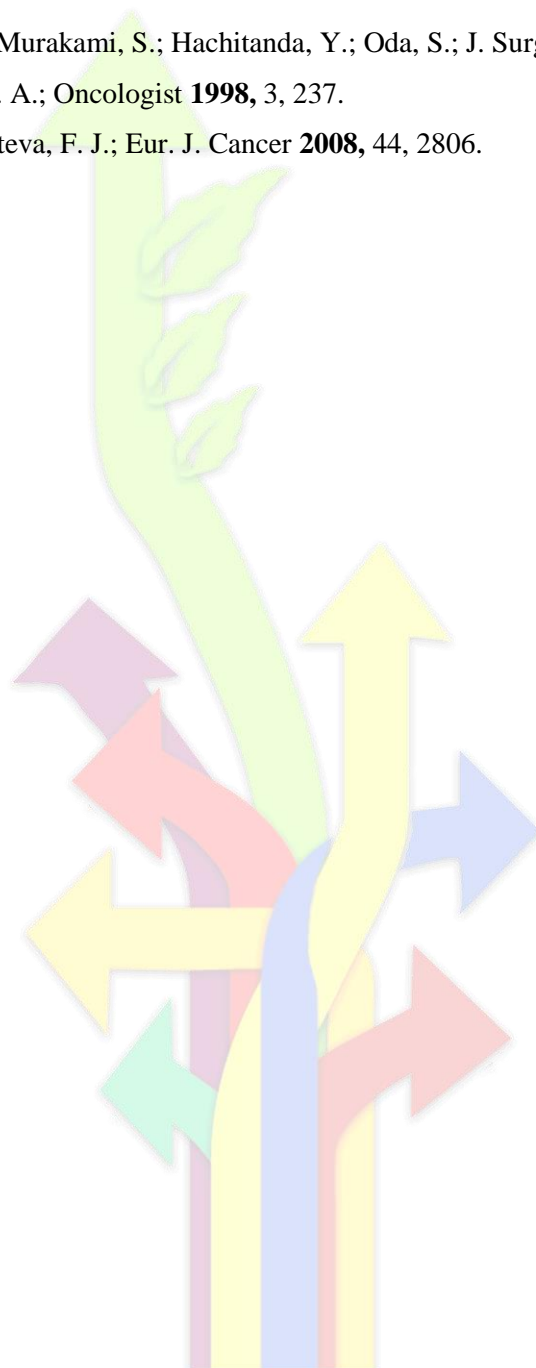
Abstract

Human epidermal growth factor receptor 2 (HER2), is encoded by a proto-oncogene located at the long arm of human chromosome 17q, plays an important role in human malignancies and is over-expressed in a high number of human breast cancers [1, 2]. The presence of HER2 on cancer cells is linked to increased cancer metastasis and tumour proliferation [3]. The aim of the present work is the fabrication of an electrochemical aptamer-based biosensor (aptasensor) for early diagnosis of breast cancer by detection of HER2. The proposed aptasensor was fabricated via covalent coupling of thiol-functionalized HER2-specific aptamer on the gold nanoparticles-graphene oxide film-modified glassy carbon (GC/rGO-AuNP) electrode. The resultant nanocomposite can provide a large surface area, excellent electrocatalytic activity, and high stability, which would improve immobilization sites for biological molecules, allow remarkable amplification of the electrochemical signal and contribute to improved sensitivity. HER2-specific aptamers were simply immobilized onto the rGO/AuNPs nanocomposite matrix. The electrochemical impedance spectroscopy (EIS), cyclic voltammetry (CV) and differential pulse voltammetry (DPV) were used to analyze the surface characterization of unmodified GCE and rGO/AuNPs modified GCE, and also the interaction between aptamer and HER2. In the presence of HER2, the aptamer on the adsorbent layer captures the target on the electrode interface, which makes a barrier for electrons and inhibits electron transfer, thereby resulting in decreased DPV signals of the modified GCE. Furthermore, the proposed aptasensor has a very low LOD of 0.05 ng mL^{-1} HER2 within the detection range of $0.02\text{--}50 \text{ ng mL}^{-1}$. The aptasensor also presents high specificity and reproducibility for HER2, which is unaffected by the coexistence of other proteins.

Keywords: *Biosensor; Breast cancer; Electrochemical impedance spectroscopy; HER2*

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13th Annual Electrochemistry Seminar of Iran

Preparation of Cu-MWCNT hybrid modified ionic liquids-carbon paste electrode and its application for determination of acetaminophen

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Abstract

Acetaminophen (N-acetyl-p-aminophenol or Paracetamol) is an antipyretic and analgesic drug commonly used against mild to moderate pain or for reduction of fever [1]. Nanomaterials due to their high conductivity and great surface area have received significant attention for modification of electrodes which exhibit good electrocatalytic activity towards oxidation of acetaminophen [2]. In this work, we have reported the preparation of copper/multi walled carbon nanotubes hybrid incorporated with ionic liquid/carbon paste electrode (Cu-MWCNT/IL/CPE) for determination of acetaminophen at phosphate buffer solution (pH=7.0). The Cu-MWCNT hybrid was synthesized by dispersion of 0.25 g MWCNT (COOH group) with appropriate amount of CuSO₄ solution and subsequent reduction the solution by hydrazine monohydrate. Modification of CPE was performed by hand mixing of graphite powder, Cu-MWCNT hybrid, ionic liquid (1-butyl-3-methyl imidazolium hexafluorophosphate) and viscose paraffin oil. The electrochemical investigations showed that the nanohybrid had electrocatalytic effect on oxidation of acetaminophen with improved current density. The oxidation peak potential of acetaminophen at the surface of Cu-MWCNT/IL/CPE appeared at 410 mV that was about 110 mV lower than that traditional CPE under similar conditions. This modified electrode had detection limit of 9.0×10^{-7} M using differential pulse voltammetry.

Keywords: Nanohybrid; Multi-walled carbon nanotubes; Copper particles; Ionic liquid; Acetaminophen

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Investigation of ionic liquid effect on the electrocatalytic performance of manganese/multi-walled carbon nanotubes nanohybrid for paracetamol oxidation

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Abstract

Ionic liquids (ILs) are kinds of important solvents with unique properties of good ionic conductivity, high viscosity, low toxicity, negligible vapor pressure, high chemical and thermal stability [1]. Hence, it has been widely applied in electrochemistry. On the other hand, carbon nanotubes (CNTs) have been receiving considerable attention in various areas due to their unique mechanical, electrical and chemical properties [2]. Due to non-covalent (π - π) interactions between the loop of imidazole IL and the π electron of CNTs, combination of IL and CNTs could provide an outstanding synergistic increase presentation like good electronic and ionic conductivity, high electro chemical stability, and good biocompatibility [3]. Herein, Mn-MWCNTs nanohybrid was prepared by mixing a suspension of MWCNTs and $\text{Mn}(\text{OAc})_2$ followed by reduction with hydrazine monohydrate in alkaline solution. Then, a homogenous mixture of the hybrid, paraffin oil, graphite powder and IL was used for construction of a novel electrochemical sensor for determination of paracetamol at phosphate buffer solution (pH=7.0). The obtained results showed that on the Mn-MWCNT/IL/CPE, the oxidation peak potential shifted to more negative directions with a higher oxidation peak current than that the CPE attributing to the presence of IL as the binder/modifier due to higher conductivity, fast electron transfer rate, good anti-fouling properties, intrinsic catalytic ability and adsorptive behavior.

Keywords: Nanohybrid; MWCNT; Ionic liquid; Paracetamol; Electro-catalysis

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Development of an electrochemical sensor based self-assembled monolayer (2-mercaptoethansulfonate) for detection of Copper (Cu^{2+})

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Abstract

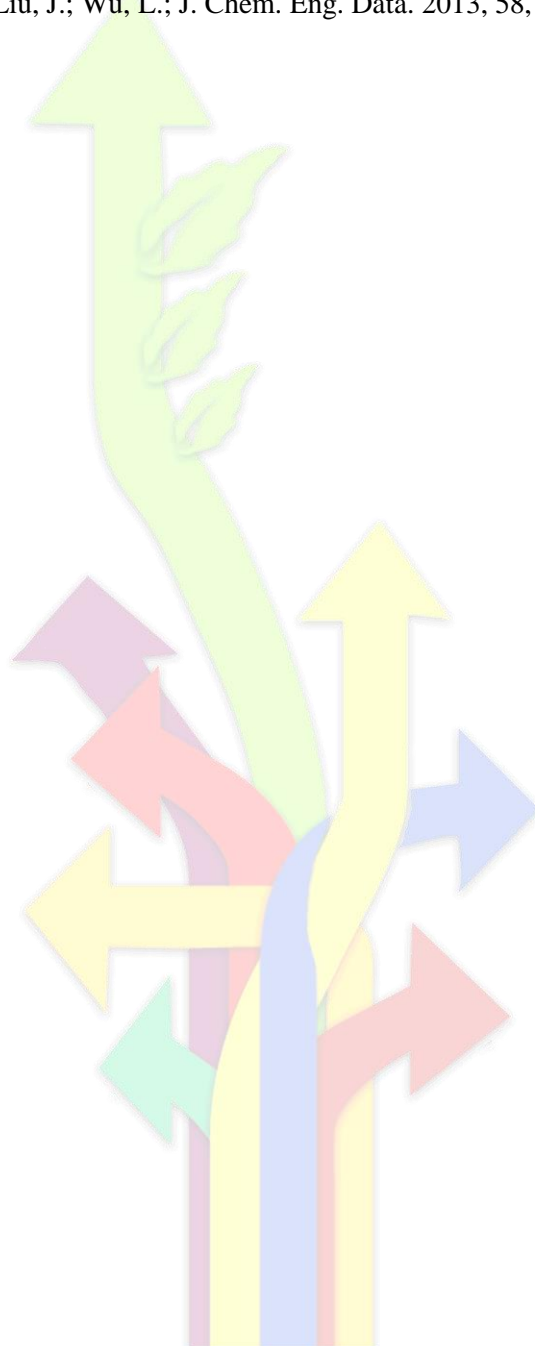
Copper (Cu^{2+}) is micronutrient element and plays an important role in the bone formation together with certain proteins and enzymes. However, it can be considered as a dangerous contamination when not maintained at the appropriate physiological concentration. Therefore, simple, reliable, selective and sensitive method for Cu^{2+} monitoring and removal are important for environmental safety and health [1]. In this project, an electrochemical sensor based on The Self assembled monolayers (SAMs) design was designed to detect copper ions. The SAMs electrode field in recent years has grown vastly in electrochemicals . The SAMs on metal electrode have been investigated widely in electrochemical, biochemical analysis or nanotechnology [2]. Many self-assembly systems have been designed, but monolayers of thiolates on gold electrodes are probably the SAMs most studied to date.the use of SAM-modified electrodes to improve the selectivity of gold electrodes has been reported. The mercapto group ($-\text{SH}$ group) has a high ability to adsorb heavy-metal ions, this unique characteristic reveals that the group has potential application in the removal of heavy-metal ions from industrial wastewater [3]. In this work, voltammetric behavior of Cu^{2+} on a gold electrode modified with the SAMs of 2-mercaptoethansulfonate has been investigated. The cyclic voltammetry (CV) and differential pulse voltammetry (DPV) was used to determine the electrode surface modification process. This negatively charged layer could act as a discriminating layer against Cu^{2+} based on the electrostatic interactions. The oxidation peak current increases linearly with the concentration of Cu^{2+} in the range of 2×10^{-8} to 2×10^{-6} M. The detection limit is 7.5×10^{-9} M. This method will be applicable to the determination of Cu^{2+} in industrial wastewater, and the good recovery of Cu^{2+} is obtained.

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Keywords: sensor; Copper (Cu^{2+}); Self-assembled monolayers; Voltammetry

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Simultaneous voltametric determination of L-tryptophan and L-tyrosine with the screen printed electrode modified by Fe₃O₄/GOX-COOH

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Abstract

L-tryptophan (Trp) and L-tyrosine (Tyr) are two main amino acids owing to their crucial roles in biological systems. They are precursors of hormones and neurotransmitters such as thyroxine and dopamine, as well as other physiologically important biomolecules [1]. Electrochemical methods have found many applications in the determination of the amino acids such as tyrosine and tryptophan owing to their electro-active aromatic groups [2]. In the many studies the chemically modified electrodes used to improve the anodic oxidation of amino acids. Other investigations have also indicates the importance of the carboxyl group in the surface adsorption of the related molecules. Moreover, the aromatic amino acids were quickly adsorbed onto the surface of the graphene oxide nano-composite with the strong π - π interactions [3].

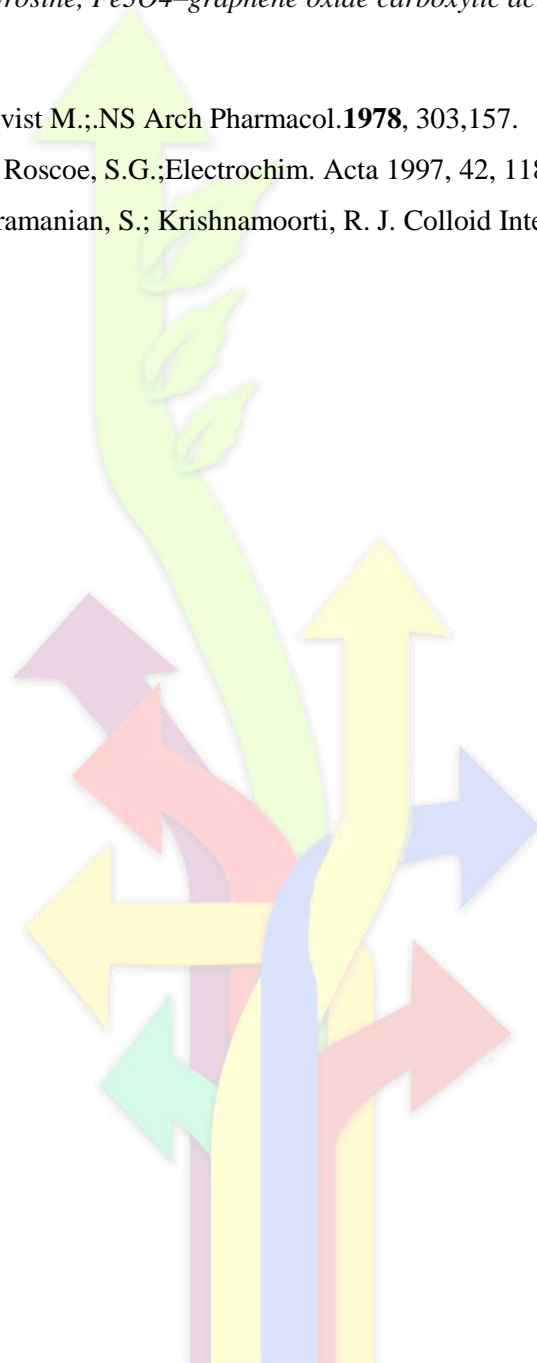
Here, the Fe₃O₄-graphene oxide carboxylic acid (Fe₃O₄/GO-COOH) nanocomposite was prepared. The composite (1-5 mg) was dispersed in 10 mL doubly distilled water and ultrasonicated for 2 h until a homogenous dispersion obtained. Subsequently a mixture of Fe₃O₄/GO-COOH and chitosan (%1 w/w) was electrodeposited onto the surface of a screen printed carbon electrode (SPCE) in a constant potential (-2 V vs. Ag/AgCl) for 80 s. The electrochemical behavior of the modified electrode exhibits an electrocatalytic activity towards the oxidation of the Trp and Tyr. The experimental conditions, including the ratio of Fe₃O₄/GO-COOH and chitosan, the electrodeposition time and potential and also the pH value were studied by differential pulse voltammetry (DPV). The peak potentials of tryptophan and tyrosine are closed at the pH values greater than 2. Therefore, their simultaneous determination by the SPCE/(Fe₃O₄/GO-COOH/Chitosan electrode in pH<2 is possible. Under the optimized conditions, the oxidation peak current was linear over a concentration range of 0.50 to 40.0 ppm for Try and 0.20 to 50.0 ppm for Tyr with a detection

limit of 0.20 and 0.15 ppm for Trp and Tyr respectively. The modified electrode was successfully applied for the simultaneous determination of Trp and Tyr in some food samples.

Keywords: Tryptophan, Tyrosine, Fe_3O_4 -graphene oxide carboxylic acid, Food, Electrochemistry

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Electrochemical determination of doxorubicin in pharmaceutical samples using nanostructure sensor

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Abstract

Cancer is a serious disease and an important cause of mortality in humans from many years ago. Many attempts have been made to treat cancer by scientist. Application of anticancer drugs in chemotherapy is the main way for treatment of cancer. Doxorubicin can be interferes with the growth and spread of cancer cells and helps to treatment of cancer and especially breast cancer. The electrochemical oxidation of oxorubicin was investigated at a ZnO nanoparticle/1-butyl-3-methylimidazolium tetrafluoroborate modified carbon paste electrode (ZnO-NPs/BMTFB/CPE). Doxorubicin could exhibit a good quasi-reversible signal with high sensitivity at a surface of ZnO-NPs/BMTFB/CPE compare to unmodified electrode. The ZnO-NPs was biosynthesized by celery extract and used for modification of carbon paste electrode. In addition, the ZnO-NPs/BMTFB/CPE exhibits two separated oxidation signals for simultaneous analysis of doxorubicin and dasatinib with $\Delta E \sim 300$ mV. Finally, the ZnO-NPs/BMTFB/CPE showed good ability for analysis of doxorubicin and dasatinib in injection and serum samples.

Keywords: doxorubicin, ZnO nanoparticle, Electrochemical sensor

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Voltammetric analysis of epirubicin in biological and pharmaceutical samples using nanostructure sensor

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Abstract

Epirubicin (EB) is a usual and useful anticancer drug for treat breast cancer. Due to lower side effect of EB compare to doxorubicin, this drug is favoured over doxorubicin for treat of breast cancer. The presence study showed that modifying a carbon paste electrode by CuO nanoparticle and 1-butylpyridinium hexafluorophosphate as a sensitive sensor to detect EB in the presence of topotecan (TP). The square wave voltammetric data showed that the oxidation of EB is catalyzed by modified electrode at pH = 7.0. The CPE/1-BPr/CuO-NPs showed the lower limit of detection of 0.008 μM and 0.3 μM for EB and TP, respectively. The CPE/1-BPr/CuO-NPs showed good ability for EB and TP analysis in real samples.

Keywords: Epirubicin, CuO nanoparticle, Electrochemical sensor

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Development of an electrochemical signal-on aptasensor for selective detection of tryptophan

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Abstract

Tryptophan (Trp) is a necessary amino acid which is required for normal growth, nitrogen equilibrium and brain functions in humans [1]. It is also used as a precursor for melatonin, serotonin and niacin [2]. Regrettably, when it was incorrectly metabolized, it begets a toxic product in the brain that makes schizophrenia, hallucinations and delusions [3].

On the other hand, because the concentration of Trp in biological samples is low, therefore, there is an urgent need for rapid, easy, sensitive, selective and less expensive techniques for quantification of Trp.

In recent years, in order to solve many of these challenges, a new class of ligands, called aptamers, is known to detect specific molecular targets. Aptamers possess several advantages that make it an ideal biosensing element such as improved temperature stability, shelf life, easy synthesis, flexible to modification, easy storage, low toxicity, more stable to biodegradation and high binding tendency and selectivity [4].

Among various aptamer-based biosensors, electrochemical aptasensors have received an increasing amount of attention for detection of analytes due to their high sensitivity, simplicity, portability, low expense and rapid response for molecular detection [5].

In the development of chemical sensors, among the many types of nanoparticles, silica is highly regarded. Silica nanoparticle (SN) can be grafted with a various types of functional groups, in order to immobilize different biomolecules such as enzymes, proteins, antibodies and DNA [6].

In this study, a simple and label-free signal-on electrochemical aptasensor is designed for sensitive and selective electrochemical detection of Trp based on the covalent attachment of

aptamer, as recognition molecule, on AuNPs/SiO₂-pr-NH₂/SPE and using hemin and [Fe (CN₆)]^{3-/4-} electrochemical probes, for “signal on” strategy.

The linear range of proposed aptasensor based on “signal on” mechanisms using “DPV” and “EIS” methods were 0.06–250 nM with corresponding LODs 0.026 and 0.01nM, respectively. The introduced strategy has several attractive advantages simultaneously such as: simplicity, fast response, no requirement for a specific label, inexpensive, efficient sensitivity, high selectivity, a useful method in medical diagnosis and it is a signal-on sensor with acceptable stability.

Keywords: *Tryptophan, Hemin, Signal-on aptasensor, Gold nanoparticles*

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Potentiometric Determination of Thallium Ions Using Carbon Paste Electrode by 18-Crown-6 as a modifier

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Abstract

Potentiometric method, using ion-selective electrodes, in comparison to other analytical methods, such as atomic absorption spectroscopy, chromatography, etc, has the advantages such as ease of use, speed and low cost. In this design, a sensitive and selective electrode was designed to measure thallium ion. The response of this electrode in the range of 10^{-7} to 10^{-3} molar concentration of thallium is a Nernstian response. The sensor has a detector value of 5×10^{-8} molar. From an environmental point of view, toxicity of thallium (I) is a problem. Thallium(I) compounds, such as thallium sulfate, thallium acetate and thallium carbonate are toxic. Therefore, ion-selective electrode is one of the best and easiest methods for measuring thallium ion concentration. The response time of this sensor is about 40 seconds. The optimal membrane structure, which resulted in favorable results, was 72.59% graphite, 1.2% (18- crown-6) ionophore, 1.27% sodium tetra phenyl borate and 24.25% paraffin, that has the best Nernstian tilt of 57.07 ± 1.09 . After several tests, it was found that the electrode response does not depend on pH changes in (5 – 7) PH range. The electrode can be susceptible for 21 days and has an acceptable response to thallium concentration changes. The sensor was also used as the detector electrode in the potentiometric titration with EDTA and the results were in good agreement with the actual value.

Keywords: *potentiometric, thallium, ion selective electrode, 18-crown-6*

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Nanostructure electrochemical sensor for determination of ferolic acid trance in food samples

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Abstract

Ionic liquid modified nickel oxide/ carbon nanotubes paste electrode (IL/NiO/CNTPE) had been fabricated by using hydrophilic ionic liquid 1-methyl-3-butylimidazolium bromide [MBIDZ] Br for determination ferolic acid trance in corn milk. In this work, production of NiO nanoparticles (NiO/NPs) by chemical precipitation is the approach utilized. Nanopowders properties are identified by scanning electron microscope (SEM), x ray diffraction (XRD) and Transmission Electron Microscope (TEM). The results obtained confirm the presence of nickel oxide nanopowders produced during chemical precipitation. In continuous, a novel NiO/NPs carbon paste electrode (NiO/NPs/CPE) had been fabricated. Electrochemical behavior of ferolic acid trance at the NiO/NPs /CPE had been investigated in pH 7.0 phosphate buffer solution (PBS) by cyclic voltammetry (CV), chronoamperometry (CA) and square wave voltammetry (SWV). Detection limit of ferolic acid trance was found to be 0.02 μM and the linear dynamic range was found 0.06-900 μM . The proposed sensor was successfully applied for the determination of ferolic acid trance in corn milk.

Keywords: Food sample analysis, Voltammetry, ferolic acid trance

Modified Carbon Paste Electrode Based on Graphen oxide nano particles and Ionic Liquid for Potentiometric Determination of Na¹⁺ Ions in Real Samples

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Abstract

in this article to determination negligible amounts of sodium was made a selective and sensitive carbon paste sensor modified with (1- Hexyl 3methyl imidazolium hexafluoro phosphate and graphene oxide nanoparticle.).in several reviews has been reported applied ionic liquids in carbon paste electrode composition (1-4). CPEs are extensively applied for coulometry, amperometry and potentiometry analysis because of considerable beneficial properties (5). the proposed electrode demonstrates admissible selectivity and low detection limit using the ionopher. for better respond to this sensor was utilized graphen.in order to more accurate response and optimization and the effect of independent variables are utilized from center composite design -method of the base of surface response method (RSM)in the help of soft designer expert. also, were investigated the effects of 5 significant factors;(graphite powder%, ionopher %, ionic liquid%, paraffin oil%,

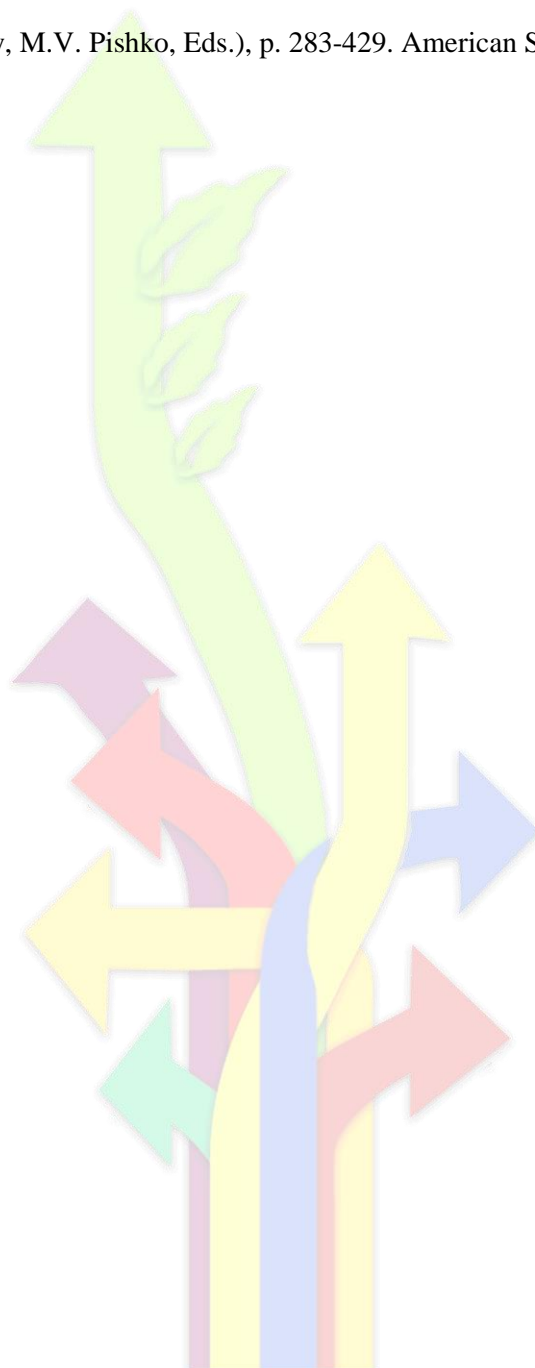
nano particle %) in 5 levels in 21 totally randomized tests. then obtained slop nerenstian 59.899 (mv per decade), linear dynamic range (10^{-1} - 10^{-7}) molar and limit of detection 10^{-7} molar for the optimized electrode with percentage of specified compound. also the electrode displayed excellent sensivity and high selectivity, for sodium compared to wide range of annoying cations low detection limit and desirable repeatability.

Keywords: Ionic Liquid, Carbon Paste Electrode, Potentiometric, nano particles

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13th Annual Electrochemistry Seminar of Iran

Direct electron transfer of choline oxidase immobilized on modified glassy carbon electrode with Fe₃O₄ nanoparticles

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Abstract

The most promising approach for the development of electrochemical biosensors is to establish a direct electrical communication between the biomolecules and the electrode surface. In this study Fe₃O₄ nanoparticles was synthesized and characterized by TEM electron microscopy. The nanoparticles were used for modification of glassy carbon electrode. Fe₃O₄ nanoparticles possess high surface area, nontoxicity, good biocompatibility and chemical stability. By immobilizing choline oxidase (ChOx) on the modified electrode, the enzyme direct electron transfer has been achieved. The electrochemical behavior of the modified electrode was studied by cyclic voltammetry, and a pair of redox peaks appeared in the cyclic voltammogram, indicating that direct electron transfer of ChOx was realized on the modified electrode. The modified electrode exhibited a pair of well-defined cyclic voltammetric peaks at a formal potential of 0.62 V versus Ag/AgCl in 0.1 M phosphate buffer solution at pH 7.0.

Keywords: Biosensor, choline oxidase, Fe₃O₄ nanoparticles, cyclic voltammetry

Highly sensitive voltammetric sensor for iodide determination based on carbon paste electrode modified with nanosized sulfate-modified α - Fe_2O_3

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Abstract

Iodide is an essential micronutrient for human body organism. It plays an important role in mental development, growth and basic metabolism^{1, 2} and its deficiency in the body is a serious threat because it is associated with thyroid and neurological disorders³. Recommended daily allowance (RDA) of iodine for adults is 140–200 $\mu\text{g}/\text{d}$ ⁴. On the other hand, deficiency and excess amount of iodine both can produce thyroid disorder, and further reduce various disease and hypothyroidism as well as hyperthyroidism^{5, 6}. Iodide is present in waters⁷, food⁸, pharmaceutical preparations⁴, clinical⁹, fodder¹⁰, biological samples¹¹ and environmental samples¹². Iodine species belongs to the toxic elements and causes pollution in the environment, especially because one of its radioactive isotopes, which have harmful impact¹³. Therefore, in the context of radioactive waste management and biological role of iodine, a number of analytical methods are employed and have been suggested to evaluate iodine speciation in the natural environment^{14, 15}. At present, several analytical methods have been proposed for the determination of iodide including, UV–vis spectrophotometry^{16–19}, spectrophotometric²⁰, chemiluminescence²¹, flow injection²², inductively coupled plasma (ICP–MS)²³, inductively coupled plasma–atomic emission spectrometry (ICP–AES)²⁴, diffuse reflectance spectroscopy²⁵, capillary electrophoresis²⁶, ion chromatography²⁷, gas chromatography with mass spectrometry detection^{15, 28, 29} and neutron activation analysis (NAA)³⁰. However, most of these techniques are complex sample preparation process, expensive for routine analysis and time consuming. By comparison, electrochemical method is less expensive and has additional advantages of fast response speed, simple operation, timesaving, high sensitivity, and real-time detection in suitable condition as an analytical technique so have been successfully applied in the determination of trace iodine.

Recently, much attention has been directed toward nanoparticle based sensors, in this approach we describe a new concept for simple and rapid determination of iodide. The nanosized $\alpha\text{-Fe}_2\text{O}_3$ particles were synthesized via sol-gel technique and then modified with sulfate groups. The synthesized nanoparticles were characterized by transmission electron microscopy, Fourier transform infrared spectroscopy (FT-IR), and X-ray diffraction (XRD) methods. The obtained material was incorporated with carbon paste electrode to fabricate an iodide sensor. It was shown that the modification of hematite nanoparticles with sulfate improved the sensor efficiency. The detection process was characterized by studying the redox behavior of iodide ions at both carbon paste and modified carbon paste electrodes by cyclic voltammetry (CV), impedance and tafel curve. The effect of electrode composition (sulfated $\alpha\text{-Fe}_2\text{O}_3$, carbon, binder, pH, scan rate and accumulation time) on its response was also investigated. The voltammetric method showed a good linear correlation coefficient ($r = 0.9979$) with a Nernstian behavior in a concentration range of 1.49×10^{-7} to 10^{-3} mol L⁻¹ and a detection limit of $0.149 \mu\text{mol L}^{-1}$ ($S/N=3$) for an accumulation time of 15 min. The relative standard deviation (R.S.D.) below 3.5% for all iodide standard solutions investigated. Determination of iodine in complex real samples such as sea water and soil were performed with high accuracy and satisfactory recovery results. The results were compared to those obtained by reference methods. The effect of different cations and anions interference was also discussed. The presence of sulfated $\alpha\text{-Fe}_2\text{O}_3$ in electrode composition was found to considerably facilitate iodide oxidation, with response currents directly related to iodide concentration.

Keywords: *iodide, electrochemistry, voltammetry, carbon paste modified electrode, sulfate-modified $\alpha\text{-Fe}_2\text{O}_3$*

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Development of an electrochemical sensor based on 2-mercaptoethansulfonate - self-assembled monolayer for detection of copper ions

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Abstract

Copper is micronutrient element and plays an important role in the bone formation together with certain proteins and enzymes. However, it can be considered as a dangerous contamination when not maintained at the appropriate physiological concentration. Therefore, simple, reliable, selective and sensitive methods for Cu^{2+} monitoring are important for environmental safety and health [1]. In this project, an electrochemical sensor based on the self assembled monolayers (SAMs) was designed to detect copper ions. The SAM electrode field in recent years has grown vastly in electrochemicals [2]. Many self assembly systems have been designed, but monolayers of thiolates on gold electrodes are probably the SAMs most studied to date. The use of SAM-modified electrodes to improve the selectivity of gold electrodes has been reported. The mercapto group ($-\text{SH}$ group) has a high ability to adsorb heavy-metal ions, this unique characteristic reveals that the group has potential application in the removal of heavy-metal ions from industrial wastewater [3]. In this work, voltammetric behavior of Cu^{2+} on a gold electrode modified with the SAMs of 2-mercaptoethansulfonate has been investigated. The cyclic voltammetry (CV) and differential pulse voltammetry (DPV) was used to determine the electrode surface modification process. The anodic peak current increases linearly with the concentration of Cu^{2+} in the range of 2×10^{-8} to 2×10^{-6} M. The detection limit is 7.5×10^{-9} M. This method will be applicable to the determination of Cu^{2+} in industrial wastewater, and the good recovery of Cu^{2+} is obtained.

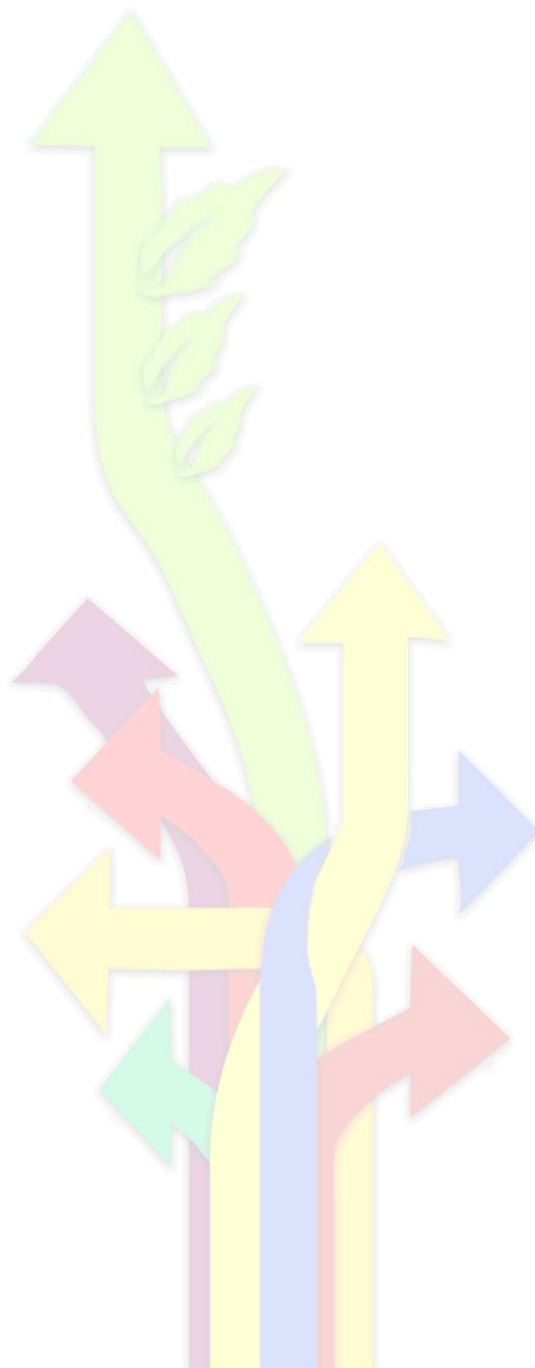
Keywords: Copper ions; Self-assembled monolayers; 2-mercaptoethansulfonate

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13th Annual Electrochemistry Seminar of Iran

Electrocatalytic determination of captopril using voltammetry methods at the nano zeolite modified carbon paste electrode

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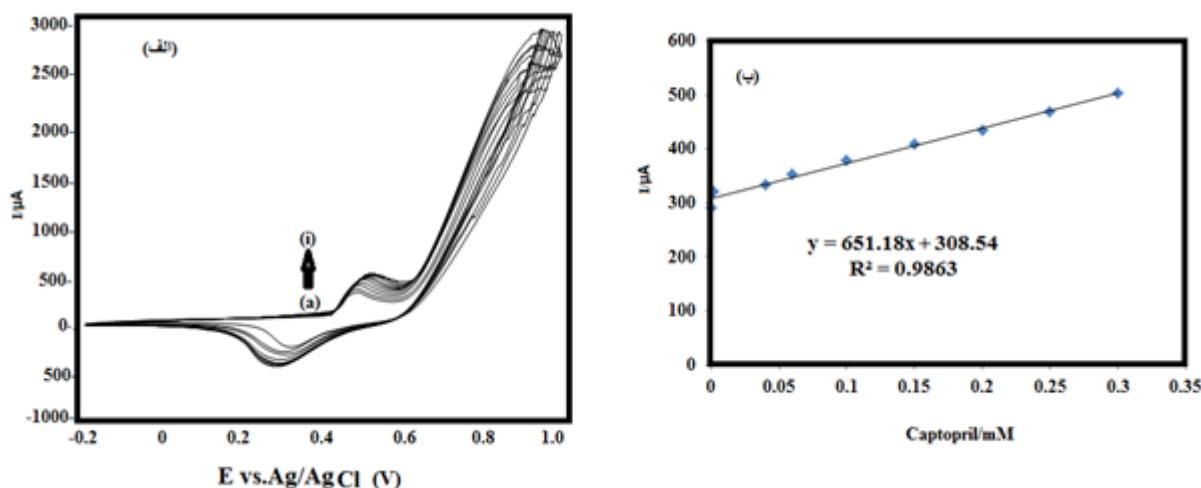
Abstract

Captopril (CAP) is an orally active inhibitor of the angiotensin converting enzyme and is generally used for the treatment of hypertensive illness alone or in combination with other drugs [1]. This compound can also be used to treat congestive heart failure [2]. Several in vitro studies indicate that captopril functions as an antioxidant both by rising the activities of antioxidant enzymes for example superoxide dismutase and glutathione peroxidase and by scavenging reactive oxygen species (ROS) [3]. Numerous methods have been applied to the determination of CAP, such as chromatography, spectrophotometry, fluorimetry, spectrophotometry, electrophoresis and electrochemical methods [1-4]. There are several limitations for electrochemical determination of pharmaceutical compounds, such as slow electron transfers kinetics, low sensitivity and reproducibility and high overpotentials. The chemical modifications with electroactive materials cause considerable advantages in the design and development of electrochemical sensors. Through the electrochemical reaction, the mediator transfers electrons between the analyte and the electrode with considerable decrease in the activation overpotential [5, 6]. The present work deals with zeolite-modified electrode doped with Fe ions for captopril oxidation. The performance of Fe-zeolite-modified carbon paste electrode towards captopril oxidation compared with unmodified carbon paste electrode using cyclic voltammetric and Chronoamperometry techniques. Cyclic voltammetry techniques proved to be very helpful in this concern.

To prepare the modified ZSM-5 zeolite with Ni ions, a prepartate of synthesized nano zeolite was immersed to 10 ml of 1M NiCl₂ solution for 3 h. Then, the ion exchanged zeolite was dried in oven at 347 K for 8 h. A mixture of 0.015g of nanosized NiZSM-5, 0.35g of graphite powder and paraffin was blended to prepare the zeolite modified carbon paste [NZMCP]. The resulting paste was then inserted in a glass tube. The electrical connection was implemented by a copper wire lead fitted into the glass tube. The carbon paste electrode (CPE), used for comparison, was prepared in the same way but omitting the zeolite addition

step. The oxidation of captopril was studied at CPE and NiZSM-5/CPE by cyclic voltammetric experiment in NiOH solution. Then the obtained results were compared with each other. The electrochemical behavior of modified carbon paste electrodes in the forms of NiZSM-5CPE and unmodified carbon paste electrode were studied using of cyclic voltammetric techniques. The obtained results show that the modified carbon paste electrode NiZSM-5CPE is the suitable electrode for electrooxidation of captopril fig 1. Also using of cyclic voltammetric techniques the linear dynamic range and detection limits for captopril were calculated 2×10^{-6} - 3×10^{-4} and 0.384mM respectively.

Keywords: Captopril, Electrocatalysis, Cyclic voltammetry, Zeolite



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A new insight to electrochemical detection of Endonuclease II enzyme

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Abstract

Highly sensitive and selective detection of low concentrations of specific biomolecules is important in biomedical diagnosis and also industrial processes. Many types of aptasensors have been developed for the detection of various analytes, but usually suffer from false positive signals and high background signals. In this work, we have developed an aptasensor platform for ultrasensitive biomolecule detection based on enzyme-assisted target-recycling signal amplification and reduced graphene oxide/Au Nano Particles (RGOAuNPs). By using a split molecular aptamer beacon and a nicking enzyme, the typical problem of false positive signals can be effectively resolved. Only in the presence of a target biomolecule, the sensor system is able to generate a signal, which significantly improves the selectivity of the aptasensor. We select endoglucanase II as model analytes in the current proof-of-concept experiments. It is shown that under optimized conditions, our strategy exhibits high sensitivity and selectivity for the detection of enzyme. In addition, this biosensor has been successfully utilized in the analysis of real biological samples.

Keywords: Nano Particles (RGOAuNPs), aptasensors, endoglucanase II

Electrochemical Investigation and Determination of Sulfite Using Modified Carbon Paste Electrode with Nano

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Abstract

Sulfite is widely used as a broad-spectrum preservative to prevent microbial spoiling and browning reactions across a wide range of consumable products. Sulfites are commonly used in the food and pharmaceutical industries as preservatives and antioxidants and brewing industry as an antibacterial agent [1-3]. In large quantities, sulfite and its oxidation products are pollutants. In particular, sulfiting agents have received extensive attention, as a result of their allergenic effect on those individuals who are hypersensitive. Previous methods for determining sulfite have included titration, chromatography, spectrophotometric and chemiluminescence methods [4,5]. In turn, electrochemical techniques provide reasonable results under laboratory conditions. On electroanalysis, modified electrodes have the capable potential to satisfy the requirements of a sensor to be fast and exact. Alternatively, it has been demonstrated that sulfite can undergo catalytic or mediated oxidation using some chemically modified electrodes (CMEs). The important group of are the transition metals compounds [6]. The presence of transition metals in zeolite framework of zeolite modified electrode (ZME) benefits its catalytic and electro-catalytic characteristics.

The goal of this research is electrochemical investigation and determination of sulfite using modified carbon paste electrode with nanozeolite. Iron (II) ions doped to nano ZSM-5 zeolite framework through ions exchange mechanism and electrochemical behavior of this electrode (CPE (unmodified carbon paste electrode) and ZSM-5/CPE (nano zeolite modified carbon paste electrode) were studied by using cyclic voltammetry technique. Iron (II) doped nano zeolite (FeZSM-5/CPE) was the optimum electrode for electrochemical determination of sulfite. The optimized pH was 7.0 and the optimized percent of modifier was 30:70 of nano zeolite – graphite. In optimum condition (linear dynamic range (LDR) of sulfite by voltammetry was 2×10^{-5} – 1.8×10^{-4} M and limit of detection was 9.1×10^{-6} M.

Keywords: Nano zeolite, Sulfite, Fe ion, Voltammetry

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A highly sensitive voltammetric platform for analysis of nitrite in foodstuff

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Abstract

Nitrite can be prevents from bacterial growth and therefore it used for the meat curing. Also this compound is an important reducing agent, in a reaction with the meat's myoglobin, gives the product a desirable pink-red 'fresh' color. It has important role in food industrial because nitrite can interact with amines to produce nitrosamines, which are well-known as carcinogenic substances [1]. The high value of nitrite content in the human blood results a decrease in oxygen transportation by the human blood follow-on methemoglobinemia, "blue baby syndrome" which is the reaction of nitrite with Fe (II), resulting transformation of hemoglobin to methemoglobin (HbFe (III)) [2]. According to the above points, the assay of nitrite is vital in environmental safety. The aim of the present study was usinCuO nanoparticles and ionic liquid for modification of carbon paste electrode. The proposed sensor was applied to the determination of nitrite. The plot of oxidation signal vs. concentration of nitrite was linear in the range of 0.03 to 500.0 μM . The new sensor was also examined for the determination of nitrite in food samples.

Keywords: Nitrite, CuO nanoparticle, Electrochemical sensor

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Sensitive and selective folic acid measurement with adsorption effect of N-Dodecylpyridinium chloride at Carbon paste electrode

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Abstract

Folic acid (FA) also called as pteroylglutamic acid (PteGlu), is essential for various biological functions. Deficiency of FA causes anemia, leucopenia, devolution of mentality, psychosis and increasing probability of heart attack and stroke. It is a necessary vitamin for healthy growth and development of foetus. Therefore, the measurement of FA is a significant research interest and several analytical methods have been reported for the determination of FA. In recent years, electrochemical methods have been used for the measurement of FA with the advantages including cheaper instruments, more convenient procedure, good sensitivity and selectivity.

A surfactant, with a hydrophobic C-H chain, can adsorb at the CPE surface via hydrophobic interaction and then change the electrode/solution interface, and finally affects the electrochemical response of drug.

Cetyltrimethylammonium bromide (CTAB) and N-Dodecylpyridinium chloride (DPC) are two types of cationic surfactants. CTAB has been widely used for surface modification of electrode, while for DPC, no successful application has been reported.

This study reports designing an electrochemical sensor for FA based on adsorption of cationic surfactant at a Carbon paste electrode (CPE) and compares the performance of DPC with CTAB to improve the electrochemical response of folic acid. In this work cyclic voltammetry (CV), differential pulse voltammetry (DPV), chronoamperometry (CA) and chronocoulometry (CC) methods were used and the experiments were performed with a certain electrode composition of 0.05 gr paraffin oil and 0.5 gr high purity graphite powder. After determining the superiority of DPC, for the first time, DPC was successfully used in electrochemical assays. Central composite design (CCD) was used to optimize the experimental parameters.

Under the optimized condition, the anodic peak current is linear to folic acid concentration in the range from (1.0×10^{-2} - $10.0 \mu\text{M}$). The detection limit was 3.1 nM and the relative standard deviation for $10.0 \mu\text{M}$ of folic acid was 2.3%. After studying the influence of the interfering ions and drugs, it was found that the proposed procedure has excellent selectivity. Finally, the method was successfully applied to the determination of folic acid in pharmaceutical tablets and urine.

Keywords: *Electrochemical studies, Folic acid, N-Dodecylpyridinium chloride, Cetyltrimethylammonium bromide, Carbon paste electrode*

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Fabrication and investigation of performance of nanocomposite electrodes composed of carbon quantum dots and copper nanoparticles for catalysis of hydrogen evolution reaction

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Abstract

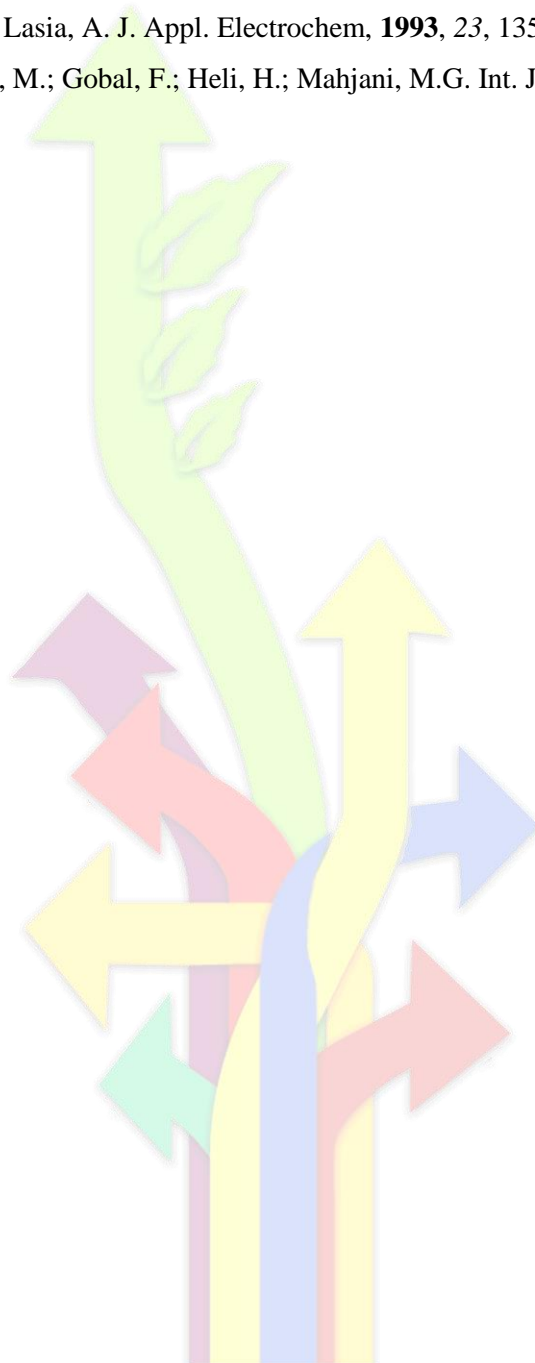
In the energy production and storage systems in an industrial plant, the system's efficiency is an important goal for the manufacturers. So, fabrication improvement and changes of the system compartments can increase the efficiency. The catalysis of reactions involved for electricity production is a good choice for efficiency improvement. Different types of catalysts are designed depending on the nature of process. They can be composed of metals, metal alloys and metal oxides. Carbon-based materials have also been used as a supporting catalyst, due to their high active surface area. In the present work, a composite composed of a carbon-based nanomaterial (carbon quantum dot) and metallic nanoparticles (copper) is used as a catalyst for catalysis of hydrogen evolution reaction. The present research project is oriented in order to introduce a new nanoporous and environmentally-friendly nanocomposite as a candidate for application in energy production and storage systems.

For fabrication of mentioned nano composite we used from electrochemical methods. Electrocatalytic behavior of prepared electrodes also have been studied by linear sweep voltammetry, chronoamperometry and electrochemical impedance spectroscopy techniques. Results show that presence of carbon quantum dots as a porous, suitable and easy synthesis based catalyst caused to increases of active surface area that this effect observed as decreases of overvoltage and increases of current density. Also, by comparing the kinetic quantity such as the tafel slope, the effect of improving the catalytic activity as a result of the presence of quantum carbon is well visible. The stability of prepared nano composite also was investigated by chronoamperometry method during 3h. Finally, the EIS results confirmed the positive effect of proposed nano composite by good electronic conductivity and weak charge resistance.

Keywords: Carbon Quantum Dot, High Active Surface Area, Catalytic Property, Nano Composite, Over Voltage

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Electrochemical determination of kojic acid in food samples using a modified carbon paste platform

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Abstract

Kojic acid is a small natural substance with wide application in cosmetic products and food samples such as skin whitening, soy sauce and vegetables. Kojic acid exhibit antibacterial (kill certain bacteria) and antifungal properties when was used in food and cosmetic products [1]. On the other hand, application of kojic acid can be cause an allergic reaction and cause unintended skin color changes. Therefore, it is very important for develop a highly sensitive method for the determination of kojic acid in trace level. In this research, we fabricated a carbon paste electrode modified with CuO nanoparticles and ionic liquids as highly powerful toll for analysis of kojic acid in the concentration range 0.02-450 μM with detection limit 9 nM. The proposed sensor was successfully used for determination of kojic acid in food samples.

Keywords: *Kojic acid, CuO nanoparticle, Electrochemical sensor*

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Preparation of flowerlike metal/metal oxides nanostructures derived from layered double hydroxide on nanoporous anodic aluminum oxide/aluminum wire as solid-phase microextraction fiber

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Abstract

In this study, a hierarchical CoAl layered double hydroxide (CoAl-LDH) has been fabricated via the in situ crystallization technique on nonporous anodic aluminum oxide/aluminum wire (AAO/Al) as both the substrate and the aluminum source. Then hierarchical peony-like metal/metal oxides ($\text{Co/Al}_2\text{O}_3$ and $\text{Co}_3\text{O}_4/\text{Al}_2\text{O}_3$) composites assembled with uniform nanosheets were successfully synthesized using CoAl Layered double hydroxides as self-sacrificial templates. The prepared wire with high surface area and compact structure was used as solid phase microextraction (SPME) fiber for separation and determination of benzene, toluene, ethylbenzene and xylene (BTEX) from aqueous solutions in combination with gas chromatography-flame ionization detector (GC-FID). SPME is a solvent-free microextraction technique applicable for the extraction of analytes from various matrices. In spite of the great applicability of SPME method, it has some major difficulties such as low chemical and mechanical coating stability, memory effect, expensive fibers, limited selection of commercially available fibers, fragility and limited lifetime of the fiber, and easy swelling in organic solvents. Therefore, most attempts such as this study have been focused on obviating the disadvantages of SPME. An experimental design optimization strategy (CCD) was applied for optimizing the important extraction parameters such as extraction temperature, extraction time, ionic strength and desorption time and temperature.

Keywords: Metal/metal oxides nanostructures, aluminum oxide, Solid phase microextraction, Nonoporous anodic aluminum oxide, BTEXs

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Multivariate optimization of mebeverine analysis using molecularly imprinted polymer electrochemical sensor based on silver nanoparticles /polypyrrole film

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Abstract

Thin film of a molecular imprinted polymer(MIP) based on electropolymerization method with sensitive and selective binding sites for mebeverine(MEB) was developed. This film was cast on graphite pencil electrode by electrochemical polymerization in solution of pyrrole and template MEB via cyclic voltammetry scans and further electro deposition of silver nanoparticles(AgNPs). All electroanalytical measurements were done at room temperature by differential pulse voltammetry (DPV). Several significant parameters controlling the performance of the MIP sensor were examined and optimized using multivariate optimization methods such as Plackett–Burman design (PBD) and central composite design (CCD). The AgNPs-MIP-PGE exhibited excellent electrocatalytic behavior for the oxidation of MEB as evidenced by the enhancement of the oxidation peak current and the shift in the oxidation potential to less positive values (66 mV) in comparison with a MIP-PGE (72 mv). Under the selected optimal conditions, MIP sensor was showed two linear ranges from

1×10^{-8} to 1×10^{-6} and 1×10^{-5} to 1×10^{-3} M with limit of detection of

8.6×10^{-9} M. The imprinted sensor showed the advantages of high porous surface structure, ease of preparation, good reproducibility and repeatability (RSD 1.1%), high selectivity and sensitivity.

Keywords: *Molecularly imprinted polymer; Electrochemical sensor; Mebeverine; Multivariate optimization; Silver nanoparticle.*

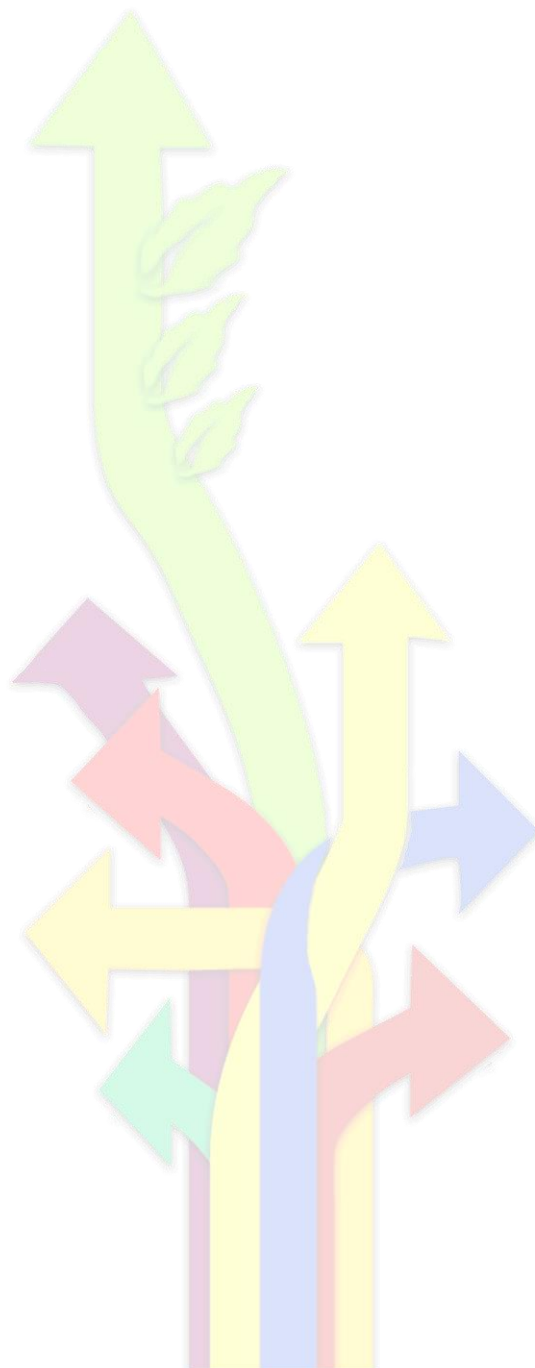
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13th Annual Electrochemistry Seminar of Iran

Comparison of electrochemical and spectrometric methods in the antioxidant activity evaluation of some fruits from north of Iran

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Abstract

The antioxidant capacity is defined as the ability of compound (or mixture of compounds) to inhibit the oxidative degradation of various compounds like preventing lipid peroxidation. These methods are usually based on the direct reaction between studied compounds and free radicals (quenching or scavenging) or on the reaction with transition metals [1].

Spectrometric methods are mainly used in the analysis of antioxidant properties. However, these methods are dependent on many parameters, such as: temperature, analysis time, character of a compound or mixture of compounds (extracts), concentration of antioxidants and prooxidants and many other substances [2,3].

Electrochemical methods used for the determination of antioxidant capacity have been still developing. These provide rapid, simple and sensitive alternative methods in the analysis of bioactive compounds associated with the scavenging of the radicals as well as the antioxidant capacity itself. They are low-cost and usually do not require time consuming sample preparation [4].

In the present work, we investigated electrochemical evaluation of antioxidant capacity of persimmon and hawthorn fruits from north of Iran. Hence, definite amounts of dried persimmon and hawthorn samples were weighted and then extracted with different solvents such as ethanol and methanol by soxhlet. After extraction, 250 μ L of obtained extracts was diluted with 15 mL of phosphate buffer 0.1M (pH 5.5). Electrochemical responses were obtained with a three electrode system consisting of a glassy carbon electrode, a Pt wire and the Ag/AgCl/KCl 3M representing the working electrode, the counter electrode and the reference electrode, respectively. The antioxidant and redox profile of fruits extracts were characterized by means of cyclic voltammetry and differential pulse voltammetry. The

antioxidant potential expressed by electrochemical index (EI) was compared to the results obtained with the 2,2-diphenyl-1-picrylhydrazyl (DPPH) method.

Each extract was dissolved in an appropriate volume of water to give final concentrations ranging from 50.00 to 300.00 µg/mL. Standard ascorbic acid solutions were also prepared in water to give the same concentration range. 1.5 mL of each solution was transferred to a test tube and mixed with 1 mL of 0.3 mmol/L DPPH. A control solution was prepared by adding methanol (1.5 mL) to 0.3 mmol/L DPPH (1 mL). The experiment was run in triplicate. The solutions were mixed well and left in the dark for 30 min. After 30 min the solutions were analyzed on a UV–vis spectrophotometer at 517 nm. The percentage of antioxidant potential was calculated using the formula:

$$\% \text{ Inhibition} = \frac{A_c - A_s}{A_c} \times 100$$

where, A_c is absorbance of the control solution, A_s is absorbance of the sample or standard. Also, the extracts were analyzed by Gas chromatography–mass spectrometry (GC-MS).

Keywords: Antioxidant activity, Persimmon, Hawthorn, Electrochemical Index.

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Electrooxidation and voltammetric determination of hydrazine in aqueous media at the surface of glassy carbon electrode in the presence of chlorohydroquinone as a homogeneous electrochemical catalyst

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Abstract

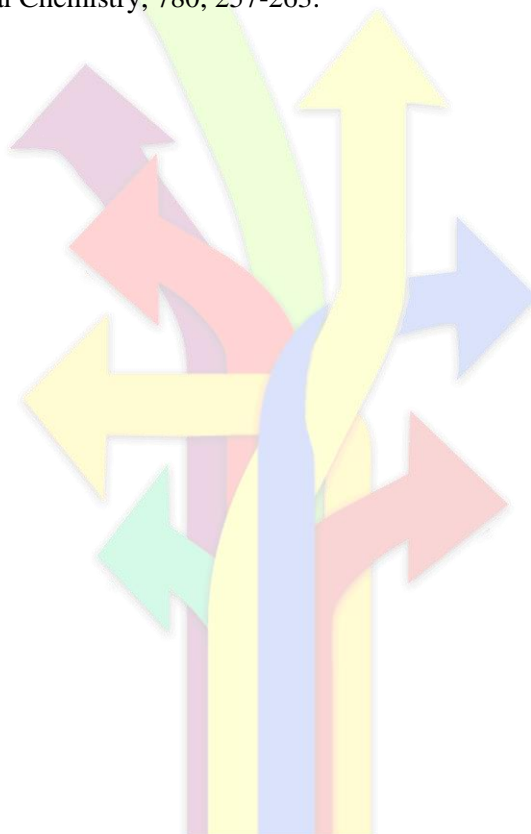
According to environmental protection agency (EPA), hydrazine and its derivatives are known as environmental pollutants, and the hydrazine limit in sewage is set to be 1 ppm [1]. Hydrazine is absorbed through the skin, lungs, and gastrointestinal tract and is spread rapidly throughout the body. In the case of acute human toxicity, such as, multiple respiratory system infections, central nervous system, liver, lungs, brain, kidneys, DNA, as well as cancer, has been reported [2]. Therefore, its measurement was of great importance, but electrooxidation of hydrazine at the surface of carbon electrodes and most other common electrodes has a slower kinetics and requires high over potential. For this reason, in the recent years, much attention has been paid to the expansion of the use of catalysts for hydrazide electrooxidation [3,4].

In this work, electrochemical oxidation and voltammetric determination of hydrazine has been studied on the surface of glassy carbon electrode (GCE) in the presence of chlorohydroquinone as a homogeneous electrochemical catalyst in hydrazinum hydroxide aqueous solution. The electrochemical behavior of chlorohydroquinone was studied in aqueous media by using cyclic voltammetry method. The electrochemical oxidation of hydrazinum hydroxide in the presence of this mediator at GCE and the effect of various factors on its electrocatalytic ability were investigated. The results show that the optimum pH value for electrocatalytic oxidation of hydrazinum hydroxide in the presence of chlorohydroquinone is pH=8.00. The electrocatalytic oxidation peak current of hydrazinum hydroxide in the presence of this mediator at GCE were linearly depended on its concentration in the range of 1.0×10^{-5} M – 2.0×10^{-2} M using cyclic voltammetry method. The detection limit (3σ) was determined as 6.040×10^{-6} M.

Keywords: Chlorohydroquinone, Hydrazine, Homogeneous electrocatalyst, Glassy carbon electrode.

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13th Annual Electrochemistry Seminar of Iran

Conductometric studies of Ag^+ , Co^{2+} , Hg^{2+} , Zn^{2+} , Ni^{2+} , Cu^{2+} , Cd^{2+} , Mg^{2+} , Cr^{3+} , Fe^{3+} and Pb^{2+} ions by new pyrazoloisoindol derivative ligand in nonaqueous solution

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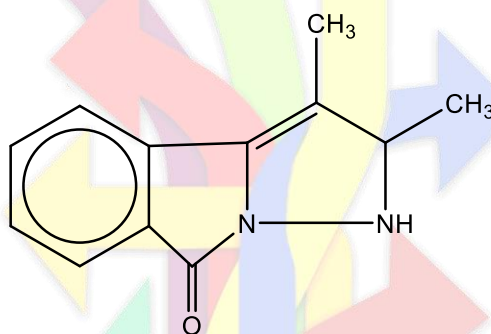
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Abstract

The complexation reaction between 2,3-dimethyl-1,2-dihydro-8H-pyrazolo[5,1-a]isoindol-8-one, ligand and Ag^+ , Co^{2+} , Hg^{2+} , Zn^{2+} , Ni^{2+} , Cu^{2+} , Cd^{2+} , Mg^{2+} , Cr^{3+} , Fe^{3+} and Pb^{2+} ions were studied conductometrically in acetonitrile solution. The formation constants of the 1:1 and 2:1 complexes (metal to ligand) were calculated from the computer fitting of the molar conductance in various mole ratios at 15, 25, 35 and 45 °C. The enthalpy and entropy changes of the complexation reactions in acetonitrile were estimated at four different temperatures.



Scheme 1. 2,3-dimethyl-1,2-dihydro-8H-pyrazolo[5,1-a] isoindol-8-one

Keywords: Conductometric, Stability constant, Pyrazoloisoindol derivative ligand

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9805. **13th Annual Electrochemistry Seminar of Iran**

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Conductometric studies of the thermodynamics complexation of Ag^+ , Co^{2+} , Hg^{2+} , Zn^{2+} , Ni^{2+} , Cu^{2+} , Cd^{2+} , Mg^{2+} , Cr^{3+} , Fe^{3+} and Pb^{2+} ions with new shift base ligand derivatives in acetonitrile solution

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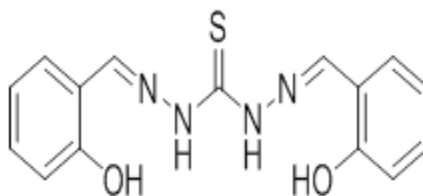
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Abstract

The complex-formation reactions between of Ag^+ , Co^{2+} , Hg^{2+} , Zn^{2+} , Ni^{2+} , Cu^{2+} , Cd^{2+} , Mg^{2+} , Cr^{3+} , Fe^{3+} and Pb^{2+} ions with (N,N'-bis (2-hydroxybenzylidene) hydrazine-1 carbothiohydrazid) (scheme 1) has been studied by conductometric methods in acetonitrile at various temperatures. Formation constants of the 1:1 and 1:2 complexes (metal ion to ligand) were calculated from the computer fitting of the absorbance-mole ratio data and molar conductance-mole ratio data at different temperatures. The enthalpy and entropy changes of the complexation reaction were determined from the temperature dependence of the formation constants.



Scheme 1. (N,N'-bis (2-hydroxybenzylidene) hydrazine-1 carbothiohydrazid)

Keywords: Conductometric, Complexation, (N,N'-bis (2-hydroxybenzylidene) hydrazine-1 carbothiohydrazid), formation constants.

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Electrochemical determination of entacapone in the presence of levodopa and carbidopa using a carbon paste electrode modified with a SnO₂/CuS nanocomposite.

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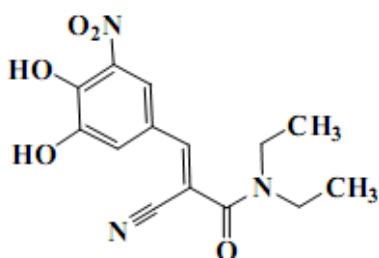
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Abstract

Entacapone (ENT), (E)-2-cyano-3-(3,4-dihydroxy-5-nitrophenyl)-N,N-diethyl prop-2-enamide belongs to the class of antiparkinson agents (Scheme 1) [1]. The simultaneous pharmaceutical analysis of multi component drugs represents a challenge due to a large total number of analytes present in the sample [2]. In this research work, a novel carbon paste modified electrode (CPME) with SnO₂/CuS nanocomposite has been developed for electrocatalytic oxidation and determination of the ENT in the presence of levodopa and carbidopa. The electrochemical behavior and determination of ENT at the SnO₂/CuS nanocomposite CPME were investigated by cyclic voltammetry and differential pulse voltammetry techniques respectively (Fig 1). The results showed that the sensor had excellent electrocatalytic activity toward determination of ENT and exhibited a wide linear range (2nM-40μM) and a low detection limit (1nM). The proposed electrode displayed excellent repeatability and long-term stability and it was satisfactorily used for determination of ENT in pharmaceutical dosage forms, human plasma and urin with high recovery.

Keywords: Nanocomposite, Entacapone, Levodopa, Carbidopa, Paste electrode



Scheme 1: Chemical structure of ENT.

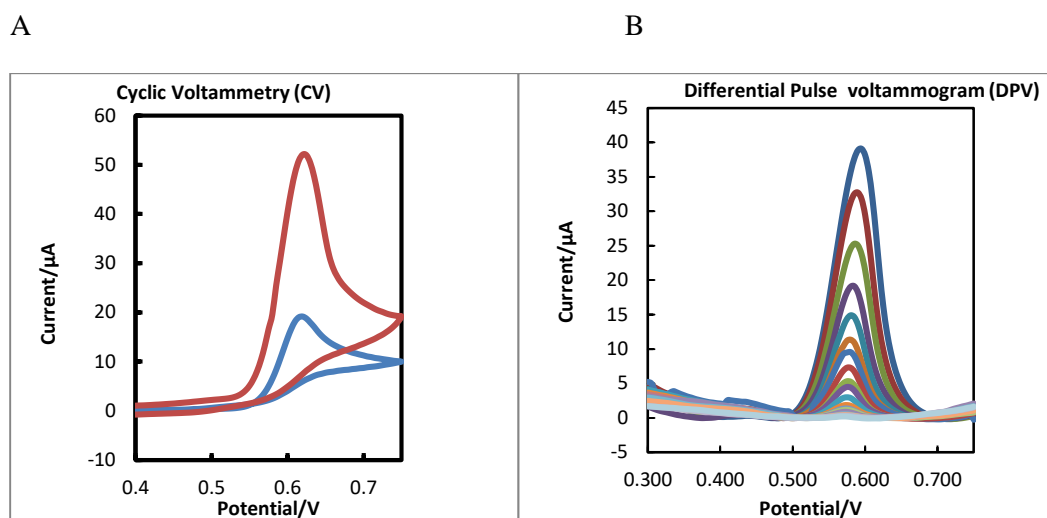


Fig 1 : A) Cyclic voltammograms of $0.5 \times 10^{-3} \text{ mol L}^{-1}$ ENT in B-R buffer pH 2 at a scan rate of 50 mV s^{-1} , carbon past bare (blue peak), carbon paste SnO_2/CuS nanocomposite (red peak). B) differential pulse voltammetry of carbon paste SnO_2/CuS nanocomposite in B-R buffer (pH 2) containing different concentrations of EN.

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Manufacturing of Electrochemical Modified Graphite Electrodes to Determine Some Metal Traces

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Abstract

This research includes preparation of new working Modified Graphite Electrodes (MGE). The first modified electrode based on 1,3-di phenyl-5-P-nitro phenyl formazan in order to Differential Pulse Anodic Stripping Voltammetric (DPASV) determination of lead and cadmium ions simultaneously in aqueous solutions and the second one based on benzaldehyde-2,4-dinitro phenyl hydrazone to determine silver ions in aqueous solutions. After that, studying the effect of the most important analytical and technical conditions (Electrolyte, Electrolyte Concentration and pH. Accumulation potential, Accumulation time, Potential scanning rate and Pulse amplitude) on the voltammetric curves. Standard deviation $SD(Cd) = 0.020\text{mg/l}$, $SD(Pb) = 0.021\text{mg/l}$ and $SD(Ag) = 0.011\text{mg/l}$.

Keywords: DPASV, modified working electrode, Lead, Cadmium, Silver, traces.

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Combination of micro liquid liquid extraction and differential pulse voltammetry for determination of TBHQ in edible oil samples by pre-anodized glassy carbon electrode in the single drop mode

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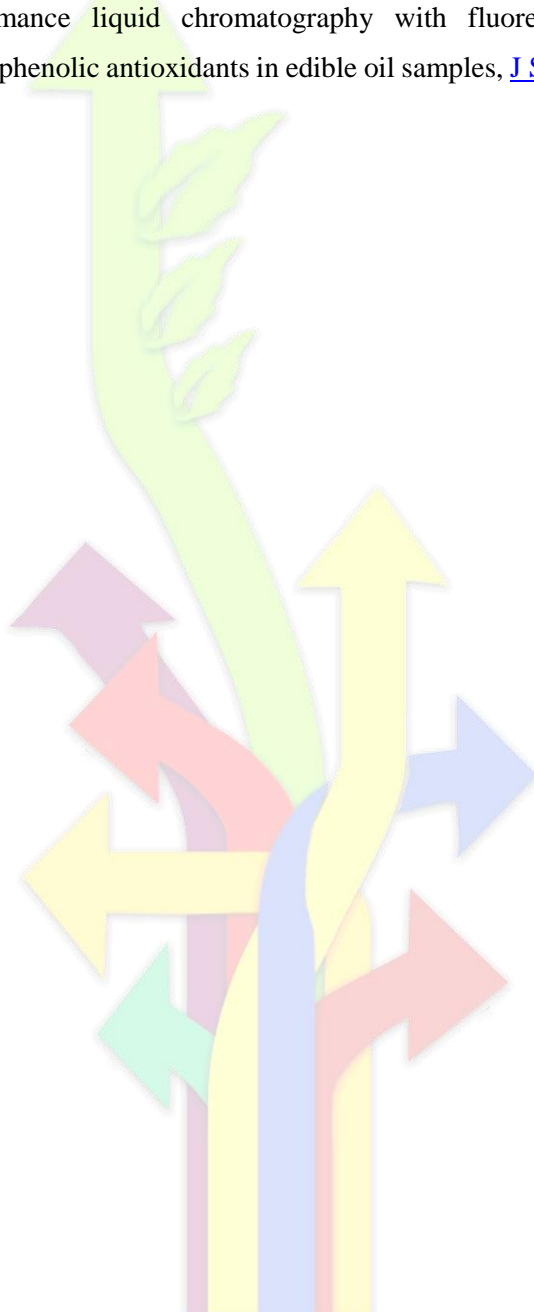
Abstract

t-butyl hydroquinone (TBHQ) is a synthetic phenolic antioxidant which is used as a preservative in edible oil sample. Recently, carcinogenic effect of TBHQ has been reported in high dosage [1], therefore, determination and control of TBHQ is very important in food samples and many attempts have been performed [2]. In this research, a combination of micro liquid liquid extraction with differential pulse voltammetry has been introduced for extraction and determination of TBHQ in edible oil samples. The amount of 300 μL methanol was used for extraction step. Pre-anodized glassy carbon (PAGC) electrode was utilized as working electrode. Counter and reference electrodes were thin platinum and silver wires. A 100 μL solution of a mixed extraction solvent and aqueous buffer was put on the surface of the PAGC. Different effective parameters such as type and volume of solvent, pulse width and height, type and concentration of pre-anodizing solution were evaluated and optimized. The analytical performances of the method were studied under optimal conditions. Dynamic range of the method was achieved in the range of 10-200 $\mu\text{g/g}$ and detection limit was 3.7 $\mu\text{g/g}$ based on $S/N=3$. Inter- and intra-day precision were in the range of 4.2-6.9 %. Finally, proposed method was performed for determination of TBHQ in different brands of edible oils in the market and the results were compared by the results of the HPLC method. The amounts were between 140 to 400 $\mu\text{g/g}$ and were not in the allowance level of Iranian standard. Relative recoveries were attained in the range of 95-105%.

Keywords: Synthetic phenolic antioxidant, Edible oil sample, Liquid phase microextraction, Differential pulse voltammetry, Pre-anodized glassy carbon (PAGC) electrode.

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Highly Sensitive Determination of Uric Acid Based on Zinc Oxide Nanoparticle/Graphene Modified Electrode

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Abstract

Determining the uric acid (UA) concentration in human body fluids (e.g., serum and urine) is required for the diagnosis of several diseases and determination of uric acid is a clinically valuable diagnostic indicator. Uric acid is the primary end product of purine metabolism in the human body. High level of uric acid in the body will lead to several diseases including gout, hyperuricemia and Lesh-Nyhan syndrome [1]. Among several mechanisms of transduction, the electrochemical one has received the largest credit, because of its easiness, low instrumentation cost, capability of miniaturization, and automation. In addition, the use of screen-printed electrodes (SPEs), appeared in the 1990s, largely contributed to this fortune, because of their reliability, reproducibility, mass production, and low cost [2]. Among carbon nanomaterials, graphene has been considered as one of the most promising materials for electrochemical sensing. However, in practical application, the detection limit and selectivity of the graphene are not good, because of their large surface-to-volume ratio leads to lower detection limits [3]. To overcome these obstacles, various materials have been used as matrices for graphene to improve their electrochemical performance. Nanostructured materials including metal oxides have been extensively used for electrochemical sensing applications due to their improved electrocatalytic ability achieved through chemical doping and composite formation [4]. The present study is aimed at the synthesis of the ZnO/GR nanocomposite and its application in the form of the modified screen printed electrode for trace, rapid, and sensitive determination of uric acid through cyclic voltammetric and differential pulse voltammetric techniques. The modified electrode exhibited remarkably anodic peak corresponding to the oxidation of uric acid over the concentration range of 1.0–100.0 μM with detection limit of 0.43 μM (S/N = 3). The fabricated sensor was further

applied to the detection of uric acid in urine samples with good selectivity and high reproducibility.

Keywords: Uric acid, ZnO/Graphene nanocomposite, Graphite screen printed electrode, Voltammetry.

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Application of Graphite Screen Printed Electrode modified with Zinc Oxide Nanoparticle/Graphene for Voltammetric determination of Dopamine

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Abstract

Dopamine is a unique neurotransmitter as it possesses both excitatory and inhibitory classification. The abnormality in the amount of dopamine produced in the central nervous system is a sign of an underlying condition of a neurological disorder in the human body. The lower level of dopamine has been strongly related to illness such as Parkinson's disease, restless leg syndrome, attention deficit hyperactivity disorder (ADHD), schizophrenia, and HIV infection. In the past few decades, various analytical methods have been established and described for the detection of dopamine [1]. Among many techniques, electrochemical method is a more suitable approach in evaluating the low concentration of dopamine under physiological conditions. Lately, SPE have been successfully used as the electrochemical sensor for various researches due to their disability, the simplicity of the apparatus, minimum sample preparation and obtaining of fast results. But, the use of the SPEs for determination of dopamine has been rarely reported, since the unmodified electrodes are unable to discriminate signals of dopamine [2]. Therefore, various inorganic and organic materials have been used to modify these electrodes. Among the nanostructure materials, zinc oxide (ZnO) with many extraordinary properties, including wide direct band gap, large exciton binding energy, nontoxicity, chemical and photochemical stability, high electrochemical activities and easy preparation has been introduced as one of the most promising candidates to modified electrodes [3]. Graphene (GR), which is a flat monolayer of carbon atoms tightly packed into a two dimensional honeycomb lattice, has drawn a great interest due to its unique structure, outstanding electrical, thermal, optical and mechanical properties and high specific

surface area [4]. According to above mention, a sensitive and selective electrochemical sensor based on zinc oxide nanoparticles and graphene nanocomposite (ZnO/GR) for detection of dopamine (DA) has been successfully developed in present work. The electrochemical characteristics and catalytic behavior of prepared electrodes for the determination of dopamine were systematically investigated by cyclic voltammetry (CV) and differential pulse voltammetry (DPV). Under optimum conditions, the ZnO/GR/SPE exhibits linear response to dopamine in the range 0.5-100 μM , with detection limit ($S/N = 3$) calculated to be 0.1 μM . Finally, the developed sensor was successfully applied to the detection of dopamine in dopamine injection and urine samples with satisfactory results. The ZnO/GR nanocomposite show promising applications in the development of electrochemical sensors for chemical or biomolecules determinations.

Keywords: Dopamine, ZnO/Graphene nanocomposite, Graphite screen printed electrode, Voltammetry.

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A new electrochemical sensor based on molecularly imprinted polymer for determination of biotin in drug sample

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Abstract

Biotin, which is an essential vitamin H, helps in the health of skin, nerve and digestive system, and assists in releasing energy and metabolism of fats, protein and carbohydrates. It also helps in the formation of embryonic development. It has wide function and effects on systemic processes and development assistance in immunity. Determination of biotin in foodstuffs is important, because appropriate biotin intake is beneficial in attaining a good quality of life, better health and development of children and adults, improved physical mechanisms that combat aging and disease and efficient mental capacity [1].

The analysis of biotin in foodstuff and tissue and body fluids by high-performance liquid chromatographic (HPLC), luminescence, spectrophotometric, fluorometric has been reported. Due to their excellent rapid response, excellent sensitivity, simplicity, low cost and vivo detection, electrochemical sensors (e.g). voltammetric, potentiometric, conductimetric and capacitance) have obtained wide application in medical, biological and environmental analysis. In the recent years there has been an increasing interest in the modification of electrode's surface with a molecularly imprinted polymer (MIP) to enhance selectivity. Generally, MIPs are synthetic polymers able to selectively recognize a template molecule in an easy and a rapid way. The synthetic procedure is cheap and MIPs are stable under harsh conditions of pH and temperature. Basically, MIPs are prepared by the polymerization of a suitable monomer and a cross-linker agent in the presence of a template. After polymerization, the template is removed and a polymer matrix, which is complementary in shape and functionality to the template, is obtained. Thus, the polymer has the ability to selectively link to the target analyte [2].

In this work, a new and selective electrochemical sensor was introduced by electropolymerization of the functional monomer and molecularly imprinted polymer (MIP) which was synthesized onto a glassy carbon electrode (GCE) in aqueous solution using

cyclic voltammetry in the presence of biotin as template. The imprinting effect was verified by comparing electrochemical response of MIP and non-imprinted polymer (NIP) tested by cyclic voltammetry between -0.6 and 0.8 V in redox peak currents of hexacyanoferrate. As shown in Fig.1. Several important parameters affecting sensor response were optimized and the DPVs of the MIP/GCE for biotin under the optimized experimental conditions are shown in Fig. 2. It can be seen that the peak current decreases as the concentration of biotin increased, which suggested that more and more binding sites in the film are occupied by biotin molecules. Furthermore, they present a fine linear relationship in the range from 1 to 95 μM the regression equation is $I_p = 0.027c + 1.34$ ($R^2 = 0.997$). The detection limit was 0.3 μM ($S/N=3$). The result of this research demonstrates that the new sensor is sensitive, simple to construct and easy to operate. This imprinted electrochemical sensor was used successfully applied to the determination of biotin in real sample.

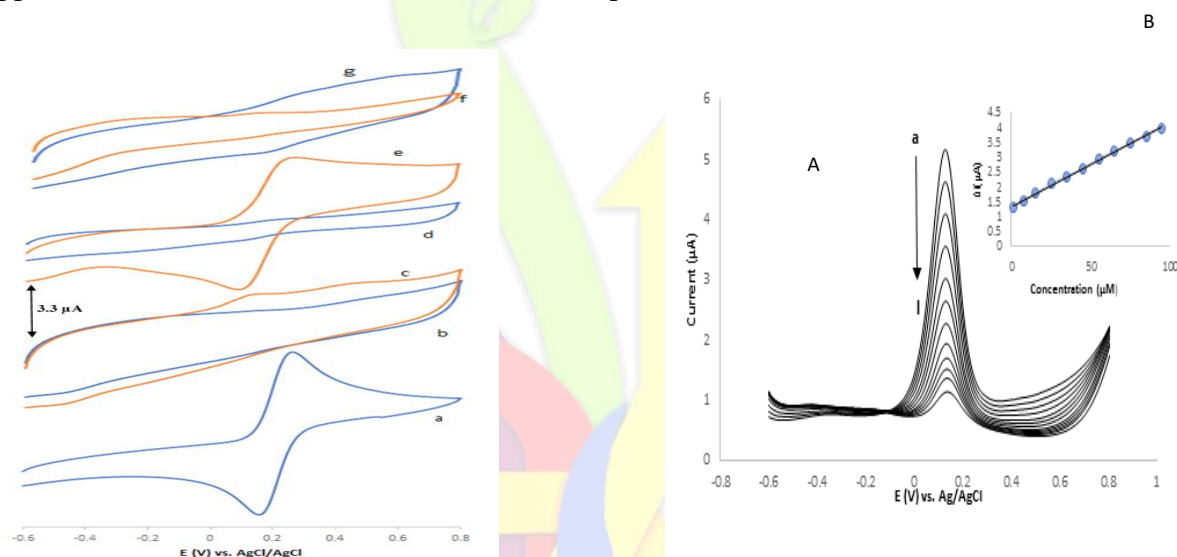


Fig. 2. CV curves (50 mVs^{-1}) of bare GCE (a), NIP/GCE (b), MIP/GCE (c), NIP/GCE after removing process (d), MIP/GCE after removing the template (e), NIP/GCE after loading the template (f) and MIP/GCE after loading the template (g) in electrolyte solution including 0.2M Na_2SO_4 and 2 mM $[\text{Fe}(\text{CN})_6]^{3-}$.

Fig. 2. (A) DPVs of biotin (1 -95 μM) on the MIP/GCE. biotin concentration: 0, 1, 8, 15, 25, 35, 45, 55, 65, 75, 85, 95 μM (from a-l). (B) Related calibration curve. Pulse amplitude = 50mV, pulse width = 50 ms, scan range = -0.6 to 0.8. Electrolytes: 2mM $\text{K}_4\text{Fe}(\text{CN})_6$ and 0.2 M Na_2SO_4 .

Keywords: Molecularly imprinted polymer, Electropolymerization, 1, 4-phenylenediamine, Biotin

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ZIF-8-based microextraction method for determination of fluoxetine in water samples

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Abstract

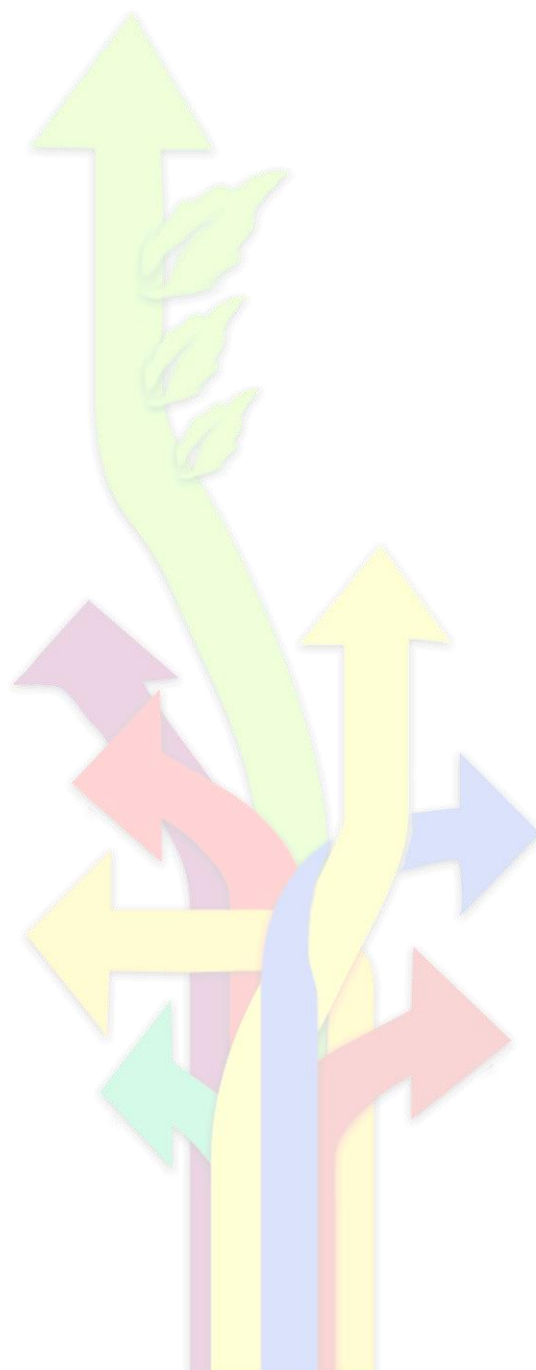
Sample extraction is a major step in environmental analyses due both to the high complexity of matrices and to the low concentration of the target analytes [1]. Sample extraction is usually expensive, laborious, timeconsuming and requires a high amount of organic solvents [2]. Actually, there is a lack of miniaturized methodologies for sample extraction and chiral analyses. Here, we developed a ZIF-8-based thin film microextraction (TFME) to extract the pharmaceuticals fluoxetine as model of basic chiral compounds, from wastewater samples [3]. Compounds were then analysed by enantioselective high-performance liquid chromatography. We monitored the influence of sample pH, extracting and dispersive solvent and respective volumes, salt addition and extracting time. The proposed method gives a very rapid, simple, sensitive and low-cost procedure for the determination of fluoxetine.

Keywords: Thin film microextraction, Electrospeinning, Fluoxetine, ZIF-8

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Bio Electrochemistry



13th Annual Electrochemistry Seminar of Iran

Sensitive I-V Biosensor Using DC Sputtered Nano-ZnO on PVA/FTO Thin Film as an Innovative Composite Transducer for Glucose Biosensing

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Abstract

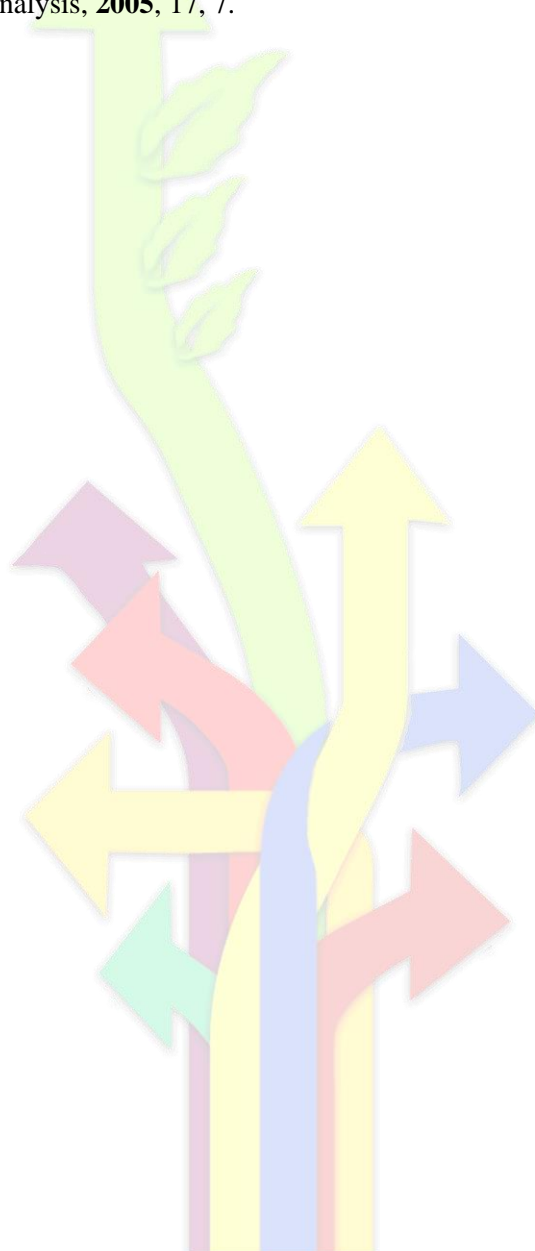
Nanostructured zinc oxide (Nano-ZnO) was sputtered by reactive direct current (DC) magnetron sputtering system on a pre-covered fluorinated-tin oxide (FTO) conductive layer by spin-coated polyvinyl alcohol (PVA) at the optimized instrumental deposition conditions, as an efficient medium for glucose oxidase (GOx) enzyme covalent immobilization via covalent linking to the exposed PVA –OH groups through cyanuric chloride (GOx/Nano-ZnO/PVA/FTO), designed for glucose biosensing by the role of PVA as an electrostatic repulsive layer for the anionic interferents at the biological pH. The structural and electrochemical analyses such as Field emission-scanning electron microscopy (FE-SEM), cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS), and I-V techniques have been applied for morphology assessment of modified electrode's surface and electrochemical behavior of glucose on the fabricated GOx/Nano-ZnO/PVA/FTO biosensor.

The I-V results indicated good sensitivity for glucose detection (0.041 mA per mM) within 0.2 – 20 mM and limit of detection was as 2.0 μ M. We believe that such kind of bio-devices has possessed a prominent potential to trace The number of bio-compounds in biological fluids along with excellent accuracy, selectivity, and precise analysis. A fast response of fabricated GOx/Nano-ZnO/PVA/FTO biosensor (less than 3 seconds (s)), can usually allow a real-time analysis.

Keywords: Covalent GOx immobilization; Direct current magnetron sputtering; Spin coating; Zinc oxide; Polyvinyl alcohol; Disposable glucose biosensor.

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Application of Molecular Imprinted Polymers In The Fabrication of Biosensor And Sensor Since 2015-2017

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Abstract

Molecularly Imprinted Polymers (MIPs) are artificial receptors that mimic natural recognition entities, with high selectivity for a given target analyte [1]. The QR imprinted poly(p-ABA) presented good recognition ability [2]. poly(o-phenylenediamine) film (PPD) was electropolymerized onto the carboxylic-group-functionalized SWCNHs (SWCNHs-COOH)-modified glassy carbon electrode to make electrochemical kanamycin MIP sensor. The resulting sensor has been successfully applied to analyze kanamycin with high sensitivity, selectivity, and recovery [3]. The developed electrochemical sensor exhibits an easy manufacture, a highly sensitive and selective sensing material [4]. Increasing interest has been given to microporous metal-organic frameworks (MMOF) as highly sensitive and selective platforms for the development of sensors [5]. Polydopamine (PDA) has been implemented as an excellent surface-adherent material for multifunctional coatings of membrane surfaces [6]. The synthetic sensor possesses advantages including simplicity, high specificity low cost of preparation, good chemical and mechanical properties, sensitive and label-free determination [7]. combining GO with MIP in the material design will result in higher affinity and exceptional sensitivity to a particular detecting molecule due to its homogeneous distribution of recognition sites [8]. Boronic acid can combine with glucose or fructose in the imprinted polymer matrix according to covalently linkage, under the circumstances, it is a potential method to improve the selective detection of sugar [9]. The prepared GO-MIP based working electrode showed excellent catalytic activity against glucose [10]. In this paper polyvinyl acetate has been chosen for its properties, such as water insolubility, low cost, simple usage and stability at room temperature [11]. Films of PPy and derivatives have good conductivity, selectivity, stability and efficient polymerization at neutral pH [12]. The MIP modified ISFET sensors also

exhibit excellent reproducibility, repeatability and stability, as well as high selectivity to urea [13]. Finally, in this article, we will try to show the hot articles of the MIP application in building the biosensor and sensor in the table.

Table 1. Selected Examples of Functional Monomers Providing Defined Recognizing Sites in Imprinted Molecular Cavities

Menomer	Template	Linear range	LOD	Refs
Para amino benzoic acid	quercetin	0.01 – 0.5 μM	5n M	[2]
o-phenylenediamine	kanamycin	0.1 – 50 μM	0.1 μM	[3]
P-aminothiophenol	tetracycline	22.4 nM – 224 f M	0.22 fM	[4]
P-aminothiophenol	gemcitabine	318 fM – 38 nM	3 fM	[5]
amino thiophenol	cholesterol	10^{-18} – 10^{-13} M	3.3×10^{-19} M	[6]
O - aminophenol	cardiac troponin	0.05 – 5 nM	0.027 nM	[7]
methacrylic acid	cholesterol	0.1 nM – 0.01 M	0.1 nM	[8]
3-hydroxyphenylboronic acid-co-phenol	glucose , fructose	0.75 – 18 mM	0.23 mM , 0.35 mM	[9]
methacrylic acid	glucose	0.01 – 0.08 μM	0.1 nM	[10]
vinylacetate	glucose	0.5 – 4.4 mM	53 μM	[11]
pyrrole	cortisol	1 pM – 10 μM	1 PM L ⁻¹	[12]
Methyl methacrylate	urea	10^{-1} M – 10^{-4} M	10^{-4} M	[13]

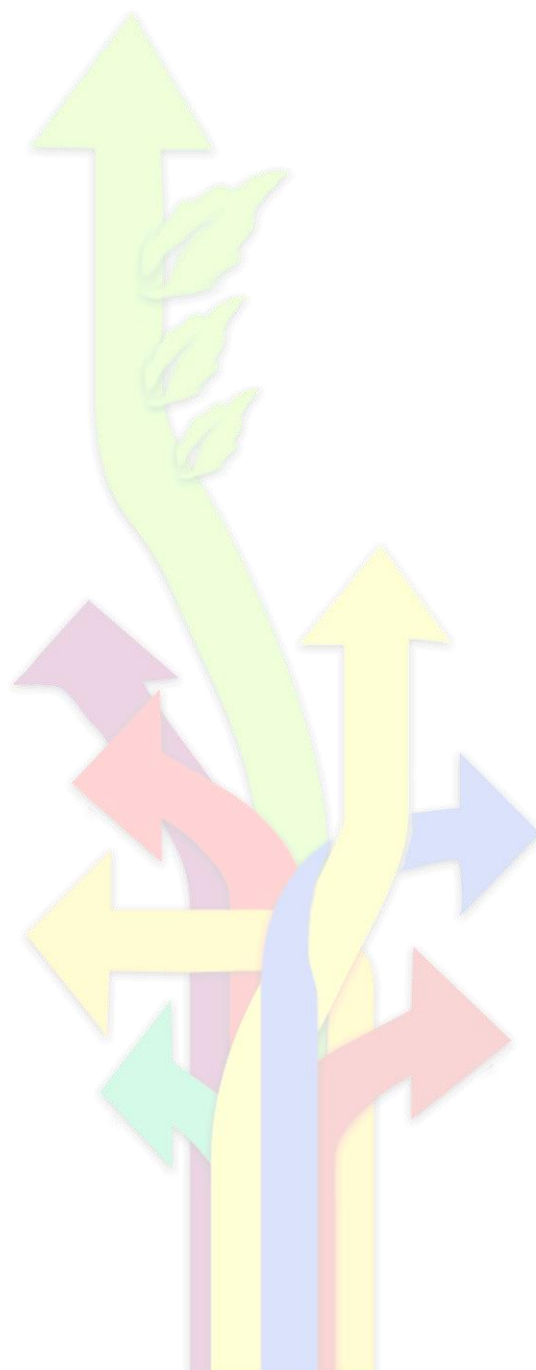
Keywords: Molecularly Imprinted Polymers, Biosensor, sensor, pyrrole

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Fabrication of new amperometric biosensor for determination of Tartrazine

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Abstract

Tartrazine, also known as Acid Yellow 23 or the additive E102 provides a vivid yellow color to the commercial beverages and foods as well as drugs and personal care products [1]. Tartrazine, has a sulfonic group as an auto chrome that makes it highly water soluble and polar. It is reported that Tartrazine is often responsible for allergic reactions, and, it is suspected to be carcinogenic and mutagenic due to its conversion into the aromatic aminesulphanilic acid in metabolic reactions [2].

The enzyme laccase (polyphenoloxidase; EC 1.10.3.2) is a member of the blue multi-copper-oxidase family. These enzymes have been studied for a long time, due to their ability to oxidize a variety of organic substrates, and to reduce molecular oxygen to water [3]. Azo dye degradation by laccases starts by asymmetric cleavage of the azo bond followed by oxidative cleavage, desulfonation, deamination, demethylation and dihydroxylation, depending on dye structure [4].

Monitoring of Tartrazine in the food industry, environmental and bio-medical analyses, by using portable, cost effective devices, has become an area of growing interest over the past decade

A new Laccase amperometric biosensor based on Fe_3O_4 /Graphene Oxide/Chitosan nanocomposite for determination of Tartrazine(TZ) was described. The nanocomposite was electrodeposited on the surface of screen printed carbon electrode (SPCE) by one-step electrodeposition of the nanocomposite at an applied potential of -2 V (vs. Ag/AgCl) for 60 s. For biosensor preparation, modified SPCE was immersed in the solution containing glutaraldehyde (2.5%) as cross-linker for 2 h, enabling the formation of a covalent bond between the aldehyde group of glutaraldehyde and the amine group of the protein (Laccase). Filed effect Scanning electron microscopy (FE-SEM), Fourier transform infrared (FTIR)

spectrophotometry, cyclic voltammetry (CV), and electrochemical impedance spectroscopy (EIS) were used for characterization of the electrode surface.

The biosensor was optimized with respect to biocomposite composition, enzyme loading, and solution pH by amperometry method the applied potential of -0.25 V (vs. Ag/AgCl). The biosensor exhibited noticeable electrocatalytic ability toward tartrazine with a linear concentration range from 0.03 to 10 $\mu\text{g/L}$ and a detection limit of 10 ng /L. The fabricated biosensor was successfully applied for measurement of Tartrazine in soft drinks.

Keywords: *Biosensor, Laccase, Tartrazine*

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Development of an electrochemical DNA biosensor for detection of breast cancer gene

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Abstract

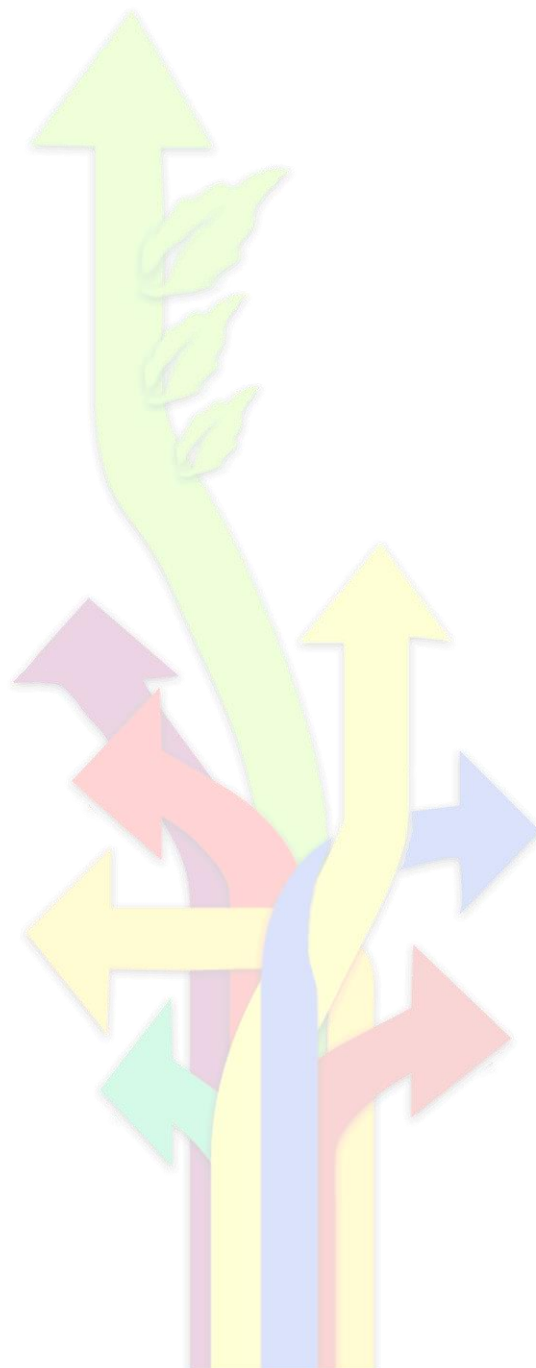
Breast cancer is one of the leading causes of death in women worldwide [1]. Several inherited gene mutations have been linked to breast cancer, mostly associated to the tumor suppressor gene BRCA1, which is known as a kind of anti-oncogene and one of the most important breast cancer susceptibility gene [2]. Hence, the detection of BRCA1 offers an opportunity to characterize the function of genetic features in breast cancer as well as to screen breast cancer patients for the presence of germ line mutations [3]. An impedimetric BRCA1 gene biosensor has been developed based on carbon nanotube-Nafion composite film. The resultant nanocomposite can provide a large surface area, excellent electrocatalytic activity, and high stability, which would improve immobilization sites for biological molecules, allow remarkable amplification of the electrochemical signal and contribute to improved sensitivity. The biosensor was fabricated by adsorbing the single-stranded DNA (ssDNA) on carbon nanotube-Nafion modified on the surface of glassy carbon electrode via the π - π^* stacking interactions. As the negative ssDNA and the steric hindrance, the electron transfer resistance of the electrodes toward the $[\text{Fe}(\text{CN})_6]^{3-/4-}$ redox couple was difficult, the electron transfer resistance value increased. In the measurement of BRCA1 gene, ssDNA probe with the target DNA to form double-stranded DNA (dsDNA), the formation of helix induced increase in the electron transfer resistance that was in logarithmically direct proportion to the concentration of BRCA1 gene over a range from 1.0×10^{-15} to 1.0×10^{-12} M. The detection limit of this sensor was 7.0×10^{-16} M. The results demonstrate that this carbon nanotube-Nafion biosensor possesses good selectivity, acceptable stability and reproducibility for BRCA1 gene detection.

Keywords: Biosensor; Breast cancer; Electrochemical impedance spectroscopy; Voltammetry

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13th Annual Electrochemistry Seminar of Iran

Preparation of an electrochemical biosensor for the study of DNA interaction with Flutamide using hemin

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Abstract

Flutamide is the first discovered compound that meets the criteria of a pure antiandrogen[1]. It is of particular interest to investigate the effect of this drug on the DNA structure as it is widely used for the treatment of prostate cancer nowadays [2, 3]. Development of DNA biosensors for the understanding of DNA interactions with molecules has been raised in recent years [4].

An improved performance for DNA biosensors could be achieved by modifying the electrode surface using different nano materials which lead to an increase in electrochemical signal. One of these candidates is mesoporous silica material that showed good aspect for surface modification in fabrication of DNA biosensors. MCM which was discovered in 1992 [5, 6] is a promising candidate for the immobilization of various bio and nano materials and can be used to develop stable biosensing systems owing to their unique properties such as high surface area, porosity, uniform pore size distributions, thermal stability and also the high stickness stability after immobilization on the electrode surface which lead to a stable and steady response.

In this study, the electrochemical biosensor was prepared by modifying screen-printed graphite electrode (SPE) with mesoporous structure of MCM41 to immobilize DNA structure. The mesoporous structure introduces a significant surface area and can increase the amount of adsorbed DNA on the surface. The interaction of Flu as the anticancer drug with ssDNA and dsDNA was studied in Tris-HCl buffer and also in the presence of Hemin (HEM) as a suitable electroactive redox label. The differential pulse voltammetry (DPV) current of HEM reduction peak decreases with the increasing concentrations of drug due to the interaction of DNA/Flu. The peak current of HEM linearly decreased with the

concentration of Flu in the range 0.7 to 10 μ M ($R^2 = 0.9907$), with a detection limit of 0.1 μ M.

Keywords: DNA, Electrochemical biosensor, Mesoporous structure, Flutamide.

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Designing a new glucose biosensor based on glucose oxidase-immobilized at biocompatible electrospun nanofiber membrane

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Abstract

Glucose biosensor technology including point-of-care devices, continuous glucose monitoring systems and noninvasive glucose monitoring systems has been significantly improved. However, there continues to be several challenges related to the achievement of accurate and reliable glucose monitoring. In this work, we have developed a polyvinyl alcohol/starch/ graphene biocompatible electrospun nanofiber membrane, to measure the glucose level. The formation of polyvinyl alcohol/starch/ graphene nano-fibre was confirmed by scanning electron microscopy, X-ray diffraction, UV-vis and FTIR spectroscopy methods. Glucose oxidase (GOx) was successfully immobilized on modified polyvinyl alcohol/starch/graphene nanofibre membrane and direct electron transfer of GOx was investigated. The amount of electroactive GOx and electron transfer rate constant were also found. The comparison of the biosensor performance with reported sensors reveals the significant improvement in overall sensor performance. Moreover, the biosensor exhibited appreciable stability, repeatability, reproducibility and practicality.

Keywords: polyvinyl alcohol/starch/graphene, electrospun nanofiber membrane, Glucose oxidase, biosensor

Electrochemical removal of Cefixime antibiotic from aqueous solution

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Abstract

the presence of antibiotic compounds in surface waters is an emerging environmental issue. Pharmaceuticals industries, health attention centers (especially hospitals) or simple civil buildings represent important points of antibiotic discharge into the environment and produce a non negligible effect on the physical, chemical and biological composition of receptor water bodies [1]. In recent years, several wastewater treatment methods have been developed to remove toxic and biorefractory organic pollutants from wastewater [2, 3]. Among these methods, a great attention has been paid to the advanced oxidation processes (AOPs). These methods are based on the in situ production of highly reactive species, the hydroxyl radicals ($\cdot\text{OH}$), which react unselectively with organic matter unto its total or quasi-total mineralization [4]. Among AOPs, electrochemical advanced oxidation processes (EAOPs) have received an increasing attention because of their efficiency in the destruction of toxic/persistent organic pollutants, environmental safety and computability and versatility [5]. One of the most popular EAOPs is the electro-Fenton process in which $\cdot\text{OH}$ are produced electrocatalytically through the Fenton reaction (1);



In this study, the Cefixime antibiotic removal, from aqueous solutions, was studied using Electro-Fenton process. Cefixime is an antibiotic useful to treat a number of bacterial infections [6]. This includes otitis media, strep throat, pneumonia, urinary tract infections, gonorrhea, and Lyme disease [6].

The electro-Fenton experiments were carried out at room temperature, in an open electrolytic cell containing a graphite cathode and an Iron anode of 10 cm^2 surface area. The electrolysis of Cefixime solutions were performed with a MEGATEK MP-3003D triple power supply at constant current density, in a non-divided cylindrical glass cell of 100 mL capacity. Homogenization of the electrolysis solution was ensured with a magnetic stirring. Aqueous solutions containing 45 ppm Cefixime and 20 ppm Na_2SO_4 were used for different trials.

Absorbance of drug solutions before electrolysis, and at different time during electrolysis, was determined using a uv-vis spectrophotometer.

Effective factors on drug removal efficiency, such as H_2O_2 concentration, pH and current density, were optimized. The results showed that in optimized condition (11mM H_2O_2 , pH 4 and current density of 10 mAcm^{-2}), the percentage of drug removal after 1 hour electrolysis was 95%. Compared to the methods, reported in the literatures, the proposed method has the potential to remove higher concentrations of the drug.

Keywords: *Cefixime Removal, Electro-Fenton, Electrolysis, Current Density.*

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The effect of bioactive glass 58S on pH variations and the ions release in the simulated body fluid medium

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Abstract

This study evaluated the effect powder of bioactive glass 58S that prepared from sol-gel method, on pH variations in the simulated fluid body (SBF) and the ions release of calcium, phosphorus and silicon. Powder with size less than 45 micron then they were immersed in SBF at 37°C for 10 minutes and 1, 2, 3, 4, 6, 8, 10, 12, 14, 16, 18, 20, 22, 24, 26 and 28 day. After removing particles and mixing SBF, the pH variations were measured with a normal glass pH electrode. The quantification of ions in the solutions for different soaking times were analyzed via optical emission spectroscopy. The results show that after 28 days, pH is alkaline and the formation of apatite on the surface of the 58S powder is similar to the silicate glass standards. The ICP test introduces the release rate of the elements and the probability of forming the HA layer, thus the glass has potential for bone repair and remodeling applications.

Keywords: Bioactive glass 58S; bioactivity; ions release; pH; ICP-AES.

Evaluation of pH variations in the interaction of different amounts of gelatin and sodium alginate in aqueous medium.

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Abstract

Complex coacervate of biopolymers, polysaccharide and protein are produced by electrostatic attraction between oppositely charged in both polymers. Properties of complex strongly depend on the molecular weight, molecular shape, surface charge, ionic strength, temperature, concentration, pH, and interactions between them. In order to investigate the behavior of gelatin and alginate complex in aqueous media, pH variations of these compounds at five different concentrations of 0.98 2.0 3.5, 5.0 and 6.02% (w/v) of gelatin, with 12 different concentrations of 0.01, 0.05, 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9 and 1.0% (w/v) of sodium alginate, at 50 ± 1 °C. The results showed that at constant concentrations of sodium alginate, pH decreased with increasing gelatin, and unlike, in constant concentrations of gelatin, pH increased with increasing sodium alginate. Since all amino acids in the highly acidic pH have a positive charge due to the protonation of the groups (COO^-) and (NH_2), and in all of these compounds, pH were acidic and proteins often have a negative charge in above the isoelectric point ($\text{pI} \approx 5$). Therefore, at the pH values below the isoelectric point, they can form electrostatic interaction with polysaccharide with negative charge. So, in order to optimal interaction between two biopolymers, we need to add acid to the reaction medium and determine the best ratio of the compound.

Keywords: Biopolymer; gelatin; sodium alginate; pH; Iso-electric point.

A Review of Current Trends and Development and application of electrochemical biosensor

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Abstract

The biosensors are based is, in general, common to chemical sensors. They may be also differentiated according to the biological elements used in the receptor. Those may be: organisms, tissues, cells, organelles, membranes, enzymes, antibodies, etc. The biosensors may have several enzymatic systems coupled which serve for amplification of the signal [1]. A chemical sensor is a device that transforms chemical information, ranging from the concentration of a specific sample component to total composition analysis, into an analytically useful signal. The chemical information, mentioned above, may originate from a chemical reaction of the analyte or from a physical property of the system investigated [2]. Electrochemistry provides a broad array of quantitative methods for detecting important analytes (e.g., proteins, nucleic acids, metabolites, metals) for personal and public health, clinical analysis, food and water quality, and environmental monitoring [3]. This statement is justified due to some of its advantageous features, such as high sensitivity and selectivity, moderate cost, portability, rapid responses, possibility of system miniaturization, and analysis of complex matrices [4,5]. This paper describes development of biosensor based sensing system that relies on electrochemical detection reporting an in the period 1948 till 2017 years.

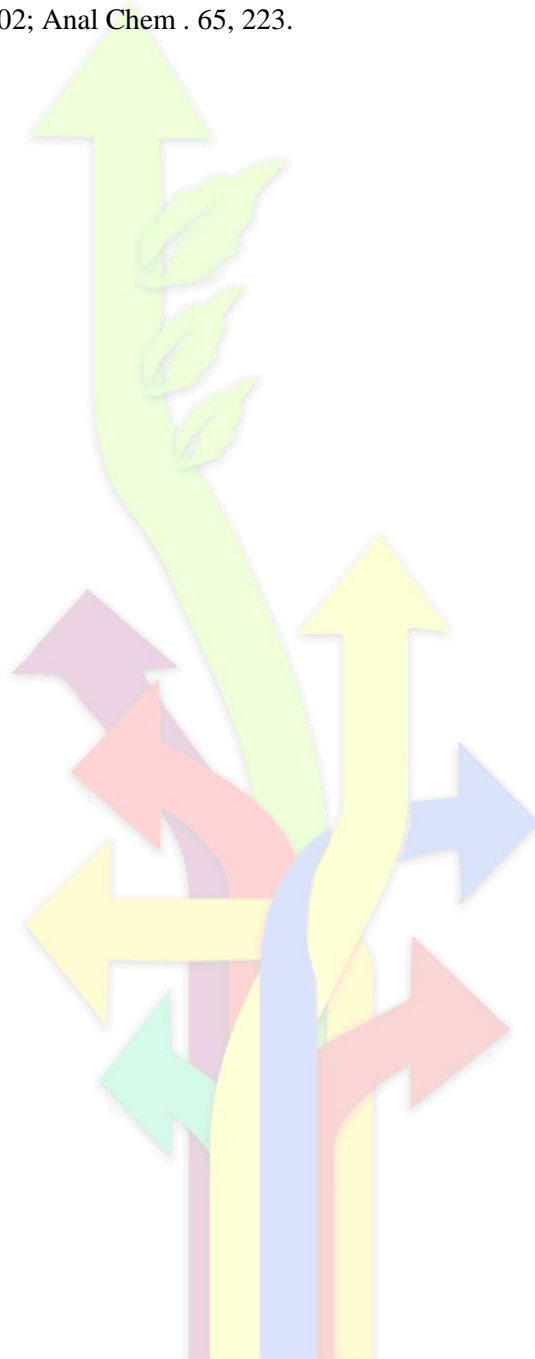
Keywords: *ELECTROCHEMICAL, BIOSENSOR*

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13th Annual Electrochemistry Seminar of Iran

A facile electrochemiluminescence glucose biosensor based on $\text{GO}_x/\text{Ni}(\text{OH})_2/\text{Ni}$ foam modified electrode

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Abstract

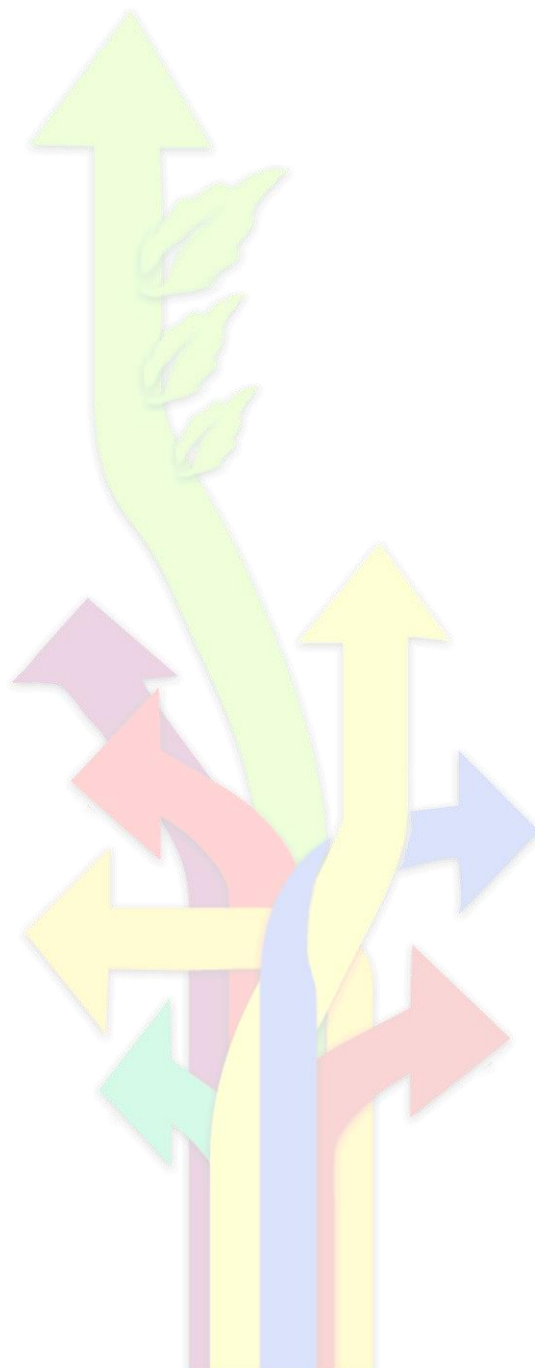
Selectivity and sensitivity are two important parameters for glucose sensing in clinical and food applications. Glucose oxidase (GO_x) which is used for the enzymatic glucose detection, plays a significant role in the selectivity of the biosensor, due to its specific interaction with glucose. The enzymatic glucose biosensors have been developed in three generations during three decades [1-3]. In this study, a new electrochemiluminescence (ECL) glucose biosensor based on immobilization of glucose oxidase into the nickel foam modified electrode was fabricated. Nickel hydroxide was formed on the surface of 3-D Ni foam by a successive scan of cyclic voltammetry in an alkaline solution. The structural characterization of the modified electrode was carried out by electrochemical impedance spectroscopy (EIS), scanning electron microscopy (SEM) and energy-dispersive X-ray analysis (EDX). The electrochemiluminescence method was used for determination of glucose in the aqueous solutions. The fabricated ECL biosensor, $\text{GO}_x/\text{Ni}(\text{OH})_2/\text{Ni}$ -foam electrode, exhibited excellent electrocatalytic properties toward the detection of glucose in 0.1 M phosphate buffer solution (pH 7.4). The linear range and limit of detection were evaluated to be 2.7×10^{-9} to 4.5×10^{-3} M, and 2.0×10^{-9} M ($S/N = 3$) respectively. The present ECL biosensor exhibited good reproducibility, stability and has been used for determination of glucose concentrations in blood serum samples in the presence of the interference species with the satisfactory results.

Keywords: Glucose oxidase, Nickel hydroxide, Ni foam, Electrochemiluminescence

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13th Annual Electrochemistry Seminar of Iran

Adsorptive potentiometric stripping analysis of anticancer drug at different electrodes and different electrolytes.

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Abstract

In this work, effect of different electrodes such as glassy carbon (GC), Au, Pt, MWCNT past and MWCNT-GC electrodes and different electrolytes such as HNO₃, CH₃PO₄, CH₃COOH and B-R buffer on the electrochemical behavior of Tamoxifen (Tam) as breast anticancer drug were investigated by differential-pulse anodic adsorptive stripping (DPAAS) voltammetric techniques. It was observed that at GCE and H₂SO₄ as electrolyte, electrocatalytic behavior for the oxidation of Tam was excellent. It was evidenced by the enhancement of oxidation peak current and shift in the oxidation potential to less positive values. Cyclic voltammetry (CV) and chronoamperometry were used to understand the electrochemical characteristics of Tam. A chronoamperograms gave fundamental electrochemical parameter including the electroactive surface coverage (Γ), the diffusion coefficient (D) and the heterogeneous rate constant (k_s). Based on the results of the recorded CV, the electrodeposition and anodic stripping behavior of the Tam were investigated at the surface of GCE. The primary experiments demonstrated that the DPAASV presents a sufficient oxidation peak current at approximately 1.03 V vs Ag/AgCl. Therefore, to find the best conditions for taking a sharp analytical peak concerning the electro-oxidation of Tam, the effects of different factors such as scan rate, deposition potential and deposition time on anodic peak have been studied and optimized. The calibration curve showed linearity in the

range of 0.5 to 80 μM and the limits of detection (LOD) and quantitation (LOQ) were calculated to be 0.12 and 0.4 μM , respectively. The mean, standard error and relative standard deviation (RSD) for 4 replicates of 15 μM were found to be 15.57 μM , 3% and 4%, respectively. Finally, it has been successfully applied to the determination of Tam in different spiked physiological samples.

Keywords: *Tamoxifen, DPAASV, Stripping voltammetry, deposition*

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Voltammetric determination of epinephrine using modified carbon paste electrode

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Abstract

Epinephrine (EP), is a very important catecholamine neurotransmitter in the central nervous system. It exists as an organic cation in the nervous tissue and biological body fluid. Many diseases are ascribed to changes of its concentration [1]. EP has, therefore, been attracted tremendous consideration in biomedically-oriented research. Hence, it is very necessary to develop sensitive, selective, and reliable methods for the direct determination of trace EP due to its physiological function and the diagnosis of some diseases in clinical medicine. Therefore, it is very important to develop sensitive and selective analytical methods for the detection of EP in biological fluids [2].

EP is an electroactive compound that can be determined by different techniques. In recent years, the development of voltammetry methods for its determination in human body fluids such as urine and serum has received considerable interest. Carbon-based electrodes are among the most commonly used electrodes in voltammetric analysis because of their low cost, wide potential windows, low electrical resistances, and versatility of chemical modification [3]. In the present work, we describe the preparation of a new carbon paste electrode modified with ionic liquid and magnetic core-shell manganese ferrite nanoparticles (MCSILCPE) and investigate its performance for the determination of epinephrine. The morphology of the modified electrode was studied using SEM. The electrochemical characterization of the modified electrodes was carried out by cyclic voltammetry (CV), chronoamperometry (CHA) and square wave voltammetry (SWV). SWV exhibits a linear response from 1.0×10^{-7} to 1.0×10^{-4} M epinephrine. The limit of detection was obtained 2.4×10^{-8} M. The modified electrode exhibited excellent accuracy and precision, the relative

standard deviation was less than 4%. Finally, the modified electrode was successfully used to accurately detect the content of EP in in real samples.

Keywords: Epinephrine; Magnetic core-shell manganese ferrite nanoparticles; Ionic liquids; Chemical modified electrodes

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Modified screen-printed electrodes for determination of imipramine

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Abstract

Imipramine (Imi) is a tricyclic antidepressant drug, which is the most effective drug presently available for the treatment of depression and other psychiatric disorders by blocking the reuptake of norepinephrine at nerve terminals [1]. To enable its clinical applications to achieve optimum therapeutic effects and minimize side effects, analytical method with high sensitivity and free from interference is needed [2]. Many methods for the determination of Imi have been developed using techniques such as high performance liquid chromatography, gas chromatography, spectrophotometry, electrochemical analysis, chemometrics and capillary electrophoresis. Electrochemical methods are more desirable than other techniques because they are convenient and low cost.

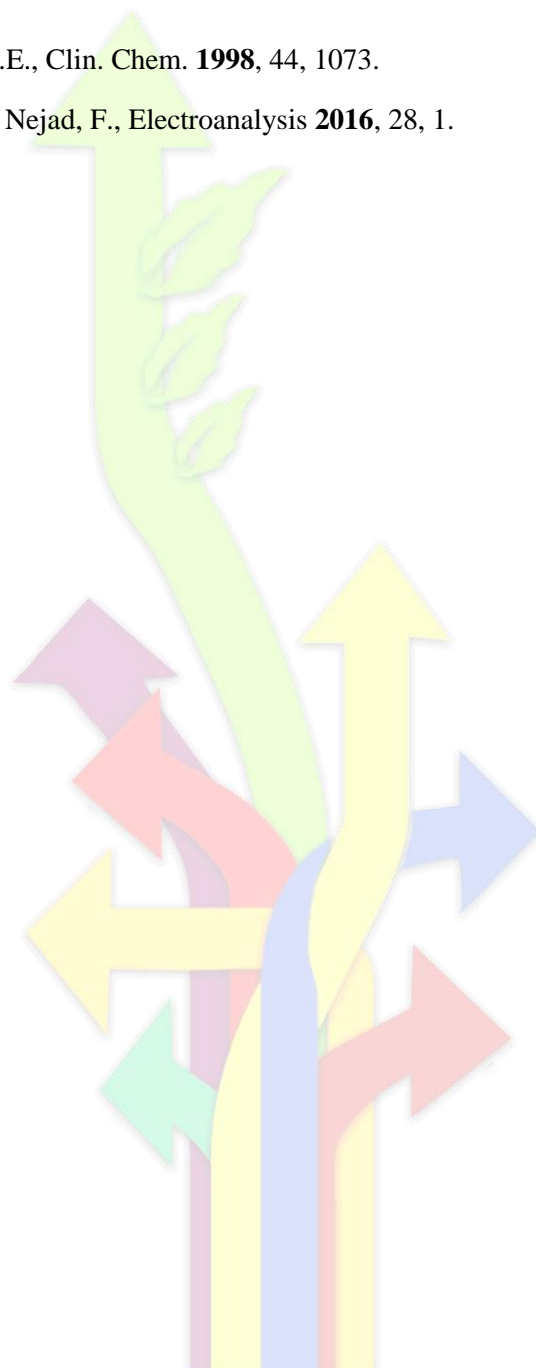
The screen-printed electrodes (SPEs) have been designed especially for miniaturization of electrochemical analytical systems [3]. SPEs are highly-versatile, easy to use, cost-effective analytical tools, also suitable to miniaturization. Furthermore, a screen printed electrode avoids the cleaning process, unlike conventional electrodes such as a glassy carbon electrode (GCE). In order to improve their electrochemical performance, SPEs have been modified with nano sized materials. The modified electrode has good electro catalytic activity, sensitivity, and selectivity; it has also a low detection limit compared to unmodified electrodes.

In the present work, we synthesized magnetic core-shell manganese ferrite nanoparticles (MCSNP) and screen printed electrodes were modified with MCSNP. The electrochemical characterization of the modified electrodes was carried out by cyclic voltammetry, chronoamperometry and square wave voltammetry. Under optimized conditions, square wave voltammetry exhibited linear dynamic ranges from 1.0×10^{-6} – 2.0×10^{-4} M with detection limit of 1.8×10^{-7} M. The modified electrode exhibited excellent accuracy and precision, the relative standard deviation was less than 4%. Finally, the modified electrode was successfully used to accurately detect the content of Imi in real samples.

Keywords: *Imipramine; Magnetic core-shell manganese ferrite nanoparticles; Screen-printed electrodes; Chemical modified electrodes.*

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13th Annual Electrochemistry Seminar of Iran

Pseudo-chloride peroxidase: Iron-Porphyrin/Cystein/PEG structural view-point

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Abstract

Enzyme mimics are designed mainly for practical applications as artificial enzymes and also for understanding enzymatic mechanisms [1]. The importance of nano-structures on dynamic functions, including catalysts, is now well recognized. In this approach, many attempts have been made to improve and enhance the activity of these pseudo-enzymes by hydrophobic pocket or scaffold. The encapsulation of metallo-porphyrin active-site into polymer scaffold can be classified in colloid chemistry as part of interfaces in solution. The solution consisted of the porphyrin-metal-cysteine complex and polyethylene glycol (PEG) (as a scaffold) has used to model the chloride peroxidase (CLP) by 28% of native enzyme efficiency. Dynamic light scattering indicated that the designed nanozyme has vesicle-like size-structure and zeta potential indicate negative charge of PEG oxygen atoms, laid on external edge of nano-particles.

Also TEM images confirmed the multi-cavity vesicular nanozyme.

Keywords: Iron-Porphyrin/Cystein, PEG, chloride peroxidase, TEM, DLS

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Potentiometric Determination of Aluminium Ions Using Carbon Paste Electrode by Benzo 15 –crown – 5 as a modifier

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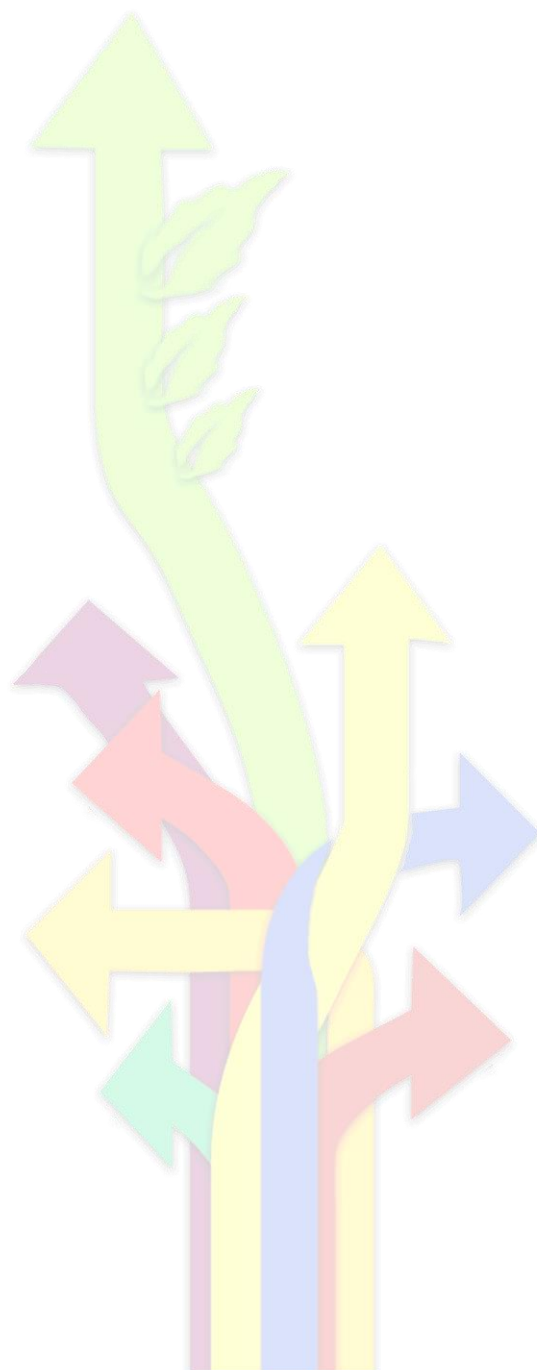
Abstract

Carbon paste is an ideal electrode substrate due to its; chemical inertness, low cost, fast response time, ease of fabrication in different configuration and size and renewal. These attractive features of carbon electrodes are the reason for the considerable attention at the production of carbon based electro analytical sensors. Recently, carbon paste technique was used in the construction of amperometric enzyme electrode, urea and potentiometric determination of drug. a new modified carbon paste electrode based on benzo 15 – crown – 5 as a modifier was prepared for the determination of Aluminium ions. The best results were obtained from the composite sensor with the electrode composition of 1.81% modifier, 23.99% paraffin oil, 1.48% sodium tetra phenyl borate and 73.88% graphite powder. The proposed electrode shows a Nerstian slope 19.54 mV in the concentration of 1×10^{-7} to 1×10^{-3} mol/L of Aluminium ions with detection limit of 5×10^{-7} M. Response of electrode in the pH is fixed between 5 and 8. The sensor exhibits good selectivity for Aluminium ions over a wide variety of other cations. It can be used as an indicator electrode in potentiometric titration of Aluminium ions. The electrode has a response time of about 40 seconds and can be used 21 days without a serious change in sensitivity.

Keywords: Aluminium, Potentiometric, Benzo-15-crown-5, Ion selective electrode

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Development of a new molecularly imprinted polymer (MIP) - based electrochemical sensor for determination of phenylephrine hydrochloride in drug sample

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Abstract

A molecularly imprinted polymer (MIP) for the determination of Phenylephrine hydrochloride (PHE) was present for the first time. molecular imprinting technology has become a well-established analytical equipment, which has been widely applied for the preparation of polymeric materials that have the ability to specifically bind species [1, 2]. Molecular imprinting involves positioning functional monomers around the target molecules (template) by covalent interaction or non-covalent interaction, followed by polymerization [3]. After removing the template, molecularly imprinted polymers (MIPs) were obtained. MIPs possess surface cavities complementary to the template. The imprinting effect was verified by comparing electrochemical response of MIP and non-imprinted polymer (NIP) tested by cyclic voltammetry between -0.6 and 0.8 V in redox peak currents of hexacyanoferrate. As shown in Fig.1. A couple of typical redox peaks of $[\text{Fe}(\text{CN})_6]^{3-}$ appeared at bare GCE (curve a). (curves b and c), when the NIP and MIP was deposited on the surface of the electrode, the peak current was not observed. This may be due to the fact that the synthesized polymer film caused to create a protective layer on the surface of the electrode. The $\text{K}_3[\text{Fe}(\text{CN})_6]$ could not pass through the layer of polymer to arrive at the surface of the electrode for electrochemical processes. When the template removal process was done on both NIP and MIP electrode, no notable changes were observed in NIP electrode behavior (curve d), but in the MIP, the redox current of $\text{K}_3[\text{Fe}(\text{CN})_6]$ observed (curve e). This well illustrates the fact that removal of the template and the formation of recognition sites or binding cavity made electron transmission possible and $\text{K}_3[\text{Fe}(\text{CN})_6]$ could pass through the cavity in MIP and reach the surface of the electrode more easily for electrochemical process. When the electrodes were loaded by the template again, but in the case of NIP, there was no change in the behavior of the electrode in a $\text{K}_3[\text{Fe}(\text{CN})_6]$ solution (curve f). But the MIP, the peaks of $\text{K}_3[\text{Fe}(\text{CN})_6]$ disappear again (curve g). It can be ascribed

that after loading with template, the cavity in MIP film occupied by template and the process of the probe to the surface of the electrode was stopped. It is also noted that compared with the bare GCE (curve a), an obvious current increased appeared when using the MIP/GCE after the template removal (curve e) in the presence of $[\text{Fe}(\text{CN})_6]^{3-}$. This may result from the high conductivity of the film coated on GCE. Some parameters affecting sensor response were optimized and then a calibration curve plotted. A dynamic linear range of 5 to 110 μM was obtained. The detection limit was 0.9 μM ($S/N=3$). This imprinted electrochemical sensor was used successfully for PHE determination in real samples.

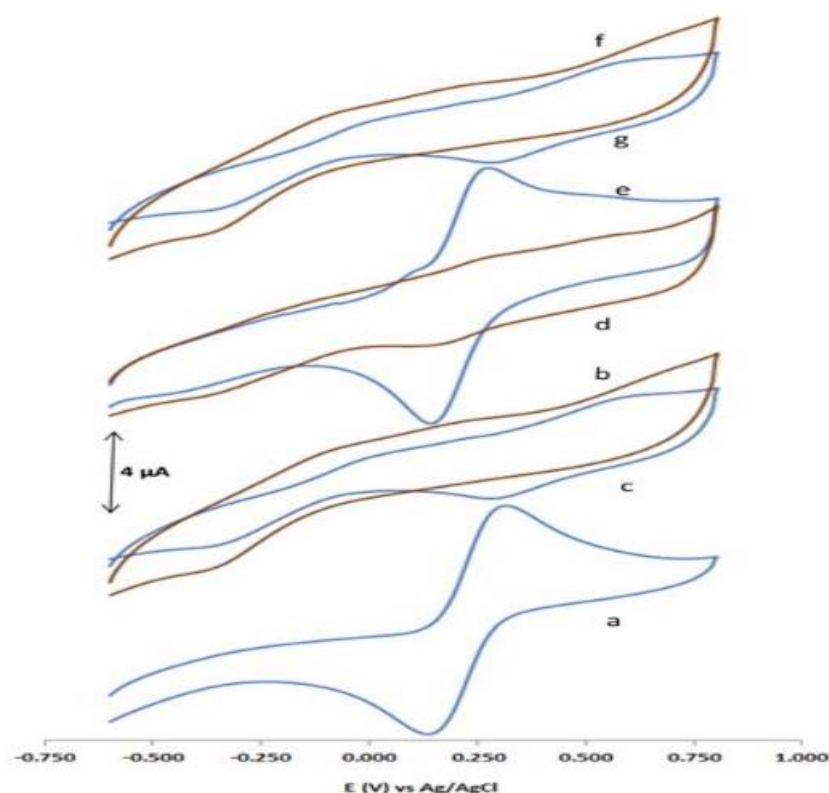
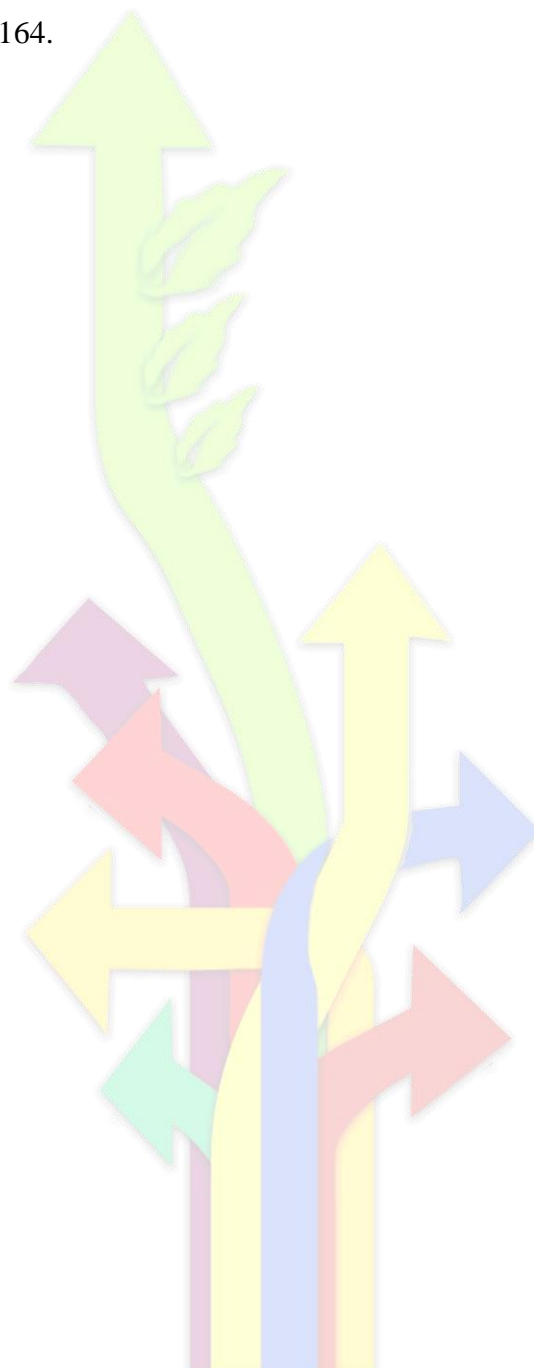


Fig. 1. Electrochemical response of MIP and none imprinted polymer (NIP) tested by cyclic voltammetry between -0.6 and 0.8 V in redox peak currents of hexacyanoferrate

Keywords: Molecularly imprinted polymer, Electropolymerization, Phenylephrine hydrochloride, 1, 4-phenylenediamine

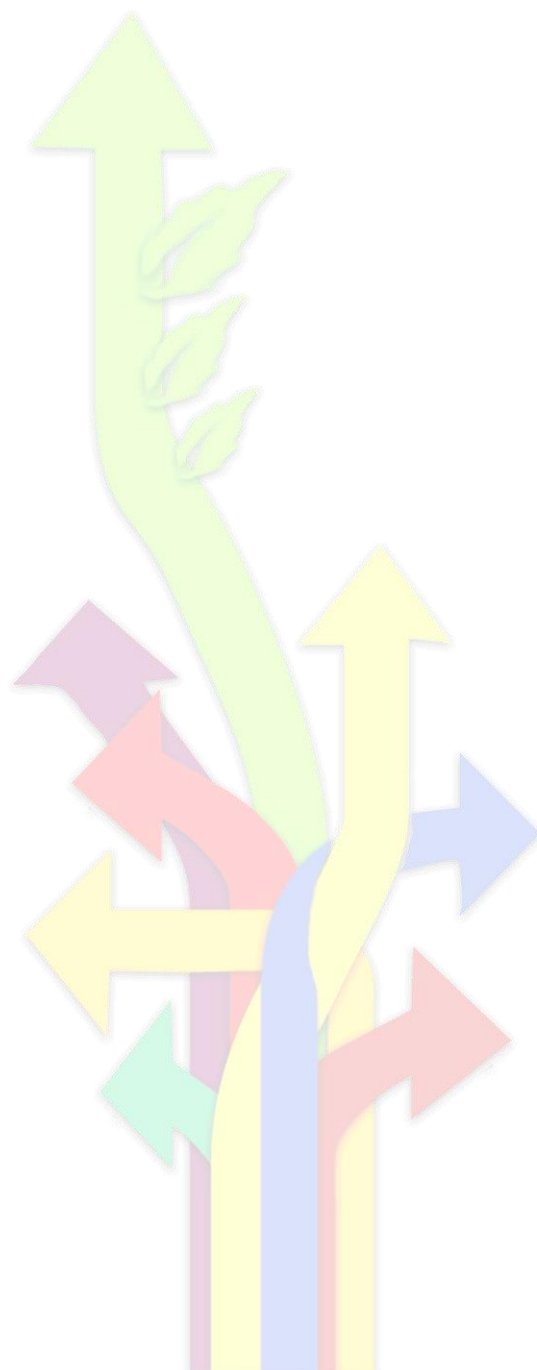
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13th Annual Electrochemistry Seminar of Iran

Environmental Electrochemistry



13th Annual Electrochemistry Seminar of Iran

Environmental Electrochemistry

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Abstract

Different industries in the world develop every day. However, these developments induce better and easier life; they are main suspected pollution increasing in the world. Air, water and soil are polluted by waste materials and these contaminants are comminatory for human lives. Most of these hazardous materials can be changed to less pollutant or no contaminant substances by use of electrochemical methods. On the other hands electrochemical process has been known as green process.

In this theoretical research, the electrochemical methods that have been used for wastewater treatment, remediation of soil and recovery of catalysts, will be introduced. Besides them, the ability of electrochemical sensors to detect and determination of pollutant has been discussed.

Keywords: *Electrochemistry, Environment, Wastewater, Electrokinetic remediation, Catalyst recovery*

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Investigation of electrochemical removal of pyrrole in aqueous media

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Abstract

Electrochemical removal of pyrrole performed in a divided cell utilizing a proton-exchange membrane in aqueous media by different electrolytes. Experiments were done at three different electrolytes (0.5 M H₂SO₄, 1 M NaOH, 1 M KOH). The electrochemical methods were carried out using CV and CA methods. The results of this study showed that removal of pyrrole in acidic electrolyte is proper than basic electrolyte. Also large peak at 0.85 V revealed the oxidation potential of pyrrole in acidic medium. It revealed that 30% of pyrrole was removed after 8 hr.

Keywords: Pt electrode, Electrochemical denitrogenation, Nitrogen compounds, Pyrrole

Introduction

Among various global environmental pollutions, recalcitrant pollutants have drawn extensive attention in the last [1]. The presence of coexisting nitrogen compounds in the fuel makes the removal of sulfur, in particular, refractory sulfur compounds, extremely difficult [2]. Pyrrole is a typical heterogeneous compound which is most frequently used as an intermediate and solvent in the synthesis of various pharmaceuticals, insecticides, pesticides, cosmetics, agrochemicals, and disinfectants [3]. A significant portion of papers has been devoted to the degradation of nitrogen-containing organic compounds based on physical and biological methods [1]. The physical means realize nitrogen-containing organic compounds removals with only transferring them to another phase, which may cause secondary pollution [4]. Right now the Conventional method of removal of nitrogen and sulfur are hydrodesulfurization (HDS) and Hydrodenitrogenation (HDN) Which are part of high-energy processes. The electrochemical method has more advantages than conventional and old methods, because of low operating temperature and low pressure, high selectivity Product and low energy consumption.

Experimental

Cell, materials, and electrodes

A potentiostat (the SP-150), was used for electronic control and data acquisition. A divided batch cell utilizing a proton-exchange membrane was used. The reference electrode was Ag/AgCl/KCl Sat' and all the potentials reported in this paper are referenced to this electrode. The working electrode used for voltammetry measurements of pyrrole adsorption on platinum electrodes was a 2 mm diameter platinum disk. All the used reagents were highly pure (Merck Millipore).

Experimental procedures

For investigation of the removal of the pyrrole in different electrolytes, CA methods were applied at oxidation and reduction voltage peak. For this purpose, at first, the reference voltammogram was taken from desired electrolytes. The potentials were applied to the electrode with a solution of 500 ppm pyrrole in the desired electrolyte for 10 min. Then, for comparison with the initial state, the voltammogram of the platinum electrode was recorded in the pyrrole-free electrolyte.

Another experiment was applying potentials to the electrode for longer times. The pyrrole removal was tracked and analyzed in electrolyte at different times by UV-visible spectroscopy.

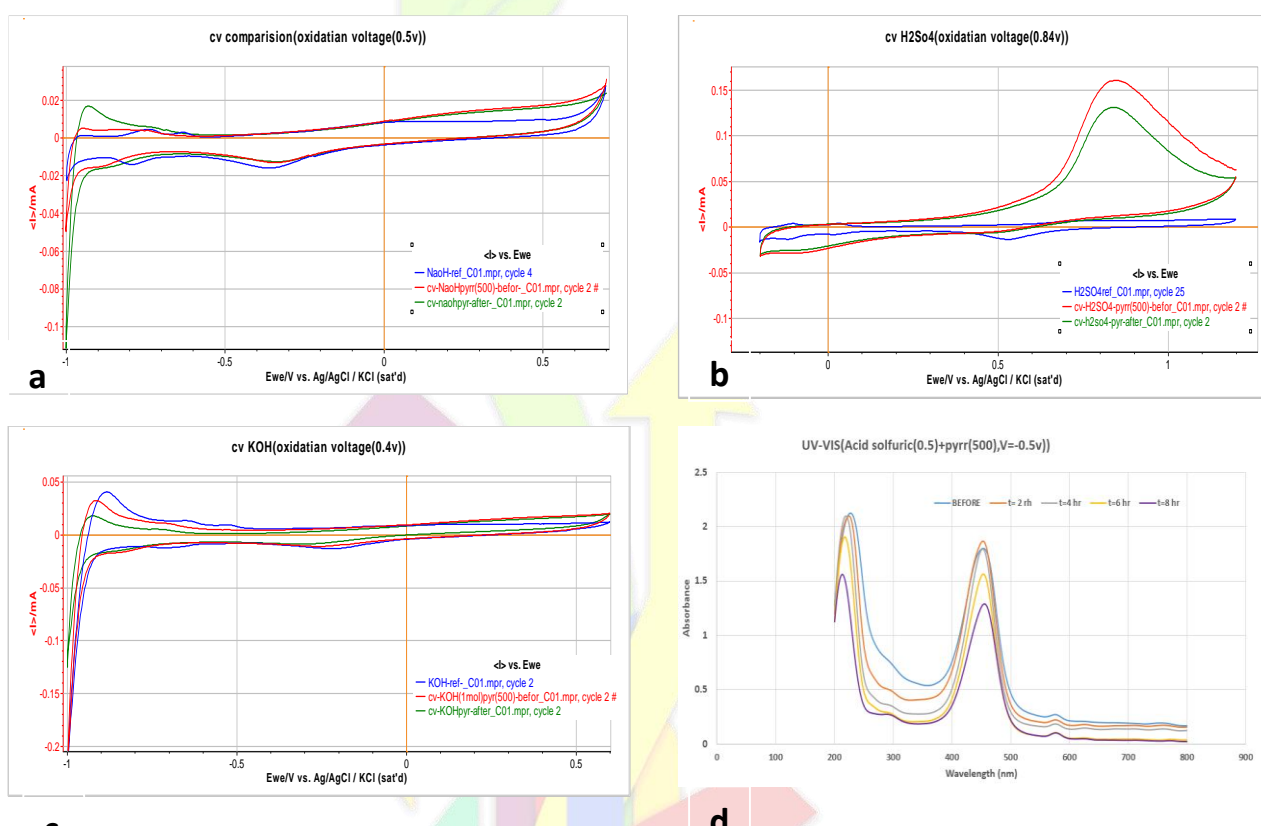


Fig.1. Cyclic voltammetric before and after applying potentials for 10 minutes a) 1 M NaOH + 0.5×10^{-3} M pyrrole, $V=0.5$ V b) 0.5 M H₂SO₄ + 0.5×10^{-3} M pyrrole, $V=0.84$ V c) 1 M KOH + 0.5×10^{-3} M pyrrole, $V=0.4$ V . d) UV spectra of pyrrole at different time intervals during electrochemical treatment.

Results and discussion

Figure 1:a, b and c, show Cyclic voltammetric before and after applying potentials for 10 minutes. The results of this study showed that the most proper electrolyte among these is H₂SO₄, Because the difference between the preceding and subsequent diagram is indicative of the pyrrole removal. Also large peak at 0.85 V revealed the oxidation potential of pyrrole in acidic medium. Figure 1:d illustrates decrease of pyrrole concentration at different time intervals during CA test (applied potential was -0.5V). It revealed that 30% of pyrrole was removed after 8 hr.

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Investigation of Electrochemical Sulfide Removal from wastewater using different electrolyte

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Abstract

Electrochemical oxidation of sulfide is a promising method for its removal from wastewaters. One of the primary product of electrochemical oxidation is elemental sulfur. Sulfur deposits on the electrode and deactivates it. In this paper electrochemical removal of aqueous sulfide in water was investigated. Two electrolytes were employed in the undivided cell, and results showed that pH of the electrolyte is an important parameter in the removal of sulfide. The results of this study showed that adsorption of sulfide in sodium hydroxide electrolyte is proper than the basic electrolyte. The optimum sulfide adsorption potential for 400 ppm sulfide in sodium hydroxide was determined -0.65 V.

Keywords: sulfide, wastewater, Pt electrode, Electrochemical desulfurization, Electrolyte.

Introduction

Sulfide present in the aqueous stream from industrial wastewater such as chemical and petrochemical industrial and mineral waste, Slaughterhouse waste, Urban sewage and so on. The threshold odor concentration of H₂S in clean water is between 0.025 and 0.25 µg/L. It attacks metals, directly and indirectly, has caused severe corrosion of concrete sewers because it is oxidized biologically to H₂SO₄ on the pipe wall. Dissolved H₂S is toxic to fish and other aquatic organisms.

The most commonly used methods involve physicochemical processes including wet air oxidation and incineration, oxidation with oxidant agents addition, precipitation and neutralization/acidification, electrochemical, biological or bio-electrochemical processes. The key limitations that restrict their application are cost, complexity, high consumption of chemicals, safety/handling issues and most importantly the lack of recovered product [1].

Vaiopoulou et al.[1] propose a novel method that relies on the simultaneous anodic oxidization of sulfide coupled to cathodic caustic generation in a two-compartment electrochemical cell. Batch experiments showed sulfide removal efficiencies of $84 \pm 4\%$ with joint $57 \pm 4\%$ efficient caustic production in the catholyte at an applied current density of 100 A.m^{-2} . Long-Term continuous experiments showed that stable cell voltages (i.e., $2.7 \pm 0.1 \text{ V}$), as well as constant sulfide removal efficiencies of $67 \pm 5\%$ at a loading rate of $47 \text{ g(S) L}^{-1}.\text{h}^{-1}$ were achieved over a period of 77 days. Lu et al. [2] were explored to remove sulfide in municipal sewage by a Pulsed electrochemical process in a single chamber electrochemical reactor, which successfully achieved higher removal rate of 93.2% compared with 73.2% by direct current (DC) power supply. Al Kharafi [3] investigated the effects of scan rate, the concentration of sulfide ions and temperature on the cyclic voltammograms. Lin et al. [4] was studied to use low price electrode materials of carbon cloth and stainless steel AISI 304. They investigated three voltages and two concentrates of sulfide in various temperatures and found out Both electrode materials showed complete sulfide removal of 10 mM at 3 V in 2 days. Pikaar et al. [5] used galvanostatic control and showed The results of the experiments from the synthetic feed at current densities of 25, 50 and 100 A.m^{-2} . The sulfide removal rates increased when the current density was increased from 25 to 100 A m^{-2} .

Experimental

Cell, materials, and electrodes

A SP-150 Potentiostat was used for electronic control and data acquisition. A conventional, three-electrode cell was used equipped with a multiple-inlet system for purging and blanketing the solution with oxygen-free nitrogen. The reference electrode was Ag/AgCl/1.0 M Cl^- and all the potentials reported in this paper are referenced to this electrode. The working electrode used for voltammetric measurements was a 2 mm diameter platinum rod. All the reagents used were highly pure (Merck Millipore).

Experimental procedures

The electrode was initially cleaned in a freshly prepared sodium hydroxide solution. For investigation of the adsorption of sulfide on platinum electrodes, the working electrode was

allowed into contact with an electrolyte + 400ppm sulfide solution for 10 min under potential control. Sulfide solution was then rinsed out from the cell with sulfide-free 1 M NaOH solution. The voltammogram of the sulfide-dosed platinum electrode was recorded in 1M NaCl + 1000 ppm of sulfide in different scan rate. It shows with NaCl in the oxidation region there are two peaks or more. we can guess the concentration of sulfide in NaOH by calculation of the area under the oxidation peak.

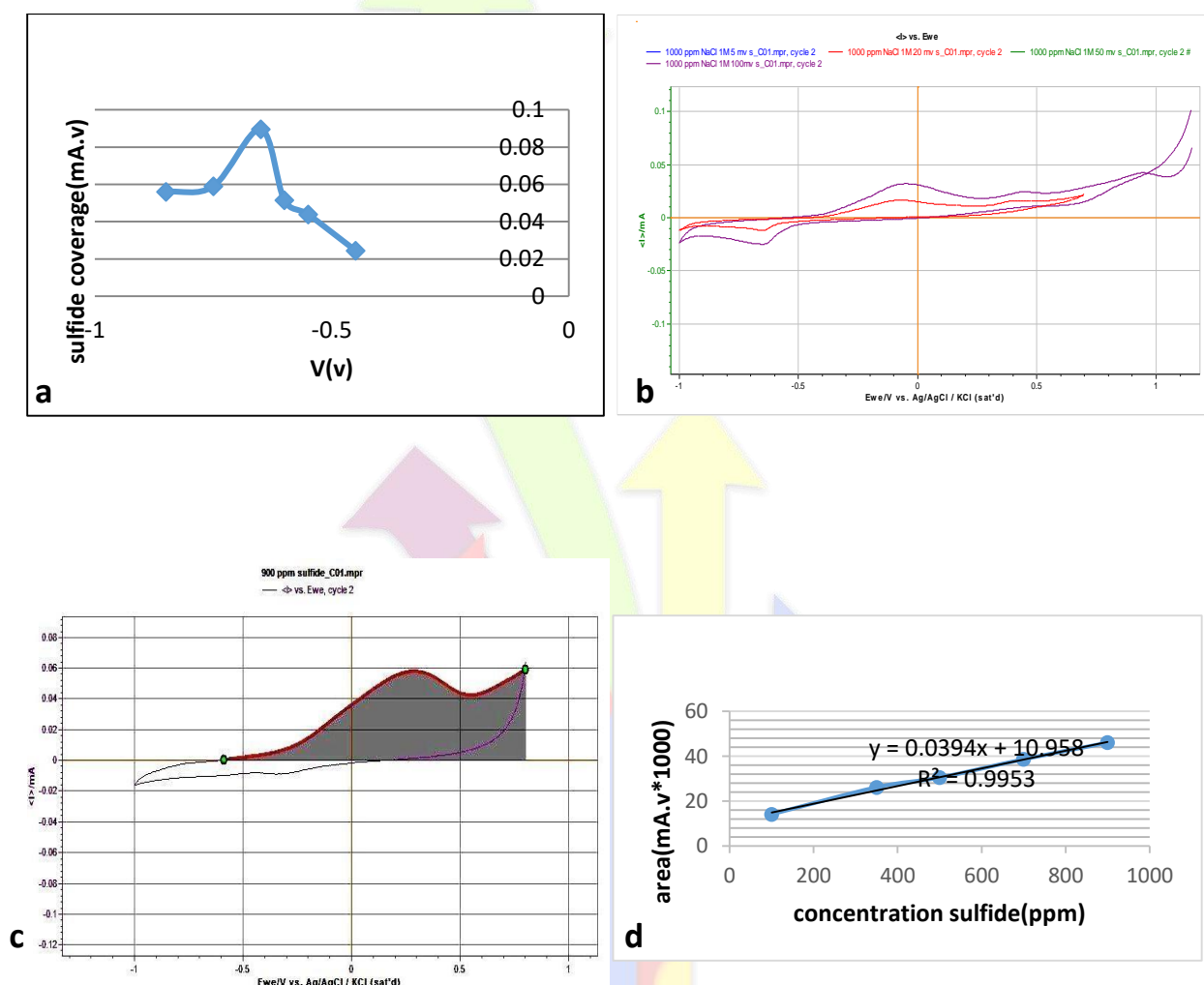


Fig. 1. **a)** The coverage of sulfide on a Pt electrode vs. the potential at which sulfide was adsorbed. Sulfide adsorption was carried out potentiostatically in 1 M NaOH + 400ppm sulfide, **b)** The voltammogram of the sulfide-dosed platinum electrode was recorded in 1M NaCl+1000ppm of sulfide in different scan rate, **c)** The voltammogram of the sulfide+NaOH was shown that area below the surface of sulfide. **d)** By making five solutions with different concentrations and calculation of the area was drawn this diagram.

Results and discussion

The diagram of sulfide adsorption in NaOH shown in Fig 1:a. The results of this study showed that the best sulfide adsorption potential for NaOH occurs at -0.65 V. Regarding CV of sulfide+NaCl in Fig. 1:b, different reactions can occur at low pH and have different products. Fig. 1:c shows the recorded voltammogram of sulfide+1 M NaOH at 900 ppm and the area of diagram that is important to find out the concentration. The Figure 1:d shows the correlation of area Vs. The concentration and revealed that with increasing the concentration of sulfide, the area is increasing.

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Manufacturing of electrochemical sensors to determine some metal traces and applied it environmentally

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Aims of this research:

1- Preparation of a modified graphite electrode for determination of studied metal ions based on Hydrazone compounds as ionophore. 2- Prepare the electrode in specific dimensions. 3- Set an Algorithm, which make the instrument accept our new working electrodes. 4- Choose the optimal conditions to determine the studied metal ions using the new working electrodes:

Techniqueal conditions: Accumulation potential, Accumulation time, Potential scanning rate and Pulse amplitude.

Analytical conditions: Choose the Electrolyte, Electrolyte Concentration and pH.

5- Statistical study. 6- Apply the new analytical system with its own conditions on environmental samples.

Significance of the research:

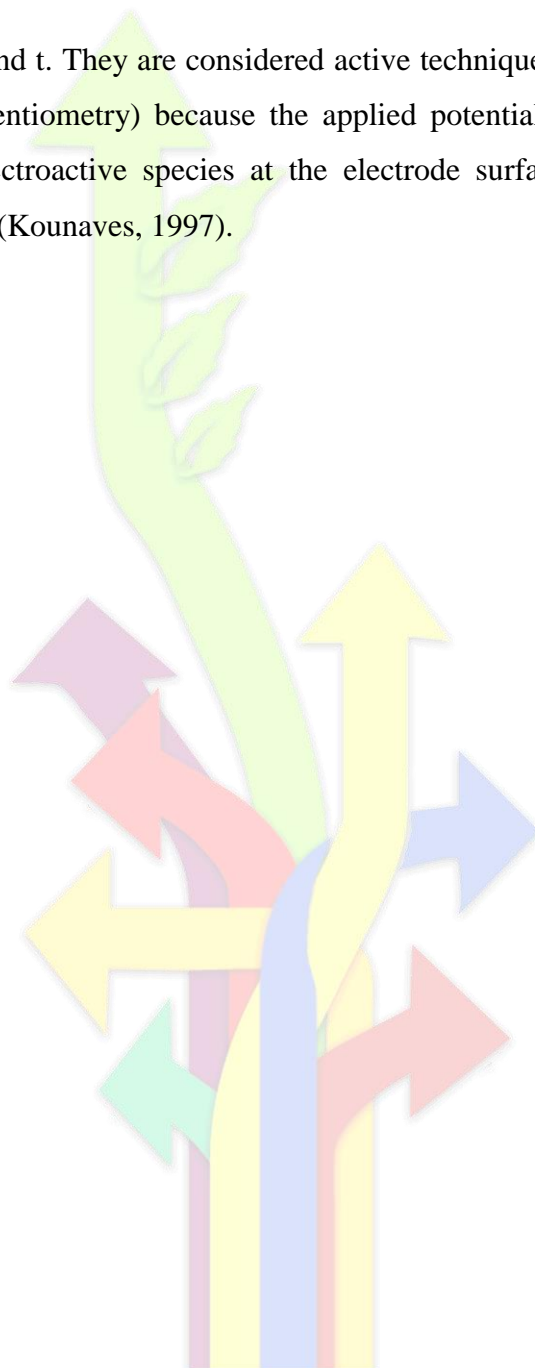
Development of an analytical system, which has a sensitivity and precision, ease and speed and low cost, by adoption of (Hybrid Method) or (Combinative) that promote the processes of separation and detection at the same time, which reduces the complex chemical procedures governing the sample before measurement, which are often the source of many errors. By using these ion selective electrodes as working electrodes, the detection limit 10-14 mg/l could be reached. Generally, working electrodes in this technique made up of gold, platinum, or silver, so it is expensive, but our new electrodes will be made up of organic materials and PVC with high analytical sensitivity and accuracy, so it has low cost.

Methodology and research techniques:

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In this research, we used Voltametric Technique to determine the ultratraces of Hg, As, Pb and Cd. The common characteristic of all voltammetric techniques is that they involve the

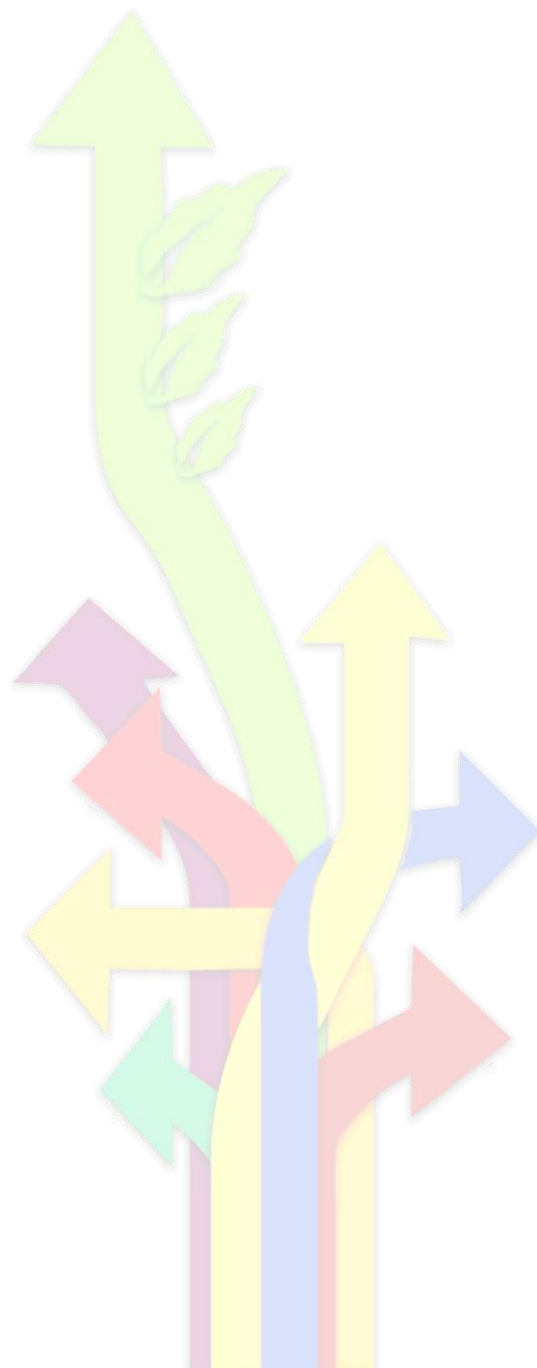
application of a potential (E) to an electrode and the monitoring of the resulting current (i) flowing through the electrochemical cell. In many cases the applied potential is varied or the current is monitored over a period of time (t). Thus, all voltammetric techniques can be described as

some function of E , i , and t . They are considered active techniques (as opposed to passive techniques such as potentiometry) because the applied potential forces a change in the concentration of an electroactive species at the electrode surface by electrochemically reducing or oxidizing it (Kounaves, 1997).



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Energy



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Effect of Silver (II) oxide Particle Size on Discharge Characterization of Cathode for Alkaline Battery

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Abstract

Silver (II) oxide (AgO) is used as a cathode material in a variety of alkaline batteries [1]. This cathode material is widely used in space and military industries due to their high energy density, capacities and average voltage [2]. The properties of the cathode discharge are one of the most important functional properties of the battery and depend on various factors such as porosity, surface area and capacity of active material, the conductivity of the adhesive and active material system, and other factors [3,4].

In various studies, the electrochemical properties and conductivity of cathodes based on AgO have been investigated, but the effect of particle size distribution on the properties of the cathode discharge has not been considered. In this research, AgO particles were synthesized by oxidation of silver nitrate with potassium persulfate in an alkaline medium. The five samples of AgO active material were synthesized in various concentration and synthesis condition. Then, characterization and discharge properties of the AgO material were investigated.

SEM micrographs show that all synthesized particles are in the form of a plate, and the difference between particles is due to differences in particle size and particle size distribution (Table 1). The synthesized AgO samples 3 and 4 have the smallest average length size and narrowest particle distribution (0.05-0.3 μm). The widest range of particle size is for the sample 5, which is a mixture of fine particles of 0.1 μm and large particles of 4.5 μm .

Figure 1 shows the discharge curves of the synthesized AgO cathodes at a current density of 0.2 A/cm². The results show that the highest output voltage (1.78 V) and the lowest voltage drop is observed for sample 5. Also, this sample cathode provides a flatter discharge profile than other samples. The reason for these outstanding properties can be seen in improving the cathode conductivity and the silver product formed in the second step of discharging. Table 1 shows the results of particle size and the capacity values of the synthesized samples. The

results show that the sample 5 has the highest capacity (0.3 Ah.g^{-1} , and 0.46 Wh.g^{-1} , respectively) in between samples. This value is about 1.5 times the capacity of sample 1 (0.18 Ah.g^{-1} and 0.27 Wh.g^{-1}). The sample 5 has a mixture of fine and large particles, which increase the connection between particles, decrease interconnections and creates a path for conduction in the cathode with the lowest contact resistance, while in samples 3 and 4, there are a large number of particles in the nanometer range that whose interconnections lead to increased contact resistance and reduced number of conduction paths in the cathode and therefore have finite conduction properties.

Keywords: AgO Cathod, Alkaline Battery, Particle Size, Discharge Properties

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Table 1. Range of particle size and capacity of synthesized AgO particles

Sample	Range of particle size (μm)	Capacity (Ah.g^{-1})	Capacity (Wh.g^{-1})
1	0.13-2.0	0.18	0.37
2	0.19-1.4	0.11	0.17
3	0.05-0.7	0.07	0.01
4	0.04-1.5	0.10	0.16
5	0.13-4.3	0.30	0.46

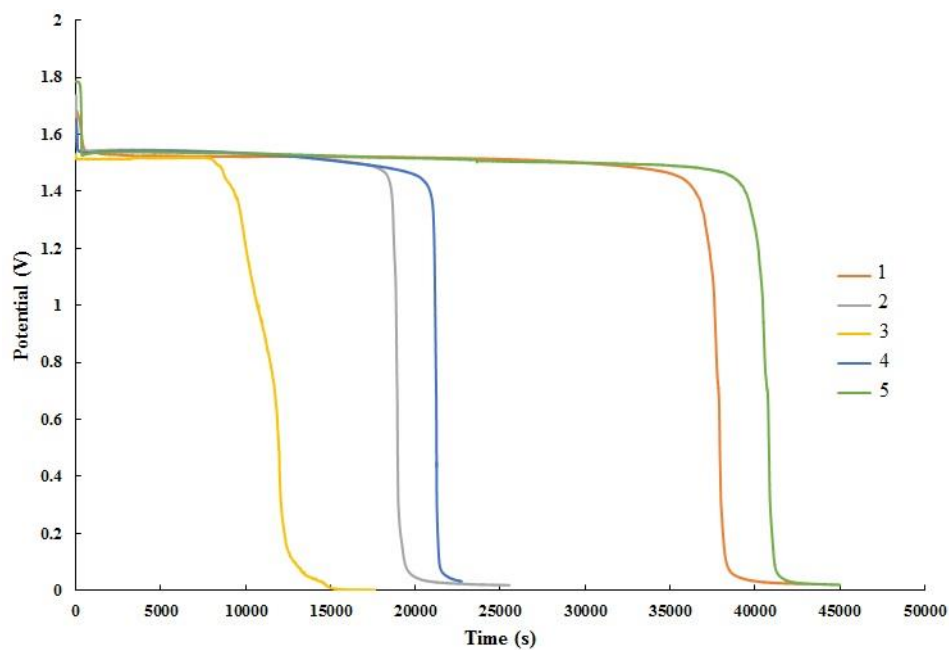


Figure 1. Discharge profile of synthesized AgO particles

Ti₃C₂T_x/ CMK-5 Nanocomposite as Sheet-like Anode For Lithium Batteries

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Abstract

Lithium ion batteries have great importance for portable electronic devices and stationary energy storage because they are rechargeable power sources, with high power, high energy density and long cycling life [1-3].

Ti₃C₂T_x nanosheets were prepared according to their related Ti₃SiC₂ MAX phase. Ten grams of Ti₃SiC₂ sample, was immersed in 50 wt.% HF solution under magnetic stirring. After washing, the powders were dried. The as-obtained d-MXene powder mixed with CMK-5 in equal weight and disperse in water/ethanol (1 and was subjected to high energy ball milling. Cells were assembled in an Argon filled glove box using Ti₃C₂T_x, Ti₃C₂T_x/CMK-5 nanocomposite and lithium foil as reference electrode.

It can be seen from Figure 1a that completely exfoliation performed and two-dimensional layered MXene was created. As seen in Figure 1b, when the Ti₃C₂T_x and CMK-5 nanoparticles were milled, CMK-5 can effectively hinder the aggregation of the individual Ti₃C₂T_x sheet by the shear force of zirconia balls and CMK-5 nanoparticles.

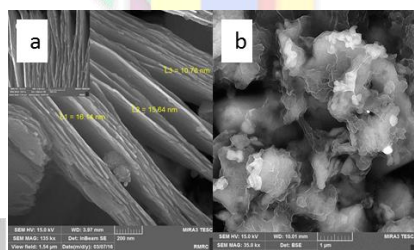


Figure 1 . SEM images a) Ti₃C₂T_x and b) Ti₃C₂T_x/CMK-5

Figure 2a shows voltage- capacity dependence profiles for $\text{Ti}_3\text{C}_2\text{T}_\text{x}$ and $\text{Ti}_3\text{C}_2\text{T}_\text{x}/\text{CMK-5}$ nanocomposite at 0.1C. For the $\text{Ti}_3\text{C}_2\text{T}_\text{x}/\text{CMK-5}$ electrode, the initial discharge and charge capacities were $1035.9 \text{ mAh g}^{-1}$ and 576.6 mAh g^{-1} , respectively. The first cycle irreversibility and initial capacity drop can be ascribed as SEI layer formation and irreversible reduction of electrochemically active surface groups.

Figure 2b displays cycling performance of $\text{Ti}_3\text{C}_2\text{T}_\text{x}/\text{CMK-5}$ and $\text{Ti}_3\text{C}_2\text{T}_\text{x}$ electrodes at 1C. The $\text{Ti}_3\text{C}_2\text{T}_\text{x}/\text{CMK-5}$ electrode shows the capacity of 342 mAh g^{-1} after 100 cycles with a coulombic efficiency of 98.6%. The voltage- capacity dependence profiles of $\text{Ti}_3\text{C}_2\text{T}_\text{x} / \text{CMK-5}$ nanocomposite as an anode in Li- ion cell at different C rates are shown in Figure 2c. As seen, by increasing current rates, the capacity decreased, however gradually.

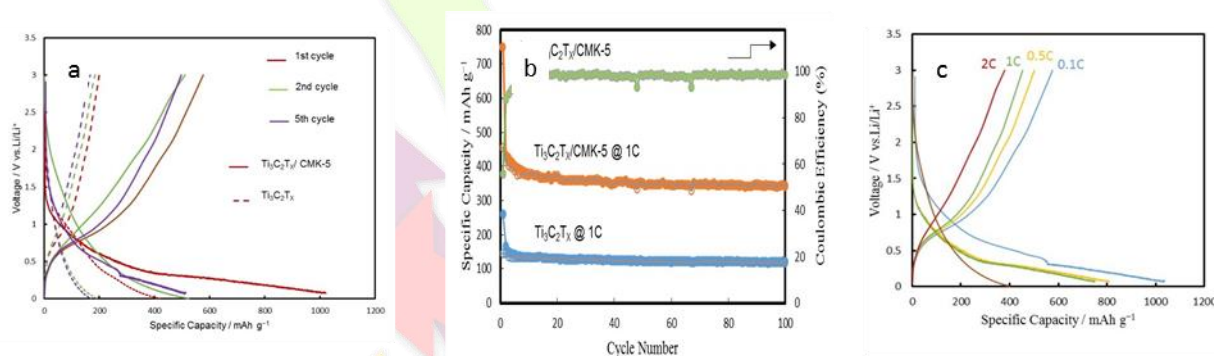


Figure 2. a) Voltage profiles of the $\text{Ti}_3\text{C}_2\text{T}_\text{x} / \text{CMK-5}$ composite and pristine $\text{Ti}_3\text{C}_2\text{T}_\text{x}$ at 0.1 C b) Cycling stability of pristine $\text{Ti}_3\text{C}_2\text{T}_\text{x}$ and $\text{Ti}_3\text{C}_2\text{T}_\text{x} / \text{CMK-5}$ composite at 1C and (c) voltage profiles of $\text{Ti}_3\text{C}_2\text{T}_\text{x} / \text{CMK-5}$ electrode at different cycling rates.

Our results revealed that $\text{Ti}_3\text{C}_2\text{T}_\text{x}/\text{CMK-5}$ nanocomposite with fast and simple method production, by improving in all MXene- based materials, can show high capacity and excellent rate capability and make it a suitable candidate for energy storage devices or other applications.

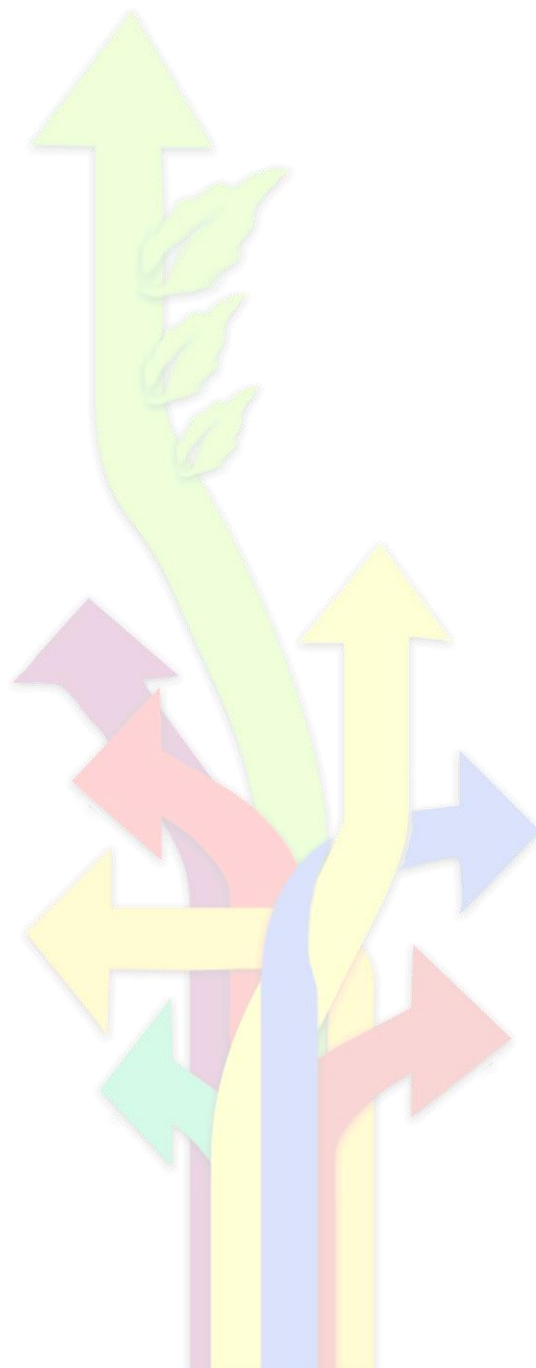
Keywords: *Lithium Batteries, Mxenes, Nanocomposite, Cycle Performance*

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13th Annual Electrochemistry Seminar of Iran

Tailored Silicon Mesoporous via Magnesiothermic Reduction for Silicon-based Lithium Ion Batteries

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Abstract

Silicon mesoporous as a most promising anodic material for high capacity lithium ion Batteries was synthesized via magnesiothermic reduction. Purity of the prepared compound was confirmed by XRD analysis as well as nanometer pores by FE-SEM. The electrochemical results delivered the first charge/ discharge capacity of 700 mAh/g and 415 mAh/g at current density of 100 mA/g. Dealloying of $\text{Li}_{3.75}\text{Si}$ to Li_2Si and Li_2Si to c-Si and alloying c-Si to a-Si were confirmed through CVs curves. Moreover, impedance spectroscopy resulted 50 Ω resistance connected to charge transfer.

Keywords: Lithium ion Battery, Silicon, Mesoporous, Impedance Spectroscopy.

Introduction

High capacity silicon-based lithium ion batteries have a broad application in the portable electronic devices due to its reversible energy storage. The swelling structure after several charge/discharge cycles is a critical issue in LIBs that can be overcome by nanostructuring the host material. Mesoporous structures with high specific surface area can facilitate the further activated sites for lithium insertion. Thereby, 4.4 lithium ions electrochemically react with one host atom on the surface of material instead of diffusing into the bulk and increase anodic capacity to near of 4200 mAh/g [1, 2].

Herein, silicon mesoporous was synthesized via salt scavenger-assisted magnesiothermic reduction and electrochemical performance was investigated through charge/discharge profile, CV curves and EIS analysis.

Methods

A specific weight of SiO_2 powder synthesized via stober method were mixed together Mg powder with mole ratio of 1:2.5 into the glovebox filled with argon. The mixture carried into

the tubular furnace at 600 °C and heated for 1h. The collected brown powder acid leached by 1M HCl and HF for removing the impurities and dried into the vacuum oven at 100 °C. Next, working electrode was prepared with loading mass of 1 mg comprising of 80 wt.% silicon, 15 wt.% carbon black and 5 wt.% PVDF binder dissolved in N-methyl-2-pyrrolidone (NMP) on copper foil. Electrochemical measurements were carried using CR2032 coin cell by lithium foil as the cathode, Celgard polyethylene membrane as the separator and 1M LiPF₆ dissolved in EC/DMC (1:1 v/v) as the electrolyte. The galvanostatic charge–discharge testing and cyclic voltammetry (CV) curves at a scan rate of 1 mV s⁻¹ in the potential range of 0-2 V (vs. Li⁺/Li) were conducted on Potentiostat PGS 2065. Electrochemical impedance spectroscopic (EIS) measurements were carried out using two-electrode coin cells at room temperature with EG & G Parstat 2273 over the frequency range between 100 mHz and 1MHz.

Results and discussion

The XRD pattern of sample in Fig. 1-a presented crystallite silicon with FCC lattice and lattice constant of 5.42 Å, matching with 27-1402 reference card. After Rietveld refinement crystallites size at (111), (220), (311), (100), and (311) was calculated to be 70, 57, 53, 52, and 47 nm using Scherrer formulae. FE-SEM micrograph in Fig. 1-b clearly showed slit-like pores. As seen in Fig. 1-c, the first specific charge/discharge capacity delivered to 700 mAh/g and 415 mAh/g at current density of 100 mA/g. In addition, silicon mesoporous could delivered 500 mAh/g and 360 mAh/g for specific charge/discharge capacity in current density of 200 mA/g. So, silicon mesoporous as the post-anode can be replace with traditional graphite anode. In CV curves in Fig. 1-d, two anodic peak at 0.3V and 0.53 V assigned to dealloying of Li_{3.75}Si to Li₂Si and Li₂Si to c-Si, respectively. While reduction Lithium ions into the silicon structure presented one cathodic peak at 0.12 V at first potential sweep that assigned to alloying c-Si to Li_{3.75}Si. Moreover, increasing intensity of anodic and cathodic peaks after three cycles is led to improve the kinetic of redox reactions. The Nyquist plot of silicon mesoporous in Fig. 1-e is shown. Electrolyte resistance was measured to be 9Ω. One depressed semi-circle at high frequencies with 50Ω resistance was referenced to charge transfer resistance into the surface electrical double-layer. Also, specific capacitance for EDL was calculated to be 1μF/cm⁻².

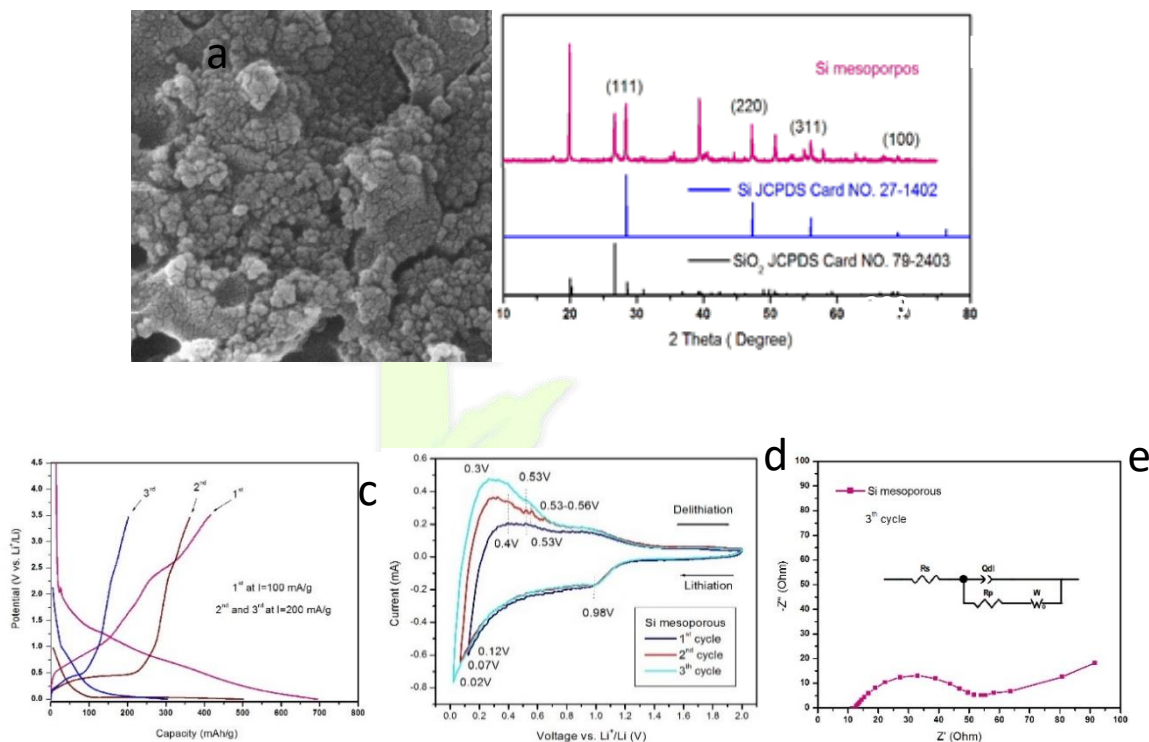


Fig1. a) The XRD pattern, b) FE-SEM micrograph, c) charge/discharge profiles at current densities of 100mA/g and 200 mA/g, d) cyclic voltammograms at the scan rate of 1 mV/s in the potential range of 0-2V, and e) Nyquist plot together equipment circuit for of silicon mesoporous.

Conclusion

Silicon mesoporous was synthesized via magnesiothermic reduction at 600°C for high capacity lithium ion Batteries. The electrochemical results delivered the first charge/discharge capacity of 700 mAh/g and 415 mAh/g at current density of 100 mA/g, and 500 mAh/g and 360 mAh/g for specific charge/discharge capacity at current density of 200 mA/g. Also, charge transfer resistance and specific capacitance of the surface electrical double-layer were measured to be 50Ω and 1μF/cm⁻², respectively.

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Electrochemical properties of Silver(III) Oxide Cathode and comparing it to Silver(II) Oxide Cathode in Zinc-Silver Oxide Cells

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Abstract

Silver can be oxidized in different oxidation states +1,+2,+3 and more. It can be prepared different compositions. The most popular oxidation states of silver oxide are monovalent (Ag_2O), divalent (AgO), and trivalent (Ag_2O_3) [1]. Because of high specific energy the batteries based on Silver are very regarded [2]. The cathodes containing this oxide has an appropriate potential and doesn't have an environmental pollution [3]. One of these oxides is Ag_2O_3 , However, it is more unstable than other silver oxides but it conductor of electricity and its impedance less than them [4].

In this work, Silver(III) oxide and Silver(II) oxide were synthesized, the cathode and the cells that containing silver oxide were made, then related tests were done to show the identification of compounds and their electrochemical properties.

Silver(III) oxide and silver(II) oxide were synthesized by ozonolysis [5] and chemical methods [6], respectively. Silver nitrate has been used in both methods. The experimental powders were characterized by thermal analysis and FT-IR methods.

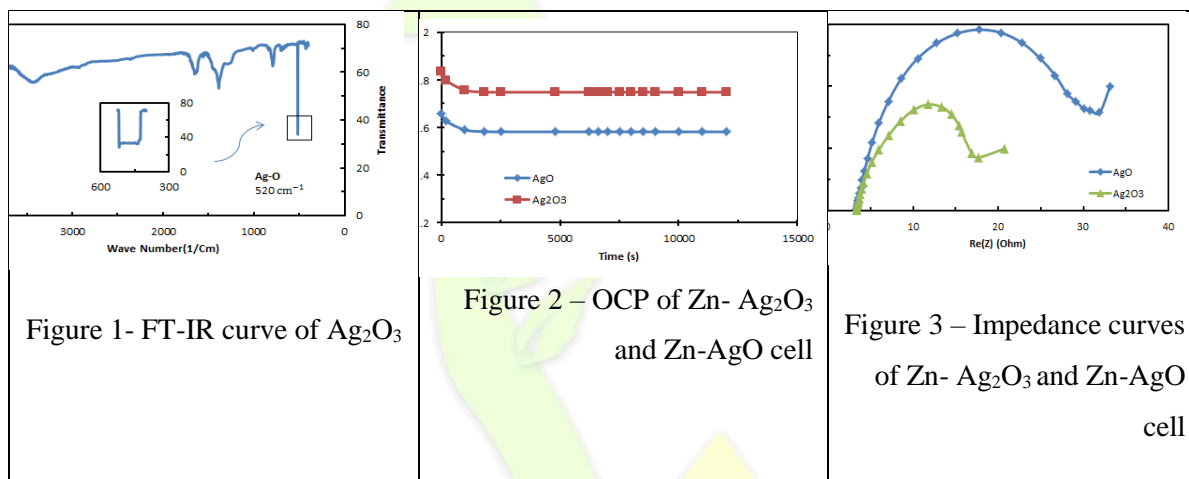
The presence of a peak at a frequency of 520 cm^{-1} endorsement the connection between silver and oxygen (figure 1). The results were showed that Ag_2O_3 and AgO were synthesized by high efficiency (99.67 and 96.7 percent).

Silver oxide cathodes Ag_2O_3 and AgO were prepared by mixing these oxides, Acetylene black and polyvinylidene fluoride.

Zinc, Silver oxides and Potassium hydroxide were used as an anode, a cathode and electrolyte, respectively. Some electrochemical tests were done such as open circuit potential (OCP) Discharge and potentiodynamic Electrochemical Impedance Spectroscopy. The results of OCP were shown the potential of Zn- Ag_2O_3 and Zn- AgO were 1.8(V) and 1.6(V),

respectively (figure 2). It means the potential of the Zn- Ag_2O_3 cell was more than the Zn- AgO cell. Discharge curves were shown the specific energy of these cells at 1C for Zn- Ag_2O_3 and Zn-AgO were 2.194 and 0.7274 (Wh/g). Impedance curves were shown the resistance of the Zn- Ag_2O_3 cell was less than Zn-AgO cell.(Figure 3)

All of these results were shown the Ag_2O_3 is the best alternate for AgO.



Keywords: Zinc-Silver Oxide Battery, Silver(III) Oxide, Ozonolysis, Silver(II) Oxide

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Synthesis of mixed binary oxides by Cathodic electrodeposition for energy storage systems

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Abstract

Recently, there is a growing demand for cheap and efficient energy storage devices. Supercapacitor as an uninterruptable power source can be coupled with batteries to provide peak power and can replace batteries for memory back up. Some benefits of supercapacitors in comparison with other energy storage devices are long life, high power density, wide thermal range and low maintenance [1]. Electrode material is the most important component of a supercapacitor. So, there is much attempt to reach electrode material with higher power and energy density. Synthesis of various mixed binary metal oxides is an example of these attempts. Elemental doping of simple pseudo-capacitive metal oxides to form mixed metal oxides has been proven to be an effective way to increase their electronic conductivity, specific capacitance and cyclic durability [2]. In this work, nano-size of binary metal oxides Mn-Al, Mn-Ce, Ce-Al were synthesized by cathodic electrodeposition method. These binary metal oxides were characterized by SEM and XRD methods. These materials will be used as electroactive materials for supercapacitors.

Keywords: Supercapacitor, Mixed oxide, Elemental doping, Cathodic electrodeposition

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Sonoelectrochemical deposition of graphene oxide-polypyrrole/cobalt oxide nanocomposite and investigation of their electrochemical properties in supercapacitors

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Abstract

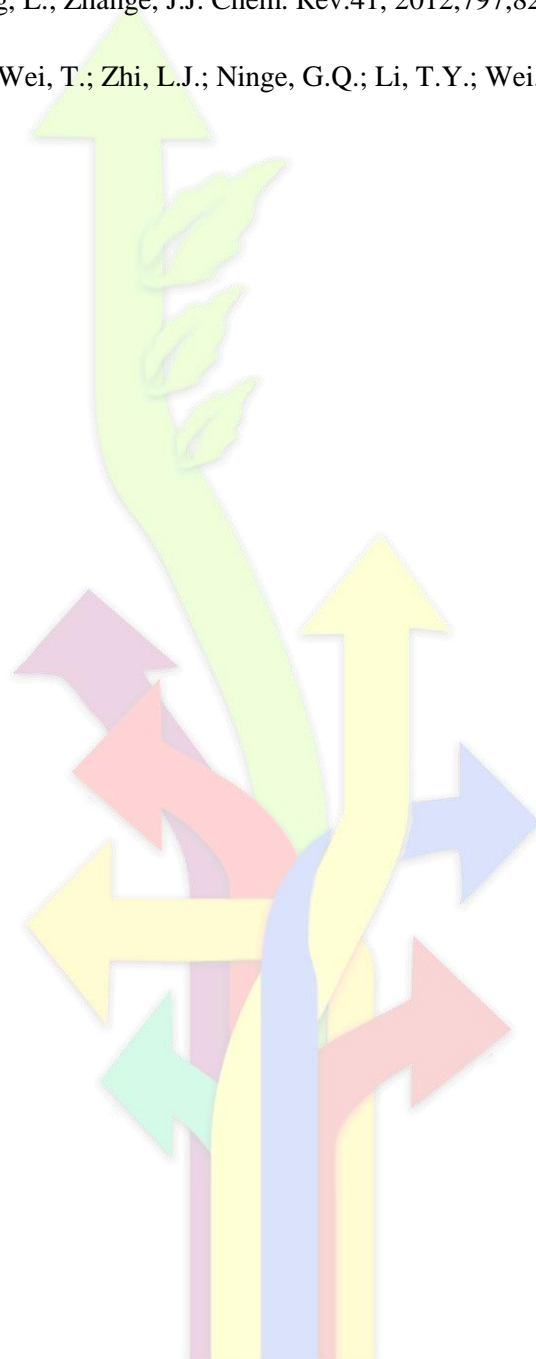
As a type of electrochemical energy storage device supercapacitors have attracted considerable attention over the past decades. owing to the higher power density and longer cycle life than secondary batteries and higher energy density compared to conventional capacitors, they have application in some systems such as hybrid electric vehicles, portable electronics, mobile communications and etc. Generally, the charge storage mechanism of supercapacitors could be classified in to two types: electrochemical double layer capacitors (EDLCs) and faradaic pseudocapacitors.

The aim of this project is the sonoelectrochemical deposition graphene oxide-polypyrrole/cobalt oxide nanocomposite and improvement of electrochemical properties in such as cycle stability, special capacity and etc. the morphology and structure of this materials were studied by using scanning electron microscopy and energy dispersive x-ray spectroscopy .electrochemical activity and capacitor behavior of prepared electrodes were studied using electrochemical techniques including cycle voltammetry, galvanostatic charge-discharge and electrochemical impedance spectroscopy.it was found the grapheme oxide-polypyrrole /cobalt oxide electrode has better capacitance and electrochemical properties thane graphene oxide -polypyrrole electrode and polypyrrole electrode ,the calculated specific capacities by cyclic voltammetry and charge-discharge confirmed this clam. The specific capacity value that obtained by cyclic voltammetry at the scan rate 10 mv.s^{-1} , consiste of 228 F.g^{-1} for the graphene oxide-polypyrrole/cobalt oxide electrode, 168 F.g^{-1} for graphene oxide -polypyrrole electrode and 22F.g^{-1} for polypyrrole electrode. The same a capacities amounts also obtained by charging – discharging technique.

13th Annual Electrochemistry Seminar of Iran
Keywords: Sonoelectrochemical deposition, Supercapacitor, Polypyrrole, Nano composite

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Design and manufacture of an electrochemical carbon-zinc battery and its use in Chem-E-Car

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Abstract

In this research, the purpose is manufacture of a battery to eliminate the limitation of the Leclanche and alkaline batteries and make them more efficient. Leclanche batteries with all its benefits categorize as wet-cell batteries and in some cases a wet-cell doesn't have any useful usage. Also dry cells aren't rechargeable. The battery designed and manufactured in this work, eliminate some of disadvantages such as it belongs to emi-dry batteries, it is reachargable and its voltage does not drop if not used for a while. Also, the voltage drop rate is very slow at the laboratory scale usage.

Keywords: Electrochemical Cell, Carbon- Zinc, Chem-E-Car

Battery description

chem-E-Car national competition is a student activity that hold annual to actualize the theory lessons for student. The target of Chem-E-Car is to find an alternative for fossil fuels which is more environmentally compatible. The most common chemical materials used as fuel are: Acetic acid, Sodium dicarbonate, baking soda, hydrogen peroxide, potassium iodide. The majority of these fuels are green fuels [1].

Common types of driving forces are electrochemical cell, fuel cell and driving force by pressure that usually use copper-magnesium electrochemical cell in this competition and Hakim Sabzevari university team eliminated acid (sulfuric acid) used as electrolyte and a completely different type of cell was manufactured.

In the designed cell, zinc was anode and greasy carbon was used as cathode. 15M solution of potassium hydroxide was used as electrolyte. For construction of this cell, the mixture of greasy carbon and manganese dioxide was used by ratio of 3 gr greasy carbon to 8 gr manganese dioxide to have better electron conductivity. This factor is an efficient parameter for having higher voltage and ampere.

Manganese dioxide works as a catalyst and carbon rod has a conductivity role. The overall structure was that the carbon rod was located in the middle of the cell and the powder mixture of manganese dioxide and greasy carbon were directly connected to the rod and the zinc plate were wrapped around it. However, it should be noted that the zinc plate didn't have direct contact with the mixture and was separated by a membrane.

Experiments were carried out on different electrolytes with various concentrations and the results show that the use of strong base has positive influence on the cell performance.

For activating the cell, each cell should be placed in potassium hydroxide solution for one minute and the cell is ready to operate.

For each cell, 100 mA and 1.2-1.3 voltage can be received. About 12 volts with approximately 1 ampere is required to start the engine up.

So, ten series each containing ten cells were made. Then these series were connected to each other in parallel mode making a 10×10 cell matrix producing the power needed for the electrical motor.

One of the advantage of this cell in comparison with other cell is that the power dropped occur slowly through time.

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Design and construction of a thin layer electrode and electrochemical Behavior of lithium reserve batteries

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Abstract

Traditional printing methods offer the advantage of well-matured technology, high accuracy of depositing inks over flexible substrates at high web speeds, and low cost of fabrication. The components of a battery the current collectors, active layers, and separator can all be deposited using conventional printing techniques by designing suitable inks. A combination of low thickness of printed electrodes, flexible packaging, battery architecture, and material properties makes printed batteries flexible.

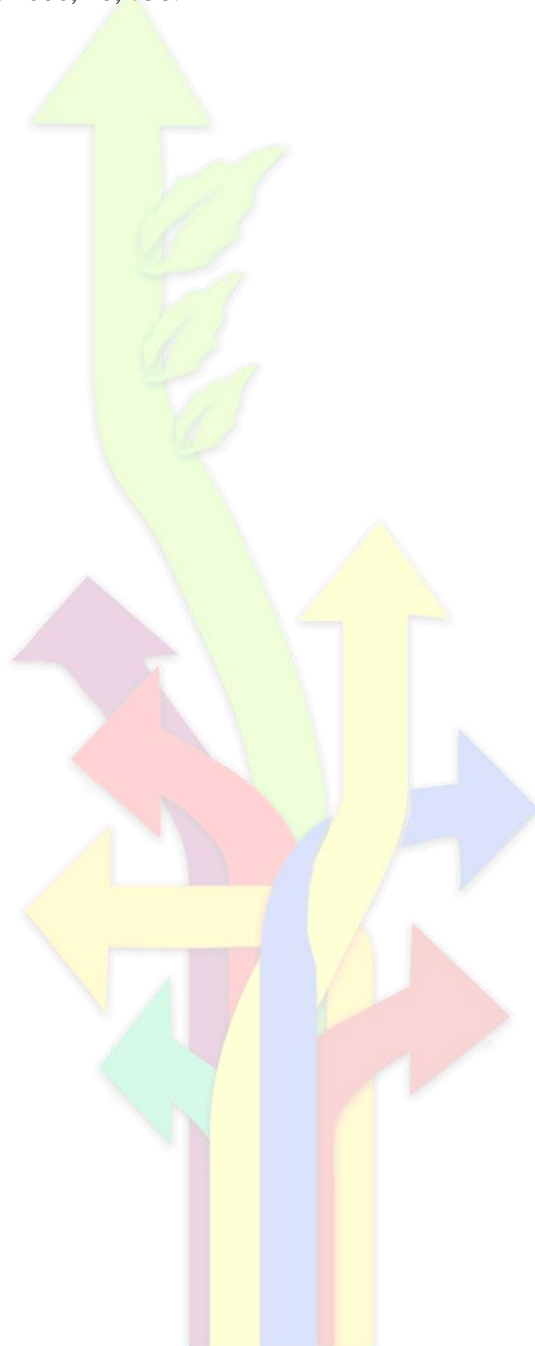
An aqueous slurry can be used to paint thermal electrodes onto a current-collector substrate With a spray gun for thin electrodes or pasting With a thickened slurry. A feedstock aqueous slurry can include thermal electrode components, thermal electrolyte components, a hinder or thickening agent, and Water. This slurry can be sprayed or pasted onto a substrate and dried. To obtain different densities, the substrate can be compressed to a desired density. Thermal electrodes of a desired size and shape can be cut or punched from the sheet. Different binders and binder concentrations can be used toad just the Viscosity or thickness of the electrode.

Keywords: Thin layer, lithium reserve battery, flexible electrode, spray pyrolysis, printing techniques

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Fabrication of graphene and its application as nanocomposite material for lithium ion batteries

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Abstract

As the man's life has significantly impressed with electronic mobile devices recently, the electronic industries have strived to prepare smaller, thinner and lower weight products. The giant electronic companies, therefore, compete in developing more efficient hardware such as batteries used inside the small metallic or polymeric frame. One of the most important materials in the production lines is the lithium-based batteries which is so famous for its ability in recharging as many times as a user needs. However, this is not an indication of being long lasted, as many of the electronic devices are frequently being used for a long time. The performance, chemistry, safety and above all cost of the lithium ion batteries should be considered when the design of the compounds are at the top concern of the engineers. To increase the efficiency of the batteries a combination of graphene and nanomaterials is recently introduced and it has shown to have enormous technological effect in enhancing the durability of the batteries. However, due to its vast amount of properties, these materials can be thought of as preparing the cathode electrode in the lithium-ion battery. Graphene is a free-standing 2D crystal with one-atom thickness of carbon atom. Those carbon atoms are sp²-hybridized atoms arranged in six-membered rings in a honeycombed network which is also considered as the fundamental foundation for fullerenes, carbon nanotubes, and graphite [1]. As a kind of carbon materials, graphene has attracted increasing attention in a variety of fields because of its large specific surface area, good flexibility, superior chemical/thermal stability, and extraordinary electrical, and thermal, mechanical properties. With so many advantages, it is desirable to utilize the unique properties of graphene in composites through the incorporation with all kinds of functional materials [2–3]. One of the most important applications of graphene-based composites is electrode material for lithium ion batteries [4-6]. In this paper, the various approaches to

characterize graphene as a composite material and the process of preparing it for the lithium-ion batteries as well as their electrical properties are discussed. We used the hummer method for synthesis of graphene as it's been discussed by prior literatures. The methods of compositing were different like sol-gel, mechanical mixing, deposition, spray drying and coating.

Keywords: *Graphene, Nanocomposite, Li ion batteries, Efficiency*

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Investigation of ESI layer formation at SiNWs/electrolyte interface for lithium ion batteries

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Introduction

The operating voltage of the commonly used negative electrodes such as graphite and silicon is lower than the lower of the stability voltage window of the commonly used electrolytes (1M LiPF₆ in solution of 1:1 (w/w) ethylene carbonate and dimethyl carbonate (EC: DMC)) which leads to surface forming solid electrolyte interphase (SEI) at surface of electrode and result capacity fading of the battery[1-3]. In this paper formed SEI layer at SiNWs anode surface and electrolyte were investigated by survey of electrochemical curve.

Methods

The silicon wafer (p-type, 8-12 Ω .cm and <111> direction) was washed and placed into these solutions mixture of [AgNO₃] =0.005 M and [HF]=4.8 M for one minute. Then the sample was placed in solution mixture of [H₂O₂] =0.8 M and [HF]=4.8 M. finally the sample was placed in solution of HNO₃:H₂O₂ by ratio of (1:1, v: v) and washed with deionized water and dried by N₂. Swagelok cell were assembled in an Ar-filled glove box (Samanetajhiz). The SiNWs was used as the working electrode and Li foil was used as both of the counter and reference electrode. The electrolyte was used included 1M LiPF₆ in solution of 1:1 (w/w) ethylene carbonate and dimethyl carbonate (EC: DMC) and Celgard 2400 polypropylene was used as the separator.

Chronopotentiometry is applied to determine the chemical species reacted in a specified time range. The current of the working electrode is held constant in 0.1 mA, and the corresponding potential on that electrode over time is recorded to generate choroncpotentiogram.

Result and discussion

Figure 1-a shows the FE-SEM SiNWs array top view that prove formation of SiNWs array. Figure 1-b shows CV curve of SiNWs electrode for 3 cycles a scan rate of 1 mV/s. The charge peak at 0.2 V (versus Li/Li+) is correspond with lithiation process of silicon with lithium. Figure 1-c illustrated voltage profiles for second, fourth, fifth and seventieth charge/discharge. The potential plateau of 0.2 V is observed in all charge curve. A short plateau in all charge curve at 1-1.5 V also observed that correspond with formation of SEI layer. Figure 1-d depicted a peak at potential range of 1-1.5 V (versus Li/Li+) in charge curve correspond with SEI layer reduction. This peak can be observed in all cycles that confirm SEI layer forms in all cycles and has been for a SEI thickness layer on SiNWs surface.

Conclusion

In summary, cyclic stability of SiNWs is completely limited due to SEI layer formation in all charge/discharge cycles. On the other word, the SEI layer and combination play an important role for SiNWs performance for anode in lithium ion batteries.

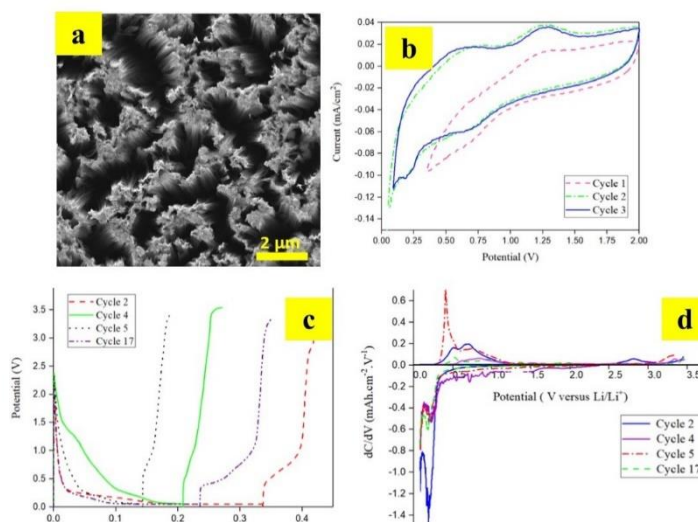


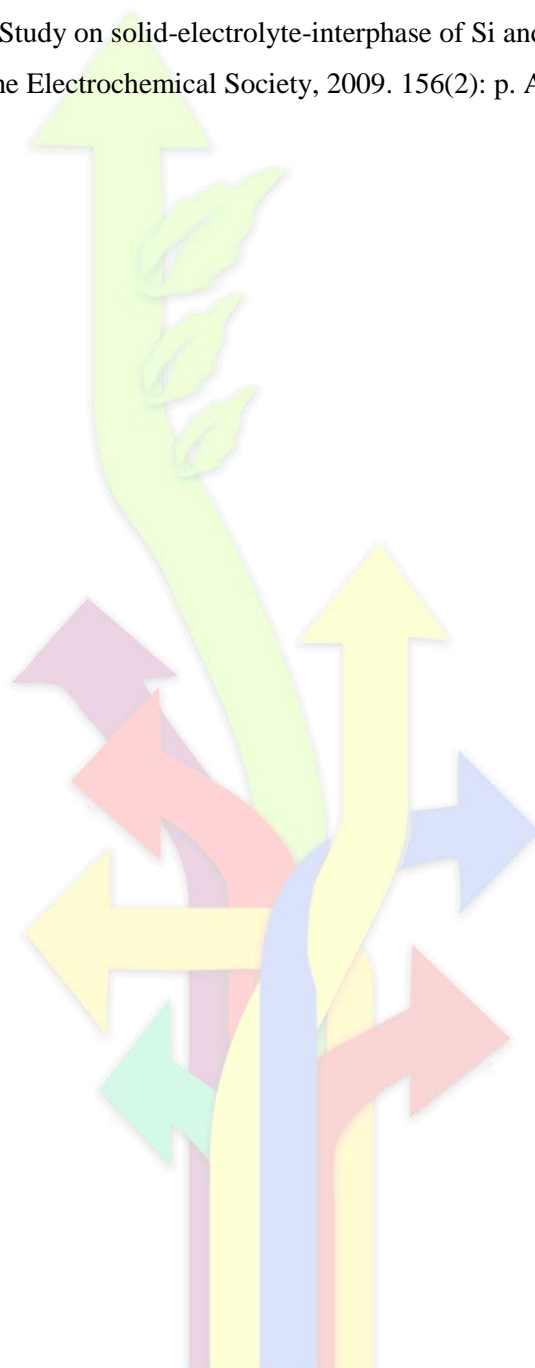
Figure 1 a. FESEM images of SiNWs array, b. CV curve, c. voltage profile and d. dC/dV versus V plot for SiNWs anode material

Keywords: lithium ion battery; SEI layer; Electrode/electrolyte interface; Silicon nanowires array;

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Facile Synthesis of CuO Nanosheets as Anode Material for Rechargeable Lithium-ion Batteries

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Introduction

Li-ion batteries are widely used as one of the most advanced rechargeable batteries today. CuO as an important transition metal oxide, has been considered as a promising anode candidate for LIBs because of its' high theoretical capacity (674 mAhg^{-1}), good rate capability, good safety, non-toxicity, chemical stability and cost effectiveness ^[1-2]. 2D transition metal oxide nanosheets (e.g. SnO_2 , Fe_2O_3 , etc.) possess many distinctive characteristics for electrochemical reactions, such as high surface area and shortened paths for fast lithium ion diffusion ^[2]. Herein, we introduce a facile procedure to synthesize 2D CuO nanosheets as an anode material of rechargeable LIBs.

Methods

For synthesis of CuO nanosheets, 2gr of $\text{C}_4\text{H}_6\text{CuO}_4 \cdot \text{H}_2\text{O}$ was dissolved in deionized water to form a solution of 0.1 M. The pH of the solution was adjusted to 14 with NaOH solution (0.1 M). The black precipitate was harvested by centrifugation, and repeatedly washed by deionized water and absolute ethanol for several times till pH reached 7.0. Subsequently, the washed precipitate was dried at 80°C for 16 h. Finally, the precursor was calcined at 500°C for 4 h.

Result and discussion

The XRD patterns of calcined CuO nanosheets are shown in Figure 1. XRD peaks confirm that the formation of CuO was in monoclinic phase and, there are no additional peaks of impurities, indicating that they are pure CuO. The characteristic main peaks located at $2\theta = 32.58^\circ$, 35.47° , 38.97° and 48.74° are assigned to (110), (002), (200) and ($\bar{2}02$) plane orientation of CuO (JCPDS 80-1268).

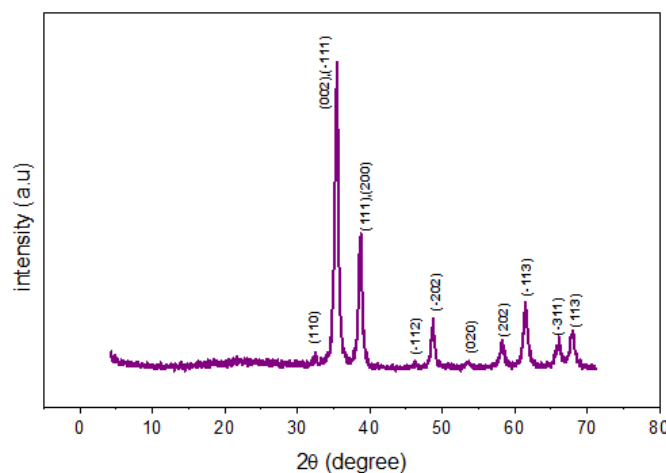


Figure. 1. XRD patterns of CuO nanosheets

Figure 2. is FE-SEM image illustrating the morphology of the product. The results indicate that sheet-like CuO were formed. EDS was carried out on the obtained CuO nanosheets (figure 2). The result of EDS confirms the presence of Cu and O peaks, and that the atomic ratio of Cu to O is 3:1. The specimens were coated by gold (Au) before FE-SEM observation.

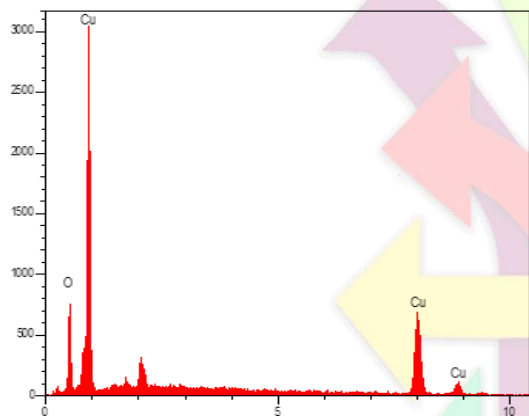


Figure. 2. EDS spectrum of the CuO nanosheets

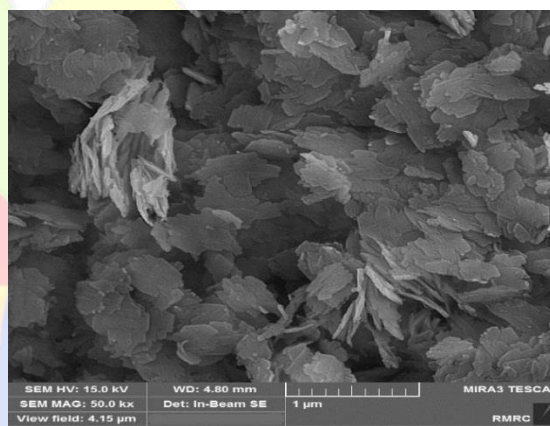


Figure. 3. FE-SEM of CuO nanosheets

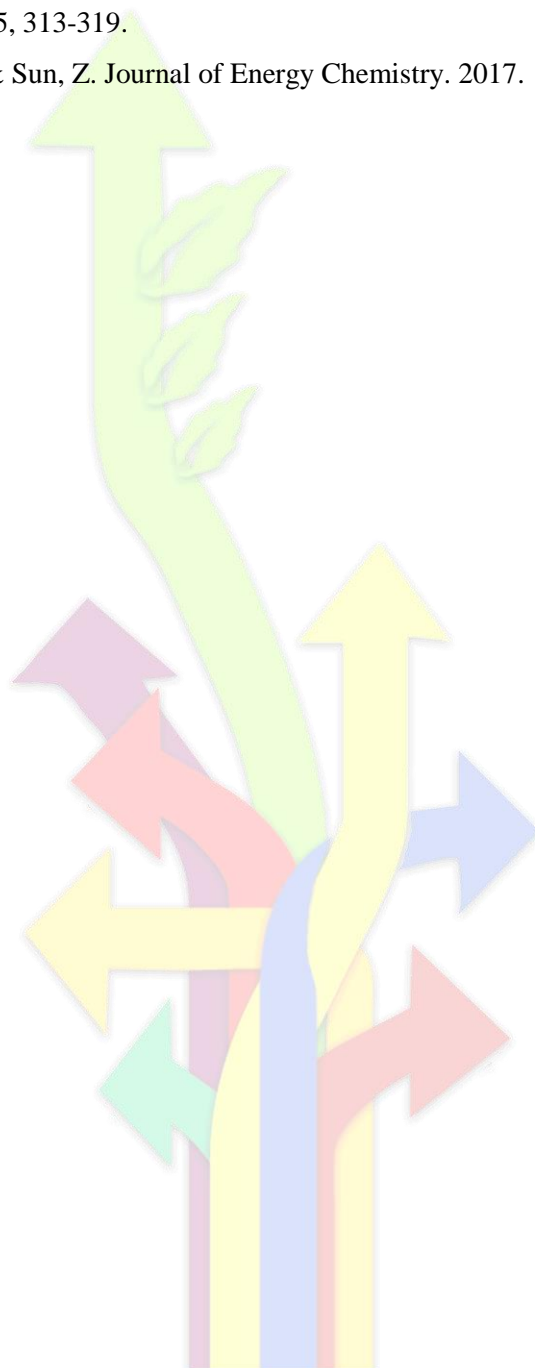
Conclusion

In summary, CuO nanosheets were successfully synthesized by a facile precipitation method. XRD and FE-SEM results confirms good formation of CuO nanosheets; therefore, CuO nanosheets can be introduced as a good candidate for anode materials of LIBs owing to the appropriate properties of 2D nanostructures metal oxides.

Keywords: CuO, Nanosheets, Precipitation method, Anode, Lithium-ion battery.

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Investigation of Electrical Surface Film in the Macroporous Silicon-based Anodes in Lithium ion Batteries

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Abstract

Silicon macroporous as an attractive anode for rechargeable lithium ion Batteries was synthesized via magnesiothermic reduction. Purity and honeycomb morphology of the macroporous silicon were confirmed by XRD and FE-SEM analyses. The electrochemical results showed the pseudo capacitive behavior of silicon at 10 mV/s scan rates, assigning to store electrostatically electrons on the silicon surface.

Keywords: *Lithium ion Battery, Silicon, Macroporous, cyclic voltammetry.*

Introduction

Lithium ion batteries are the most advanced secondary electrochemical systems for reversible energy storage. High capacity, power density, safety and cycle ability are crucial properties of LIBs. Graphite as the first anode material that have been commercialized firstly by Sony in 1991 suppressed of its low capacity of 372 mAh/g. So, silicon with 4200 mAh/g theoretical capacity as a promising anodic candidate was introduced instead of graphite. Nonetheless, surface electrode-electrolyte interface (SEI) layer that have been formed through decomposition of electrolyte compounds on the silicon surface as well as electrical double layer led to increase charge transfer resistance, decrease lithium-ion diffusivity, and increase the capacitive capacity. Herein, the capacitive capacity of macroporous silicon were investigated by cyclic voltammogram [1].

Methods

A specific weight of SiO₂ powder were mixed together Mg powder with mole ratio of 1:2.5 and pressed. The green compact was placed into the alumina crucible and carried into the tubular furnace at 670 °C and heated for 2.5h. The collected brown powder acid leached by 1M HCl and HF for removing the impurities and dried into the vacuum oven at 100 °C. Next, working electrode was prepared with loading mass of 1 mg comprising of 80 wt.% silicon, 15 wt.% carbon black and 5 wt.% PVDF binder dissolved in N-methyl-2-pyrrolidone (NMP) on copper foil. Electrochemical measurements were carried using CR2032 coin cell by lithium foil as the cathode, Celgard polyethylene membrane as the separator and 1M LiPF₆ dissolved in EC/DMC (1:1 v/v) as the electrolyte. The galvanostatic charge-discharge testing and cyclic voltammetry (CV) curves at a scan rates of 1, 5, and 10 mV s⁻¹ in the potential range of 0-2 V (vs. Li⁺/Li) were conducted on Potentiostat PGS 2065.

Results and discussion

The XRD pattern of sample in Fig. 1-a presented pure silicon with FCC lattice and lattice constant of 5.43 \AA , matching with 27-1402 reference card. After Rietveld refinement crystallites size at (111), (220), (311), (100), and (311) was calculated to be 70, 60, 50, 50, and 50 nm using Scherrer formulae. FE-SEM micrograph in Fig. 1-b clearly showed honey-comb pores with uniform distribution. As seen in Fig. 1-c, the first specific charge/discharge capacity delivered to 100 mAh/g and 50 mAh/g at current density of 200 mA/g. Loss of 50% of discharge capacity is due to SEI surface layer formation.

In Fig. 1-d, CV curve at scan rate of 1mV/s showed no redox peak but one anodic peak at 0.5 V at scan rate of 5 mV/s assigned to dealloying of Li_2Si to c-Si. With increasing scan rate to 10 mV/s presented one reduction peak at 0.3 V for diffusion lithium ions into the silicon lattice for formation Li_2Si alloy. Beside, rectangular-like shape of curves at higher scan rates connected to store the electrostatic charge on the silicon surface and pseudo capacitive behavior. Thereby, diffusivity of lithium ions into host lattice were restricted and loss of specific capacity was observed.

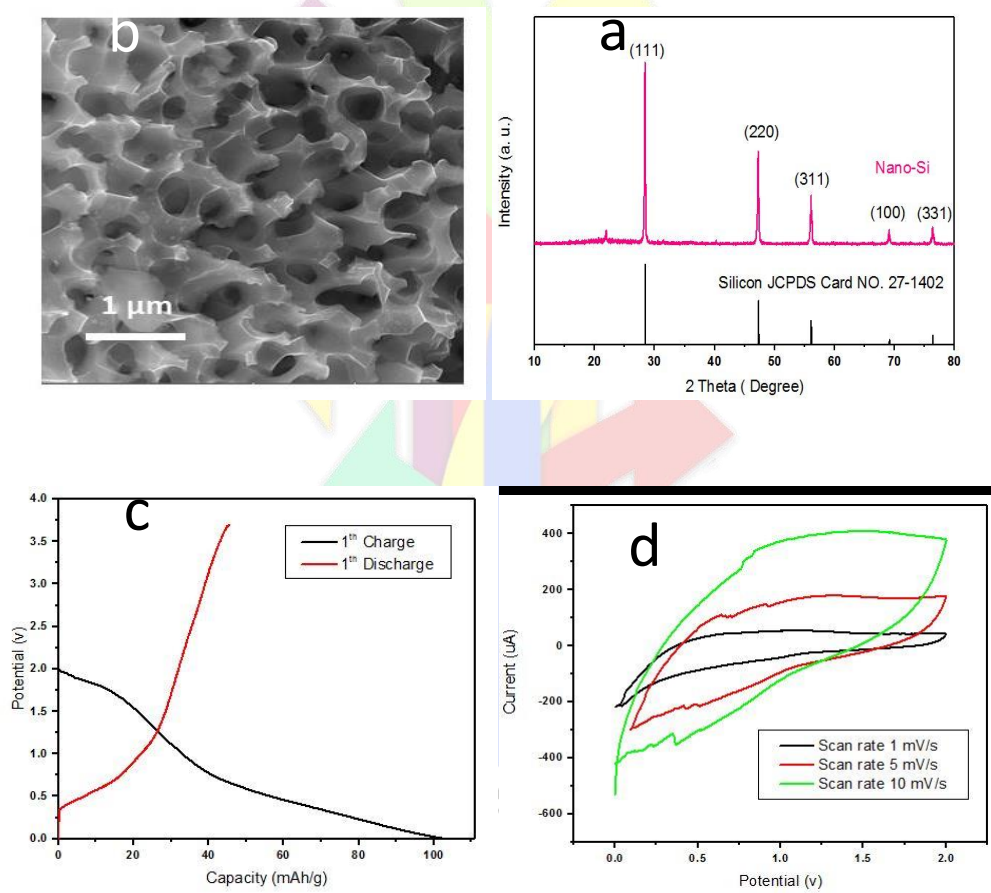


Fig1. a) The XRD pattern, b) FE-SEM micrograph, c) charge/discharge profiles at current density of 200 mA/g, d) cyclic voltammogram at the different scan rates in the potential range of 0-2V.

Conclusion

Silicon macroporous was synthesized via magnesiothermic reduction at 670°C during 2.5h as an anode material for lithium ion Batteries. Honeycomb morphology of pure silicon was confirmed using FE-SEM analysis. The electrochemical results delivered the first charge/discharge capacity of 100 mAh/g and 50 mAh/g at current density of 200 mA/g. In addition, pseudo capacitive behavior of silicon at high scan rates assigned to store electrostatically electrons on the silicon surface.

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Investigation of optimizing electrospun carbon nanofibers as an anode for Lithium ion batteries

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Abstract

The utilization of green energy, including solar and wind power, is believed to be one of the most promising alternatives to support more sustainable economic growth. In this regard, lithium-ion batteries (LIBs) can play a critically important role. To further increase the energy and power densities of LIBs, silicon anodes have been intensively explored due to their high capacity, low operation potential, environmental friendliness, and high abundance. Additionally, CNFs show excellent lithium- and sodium-storage performance when used directly as anode materials *via* template and activation strategies to produce numerous intercalation sites. nanofibers of Carbon with diameters that achieved into nanometer range have attracted growing attention in recent years due to their superior chemical, electrical, mechanical and also sacrificial properties in combination with their unique 1D nanostructures [1-2]. Unlike other synthesis methods, electrospinning polyacrylonitrile (PAN) followed by stabilization and carbonization has become convenient route to make carbon nanofibers [3]. Electrospinning utilizes electric forces and hence the electrical properties of the solution it's a direct process to produce polymer fibre with high specific surface area ratio [4]. In addition, This method is a direct process that produces continuous fibers with nano-sized diameters and high specific surface area ratios Apart from polymer fiber producing; which the benefit of this process is the fiber can be produced straight away with lower cost than conventional melt spinning process. Recently, successful attempts have been made to produce polymer fiber by adjusting the parameters of electrospinning such as the collector distance, needle size, polymer concentration voltage applied [4-6]. In this study we're willing to illustrate the effect of conductive additive, temperature, and solution concentration on optimizing the electrospinning process with polyacrylonitrile (PAN) to reduce diameter size, enhance mechanical properties, and

improve orientation (alignment) of the nano-fibers. Polyacrylonitrile (PAN) dissolved in dimethylformamide (DMF) was used as a precursor. Fiber diameters and orientation were measured through Scanning Electron Microscope analysis. Fibers with diameters of ~80-150 nm were successfully collected. For diameter analysis, voltage is found to be the most significant parameter in determining fiber diameter. Flow rate of syringe pump is the less-significant factor. The parameters were examined between voltage of 10-25kV, weight percentage PAN of 8-15wt%, and flow rate of 1-4ml/hr. and the temperature of precursor synthesis was 50-90 celsius. The fibers exhibited fine surfaces and were homogenous in terms of cylindricity. we were successful to find collaborations between the parameters with the required outputs.

Keywords: carbon nanofibers, Electrospinning, optimization, Li ion batteries

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Electrochemical and Morphological Characterization of Zn-Al-Cu Layered Double Hydroxides as a Negative Electrode in Aqueous Zinc-Ion Batteries

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The zinc electrodeposition efficiency is a primary key for improving long-term cycling of aqueous zinc-ion batteries. In order to improve the efficiency of electrodeposition in this type of batteries, it is possible to use layered double hydroxides (LDHs) as the negative electrode. For this purpose, Copper-doped Zn-Al-CO₃ layered double hydroxides (LDHs) have been synthesized by co-precipitation method under constant pH. The X-ray diffraction patterns in combination with scanning electron microscope images declare that the as-synthesized LDHs are well crystalline and hexagonal platelet-like. LDH was mixed with zinc powder in different ratios and the electrochemical performance of the mixtures were characterized by galvanostatic cycling with potential limitation (GCPL) at different current rates. The results show that in an appropriate combination of zinc and LDH, the electrodeposition efficiency of zinc can increase from 88% to 98%. Moreover, this electrode shows a proper efficiency of zinc electrodeposition at high rate of charging and discharging which is a vital parameter for grid-scale energy storage.

Electrochemical Characterization of Organic Electrolyte Additive on Zinc Electrodeposition Mechanism in Aqueous Zinc-Ion Batteries

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The efficiency of zinc electrodeposition is of fundamental importance for improving long-term performance of aqueous zinc-ion batteries. To improve the efficiency of zinc electrodeposition, it is possible to use organic additives in the electrolyte. Here, the effect of branched polyethyleneimine (BPEI) as an electrolyte's additive on the kinetics and morphology of zinc electrodeposited layer in 0.5 M of ZnSO_4 solution is investigated. The presence of BPEI changes the morphology of the electrodeposited layer from hexagonal large crystals to compact layer without preferential growth morphology. Moreover, we observed that BPEI adsorption on the surface of the substrate suppresses the kinetic of zinc electrodeposition and decreases the grain growth rate, thus favoring the nucleation over the growth. Finally, BPEI ensures a homogeneous distribution of the current densities and can guarantee uniformity of the deposited layer.

Facile synthesis silicon-based nanosheets as anodes for lithium ion batteries

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Introduction

Silicon (Si)-based materials have the highest capacity among the investigated anode materials and have been recognized as one of the most promising materials for lithium-ion batteries. However, it is still a significant challenge to obtain good performance for practical applications due to the huge volume change during the electrochemical process. To date, the most successful strategy is to introduce other components into Si to form composite or nanostructures[1]. In this research, reviewed the effect of heating rate for fabrication silicon nanosheets .

Methods

1 g of $\text{CaSi}_2/\text{SnCl}_2$ was mixed with 10 g of a eutectic composition of LiCl/KCl and milled homogeneously by using a mortar and pestle in an argon-filled glove box. The powdered mixture was then filled into a ceramic crucible and sintered at 400 °C under an Ar atmosphere. All the obtained products were washed with water or ethanol, and then dried in a vacuum furnace at 80 °C for over 24 h[2].

Results and discussion

Figure 1 shows the effect of the precursor morphology on the synthesis compound. It is obvious that porosity resulting from washing with NaOH in the precursor plays an effective role in the uniformity of the nano sheets. However, it is observed in the structure that nanosheets are stucked together due to the high heating rate. Therefore, the effect of heating temperature on the initial mixture was investigated for the sheets layering. In figure 2, It was observed that when the heating rate decreased from 10 to 3, homogenous and completely uniform nanosheets were synthesized. Two-dimensional silicon nanosheets are expected to increase the capacity of today's batteries compared to conventional batteries.

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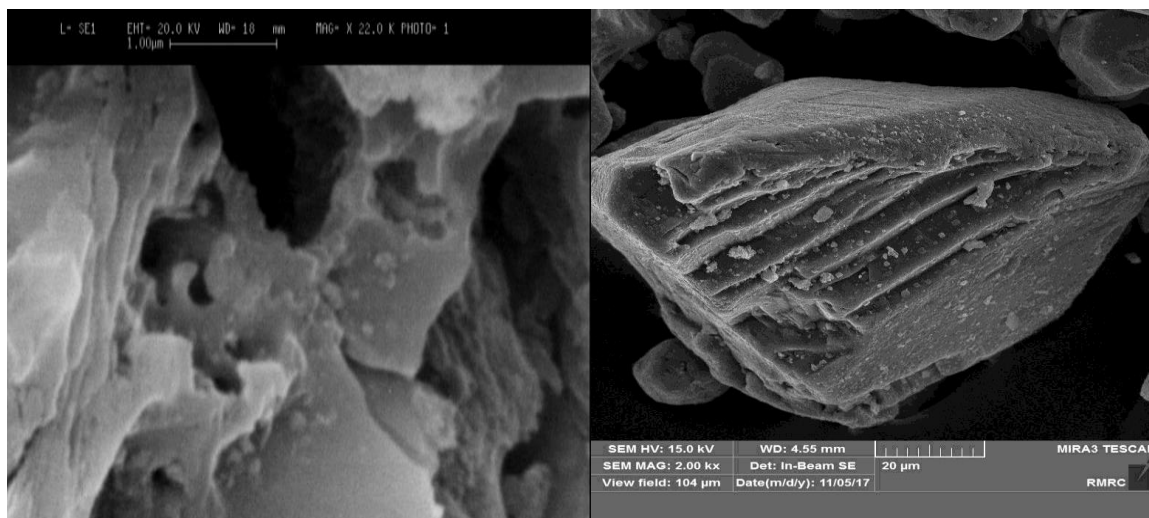


Figure.1. first sample

figure.2. second sample

Conclusions

We used topochemical process for fabrication silicon nanosheets as an anode material for LIBs. The both samples were under 400 °C for 5 h. SEM pictures showed that the nanosheet is formed uniformly in rate of three degrees per minute.

Keywords: *lithium ion batteries – Silicon nanosheets - topochemical*

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Composition and Manufacturing Effects on Electrical Conductivity of Li/CoS₂-FeS₂ Lithium reserve Battery Cathodes

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Abstract

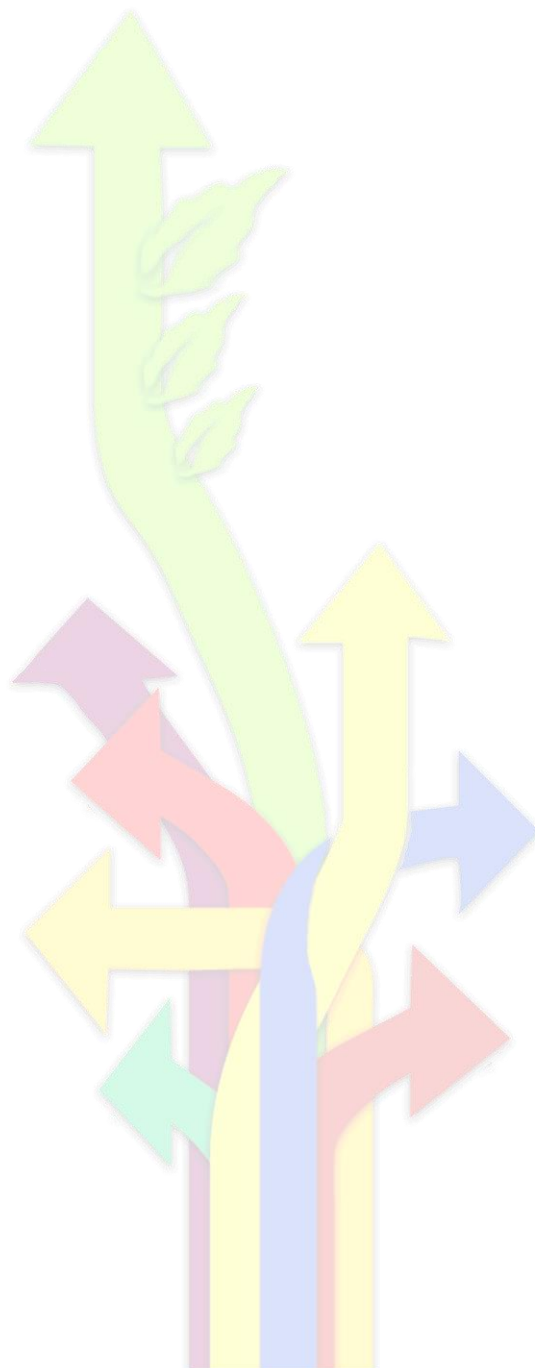
Electrical conductivity is key to the performance of Lithium reserve Battery cathodes. In this work we present the effects of manufacturing and processing conditions on the electrical conductivity of Li/ CoS₂-FeS₂ thermal battery cathodes. We use finite element simulations to compute the conductivity of three-dimensional microcomputed tomography cathode microstructures and compare results to experimental impedance spectroscopy measurements. A regression analysis reveals a predictive relationship between composition, processing conditions, and electrical conductivity; a trend which is largely erased after thermally-induced deformation. The trend applies to both experimental and simulation results, although is not as apparent in simulations. This research is a step toward a more fundamental understanding of the effects of processing and composition on thermal battery component microstructure, properties, and performance.

Keywords: Thin layer, lithium reserve battery, flexible electrode, spray pyrolysis, printing techniques

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Synthesis of cathode active material with (NMO) composition by modified co-precipitation method for use in lithium ion batteries.

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Abstract

Nowadays high C-rate and long life cycles in lithium ion batteries are more considered as a power plant according to industry's interest for high power applications such as electrical motors. LIB cathode material and its characteristics is the critical element from this point of view. In this research, the cathode active material (NMO) with a composition (Li Ni_{0.5} MnO_{0.5} O₂) has been synthesized in a co-precipitation modified method to increase the capacity and prevent the loss of capacity in long life cycles. For this purpose, the concentrated gradient method has been used to synthesize the active cathode material; with this design, the reaction speed is increased at the surface of the particles and in the center of particles there are high stability compounds to prevent the reduction of battery performance in large cycles. Phases are identified by XRD. BET has also been used to obtain the specific surface of the synthesized material. The study of surface morphology, particle distribution, ... has also been performed by scanning electron microscopy (SEM) images. The capacity of the cathode synthesized active substance is obtained by the Autolab Potentiostatic Device, and the battery tests, including operating voltage, cyclic test and battery life, have been performed by the kimia Stat battery tester. The results indicate that NMO is well synthesized by composition (Li Ni_{0.5} MnO_{0.5} O₂) and has a capacity of 150 mAh/g in high cycles.

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Fabrication of Polypyrrole-Graphene oxide / Nickel Oxide nanocomposite and investigation of their electrochemical treatment in supercapacitor

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Abstract

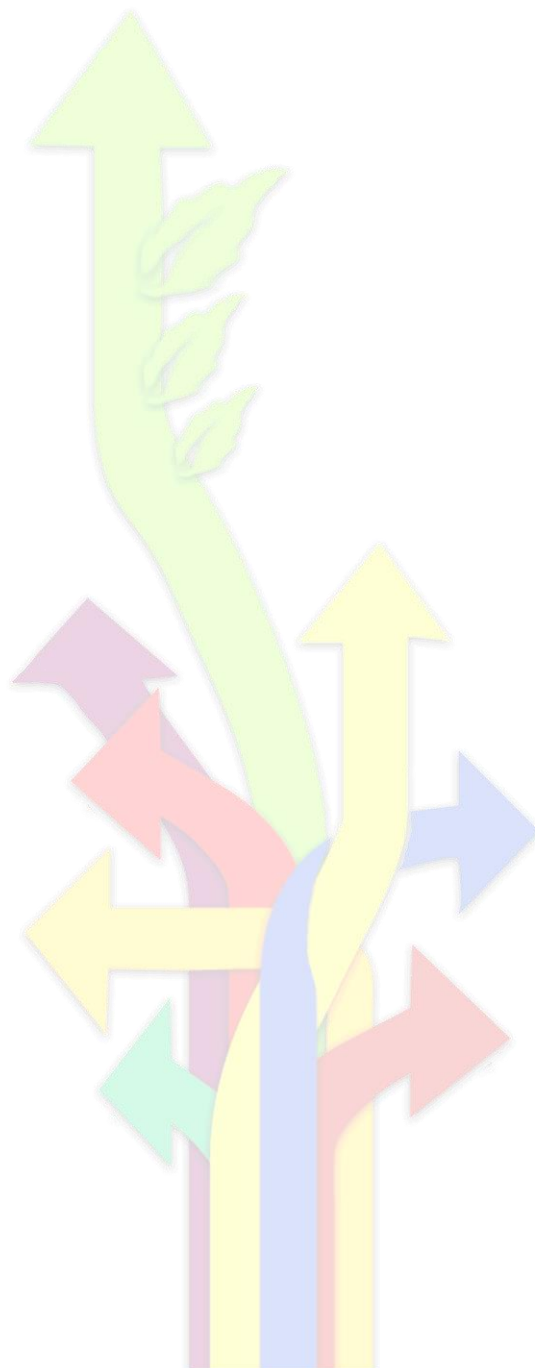
One of the great challenges in the twenty-first century is unquestionably energy storage. In response to the needs of modern society and emerging ecological concerns, it is now essential that new, low-cost and environmentally friendly energy conversion and storage systems are found; hence the rapid development of research in this field. The performance of these devices depends intimately on the properties of their materials. Supercapacitors and batteries are two most important technologies which are being viewed as possible solutions for storing the energy generated using alternative sources of energy. Charge transfer in supercapacitors are highly reversible and have long cycle life due to suitable power and energy density. In general, electrodes of electrochemical supercapacitors are made of nano-materials with high surface area and high porosity. Charges can be stored and separated at the interface between the conductive solid particles (such as carbon particles or metal oxide particles) and the electrolyte. This surface can act as a capacitor with a capacitive double layer electrical conductivity.

The present study is aimed at the preparation of polypropylene, graphene oxide and nickel oxide nanocomposites for the study of its capacitive behavior. For fabrication of mentioned nano composite we used from electrochemical methods. Its capacitive behavior has also been studied by cyclic voltammograms techniques, galvanostatic charge-discharge tests and electrochemical impedance spectroscopy. The results showed that, the presence of graphene oxide in the electrode composition due to its high surface area improves the electrochemical properties of the electrode. On the other hand, the electrodeposition of nickel oxide also improves the capacitance of the electrode.

Keywords: Supercapacitor, Nano Composite, Nickel oxide, Graphene oxide, polypyrrole

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- (1) Wu, M.; Huang, Y.; Yang, Ch.; Jow, J. hydrogen. energy, **2007**, 32, 4153.
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13th Annual Electrochemistry Seminar of Iran

Electrosynthesis of the CdMn_2O_4 Nanoplates as High Performance Supercapacitor by Controlled Current Density

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Abstract

The ever-growing demands for high capacity energy storage devices have prompted worldwide research community to develop an advanced high performance energy storage system. Supercapacitors, also known as electrochemical capacitors, have attracted much attention in recent years due to their significant potential for energy storage and electric vehicles. Several mixed transition metal oxides such as Fe_3O_4 , Co_3O_4 , NiO , Mn_3O_4 , CuCo_2O_4 , ZnMn_2O_4 , CoMn_2O_4 etc., have been explored as electrode material for energy storage device. In this work, Cd-Mn binary oxide nanostructures have been prepared by cathodic electrodeposition of mixed Cd/Mn nitrate solutions. The electrodeposition step has been carried out galvanostatically at current density of 4 mAcm^{-2} . The synthesized nanostructures were characterized by X-ray diffraction (XRD), Fourier Transform Infrared (FTIR) spectrometry and Scanning Electron Microscopy (SEM). The storage ability of the samples was investigated by cyclic voltammetry in 1M KOH aqueous solution at different scan rates ranging from 1 to 100 mVs^{-1} . High capacitance of 210 Fg^{-1} for the sample prepared can be achieved at the scanning rate of 1 mv/s. The proposed method provides a facile, cost effective and high performance strategy for the synthesis of CdMn_2O_4 for supercapacitor applications.

Keywords: Electrosynthesis, Nanoplates, Cd-Mn Binary oxide, Supercapacitor

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Ni₃(VO₄)₂ Nanoparticles for High-Performance Supercapacitor Applications

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Abstract

Nickel vanadate Ni₃(VO₄)₂ nanoparticles were successfully synthesized by a sonochemical method. The characterization of this material was examined by Fourier transform infrared spectroscopy (FT-IR), energy dispersive X-ray diffraction (EDS), field emission scanning electron microscopy (FE-SEM), Raman spectroscopy X-ray diffraction (XRD), and thermal gravimetric analysis (TGA). The prepared sample was coated on nickel foam. The capacitive behavior of nanomaterials were investigated by cyclic voltammetry (CV), galvanostatic charge/discharge test and electrochemical impedance spectroscopy (EIS) by a three-electrode system in an aqueous solution of 6 M potassium hydroxide as the electrolyte. The electrochemical properties were studied at various scan rates in the potential range of 0.0 - 0.55 V. Electrochemical testing showed that Ni₃(VO₄)₂ nanoparticles performance with high specific capacitance ~ 400 F g⁻¹ at 2 A g⁻¹ with enhanced rate capability and excellent capacity retention more than 85% after 1000 consecutive charge–discharge cycles.

Keywords: Ni₃(VO₄)₂, Nanoparticle, Sonochemical, Supercapacitor, Nickel foam

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Evaluation of charge storage ability of zinc doped Co₂O₃ nanostructures derived by cathodic electrodeposition

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Abstract

Supercapacitor, as one of the promising advanced energy storage devices, has attracted considerable attention because it can offer much higher charge/discharge rate than batteries, higher energy density than conventional capacitors, and long cycle life and environmental friendliness. In this work a facile synthetic route has been proposed to prepare nanostructures of Zn doped Co₂O₃. The synthesis was carried out by constant current cathodic electrodeposition from Co²⁺ nitrate solutions. X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) were used to characterize the nanostructures. The storage ability of the obtained nanostructures was investigated by cyclic voltammetry (CV) in 3 M KOH solution. The results indicated that the Zn doped cobalt oxide material shows better performance than the non-doped one, and the charge capacity (SC) of doped cobalt oxide (630 F/g) was higher than pristine cobalt oxide (211 F/g).

Keywords: Electro synthesis, Nanoparticles, Energy storage, Supercapacitor.

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Carbon/CoFe₂O₄ composite nanoparticle for energy storage

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Abstract

supercapacitors with excellent cycle life, rapid charging and discharging, wide thermal operating potential range, low cost, eco-friendly and safety able to storage electrochemical energy like batteries and fuel cells [1]. According to the characteristics of supercapacitors, they can be used in different applications requiring high power density such as consumer electronics, memory back-up systems and smart grids [2]. In this work, we report the studies concerning the structure and electrochemical properties of carbon/CoFe₂O₄ composite nanoparticle produced by co-precipitation. The prepared samples were characterized by means of X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM). The electrochemical performance of the material is investigated by cyclic voltammetry (CV), galvanostatic charge/discharge cycling and electrochemical impedance spectroscopy (EIS). The results indicate that carbon/CoFe₂O₄ composite nanoparticle a good supercapacitive behavior.

Keywords: Carbon composite, energy storage, metal oxide, nanoparticle

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Studying the Supercapacitive behavior of Co_3O_4 Decorated on the Chitosan/GM

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Abstract

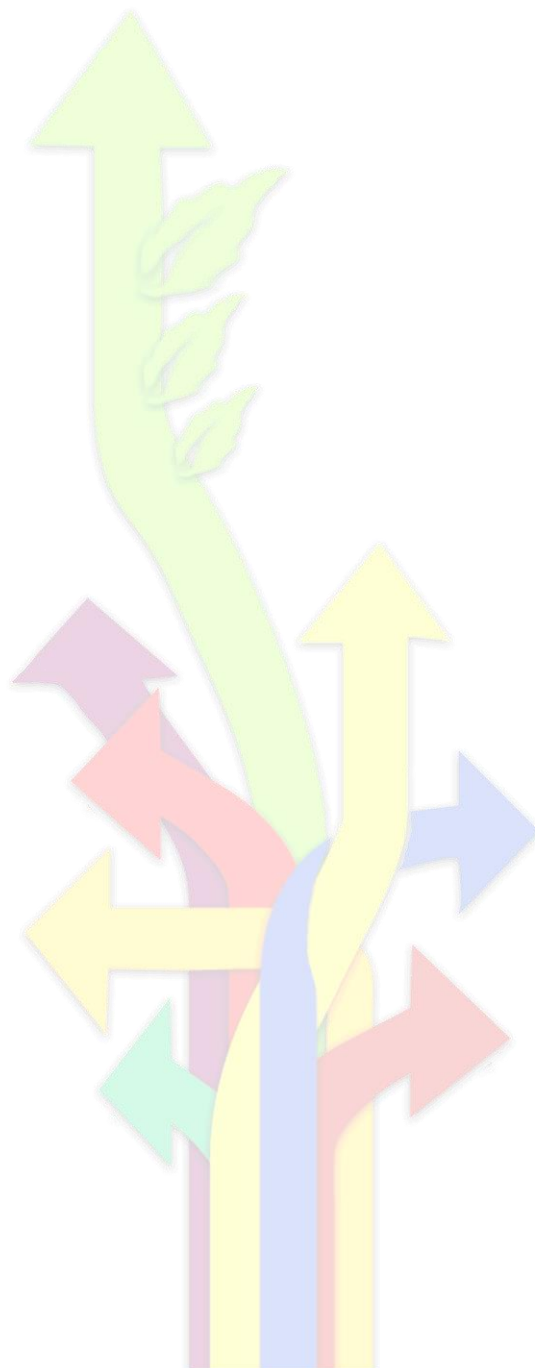
Supercapacitors have been in the center of attention because of their high power density, long cycle life ($>100\ 000$ cycles), and rapid charging-discharging rates. According to the mechanism through which they store the charge, they are basically classified into two types: electric double layer capacitors (EDLCs) based on carbon electrodes and pseudo-capacitors with certain metal oxides (RuO_2 , IrO_2 , Co_3O_4 , MnO_2 , NiO) or conducting polymers as electrode materials [1-4]. Co_3O_4 is being studied as a candidate material for pseudo-capacitor electrode because it has a high theoretical specific capacitance and good electrochemical capability [5-7]. In this work we synthesized urchin-like Co_3O_4 , which is composed of nanorods, deposited on Chitosan/GO-MWCNT (CS/GM) as a template, via simple hydrothermal method. The synthesized nanocomposite has been characterized by Field emission scanning electron microscopy (FE-SEM), X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), as well as energy dispersive X-ray spectroscopy (EDX). The electrochemical performance of CS/GM/ Co_3O_4 was studied by cyclic voltammetry (CV), galvanostatic charge-discharge (CD) and electrochemical impedance spectroscopy (EIS) techniques in 0.5 M KOH. The high specific capacitance of $1632.28\ \text{F g}^{-1}$ was obtained. This nanocomposite not only exhibits a high specific capacitance but also have a good cycling stability even at high current density of $100\ \text{A.g}^{-1}$, which can keep capacitance retention of 90.82% after 500 cycles.

Keywords: Supercapacitor, Co_3O_4 , Electrochemical Impedance Spectroscopy, Specific Capacitance

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13th Annual Electrochemistry Seminar of Iran

Facile electrochemical synthesis of new ternary mixed metal oxide: characterization and energy storage studies

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Abstract

Nowdays, much of interest were focused on supercapacitors as one of energy storage devices due to their high power density, long life services and good cycleability. Much effort have focused on development of electrode materials to fabricating of supercapacitors with better performance [1]. Doping of two or more metals is one of the key ways to increase the saturation magnetization of oxide samples. Presence of two different transition metal ions simultaneously in a host material produces magnetic property that can be different from the magnetic property due to single transition metal ions [2]. On the other hand, using a facile and cost effective sythesis method is essential. Cathodic electrodeposition is an efficient route for synthesis of high pure mixed metal hydroxide/oxides. In this work, nano ternary metal oxide of Mn-Ce-Al was synthesized by means of cathodic electrodeposition method. The nano-structure characterization was studied by variety of methods including X-ray diffraction (XRD) and scanning electron microscopy (SEM). The ternary metal oxide will be used for fabricating supercapacitors.

Keywords: Supercapacitor, Mixed Oxide, Doping, Electrodeposition

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Fabrication of Polypyrrole-Graphene oxide / Nickel Oxide nanocomposite and investigation of their electrochemical treatment in supercapacitor

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Abstract

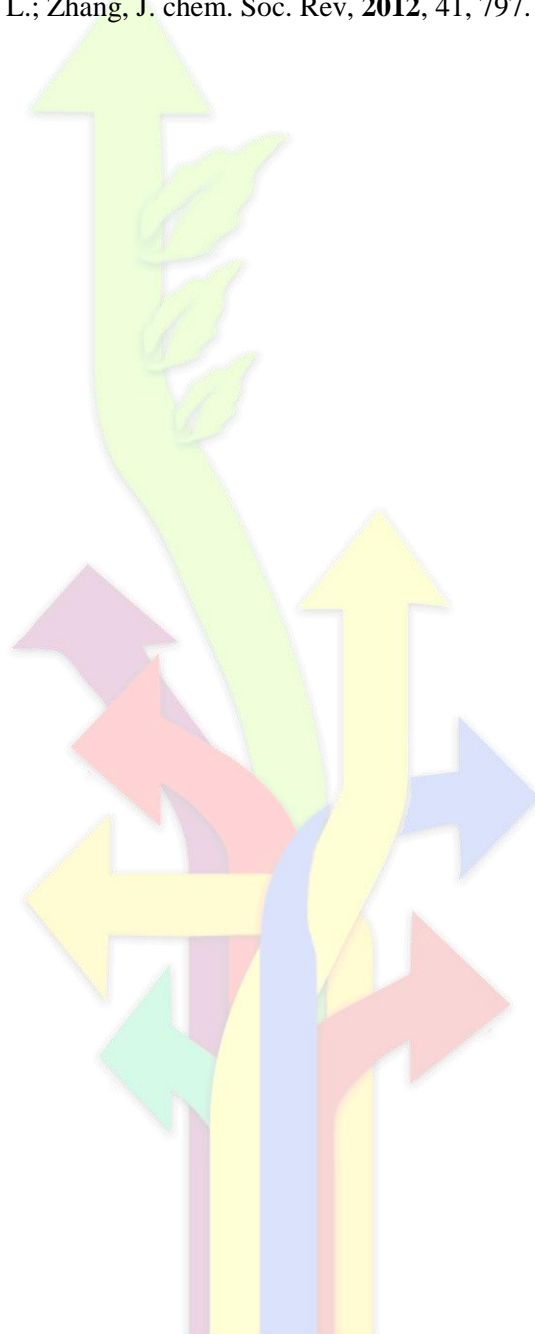
One of the great challenges in the twenty-first century is unquestionably energy storage. In response to the needs of modern society and emerging ecological concerns, it is now essential that new, low-cost and environmentally friendly energy conversion and storage systems are found; hence the rapid development of research in this field. The performance of these devices depends intimately on the properties of their materials. Supercapacitors and batteries are two most important technologies which are being viewed as possible solutions for storing the energy generated using alternative sources of energy. Charge transfer in supercapacitors are highly reversible and have long cycle life due to suitable power and energy density. In general, electrodes of electrochemical supercapacitors are made of nano-materials with high surface area and high porosity. Charges can be stored and separated at the interface between the conductive solid particles (such as carbon particles or metal oxide particles) and the electrolyte. This surface can act as a capacitor with a capacitive double layer electrical conductivity.

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Keywords: Supercapacitor, Nano Composite, Nickel oxide, Graphene oxide, polypyrrole

Reference

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Electro-oxidation of ethanol on copper in alkaline solution

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Abstract

The electro-oxidation of ethanol on copper in alkaline solutions has been studied by the method of cyclic voltammetry. The electro-oxidation of ethanol was studied on the surface of the pre-treatment copper electrode in the sodium hydroxide solution. The main purpose of this research is promotion of electrocatalytic property of copper which can be used as an anode in the fuel cell and is usable in many industries like military, automotive and aerospace.

Keywords: Ethanol Oxidation, Copper, Ethanol, Voltammetry.

1. Introduction

The electrochemical oxidation of alcohols as a fuel in fuel cells requires the use of a catalyst to achieve the high current densities necessary for practical applications. Among the different small organic compounds, ethanol is considered as an ideal fuel because it is easy to handle and to store and widely available.

2. Experimental

All the chemicals used in this work were analytical grade of Merck origin and were used without further purification. All solutions were prepared by doubly distilled water. The electrochemical measurements were done by a computer-controlled potentiostat/galvanostat (Auto Lab, PGSTAT302N). A dual Ag/AgCl-saturated KCl, a platinum wire and a copper disk electrode were used as the reference, counter and working electrodes, respectively. All experiments were performed at room temperature (25 ± 2 °C).

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3. Results and discussion

Cyclic voltammograms of pre-treatment copper electrodes in 1 M NaOH solution containing 0.5 M ethanol are presented in Fig. 1 at scan rate of 10 mV s^{-1} . As seen, ethanol oxidation current increases considerably for all surface treatments. The current response for the pre-treatments including copper deposition and corrosion in the presence of NaCl solution is higher than the one obtained for the electrode without surface treatment and also for the electrode after corrosion in H_2SO_4 . So, this treatments produce a higher surface area and active sites for electro-catalytic ethanol oxidation.

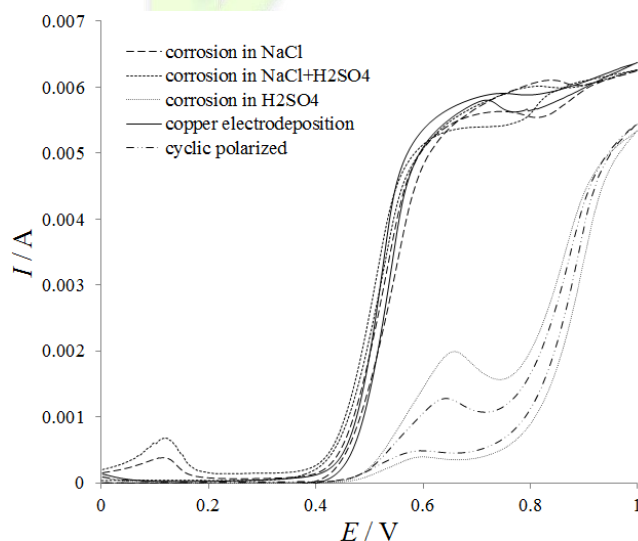


Fig. 1. Cyclic voltammograms for ethanol oxidation on copper electrode after different surface treatments. Solution contains 1 M NaOH and 0.5 M ethanol .Scan rate: 10 mV s^{-1} .

4. Conclusion

This work presents the electro-oxidation of ethanol on poly-crystalline copper in alkaline solution. It is concluded that the electro-oxidation of ethanol that starts around 640 mV/Ag, AgCl occurs through a mediated electron transfer mechanism catalysed by Cu^{III} species which form in the same potential region.

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Pt₃Ni supported on Co₃O₄ as a high performance electrocatalyst for ORR in alkaline fuel cells

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Abstract

Alkaline fuel cells as a promising energy sources have attracted remarkable interest amongst low-temperature fuel cells due to enhanced electrocatalysts stability and cell durability. Furthermore, operation in alkaline media allows use of fuels other than hydrogen, such as alcohols. Different kind of catalyst materials have been investigated as an electrocatalyst for fuel cell reaction. Platinum (Pt) is known as an affective electrocatalyst with great activity for oxygen reduction reaction (ORR), good electronic conductivity and stability. Nevertheless, high cost of Pt limits its extensive use in fuel cells. One approach to reduce the amount of Pt is to incorporate it with other transition metals such as nickel, cobalt, iron or copper. In this regard, PtNi/C has been extensively studied for various important electrochemical reactions, including ORR. Sakamoto et al. reported the ORR performance of Pt₃Ni to be 10 fold higher than that of bare Pt in acid media. However, it is well-known that the rate of the electrochemical reaction is not only significantly affected by the catalytic activity of the used metal but also by the employed catalyst support. Carbon materials generally used as supports, such as Vulcan, offer high specific surface area, but have low involvement towards ORR. In this work, PtNi NPs were supported onto Co₃O₄ prepared by hydrothermal method. The probable synergistic phenoamena between transition metal oxide (Co₃O₄), Pt and Ni will be studied, in exploration of catalyst simultaneously active for ORR. The cobalt (II, III) oxide-supported platinum (Pt-Ni/Co₃O₄) catalyst was prepared employing a conventional impregnation method. A determined amount of Co₃O₄ powder was added to the Pt and Ni precursor solution and homogeneously dispersed by sonication for 40 min. The resulting suspension was then heated in a furnace at 250°C for 5 h under air atmosphere. Powdered samples. were cooled to room temperature and thoroughly mixed by grinding. The samples milled above were further sintered in an air atmosphere at 450° C for 2 h.

The catalytic inks were prepared by dispersing each electrocatalyst in 125 μL of 2% solution of polyvinylidene fluoride (PVDF, Alfa Aesar) in *N*-methyl-2-pyrrolidone (NMP, Sigma–Aldrich) and then ultrasonically treating them for ca. 30 min. PVDF was used to bind the electrocatalyst particles onto the conductive support in 125 μL of 2% solution of polyvinylidene fluoride (PVDF, Alfa Aesar) in *N*-methyl-2-pyrrolidone (NMP, Sigma–Aldrich) and then ultrasonically treating them for ca. 30 min. PVDF was used to bind the electrocatalyst particles onto the conductive support. For comparison purposes, CVs of $\text{Pt}_3\text{Ni}/\text{Co}_3\text{O}_4$ and Pt/C were also recorded under the same conditions. The electrocatalytic performance of the as-prepared $\text{Pt}_3\text{Ni}/\text{Co}_3\text{O}_4$ alloy catalyst, compared with commercial Pt/C , and the results are as shown in Figure 1. CVs of PtNi alloy(Figure 1) show typical Pt-like H underpotential deposition features, while the reduction peaks ($\sim 0.86\text{ V}$ vs. RHE) in the cathodic scan for PtNi alloy have a positive shift compared to that of Pt/C catalyst ($\sim 0.77\text{ V}$ vs. RHE). This shift can be attributed to the reduced adsorption forte of oxygenated species ($-\text{O}$ or $-\text{OH}$) on alloy surfaces, and more essentially, due to the downshift of the d band center. The reduced surface coverage of oxides resulted in a considerable improvement in the ORR catalytic performance for PtNi alloy catalyst, just as demonstrated by the positive shift (27mV) of half wave potentials in the polarization curves.

Keywords: Oxygen Reduction, Pt alloy, cobalt oxide, Fuel cell

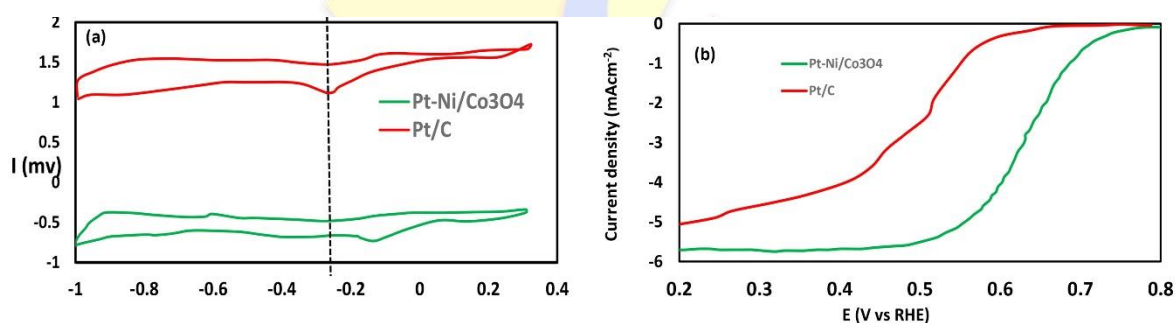
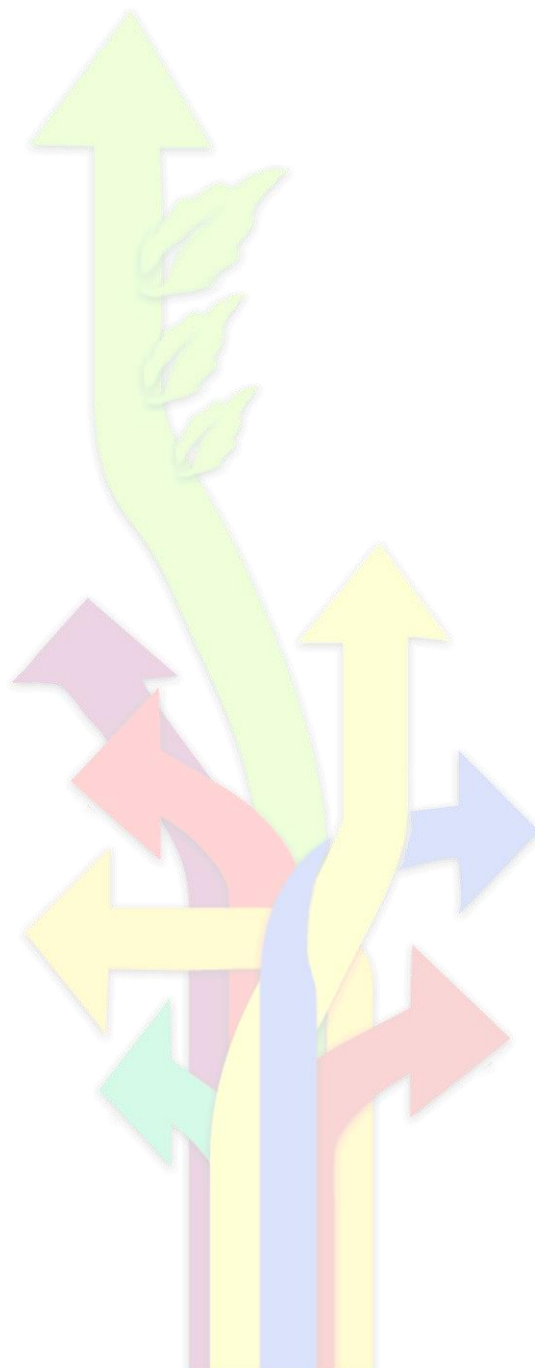


Figure 1.(a)cyclic voltammetry in argon-purged electrolyte; (b) polarization curves for the ORR; RDE electrochemical characterization in 0.1 M HClO_4

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Electrochemical characterization of electroplated Ni-Mo and Ni-Mo-P alloy coated stainless steel bipolar plates for PEMFC

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Abstract

Stainless steel bipolar plates (BPPs) are the preferred choice for proton exchange membrane fuel cells (PEMFCs); however, a surface coating is needed to minimize contact resistance and corrosion. In this research, Ni–Mo and Ni–Mo–P coatings were electroplated on stainless steel BPPs and investigated by SEM/EDS, AFM and contact angle measurements. The Ni and Mo alloy elements were selected because they are known to have high corrosion resistance [2]. The performance of the BPPs was studied by corrosion and conduction tests and by measuring their interfacial contact resistances (ICRs) *in-situ* in a PEMFC set-up. The durability of BPPs was investigated in a PEM single cell, using a commercial Pt/C Nafion membrane electrode assembly (MEA). The effect of the BPP coatings on the electrochemical performance up to 115 h was evaluated from polarization curves, cyclic voltammetry and electrochemical impedance spectroscopy. The results revealed that the applied coatings significantly reduce the ICR and corrosion rate of stainless steel BPP. The Ni–Mo coating showed the lowest and most stable ICR and the smallest effects on MEA performance, including catalyst activity/usability, cathode double layer capacitance, and membrane and ionomer resistance build up with time. After electrochemical evaluation, the BPPs as well as the water effluents from the cell were examined by Scanning Electron Microscopy, Energy Dispersive and Inductively Coupled Plasma spectroscopies. No significant degradation of the coated surface or enhancement in metal release was observed. However, phosphorus addition to the coating did not show to improve its properties, as deterioration of the MEA and consequently fuel cell performance losses was observed.

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2. Methodology

A commercial AISI 316L stainless steel plate was used as substrate. All samples surfaces were polished and then ultrasonically cleaned in ethanol followed by Milli-Q water. Ni–Mo and Ni–Mo–P coatings were electroplated on AISI 316L stainless steel from citrate–ammonia electrolyte baths. Then, the samples were electroplated using an electrolytic citrate bath.

3. Results

The Ni–Mo coatings deposited at current density of 100 mA cm^{-2} exhibits a water contact angle around 105° , which is significantly higher compared to bare stainless steel and also higher compared to graphite for which a 95° water contact angle was measured [2]. Thus, the results indicate that the water management potentially could be improved by using Ni–Mo coated 316L BPPs instead of graphite BPP.

4. Conclusion

To summarize, the BPP properties, including corrosion resistance, ICR and hydrophobicity, all affect the performance of the PEMFC depending on the superficial composition of the coatings. The superior performance of Ni–Mo (100) coated 316L BPP is argued to be associated to its high conductivity and corrosion resistance properties. Hence, the inferior performance of the uncoated 316L BPP can be related to high electrical resistance of the formed surface oxide layer and lack of corrosion resistance in the PEMFC harsh environment. The experiments show that the phosphorus element in the coating does not enhance the overall cell performance.

Keywords: *Bipolar Plate, Electroplating, Alloy Coatings, Fuel Cells, Electrochemical evaluation, Wettability*

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Measurement and controlling the residual solvent of sulfonated poly ether ether ketone proton exchange membrane

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Abstract

Introduction: Nowadays using renewable and clean energy is one of the targets of researchers. One of these modern energy convertor is proton exchange membrane fuel cell (PEMFC) with minimum harm to the environment. Proton exchange membranes (PEM) could be categorized into perfluorinated and nonfluorinated hydrocarbon based on the material. Sulfonated Poly-Ether-Ether-Ketone (SPEEK) which is a hydrocarbon PEM was used in this research because of its low cost, good accessibility and high thermal stability (1).

SPEEK membrane usually prepared by the solution casting method. In this method the polymer is dissolved in a solvent and casted. The solvent is supposed to evaporate through the casting process. However, an amount of solvent would remain in the casted membrane after drying that is called residual solvent (RS). It is noteworthy that RS content affects many of membrane electrochemical and structural properties and are the main focus of the recent studies in this field (2). Dimethylformamide (DMF), dimethylsulfoxide (DMSO), Dimethylacetamide (DMAc) and N-methyl-2-pyrrolidone (NMP), are the most commonly used solvents for casting membranes. These solvents interact differently with the membrane. Hence the amount of RS content in the membrane casted with each of them would differ. In this research the amount of RS in the membrane casted with various solvent is measured. Additionally, acidic and thermal treatment were used to reduce the RS.

Materials and methods:

Sulfonated poly ether ether ketone (SPEEK) was synthesized via post-sulfonation of PEEK using concentrated sulfuric acid (3). The DS of the polymer was evaluated 0.935 by ¹H NMR.

Membrane preparation:

Membranes were prepared using NMP, DMAc and DMSO as casting solvents. In a typical experiment SPEEK was dissolved in solvent in a 10:1 g L⁻¹ ratio. The resulting solution was mixed under continuous stirring for 24 h to obtain a homogeneous liquid. The solution was then coated on a glass plate.

Residual solvent measurement:

RS content measured from weight difference between solution before cast and casted membrane.

Results and discussion:

Results indicated that NMP remains more than other solvent in the prepared membrane (Table.1). This result is compatible with fact that this solvent interacts more with the polymer molecule (4). The more RS content of the membrane, the more properties are affected by the solvent. For instance, ion exchange capacity would decline dramatically if the RS is not removed.

Table.1: RS content in two conditions with different solvents

Solvent	As-cast membrane RS content (%)	Thermal treated membrane RS content (% _°C)
DMSO	52.95	34.28 (100 °C)
DMAc	49.67	30.53 (100 °C)
NMP	79.48	47.41 (120 °C)

In order to reduce the RS, different methods were used (Table.2). The most effective method was thermal treatment.

Table.2: different methods for RS reduction in SPEEK membrane casted with DMSO solvent

Thermal treatment (vacuum /temperature (°C) /time (h))	Acidic treatment (molarity (M)/time (h))	As-cast membrane RS (%)	Treated membrane RS (%)
Thermal treatment (not vacuum / 60 / 8)		59.85	57.04
Thermal treatment (vacuum / 100 / 12)		52.95	34.28

Thermal treatment (vacuum / 50 / 24)	55.38	33.48
Acidic treatment (1/12)	58.09	42.46
Acidic treatment (2/12)	56.52	39.54

Conclusion:

RS resulted from different treatment methods and casting solvents were investigated in this study. The results lay a basic foundation for further study to control the RS amount in the SPEEK membrane for enhancement of the desired properties.

Keywords: Residual Solvent, DMSO, DMAc, NMP, SPEEK, PEMFC

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Electrocatalytic Activity of Pd-Based Cathode Catalysts for Direct Methanol Fuel Cells

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Abstract

Palladium-based alloys supported on carbon were successfully synthesized and employed as cathode materials for improved oxygen reduction reaction (ORR) in direct methanol fuel cells (DMFCs). Several instrumental methods were used to investigate the physical properties of the prepared samples. The morphological characterizations exhibit that the alloyed nanoparticles were uniformly distributed on the substrate. The ORR catalytic performances of these Pd-based catalysts were excellent in comparison with the Pd/C catalyst in acidic environments. The results revealed that the Pd-based alloyed materials can be practiced as potentially effective, methanol-tolerant, highly durable, and low-cost replacements for Pt-based samples in DMFCs [1-3].

Keywords: *Palladium, Alloys, Oxygen reduction reaction, Direct methanol fuel cells.*

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A series of nickle-cobalt binary alloys with different atomic ratio on graphite electrode for methanol and ethanol oxidation

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Abstract

Direct methanol fuel cell (DMFC) has been considered greatly because of methanol reasonable price, it's also easily handle and stored.[1] However it is toxic, inflammable and non-renewable.[2] So ethanol is a good alternative feul to methanol and it doesn't have the drawbacks of methanol[3]

One of the impotant keys in direct liquid fuel cell(DLFC) is electrocatalyst. So finnding a new catalyst with greater performance and lower poisoning is necessary. Demirci [3] attempted to suggest new binary alloy as catalyst for direct liquid feul cell. His works were based on theories about metal d-shift band center and the segregation. One of the studied binary alloy by Demerci [3] is Ni-Co which has reasonable price and seems to be potentially good catalyst.

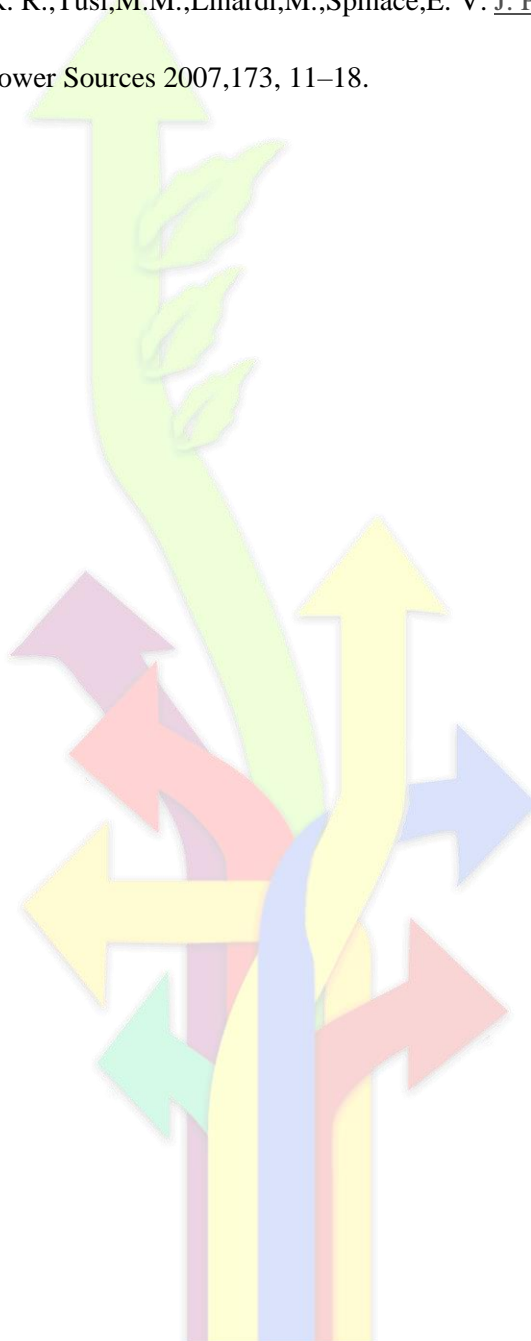
In this work the electro-oxidation of methanol and ethanol were studied on the Ni-Co binary alloys on the graphite with varying the atomic percentages in the alloys. The catalysts were prepared by cycling over cathodic potential. The catalytic performances were investigated by cycling voltammetry (CV), chronoamperometry (CA) and electrochemical impedance spectroscopy (EIS).

The results show the catalyst which has more cobalt ratio has significant higher response for both ethanol and methanol oxidation. On the other hand, the current magnitude of methanol oxidation is greater than ethanol in the same concentration. it seems that the future tracks for searching active catalyst are examining the other bimetallic alloys which seem to be active catalysts according to theoretical studies.

Keywords: Fuel cell, Cyclic voltammetry, Chronoamperometry, Electrochemical impedance spectroscopy

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Direct hydrazine-hydrogen peroxide fuel cell using Ni@Pd/rGO as anodic electrocatalyst

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Abstract

A fuel cell is an electrochemical device that directly converts the chemical energy of fuels into electrical energy [1]. Different types of fuel cells have been developed over the last decades that direct hydrazine fuel cell is one of them [2]. In this study Ni@Pd on reduced graphene oxide (rGO) were synthesized using a two-step successive reduction method and characterized by field emission scanning electron microscopy (FE-SEM) and transmission electron microscopy (TEM). Hydrazine oxidation on Ni@Pd/rGO was studied by cyclic voltammetry (CV), chronoamperometry (CA), chronopotentiometry (CP) and electrochemical impedance microscopy (EIS). Hydrazine oxidation current density on Ni@Pd/rGO was 19722 A.g⁻¹. Also, in single fuel cell studies, the influence of hydrazine concentration, hydrogen peroxide concentration and temperature on the cell performance was investigated. Maximum power density was obtained at 1 M N₂H₄, 2 M H₂O₂ and 60°C equal with 204.79 mWcm⁻².

Keywords: Hydrazine, Hydrogen peroxide, fuel cell.

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Crossover of feed gases through Sulfonated poly ether ether ketone proton exchange membrane in fuel cell application

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Abstract

Introduction: Proton exchange membrane fuel cells (PEMFCs) are among the most promising energy carrier of the future. The proton exchange membrane as the key part of these cells plays an important role in their cost and functionality. Nonfluorinated hydrocarbon PEMs are recently focused in this field due to their lower price, good thermal stability and being more environmental friendly. Among these sulfonated poly ether ether ketone (SPEEK) is distinguished. One of the advantages of using SPEEK membrane is that this membrane shows less feed gas crossover in fuel cells [1]. Feed gas crossover is considered harmful for two reasons. On the one hand, it can reduce fuel cell's efficiency and on the other hand, electrochemical reaction that may occur in presence of hydrogen and oxygen gases may lead to radical components formation on the electro-catalyst surface on the cathode. These reactions produce destructive species such as hydrogen peroxide which are likely to attack polymeric structure of membrane [2]. In this study a home setup has been developed to measure the feed gas permeation through the PEMs. The setup is used to estimate hydrogen and oxygen permeation of SPEEK membrane.

Materials and Methods: Sulfonated poly (ether-ether ketone) was synthesized via post sulfonation of PEEK using concentrated sulfuric acid. 5 grams of purchased PEEK polymer was added gradually into a three-neck flask containing 100 ml of sulfuric acid equipped with condenser and the temperature was monitored. Reaction temperature was kept at 50 °C for 6 h in nitrogen atmosphere. Then reaction stopped by placing the flask into ice-water bath. Polymer fibres were decanted into ice-water with mechanical agitation afterwards, filtered and washed until reaching neutral pH. Final fibers dried in 60 °C for 13 h followed by vacuum oven at 120 °C overnight. **Membrane preparation:** SPEEK polymers were

dissolved in DMAc at 45 °C in oil bath while were stirred with a magnetic stirrer thoroughly for 24 h. Then obtained solution was cast onto a clean flat glass and heated at oven at 60 °C for 16 h afterward. Membranes were peeled off by immersing glass plate in deionized water and followed by further drying at 120 °C under vacuum for 12 h.

Gas permeation measurement: the fact that the permeation of PEM is expected to be low make it the common permeation test setup to be inaccurate [3]. Hence a dedicated setup is fabricated based on ASTM D1434 measurement methodology for thin films. Flux and permeability have measured in two different pressures (1.5 and 2bar).

Results and discussion: In this study we investigated the flux and permeability of hydrogen and oxygen through SPEEK. Results are summarized in table.1 and table.2. According to the results hydrogen has higher permeability that seems to be due to hydrogen's smaller size.

Table.1 Flux of hydrogen and oxygen through SPEEK membrane

Unit : $\frac{\text{microlitre}}{\text{cm}^2 \cdot \text{s}}$	Pressures (bar)	
	1.5	2
gas		
Hydrogen	0.005921	0.00799
Oxygen	0.00347	0.004106

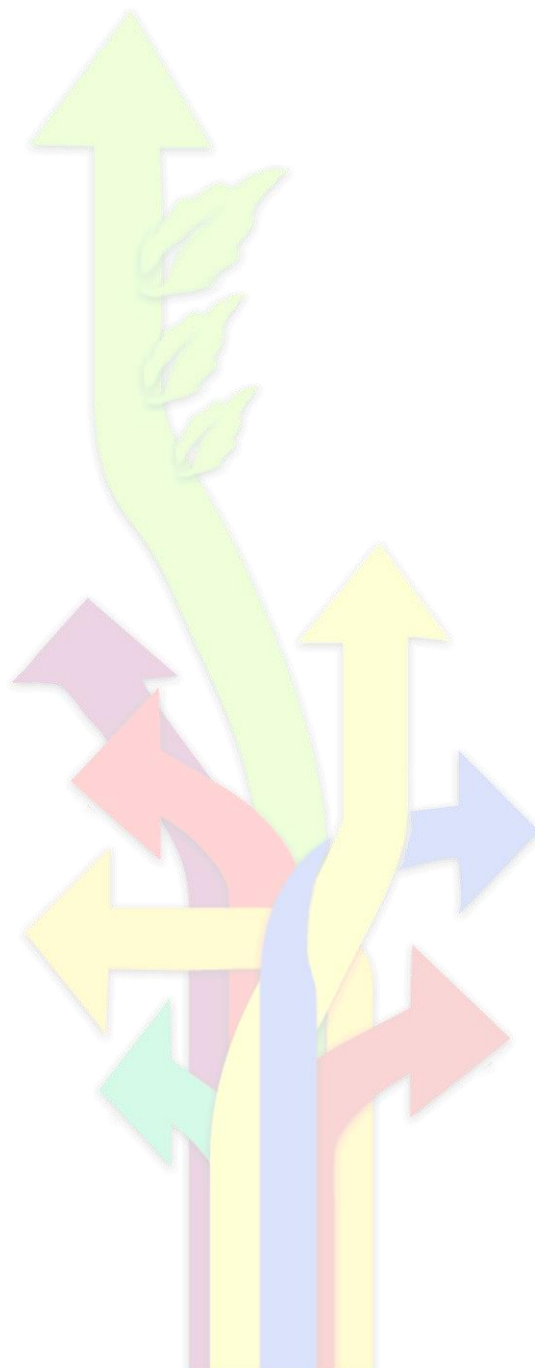
Table.2 Permeability of hydrogen and oxygen through SPEEK membrane

Permeability (barrer)	
Hydrogen	Oxygen
4.172 ± 0.025	2.618 ± 0.2077

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13th Annual Electrochemistry Seminar of Iran

A ternary catalysts of Fe-Co-Ni/rGo with high activity for alkaline glucose electrooxidation

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Abstract

In this work, a ternary catalyst of Fe_{0.33}-Co_{0.33}-Ni_{0.33} /rGo is synthesized as anode electro-catalysts for glucose oxidation. The electrochemical performance is evaluated via Cyclic Voltammetry (CV) and Chronoamperometry techniques. Results confirmed high catalytic activity of the Fe-Co-Ni/rGo electrode and glucose electrooxidation on this electrode, which make it an excellent candidate for glucose fuel cell electrocatalyst.

Keywords: Glucose, Electro-Catalyse, Cyclic Voltammetry, Fe-Co-Ni/rGo.

Introduction

In recent years, there has been growing interest in the development of battery-independent power supply systems for medical implants, such as cardiac pacemakers. Among the investigated concepts are implantable fuel cells that generate electricity from the electrochemical reaction of glucose and dissolved oxygen [1]. The detection of glucose is an important issue, because of the clinical significance of measuring blood glucose. The normal concentration of blood glucose in human body ranges from 4 to 8 mM, but the concentration may become much higher or lower for a diabetic. Thus, diabetics need a tight monitoring of their blood glucose levels [2]. In this work, we have used CV and Chronoamperometry techniques to study of Fe-Co-Ni/rGo as anode electro-catalysts for glucose oxidation.

Materials and Methods

A glassy carbon electrode was polished carefully with 0.05 μm alumina powders and then cleaned with deionized water, respectively. The catalyst ink was prepared by mixing 0.2 mg catalyst powder into 0.26 mL solution (50% Ethanol ,50% water) and 10 mg Nafion solution (5%) with stirring for 24 hr. Then 1 μL of the catalyst ink was dropped onto the clean-washed GCE and dried under room temperature. Cyclic Voltammetry (CV) and Chronoamperometry was performed in a conventional three-electrode equipped with a glassy carbon (GC) electrode, a platinum sheet counter electrode and an Ag/AgCl electrode (saturated with KCl) as the reference electrode. For cyclic voltammetry experiments, the electrode was conditioned in an aqueous solution of 1 M KOH and in the presence of glucose, by the potential cycling from -0.8 V to 0.6 V (vs. Ag/AgCl/KCl(sat.)) at a scan rate of 50 mV s^{-1} .

Results and discussion

The CV curve of a Fe-Co-Ni/rGo electrode in 1.0 M KOH solution are shown in Fig. 1. It can be seen that one anodic peak emerges at about 0.34 V (vs. Ag/AgCl/KCl sat'd). This anodic peak is attributed to

the irreversible reaction of Ni to form Ni(OH)_2 on the electrode surface. The second anodic peak at 0.40 V corresponds to the reversible reaction from Ni(OH)_2 to NiOOH . After the addition of glucose, a remarkable increase in the anodic peak current density can be observed in the corresponding CV curve, with reference to the anodic peak current density of the background CV curve obtained before glucose addition, as shown in Fig. 1. Therefore, Fe-Co-Ni/rGo electrode exhibits a steady state activity for glucose electrooxidation reaction, as shown in Fig. 2.

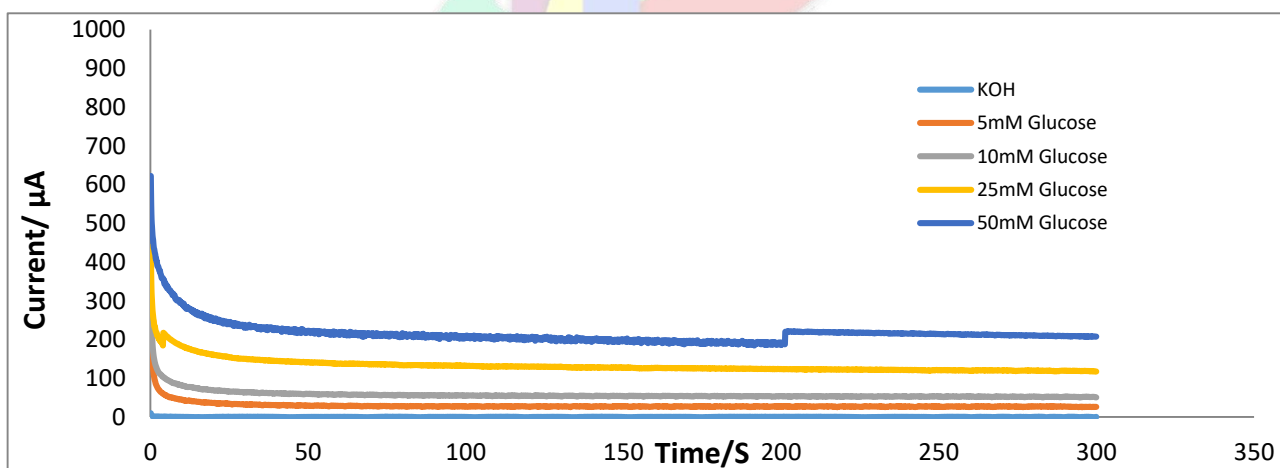
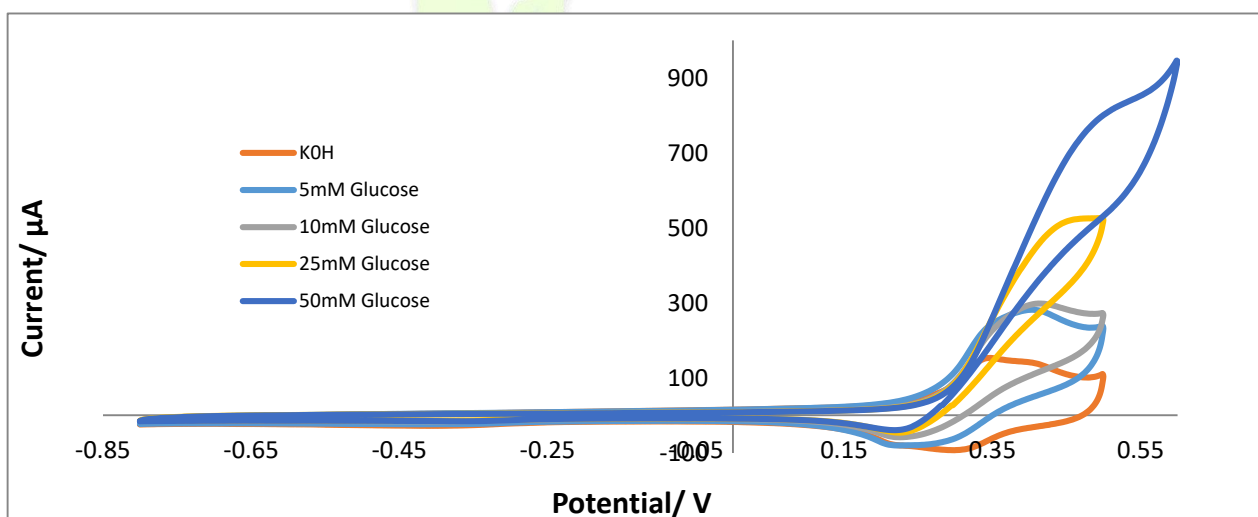


Fig. 2: Chronoamperometry curves of the Fe-Co-Ni/rGo electrode measured in 1.0 M KOH solution containing various concentrations of glucose

Conclusions

In summary, the present work represents a good activity of Fe-Co-Ni/rGo electrode for glucose electro-oxidation in alkaline media. Cyclic voltammetry showed a remarkable increase in the anodic peak current density, after addition various concentration of glucose. Chronoamperometric measurements illustrated that Fe-Co-Ni/rGo electrode exhibits a steady state activity for glucose electro-oxidation.

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A comparison between different novel carbon supported platinum nano-catalysts for Methanol oxidation reaction

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Abstract

In this study, a reduced graphene oxide and a nitrogen-doped graphene-based platinum catalyst were prepared and compared to a commercial Pt/C catalyst. X-ray diffraction patterns were recorded in order to prove successful synthesizing of graphene oxide. Scanning electron microscopic pictures were taken to show the surface and morphology of graphene-based catalysts. Excellent distribution of doped heteroatoms of nitrogen and platinum nanoparticles was showed by elemental mapping and a standard three-electrode half-cell method was employed to measure electrochemical characteristics of under investigation catalysts. To evaluate the electrochemically active surface area a CO-stripping method was used. As a result, the N-doped graphene-based catalyst showed a phenomenon performance towards methanol oxidation reaction (MOR).

Keywords: Fuel cell, Catalyst, Methanol Oxidation, Graphene, N-doped Graphene

Introduction

The main approach in Direct Methanol fuel cells is to oxidize methanol completely to carbon dioxide that consists of a complex six electron pathway [1]. To develop the kinetics of this reaction and reducing the loading of metal on the surface, many idea's have been investigated [1-5] and changing the support to a novel carbon structure especially, to graphene [6] and in addition, modifying graphene by doping N atoms seems to be good solutions for a better proceeding towards this aim [7-9]. In this research, the effect of using the mentioned supports for platinum nano-particles for MOR was investigated and the result is quite interesting.

Experimental

Graphene oxide was synthesized by a modified Hummer's method. For preparing the platinum on graphene catalyst (Pt/G) an impregnation method was employed using H_2PtCl_6 and NaBH_4 . For doping of N heteroatoms into the structure of graphene oxide, a thermal pyrolysis approach was carried out on a (1:5) mixture of GO and melamine at 600 °C under N_2 atmosphere and the procedure of loading of platinum was the same as Pt/G. For physicochemical characterizations, XRD, ICP-OES, SEM, EDS line scan and elemental mapping were utilized and for electrochemical measurements, a standard three-electrode half-cell method was employed. For methanol oxidation, a 0.5M H_2SO_4 and 2M methanol was prepared as electrolyte and for CO-stripping only H_2SO_4 0.5M. An Ag/AgCl as reference, a Platinum plate as counter and a 5mm diameter glassy carbon as working electrode was utilized in the cell.

Results and discussions

Figure 2 (Left) is showing the characteristic peak of GO that can be seen at about 12 degrees. In Pt/G and Pt/NG patterns different facets of Platinum nano-crystals are presented and at about 27 to 29 degrees the peak corresponds to carbon support. In Figure 2 (Right) cyclic voltammetry of mentioned catalyst was recorded. As it can be comprehended peak current of Pt/NG and Pt/G are much higher than that of in Pt/C. Also, the ratio of forwarding peak to backward peak (I_f/I_b) in Pt/NG is higher than other two, announcing that Pt/NG is more stable towards intermediates of electrooxidation of methanol, mostly CO, hence higher stability is achieved versus CO-poisoning of Pt nanoparticles. The forwarding peak of Commercial Pt/C is happening in more negative potentials in comparison to Pt/NG and Pt/G but as it was mentioned before a low current density achieved. Moreover, the onsets of three catalysts were almost the same but, due to higher current densities of synthesized catalysts, Pt/G and Pt/NG showed a much more faster pace to the forwarding peaks.

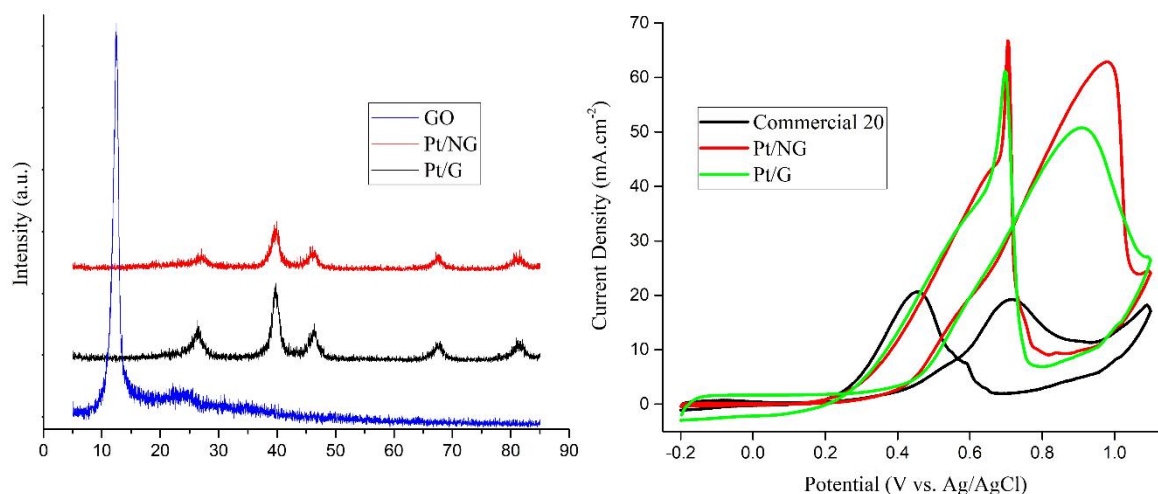


Figure 2- Left: XRD pattern of Pt/NG (Red), Pt/G (Black), and GO (Blue), Right: MOR reaction in a three-electrode half-cell measurement under Ar atmosphere at RT, 0.5M sulfuric acid and 2M methanol, normalized by CO-stripping surface area

Uniform and proper dispersion of Nitrogen and Platinum is viewed in Figure 3. As it can be seen nitrogen atoms are very well distributed along the surface and edges but since higher resolution could not be achieved due to lack of equipment, a very thorough investigation was not possible.

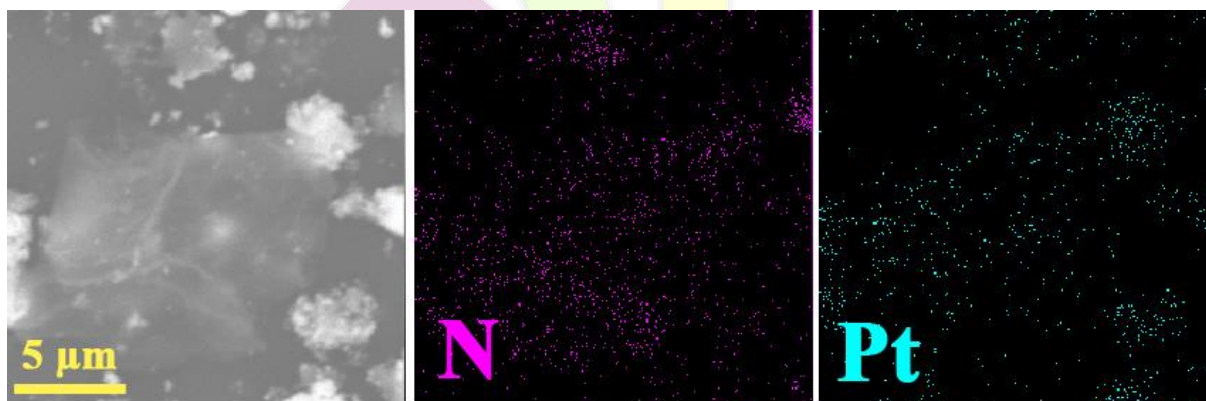


Figure 3- Elemental mapping of Pt/NG recorded by SEM.

Conclusions

Novel Pt-based catalysts supported on graphene and N-doped graphene were evaluated for the methanol and CO electrooxidation and compared with Pt/C commercial catalyst. The Pt/NG catalyst showed significantly higher electrochemical catalytic activity compared to other investigated catalysts. The enhanced performance is mainly attributed to the presence

of nitrogen functional groups in the graphene sheets which alter the defect and electronic structure of the support.

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Synthesis of New-Electrocatalyst as Pt₃Sn by Chemical Reduction Method for Methanol Electro-oxidation

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Abstract

Low temperature direct alcohol fuel cells (DAFCs) are extremely attractive as power sources for transportation, mobile and portable applications because it is not necessary to convert the fuel in a reformer into hydrogen. However, alcohols are very difficult to electro_ oxidized completely and up to now methanol has been considered the most promising fuel, because it is more efficiently oxidized than other alcohols and thus direct methanol fuel cells (DMFCs) have been developed for the portable power market. The methanol oxidation reaction (MOR) by itself is rather slow and requires active catalytic sites for adsorption and oxidation of methanol. Pt or Pt_ based alloys, supported by carbon materials are commonly used catalysts in direct methanol fuel cells (DMFCs) in acidic solution. Polyaniline (PANI) with porous structure and high surface area is a particularly attractive material as catalyst support.

In this work, platinum and platinum_ tin catalysts with an atomic ratio Pt/Sn (3:1) supported on Multiwall carbon nanotubes and Vulkan carbon are prepared by a deposition process using NaBH₄ as the reducing agent and from this category of materials was used as the anodic electrocatalysts in direct methanol fuel cells. Specificinty structural, morphology and electrochemical behavior synthesized electrocatalysts by methods of X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX) and cyclic voltammetry (CV) are investigated. From the XRD patterns, the Pt(220) peak of the PtSn/C, PtSn/MWCNT catalysts shift slightly to lower 2θ values with increasing Sn compared with that of the Pt/C, Pt/MWCNT catalysts, suggesting the alloy formation. The cyclic voltammetry reveals that PtSn/MWCNT electrocatalyst in methanol oxidation shows a greater oxidation current and a lower onest potential, compared to other electrocatalysts. Then the effect of glassy carbon electrode (GC) surface modification by a thin layer of

polyaniline is investigated. The results show that in present a thin layer of polyaniline and platinum-tin electrocatalytic particles supported on multiwall carbon nanotube (GC/PANI/Pt-Sn/MWCNT) the oxidation of methanol in aqueous acid media is considerably enhanced.

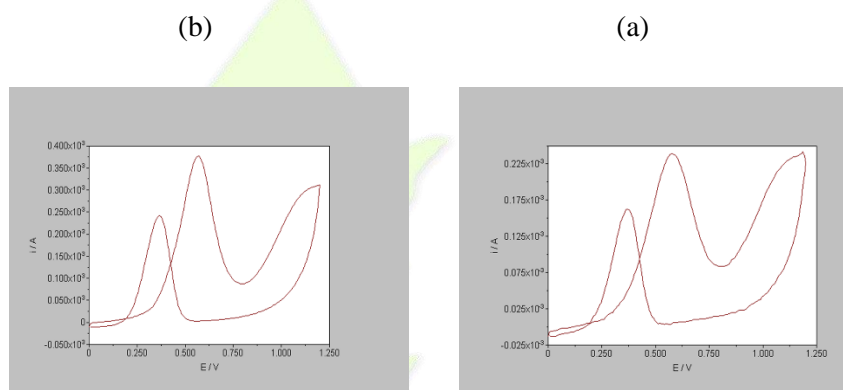


Fig.1. CVs of Pt/MWCNT (a), Pt-Sn/MWCNT in 0.5M CH₃OH and 0.1 H₂SO₄.

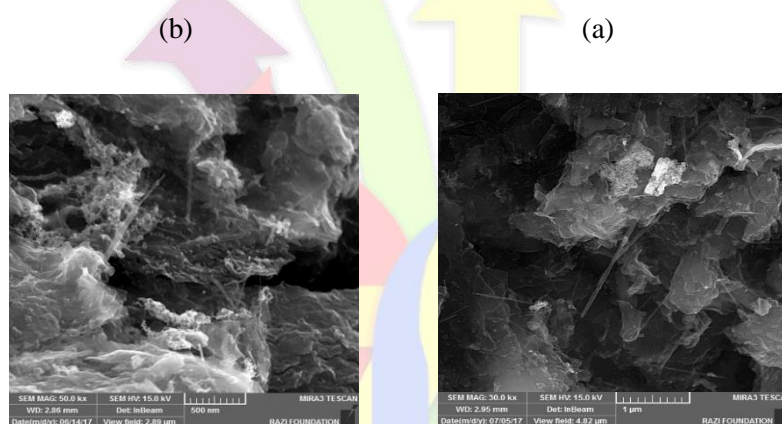


Fig.2. FESEM images of Pt-Sn/MWCNT (a), Pt/MWCNT(b).

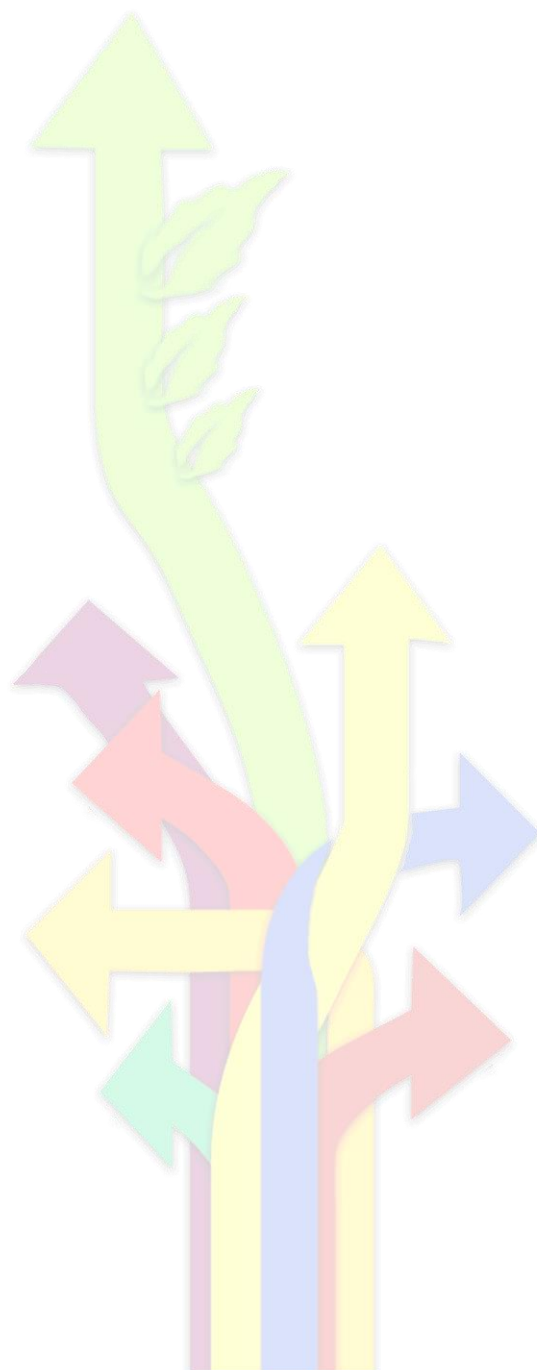
Keywords: Electrocatalyst, Platinum, Polymer, Methanol Oxidation

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13th Annual Electrochemistry Seminar of Iran

Microbial fuel cell desiging by graphite-anode electrode modified by carbon nanotubes containing Metal oxide nanoparticles

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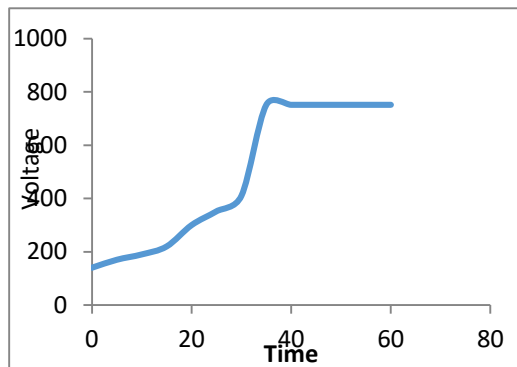
Abstract

Recently, great attentions have been paid to microbial fuel cells (MFCs) due to their mild operating conditions and using variety of biodegradable substrates as fuel. The traditional MFC consisted of anode and cathode compartments but there are single chamber MFCs. Microorganisms actively catabolize substrate, and bioelectricities are generated. MFCs could be utilized as power generator in small devices such as biosensor. Besides the advantages of this technology, it still faces practical barriers such as low power and current density. In the present article different parts of MFC such as anode, cathode and membrane have been reviewed and to overcome the practical challenges in this field some practical options have been suggested. Also, this research work demonstrates the improvement of MFCs with summarization of their advantageous and possible applications in future application. Also, Different key factors affecting bioelectricity generation on MFCs were investigated and these key parameters are fully discussed.

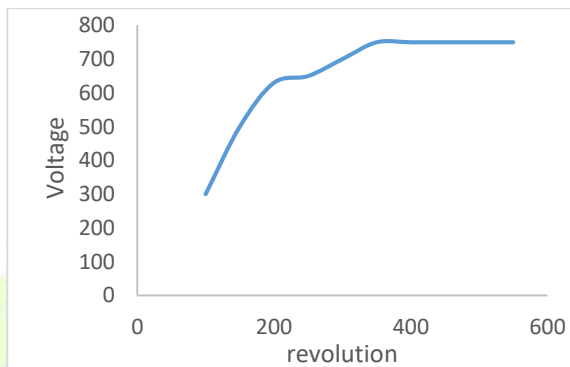
Keywords: Microbial fuel cell, Biosensor, Biocatalyst, Anaerobic anode, Bioelectricity



Microbial fuel cell picture



(A) Bent voltage in Time revolution



(B) Bent voltage in revolution

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Investigating the effect of Polyvinylpyrrolidone on the Pt-Ni/Ni/carbon paper electrode manufacturing process to improve its performance for methanol oxidation reaction in alkaline media

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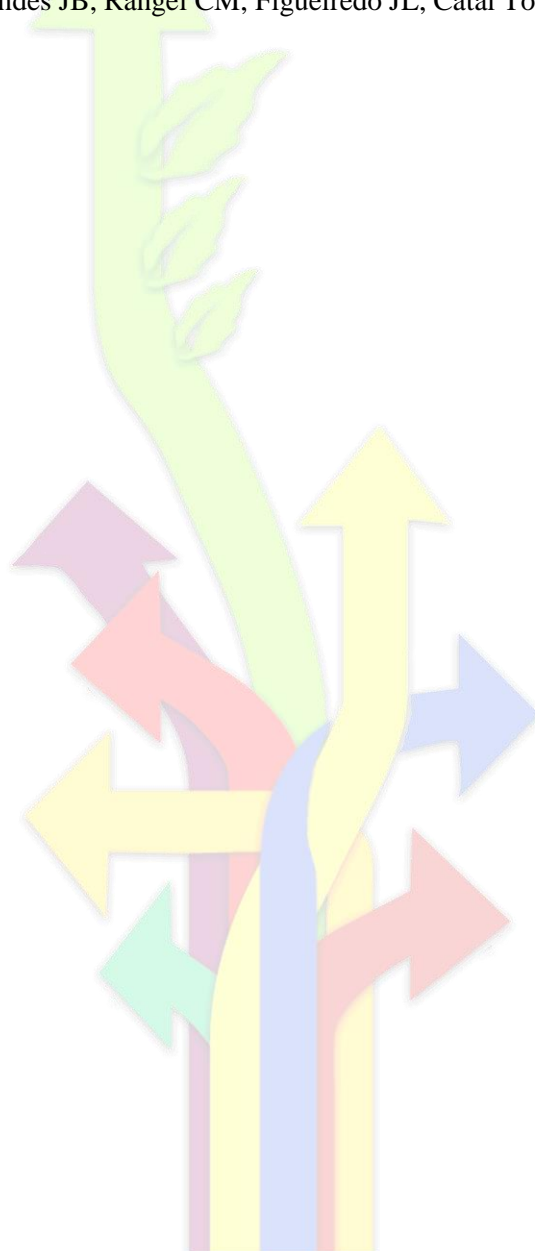
Abstract

Methanol electrochemical oxidation reaction is interesting for researchers in order to design electrodes to respond to this reaction at a high rate. One of the methods of making an electrode is the use of electro-deposition treatment. Platinum and Nickel are common electrocatalysts which can be electrodeposited on carbon paper as electrode for methanol oxidation reaction (MOR). The electro-deposition processes can be affected with various factors. Polyvinylpyrrolidone (PVP) presence in electro-deposition solution is the factor can influence on property of prepared electrocatalysts for MOR. At this work for preparing modified electrodes, first, the nickel is deposited on the carbon paper, and then on it, a mixture of platinum and nickel is deposited by cyclic voltammetry method (potential range: -0.850 V to 0.3 V vs. Ag/AgCl for Nickel and -0.850 V to 0.650 V vs. Ag/AgCl for Platinum and Nickel mixture, scan rate 50 mV s⁻¹, cycle number 40). For studying the PVP (0.005 M) effect on property of electrodeposited particles, the PVP was added to electrodeposition solution at three states: Ni electro-deposition, Pt-Ni electro-deposition and Pt-Ni/Ni electro-deposition. Electrochemical methods like as linear sweep voltammetry (LSV), cyclic voltammetry (CV) and impedance spectroscopy (EIS) were used for investigating fabricated electrodes for MOR. Based on electrochemical analysis of prepared electrocatalysts, the presence of PVP to both electro-deposition processes (Ni and Ni-Pt) is more effective than other states for the performance of fabricated electrodes to MOR in alkaline media.

Keywords: Methanol fuel cell, Nano Platinum-Nickel electrodeposited electro catalyst, Methanol oxidation reaction, Polyvinylpyrrolidone.

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13th Annual Electrochemistry Seminar of Iran

Polarization and Parametric Analytical Model of Solid Oxide Fuel Cells

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Abstract

A polarization modeling of Solid Oxide Fuel Cells (SOFCs) has been developed considering activation overpotential (modified Butler-Volmer equations), ohmic resistance and concentration overpotential. Then, a multi-parametric analysis has been carried out to highlight which parameters are most relevant in the overall cell performance as measured by polarization curve.

Keywords: SOFCs, Polarization Modeling, Multi-parametric Analysis, Optimization

Introduction

Fuel cell is an emerging technology that is to a great extent promising for the achievement of higher energy efficiency and lower environmental load. Therefore, a responsible use of energy is proportional to the high energy efficiency and its minimum impacts on the environment [1].

Methods

Activation over-potential is much more frequent in low- and average-temperature fuel cells and it is more considerable in terms of cathode. The general Butler-Volmer equation has been used here for calculating the corresponding anodic and cathodic voltage loss [2]:

$$\begin{cases} j = j_o \left[\exp\left(\frac{a \cdot n \cdot F \cdot \eta_{act}}{RT}\right) - \exp\left(\frac{(1-a) \cdot n \cdot F \cdot \eta_{act}}{RT}\right) \right] \\ \eta_{act,i} = \frac{RT}{F} \sinh^{-1} \left(\frac{j}{2j_{o,i}} \right) \end{cases} \quad (1)$$

Ohmic over-potential in which electrolyte, electrodes and the interconnectors of the fuel cell are involved occurs because of the resistance to the flow of ions in ionic conductors and also the

resistance to the flow of electrons in electronic conductors. Since these resistances follow the Ohm's Law, the total ohmic voltage loss would be [3]:

$$\eta_{ohm} = IR \quad (2)$$

Concentration over-potential represents the effects of pressure and concentration on voltage. Here, in order to avoid much reliance on a complicated relationship for determining the limiting current density, total voltage loss equations which have been previously formulated could be used to define concentration loss [4]:

$$\eta_{Conc} = C * \ln \left(\frac{j_L}{j_L - j} \right) \quad (3)$$

Results and discussion

Polarization and power density curves as a function of current density are illustrated at Figure 4:

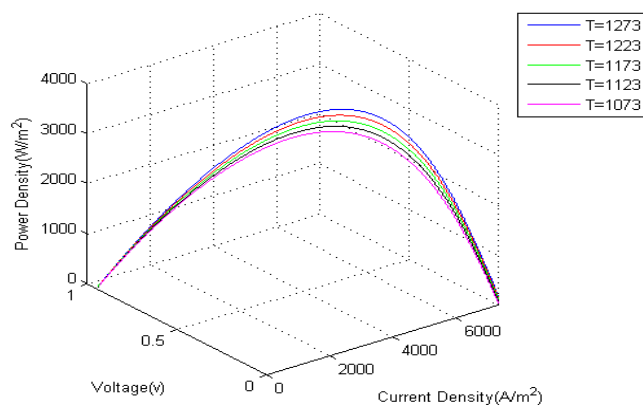


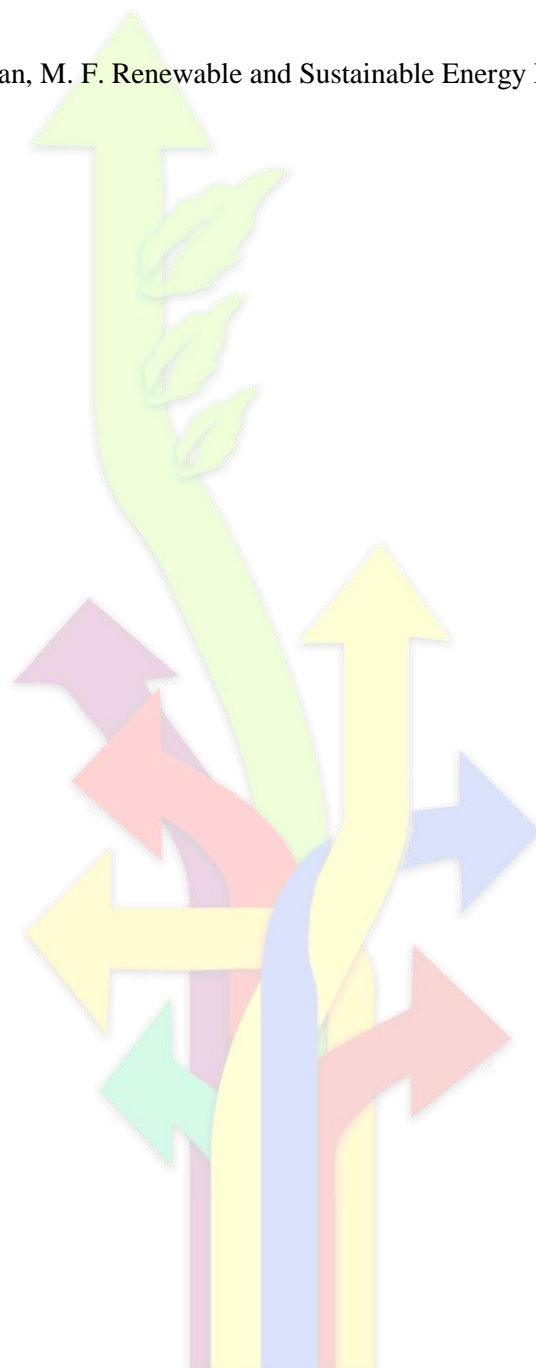
Fig. 4.1: Polarization and power density curves as a function of current density

Conclusion

A polarization modeling of Solid Oxide Fuel Cells (SOFCs) was our main objective in this research. Also, a multi-parametric analysis carried out to point out which physical parameters mostly influence the cell performance. In the Polarization model, three optimum outputs obtained were: cell voltage 0.57 V, current density 5619 W/m and power density 3206 A/m².

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Nanostructuring platinum nanoparticles on Ni/Ce_{0.8}Gd_{0.2}O_{2-δ} anode for low temperature solid oxide fuel cell via single-step infiltration: a case study

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Abstract

With the aim of promoting the Ni/Ce_{0.8}Gd_{0.2}O_{2-δ} (Ni/GDC20) cermet anode performance of low temperature solid oxide fuel cell (LT-SOFC) [1], nanostructuring platinum nanoparticles on NiO/GDC was done by single-step wet-infiltration of H₂PtCl₆.6H₂O. The anodic polarization resistance was measured using symmetric Ni–GDC20|GDC20|Pt electrolyte-supported cell at 400-600 °C. Microstructural refinement before and after hydrogen reduction at 600 °C and also after anodic performance test was studied by field emission scanning electron microscopy (FE-SEM) and high resolution transmission electron microscopy (HR-TEM) in comparison to the pure anode itself. Nanostructuring Pt-nanoparticles with average particle size of 12.5 nm on Ni/GDC20 anode indicated the lacking of electrocatalytic enhancement with the addition of platinum for H₂ oxidation reaction in LT-SOFC.

Keywords: LT-SOFC, Ni/GDC20 Anode, Platinum Infiltration, Pt-Nanoparticles, H₂PtCl₆.6H₂O

Introduction

The anodes with active triple phase boundaries (TPBs) have an important role on the performance of LT-SOFC [2]. Among the methods to improve the microstructure of co-sintered anodes, nanostructuring by infiltration, has been led to higher performance of the infiltrated anodes than the conventional anodes [3]. For Ni/GDC, which yielded good LT-SOFC performance, only a few articles have reported the infiltration using metals [4,5]. One of the most valuable electrocatalysts is platinum, which used for the H₂ oxidation reaction at the anode due to high selectivity, activity, stability, and electronic conductivity [6]. In this article, based on the necessity of cost-effective application of precious metals, the effect of once infiltration of Pt-precursor solution on the microstructure and performance of the Ni/GDC20 anode is considered.

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Methods

The configuration of planar electrolyte-supported Ni–GDC20|GDC20 half-cell was used [1].

Only a droplet of 0.1 mol/L $\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$ solution placed on the porous NiO/GDC20 composite, followed by calcination at 600 °C for 1h and H_2 reduction. Anodic impedance of the $\sim 0.03 \text{ mg/cm}^2$ Pt-infiltrated anode (T-R) was tested in comparison to the original anode (O-R) [1] and microstructure of the anodes was investigated by SEM and TEM [4].

Results and Discussion

TEM evaluation of T-R (Fig. 1) shows the isolated pseudo-spherical nanoparticles with the average nanoparticle size of $\sim 12.5 \text{ nm}$. Less coarsening of the Pt-nanoparticles in the H_2 at LT-SOFC is associated with the low mobility of metal platinum [7] and the low partial pressure of Pt [8], which can preserve the high activity of Pt-nanocatalysts. The anodic polarization resistance (R_a) data (Table 1) indicate too much increase of the R_a in T-R ($\geq 36.5\%$) than that in the O-R. Although the supreme catalytic ability of platinum in LT-SOFCs has been confirmed, but the lack of coarsening propensity of Pt-nanoparticles at LT-SOFC conditions also cannot be a help for its effective electrocatalytic activity on the Ni/GDC20 anode and lowering anode polarization resistance of O-R. Therefore, besides the catalyst composition, its local abundance on the microstructure can be important. Hence, Pt is an ineffectual nanocatalyst in the current study.

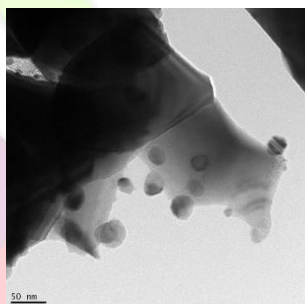


Fig.1. Typical TEM-BF image of T-R.

Table.1. Anode polarization resistance values for the H_2 oxidation reaction on the O-R and T-R anodes at testing temperatures in 97% H_2 /3% H_2O at open circuit.

T (°C)	$R_a (\Omega \cdot \text{cm}^2)$				
	600	550	500	450	400
O-R	0.64	3.96	37.96	181.36	268.62
T-R	1.23	8.26	52.12	247.47	729.06

Conclusions

The investigation of nanostructuring via Pt-infiltration of Ni/GDC20 anode with the sole addition of one drop of $\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$ solution on the anodic polarization resistance for H_2 oxidation reaction at 400-600 °C in comparison to base anode was performed. Regardless of the less coarsening propensity of Pt-nanoparticles with an average size of $\sim 12.5 \text{ nm}$ as well as the negligible volatility of platinum during the cell testing in a hydrogen environment at temperatures of LT-SOFC, the anodic performance of Pt-infiltrated anode was much higher ($\geq 36\%$) than that in the base Ni/GDC20 anode. This indicated that platinum is an ineffectual infiltrated nanocatalyst in the present study.

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Platinum–Iron NPs/C as an active catalyst for methanol electro oxidation

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Abstract

Platinum–Iron nanoparticles are synthesized by chemical reduction method as an anode catalyst for the methanol electro oxidation. The characterization of the catalyst has been investigated using physical and electrochemical methods. Prepared catalyst was characterized by scanning electron microscopy (SEM). Pt and Pt-Fe nanoparticles are uniformly dispersed on the surface of carbon powder. The catalytic properties of the catalyst for methanol electro-oxidation were studied by electrochemical methods that involved in the cyclic voltammetry, linear sweep voltammetry (LSV) and electrochemical impedance spectroscopy (EIS). The Pt-Fe/C shows high electro catalytic activity for electro-oxidation of methanol. Therefore, the Pt-Fe/C catalyst is a able catalyst for application in direct methanol fuel cells.

Keywords: Anode catalyst; Direct methanol fuel cell; Platinum; Iron

Study of glucose oxidation reaction in neutral condition on platinum doped in Nickel oxide electrocatalyst

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Glucose is used as fuel in fuel cells. Glucose fuel cells in a neutral medium could be good candidate for use in living organisms. Because glucose is found in living beings abundantly, and in addition to its abundance, glucose is not toxic and does not volatile. The energy that is produced by the oxidation of glucose in neutral medium is used in artificial tissues. The glucose oxidation reaction (GOR) is slow and much effort is being made to create new electrocatalysts to facilitate this reaction. The platinum is common electrocatalyst for GOR. At this research, new catalyst was introduced based on doping platinum in Nickel oxide (NiO) crystalline structure. First, Nano-particles of nickel oxide was synthesized by hydrothermal method, and then, the prepared Nickel oxide was identified by X-ray Powder Diffraction (XRD) spectroscopy, in the next step, platinum nanoparticles was doped in Nickel oxide with 2% concentration. For studying GOR at prepared particles, the electrodes were fabricated with various amounts of Pt-NiO (10% to 90%) on carbon paper. Electrochemical methods was used for investigating fabricated electrodes for GOR at glucose 5 mM solution. The pH of glucose solution was fixed at 7.4 by using phosphate buffer solution. The temperature of study was 37 °C . According these results, the amount of synthesized catalyst (2% platinum doped in NiO) in reaction layer has effect on the performance of fabricated electrodes for GOR. The optimized concentration was determined at 40 % Pt-NiO.

Keywords: Glucose oxidation, electrocatalyst, fuel cell, Nickel oxide, Platinum.

Effect of treatment temperature on electrocatalytic activity of manganese/ ruthenium mixed oxide composites for oxygen evolution reaction

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Abstract

Recently, development of efficient catalysts toward OER has been a challenging task. Oxygen evolution occurs on noble metal catalysts but noble metal oxides such as RuO_2 are generally more active electrocatalysts [1]. The synergic effect of two elements in the mixed oxide has been studied to obtain the more stable and low-cost efficient catalysts [2, 3]. The most stable anode electrodes are prepared by thermal decomposition of metal precursors deposited on titanium substrates [4].

In this work, we prepared manganese-Ruthenium composites by the method of thermal decomposition and were surveyed the Effect of treatment temperature on electrocatalytic activity of OER over three annealing temperatures.

Titanium sheets which were used as coating bases, first were polished and were degreased with caustic soda, hydrochloric acid, and finally, with acetone. In order to investigate the effect of manganese ratio on the catalytic properties of ruthenium coating, six different manganese-ruthenium weights were prepared from $\text{MnCl}_2 \cdot 6\text{H}_2\text{O}$ and $\text{RuCl}_3 \cdot x\text{H}_2\text{O}$, according to table 1. Also, isobutyl alcohol was used to investigate the effect of alcohol in sol-gel method. After the heating process, the thickened solution was applied to coating titanium electrode samples by repeated immersion-drying method. Finally, the surface oxidation by heat treatment was performed in a temperature-controlled furnace at a temperature of 350, 450 and 600 °C for nine hours.

The voltammetry techniques were used to survey the efficiency and compare the results of the prepared prototypes. A conventional three electrode setup consisting of the prepared working electrode, Ag/AgCl reference electrode and Pt rod as the counter electrode in the

presence of 1 M KOH electrolyte. The material specification of coating specimens is shown in Table 1.

As an example, the cyclic voltammograms of specimens with different ratio of Mn/Ru were compared in Figure 1. It is clear that the catalysts containing %100 Mn have high overpotential than the rest of the catalysts. Adding Ru reduces this gradually. It can be concluded that the addition of ruthenium ratio increases catalyst activity. However, the low price of manganese against ruthenium suggests that the optimum Mn:Ru ratio is 50:50, which does not show significant difference compared with 25:75 ratio. Furthermore, the addition of alcohol to a sol-gel solution increases the performance of the catalyst.

The results of temperature test in thermal treatment also shows that ascending the temperature of oxidation increases the overpotential and the TOEFL gradient and consequently the catalytic activity decreases. This is related to different compositions of manganese oxides (Mn_xO_y). SEM images confirm the effect of temperature and the ratio of Mn/Ru in surface layout on catalytic efficiency. It can be seen outstanding changes in surface morphology consist of abundant cracking and roughness at oxidation temperatures of 450 and especially 600 °C.

Table 1- material specification of coating specimens in a certain annealing temperature.

Sample code	Base metal	Sol Components		
		% Ru	% Mn	i-Butanol /ml
C1	Ti	0	100	0
C2	Ti	10	90	0
C3	Ti	25	75	0
C3b	Ti	25	75	0.5
C4	Ti	50	50	0
C4b	Ti	50	50	0.5
C5	Ti	75	25	0
C5b	Ti	75	25	0.5
C6	Ti	100	0	0

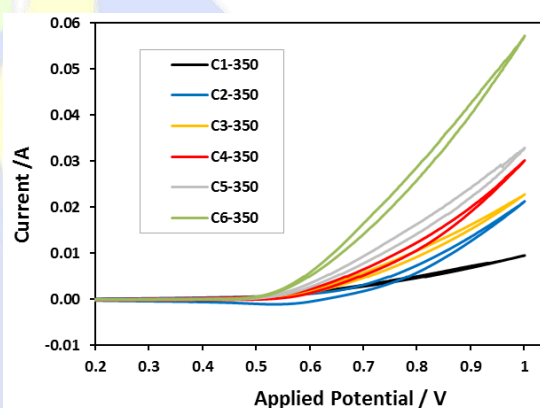
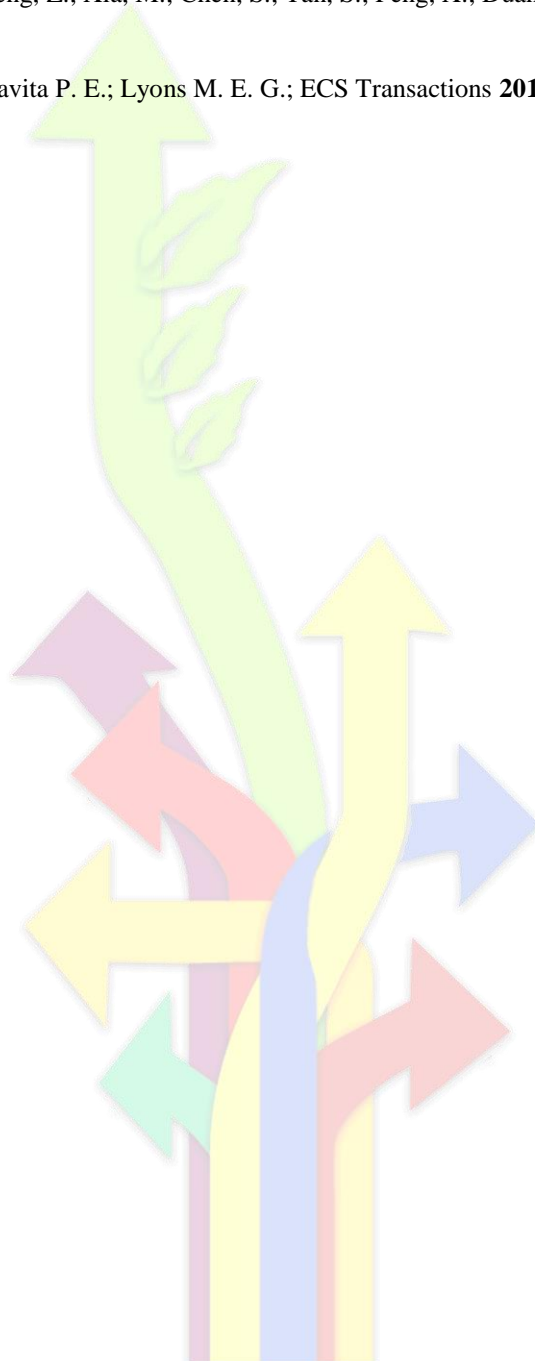


Figure 1. Cyclic voltammograms of specimens at 350 °C.

Keywords: RuO_2 - MnO_x composite, electrocatalyst, oxygen evolution reaction (OER), thermal treatment.

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13th Annual Electrochemistry Seminar of Iran

Galvanic replacement of Pt nanowires on 3D-Ni Foam helped with Silica Template for methanol fuel cell

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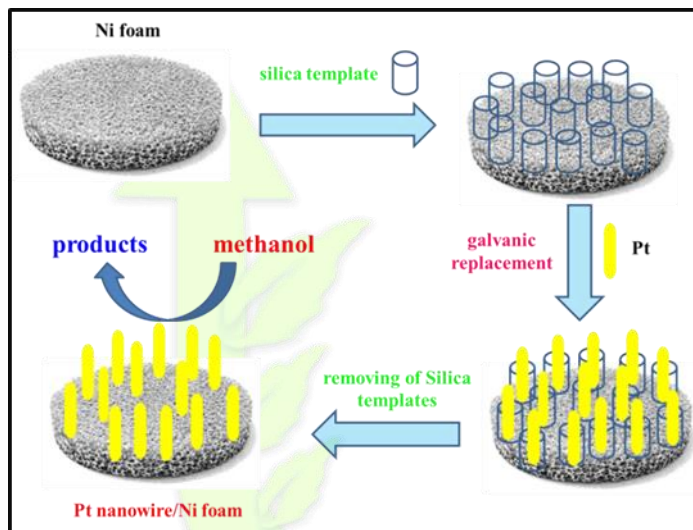
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Abstract

Generation of energy by clean, efficient and environmentally-friendly means is now one of the major challenges for engineers and scientists. Fuel cells convert chemical energy of a fuel directly into electrical work, and are efficient and environmentally clean, since no combustion is required. Moreover, fuel cells have the potential for development to a sufficient size for applications in commercial electricity generation [1]. In the presented work, modified nickel foam (NF) electrode was designed for using in direct methanol fuel cell. For this purpose, the commercial Ni foam was cut and connected with a metal wire as working electrode. The Ni foam electrode was cleaned first by the following procedure. The Ni foam was soaked and sonicated first in acetone for 15 minutes. After being cleaned in acetone, it was sonicated in deionized water for 15 minutes. Then, the electrode surface was modified with mesoporous silica as template for fabrication porous metal electrode. The silica mesoporous was formed by electro-assisted self-assembly (EASA) on the three dimensional Ni foam electrode. Pt nanowires were successfully placed on Ni foam support by employing the silica templates and then applied as an efficient catalyst. After silica removing, methanol electro-oxidation was investigated [2]. The physical properties of Pt nanowire/Ni foam modified electrode were characterized via scanning electron microscopy (SEM) and X-ray diffraction (XRD). The electrochemical properties of the catalysts were evaluated through electrochemical experiments. The electro-oxidation of methanol in 0.5 M NaOH was studied using cyclic voltammetry technique. Effective parameters included deposition potential of silica, deposition time of silica, immersion time of nickel foam in platinum solution, Concentration of platinum solution, concentration of NaOH solution and the furnace temperature were optimized. In the optimal condition, the prepared electrode showed excellent electrochemically active surface areas (ECSAs), catalytic activity, and stability toward the methanol electro-oxidation reaction (MOR) in the alkaline media.

Graphical abstract



Keywords: *Methanol fuel cell, Nickel foam, Platinum nanowire, Silica mesoporous, Electro-assisted self-assembly*

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Durability study of activated membrane electrode assembly using accelerated degradation technique

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Abstract

In this work, an MEA was degraded using accelerated stress technique and the MEA performance was studied by polarization curve, Electrochemical Impedance Spectroscopy (EIS) and Cyclic Voltammetry (CV). The obtained results indicated that during application of MEA under normal operation conditions, catalyst layer is the first part of MEA that is subject to degradation. Increase of charge transfer resistance and reduction of electrochemical surface area (ECSA) represent this issue.

Keywords: PEM fuel cell, MEA degradation, MEA durability, Aging procedure.

Introduction

Durability is one of the major barriers to proton exchange membrane fuel cells (PEMFCs) being accepted as a commercially viable product. In addition to structural parameters, PEMFC load cycles and operating conditions have a significant effect on the durability of PEM fuel cell [1,2]. In recent years, to reduce research costs and testing time, accelerated degradation techniques have been developed to study the fuel cells durability [3,4]. In this work, we have used the degradation cycles as aging procedure and polarization curve, EIS and CV as performance evaluation techniques to study of MEA aging during normal operation conditions of MEA.

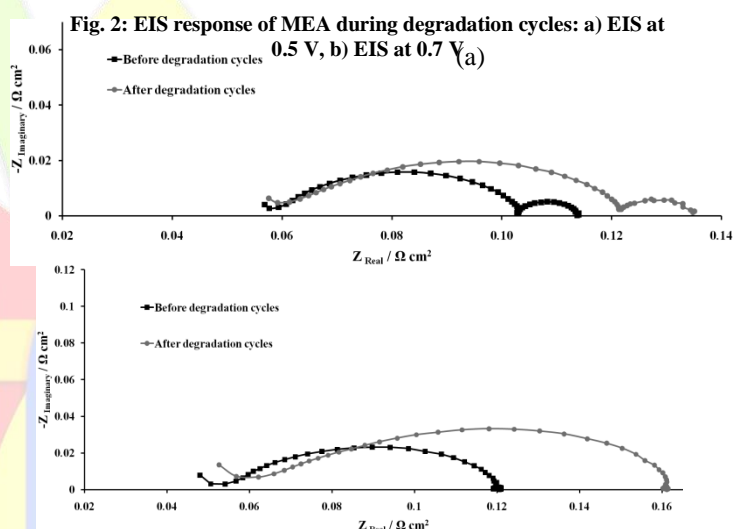
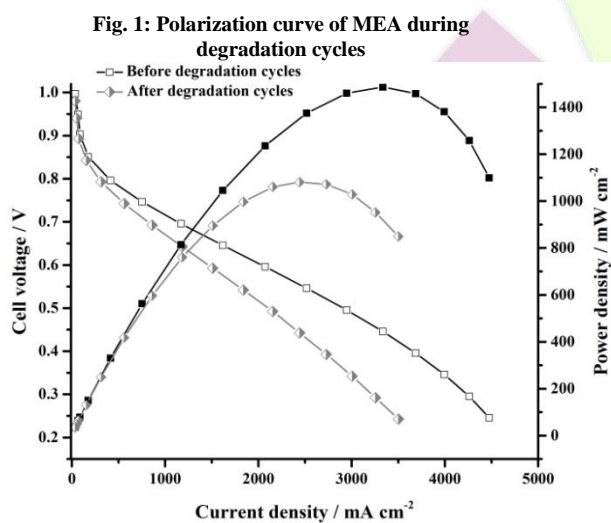
Materials and Methods

The 20 wt.% Pt/C (BASF) and 5 wt.% Nafion solution (Aldrich) were used for catalyst ink preparation. The catalyst ink was coated on a TGP carbon paper (Toray) by painting method and dried at 80 °C for 60 min. To prepare MEA, Nafion 212 membrane sandwiched between two gas diffusion electrodes. During the polarization curves and EIS tests, cell temperature was fixed at 75 °C, hydrogen was supplied to anode at 100% humidity and oxygen was supplied to cathode at 30% humidity. In CV tests, hydrogen and nitrogen was supplied to the anode and cathode, respectively and the cell temperature was fixed at 50 °C.

Results and discussion

Fig. 1 shows the polarization curve of MEA before and after degradation cycles. As seen in fig.1, the performance has reduced during the aging process. The maximum output power density of MEA has decreased from 1486 mW cm^{-2} to 1024 mW cm^{-2} .

EIS response of MEA at 0.5 V (that is related to ohmic polarization region) is shown in fig. 2-a. According to fig. 2-a, solution resistance that is attributed to membrane proton conductivity has no significant change during aging process. This issue shows that membrane has not been damage during degradation cycles. AC impedance measurements at 0.7 V are shown in Fig. 2-b. It can be seen from the fig. 2-b that the diameter of the fuel cell MEA Nyquist plot increased with time. This arc is attributed to charge transfer limitations. During degradation cycles, charge transfer resistance (R_{ct}) has changed from $67 \text{ m}\Omega \text{ cm}^2$ to $100 \text{ m}\Omega \text{ cm}^2$ that shows the increase of R_{ct} by 33%. The obtained results from EIS at 0.7 V are in agreement with the results of CV test that are shown in fig. 3.



As seen in fig. 3, electrochemical surface area has changed from $521 \text{ cm}^2 \text{ mg}^{-1}$ to $419 \text{ cm}^2 \text{ mg}^{-1}$ that shows the decrease of ECSA by 19%. Reduction of ECSA and increase of R_{ct} cause the reduction of PEM fuel cell performance during MEA aging

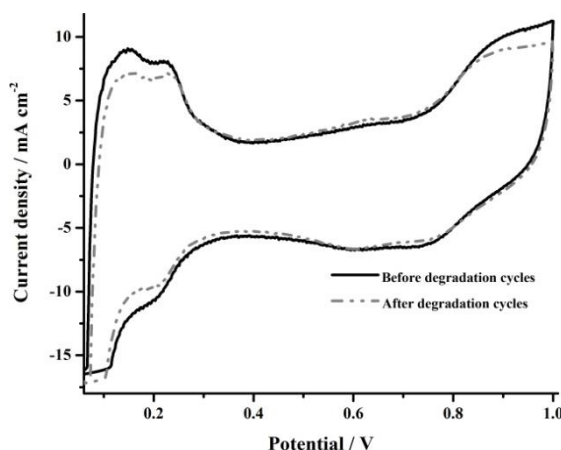


Fig. 3: Cyclic voltammogram of MEA during degradation cycles

Conclusions

The performance of MEA during degradation cycles was evaluated in fuel cell normal operation conditions. According to the obtained results, membrane has not been damaged during the aging procedure. On the other hand, changes of ECSA and R_{ct} indicate that under normal operation conditions, catalyst layer is the first part of MEA that is subject to degradation

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One step electrochemical synthesis of Ni-GO nano composite electrode as a catalyste for ethanol electro oxidation

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Abstract

Development of anode catalysts and catalyst supporting carbonaceous material containing non-precious metal have attracted tremendous attention in the field of direct ethanol fuel cells (DEFCs). Pt and pd have most catalytic activity in EOR, but because of their some disadvantages such as their high cost, application of them in various systems is limited and other metals have been substituted them. Other ways for increasing the catalytic activity of catalysts is using of supports such as carbon Vulcan, carbonized TiO₂ nanotube, multi-walled carbon nanotubes and carbon microspheres. It was found that reduced graphene oxide has very intriguing physical properties that suggest it can have a wide variety of applications.

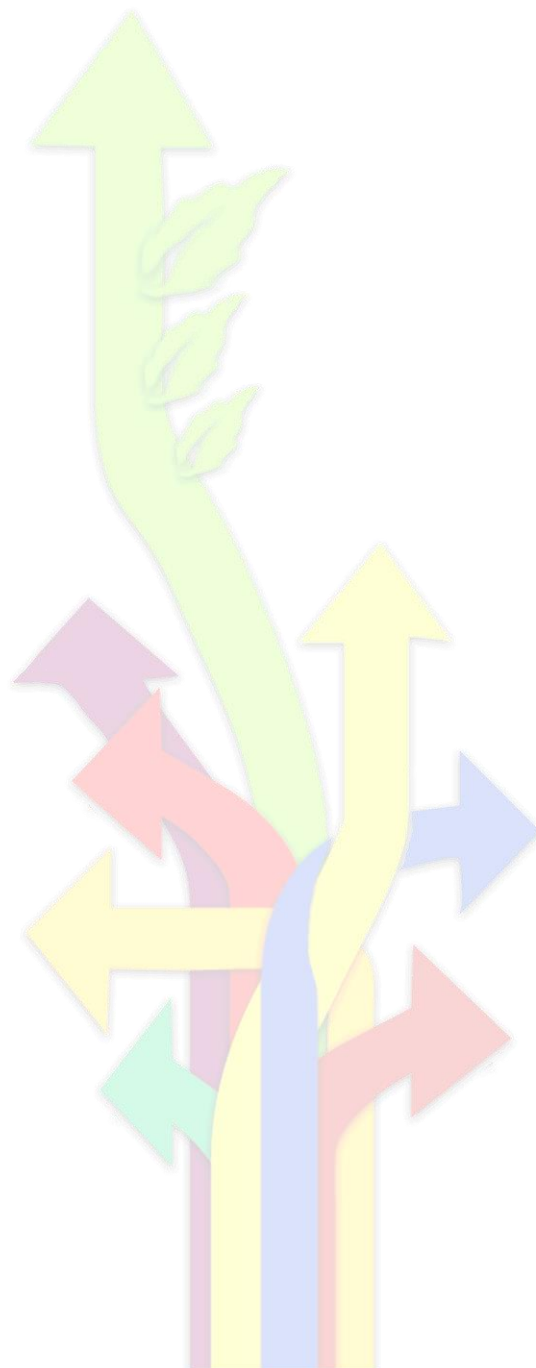
In this work we fabricate a Ni-GO catalyst, for ethanol oxidation in alkaline media. Graphene oxide was synthesized by oxidation of graphite at room temperature with modified hummer method. Then Nickel and Graphene oxide were electrodeposited on copper surface. We used electrochemical techniques such as cyclic voltammetry, Chronoamperometry, and electrochemical impedance spectroscopy. By comparing the electrochemical behavior of two synthesized electrode (Ni and Ni-graphene oxide) it can be conclude that Ni-GO can present a better catalytic activity and stability toward EOR, than Ni electrode, at room temperature. This behavior improvement is due to the presence of graphene oxide as a porous base material that can increase the active surface area of electrode.

Keywords: Nickel, Graphene oxide, High Active Surface Area, Catalytic activity

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13th Annual Electrochemistry Seminar of Iran

Retrofitting technology to achieve zero gap membrane electrolyzers in chlor-alkali industries

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Abstract

The chlor-alkali industry is the industry that produces Chlorine (Cl_2) and alkali, Sodium hydroxide (NaOH) or Potassium hydroxide (KOH), by electrolysis of a salt solution. The main technologies applied for chlor-alkali production are mercury, diaphragm and membrane cell electrolysis, mainly using sodium chloride (NaCl) as feed or to a lesser extent using potassium chloride (KCl) for the production of potassium hydroxide. The diaphragm cell process (Griesheim cell, 1880s) and the mercury cell process (Castner-Kellner cell, 1890s) were both introduced in the late 1800s. The membrane cell process was developed much more recently (1970). Each of these processes represents a different method of keeping the chlorine produced at the anode separate from the caustic soda and hydrogen produced, directly or indirectly, at the cathode. Currently, 95% of world chlorine production is obtained by the chlor-alkali process. Chlorine is largely used in the synthesis of chlorinated organic compounds. VCM for the synthesis of PVC still remains the driver of chlor-alkali production in most European countries. The Mercury cell process, being phased out worldwide because of the toxic character of Mercury and the diaphragm cell process. Best Available Techniques for the production of chlor-alkali is considered to be membrane technology [1]. Membrane electrolytes are advantageous to others, including; environmental friendly, less power consumption, high efficiency and easy operation. There are several electrolyzer design licensors, each of which has its own technology, such as ThyssenKrupp, Ineos. AKC, AGC. Uhdenora (one of ThyssenKrupp's subsidiaries) is one of the most famous licensor which established BM-2.72 single element (one anode, one cathode and one membrane in between). Version I/II of this type was introduced in early 1990s and was developed until the last version (ver. 6) in 2012. The main criteria of new version is reducing of power consumption. For example, in version 3 (which has been used in Iran Chlorine plants like Arvand Petrochemical Complex), the distance between anode and cathode is 1.2 mm (called

Narrow Gap Cells) and power consumption is about 2170 kWh/1 ton NaOH 100% at current density of 6 kA/m². By reducing the gap between the electrodes, Uhdenora started to decrease energy consumption and finally found version 6 (called Zero Gap) and much less electricity consumption (about 2020-2035 kWh/1 ton NaOH 100% at current density of 6 kA/m²). Uhdenora is trying to invent version 7 with the lower energy consumption which is still unknown for clients. One interesting technology which is going to be common in the world is direct modification of single electrolyzer version 3 or 4 to almost zero gap [2, 3, 4, 5]. This change in the distance of the electrodes is carried out with specific knowledge and instruments and really is worth the research.

Keywords: *Chlor-alkali, Membrane Electrolyzer, Zero Gap Electrolyzer, Retrofitting Technology.*

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Electrochemical evaluation of Pb – Ca – Sn - Sr alloy for positive grid of lead-acid battery

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Abstract

The electrochemical behavior of positive grid alloy is a critical issue for lead-acid battery. In order to develop a favorable grid alloy, considerable efforts have been made in the way of adding various alloying elements. Due to high water consumption of batteries with antimony in grid alloys, positive grids containing Pb-Ca-Sn alloy were introduced as the first choice for lead acid batteries due to their effect on the mechanical and electrochemical properties of the lead. However, the grids containing calcium which enhance the age harden ability of grid suffers from early losing the capacity during cycling, called premature capacity loss (PCL). This phenomenon was found to be due to formation of a layer composed of PbO, PbO_n (1 < n < 2) and PbO₂ at the grid/positive active mass interface. As known, the PbO layer has high ohmic resistance. If the rate of PbO formation is higher than the rate of its oxidation to PbO₂, a thick layer of PbO with high ohmic resistance formed on the grid surface which leads to high polarization of the plate on discharge and eventually to capacity loss. Therefore, the Sn with value $\geq 1.2\%$ wt is introduced to accelerate the oxidation of PbO to oxides of higher valency and higher electrical conductivity. In case of new application of lead acid batteries called EFB, thin full frames grids with high corrosion resistance is

needed therefore calcium should be kept in a low range to suppress the PCL effect and increase the corrosion resistance of thin grid by increasing sn/ca ratio in positive alloy.

however, the lowered Ca content will drop the grid hardness and grid casting ability. Thus a forth element must be employed to be supplied. The aim of this presentation is to investigate the electrochemical and mechanical behavior of a Pb-Ca-Sn-Sr alloy for positive grid of lead acid battery.

To run the electrochemical experiments, the conventional three-electrode cell was used in which the saturated calomel electrode (SCE) was as reference electrode and platinum foil served as a counter electrode.

All the electrochemical measurements were implemented using Gill AC potentiostat (ACM instruments). The electrochemical cell was a 250 ml beaker. All the electrochemical experiments were carried out at $25 \pm 1^\circ\text{C}$ in 4.8 M H_2SO_4 solution and after each test the solution was renewed.

the cyclic voltammetry test were run from +1800 mV/SCE to +2800 mV/SCE with scan rate of 1500 mV/min for 50 cycles,

Additionally, battery tests were performed on the sample with the best mechanical and electrochemical properties.

The idea of reducing the calcium content of positive plates grid alloy to reduce the PCL effect without losing hardness and castability properties was accomplished with introducing strontium as a new additive to the grid alloy. The results indicated that the effect of adding Sr on electrochemical properties is more profound than only reducing calcium content. To sum up, the potentiostatic test results reveal more than four times lower passive current density for the first three samples, comparing to the standard sample. Indeed, cyclic voltammetry test results show about 40% lower PbO formation peak and about 67% reduced current density at 2400 mV/SCE for sample 1 and 2, which implies lower water consumption comparing to sample 5.

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Due to hardness test results, sample 2 was chosen to make 66 Ah batteries in Nirugostaran battery manufacturing company. The grid casting (gravity casting technology), paste making,

pasting, curing, assembly and formation processes were done in the same condition and time for sample 2 and standard sample (5) and then the manufactured batteries were tested according to B255210-D standard. The result shows about 10% improvement in cold cranking ability, 53% increased charge acceptance, 39% improved cycle life and 50% lower capacity loss after cycle life test which is in line with previously performed electrochemical tests.

Keywords: *Lead-acid battery, Strontium, Cyclic voltammetry, Charge acceptance, Cold cranking*

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Fabrication and investigation of performance of nanocomposite electrodes composed of carbon quantum dots and copper nanoparticles for catalysis of hydrogen evolution reaction

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Abstract

In the energy production and storage systems in an industrial plant, the system's efficiency is an important goal for the manufacturers. So, fabrication improvement and changes of the system compartments can increase the efficiency. The catalysis of reactions involved for electricity production is a good choice for efficiency improvement. Different types of catalysts are designed depending on the nature of process. They can be composed of metals, metal alloys and metal oxides. Carbon-based materials have also been used as a supporting catalyst, due to their high active surface area. In the present work, a composite composed of a carbon-based nanomaterial (carbon quantum dot) and metallic nanoparticles (copper) is used as a catalyst for catalysis of hydrogen evolution reaction. The present research project is oriented in order to introduce a new nanoporous and environmentally-friendly nanocomposite as a candidate for application in energy production and storage systems.

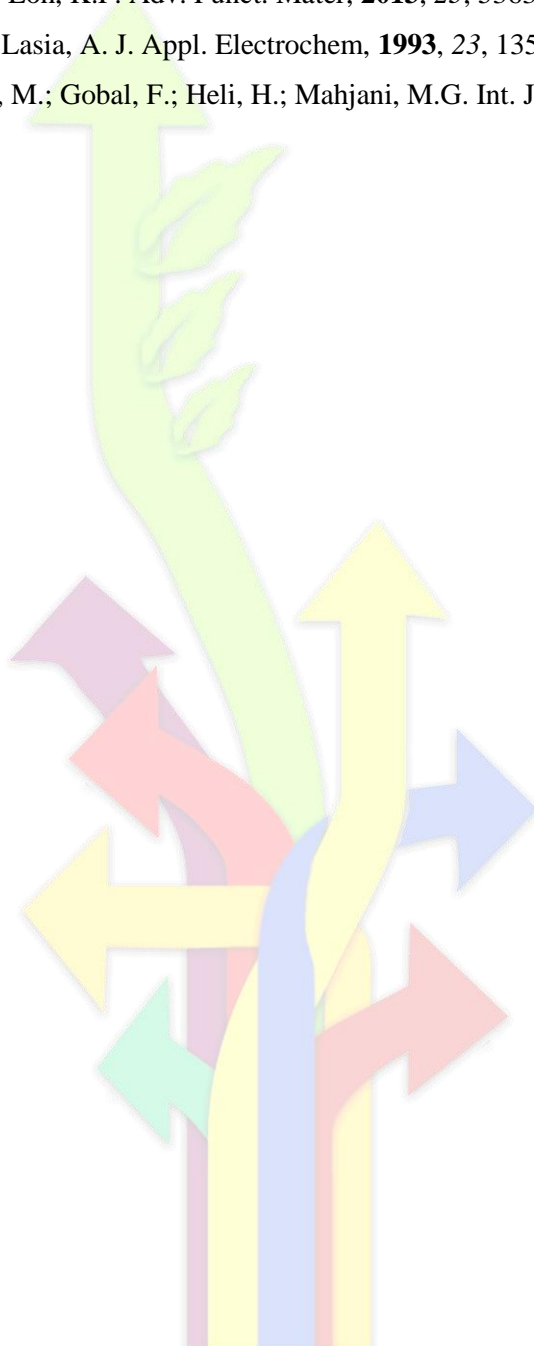
For fabrication of mentioned nano composite we used from electrochemical methods. Electrocatalytic behavior of prepared electrodes also have been studied by linear sweep voltammetry, chronoamperometry and electrochemical impedance spectroscopy techniques. Results show that presence of carbon quantum dots as a porous, suitable and easy synthesis based catalyst caused to increases of active surface area that this effect observed as decreases of overvoltage and increases of current density. Also, by comparing the kinetic quantity such as the tafel slope, the effect of improving the catalytic activity as a result of the presence of quantum carbon is well visible. The stability of prepared nano composite also was investigated by chronoamperometry method during 3h. Finally, the EIS results confirmed the positive effect of proposed nano composite by good electronic conductivity and weak charge resistance.

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Keywords: *Carbon Quantum Dot, High Active Surface Area, Catalytic Property, Nano Composite, Over Voltage*

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Hydrothermal growth of TiO_2 nanorods on the nanocrystalline TiO_2 layer and their application in ZnS and SiO_2 passivated photoanode of CdS sensitized solar cells.

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Abstract

In this research TiO_2 nanorods were grown on the surface of TiO_2 nanocrystalline layers and applied in the ZnS and SiO_2 passivated photoanode of CdS sensitized solar cells. In the ~~st~~ ^{first} part of experiments, TiO_2 nanoparticles were prepared by hydrothermal methods in an acidic medium using titanium tetraisopropoxide (TTIP) as the Ti precursor. Then, a layer of these nanoparticles was deposited on FTO glass substrates with 6- μm thickness. This layer was called as H1 and was sensitized with CdS quantum dots through the successive ionic layer adsorption and reaction (SILAR) process. For improving the photovoltaic performance of quantum dot sensitized solar cells (QDSCs), the photoelectrodes were coated with four cycles of ZnS by immersing in 0.1 M, $\text{Zn}(\text{OAc})_2$ and 0.1 M, Na_2S aqueous solutions for two successive 1 min. Then they were coated with SiO_2 layer by soaking the electrodes in 0.01 M tetraethyl orthosilicate ethanol solution for 1 h followed by rinsing with ethanol and drying in air [1]. The two ZnS and SiO_2 nanocrystalline layers were also deposited as the electron blocking components to decrease the unwanted e-h recombinations. The results show that the QDSC with a photoanode sensitized through 4 cycles of CdS deposition and passivated with ZnS/ SiO_2 layers represents the J_{sc} of $7.18 \text{ mA}/\text{cm}^2$, V_{oc} of 634 mV and energy conversion efficiency of 2.21% (Fig.1a). This efficiency was increased about 78.2% compared to that of the reference cell with H1/CdS/ZnS Photoelectrode.

In the second part of experiments, TiO_2 nanorods were grown on H1 layer by hydrothermal method with titanium tetra-butyl titanate as the Ti precursor. This bi-layer was named as the H1/INRs and was analyzed for the surface morphology. Fig. 2(a, b) demonstrate the top view and cross-sectional SEM images the H1/INRs layer. According to the images the inclined TiO_2 nanorods were grown on the surface of TiO_2 NCs layer. The diameter and length of the NRs were about 75 nm and 2 μm , respectively. The thickness of TiO_2 NRs layer was also measured about 1.9 μm . The photoelectrodes were similarly sensitized with CdS NCs using

SILAR method. The ZnS and SiO₂ blocking layers were also over-deposited on the photoanodes surface for better photovoltaic performance. The results demonstrated that the QDSC with a photoelectrode sensitized through 4 cycles of CdS deposition and passivated with 4 cycles of the ZnS formation represents a J_{sc} of 11.25 mA/cm², V_{oc} of 586 mV and energy conversion efficiency of 3.04% (Fig 3). This efficiency was improved about 13.43% compared to that of the reference cell with H1/INR/CdS/ZnS` photoelectrode.

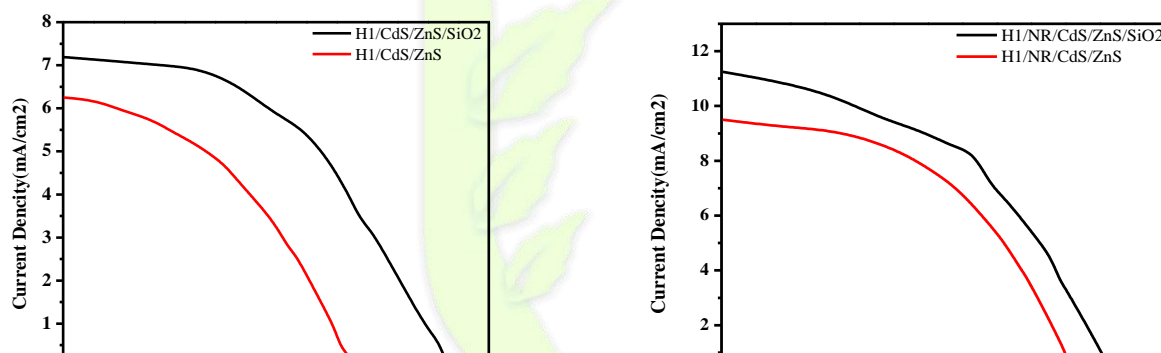
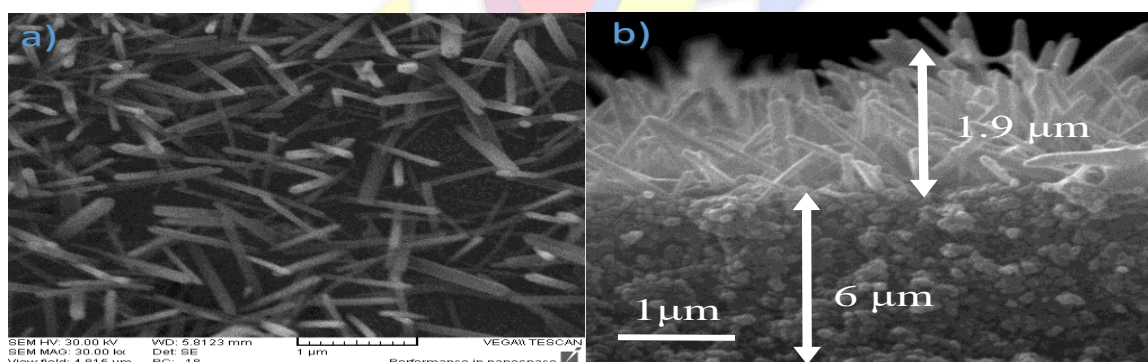


Figure 2: Top (a) and cross-sectional (b) SEM images of the H1/INRs TiO₂ bi-layer.

characteristic of the QDSCs with H1/CdS/ZS and H1/CdS/ZnS/SiO₂ photoelectrodes.

Figure 3. Photo current density-voltage (J-V) characteristic of the QDSCs with H1/INR/CdS/ZS and H1/INR/CdS/ZnS/SiO₂ photoelectrodes



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Keywords: TiO₂ nanorods, Quantum dot- sensitized solar cells, CdS, SiO₂

Preparation of Electrochemically Reduced Graphene Oxide/ Bimetallic Cu-Pt Nanohybrid as Counter Electrode for Fabrication of Dye-Sensitized Solar Cell

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Abstract

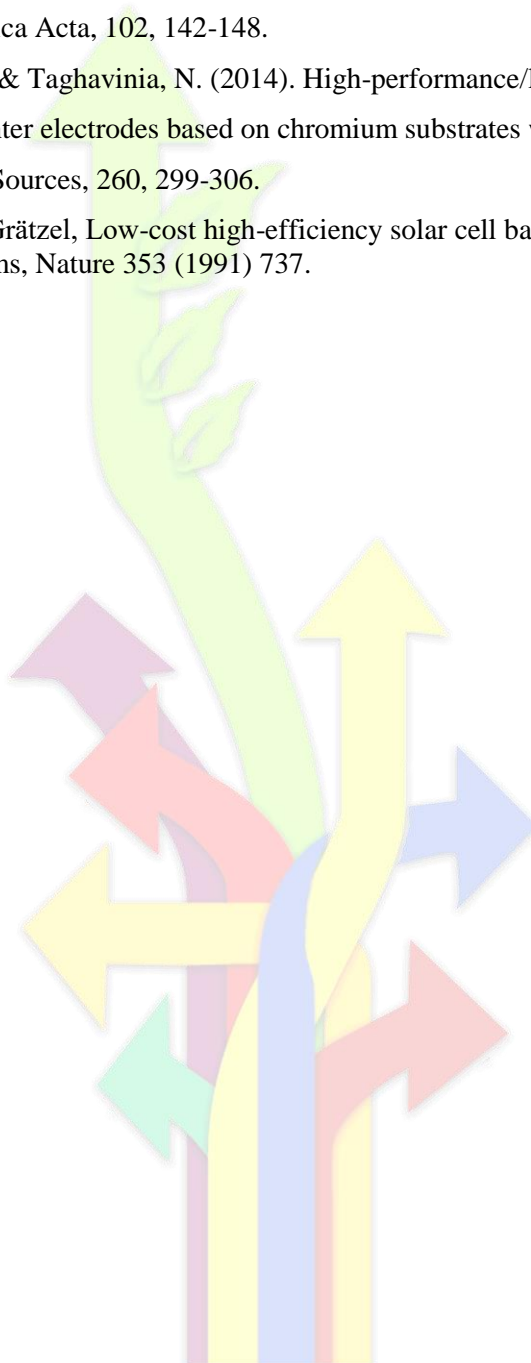
This research was conducted to prepare Cu-Pt nanoparticles incorporated onto electrochemically reduce graphene oxide (ERGO) as the counter electrode (CE) material for the production of dye sensitized solar cell (DSSC). At first, GO was introduced by electrophoretic deposition method on the surface of fluorine doped tin oxide (FTO) electrode. Then, GO nanosheets were converted to ERGO nanosheets by chronoamperometry technique. The Cu nanoparticles were grown on FTO/GO electrode surface by a chronoamperometric method from CuSO_4 (1mM) aqueous solution at specified reduction potential of -0.18 V and during an optimized time of 50 s. Then, the electrode was immersed in H_2PtCl_6 (2mM) to deposit Pt nanoparticles through galvanic replacement with Cu particles. The provided electrodes were studied by FESEM, FT-IR, EDX and ICP spectroscopy. FESEM analysis shows that the surface of the ERGO electrode are decorated with bimetallic Cu-Pt nanoparticles. Some features of CE such high catalytic activity for the reduction of triiodide to iodide ($\text{I}^- / \text{I}_3^-$) and a low charge transfer was investigated using electrochemical impedance (EIS), cyclic voltammetry (CV) and Tafel polarization. Also, the TiO_2 photoanode was prepared onto pre-cleaned FTO glass by using a doctor-blade technique. The photoanode and ERGO/Cu-Pt CE were assembled into cell with a Surlyn. Finally, the energy conversion efficiency of prepared cell was investigated using an AM 1/5 beam.

Keywords: Dye-sensitized solar cell, Counter electrode, Graphene Oxide, Pt-Cu nanoparticles, Electrophoretic deposition, Galvanic replacement

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Preparation of nanoporous silicon structure for antireflection applications

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Abstract

A simple and effective method is presented for producing light-emitting nanoporous silicon (PSi). Porous silicon structure was fabricated via metal-assisted chemical etching procedure. The effect of molar etching rate (ρ) was investigated in order to preparing various nanoporous structures. The field emission electron microscopy (FE-SEM) results showed that all etched samples had nanoporous structure and the sample which was immersed into solution with $\rho \sim 80\%$ had smallest porosities which are measured by digimizer software about 88 nm. Also, the reflectivity as low as 5% could be achieved using this molar etch rate.

Keywords: metal-assisted chemical etching, porous silicon, antireflection properties

Introduction

Silicon based nanocrystallines/nanoporous are some new photoelectronic and informational materials developed rapidly in recent years. For a long time, silicon has been considered unsuitable for optoelectronic applications because bulk silicon emits hardly any useful light due to its indirect band gap nature [2]. This opinion was deeply changed after the discovery of bright emission from porous silicon (PS) and nanocrystals [3]. PSi was first discovered in 1956 by Uhlir during silicon electropolishing experiments [4]. In this study, we fabricated various nano-porous structures by metal-assisted chemical etching. In order to investigation of their optical properties, their antireflection properties were investigated.

Method

Poly crystal silicon wafers with (100) orientation doped with boron (p-type) and resistance about 1-3 $\Omega \cdot \text{cm}$ (Bayern, Germany) were used. The wafers were cleaved into $1 \times 1 \text{ cm}^2$ pieces. Substrates were immersed into a solution containing HNO_3 (65%, Merck)/HF (40%, Merck)/ CH_3COOH (99%, Merck) with volume ratios of 75/10/25 for 15 min prior to use. For removing native oxides on substrates, samples were immersed into Ethanol (96%, Merck) and HF 5% for 10 min and 5 min respectively. Chemical metallization of silver particles on surface of samples was performed by immersing sample in a mixture of HF (0.14M) and AgNO_3 ($5 \times 10^{-4} \text{ M}$) for 3 min. Afterwards the samples were immersed into a solution containing H_2O_2 (35%, Merck)/HF (40%)/Deionized water (DIW) for penetration of silver particles and creation of primary passive porous layer. Details of concentration were presented in table1. By starting the etch process on surface bubbles were formed on the

surfaces. Then the samples were rinsed by DIW and dried at 100 °C for 10 min. Potassium hydroxide (KOH) 1M solution was used for creating post-passive porous layer for 60 s. After second washing, the samples were dried at 100 °C for 10 min. Finally, silver particles were removed by immersion into HF/HNO₃ solution with equal ratio for 2 min.

Table 1: Combination of etching solution and molar etching ratio

Samples	Molar etching ratio (%)	Concentration of HF/H ₂ O ₂
1	80	14/3.5
2	84	14/2.6
3	88	14/1.9

Results and Discussion

The FESEM images of metallized Si samples shows the uniform island layer (in fig.1B). The average size of silver nano spheres has been calculated using digimizer software and determined to be about 70 nm. The average distance of nano spheres has been estimated to be around 58 nm.

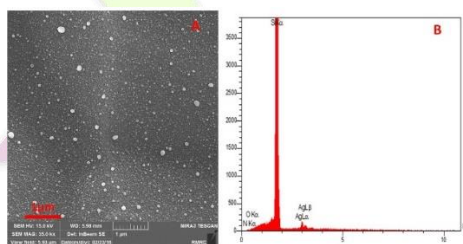


Fig.4: FE-SEM image of coated silver nanoparticles on a silicon substrate and (b) EDS analysis image of the sample surface

The morphology and the surface structure of etched wafers after immersing in etch solutions with different ratios of HF/H₂O₂ are shown in Fig.2. The surface of all samples has almost uniform porosity. Increasing amount of H₂O₂ results in increasing the size of porosities

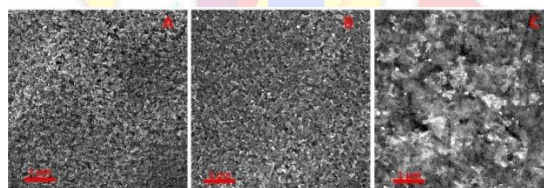


Fig.2: FE-SEM images of the etched surface samples in different concentrations of etching solutions (a) sample1, (b) sample2, and (c) sample3

The reflection spectra of visible beam has been showed in fig.3. It is reflected from surface of etched samples at different molar etching ration relative to the reflection spectra of standard sample. As can be seen the third sample has the effective reflectance up to 5 % which is lowest reflection.

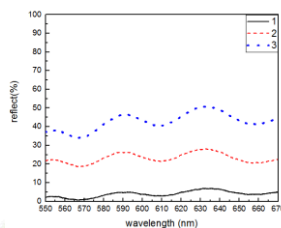


Fig. 3. Optical reflection spectra of the etched silicon wafers in different molar etching ratio (1) sample1 , (2) sample2 , and (3) sample3

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The effect of etching time of porous silicon On solar cell performance

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Abstract

Silicon is one of the most useful and low cost semiconductor material among others and its technology is the most advanced material, well-developed and least expensive compared to the technology of complex other semiconductor and it is one of the most widespread surrounding materials and one of the most common [1]. Silicon based nanocrystallines/nanoporous are some new photoelectronic and informational materials developed rapidly in recent years. For a long time, silicon has been considered unsuitable for optoelectronic applications because bulk silicon emits hardly any useful light due to its indirect band gap nature [2]. This opinion was deeply changed after the discovery of bright emission from porous silicon (PSi) and nanocrystals [3]. PSi was first discovered in 1956 by Uhler [4] during silicon electropolishing experiments. Since then not much attention was paid to this PSi layer but from the 1990s it has been under extensive investigation after the discovery of the light emitting properties of nano Psi in the visible region by L. Canham [5], who showed room-temperature photoluminescence of an anodized p-type silicon wafer [6]. Porous silicon (PS) layers based on crystalline silicon (c-Si) n-type wafers with (1 0 0) orientation were prepared using electrochemical etching process at different etching times. The optimal etching time for fabricating the PS layers is 20 min. Nanopores were produced on the PS layer with an average diameter of 5.7 nm. These increased the porosity to 91%. The reduction in the average crystallite size was confirmed by an increase in the broadening of the FWHM as estimated from XRD measurements. The photoluminescence (PL) peaks intensities increased with increasing porosity and showed a greater blue shift in luminescence. Stronger Raman spectral intensity was observed, which shifted and broadened to a lower wave numbers of 514.5 cm⁻¹ as a function of etching time. The lowest effective reflectance of the PS layers was obtained at 20 min etching time. The PS exhibited excellent light-trapping at wavelengths ranging from 400 to 1000 nm. The fabrication of the solar cells based on the PS anti-reflection coating (ARC) layers achieved its highest efficiency at 20

min etching time. The I–V characteristics were studied under 100 mW/cm² illumination conditions.

Keywords: *porous silicon, etching, solar cell, performance*

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Study of photoelectrochemical effect of PbS with CuS

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Abstract

Solar energy has been considered as a renewable energy source in recent years. To avoid global warming and provide energy that people needs, the energy production of the sun plays an essential role. installing solar cells can provide the electricity, also with this way the cost of the electricity is getting reduced by half, that the companies demand from subscribers. Nowadays in industry uses silicon cells that have high costs and efficiency of 10%. For this reasons, researchers are looking for a replacement for silicon. In this research $(pbs)_x(cus)_x$ were grown on ITO glass substrates by successive ionic layer adsorption and reaction (SILAR) technique (1). Different thin film cells were made of various cus and pbs ratio. Film's behavior was studied in light and dark with electrochemical techniques (cyclic voltammetry and chronoamperometry) in temperature room(2,3). $(pbs)_x(cus)_x$ film was much more sensitive to light and dark than ITO glass. As the ratio of cus to pbs increased, this sensitivity became more.

Keywords: Solar cells, Electrochemical methods, Renewable energy, SILAR method

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Synthesis of ZnO-Cu doped ZnO homojunction photoanode for photoelectrochemical water splitting under visible light irradiation

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Abstract

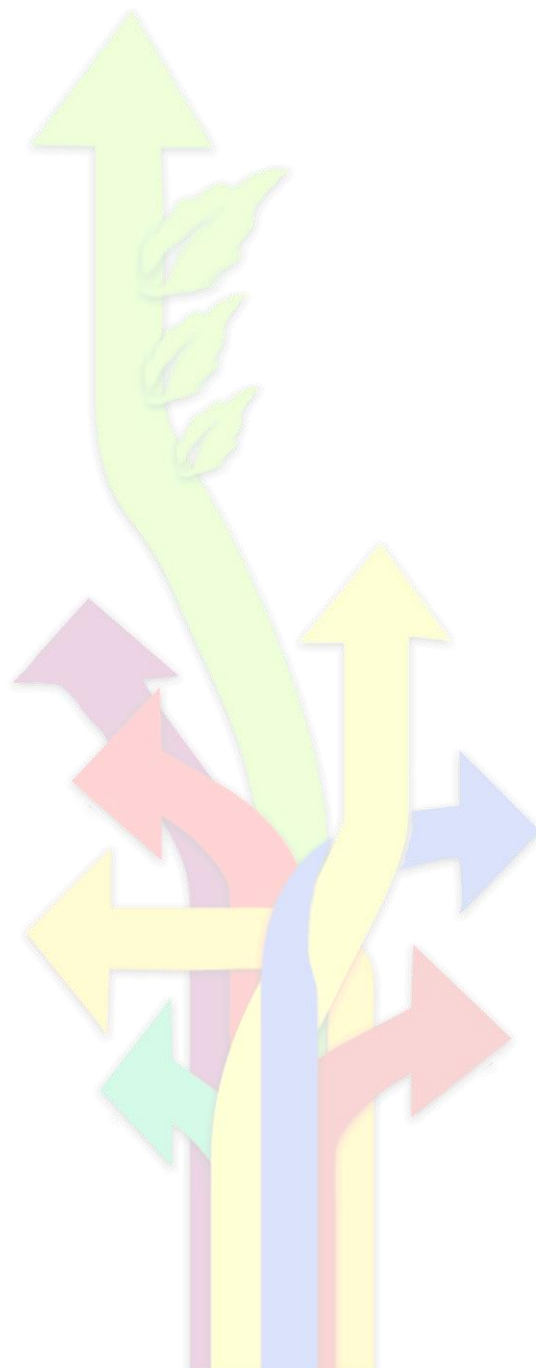
The photoelectrochemical (PEC) splitting of water utilizing sunlight to produce hydrogen has attracted much attention in light of its potential to store solar energy in a convenient way [1, 2]. Among materials for PEC applications, ZnO has been widely investigated as a photoanode because of its favorable band-edge position, high photocatalytic activity, high electron mobility, low cost, non-toxicity, thermal and chemical stability but, photocatalytic properties of ZnO often limited by recombination of photoexcited electron-hole pairs [3]. This paper describes the fabrication of a ZnO-Cu doped ZnO homojunction (ZnO/ZnO:Cu) photoanode by facile and cost-effective electrodeposition method at low temperature to improve the charge separation for enhanced photoelectrochemical water oxidation. ZnO/ZnO:Cu photoanode achieved a maximum photocurrent density of $\sim 50 \mu\text{A}/\text{cm}^2$ at 1 V vs. saturated calomel electrode (SCE), which was about 2 orders of magnitudes higher than the pristine ZnO nanorods (ZnO NRs) under visible light irradiation. This study reveals that ZnO/ZnO:Cu photoanode could facilitate the separation and restrain the recombination of photo-generated electron-hole pairs. Although both Cu-doped ZnO (ZnO:Cu) and ZnO/ZnO:Cu photoanodes exhibit better PEC performances in compare with ZnO NRs photoanode, ZnO/ZnO:Cu photoanode presents an additional charge separation effect due to band bending structure. Motte-Schottky plots show that the flat band potential of ZnO/ZnO:Cu photoanode is more negative than that of pure ZnO NRs, which is beneficial for water splitting.

Keywords: Charge separation, Electrodeposition, Photoelectrochemical water splitting

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13th Annual Electrochemistry Seminar of Iran

Strontium Manganese Oxide Nanostructures as an Efficient Catalyst for Water Splitting Reaction

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Abstract

Hydrogen is known as an important carriers of energy that could be releases the energy by directly combustion, production of steam, operation of fuel cell and combustion by catalysis. Hydroelectric, wind energy, solar energy, geothermal energy and photovoltaic energy could be used for oxidation of water and producing the hydrogen [1]. Therefore, the oxidation of water is an efficient process that limited by the selection of methods and efficiency of used catalysts [2]. So finding the low cost and environmental friendly catalyst for splitting of water. Among the different elements, Mn is an attractive element for water splitting because of the suitable properties such as earth abundance, environmental friendly and low cost [3]. On the other hand, it is an efficient catalyst that used by nature for oxidizing water on plants, cyanobacteria and algae which called water oxidizing complex (WOC) [4]. Different compound of manganese are used in various fields such as catalysis, batteries, magnetite and electrochemistry [5]. Manganese is known as one of the important transition metals which is required for the survival and growth of numerous alive organisms. Attention to the chemistry of manganese compound depicts that the electron transfer reaction is the main reaction on biological systems [6]. One of the requisite for having a sustainable hydrogen economy is molding of technical methods to split water via the sun light and using of catalysts which used in photosystem (II) to create the aerobic atmosphere on the earth. Generation of hydrogen by water splitting is an import goal to store the solar energy. Finding and developing the efficient catalysts for splitting of water to H₂ and O₂ is one of the greatest challenge to create the big plan for oxidation of water [7]. The principal focus of this investigation is to prepare strontium manganese oxide by theultrasonic, methods. The influence of calcination temperature, and ultrasound irradiation power, and the presence of surfactant were investigated on morphology and size of strontium manganese oxid nanostructures. As-prepared nanoparticles were characterized by X-ray diffraction (XRD),

scanning electron microscope (SEM), transmission electron microscopy (TEM), Fourier transform infrared spectroscopy (FT-IR), X-ray energy dispersive spectroscopy (EDS) and ultraviolet–visible (UV–Vis) spectroscopy. Results have been indicated that by changing in method and reaction condition, product appeared in different size, morphology, and uniformity. The morphology and size of nanostructures have been influenced on the properties of nano-SrMnO₃. For investigation the properties of the SrMnO₃ was used in catalytic water splitting for O₂ evolution in presence of (NH₄)₂Ce(NO₃)₆. The effect of nano-catalysts and the concentration of (NH₄)₂Ce(NO₃)₆ have been studied on O₂ evolution reaction. Results show that the efficiency of water splitting have been increased by enhancement in the size and uniformity of catalysts and the results were introduced the SrMnO₃ nanostructures as a new and efficient catalyst for O₂ evolution reaction. Figure 1 shows the TOF and TON diagram for water splitting reaction by SrMnO₃ catalyst.

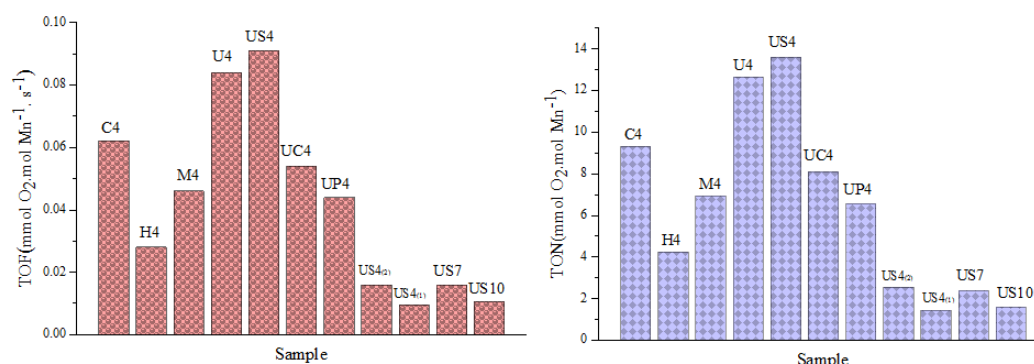


Figure 1 The TOF and TON diagram

Keywords:

Water splitting, H₂ and O₂ generation, SrMnO₃, Chemical synthesis, Nanoparticles

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Sol-gel preparation and electrocatalytic properties of RuO_2 for oxygen evolution reaction in alkaline water electrolysis

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Abstract

Electrochemically splitting water into oxygen, electrons and protons occurs at the anode. The protons transfer to the cathode, to be reduced to molecular hydrogen which is noteworthy today as a carbon-free renewable energy carrier [1]. Alkaline water electrolysis is proposed as a harmless and reliable technology. Currently, it is well-known the overpotential of anodic oxygen evolution reaction (OER) depends on the catalyst components [2]. RuO_2 as efficient and stable OER catalyst displays an exchange current density comparable to Pt, but much lower cost [3, 4].

In this work, we report a sol-gel synthesis method of ruthenium oxides in alcoholic medium followed by thermal decomposition of polymeric precursors that allows to minimize final price.

We used nickel sheets as the coating bases. The nickel sheets were first polished completely and were degreased with caustic soda, hydrochloric acid, and finally, acetone. The sol-gel method was used for coating the bases. For this purpose, a suspension of $\text{RuCl}_2 \cdot x\text{H}_2\text{O}$ and isopropyl or isobutyl alcohols precursors was made as the sol solution. Also, to study the effect of nickel ion, $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ was added simultaneously. For covering the nickel sheets by sol suspension, the method of immersion-drying was used. Coated specimens were transferred to the furnace at 350°C for thermal treatment. The immersion-drying steps followed by thermal curing were repeated 8 times. Finally, the sample was heated to 400°C for 30 minutes.

The voltammetric techniques were used to survey the efficiency and compare the results of the prepared prototypes. The cyclic and linear sweep voltmetry was performed using an Autolab potentiostat-galvanostat with a scanning rate of 10 mV/s in a three electrodes setup consisting of the prepared working electrode, platinum rod as the auxiliary electrode and Ag/AgCl reference electrode in the presence of 1 M KOH electrolyte. Working electrode

were possessed the surface area of 0.5 cm^2 coating. The material specification of coating specimens is shown in Table 1.

Figure 1 compares the obtained results. It can be seen that the sol-gel coatings of ruthenium oxide improve significantly the catalytic properties of oxygen production in OER than bare nickel. Ruthenium metal is expected to be much more active in terms of chemical activity, having the overpotential to be less. Therefore, the presence of ruthenium and nickel depletion in coatings leads to lower onset potentials and larger TOEFL slopes. On the other hand, the catalytic activity of the anode is limited by the excessive increase in ruthenium ratio in the coating content. This proves that metal/metal oxide nature and composition of composite coating is very important in the catalytic activity of the anode. Also, the addition of nickel ions to sol suspension is accompanied by a decrease in the TOEFL slopes, which confirms the weaker catalytic ability of nickel oxides for the OER. However, optimizing the amount of nickel salt in the coating layer can significantly reduce the cost of production.

Table 1- material specification of coating specimens.

Sample code	Base metal	Sol Components			
		$\text{RuCl}_2 \cdot x\text{H}_2\text{O}$	Iso Butanol	Propanol	$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$
		/g	/ml	/ml	/g
A1	Ni	0	0	0	0
A2	Ni	0.1	0.5	0	0.1
A3	Ni	0.04	0.5	0	0
A4	Ni	0.04	0	0.5	0
A5	Ni	0.04	0.5	0	0.1
A6	Ni	0.1	0.5	0	0

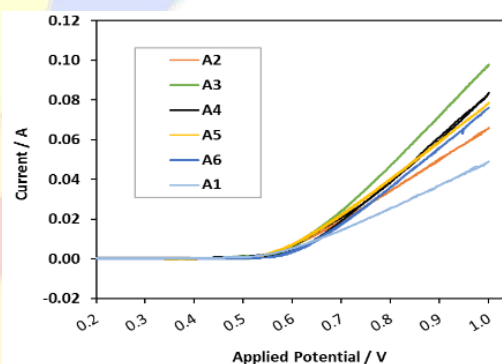
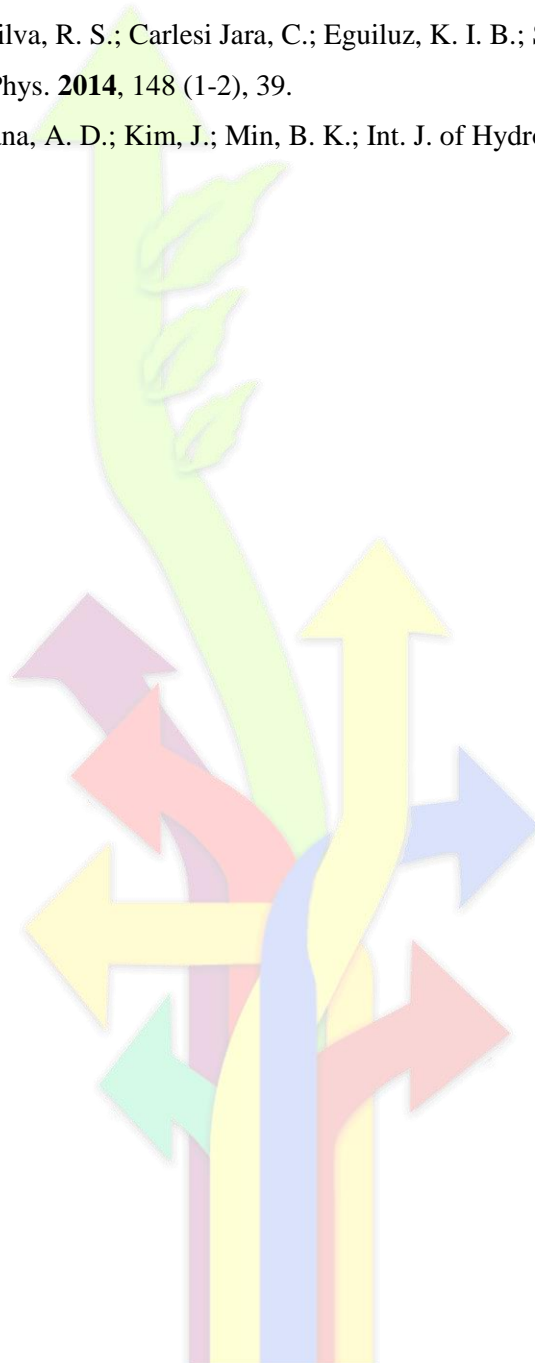


Figure 1. Cyclic voltamograms of specimens.

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Sonoelectrochemical synthesis of polypyrrole-cadmium sulfide-graphene oxide nanocomposite as an efficient photocatalyst for photoelectrochemical hydrogen evolution

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Abstract

Considering the consuming of fossile fuel and growing of environmental pollution, hydrogen energy has attracted great interest because of its environmentally clean nature and high value of combustion. Today electrochemical energy conversion is growing dramatically due to the growth in global demand for energy sources. However, due to the existance of a large overpotential for hydrogen evolution reaction, suitable catalysts are necessary to increase the hydrogen production efficiency. Platinum metal is commonly used as a well-stablished electrocatalyst owing to its low overpotential. However, Because of high price and limited world-wide supply of noble metals, their amounts used in energy technologies should be decreased. In recent years, research on semiconductor-based photocatalysts has received more and more attention because of the problems of solar energy and environmental pollution. Among semiconductor photocatalysts, CdS is one of the most efficient visible-light-driven photocatalysts because its bandgap is narrow (2.4 eV) and its condction band edge is more negative than the $\text{H}_2\text{O}/\text{H}_2$ electrode potential. However, the bare CdS still suffers from the quick recombination of photogenerated charge carriers and the instablity owing to photocorrosion. It is commonly accepted that co-catalysts play an important role in transfer photoinduced electron and improve dispersion of photocatalyst. Conducting polymers such as polypyrrole have aroused great interest to the researchers because of their excellent electronic, magnetic and optical properties and using conducting polymers as photosensitizers to modify semiconductors photocatalysts has been studied. Graphen oxide (GO) due to its high specific surface area, high carrier mobility and also high content of the oxygen containing functional groups in the basal planes and the edges can provide anchoring groups wich caused to bind inorganic materials onto GO [1-6]. The present article reports the sonoeleostrosynthesis of PPy-CdS-GO nanocomposite as a photocatalyst for H_2 evolution reaction. For this purpose, CdS nanospheres synthesized by sonochemical method with

average size distribution about 25.86 nm that was determined by XRD and SEM techniques. Then GO was synthesized by modified Hummer method and at last an in situ sonoelectrochemical method was employed to synthesis of PPy-CdS-GO nanocomposite on anodized copper substrate. After design, preparation and characterization of nanocomposite, its photoelectrochemical performance for photoelectrochemical hydrogen evolution was investigated in neutral solution under visible light irradiation by linear sweep voltammetry, electrochemical impedance spectroscopy and chronoamperometry. Results of electrochemical investigations proved that nanocomposites containing CdS nanoparticles are sensitive to visible light. Also investigations showed the decrease of overpotential and enhancement of current density for nanocomposite compared with polypyrrole coating. The overpotential for PPy-CdS-GO nanocomposite, 0.9 V/SHE was determined. Tafel slope evaluation exhibit that hydrogen desorption from the substrate obeys from Volmer-Tafel mechanism. Also mechanism of photocatalytic hydrogen evolution discussed that graphene oxide nanosheets have a predominant effect on charge transfer and enhancement of H₂ evolution on electrode surface. In fact in this study an efficient photocatalyst with composed of conductive polymer polypyrrole, cadmium sulfide semiconductor and graphene oxide nanosheets was prepared and their synergistic effect considered.

Keywords: *Hydrogen evolution reaction, Photoelectrochemical hydrogen evolution, Nanocomposite, Photocatalyst, Sonoelectrochemical synthesis.*

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Nickel oxide/porous reduced graphene oxide as active hybrid material for oxygen evolution reaction

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Abstract

Porous reduced graphene oxide (prGO) possesses all the advantages of rGO nanosheets for electrochemical sensing (1) such as increased active area and facilitated mass transport, both being advantageous for sensitive sensing. Surprisingly is that while prGO architectures have been widely used for the fabrication of high-performance energy storage devices (2,3), the use of prGO for sensing is limited to some reports. Here, a simple and efficient routine is presented by decoration of prGO with nickel oxide (NiO) particles. The morphology of NiO-prGO was investigated by using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS). In alkaline media, the carbon paste electrode (CPE) modified with NiO-prGO catalyses oxygen evolution reaction (OER) with an onset potential of 0.6 V (vs. Ag|AgCl|KCl_{3M}), showing a synergistic effect between prGO and NiO particles. After the long-term *I-t* measurment, high electrochemical stability confirms the improved electrocatalytic performance of NiO-prGO. This study affords us NiO-prGO based electrocatalyst with high performance and strong durability under alkaline conditions, which can be applied to energy conversion and storage.

Keywords: *Porous reduced graphene oxide, Nickel oxide particles, Oxygen evolution reaction, Electrocatalyst*

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Electrocatalytic Oxidation of Methanol on a Glassy Carbon modified electrode by Carbon Nanotubes, Poly(4-methyl-ortho-phenylenediamine) and platinum NanoParticles

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Abstract

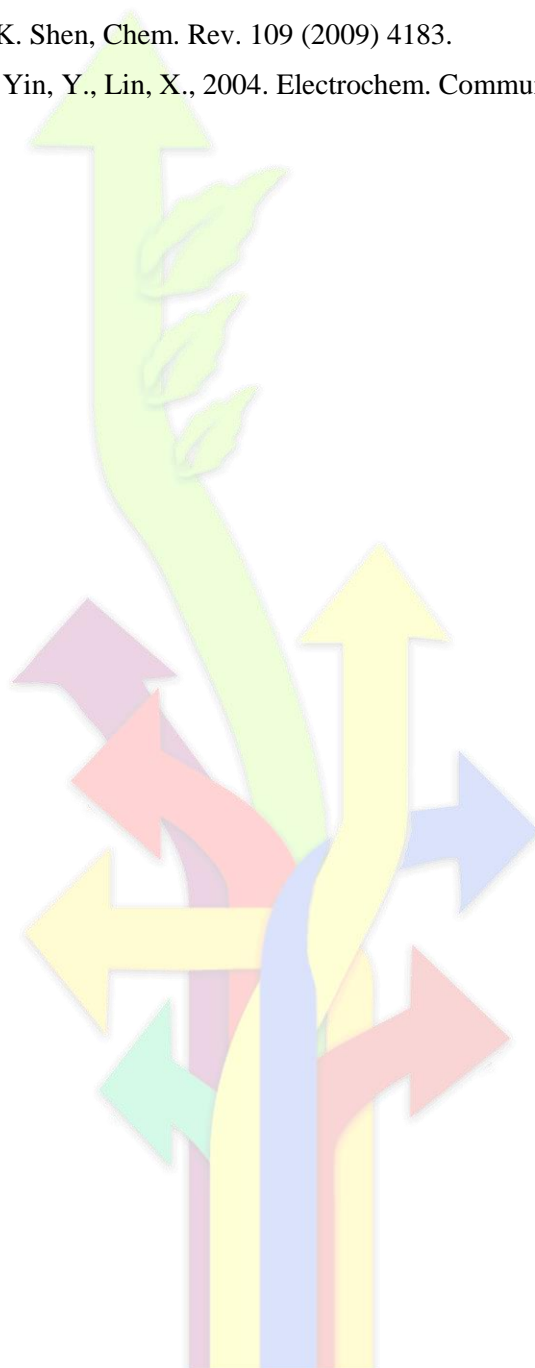
In recent years, fuel cells have become a hot topic because of the decrease in fossil fuel reserves and the serious environmental pollution caused by the use of these fossil fuels. Fuel cells have shown great potential as highly efficient and low-emission power sources for portable electronic devices. Alcohols, like methanol and ethanol, are widely recognized as the most promising candidates for fuel cells (1-4).

Electrocatalytic oxidation of methanol on a glassy carbon electrode coated with carbon nanotubes, poly (4-methyl-ortho-phenylenediamine) (4-MoPD) and platinum nanoparticles was studied by cyclic voltammetry in an alkaline medium (0.50 M NaOH). The Pt nanoclusters were electrochemically deposited on the electrodes under multi-cyclic voltammetry in 0.5 mol/L H₂SO₄ and K₂PtCl₆ solution in the potential window of -0.5 to +0.5V versus Ag/AgCl. The physical properties of modified electrode were characterized via scanning electron microscopy (SEM) and X-ray diffraction (XRD). The electro-oxidation of methanol in 0.5 M NaOH was studied using cyclic voltammetry method. The effective parameters included concentration of polymer solution, concentration of platinum solution, number of cycles for polymerization, scan rate for polymerization, number of cycles for electrochemically deposition of Platinum nanoparticles, scan rate for deposition of Platinum and concentration of NaOH solution on the electro-oxidation of methanol were also investigated. In the optimum condition, the prepared electrode showed excellent electrochemically active surface areas (ECSAs), catalytic activity, and stability toward the methanol electro-oxidation reaction in the alkaline media.

Keywords: Methanol, fuel cell, polymer, Platinum nanowire, modified electrode, electro deposition, electro oxidation of methanol.

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13th Annual Electrochemistry Seminar of Iran

A hydrothermal synthesis of porous Pt/NiO/graphene nanocomposite modified glassy carbon electrode and its application in methanol electro-oxidation

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Abstract

The conversion of chemical energy into electrical energy became more important due to the increase in the use of electricity in recent years [1]. The exhaust of fossil fuels and the accompanying environmental pollution have triggered significant interests in developing novel electrode materials for sustainable and clean energy devices. Direct methanol fuel cells (DMFC) have the advantages of high efficiency, low operating temperature, and low pollutant emission, and they are considered as promising power sources [2].

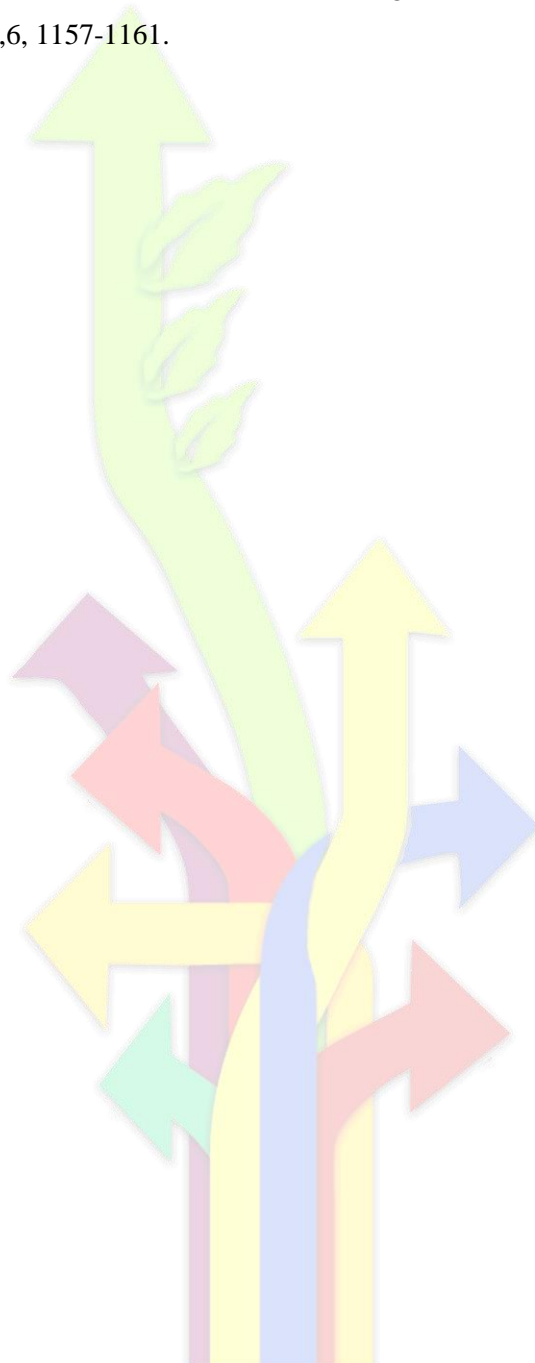
In this work, a glassy carbon electrode was modified with a new porous Pt/NiO/graphene. At first, the glassy carbon electrode surface was cleaned mechanically by polishing with 0.05 μm alumina powder in water slurry on *micro cloth* pads and rinsed thoroughly with distilled, deionized water before modification. Then, the fresh GC electrode was modified with NiO/graphene which has been prepared successfully by the hydrothermal method. The Pt nanostructures were electrochemically deposited on the surface of the modified electrode under successive scans of cyclic voltammetry in 0.5 M H_2SO_4 . The electrochemical properties of the catalysts were evaluated through electrochemical experiments. Structural characterization of the modified electrode was carried out by using electrochemical impedance spectroscopy (EIS), X-ray diffraction (XRD) and scanning electron microscopy (SEM). The electro-oxidation of methanol in 0.1 M NaOH was studied using cyclic voltammetric method. In the optimal condition, the modified electrode exhibits an excellent electrocatalytic activity towards methanol oxidation in the alkaline medium with a high electrochemical active surface areas (ECSAs) and an acceptable stability.

Keywords: Methanol fuel cell, Glassy carbon electrode, Platinum nanostructure, Graphene

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Surface morphology and electrochemical impedance correlation in Co-Pi modified TNA photoanodes

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Abstract

Hydrogen production via photocatalytic water splitting is one of the most auspicious strategies so as to overcome the energy crisis. Despite the existence of various pathways for the conversion of water and sunlight into hydrogen, photoelectrochemical (PEC) water splitting with semiconductor photoelectrodes has attracted much interest [1,2]. One main issue to increase solar hydrogen production in semiconductor PEC systems is reducing required over potential in water oxidation half reaction [3]. Here, an optimized photoanode was designed and electrochemically fabricated in which TiO₂ nanotube arrays (TNA) were fabricated by the anodizing approach and then modified with cobalt phosphate fine particles as electrocatalyst using a potentiostat electro-deposition method. The amount of loaded electrocatalyst was changed by variation of deposition time duration from 10 min to 16 hours and its influence on the photoanode surface morphology and related charge transport resistance was studied using field emission scanning electron microscopy (FESEM) and electrochemical impedance spectroscopy (EIS), respectively. As illustrated in Figure 1a, charge transfer resistance of the bare TNA was about 5.5 k Ω which decreased to 4.5, 4.2 and 4.0 k Ω for the samples modified for 10, 20 and 60 min, respectively and then increased to 5.0 k Ω for the 16 hours Co-Pi electrodeposited TNA. According to FESEM results, the addition of co-catalyst led to filling interspace of nanotubes as well as thickening their walls (Figure 1b, 1c and 1d). More amount of loading resulted in tube mouth closing and accumulation of Co-Pi particles on the whole electrode surface (Figure 1e). The latter case caused the increase in the charge transfer resistance of the sample modified for 16 hours.

Keywords: Co-Pi co catalyst, TiO₂ nanotube, Water splitting, Charge transport.

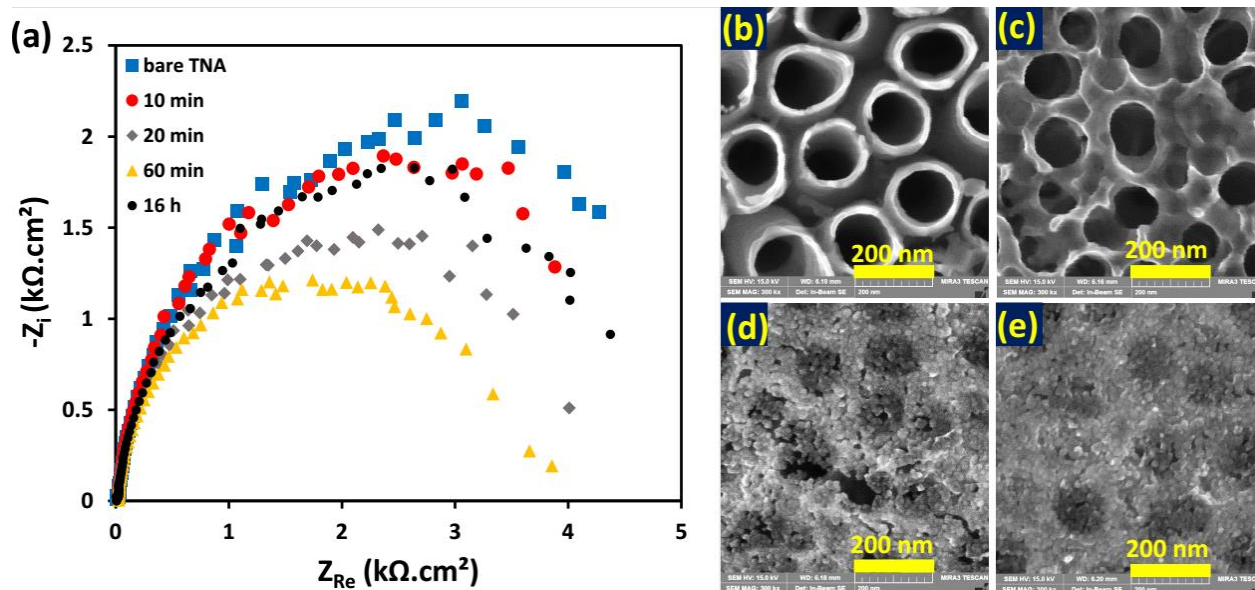
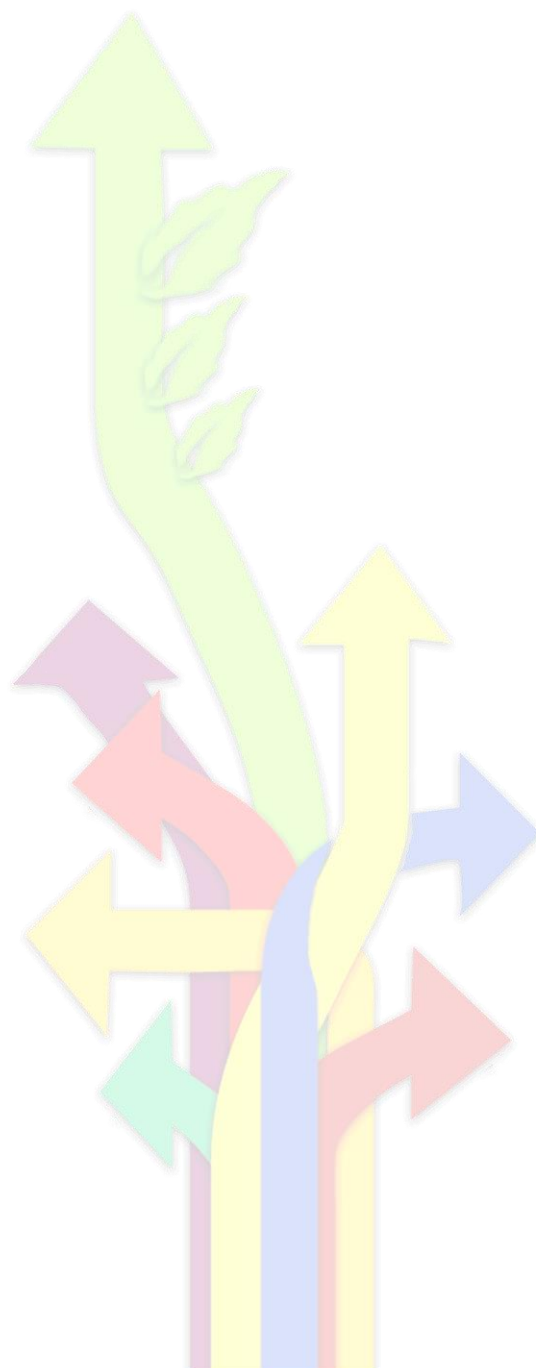


Figure 1. (a) Nyquist plots and FESEM images of (b) bare TNA, TNA modified for (c) 10 min, (d) 60 min and (e) 16 hours.

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Computational



13th Annual Electrochemistry Seminar of Iran

A theoretical atomic-scale study of polymer/metal oxide interface

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Abstract

Organic coatings are thin protective layers that by creating them on the surface of metals, the relationship between the surface and the environment can be interrupted. These materials often include paints, varnishes, rubber and plastic and bitumen coatings [1]. Thin films absorbed on the surface of metal layers create a barrier between metal and corrosive materials [2]. Recently, the use of molecular dynamics simulation software to evaluate the anti-corrosion coatings for metals has increased. Here, molecular dynamics (MD) simulations were applied [3] for comparison of the adhesion of polyethylene terephthalate, polyethylene isophenolate, polybutylene terephthalate and polybutylene isophthalate (Figure 1 (a,b,c,d)) coatings on the iron oxide surfaces. Binding energy parameter ($\Delta E_{\text{binding}}$) calculations show that the polymers coating strongly attached to surfaces of FeO and Fe₂O₃. It was found that among polymers, polyethylene isophthalate had the highest amount of adsorption energy on the Fe₂O₃ (100) surface. The binding energies of polybutylene isophthalate with FeO and Polyethylene isophthalate with Fe₂O₃ were -267.01 and -860.90 kcal/mol, respectively. These negative values quantitatively confirm the interfacial interactions exist between iron oxides surface and coating films.

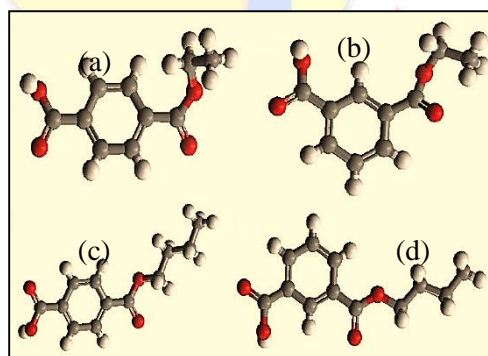


Figure 5- Molecular structures of (a) ethylene terephthalate, (b) ethylene isophenolate, (c) butylene terephthalate, (d) butylene isophthalate.

Keywords: Corrosion, anti-corrosion coating, molecular dynamics simulation.

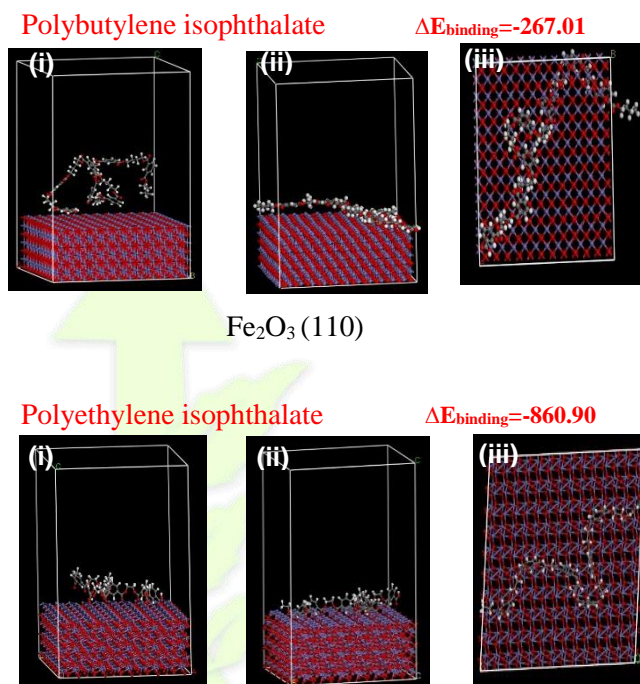


Figure 2- The initial (i) and final (ii) and top views (iii), snapshots of iron oxide substrates interacting with coating polymers.

Table 1- $\Delta E_{\text{binding}}$ that is calculated for each simulation.

FeO (100)	Polyethylene terephthalate	-223.01
	Polyethylene isophthalate	-175.14
	Polybutylene terephthalate	-40.21
	Polybutylene isophthalate	-267.01
Fe ₂ O ₃ (110)	Polyethylene terephthalate	-671.83
	Polyethylene isophthalate	-860.9
	Polybutylene terephthalate	-428.52
	Polybutylene terephthalate	-825.99

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A First-principles study of Na adsorption and diffusion on graphene nanoflakes under the effect of external electric field as an anode material for Na-ion battery

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Abstract

In this work the interaction between Na ion and graphene surface with the absence and the presence of external electric field (EEF) applied perpendicular to the surface was investigated. M06-2X/6-31G(d) DFT calculations were performed to describe the adsorption and diffusion properties. Meaning of the binding energy and scanning the potential energy surface (PES) of the charged molecular systems in the presence of EEF is different from that in the absence of electric field as the binding energy is an anisotropic characteristic which depends on the orientation of molecules with respect to the EEF. With the electric field increased from 0.0 a.u to 0.02 a.u , the diffusion barriers were decreased from 24 to 20 KJ/mol , for the Na ion in the graphene nanoflakes. We concluded that the external electric field can increase the charging speed of rechargeable ion batteries based on the graphene anode materials.

Keywords: External electric field; Grapheme; Rechargeable ion batteries

Introduction

Layered structural materials are convenient for intercalation/deintercalation of metal ions, thus can be used as the appropriate materials for rechargeable ion batteries [1-2]. Na ion batteries have been the subject of intense investigations due to their good cycling performance, high storage capacity and high energy density. Despite all the studies, finding excellent anodes with good electrical conductivity and high reversible Na storage are still under development. Among them, graphite is the key anode material for the commercial Na ion batteries with an energy capacity of 372 mAhg⁻¹. Some experimental results have shown that graphene, a single atomic-layer thickness of graphite, can adsorb higher amounts of Na (e.g. specific capacity of ~540 mAhg⁻¹) than graphite.

In this work, we investigated the effects of an external electric field on adsorption and diffusion of Na ion in the graphene monolayer based on DFT analysis. We have reported that adsorption and mobility of the transporting ion can be significantly enhanced in the graphene monolayer with an external electric field.

Materials and method

First, the model cluster with a molecular formula of $C_{88}H_{26}$ was constructed and fully optimized using the M06-2X meta-hybrid GGA functional merged with the 6-31G(d, p) basis set as implemented in the GAMESS program [3]. Then the Na ion was positioned above the surface. Three different orientations of the ion with respect to the graphene surface, namely, top, hollow and bridge, were considered as starting structures (Fig. 1). In the top site geometry, the Na ion located directly above the C-atom, in the hollow site conformation the ion is above the centre of a hexagon in the graphene layer and in the bridge site structure the ion positioned above the centre of a C-C bond. During the calculation in the absence and presence of different homogeneous external electric fields in which the electric field was directed perpendicularly from the Na ion to the graphene flake, the rigid model was used: the geometry of the cluster was frozen as it obtained in the first step and the optimized ion position was determined by varying the distance between the ion and the graphene plane until the most stable position is found. The site with the largest adsorption energy was ascribed as the most stable site of adsorption.

The binding energies for our systems in the presence of EEf were calculated from Equation

$$E_{binding} = E_{complex}^{field} - (E_{Li}^{field} + E_G^{field}) + FQ(R_1 - R_0)$$

In this equation, complex, Na, and G subscripts denote the complex, Na ion, and graphene surface, respectively. The term $FQ(R_1 - R_0)$ corrects the energy of the reference Na ions for their distance to the coordinate origin in the complexes. In other words, this term measures the electric work which is required to move free ions from the origin to their coordinates in the complexes.

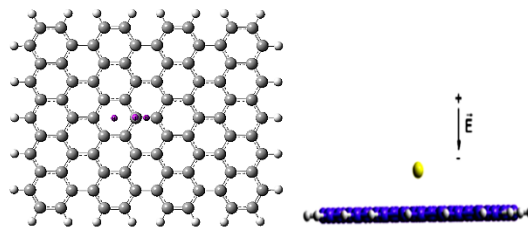


Fig.6: The different orientations of the ion with respect to the graphene surface: (T) top, (H) hollow and (B) bridge (down).

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Quantum Chemical Study of Molecular Nanoelectronic Systems: Graphene Devices

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Abstract

In this work, to study local molecular charge energy transfer in a proposed molecular nanoelectronic field-effect system (as a organic- Graphene device) atomic electronic responses to external electric field (EF) are computed. In addition, the electronic molecular transport coefficient, L_T , is computed based on the density (charge) and energy (kinetic/potential) transfers between different parts/junctions or atomic basins of the molecule, using quantum theory of atoms-in-molecules (QTAIM). In addition, dependencies of the L_T on the electric field intensity are studied. Analysis of the results show that L_T depends approximately linearly on the EF intensity.

Keywords: QTAIM, Molecular Nanoelectronic, Graphen, n/p-molecular Devices, charge/energy transfer

Introduction

The quantum theory of atoms in molecules (QTAIM), is a generalization of quantum mechanics to open systems (as opposed to closed whole systems). QTAIM defines the open systems in terms of the topology of the electron density, $\rho(r)$, that we can calculate, and that determines chemical behavior and reactivity [1-2]. Based on the QTAIM, the total atomic electronic energy, $E_e(\Omega)$, is the sum of involving kinetic and potential energies, $K(\Omega)$ and $V(\Omega)$, as $E_e(\Omega) = K(\Omega) + V(\Omega)$, here Ω denotes the atomic basin. The expectation value of a quantity corresponding to an operator averaged over all space is given by the sum of the expectation values of this operator averaged over the atomic basins, as $\langle A \rangle_{\text{Molecule}} = \sum_{\Omega}^{\text{All Atoms}} A(\Omega)$

Therefore, based on the QTAIM theory, the local intra-molecular electron density, $\rho(r)$, and its Laplacian, $\nabla^2 \rho(r)$, and thus intra-molecular virial forces, of the molecular nanoelectronic (field-effect) systems can be studied. In this work, the electronic and vibrational atomic/molecular responses to external electric field (EF) are computed to detail intramolecular charge and energy transfer in a proposed molecular nanoelectronic field-effect system, as a Graphene Devices, Fig. 1.

Computational Details

In this work, electric field intensity (\mathcal{E}_F) is applied in the x direction and geometry of the molecule is optimized, and electronic properties were studied, for a typical molecular nanoelectronic system, shown in Fig. 1, using DFT/UB3LYP/6-31+G* level of theory. In

addition, starting from the molecular electronic wave functions and using QTAIM, the atomic basins of the Graphen system are determined (using density gradient method) and thus the total atomic electronic energies and their changes are calculated at different EF intensities. In addition, we compute molecular transport coefficient (L_T) based on the response of the molecular electronic density distribution to electric field by $\vec{\nabla} E = L_T \cdot \vec{F}$, $F = \varepsilon_F L$. In this equation, $\vec{\nabla} E$ is the energy transfer (between molecular junctions) due to the electric field (F). Also, ε_F and L are electrical field intensity and distance of the two junctions (or ends) of the molecule.

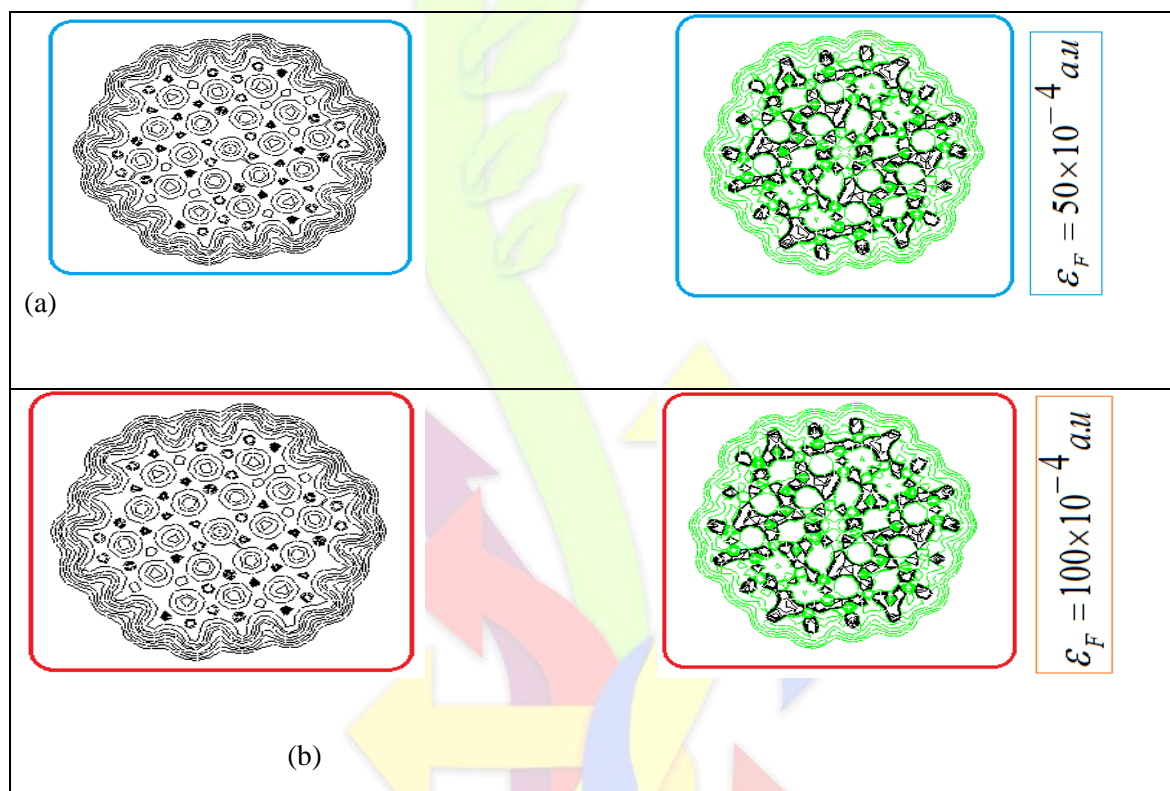


Fig. 1. Electric field effect on the electron density contour maps (left) and its Laplacian (right) of atomic basins of the organic-Graphen system.

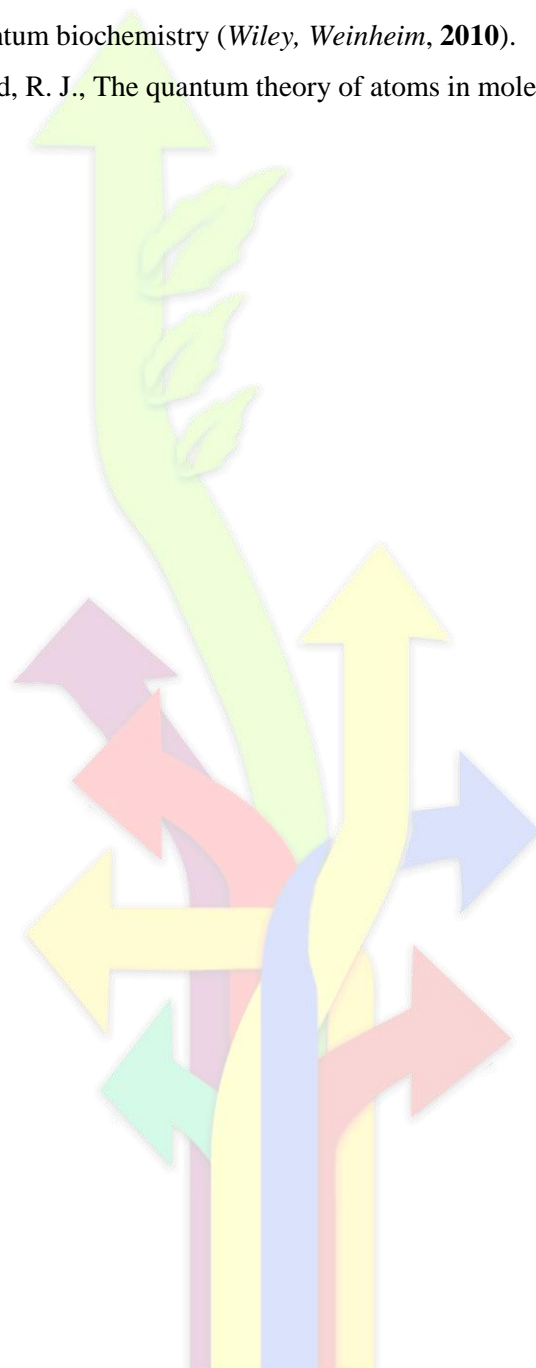
Results and Conclusions

Analysis of these results (not reported here for brevity) show that the atomic electronic properties are generally dependent on the bonding and electronic molecular structures. Also, based on the QTAIM, it is predicted that a single electro-chemical molecular system can be grouped into *p-type-like* (as intra-molecular donor section) and *n-type-like* sections (as intra-molecular acceptor section). In addition, once local intra-molecular charge/energy transfer can be studied (described) via quantum mechanical calculations; it will be possible to calculate the intra-molecular *electro-chemical transport coefficients* also. In addition, these results show that L_T depends approximately *linearly* on the EF

intensity. Such dependencies of the electronic L_T coefficients on the EF intensity obtained here are compatible with what already observed and reported for the electronic transport coefficient of the molecular (n/p)-type semiconductors.

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Nano Electrochemistry

Synthesis of Zinc Sulfide Nanopowder by Chemical Method and purifying it to improve the optical properties

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Abstract

Semiconductor nanoparticles, have different properties in comparison to their bulk particles due to their small dimensional quantum effects. So they have different and special electrical and optical properties. Controlling the synthesized particles size and reducing particle size due to increased energy gap and optimizing the physical and chemical properties is very effective. So one of the most important research areas in the world is producing semiconductor nanoparticles with controlled size. It was also found that the purity of the used materials have high influence on optical properties of sintered parts. As a result, selecting the appropriate method for the processing of optical material with high purity is very important. In this research by choosing the appropriate method, ZnS nanopowder is synthesized and produced with a specific process and then purified to improve the optical properties.

At first, zinc acetate and sodium sulfide were selected as suitable sources for synthesis of zinc sulfide powder. Then by using de-ionized water, the solubilizing of powders were performed. The solutions were then combined into ultrasonic. The Zinc acetate solution was placed into the three-neck round bottom and was heated, in an inert atmosphere consisting of either ultra high purity argon. A sodium sulfide solution was slowly added dropwise to the Zinc acetate solution. The ZnS nanoparticles obtained were separated from the reaction mixture by centrifugation. Washed with ultrapure deionized water and ethanol to remove any trace level impurities. The washed ZnS nanoparticles were then dried in a vacuum oven. The produced nanopowder was placed in a suitable tunnel furnace. And different gases such as air, argon, hydrogen sulfide and hydrogen are blown in order. And then the Purification operation of ZnS nanopowder will be over. Structure of synthesis phases and Particles morphology have investigated by X-Ray diffraction (XRD) and scanning electron microscope (SEM). The average size of nanoparticles synthesized by the acidic method is between 35 and 45 nanometer. And all impurities, including carbon and sulfur, were

extracted from the structure after purification operation. The transition of IR (infrared wave) was measured by FTIR (Fourier transform infrared spectroscopy) more than 50% .

Keywords: *Synthesis, Nanopowder, Zinc Sulfide, Optic*

Reference

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Degradation of Some Environmental Pollutants by Mg-Doped TiO₂ Nanoparticle under UV Light in Aqueous Solution

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Abstract

In this research Mg-doped TiO₂ nanopowders were synthesized by the sol–gel method. The Mg-doped TiO₂ photo-catalyst was prepared using Titanium tetra-iso propoxide and Magnesium sulfate as the dopant precursors. Rhodamine B, Nonylphenol ethoxylates 6 mol, Pseudoephedrine hydrochlorid, and Nicotine were used to study the photocatalytic performance of the Mg doped TiO₂ under UV irradiation. The synthesized samples were characterized by X-ray diffraction, X-ray photoelectron spectroscopy, Transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS) and Photoluminescence (PL). The photocatalytic degradation shows that after 60, 90, 120, and 120min of irradiation time with UV light, 99%, 98%, 98% and 98% of Rhodamine B, Nonylphenol ethoxylates 6 mol, Pseudoephedrine hydrochloride, and Nicotine were decomposed by Mg-doped TiO₂ sample, respectively.

In this work, we synthesized Mg-doped TiO₂ nanopowders by sol-gel method. This photocatalyst were characterized by XRD, TEM, XPS and PL analysis. After than the photocatalyst was applied for degradation of four pollutants such as Rhodamine B, Nonylphenol ethoxylates 6 mol, Pseudoephedrine hydrochlorid, and Nicotine. The maximum degradation for these pollutants were obtained between 98-99%.

Keywords: Mg-doped TiO₂; Photocatalytic degradation; Sol-gel method; environmental pollutant, Photoluminescence.

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Study of the surface area of $\text{LaFe}_{0.95}\text{Zn}_{0.05}\text{O}_3$, $\text{LaFe}_{0.5}\text{Zn}_{0.5}\text{O}_3$ nanoblocks before and after Calcination

M.Moradi, M. Khodabakhshi, R. Gholipour, S.N. Mirnia

Abstract

The improvement of structural properties and the surface area for practical applications is significant. Meanwhile, Perovskite metal oxides are important due to mesoscopic, electrical and optical properties. Increasing the surface area of these materials is very significant. Because the increase of the special surface can improve the electrical, optical and gas properties of the gas. Therefore, due to the electrical, optical and magnetic properties of lactate ferritic oxide, LaFeO_3 , we investigated zinc ion in iron ion locations. In this research, zinc ion was applied at concentrations of 0.05 and 0.5 with sol-gel method. Following the synthesis and clustering of the surface area of nanoparticles, X-ray diffraction (XRD), scanning electron microscopy (SEM) and coherent absorption and desorption charts (BET) were investigated.

Keywords: Specific surface area, Perovskite oxide, Doping, Calcination

Synthesis of Zinc Sulfide Nanopowder by Chemical Method and purifying it to improve the optical properties

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Introduction

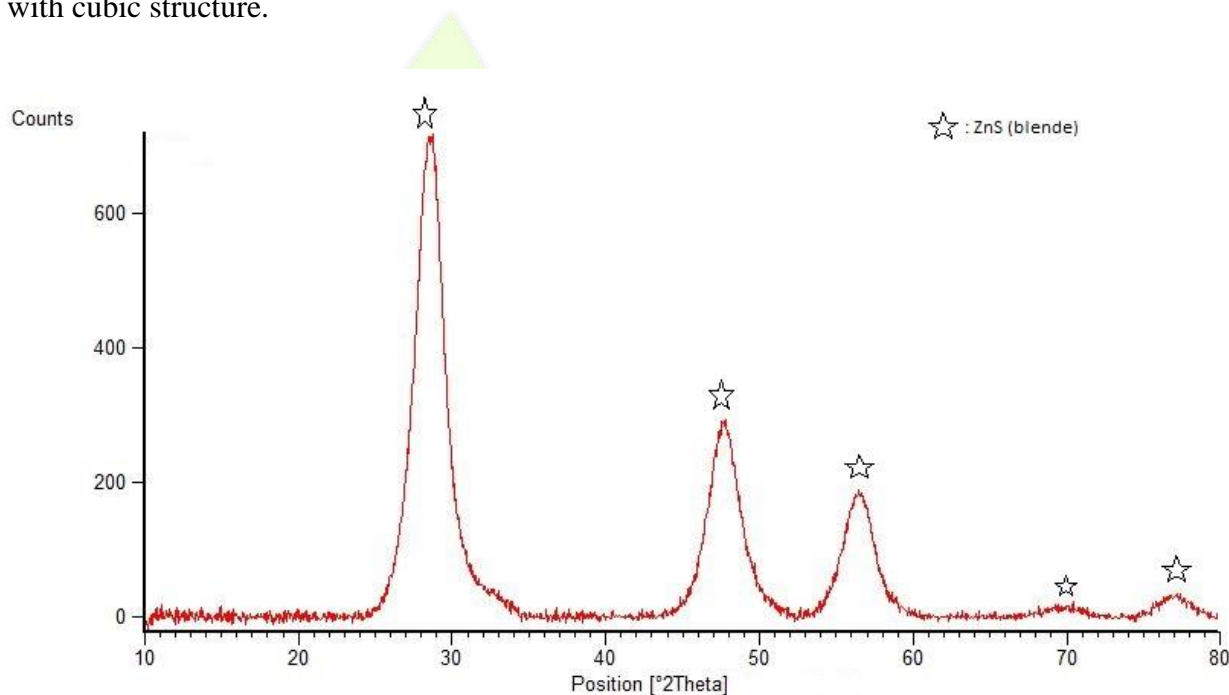
Semiconductor nanoparticles, have different properties in comparison to their bulk particles due to their small dimensional quantum effects. So they have different and special electrical and optical properties. Controlling the synthesized particles size and reducing particle size due to increased energy gap and optimizing the physical and chemical properties is very effective. So one of the most important research areas in the world is producing semiconductor nanoparticles with controlled size. It was also found that the purity of the used materials have high influence on optical properties of sintered parts. As a result, selecting the appropriate method for the processing of optical material with high purity is very important. In this research by choosing the appropriate method, ZnS nanopowder is synthesized and produced with a specific process and then purified to improve the optical properties.

experimental

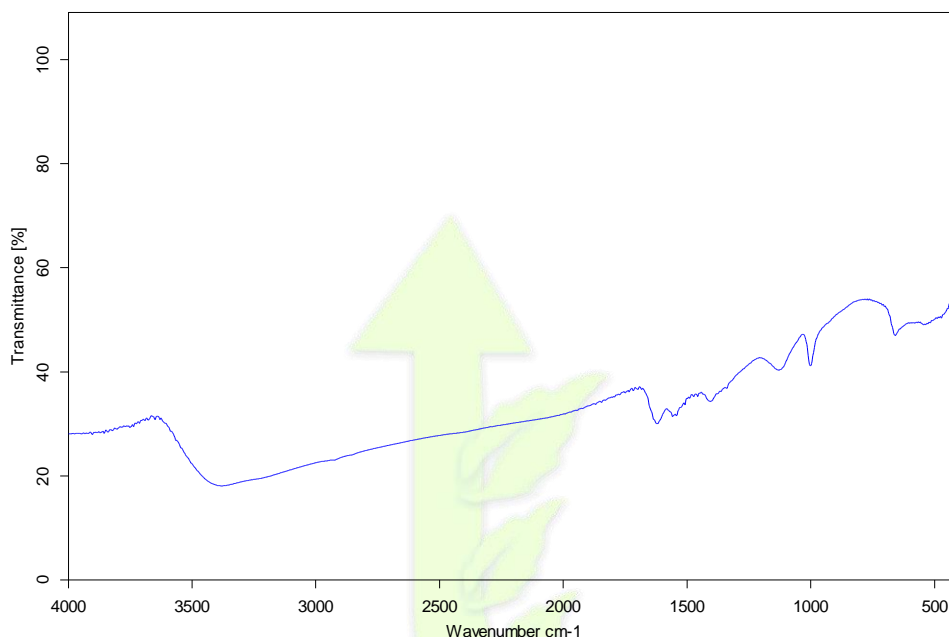
At first, zinc acetate and sodium sulfide were selected as suitable sources for synthesis of zinc sulfide powder. Then by using de-ionized water, the solubilizing of powders were performed. The solutions were then combined into ultrasonic. The Zinc acetate solution was placed into the three-neck round bottom and was heated. in an inert atmosphere consisting of either ultra high purity argon. A sodium sulfide solution was slowly added dropwise to the Zinc acetate solution. The ZnS nanoparticles obtained were separated from the reaction mixture by centrifugation. Washed with ultrapure deionized water and ethanol to remove any trace level impurities. The washed ZnS nanoparticles were then dried in a vacuum oven. The produced nanopowder was placed in a suitable tunnel furnace. And different gases such as air, argon, hydrogen sulfide and hydrogen are blown in order. And then the Purification operation of ZnS nanopowder will be over.

Results and discussion

Structure of synthesis phases and Particles morphology have investigated by X-Ray diffraction (XRD). The XRD result indicate that the ZnS powder was completely synthesized with cubic structure.



The average size of nanoparticles synthesized by the acidic method is between 35 and 45 nanometers. And all impurities, including carbon and sulfur, were extracted from the structure after purification operation. The transition of IR (infrared wave) was measured by FTIR (Fourier transform infrared spectroscopy) more than 50%.



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The process of preparing nano-gel adsorbent from *Descurainia Sophia* nano plant

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Abstract

In this study the production of *Descurainia Sophia* plant in nano dimensions using a disk grinding mill were studied. To prepare the nanogel, the top-down method was used. In preparation of adsorbent nanogel, two milling and crushing steps were necessary, to prepare the adsorbent, first the obtained stems were washed to remove any dust and contaminants. After complete drying of the stems, the stems were reduced to smaller sizes by an electric mill, and the adsorbent grading was carried out using the standard sieve mesh 60.

Key word: Nano gel, Descurainia Sophia nano, Chemical Activation, Crushing,

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Morphology and Electrical Property of TiO₂ Blocking Layer on FTO Prepared by Spin Coating Method

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Abstract

A spin coated TiO₂ thin film was prepared by using TIP/AcOh/PVP ternary system in 2-propanol which can be used as an intermediate buffer layer between the conductive glass substrate and the TiO₂ mesoporous layer in electrochemical solar cells. The effect of the number of deposited layers on the morphology of the thin film was characterized by atomic force microscopy (AFM) after final annealing at 500 °C for 3 h. cyclic voltammetry was used to establish the electrochemical characteristic of the prepared thin films.

Keywords: Titanium dioxide, Thin films, Spin coating, Morphology, AFM, Cyclic volyammetry

Introduction

Spin coating method which generates a solid film was first described by Emslie et al. [1] and Meyerhofer et al. [2] is constitute of several steps including deposition of the coating fluid onto a flat substrate, accelerating the substrate up to its final, desired, rotational speed, spinning of the substrate in order to dominate the fluid thinning behavior over the fluid viscous forces, and evaporation of the solvent.

In this work, a TiO₂ blocking layer was obtained by spin coating a sol-gel solution of TIP/AcOh/PVP (1%) ternary system in 2-propanol on the FTO glass samples. Effect of number of deposited layers on the morphology of the prepared thin film was characterized by atomic force microscopy (AFM) after final annealing at 500 °C for 3 h. Cyclic voltammetry was used for the electrochemical characteristic of the prepared thin films to established their blocking effect.

Experimental

The spin-coating of the TiO_2 thin films was carried out in air with a two step spinning speed of 500 rpm for 8 s and 3000 rpm for 20 s on 1.5×2 cm FTO glasses. The precursor was dried at 150°C for 10 min on a hot plate, following the deposition process. The spinning – drying cycle was repeated 1, 2 and 4 times in order to get films of different thickness (named S1, S2, and S4, accordingly) with final annealing at 500°C . The 0.5 cm of top of FTO samples was cleared of the formed film for providing electrical contact.

Results

Fig. 1 shows the AFM micrographs of the surfaces of bare FTO and S1, S2 and S4 samples and Table 1 presents the root-mean-squared roughness (S_q) of the surface of these samples. It can be seen that by increase of the spinning-drying cycles, surface roughness of the samples increased, which is related to decrease of the size of the nanocrystals of TiO_2 on the surface of the samples [3].

Fig. 2 shows voltammograms of bare FTO, S1, S2, and S4 samples in (2 mM $\text{K}_4[\text{Fe}(\text{CN})_6]$ + 0.25 M KCl) electrolyte in DI water (scanning rate 0.5 V/s).

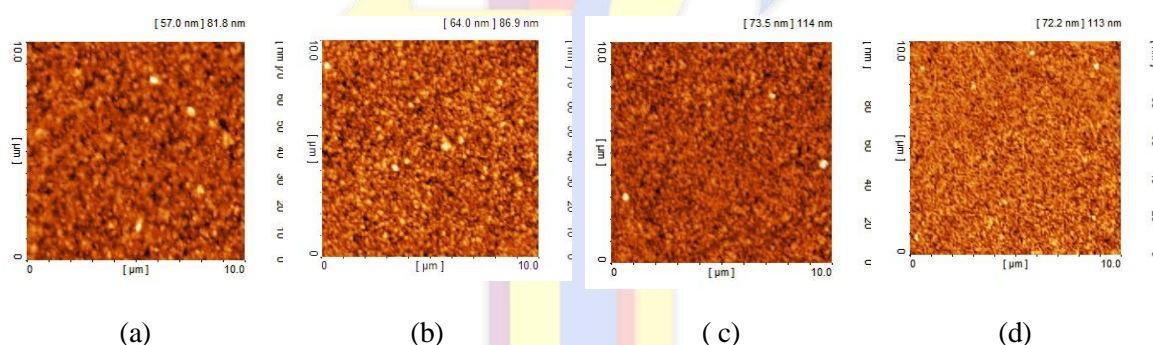


Fig. 1 AFM micrographs of: (a) bare FTO, (b) S1, (c) S2, and (d) S4 samples

Table 1 variation of surface roughness
of the samples

Sample code	Coating layer	S_q (nm) (10 $\mu\text{m} \times 10$ μm)
FTO	-	6.9
S1	1	10.1
S2	2	10.36
S4	4	10.80

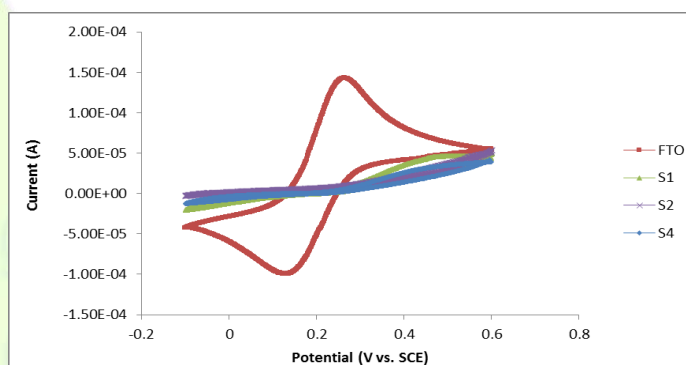


Fig. 2 Cyclic voltammograms of bare FTO, S1, S2, and S4 samples

Conclusion

It is clear that a certain thickness of the TiO_2 film is required to obtain a closed film without cracks or pinhole. Sample S2 shows the minimum anodic current compared to bare FTO and S1 and S4 samples. It shows that S2 sample has the required morphology to be used as a blocking layer for further purpose.

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Characterization and Study of electrodeposited Ni/Cu multilayers on Single and Poly Crystalline Substrates

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Abstract

In this work, Ni/Cu multilayers were deposited by single bath electrodeposition method on single crystal and poly crystal of Cu substrates. Study of CV (Cyclic Voltammetry) of electrolyte consist of Ni^{+2} and Cu^{+2} ions show The effect of temperature and pH on position of cathodic pikes from deposits Ni and Cu. Also, Investigation of X-ray diffraction for $[\text{Ni}(3\text{nm})/\text{Cu}(1\text{nm})]_{n=100}$ multilayers display effects of single crystalline and poly crystalline substrates in number of main pikes and sattlelic pikes.

Keywords: Electrochemistry, single crystal, Poly crystal, X-ray diffraction

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Preparation and characterization of electrospun ZIF-8/PAN nanofiber for thin film microextraction of fluoxetine in aqueous samples

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Abstract

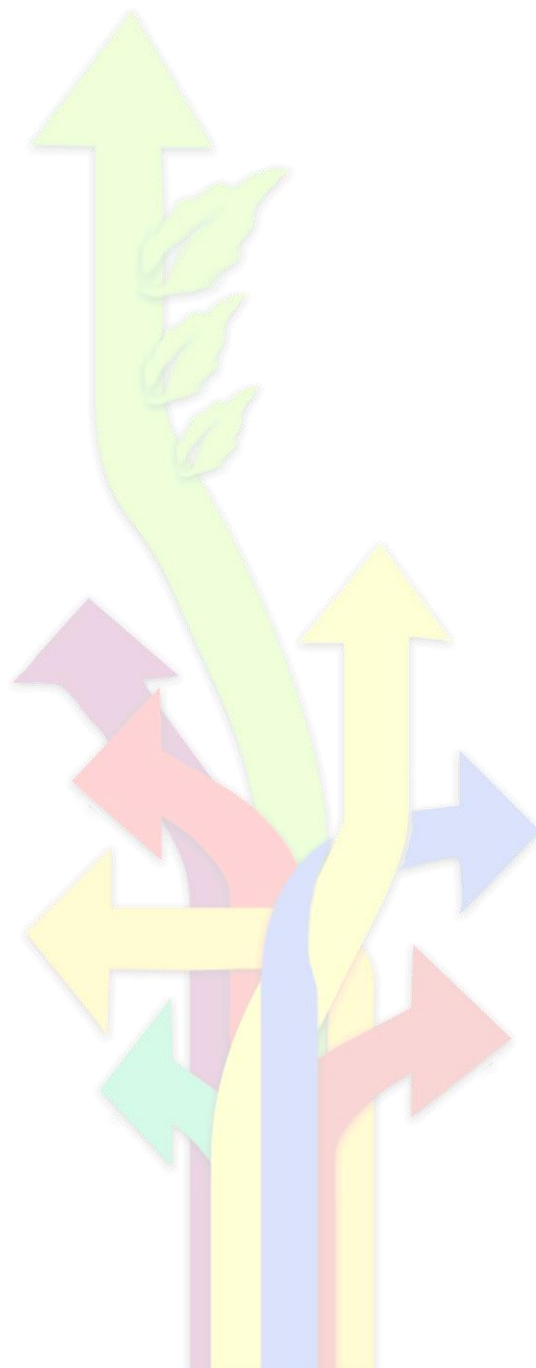
Metal-organic frameworks (MOFs) are a class of hybrid porous crystalline materials comprising of metal centers coordinated to organic linkers [1]. Owing to their well-defined pores and cavities in the scale of molecules combined with abundant surface chemistry, MOFs offer unprecedented opportunities for a wide range of applications including membrane-based separation [2]. In this work, the zeolitic imidazolate framework-8 (ZIF-8)/polyacrylonitrile (PAN) nanofiber was used in thin film microextraction procedure [3]. Firstly, the nanoparticles of ZIF-8 was synthesized [4] and different amounts (0-50%) of it were doped in 10% PAN solution and electrospun. Then the prepared nanofiber characterized using X-ray diffraction (XRD) patterns, field emission scanning electron microscopy (FESEM) images and Fourier transform-infrared spectroscopy (FT-IR). After that, this nanofiber was used as adsorption phase in thin film microextraction for fluoxetine extraction from aqueous sample. It was observed that, increasing the amounts of the doped ZIF-8 in the nanofiber enhanced the extraction efficiently linearly. Therefore the effect of ZIF-8 coatings on the extraction performance of TFME fiber was investigated. The developed nanofiber showed satisfactory extraction efficiency for the analyte and was successfully used in analyzing water samples.

Keywords: Thin film microextraction, Zeolitic imidazolate framework-8, Electrospinning, Fluoxetine

Reference

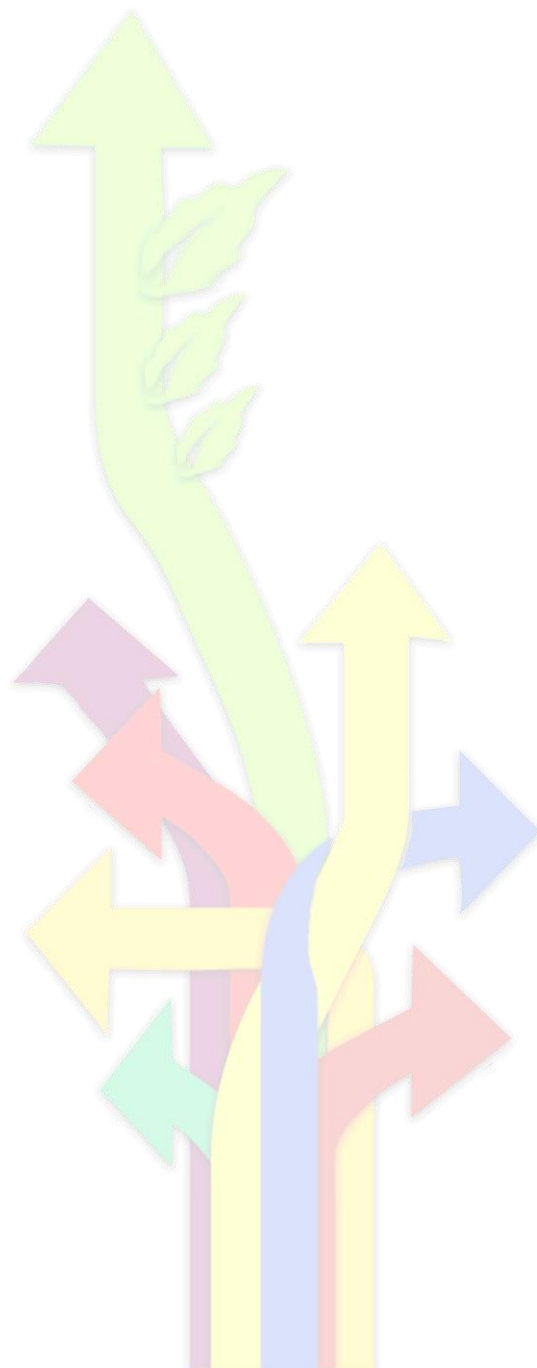
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13th Annual Electrochemistry Seminar of Iran

Corrosion



13th Annual Electrochemistry Seminar of Iran

Study of the passive film behavior formed on 316L stainless steel at different temperature using the Mott-Schottky test

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Abstract

In this research, the behavior of passive layer formed on 316L stainless steel was studied in a 3% sodium chloride solution at different temperature using the Mott-Schottky test. The results showed that an increase in temperature caused an increase in the density of electron carriers and the double layer capacitance and a decrease in the polarization resistance of the steel. The density of electron carriers on the passive layer was calculated at various temperature and the passive layer has the properties of a semiconductor type n and p

Keywords: 316L Stainless Steel, Passive Film, Corrosion, Mott-Schottky.

1. Introduction

Stainless steels is an alloy that has been developed for corrosion resistance. The presence of chromium element in stainless steel causes it to have an anti-corrosive effect and it improves the corrosion resistance of stainless steels. This improvement is due to the formation of a passive layer of chromium oxide on the surface of stainless steel. The passive layer shows the behavior of a semiconductor [1-3]. The Mott-Schottky test can provide useful information on the semiconductor behavior of passive layer formed on steel [4].

2. Experimental

All the chemicals were analytical grade of Merck origin. The electrochemical measurements were done by a computer-controlled potentiostat/galvanostat (AutoLab, PGSTAT302N) contained a three-electrode system of Ag/AgCl-saturated KCl, a platinum wire and a commercial 316L stainless steel as the reference, counter and working electrodes, respectively. The Mott-Schottky tests were done at open-circuit potential and frequency of 1000 Hz.

3. Results and discussion

Fig. 1 shows the Mott-Schottky plots (the inverse square of the capacitance of the space charge region in terms of applied potential) of passive layer formed on steel in 3% sodium chloride solution at different temperature. The maximum value of capacitance is obtained at 80 °C .

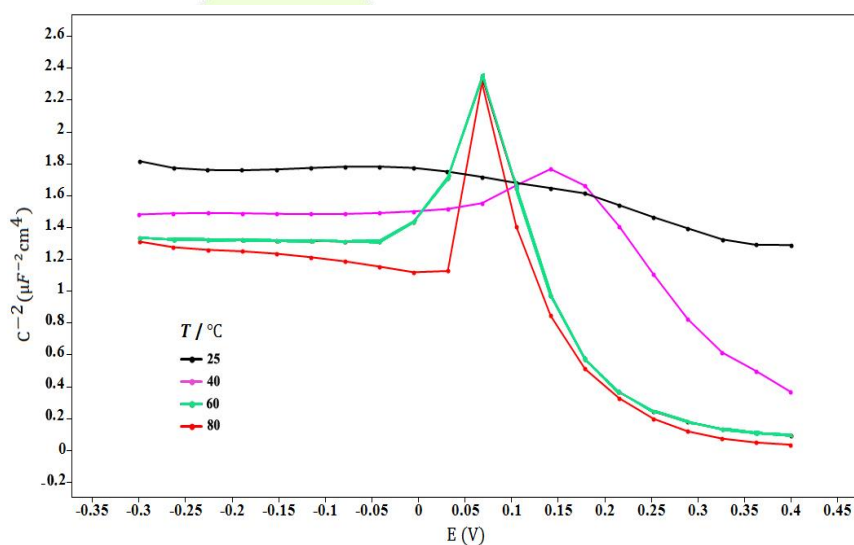


Fig. 1. Mott-Schottky plots of stainless steel in 3% Sodium Chloride Solution at different temperature.

For semiconductors of type p and n, the curve of C^{-2} against E varies linearly with a negative and positive slope, respectively.

4. Conclusion

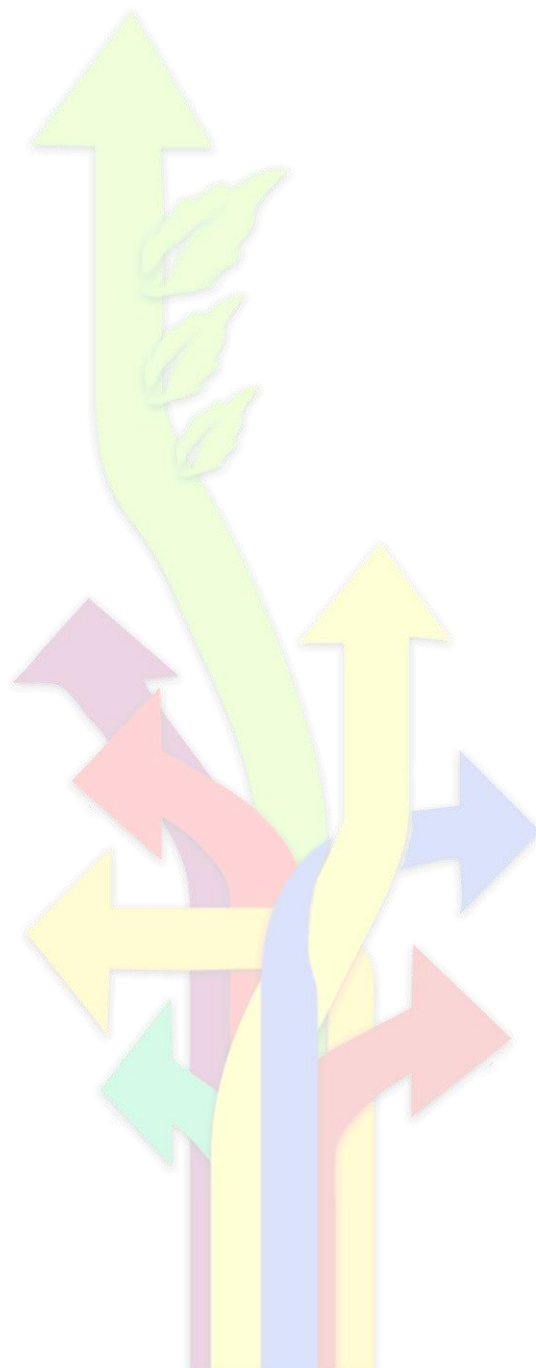
The density of electron carriers increases with increasing temperature and was calculated for various temperatures in the range of $10^{37} - 10^{39} \text{ cm}^{-3}$. Both type of semiconductor (n and p) were detected in the passive layer.

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13th Annual Electrochemistry Seminar of Iran

Behavior of corrosion inhibitor as demulsifier for W/O emulsion

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Abstract

the corrosion inhibitor has been used in this work as a demulsifier. Because of this material have surface active property, therefore they can adsorb rigid film surrounding water drops and destroy it. Functional groups of corrosion inhibitor detected by FT-IR as well as bottle test has been applied for the study of dehydration from crude oil. The results indicate the existence of O-H, C-O, and C-H groups in the structure of corrosion inhibitor. Bottle test showed that a 160 ppm of demulsifier can separate water from crude oil, completely. Studies on temperature effect indicate this factor as a cause of increase in the efficiency of dehydration. The rate of demulsification increased owing to both the decreased viscosity of crude oil when temperature increased and the amplification of motion of water drops that lead to coagulate drops.

Keywords: Crude oil, Demulsifier, corrosion inhibitor, W/O emulsion

Corrosion Resistance of Porphyrin-Nickel electrode in 8 M NaOH solution

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Abstract

Nickel is one of the most important metals that was used in different industries such as electroplating, electroforming [1] and production of stainless steel [2]. In alkaline media, hydrogen was diffused in Nickel electrode and its corrosion was occurred. In this work the corrosion behavior of Porphyrin-Nickel electrode in 8 M NaOH solution was investigated using cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS) and polarization techniques. The porphyrin molecule, as a Lewis acid with a network of conjugated π -electron systems and four nitrogen atoms at its core, possesses the molecular frame of a potential corrosion coating. It is a tetradentate chelating agent with strong bonding capability and evident ability to interact with surfaces by several physical and chemical mechanisms [3]. The Tafel plots reports that porphyrin coating decrease the corrosion rate of Ni electrode. The result show that Porphyrin coating also exhibited higher charge transfer and polarization resistance compared to plain Nickel electrode.

Keywords: Corrosion, Nickel, Porphyrin, Alkaline solution.

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Simulation and investigation of anode role in iron corrosion with concrete coating

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Abstract

The equipment used in the chemical processes and the foundation is made of various metals that expose them to corrosion. Corrosion is an electrochemical process in which iron is corroded and converted into iron ions in a limited volume of pores in the pores of concrete around steel. The process of starting and releasing corrosion is discussed in this environment. The role of multi-metal as anode and its effect on reducing corrosion by cathodic protection method is discussed. In this study, the simulation of corrosion of iron rebar inside the concrete and the saturated pores of intrusive oxygen, has been carried out by Comsol software. The results show that in the cadmium alloy a sudden drop occurs at a concentration of $PS = 0.7$ while for zinc and chromium alloys in the range of $PS = 0.65$. According to the results, zinc and chromium alloys are more suitable for corrosion prevention.

Keywords: Corrosion, Simulation, Comsol, Iron, Anode, Cathode

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Study Thermodynamic and Kinetic Diphenylamine – 4 – Sulfonic Acid Sodium Salt as a new inhibitor on corrosion stainless steel 304

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Abstract

This study uses electrochemical techniques such as linear sweep voltammetry (LSV) and the use of Tafel chart was investigated at different temperatures.

It was found that this inhibitor at 15°C had the best inhibitory effect on stainless steel 304, at this temperature the percentage of deterrence is 98%.

Using the adsorption isotherm of Langmuir, the absorption heat and entropy changes were calculated in the process of absorbing this inhibitor on the surface. As the temperature increases, the strength of the inhibitory bond decreases with the surface. And the inhibitors of absorption on the surface will be reduced. And Tafel showers, the corrosion rate increases at high temperatures. Also, at high temperatures entropy, the absorption inhibitory process increases on the surface.

Keywords: *Corrosion, tafel curve, linear sweep voltammetry, isotherm Langmuir*

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Ionic liquids as green inhibitors for protection of mild steel in acidic solution: electrochemical and DFT study

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Abstract

A literature review of abundant publications reveals the crucial role of inhibitors as one of the most practical methods for corrosion protection of metal bodies in industrial processes such as acid pickling, acid cleaning, chemical and electrochemical etching. The use of an additive is one of the major solutions for this problem. Hence, various additives are used to protect iron and its alloy against corrosive attack. The use of organic molecules containing functional groups and p electrons in their structure, as corrosion inhibitors, is one of the most practical methods for protecting metals against corrosion and it is becoming increasingly popular. Ionic liquid compounds possessing unique properties have attracted the attention of corrosion science and engineering researchers [1-3]. In recent years, several publications have provided evidence for a revolution in ionic liquid compound chemistry, leading to a probable switchover from volatile organic solvents in the future.

In the present work, different imidazolium type ionic liquid were synthesized and their inhibiting action on the corrosion of stainless steel (SS) in HCl was investigated by means of potentiodynamic polarization and electrochemical impedance spectroscopy (EIS). The results of the investigation show that the newly synthesized compounds show excellent inhibition efficiencies against the corrosion of SS in acidic solution. The adsorption of ionic liquid ions onto the SS surface followed the Langmuir adsorption model. Electronic properties such as highest occupied molecular orbital (HOMO) energy, lowest unoccupied molecular orbital (LUMO) energy and frontier molecular orbital coefficients for inhibitors have been calculated.

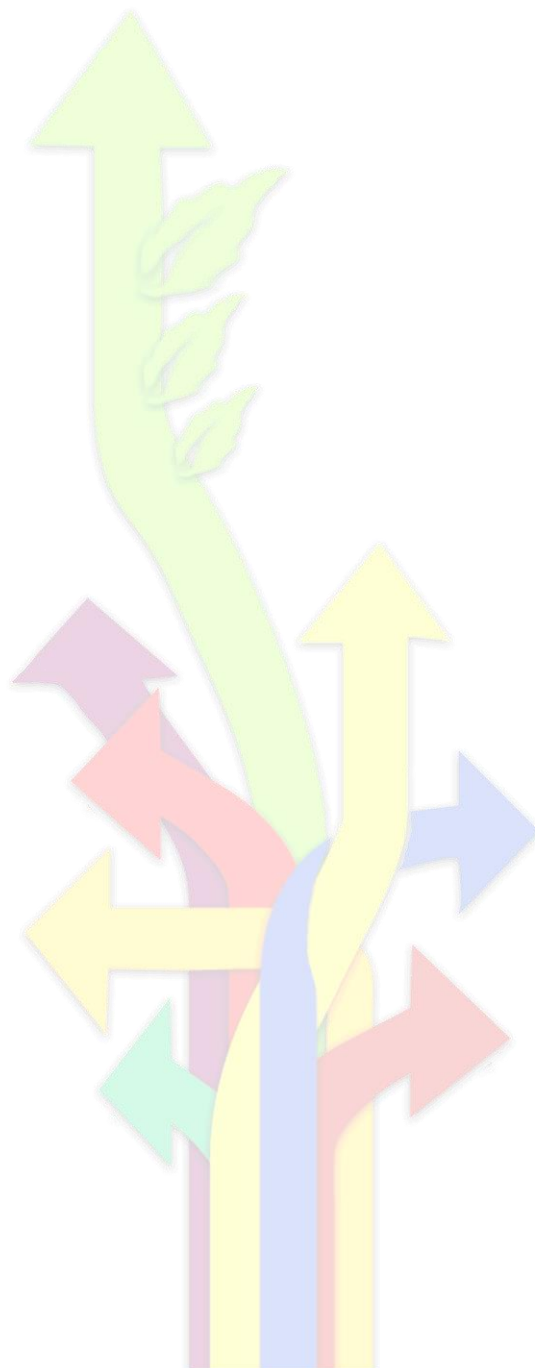
Keywords: Corrosion protection, mild steel, ionic liquid inhibitor, EIS, quantum chemical

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13th Annual Electrochemistry Seminar of Iran

Exploring the Effect of Deicing Salts Used in Highway Maintenance on the Electrochemical Corrosion Behavior of Mild Steel in 3.5 wt.% NaCl Solution

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Abstract

Deicing chemicals are widely used for maintenance of highways in winter. In this regard, sodium chloride (NaCl) is the main deicer which is applied in large-scale due to its high efficiency at commonly experienced temperatures and low cost. Other deicing agents such as CaCl_2 , MgCl_2 , and calcium magnesium acetate (CMA) are other chemicals utilized for improving the deicing performance of NaCl. However, these materials have side effects on the roadway and environment. Indeed, the deicers have detrimental effects on concrete infrastructure via interacting with concrete paste and reducing the integrity and strength of concrete; encouraging the diffusion of moisture, oxygen and other aggressive agents onto rebar surface and promoting the rebar corrosion [1-3].

Therefore, in this research, the effect of deicing salts including CaCl_2 , MgCl_2 , CMA, and urea on corrosion behavior of mild steel substrates in corrosive electrolyte is considered. The prepared electrolyte consists of 3.5 wt.% NaCl solution containing 0, 0.05, 0.1, 0.3, and 0.8 wt.% deicing salts. The corrosion performance of samples is studied by potentiodynamic polarization tests.

The results indicate that the type and the concentration of deicing salt are two important parameters affecting the corrosion behavior of mild steel. The presence of other deicing salts such as CaCl_2 , MgCl_2 , and urea increases the corrosion rate of mild steel in NaCl solution. This behavior can be attributed to decrease of pH and providing chloride ions which can reduce the passive protection. However, the corrosion rate of samples in electrolyte containing 0.05 wt.% CMA increases and then, the corrosion rate decreases by adding extra amount of CMA. The corrosion rate of mild steel in 3.5 wt.% NaCl solution containing 0, 0.05, 0.1, 0.3, 0.8, 1.5, 3, and 5 wt.% CMA is 0.1152, 0.1564, 0.1611, 0.1290, 0.0886,

0.0401, 0.0670, and 0.0857 mm/year, respectively; i.e. the NaCl solution loaded with 1.5 wt.% CMA shows the lowest corrosion rate. Indeed, both calcium hydroxide and magnesium hydroxide precipitate via adding low amounts of CMA, leading to decrease of pH and reducing the passive protection. By adding extra amount of CMA, fully passive condition is attained and the corrosion rate decreases.

It is believed that the results of this research will show corrosion aspects of deicing agents used in highways.

Keywords: Deicing salts, Mild steel, NaCl solution, potentiodynamic polarization tests

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Investigating the Accelerated Electrochemical Corrosion Protection Performance of Coal-Tar and Bitumen Enamel Coatings for Pipelines

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Abstract

Corrosion of pipelines is an extreme challenge for oil and gas industry. The harsh environment of pipelines application has great effect on selecting the appropriate coatings for them. Different types of coatings are used for corrosion protection of pipelines in industry and meanwhile, coal-tar and bitumen enamel coatings are widely applied for enhancing their service-life [1-3].

Therefore, in this research, the effects of industrially used coal-tar and bitumen enamel coatings on mild steel substrates are considered. The degradation process of coatings is examined in the synthetic groundwater by electrochemical methods using Potentiostat/Galvanostat (PGSTAT 30). The electrochemical cell consists of the sample, Pt wire, and saturated calomel electrode (SCE) as working, counter, and reference electrode, respectively. Accordingly, the polarization tests are repeated 7 times for accelerating the degradation of coatings. For potentiodynamic polarization tests, open circuit potential (OCP) of system at equilibrium state after immersion is recorded as corrosion potential (E_{corr}). Then, the polarization curve was plotted by sweeping the applied potential from -0.4 to +0.4 V with respect to OCP at scan rate of 1 mV/sec on 1 cm² anode. Corrosion current density (i_{corr}) was determined from Tafel plot by extrapolating the linear portion of the curve to E_{corr} . Tafel constants including anodic (β_a) and cathodic (β_c) slopes were calculated for anodic and cathodic parts of Tafel plot, respectively. Then, the corrosion protection performance of coatings is determined by electrochemical impedance spectroscopy (EIS). The EIS tests were carried out in frequency range of 10⁻² to 10⁵ Hz with AC amplitude of 10 mV at OCP. All electrochemical tests are repeated three times.

The variation of OCP during immersion in synthetic groundwater indicates that the coated substrates have more positive OCP values compared to bare one. Meanwhile, the OCP value of the coal-tar enamel coated sample is more noble than bitumen enamel coated sample. The results derived from potentiodynamic polarization tests show that coal-tar coated sample has more positive E_{corr} , lower i_{corr} , lower porosity, higher polarization resistance (R_p) and higher corrosion protection efficiency (PE); revealing enhanced corrosion protection capability of coal-tar enamel coating compared to bitumen enamel coated sample. Indeed, the corrosion rate for bare substrate, bitumen enamel coating, and coal-tar enamel coating is 0.07502,

0.00904, and 0.00159 mm/year, respectively. Moreover, the EIS results including Bode, phase and Nyquist plots present that the coal-tar enamel coating is more appropriate for achieving enhanced corrosion protection for pipelines due to their higher corrosion resistance. The results of EIS are fitted by electrical equivalent circuit by using NOVA software which show that the coating resistance and charge transfer resistance for coal-tar enamel coating is significantly more than bitumen enamel coating. The enhanced corrosion protection capability of this sample can be attributed to the pore structure and chemical composition of coal-tar enamel coating.

Figure 1 shows the overview of the obtained electrochemical data.

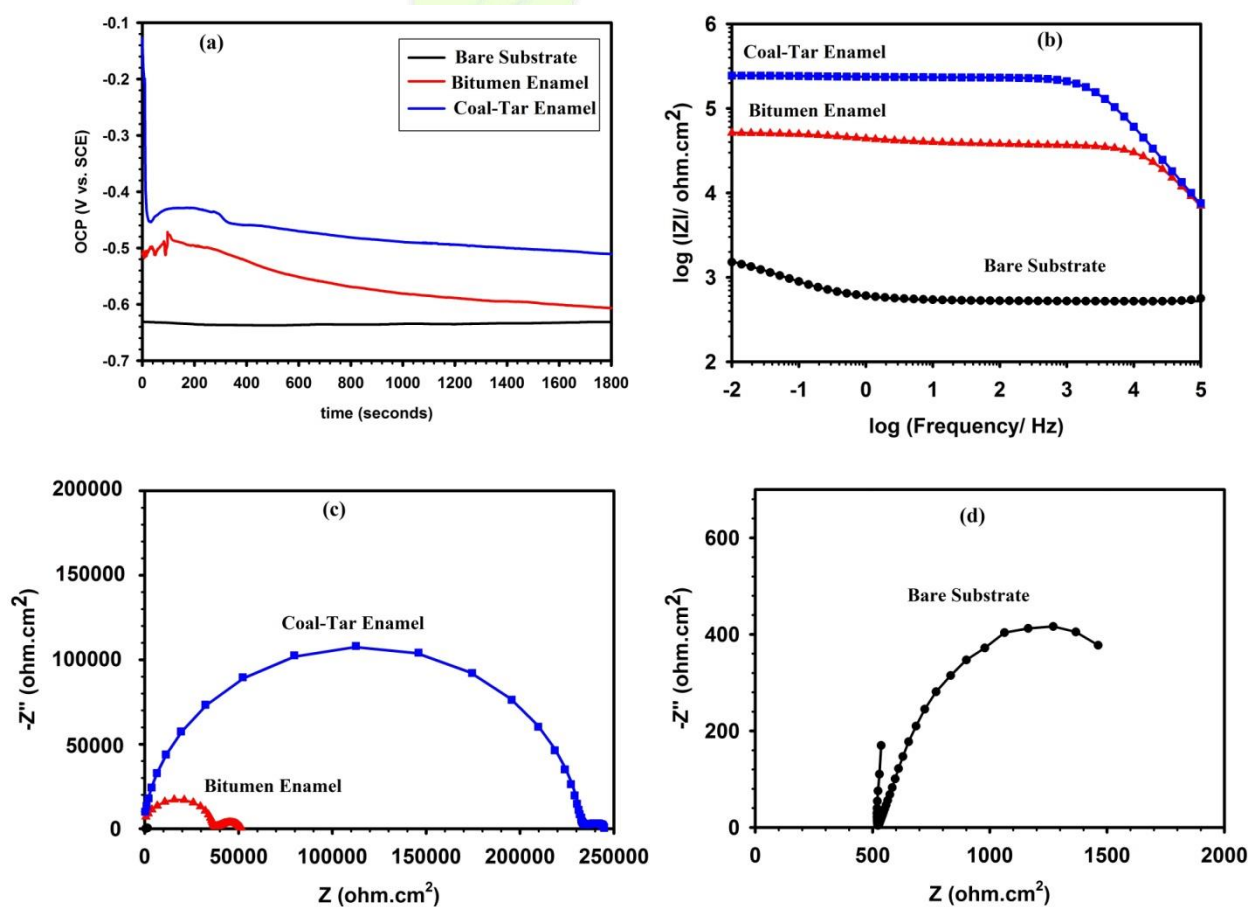


Figure 1. (a) The variation of OCP; (b) The Bode plots; (c, d) The Nyquist plots for bare substrate, than bitumen enamel coating, and coal-tar enamel coating.

Keywords: Coal-Tar Enamel, Bitumen Enamel, Organic Coatings, Electrochemical Corrosion Tests

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Effect of nano alumina addition on the corrosion behavior of NiTi

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Abstract

In this study, NiTi and NiTi-6 wt.% nano alumina composites were prepared with hot isostatic pressing method. The electrochemical corrosion behavior of NiTi compared with NiTi-6 wt.% nano Al₂O₃ in simulated body fluid (SBF) corrosion media were characterized using potentiodynamic polarization method. Results showed that in SBF electrolyte, the composite samples showed improved corrosion when compared with unreinforced NiTi samples, which is attributed to the finer grain size of composite samples which facilitate the formation of protective passive layer.

Keywords: NiTi, nano Al₂O₃, corrosion, microstructure.

Introduction

NiTi alloy has been one of the most attractive metallic implant materials during the last 20 years, due to its shape memory effect, superelasticity, good biocompatibility and high corrosion resistance [1].

The NiTi alloy is still a controversial biomaterial because of its high Ni content which can trigger the risk of allergy and adverse reactions when its ion releases into the human body [2,3]. The Ni-ion release rate to the body is directly related to its corrosion behavior. Therefore, in order to enhance the safety of NiTi replacement in the body, this alloy's corrosion resistance should be improved. It has been shown that the increasing of grain boundaries in NiTi alloy facilitates the formation of protective passive layers which improves the alloy corrosion resistance. Also, it is well-known that the phase constituents of NiTi directly affect its properties.

In the present study, to control the grain growth of the alloy, 6 wt% alumina nanoparticles were added to the NiTi matrix and the corrosion behavior of monolithic NiTi and NiTi-6 wt.% nano Al₂O₃ samples were studied in simulated body fluid (SBF) corrosion media.

Experimental

High-purity pre-alloyed NiTi powders with a composition of Ti-56wt%Ni and average particle size of 15 μm were used as raw materials. Stress-free transformation temperatures of the NiTi powders, which were measured using a differential scanning calorimetry (DSC) technique, are as follows: martensite start (M_s) temperature: 3°C, martensite finish (M_f): -22°C, austenite start (A_s): 6°C, and austenite finish (A_f): 28°C. Six wt.% $\alpha\text{-Al}_2\text{O}_3$ nanoparticles with an average particle size of 80 nm were mixed with the NiTi powder with a planetary ball mill. A HIP method was used for bulk sample production. Consolidation procedure of NiTi was performed under a condition according to reference [6]. An EG&G Princeton Applied Research Model 2273A instrument was used to determine the electrochemical properties of the samples. A large area platinum flat electrode was used as the counter electrode. An Ag/AgCl reference electrode was placed close to the working electrode surface. All electrochemical experiment was carried out in SPF solution.

Result and discussion

In order to evaluate corrosion behavior of materials, potentiodynamic polarization curve can be used. The results in table 1 indicate that the corrosion potential of NiTi-6 Al_2O_3 sample is higher and the corrosion current of this sample is lower than that in the monolithic NiTi which implies the composite samples better corrosion resistance in SBF media. To figure out the origin of this behavior, the microstructural aspects and phase constituents of these samples were studied and correlated to the corrosion resistance.

Table 1. Corrosion parameters of NiTi and NiTi-6 wt.% nano alumina samples in SBF solution.

sample	NiTi	NiTi-6 wt.% nano alumina
E_{corr}	-0.49	-0.26
$I_{\text{corr}}(\mu\text{Acm}^{-2})$	7.2	9.3×10^{-2}

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Electrochemical Comparing the Effect of Frula-Asafotedia and it's Nanocomposite with Chitosan on Cu Corrosion.

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Abstract

Corrosion of metals and alloys is an important industrial problem specialty in our country, Iran. Different organic and inorganic compounds containing N, O and S atoms are invest used as effective corrosion inhibitors [1]. Unfortunately, most of the organic inhibitors are toxic, very expensive and environmentally unfriendly. Therefore, recently some research is oriented to the development of green corrosion inhibitors with good efficiency and low risk of environmental pollution. Plant's extracts are containing mixtures of compounds such as N and S as constituent atom were studied as corrosion inhibitor and are biodegradable in nature [2]. In other hand, in recent years, there has been considerable interest in developing biodegradable nanoparticles and nanocomposites for increase inhibition efficiency.

In present work, we synthesis nanocomposite of chitosan with essential oil of Ferula Asafotedia (F.Af) (anghouzeh in Persian). Then have studied the corrosion resistance of Cu plate in 3.5% salt solution in presence of Asafotedia (Af) alone and nanocompsite it with chitosan. F.Af is one of the most important species of Iranian Ferula which grows wildly in several areas of Iran. Af is the oleo-gum resin which is exudates from F.Af. The gums were also known as green corrosion inhibitors [3]. Chitosan (Ch) is a natural polymer easily extracted from the shells of crustaceans. The anticorrosive behavior of chitosan is attributed to the presence of hydroxyl and amino groups, but it exhibits low inhibition efficiency if it is used alone [4]. Chitosan is receiving a lot of interest in the encapsulation of bioactive compounds due to its biocompatibility, low toxicity and biodegradability. For synthesis of nanocomposite, after extract oil of Af using Clevenger, Chitosan/essential oil of Af (COA) nanocomposite, were prepared according to a procedure modified from the ones reported by Esmaeili et al [5], and was characterized with different techniques. Electrochemical corrosion behavior of sealed epoxy resin Cu specimen with 1 cm² area were evaluated in 3.5

wt.% NaCl solution in absence and presence 1 g/l of As or COA nanocomposite with three electrode cell using a potentiostat/galvanostat model IVIUM. The corrosion resistance of solutions were analyzed by Tafel plot (Fig 1a) and electrochemical impedance spectroscopy (EIS) (Niquist plot, Fig 1b) measurements.

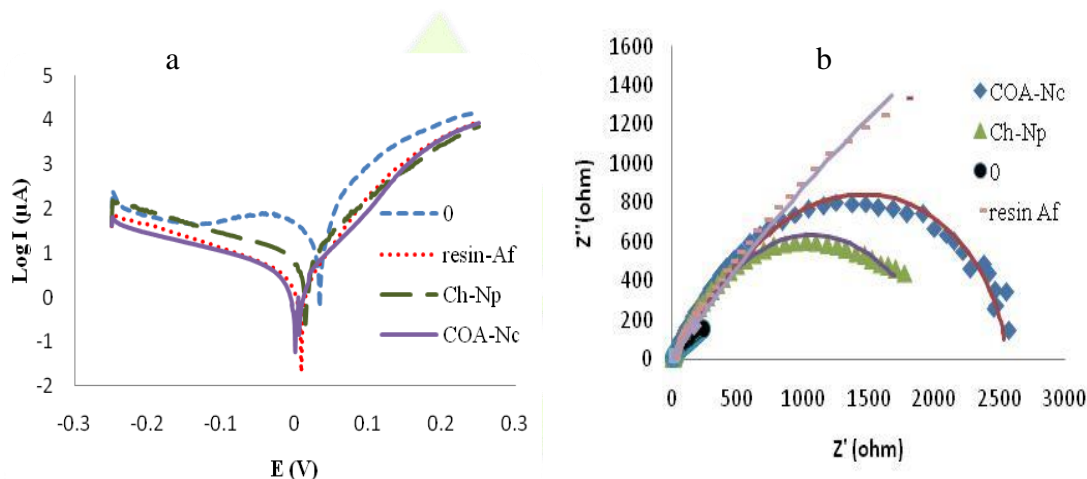


Fig 1. Tafel plot (a) and Niquist plot (b) of Cu in 3.5% salt alone (0) and in presence of 1 g/l resin Asafotedia (Af), Chitosan nanoparticles (Ch-Np) and COA nanocomposite (COA-Nc).

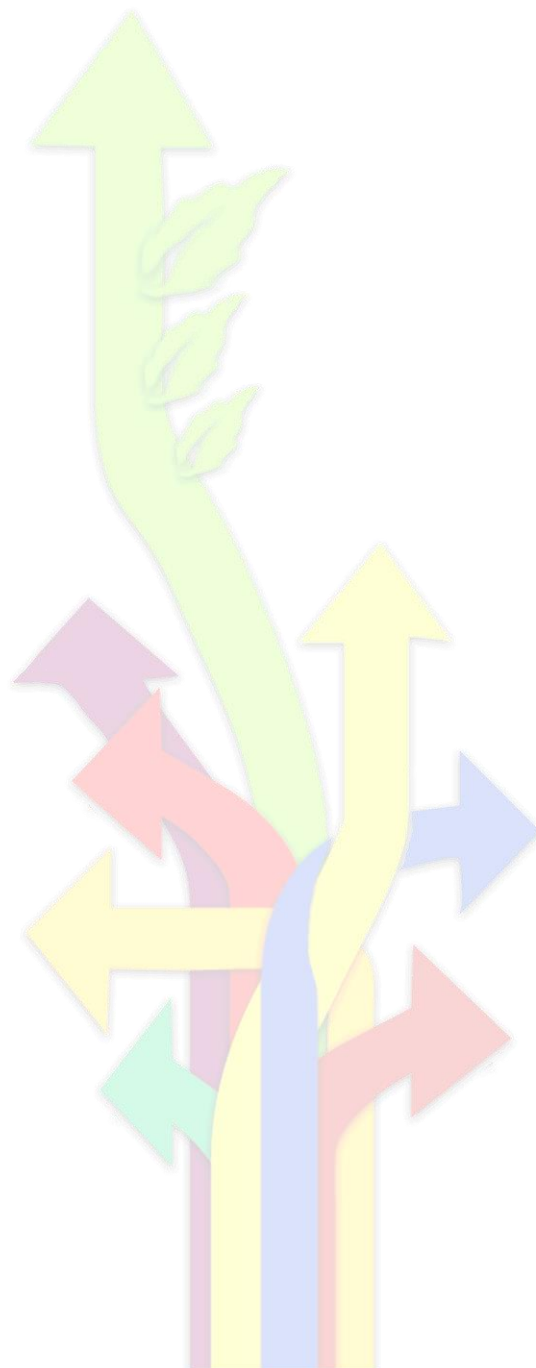
As observed in Fig. 1a shift of corrosion potential and decrease in current (I_{corr}) indicates the higher corrosion resistance property of the Cu specimen in presence of Af and COA composite. Also Fig 1b show that Cu in solution of Af and COA composite has larger charge transfer resistance (R_{ct}) (diameter in the semicircle in middle frequency range) than 3.5% salt solution alone.

Keywords: Frula-Asafotedia, Chitosan, Nanocomposite, Electrochemical Corrosion,

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Electrochemical Synthesis



13th Annual Electrochemistry Seminar of Iran

Effect of Annealing in the Molybdenum Diselenide Formation of using Electrochemical Deposition Method

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Abstract

Mo-Se thin films have been electrodeposited on conducting tin oxide (SnO₂) coated glass substrates from basic and acidic solution containing Na₂MoO₄ and H₂SeO₃. The Mo-Se films obtained in different electrolytes were analyzed for their composition. It was found that the chemical composition of the Mo-Se films depends on the Na₂MoO₄/H₂SeO₃ ratio in the electrolyte. Also the time of electrodeposition optimized. In this work the influence of pH value and the concentration of precursor and influence of annealing temperature on the electrochemical property of the MoSe₂ were investigated. Some unreacted MoO₂ was detected and also some excess elemental Se was also removed, but were removed by thermal annealing. X-ray diffraction analysis showed the presence of a highly textured MoSe₂ film and polycrystalline nature. SEM studies shows that the unheated films are uniform and pin-hole free. As the temperature rises, the pin-hole increase on the surface.

Keywords: Electrodeposition, Molybdenum diselenide, Thin film

Reference

- (1) Zhai, Y. J., et al. Journal of Alloys and Compounds 672 (2016): 600-608.
- (2) ANAND, T. JOSEPH SAHAYA, et al. (1999): 125-130.
- (3) Tsang, Chu F., Maria A. Ledina, and John L. Stickney Journal of Electroanalytical Chemistry (2017).

Effect of Annealing on Electrodeposited Molybdenum Diselenide Properties

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Abstract

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UV-assisted pulsed-current photo-electrochemical etching of silicon carbide thin films on silicon substrates

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Abstract

In order to fabricate porous silicon carbide (PSC) thin films, photo-electrochemical etching of silicon carbide (SiC) thin films was carried out using a pulsed-current generator with specific pulse properties. Prior etching, thin films of SiC were coated on polished surface of silicon (Si) using radio frequency (RF) magnetron sputtering. For best quality of SiC thin films, the surface of Si substrate was chemically cleaned using RCA clean method.

Silicon carbide was immediately deposited on freshly prepared Si substrates using an RF magnetron sputtering unit (Edwards A500, UK). The RF power was set to 200 W and the substrate temperature was fixed at 400 °C. Substrate heating can act like an in-situ annealing process that promotes the allocation of the sputtered SiC atoms. After 2 h of sputtering, the obtained thickness of the deposited SiC was 1 μm (deposition rate = 500 nm/h).

The samples were then placed in a quartz carrier and annealed in a tube furnace. Thermal annealing was performed for 1 h at 1200 °C under a nitrogen flow. The samples were then allowed to cool to room temperature inside the furnace.

For formation of PSC, etching is an essential technique. Recently, dry etching methods based on electron cyclotron and inductively coupled plasma are mostly applied. However, these methods needs expensive and sophisticated techniques and suffer from the risk of creating damage in the surface [1-4]. In many cases, wet-chemical etching is an attractive alternative [5]. There are two main methods for wet chemical etching of SiC substrates: metal-assisted electroless chemical etching and photo-assisted electrochemical etching. The former has been suffering from the lack of controllability of the pore size and distribution. But the latter technique which was pioneered by Shor et al. [6, 7] can be controlled by several parameters like current density and shape of current. Many studies on SiC wafers were reported, but less effort has been focused on SiC thin films on Si substrates [8-10]. For electrochemical etching of p-type SiC, by applying an anodic potential to the substrate, holes accumulate at the surface and cause oxidation and dissolution. This process takes place in dark [10].

The UV assisted-electrochemical etching method can be assumed as an attractive technique for fabricating porous n-type SiC and producing optical waveguides because of the ease with which layers can be fabricated over a large area of the substrate, and the uniformity of porous layers which are created by this method. At the SiC/HF electrolyte interface, the chemical treatment of SiC leads to the formation of SiF_6^{2+} complex which is soluble in the solution [5]. The role of ultraviolet radiation for enhancing the etch rate is partially due to the relatively shallow absorption depth of UV light. This phenomenon allows more carriers to be photo-generated in the space charge layer.

In the current research, a two-electrode setup was used for the electrochemical etching of SiC thin films. The substrate (SiC) functioned as the anode electrode, whereas an inert metal wire (Pt) was used as the cathode. A pulsed current with a period time (T) of 14 ms and a pause time (T_{off}) of 4 ms was supplied by an integrated SourceMeter instrument (Keithley 2400). Current densities of 10, 15, and 20 mA/cm^2 were used to prepare the porous samples A, B, and C, respectively. Then, all samples were washed with deionized water and air-dried in the laboratory at room temperature.

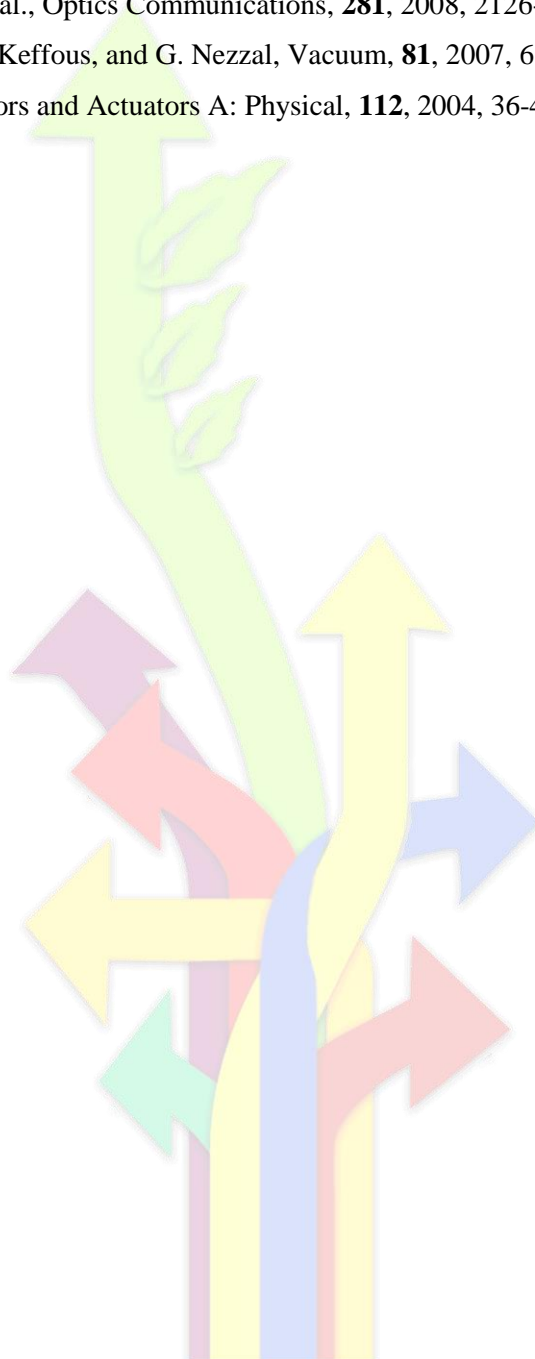
Here, the improvement in optical characteristics of PSC/Si by optimization of the current density in UV-assisted electrochemical etching of SiC thin films on Si substrates is reported. It has been illustrated that current density can be considered as an important parameter for controlling the etching rate and morphology of the porous samples. Thus, it can enhance the optical properties of electrochemically etched PSC layers. Scanning electron microscopy (SEM) images demonstrate that by this technique the porosity and uniformity of PSC thin films can be controlled. Hence, the photoluminescence properties of porous samples can be optimized. Therefore, PL of PSC has been related to the morphology of the surface which is controllable by etching parameters. The PL Peak intensities of PSC samples are shown to be enhanced and the optical properties are improved with increase in etching current density as compared to non-porous SiC thin film.

Keywords: Electrochemical Etching, Porous Silicon Carbide, RF Sputtering, Optical Properties

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13th Annual Electrochemistry Seminar of Iran

The effect of etching time on physical properties of porous silicon fabricated via photo-electrochemical etching technique

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Abstract

porous silicon (PSi) has received a great deal of attention due to the high specific surface area and easy fabrication using some established processes of the usual silicon technology. Porous silicon has been demonstrated being a sensitive sensing platform because of its large internal surface area. The optical and electrical properties of PSi such as refractive index, photoluminescence and impedance are very sensitive to the presence of biological or chemical species inside the pores. Recently, PSi sensor for detection of different targets such as gases, DNA, and proteins have been reported. An essential requirement for PSi sensor is that the pore's diameter should be large enough to allow easy infiltration of the biological targets but small enough to preserve the large internal surface area to volume ratio that increases the sensitivity. In the present work, we report the behavior of anodized silicon under various etching time parameter and achieving optimum etching time for these processes. All experiments were conducted on square samples (10 mm×10 mm) cut from a single crystalline n-type [100] silicon wafer with a surface resistivity of 0.1-10 Ω/\square . For cleaning procedure, the silicon substrates were dipped in $\text{H}_2\text{SO}_4\text{:H}_2\text{O}_2$ 3:1 %wt for 10 minutes and then HF: H_2O in 1:9 for 4 minutes, Afterwards, the samples were washed with deionized water and dried under an ambient nitrogen flow.

The PS layers were created by etching crystalline silicon (c-Si) in a HF-based photo-electrochemical bath at room temperature under the illumination of a 100W tungsten lamp placed 20 cm above the samples. The electrochemical cell was a Teflon container 10 mm in diameter and 25 mm in height. The solution contained a mixture of hydrofluoric acid (49%), ethanol (95%) and with the volume ratio of 1:4. The silicon substrate was used as an anode electrode, and the cathode was an inert metal (Pt) wire.

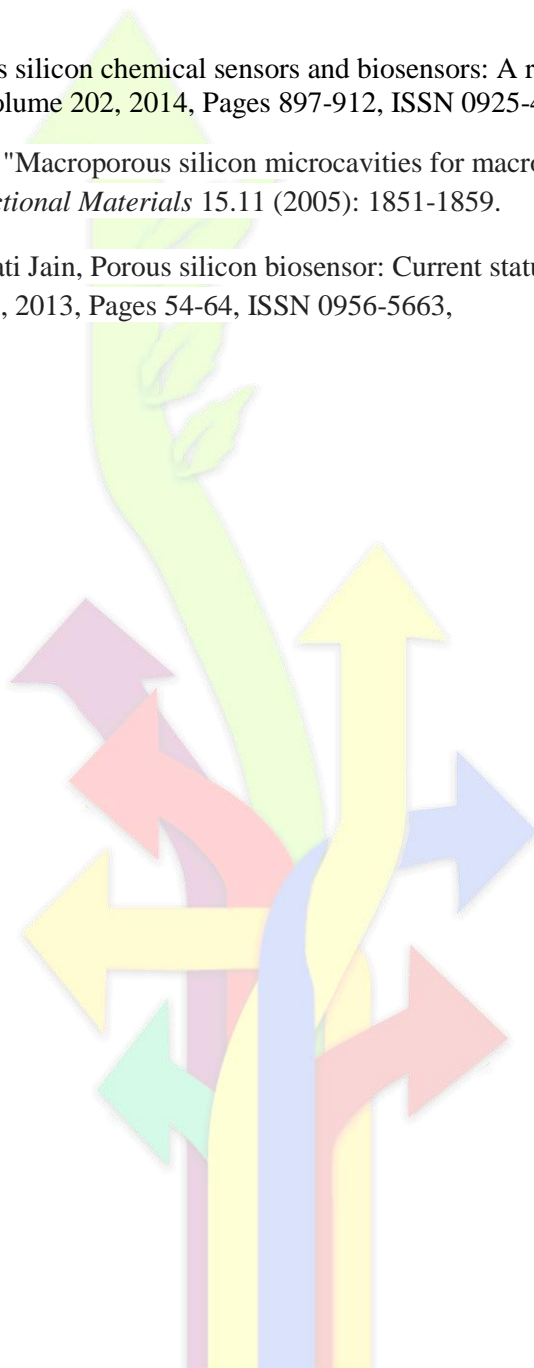
The etching current density was fixed at 40 mA/cm² for different etching time of 10, 15, 20, 25, 30 and 35 minutes. It was found that etching time is an important parameter for photo-

electrochemical etching of n-type silicon. Therefore, by optimizing etching time, the physical properties of PSi samples can be optimized for different applications [1-3].

Keywords: Porous silicon, Etching time, Electrochemical etching

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13th Annual Electrochemistry Seminar of Iran

Electrochemical Synthesis of Nanometal Oxides based on Iron and Cobalt Nanoparticles

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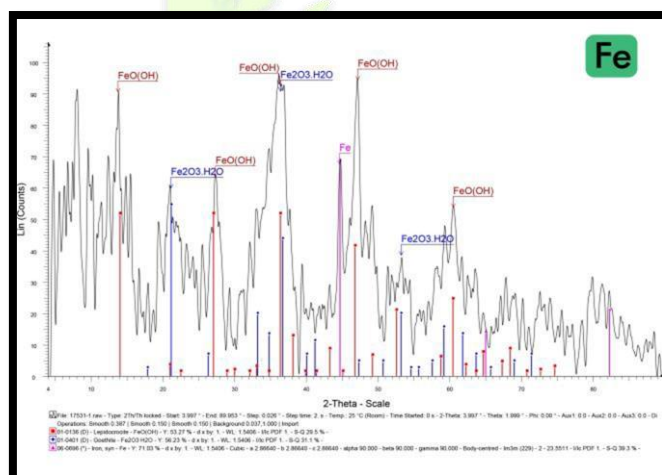
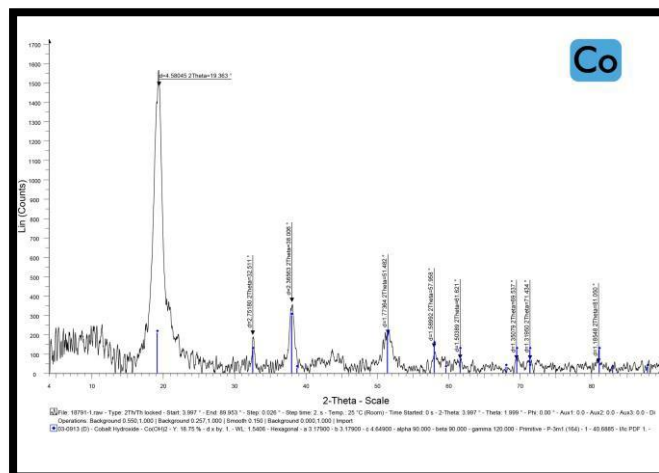
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Abstract

Nanotechnology is one of the most important issues in today societies that is developing fast in all industrial and scientific fields. Perhaps one of the simple nanostructures that has been used widely in industry nowadays is nano metal oxides. One of the best methods for synthesizing nano oxides such as Iron, Cobalt, Copper, etc. is electrochemical deposition that cathodic electrodeposition is one of the subgroups of this method. In this method hydroxide was deposited via cathodic electrodeposition from low temperature 0.005 M FeNO₃ bath. The cathodic current density was 2 mA cm⁻². Producing alkali is done near the surface of electrode under the effect of reaction of oxygen pre-reactor such as Nitrat ions. Electrochemical cell include cathode whose material is stainless steel. The XRD pattern confirmed that the prepared sample has a crystal phase FeO(OH).

One of the disadvantages of this method that is special to Ironoxide is the low adhesion of the particle to the electrod surface. This problem has been solved approximately by decreasing the movement of solvent and sample molecules via decreasing bath temperature.



Keywords: Electrodeposition Nanostructures FeO(OH)

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13th Annual Electrochemistry Seminar of Iran

Electrochemical synthesis of crystallographic phase controlled Zinc hydroxide

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Abstract

Controlled potential dissolution of Zn anode is an important technique from both scientific and industrial approach. Significance of zinc hydroxide as precursor of ZnO nanostructures, as well as its application in skin lotions, baby powders ointments and other cosmetics is the motivation of researchers in this era. In this work, electrochemical synthesis of zinc hydroxide has been studied in presence of cyanoguanidine as complexing agent in a simple one compartment cell containing zinc metal electrodes as anode and cathode. Cyanoguanidine has been selected for the above mentioned reaction due to existence of CN and amine functional groups in its structure. Sono-electrochemistry comparison with conventional mixing the solution during electrosynthesis was also investigated.

Electrochemically precipitated materials were investigated by x-ray diffraction and FT-IR spectroscopy techniques. Results showed that controlled potential electro-dissolution of zinc in sodium nitrate electrolyte solutions containing cyanoguanine results in solely the Wulffite crystallographic structure of $\text{Zn}(\text{OH})_2$.

Keywords: Electrosynthesis, $\text{Zn}(\text{OH})_2$, Wulffite, Complexing agents

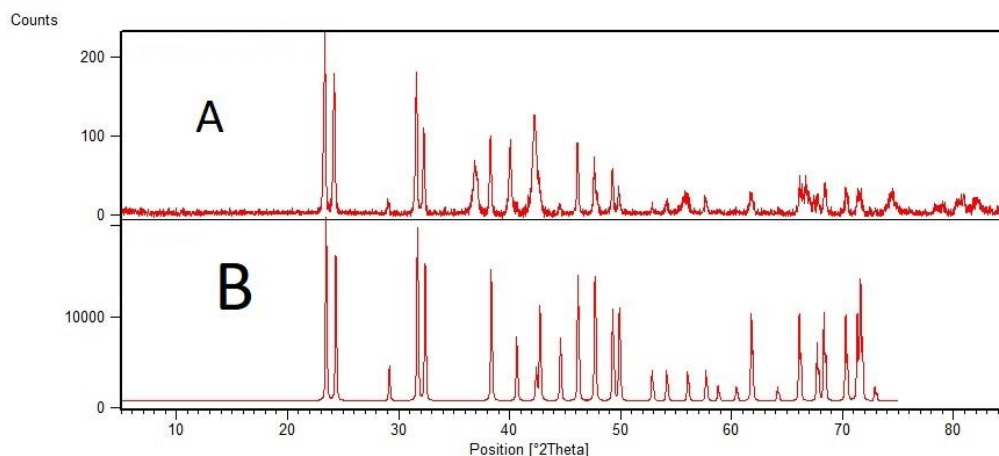


Fig. 1. XRD pattern of a) electrochemically synthesized zinc hydroxide b) Wulffite as reference

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Cathodic Electrodeposition: Synthesis, Characterization and Luminescence properties of YAG: (Ce, Sm and Gd) Nanophosphor

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Abstract

YAG: RE (RE: Ce³⁺, Sm³⁺ and Gd³⁺) nanophosphor were synthesized by a novel method based on the cathodic electrodeposition of some rare earth cations. In this method, hydroxide precursors were grown on the surface of cathode and then the final product was obtained via heat treatment of hydroxide powders at 1100 °C for 4 h. The formation of oxide product was confirmed by XRD, XPS, FTIR, EDX and SEM analysis. The photoluminescence properties of prepared materials were studied and results revealed that cathodic electrodeposition is a suitable and versatile approach for preparation of YAG: RE nanophosphors with emission spectra in the visible range.

Keywords: Cathodic electrodeposition; Nanophosphor; YAG:RE; Photoluminescence

Cathodic Electrodeposition of YAG Nanostructure: Effects of Current Density on the Crystal Structure and Morphology

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Abstract

Yttrium aluminum garnet (YAG) was successfully prepared by cathodic electrodeposition process from the mixture of YCl_3 and AlCl_3 dissolved in water/ethanol 1:1 solution through applying different current densities. First, hydroxide precursors were cathodically grown on the cathode surface at the different current densities, then hydroxide powders were heat-treated at 1100 °C for 4 h. The oxide products were characterized by XRD, FTIR, DSC–TGA and SEM techniques. The results of the SEM as well as the effect of applied current density on the morphology and particle size of nanostructures of YAG were investigated. The results revealed that cathodic electrodeposition followed by heat-treatment can be used as a facile method for preparation of YAG nanostructures with different morphology.

Keywords: Yttrium aluminum garnet; Nanostructure; Cathodic electrodeposition: Morphology Electrochemistry - Industry

Electrochemical deposition of highly porous nanostructured copperoxide foam fiber as a novel sorbent for solid phase microextraction method

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Abstract

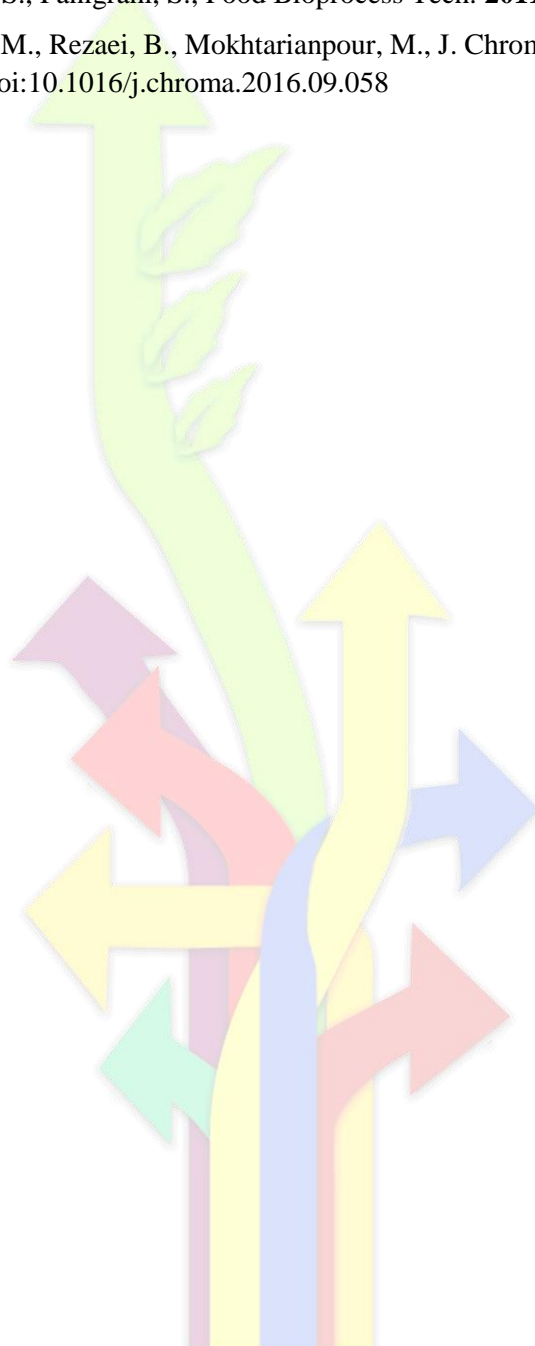
In the past two decades, the determination of different compounds such as BTEXs in different environmental samples has received great attention because of their toxicity for human and the environment. Direct analysis of the pollutants is not possible because of low concentration of these compounds in environmental samples. So separation and pre-concentration steps are required prior to final analysis. Solid phase microextraction (SPME) is a solvent-free microextraction technique applicable for the extraction of analytes from various matrices. In spite of the great applicability of SPME method, it has some major difficulties such as low chemical and mechanical coating stability, memory effect, expensive fibers, limited selection of commercially available fibers, fragility and limited lifetime of the fiber, and easy swelling in organic solvents. Therefore, most attempts have been focused on obviating the disadvantages of SPME. Nevertheless, the synthesis procedures are relatively complex, time consuming and difficult.

Herein, a new headspace solid phase microextraction technique based on using a copper oxide foam nanostructure substrate followed by gas chromatography-flame ionization detection was developed for the determination of BTEXs in water and wastewater samples. The copper foam with highly porous nanostructured walls was fabricated on the surface of a copper wire by a rapid and facile electrochemical process during 3 seconds. Then the prepared copper foam was converted to copper hydroxide by hydrothermal method and finally changed to copper oxide foam by the thermal process. Finally, the fiber used for the microextraction of benzene, toluene, ethylbenzene and xylenes. The experimental parameters such as desorption temperature, desorption time, salt concentration, sample temperature, equilibrium time and extraction time, were investigated and optimized by experimental design method. The fiber-to-fiber reproducibility for three fibers prepared under the same condition was 5.6-8.4%.

Keywords: *Copper oxide nanofoam, Electrochemical deposition, Solid phase microextraction, Copper foam, BTEXs*

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<http://dx.doi.org/doi:10.1016/j.chroma.2016.09.058>



13th Annual Electrochemistry Seminar of Iran

Fabrication of bimetallic Pd/Pt nanostructure deposited on copper nanofoam substrate as a novel sorbent for solid phase microextraction method

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Abstract

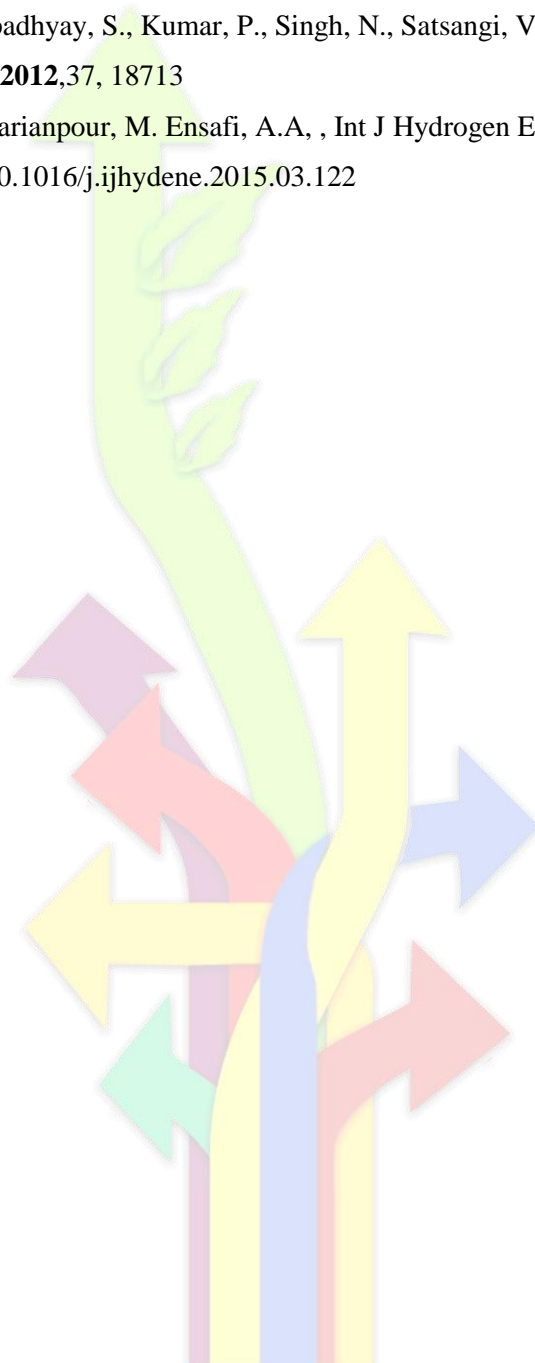
Nano materials and structures play an important role in technologies such as energy applications, catalytic properties, separation science, fuel cells and water splitting. The structure of supporting materials is very important. The producing 3-D nanofoam structure of metals with highly porous dendritic is a simple and very effective method. This process is done by the electrochemical deposition accompanying hydrogen evolution. In this technique, produced hydrogen bubbles are as a dynamic template for metal deposition with dense metallic components and unique pore size distribution. Solid phase microextraction (SPME) is a simple, sensitive, solvent free, portable, easy to automate and environmentally friendly microextraction technique applicable for the extraction of analytes from various matrices. The main disadvantages of SPME are the limited selection of commercially available fibers, fragility and limited lifetime of the fiber, and easy swelling in organic solvents. Therefore, most attempts have been focused on obviating the disadvantages of SPME.

In this work, the Pd/Pt bimetallic nanofoam was fabricated on Cu nanofoam substrate. The substrate was electrochemically prepared by copper reduction during 3 seconds and covered with Pd/Pt by galvanic replacement reaction in aqueous solutions of Pd (II) and Pt (IV). Then the prepared fiber was used as the sorbent for solid phase microextraction (SPME) method for the extraction and analysis of BTEX from the environmental samples as the HS-SPME method. The experimental parameters such as desorption temperature, desorption time, salt concentration, sample temperature, equilibrium time and extraction time, were optimized by experimental design method. The fiber-to-fiber reproducibility for three fibers prepared under the same condition was 6.4-7.8%.

Keywords: Copper nanofoam, Electrochemical deposition, Solid phase microextraction, Pd/Pt bimetallic nanofoam

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13th Annual Electrochemistry Seminar of Iran

In situ synthesis of ZIF-67 in porous nanostructured copper foam substrate as a sorbent for solid phase microextraction method

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Abstract

Metal-organic frameworks (MOFs), a class of microporous crystalline hybrid materials was first introduced in 1995 by Yaghi et al. MOFs are formed by coordination of metal cations (or cationic clusters) with multifunctional organic linkers. The significant properties of MOFs, including large surface area and porosity, uniform pore sizes, tunable surface chemistry, structural diversity, excellent thermal and solvent stability, make MOFs highly promising in diverse applications including gas separation and storage, catalysis, chemical sensing, biomedical imaging, separation science and drug delivery. In recent years, various MOFs are synthesized and explored as separation media in analytical chemistry. To some extent, MOFs are usually synthesized via one-pot self-assembly reactions between ligands and metal salts in solutions at a certain temperature. The synthetic conditions are mild and easily controlled to obtain flexible and ideal structure. Ramification of electrodeposited metals such as copper and tin has been a very interesting topic for scientists. In particular, three-dimensional (3D) nanoramified metal deposits are suitable for sensors, batteries and fuel cells.

In the present study, a simple and very rapid procedure was used to electrodeposit a copper foam coating with highly porous nanostructured walls on the surface of a copper wire. The porous structure of coating was impregnated Co and Zn for using as holder and substrate for in-situ approach for synthesis of ZIF-8 and ZIF-67. Finally the prepared fiber was used as extraction device in head space solid phase microextraction (HS-SPME). Herein benzene, toluene, ethylbenzene and xylenes were selected as the model compounds. Various experimental parameters affecting the extraction efficiency, such as desorption temperature, desorption time, salt concentration, temperature effect, equilibrium time and extraction time, were investigated and optimized.

Keywords: Electrochemical deposition, Copper foam, Metal-organic framework, Zeolitic-imidazolate frameworks, ZIF-8, HS-SPME

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Growth ZIF-8 on electrospun PAN nanofiber for TFME procedure

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Abstract

As a new geometry for solid-phase microextraction, thin-film microextraction (TFME) has become an attractive sample-preparation technique[1]. The high surface area-to-volume ratio together with the increase of extraction-phase volume enhanced the sensitivity of this technique without sacrificing the sampling time compared to other microextraction approaches. Comprehensive research has demonstrated the good agreement of the experimental data with the fundamental principle of this technique. In this work, Zeolitic imidazolate framework-8 (ZIF-8)-based PAN nanofibrous films were successfully fabricated by a simple strategy of in situ loading on a PAN substrate. In addition, the thin film was confirmed by scanning electron microscopy and X-ray diffraction. This thin film nanofibrous was applied in water treatment [2] by combining the unique properties of microporous material as active sizes for TFME procedure with a high surface area of electrospun polyacrylonitrile (PAN) film as the porous substrate.

Keywords: Thin film microextraction, PAN, Zeolitic imidazolate framework-8

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Microwave synthesis of CuO/NiO magnetic nanocomposites and its application in photo-degradation of methyl orange

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Abstract

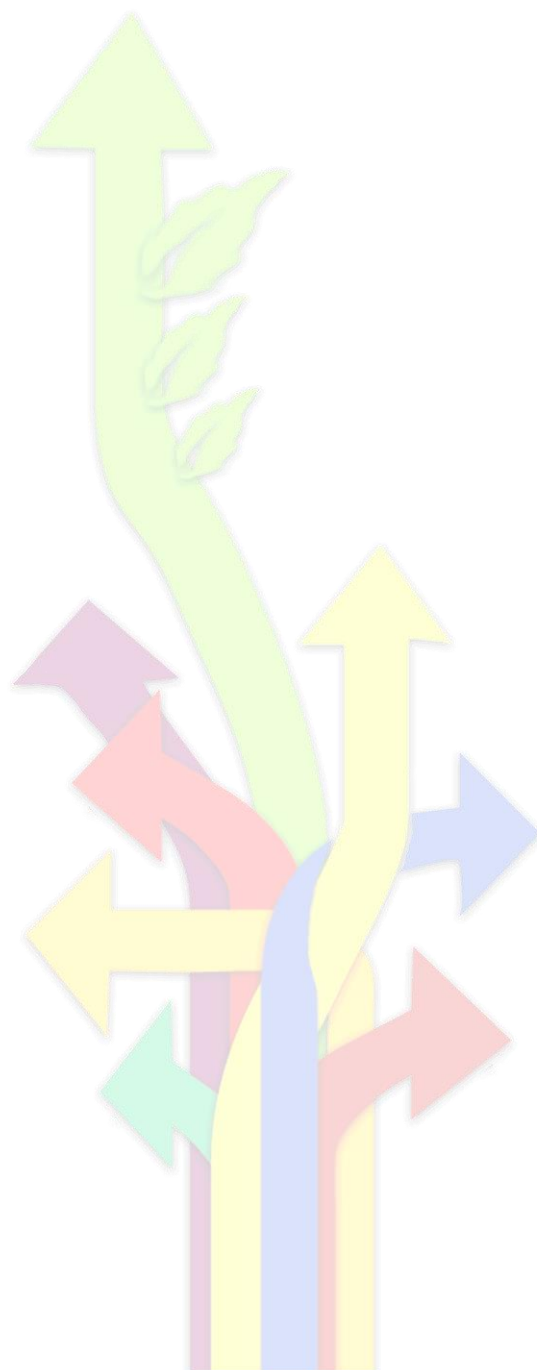
In this work CuO/NiO nanocomposites were synthesized via a fast microwave method at short period of time. The prepared products were characterized by X-ray diffraction pattern (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and Fourier transform infrared (FT-IR) spectroscopy. Alternating gradient force magnetometer (AGFM) illustrated ferro-magnetic behaviour of CuO/NiO nanocomposites. The photocatalytic behaviour of CuO/NiO nanocomposites was evaluated using the degradation of methyl orange under ultraviolet light irradiation. The results show that nanocomposites have applicable magnetic and photocatalytic performance.

Keywords: Microwave, Nanocomposite, Photocatalytic

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Physical Electrochemistry



13th Annual Electrochemistry Seminar of Iran

Electrocatalytic glucose oxidation via nanomaterial catalyst: Ni-Fe Layered Double Hydroxide/ Graphene

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Abstract

Nickel-Iron Layered Double Hydroxide nano composites were electrochemically synthesized on graphene/ glassy carbon electrode. This electrode was studied for glucose electro-oxidation reaction using cyclic voltammetry, chronoamperometry. Results confirmed high catalytic activity, stability of the graphene/Ni-Fe LDH electrode and glucose electro oxidation reaction on this electrode is under the effect of diffusion process.

Keywords: Nickel-Iron Layered Double Hydroxide, Catalytic activity, Glucose, Electro-oxidation

Kinetic study of modified copper electrode for ethanol oxidation reaction

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Ethanol as fuel can be used in alkaline fuel cells. The sluggish ethanol oxidation reaction has been attracted many researchers for investigate how can they fast the kinetic of this reaction. At this work simple electrodeposition method was introduced for preparing modified copper electrode. The copper electrode was successfully synthesized by applying constant potential at -0.4 v versus saturated calomel electrode in electrodeposition solution ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ 1 M, H_2SO_4 0.7 M) on copper sheet for 120 s. The prepared electrode was studied for ethanol oxidation reaction by electrochemical methods. The concentration of ethanol and KOH in solution were 7 mM and 1 M respectively. For calculating activation energy, the linear sweep voltammetry (lsv) method was used at various temperature. According the maximum current density related to ethanol oxidation reaction at different temperature, the activation energy was calculated by Arrhenius equation. This amount was 33.71 kJ. The response of prepared electrode for ethanol oxidation with concentration was investigated. For doing that, the ethanol concentration was varied from 1 mM to 10 mM. based on lsv method results, there is linear relation between maximum current density and ethanol concentration. According these results, the fabricated electrode could be good candidate for ethanol sensors at mentioned ethanol concentration.

Keywords: Ethanol oxidation ,activation energy ,ethanol sensor, fuel cell

Effect of temperature and solvent on the stability constant of complex formation between Zn^{2+} and Cu^{2+} cations with 1, 8-dihydroxyanthraquinone and 1-(methylamino)anthraquinone ligands using Response surface method.

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Abstract

In this study, response surface methodology was applied to determine the optimized condition, the stability constant between Zn^{2+} and Cu^{2+} cations with 1,8-dihydroxyanthraquinone (DHAQ) and 1-(methylamino)anthraquinone (MAAQ) ligands in acetonitrile (AN) - methanol (MeOH) binary solutions at different temperatures using conductometric method. The stability constant of complexes is obtained from fitting of molar conductivity data using GENPLOT computer program. The statistical analysis was performed using Minitab 16 software and fitted to a second-order polynomial regression model containing the coefficient of linear, quadratic and interaction terms. Central composite design (CCD) was used to investigate the effects of temperature and mol% AN as independent and dependent variables.

An analysis of variance (ANOVA) with 95% confidence level was carried out for response. Regression coefficients of predicated polynomial models are summarized in Table 1.

Table 1. Regression coefficients of predicated polynomial models

Coefficient ^a	(DHAQ. Zn^{2+})	(DHAQ. Cu^{2+})	(MAAQ. Zn^{2+})	(MAAQ. Cu^{2+})
β_0	2.71*	3.79	3.94	3.45
β_1	0.52	0.67	0.45	0.67
β_2	0.09	0.13**	-0.22*	0.01****
β_{11}	0.33	-0.26	-0.27*	***
β_{22}	0.35	****	-0.28	0.25
β_{12}	****	****	****	0.26
Model	0.001	0.000	0.001	0.000
Linear	0.000	0.000	0.000	0.000
Quadratic	0.001	***	***	***
Lack of fit	ns	ns	ns	ns
R^2	96.21	95.90	98.23	97.58

^aPolynomial model $Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i=1}^{k-1} \sum_{j=i+1}^k \beta_{ij} X_i X_j$

where β_0 is the constant coefficient, β_i is the linear coefficient, β_{ii} is the quadratic coefficient, and β_{ij} is the two factors interaction coefficient

* significant $p \leq 0.005$; **significant $p \leq 0.001$; ***significant $p \leq 0.01$; **** not significant; ns, not significant $p \geq 0.01$

Response surface plots of the effect of temperature and mol%AN on the stability constant (DHAQ. Zn^{2+}), (DHAQ. Cu^{2+}), (MAAQ. Zn^{2+}) and (MAAQ. Cu^{2+}) are presented in Figure 1.

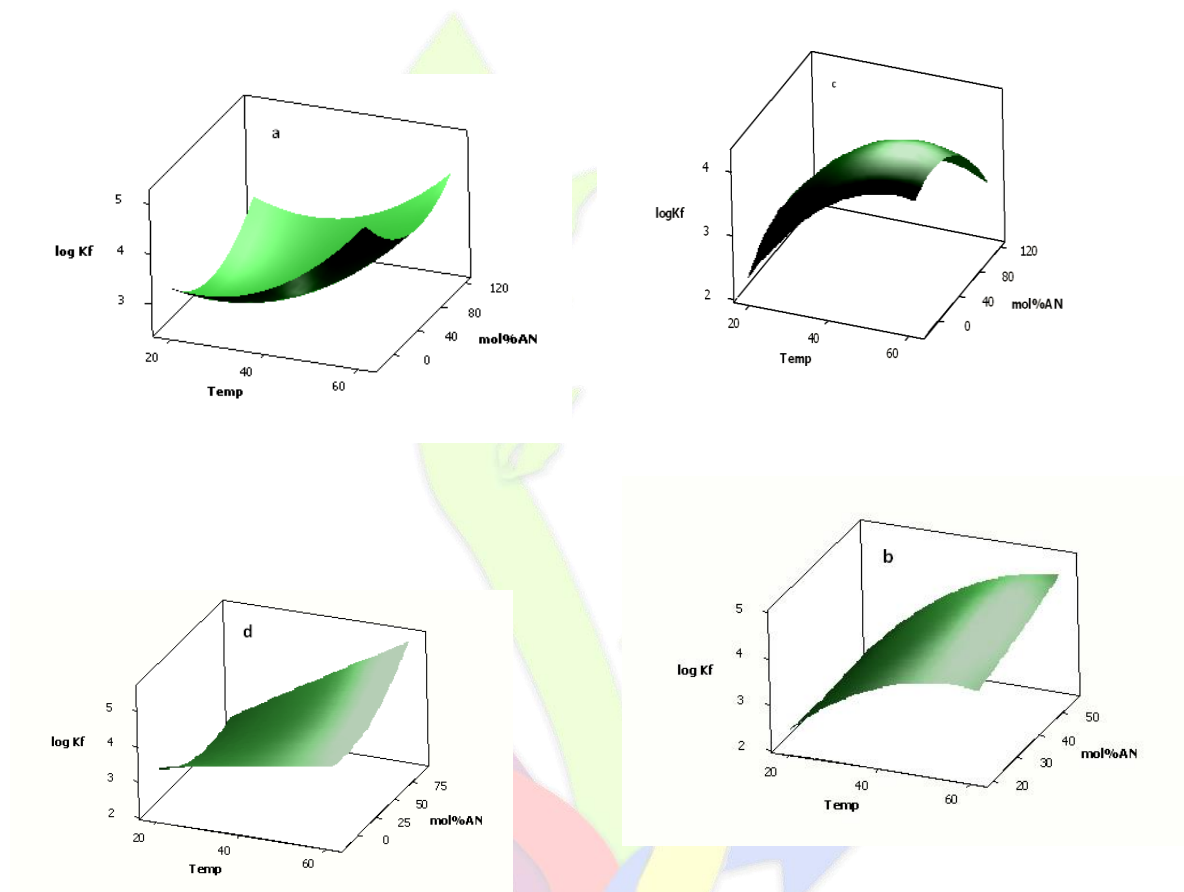


Figure 1. Response surface plots showing the effects of temperature and mol%AN on the stability constant a:

(DHAQ. Zn^{2+}), b: (DHAQ. Cu^{2+}), c: (MAAQ. Zn^{2+}) and d: (MAAQ. Cu^{2+}).

As seen from Figure 1, the complexation reaction between Zn^{2+} and Cu^{2+} cations with 1,8-dihydroxyanthraquinone (DHAQ) and 1-(methylamino)anthraquinone (MAAQ) ligands are endothermic and the $\log K_f$ increase with the increasing mol % AN in AN-MeOH binary solution.

Keywords: Central composite design, the stability constant, 1,8-dihydroxyanthraquinone, 1-(methylamino) anthraquinone, Zn^{2+} and Cu^{2+} , conductometry.

Conductometric study of complexation reactions between Benzyl bis semicarbazone and 3-Methylpyrazol-5-one with Cd^{+2} cation in pure and binary non-aqueous solvents

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Abstract

Semicarbazones are compounds which are synthesized by the condensation of semicarbazide and aldehydes/ketones [1]. An interesting aspect is that the semicarbazones show a variety of coordination modes with transition metals [2]. Pyrazolone is a five-membered lactam ring, containing two nitrogen and one ketonic group in its structure [3].

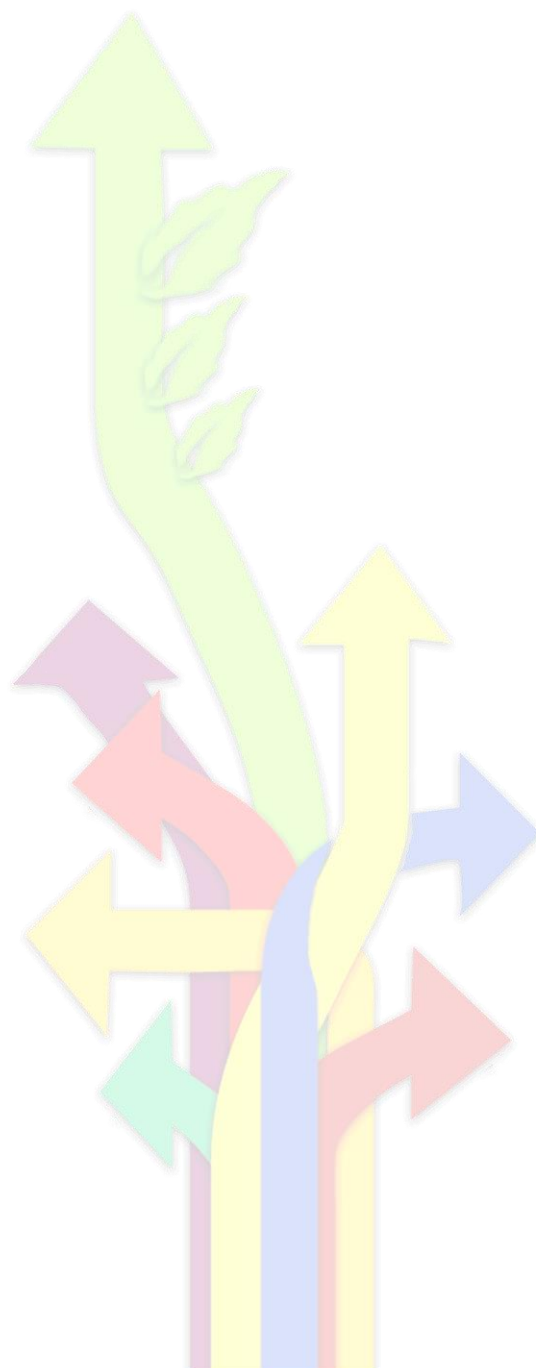
In this paper, the complexation reactions between Cd^{+2} cation with benzyl bis semicarbazone (BSC) and 3-methylpyrazol-5-one (3-MP) ligands were studied in acetonitrile-ethylacetate (AN-EtOAc), acetonitrile-dimethylformamide (AN-DMF) and ethylacetate-dimethylformamide (EtOAc-DMF) binary mixtures at different temperatures using conductometric method. The results show that in all cases, the stoichiometry of the formed complexes is 1:1 (ML). The stability constants of the resulting complexes were calculated from the computer fitting of the molar conductance-mole ratio data at various temperatures. The selectivity order of Cd^{+2} cation for BSC and 3-MP ligands in most cases was found to be: $(\text{BSC.Cd})^{+2} > (\text{3-MP.Cd})^{+2}$. A nonlinear relationship was observed for changes of $\log K_f$ of complexes versus the composition of the binary mixed solvents. The values of standard thermodynamic quantities (ΔH°_c , ΔS°_c) for formation of $(\text{BSC.Cd})^{+2}$ and $(\text{3-MP.Cd})^{+2}$ complexes were obtained from temperature dependence of the formation constants using the van't Hoff plots. The results show that the values and also the sign of these parameters are influenced by the nature and composition of the mixed solvents.

Keywords: Cd^{+2} ; Benzyl bis semicarbazone; 3-Methylpyrazolone; conductometry; non-aqueous.

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13th Annual Electrochemistry Seminar of Iran

Effects of Cl in Electrodeposition Baths on Morphologies of Zinc Oxide Thin Films

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Abstract

Thin compact layers of ZnO were grown on the conductive transparent substrate of FTO by electrochemical deposition method at 85°C for application perovskite solar cells. Different Cl concentrations and potentials are investigated to optimize the final ZnO layer. The quality of layer is evaluated by scanning electron microscopy (SEM), diffused transmittance spectroscopy (DTS) and electrochemically by ferro/ferricyanide as the model redox probes. FE-SEM images demonstrate that the FTO surface is effectively covered by ZnO for deposition times more than 400 seconds. XRD spectra indicates the preferential growth along the (001) direction for ZnO films. Evidence from cyclic voltammograms show that ZnO blocking layers deposited at $t > 400$ s successfully suppress interfacial charge recombination. DTS data demonstrate that as Cl concentration increases, as ZnO film transmittance decreases.

Keywords: Zinc oxide, Chlorine, Electrochemical

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