Electrochemical Preparation and Characterization of Some Metallic and Metallic Oxides Nanostructures and Their Biological Study

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ABSTRACT

This thesis comprises five main chapters:

Chapter 1: contains an introduction to the most important methods that were used to study the metallic and metallic oxides nanoparticles. This chapter also debriefs the literatures of some analogous studies.

Chapter 2: lists some important details of the materials and chemicals used in this work and describes the experimental procedures concerning samples preparations, physical measurements, methods and calculations.

Chapter3: In this chapter, TiO_2 nanotubes were synthesized electrochemically by anodization of pure Ti sheet in 0.5% HF solution (20 V, 20 min) to obtain (TiO_2 NTs) then the nickel oxide was deposited on the support TiO_2 NTs by immersing TiO_2 NTs in mesoporous sol-gel template Ni nanoparticles method for overnight and then annealed in N_2 atmosphere to obtain the electro catalyst NiO NPs/TiO_2 NTs-N_2. The physicochemical characterizations of NiO NPs/TiO_2 NTs-N_2 were performed by scanning electron microscopy (SEM), X-ray diffraction (XRD) analysis. The catalyst NiO NPs/ TiO_2 NTs-N_2 was electrochemically characterized in basic solution of methanol by cyclic voltammetry (CV) measurement. XRD characterization indicates the presence of nickel oxide nanoparticles with diameter less than 43 nm and regularly combined into TiO_2 nanotubes. NiO NPs/ TiO_2 NTs-N_2 catalyst exhibited a remarkably high electrocatalytic activity and have high electrocatalytic stability for methanol electrocatalytic oxidation reaction. The electrochemical studies showed that NiO NPs/ TiO_2 NTs-N_2 in alcoholic basic solution exhibited higher catalytic activity and stronger poisoning-tolerance for

methanol oxidation than that in non-alcoholic basic solution. This suggests that the NiO NPs catalyst supported on TiO_2 NTs-N_2 has promising potential applications in electro catalyst reactions. Nickel oxide supported on the conductive support TiO_2 NTs-N_2 is used as electro catalyst in alkaline fuel cells. Results showed best improvement in physico-chemical properties of these catalysts in comparison with previous studies. The antibacterial properties of the TiO_2 NTs-N_2, NiO nanoparticles and NiO NPs/ TiO_2 NTs-N_2 were assessed against two common bacterial strains using disc diffusion assay. The minimum inhibitory concentration (MIC) was measured.

Chapter 4: In this chapter, TiO_2 nanotubes were prepared electrochemically as pure Ti sheet in 0.5% HF anodized and annealed below nitrogen acquiring the TiO_2 NTs-N_2 support .By chemical deposition method using $NaBH_4$ as reducing agent, platinum was deposited on TiO_2 NTs-N_2 support to gains the Pt NPs/TiO_2 NTs-N_2 electrocatalyst. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) have been applied for the physicochemical characterization of Pt NPs/TiO_2 NTs-N_2. While, the electrochemical characterization of Pt NPs/TiO_2 NTs-N_2 was measured in basic solution of methanol using cyclic voltammetry. XRD exhibits the an existence of platinum nanoparticles with diameter much less than 54.8 nm, which is regularly combined with TiO_2 nanotubes . The Pt NPs/TiO_2 NTs-N_2 catalyst indicates considerably high electrocatalytic activity and stability for methanol electrocatalytic oxidation reaction. The catalyst Pt NPs/TiO_2 NTs-N_2 was electrochemically characterized in a basic solution of methanol using cyclic voltammetry (CV) measurement in the absence and an existence of light. The higher poison tolerance and higher methanol oxidation current density observed for the Pt NPs/TiO_2 NTs-N_2 catalyst suggests that this catalyst supported on TiO_2 NTs-N_2 has a promising potential applications in the electro catalyst reactions. In addition, synthesized platinum nanoparticles deposited on TiO_2 nanotubes reveals a tremendous effective of antibacterial activity towards pathogenic microorganism's.

Chapter 5: In this chapter, TiO_2 NPs powder (titanium oxide nanoparticles powder) is prepared by anodization in 0.7 M $HClO_4$ is annealed in N_2 at 450°C for 3 h to obtain the TiO_2 NPs -N_2 powder as catalyst support to which Au is loaded by photodeposition technique using tetrachloroauric acid ($HAuCl_4$) and isopropanol as sacrificial donor. The physicochemical characterization of TiO_2 NPs arrays powder were performed by energy dispersive X-ray Diffractive (EDX) analysis, transmission electron microscopy (TEM), X-ray diffraction (XRD), and surface area and pore size analysis. The resulting Au NPs/TiO_2 NPs-N_2 powder catalyst exhibits BJH high surface area of 121.128 m^2/g. XRD characterization indicates the presence of Au nanoparticles with average diameter about 26.8 nm and uniformly incorporated into TiO_2 NPs -N_2. The XRD patterns and EDX spectra showed that the prepared Au NPs/ TiO_2 NPs-N_2 were crystalline and Nano-sized. The TEM image suggested that Nano particles were spherical and there was tendency of agglomerations. The electro-catalytic activity of Au NPs/TiO_2-NTs-N_2 catalysts in ferrocyanide system , in KOH and in H_2SO_4 electro-oxidation was examined by cyclic voltammetry and contrasted with a financially accessible polycrystalline gold electrode. Au-TiO_2 NPs-N_2 powder electrode catalyst displays a strikingly high electrocatalytic activity in KOH , in H_2SO_4 and in (2 M KOH + 0.1 M glycerol) solutions. Electrocatalytic activity of gold nanoparticles (Au NPs) loaded on TiO_2 NPs toward oxygen reduction reaction (ORR) has been examined by cyclic voltammetry (CV). The electrocatalytic activity of AuNPs toward oxidation of glycerol was assessed in the

alkaline medium by cyclic voltammetry. The results indicate Au NPs/TiO_2-NTs-N_2 catalysts as a promising support material improve the excellent electro-catalytic activity for acidic and basic oxidation greatly. So Au NPs/TiO_2-NPs-N_2 electrode can be used repeatedly and exhibits stable electro-catalytic activity for the acidic and basic oxidation.

The catalyst supports TiO_2 NPs arrays powders are electrochemically characterized in basic solution of glucose by cyclic voltammetry (CV measurements). Au- TiO_2 NPs-N_2 powder electrode catalyst exhibits a remarkably high electrocatalytic activity and has high electrocatalytic stability for glucose electrocatalytic oxidation reaction. Au supported on the conductive support TiO_2 NPs -N_2 is used as electro catalyst in Glucose Alkaline Fuel Cell (GAFC) applications. The electrocatalytic properties of nanostructured gold electrodes for glucose electro-oxidation in KOH were investigated by cyclic voltammetry and compared with a commercially available polycrystalline gold electrode. Gold on treated TiO_2 nanoparticles turned out to be a very promising anode for glucose electro-oxidation.

The antimicrobial activity of TiO_2 nanoparticles and Au NPs/TiO_2 NPs was analyzed by subjecting it to human pathogenic bacteria (Gram-positive *Staphylococcus aureus* (*S. aureus*) and Gram-negative (*Escherichia coli* (*E. coli*) using disc diffusion method. The TiO_2 nanoparticles and Au NPs/TiO_2 NPs showed the efficient antibacterial and activity of above-mentioned microbes. It was confirmed that Au NPs/TiO_2 NPs have the best antimicrobial agent compared to the standard antibiotics (Gentamicin).