SYNTHESIS AND CHARACTERIZATION OF CARBON NANOTUBE-QUICKLIME NANOCOMPOSITES AND REDUCED GRAPHENE OXIDE HYBRIDS FOR SCREEN PRINTED ELECTRODE MODIFICATION

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By

RUZANNA BINTI IBRAHIM

Thesis submitted to the School of Graduate Studies Universiti Putra Malaysia, in the Fulfilment of the Requirements for the Degree of Doctor of Philosophy

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DEDICATIONS

To my parents and my husband

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

SYNTHESIS AND CHARACTERIZATION OF CARBON NANOTUBE-QUICKLIME NANOCOMPOSITES AND REDUCED GRAPHENE OXIDE HYBRIDS FOR SCREEN PRINTED ELECTRODE MODIFICATION

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October 2019

Chair: Prof Mohd Zobir Bin Hussein, PhD
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Carbon nanomaterials such as carbon nanotubes (CNT) and reduced graphene oxide (RGO) have become the materials of interest due to many desirable properties. A nanocomposite containing both CNTs and calcium oxide (CaO) or quicklime is beneficial since the high conductivity of CNTs are favourable for electrochemical detection and CaO may increase thermal stability. Additionally, it has been shown that the performance of CNTs or RGO alone in applications such as electrochemical detection is constricted due to agglomeration of both materials caused by π-π interactions. Decorated nanomaterials and nanomaterial hybrids have also demonstrated better performance as sensor platforms compared to the base materials. In this work, CaO-supported catalysts (Ni/CaO, Co/CaO and Fe/CaO) were used to synthesize carbon-nanotubes quicklime nanocomposites (CQNs) via chemical vapour deposition (CVD) of hexane with studies on the effects of catalyst composition and CVD temperature. The CQNs were then characterized using X-ray diffraction (XRD), Raman spectroscopy, nitrogen adsorption desorption isotherms, field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM) and thermogravimetric analysis (TGA).

Various RGO hybrids were also prepared including RGO-carbon nanotubes quicklime nanocomposites (RGO-CQN), RGO-carboxylated multiwalled carbon nanotubes (RGO-MWNT) and RGO-silver nanoparticle hybrid (RGO-Ag). The RGO was synthesized using the improved Hummer's method followed by reduction using hydrazine. RGO and silver nanoparticles hybrids (RGO-AgNPs) were synthesized via the reduction of GO and silver nitrate ($AgNO_3$) using a combination of hydrazine hydrate and sodium citrate. RGO-Ag were characterized using XRD, FESEM, TEM, UV-Vis and FTIR. CQNs-modified screen printed carbon electrodes (SPCE) and RGO-hybrids-modified SPCE were subjected to cyclic voltammetry (CV) studies with potassium ferricyanide ($K_3Fe(CN)_6$) redox probe in order to see their potential use in electrochemical detection.

Using Ni/CaO (10 wt%) (800 °C), Co/CaO (15 wt%) (800°C) and Fe/CaO (20 wt%) (850 °C) catalysts produced CQNs with the highest graphitization possessing I_G/I_D values of 1.30, 1.15 and 1.36, respectively. As SPCE modifier, the CQNs showed relatively high CV response compared to the bare electrode namely HNi10-800, HCo10-800 and HFe15-900 as indicated by their high anodic peak current values and low peak-to-peak potential separation, ΔEp. However, graphitization can be correlated with high electrochemical performance only for Ni/CaO and Co/CaO catalyzed CQNs since for Fe/CaO catalyzed CQNs other factors may attribute to the electrochemical performance. When compared to MWNT-modified SPCE, the CV response of the HNi10-800-modified and HFe15-900-modified SPCEs displayed comparable electron transfer and they also exhibited higher anodic peak currents.

Both RGO-CQN nanocomposites (RGO-HNi and RGO-HFe) showed increased CV response when they were used to modify SPCE. It can be seen that the modification of SPCE using RGO-CQN show better electrochemical response than using only RGO. It was also found that the RGO-CQN-modified SPCEs presented higher electroactive surface area compared to RGO-MWNT-COOH modified SPCE. The AgNPs grown on the RGO-Ag hybrids as observed from TEM and FESEM micrographs were polydispersed with the lowest mean diameter of around 20 nm for RGO-Ag5 (5 mM Ag). RGO-Ag10-modified SPCE displayed the highest CV current for all RGO-Ag synthesized in this work, presumably due to its better reduction and well dispersed AgNPs. However, RGO-Ag were found to be unstable on the SPCE surface. Meanwhile, RGO-Ag/MWNT-COOH-modified SPCE showed slower electron transfer due to significant increase in the peak potential separation but the stability of RGO-Ag/MWNT-COOH on SPCE is better compared to RGO-Ag.

Based on this work, the suitable material for SPCE modification would be CNT-Quicklime nanocomposite synthesized using Ni/CaO (HNi10-800) and Fe/CaO (HFe15-900) and RGO-CQN as they presented relatively high CV currents and low ΔEp values indicating fast electron transfer. It is anticipated that the carbon nanomaterial-modified electrodes from this work can be used for the electrochemical detection of heavy metal ions.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
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SINTESIS DAN PENCIRIAN KOMPOSIT TIUB NANO KARBON-BATU KAPUR DAN HIBRID GRAFIN OKSIDA TERTURUN UNTUK PENGUBAHSUAIAN ELEKTROD KARBON PAPARAN TERCETAK

Oleh

RUZANNA BINTI IBRAHIM

Oktober 2019

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Bahan karbon nano seperti tiub nano karbon (CNTs) dan grafin oksida terturun (RGO) adalah bahan nano yang sering menjadi fokus kajian kini kerana pelbagai ciri-ciri hebat yang dimiliki. Komposit nano yang terdiri daripada CNT dan kalsium oksida (CaO) atau CQNs adalah bermanfaat kerana konduktiviti elektrik CNT yang tinggi berguna dalam pengesanan elektrokimia dan kandungan CaO berkemungkinan meningkatkan kestabilan terma komposit tersebut. Di samping itu, keberkesanan setiap bahan karbon nano untuk aplikasi seperti sensor elektrokimia adalah terhad kerana penggumpalan setiap bahan disebabkan interaksi π–π. Kajian-kajian terdahulu mendapati bahawa prestasi bahan nano yang didekorasi atau berbentuk hibrid adalah lebih baik sebagai platform pembuatan sensor berbanding dengan bahan asalnya. Kajian ini melibatkan sintesis komposit CNT-CaO (CQNs) menggunakan kaedah pengenapan wap kimia (CVD) heksana dengan pemangkin Ni/CaO, Co/CaO dan Fe/CaO. Kesan komposisi pemangkin dan suhu CVD terhadap ciri-ciri CQNs yang terhasil telah diselidiki dalam kajian ini. CQNs yang dihasilkan telah dicirikan menggunakan kaedah pembelauan sinar-X (XRD), spektroskopi Raman, penyerapan-pembebasan nitrogen, mikroskopi imbasan elektron pemancar medan (FESEM), mikroskopi transmisi elektron (TEM) dan analisis termogravimetri (TGA).

Turut dikaji adalah pelbagai hibrid RGO termasuk komposit RGO-tiub nanokarbon/CaO (RGO-CQN), RGO-tiub nanokarbon berbilang dinding (RGO-MWNT) dan hybrid RGO-nanopartikel-perak (RGO-Ag). RGO telah disintesis menggunakan kaedah Hummer terubahsuai diikuti dengan penurunan menggunakan hidrazin. Hibrid RGO-Ag telah disintesis secara penurunan grafin oksida (GO) dan argentum nitrat ($AgNO_3$) dengan menggunakan kombinasi hidrazin dan natrium sitrat. RGO-Ag telah dicirikan menggunakan XRD, FESEM, TEM, UV-Vis dan FTIR. Elektrod karbon paparan tercetak (SPCE) yang diubahsuai menggunakan CQN dan hybrid-hibrid RGO telah dianalisis secara voltametri siklik (CV) menggunakan bahan redox kalium ferricyanide

($K_3Fe(CN)_6$) untuk melihat potensi bahan-bahan tersebut dalam pengesanan elektrokimia.

Kajian ini mendapati CQNs yang disintesis menggunakan pemangkin Ni/CaO(10 wt%) (800 °C) (HNi), Co/CaO (15wt%)(800°C) (HCo) and Fe/CaO(20 wt%) (850 °C) (HFe) menunjukkan pengrafitan terbaik dengan nilai nisbah I_G/I_D masing-masing adalah 1.30, 1.15 dan 1.36. Manakala apabila digunakan sebagai pengubahsuai SPCE, semua sampel CQN menunjukkan tindak balas CV yang lebih baik berbanding SPCE yang tidak diubahsuai terutamanya CQN HNi10-800, HCo10-800 dan HFe15-900. Walau bagaimanapun, kadar pengrafitan hanya boleh dikaitkan dengan prestasi elektrokimia untuk CQNs yang dimangkin oleh pemangkin Ni/CaO dan Co/CaO sahaja kerana berkemungkinan bagi CQN dari Fe/CaO terdapat faktor-faktor lain yang mempengaruhi ciri elektrokimia bahan tersebut. Prestasi bagus untuk CQN terbabit ditunjukkan oleh nilai arus puncak anodik yang tinggi dan jarak antara puncak upaya (ΔEp) yang rendah. Hanya SPCE terubahsuai dengan HNi10-800 dan HFe15-900 menunjukkan kadar perpindahan elektron yang setanding dengan MWNT-COOH komersil di samping memberikan nilai arus puncak anodik yang lebih tinggi.

Kedua-dua komposit nano RGO-CQN (RGO-HNi dan RGO-HFe) meningkatkan tindak balas CV apabila digunakan untuk mengubahsuai SPCE. Pengubahsuaian SPCE munggunakan RGO-CQN menunjukkan tindak balas elektrokimia yang lebih baik berbanding dengan hanya menggunakan RGO sahaja. Didapati juga bahawa luas permukaan elektroaktif bagi SPCE yang diubahsuai menggunakan RGO-CQN adalah lebih tinggi berbanding SPCE yang diubahsuai menggunakan RGO-MWNT-COOH. Mikrograf-mikrograf TEM dan FESEM menunjukkan AgNP yang dihasilkan dalam RGO-Ag adalah tersebar secara rawak dengan pelbagai bentuk dan saiz. Diameter purata terkecil ialah 20 nm bagi RGO-Ag5 (5 mM Ag). RGO-Ag10 menunjukkan arus puncak CV tertinggi apabila digunakan untuk mengubahsuai SPCE, mungkin kerana penurunan yang lebih baik dan penyebaran AgNP yang lebih bagus. Walau bagaimanapun, didapati bahawa RGO-Ag adalah tidak stabil di atas permukaan SPCE. Manakala untuk SPCE yang diubahsuai menggunakan RGO-Ag/MWNT-COOH, perpindahan elektron adalah lebih perlahan apabila nilai jarak antara puncak keupayaan CV didapati semakin besar tetapi RGO-Ag/MWNT-COOH adalah lebih stabil di atas permukaaan SPCE berbanding RGO-Ag.

Bahan komposit nano yang sesuai untuk pengubahsuaian SPCE dari kajian ini ialah komposit CQN menggunakan pemangkin Ni/CaO (HNi10-800) dan Fe/CaO (HFe15-900) dan RGO-CQN kerana arus puncak CV yang tinggi dan nilai ΔEp yang rendah menunjukkan perpindahan elektron yang pantas. Dijangka bahawa elektrod yang diubahsuai menggunakan bahan komposit karbon nano dari kajian ini boleh digunakan sebagai bahan untuk pengesanan ion logam berat.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

AgNPs	Silver Nanoparticles
Ag/AgCl	Silver, silver chloride
AuNPs	Gold Nanoparticles
ASV	Anodic Stripping Voltammetry
μA	micro Amperes
BET	Brunauer Emmet Teller
BJH	Barret Joyner Halenda
CNM	Carbon Nanomaterials
CNT	Carbon Nanotubes
CPE	carbon paste electrode
CQNs	Carbon Nanotubes-Quicklime Nanocomposites
CV	Cyclic Voltammetry
DPV	Differential Pulse Voltammetry
DPASV	Differential Pulse Voltammetry Anodic Stripping Voltammetry
DTG	Derivative thermogravimetry
EDX	Energy Dispersive X-ray
ESA	Electroactive surface area
FESEM	Field Emission Scanning Electron Microscopy
FTIR	Fourier Transform Infrared
GO	Graphene Oxide
HCo	CQNs synthesized using Co/CaO
HFe	CQNs synthesized using Fe/CaO
HNi	CQNs synthesized using Ni/CaO
HRTEM	High-resolution Transmission Electron Microscopy
LSV	Linear sweep voltammetry
LSASV	Linear Sweep Anodic Stripping Voltammetry
MWNTs	Multi-walled Carbon Nanotubes

MWNT-COOH	Carboxylated Multi-walled Carbon Nanotubes
NM	Nanomaterials
NP	Nanoparticle
Redox	Reduction-oxidation
RGO	Reduced Graphene Oxide
RGO-Ag	Reduced Graphene Oxide Silver Nanoparticles
RHFe	Reduced graphene oxide-HFe nanocomposite
RHNi	Reduced graphene oxide-HNi nanocomposite
RGOM	Reduced graphene oxide-MWNT-COOH nanocomposite
SPCE	Screen printed carbon electrode
SPE	Screen printed electrode
SWNTs	Single-walled carbon nanotubes
SWV	Square Wave Voltammetry
SWASV	Square Wave Anodic Stripping Voltammetry
UV-Vis	Ultra violet-visible
XRD	X-ray diffraction

CHAPTER 1

INTRODUCTION

1.1 Background

Materials have always been the defining element in the development of human civilizations. History has recorded the Stone Age, Bronze Age and Iron Age and the material of today would be nanomaterials. Nanomaterials presents a large class of material with at least one dimension lower than 100 nm (1 nm = 1 billionth of a meter). These materials offers great excitement to scientists and researchers as they have shown to exhibit characteristics which are more superior to the bulk materials and this presents great potential for use in technological advancements.

Nanomaterials are now at the forefront of technological developments such as electronics, composites and also medical technology. One main area which nanomaterials are applied is in electrochemical detection. Compared to chromatographic and spectroscopic methods, electrochemical detection strategies via voltammetric techniques such as cyclic voltammetry (CV), differential pulse voltammetry (DPV) and square wave voltammetry (SWV) provide a simpler, economical, rapid and on-site detection of organic and inorganic molecules (Ishikawa et al. 2009; Zhao et al., 2016) . This is because electrochemical sensing devices are easier to be miniaturized, have less power and space requirement and is also cost effective. Additionally, electrochemical sensing devices are also sensitive, selective and possesses a wide linear range (Cui et al., 2015; Hayat & Marty, 2014) making it more favourable compared to conventional detection techniques. For instance, in the case of heavy metal detection, conventional procedures are atomic absorption spectroscopy (AAS) and also inductively coupled plasma - mass spectrometry (ICP-MS) which although highly sensitive, suffer from shortcomings such as they require laboratory environment, tedious sample preparation, difficulty in operation and expensive equipment (Gao et al., 2013; Zhao et al., 2016).

Electrochemical sensing can be used to detect a wide variety of target analytes and are widely used for the detection of contaminants in food and water. One of the main contaminants in food and water are heavy metal ions that may be detrimental to human health. Meanwhile, some other metals which are required for the human body function will still pose a risk to health if it is present in excessive amount in the body (Aragay & Merkoci, 2012). Although it may not be possible to completely eliminate the presence of heavy metal contaminants in water, a sensitive detection procedure is important to ensure that the contamination level is kept at a minimum. In addition of identifying and quantifying amount of contaminants in water, the detection of heavy metal ions may also indicate the source of the water pollution (Luong et al., 2014).

Interests of using nanomaterials in electrochemical detection are mainly due to the suitable electrical properties of nanomaterials such as metal nanoparticles and carbon nanomaterials. For instance, the sensitivity of nanomaterial based non-enzymatic glucose sensors was found to be higher than conventional enzymatic glucose sensors (Piao et al., 2014). Since the electrochemical detection performance is influenced by properties of the electrode surface as well as the utilized electrochemical techniques, nanomaterials are applied in electrochemical detection mainly as electrode material or electrode modifiers (Chen et al. 2012; Ricci et al. 2003; Manea 2014) . In biodetection, nanomaterials are also sometimes used as labels for antibodies and DNA (Kumar et al., 2013; Leng et al., 2011; Zhang et al., 2009).

The motivation of using carbon and carbon based materials as electrodes in electrochemical detection stems from numerous advantages such as low cost, easy preparation, reproducibility, low ohmic resistance, low background current and stability. However, these early carbon-based electrodes/sensors are limited in terms of detection limits as they lack the surface architecture needed for better sensitivity (Grieshaber et al., 2008; Manea, 2014; Tiwari, et al., 2016). This limitation can be possibly addressed using carbon nanomaterials as electrode modifiers as they offer various types of surface architectures with very high surface area.

Carbon nanomaterials (CNM) such as carbon nanotubes and graphene/reduced graphene oxide are used in electrochemical sensors as previous studies have reported that CNM-modified electrode exhibits better electrochemical behaviour compared to the bare electrodes. For instance, studies using carboxylated single-walled carbon nanotubes (SWNTs) showed stronger electrochemical activity to uric acid compared to the bare gold electrode and raw SWNT modified electrode. This is due to the increment of active sites on the electrode surface by the assembled carboxylated SWNTs (Gao et al., 2012). Additionally, a nanocomposite containing CNTs and CaO may produce a more conductive metal oxide material that can be used for electrode modification for electrochemical sensing purpose. Furthermore, hybrids of reduced graphene oxide with CNT (RGO-CNT) and silver nanoparticles (RGO-Ag) are expected to increase the electrochemical performance of screen printed carbon electrodes.

1.2 Scopes of Study

In this work the CNT nanocomposites were prepared using Ni, Co and Fe catalysts supported on calcium oxide (CaO) or quicklime derived from carbonate stones. The catalysts were denoted as either Ni/CaO, Co/CaO, Fe/CaO depending on the metal used. Synthesis of carbon nanotubes-quicklime nanocomposite (CQNs) were conducted via CVD method using hexane as carbon source. The characterization of the synthesized CQNs was done using analytical techniques such as X-ray diffraction (XRD) for identification of the phase present, morphology was determined by field emission scanning electron microscopy (FESEM) and transmission electron microscopy (TEM), thermogravimetric analysis (TGA) was used to investigate thermal decomposition of the samples, nitrogen adsorption-desorption for the determination of BET surface area and porosity, while Raman spectroscopy was used to assess graphitization of the samples.

Synthesis of reduced graphene oxide (RGO) used in RGO-CQN were carried out using improved Hummer's method followed by reduction using hydrazine. The formation of RGO-CNT nanocomposites was done using sonication of RGO and CNT-quicklime nanocomposites. Synthesis of RGO-Ag was conducted via reduction of GO and silver salts using hydrazine and sodium citrate. Addiotnal characterizations of RGO-Ag included UV-Visible spectroscopy (UV-Vis), Fourier transform infra-red spectroscopy (FTIR).and carbon, hydrogen, nitrogen (CHN) analysis

Modification of the screen printed carbon electrodes (SPCE) was conducted using drop casting method. Evaluation of electrochemical performance of nanomaterial modified-SPCE were conducted using cyclic voltammetry in potassium ferricyanide solution.

1.3 Problem Statement

Nanomaterials such as CNTs have exceptional qualities that make them attractive for numerous applications. However, the price of nanomaterials are quite expensive because they require tedious purification steps to obtain the high purity material. It is therefore worthwhile to find cost cutting measures that will make these carbon nanomaterials more economical. Additionally, nanomaterials have been applied in electrochemical detection as electrode modifier in order to increase the electrochemical performance of the electrode. On-site detection of heavy metals usually achieved using portable devices with disposable electrodes such as the screen printed electrode (SPE). However, screen printed electrode usually suffers from the non-electroactive component of the in formulation. Addition of carbon nanomaterials as electrode modifiers may be able to increase the electrode performance of SPE.

This work explores the use of quicklime, CaO as a catalyst support cum composite component for the chemical vapour deposition synthesis of CNTs using Ni, Co, Fe catalysts. These catalysts were chosen as theynare highly soluble in carbon. The combination of these two materials will produce a CNT-quicklime nanocomposite that is conductive and will be possible for numerous applications where CNT and CaO plays a major part. The final product does not require any purification steps and remaining catalyst particles can actually be made use of instead of being eliminated.

The use of carbon nanomaterials such as CNT and graphene are usually limited due to agglomeration. In this work, RGO-CNT-quicklime composites and RGO-silver nanoparticle hybrids were investigated to increase the electrochemical performance of screen printed carbon electrodes (SPCE). The combination of both RGO and CNTs are mainly to prevent aggregation of both nanocarbon materials and to improve the electrochemical properties of the CQNs. In addition silver nanoparticles are also attractive for use as electrode modifiers for electrochemical detection and RGO-silver nanoparticle hybrids are also studied in this work. It is anticipated that usage of both CQNs, RGO-CQNs and RGO-Ag nanocomposites will be able to enhance the electrochemical performance of SPCE better than commercially available CNTs.

1.4 Hypothesis

A nanocomposite containing carbon nanotubes (CNTs) and quicklime will be an economical material to use for screen printed electrode modification. CaO can be obtained from a natural source such as carbonate stones. Using CaO as the catalyst support will enable easier purification of the CNTs as the CaO can be easily washed and removed using mild acid if the need arises. CNT-quicklime nanocomposites containing Ni, Co and Fe catalysts should give improved electrochemical response compared to commercially available CNTs when used as electrode modifier due high material graphitization. Additionally, reduced graphene oxide (RGO)-carbon nanotubes nanocomposite should give increased electrochemical response compared to carbon nanomaterials alone due to the prevention of agglomeration of both carbon nanomaterials. A hybrid consisting of RGO and silver nanoparticles may give improved electrochemical response due to the high electrical conductivity of both nanomaterials. Preparation of RGO-silver nanoparticle hybrid using combination of hydrazine and sodium citrate presents a fast and efficient approach compared to using other reductant and reducing techniques.

1.5 Objectives of Study

- To synthesize CNT-quicklime nanocomposites via CVD of hexane to study the effect of catalyst composition and CVD temperature on the properties of the CNT-quicklime nanocomposites.
- To synthesize RGO-Ag using reduction of silver salts using hydrazine and sodium citrate as stabilizer. Characterization of RGO-Ag to study the effect of sodium citrate concentration and silver concentration on the morphology of the resulting AgNPs grown on the RGO.
- To study effects of using the synthesized nanomaterials as modifiers of screen printed electrode via cyclic voltammetry.
- To compare the effects of the nanocomposites on the SPCE performance with RGO and commercially available CNTs.

1.6 Significance of Study

This study presents a novel nanocomposite of CNT-quicklime where the CaO catalyst support is from natural source which is carbonate stones. This study also offers a unique combination of RGO-CNT-quicklime nanocomposites for screen printed electrode modification. Apart from that, this work also presents the fast and efficient synthesis of RGO-AgNPs using a combination of hydrazine and sodium citrate with systematic optimization steps.

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