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PHOTOELECTROCHEMICAL SENSOR BASED ON MODIFIED CADMIUM SULFIDE NANOMATERIALS FOR COPPER (II) IONS DETECTION

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By

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Chair : Associate Professor Janet Lim Hong Ngee, PhD
Faculty : Science

Discovering the distinctive photophysical properties of semiconductor nanomaterials has made these a popular subject in recent advances in nanotechnology-related analytical methods. Semiconductors are well-known materials that have been widely used in photovoltaic devices such as optical sensors and bioimaging, and dye-sensitized solar cells (DSSCs), as well as for light-emitting diodes (LEDs). The use of a narrow-bandgap semiconductor such as cadmium sulfide nanoparticles (CdS NPs) in the photoelectrochemical (PEC) sensor of chemicals and biological molecules plays a key role as a photosensitizer and promotes some specific advantages in light-harvesting media. Their size-controlled optical and electrical properties make nanomaterials fascinating and promising materials for a variety of nanoscale photovoltaic devices. Moreover, charge injection from the narrow bandgap to the adjacent material leads to efficient charge separation and prolongs the electron lifetime by the elimination of the charge carrier recombination probability. In this regard, a single photon enables the production of multiple photogenerated charge carriers in CdS NPs, which subsequently boosts the effectiveness of the photovoltaic devices. In particular, this thesis highlights the recent emerging PEC detection based on CdS NPs, specifically related to the interactions of CdS NPs with target analytes of copper ions (Cu^{2+}). The investigation and justification of different CdS nanocomposites were discussed in terms of different structural morphologies, and its impact on sensitivity and selectivity towards the targeted Cu^{2+} ions. Thus, it eventually provides a significant insight in achieving real-world applications of CdS-based PEC sensing.

In the first studies, the nanospherical-like morphology of CdS with a narrow diameter distribution of about 350–400 nm was being employed and assembled with a transparent ultrathin reduced graphene oxide (rGO) layer. The nanostructured CdS adhered securely to a continuous network of rGO that also

acted as an avenue to facilitate the transfer of electrons from the conduction band (CB) of CdS. The CdS-rGO photoelectrode response for Cu^{2+} ion detection had a linear range of 0.5–120 μM, with a limit of detection (LoD) of 16 nM. The low LoD demonstrated the favourable structure of CdS-rGO as photoactive materials in PEC sensing platform.

In the second studies, the smaller particle diameters in an average of 25−30 nm of nanospherical CdS was obtained. The hydrothermal synthesis of CdS NPs were decorated with gold quantum dots (Au QDs) via stepwise *in situ* approaches, along with notable PEC performance. The introduction of Au which induced a plasmonic effect on photoactive materials like CdS semiconductors has prompted an intensive interest in PEC sensing applications. The hybrid structure of CdS-Au resulted in the amplification of the photocurrent signal because of the enhanced absorption of photon-generated photoelectron on the CdS. Therefore, it contributed to a sensitive Cu^{2+} ions detector with the lowest LoD of 6.73 nM in a linear range of 0.5−120 nM.

In the third studies, huge efforts have been dedicated to intensifying the PEC performance by modifying the morphology and structure of CdS. One-dimensional (1D) nanostructure (e.g. nanotubes, nanorods, nanofiber and nanowire) of CdS were found to have a practical and substantial potential due to its specific directionality for the transportation of charge carrier, thus decreasing the probability of the recombination of charge carrier. In this regards, the 1D nanorods (NRs) structure of CdS was prepared and the outcomes consistently portray a much better PEC performance than the other counterpart particulate nanostructure. A multi-functional hybrid nanostructure of CdS NRs with Au NPs and graphene quantum dots (GQDs) has been successfully designed. The calculated LoD was 2.27 nM in a range of 0.1-290 nM. A clear trend can be observed based on the obtained LoD from all the three studies, and ultimately proven that the structure, particle size and the nanocomposite materials- based CdS could greatly influence the PEC sensing performance of Cu^{2+} ions.

It has been a pressing need to develop a new materials for simultaneous detection and removal of Cu^{2+} ions from water sources, due to its acute and chronic effect on human health upon exposure to excessive copper. Thus, in the final studies, a ternary hybrid of cellulose acetate (CA) with CdS and methylene blue (MB) in a bead composition was synthesized and investigated as a photosensor-adsorbent of Cu^{2+} ions. The PEC detection of Cu^{2+} ions possessed a lower LoD of 16.9 nM and a notable removal efficiency of 96.3% in the linear range of 0.1-290 nM.

Conclusively, these research have given rise to a neoteric finding and provided an important leap in the employment of CdS as potential semiconductor materials in PEC sensing applications. Even though, only a few CdS-based products that have successfully penetrated the market, but the thorough study and investigation of CdS- based nanocomposite in this thesis can eventually disclose its real potential. Ultimately, it may become a kick-start to researchers and innovators to come up with new CdS-based photosensor device.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

SENSOR FOTOELEKTROKIMIA BERASASKAN KADMIUM SULFIDA NANOBAHAN YANG DIUBAH SUAI UNTUK PENGESANAN ION KUPRUM

Oleh

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Penemuan ciri-ciri foto fizikal tersendiri bagi semikonduktor nanobahan telah menjadikan subjek ini terkenali dalam kemajuan nanoteknologi terbaru yang berkait rapat dengan kaedah analisis. Semikonduktor adalah bahan yang digunakan secara meluas dalam peranti fotovoltan seperti sensor optik dan bio-pengimejan, *sel suria tersintesis warna* (DSSC), dan juga *diod pancaran cahaya* (LED). Penggunaan semikonduktor yang mempunyai jurang jalur sempit seperti *kadmium sulfida nanozarah* (CdS NPs) dalam sensor *fotoelektrokimia* (PEC) bagi molekul kimia dan biologi memainkan peranan penting sebagai pefotopeka dan menggalakkan beberapa kelebihan tertentu dalam media pungutan cahaya. Saiz nanobahan yang mampu mengawal ciri-ciri optik dan elektrik telah menjadikannya menarik dan berpotensi untuk digunakan dalam pelbagai peranti fotovoltan yang berskala-nano. Tambahan pula, penyuntikan cas daripada jurang jalur sempit ke bahan berdekatan telah menyebabkan pemisahan cas yang cekap dan memanjangkan jangka hayat elektron dengan penghapusan kebarangkalian penggabungan pembawa cas. Dalam hal ini, foton tunggal yang membenarkan pengeluaran berbilang cas dari foto-terjana di dalam CdS NPs telah meningkatkan keberkesanan peranti fotovoltan. Khususnya, thesis ini menekankan pengesanan PEC terkini berasaskan CdS NPs yang secara amnya berkaitan dengan interaksi CdS NPs dengan analit sasaran iaitu *ion kuprum* (Cu^{2+}). Penyiasatan dan justifikasi bagi nanokomposit CdS yang berbeza akan dibincangkan dari segi struktur morfologi CdS, dan kesannya terhadap kepekaan dan pemilihan terhadap ion Cu^{2+} yang disasarkan. Akhirnya, ia akan memberikan pandangan yang penting dalam penghasilan peranti pengesanan PEC untuk penggunaan dunia yang berasaskan CdS.

Dalam kajian pertama, morfologi berbentuk seperti nanosfera dengan taburan diameter yang sempit kira-kira 350-400 nm telah digunakan dan dihimpunkan dengan lapisan lut sinar ultranipis *grafin* (rGO). Struktur nano CdS telah melekat dengan kuat pada rangkaian berterusan rGO yang juga bertindak sebagai saluran untuk memudahkan pemindahan elektron dari *jalur pengaliran* (CB) CdS. Gerak balas fotoelektrod CdS-rGO bagi pengesanan ion Cu^{2+} mempunyai julat linear 0.5-120 μM, dengan *had pengesanan* (LoD) sebanyak 16 nM. LoD rendah yang diperolehi menunjukkan bahawa struktur CdS-rGO yang baik sebagai bahan fotoaktif dalam platform pengesanan PEC.

Dalam kajian kedua, diameter zarah bagi nanosfera CdS yang kecil dalam purata 25-30 nm telah diperolehi. Pensintesisan hidraterma CdS NPs yang dihiasi dengan *titik kuantum emas* (Au QDs) melalui pendekatan berperingkat *in-situ*, menunjukkan prestasi PEC yang ketara. Penggunaan Au yang mencetuskan kesan plasmonik pada bahan fotoaktif seperti semikonduktor CdS telah mendorong minat yang bersungguh-sungguh terhadap penggunaannya di dalam pengesanan PEC. Struktur kacukan CdS-Au menghasilkan isyarat fotoarus yang kuat kerana peningkatan penyerapan foton yang menjana fotoelektron pada CdS. Dengan itu, ia menyumbang kepada pengesanan peka bagi ion kuprum dengan LoD terendah sebanyak 6.73 nM dalam julat linear 0.5-120 nM.

Dalam kajian ketiga, usaha yang bersungguh-sungguh telah didedikasikan untuk meningkatkan prestasi PEC dengan mengubah morfologi dan struktur CdS. Satu dimensi (1D) struktur nano (contoh: nanotiub, nanorod, nanofiber dan nanowayar) CdS didapati bersesuaian dan mempunyai potensi yang besar kerana kaedah penghantaran casnya yang mempunya arah yang khusus, seterusnya menurunkan kebarangkalian untuk penggabungan semula pembawa cas. Dalam hal ini, struktur 1D *nanorod* (NRs) CdS telah dihasilkan dan ia menunjukkan prestasi PEC yang konsisten dan lebih baik daripada struktur nano partical yang lain. Kacukan pelbagai fungsian struktur nano CdS NRs dengan Au NPs dan *titik kuantum grafin* (GQDs) telah berjaya direka. LoD yang telah dikira adalah 2.27 nM dalam lingkungan 0.1-290 nM. Perkembangan yang jelas dapat diperhatikan berdasarkan LoD yang diperolehi dari ketiga-tiga kajian, dan akhirnya membuktikan bahawa struktur, saiz zarah dan bahan nanokomposit yang berasaskan CdS sangat mempengaruhi prestasi pengesanan PEC bagi ion Cu^{2+}.

Desakan untuk pengeluaran bahan baru bagi pengesanan dan penyingkiran ion Cu^{2+} secara serentak dari sumber air adalah semakin meningkat. Ini disebabkan oleh kesan buruk dan kronik terhadap kesihatan manusia apabila terdedah kepada kuprum yang berlebihan. Oleh itu, dalam kajian terakhir, kacukan pertigaan *selulosa asetat* (CA) dengan CdS dan *metilina biru* (MB) dalam komposisi manik telah disintesis dan disiasat sebagai foto pengesanan-zat penjerapan ion Cu^{2+}. Pengesanan PEC bagi ion Cu^{2+} mempunyai LoD yang rendah sebanyak 16.9 nM dan kecekapan penyingkiran ketara sebanyak 96.3% dalam julat linear 0.1-290 nM.

Secara keseluruhannya, penyelidikan ini telah menimbulkan penemuan neoterik dan memberikan lonjakan penting dalam pengajian berkaitan CdS sebagai bahan semikonduktor yang berpotensi dalam penggunaan pengesanan PEC. Walaupun hanya beberapa produk berasaskan CdS yang berjaya menembusi pasaran, tetapi kajian menyeluruh terhadap nanokomposit CdS yang dilaksanakan di dalam tesis ini mampu mendedahkan potensi sebenarnya. Akhirnya, ia mampu menjadi titik permulaan bagi penyelidik dan inovator untuk menghasilkan peranti foto pengesanan baharu berasaskan CdS.

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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

0D	Zero-dimensional
1D	One-dimensional
1LO	First- longitudinal optical
2LO	Second- longitudinal optical
3LO	Third-order longitudinal optical
τ_e	Electron lifetime
λ-Exo	λ-Exonuclease
Au QDs	Gold quantum dots
AA	Ascorbic acid
AACVD	Assisted chemical vapor deposition
AAP	Ascorbic acid 2-phosphate
AAS	Atomic absorption spectroscopy
Ab	Antibodies
AChe	Acetylthiocholine esterase
Ag	Antigen
ATC	Hydrolyzing acetylthiocholine
ATP	Adenosine triphosphate
Av-ALP	Avidin-ALP
BET	Brunauer, Emmett, and Teller
$C_4H_{11}N$	N-butylamine
$C_{16}H_{18}ClN_3S$	Methylene blue
CA	Cellulose acetate
CA/CdS/MB	Cellulose acetate/cadmium sulfide/methylene blue
CB	Conduction band
CdS-Au	Cadmium sulfide/gold
CdS/Au/GQDs	Cadmium sulfide/gold/graphene quantum dots
$Cd(CH_3COO)_2{\cdot}2H_2O$	Cadmium acetate dihydrate
CdS-rGO	Cadmium sulfide/reduce graphene oxide
CdS:Mn-NH_2	Amino-functionalized cds:Mn
CdS NPs	Cadmium sulfide nanoparticles
CH_3OH	Methanol
$(CH_3)_2CO$,	Acetone

$CH_3CH_2OH_2$	Ethanol
$(CH_3)_2SO$	Dimethyl sulfoxide
CH_4N_2S	Thiourea
Cu^{2+}	Copper ions
$CuSO_4.5H_2O$	Copper(II) sulfate pentahydrate
CV	Cyclic voltammetry
DPV	Differential pulse voltammograms
DSN	Duplex-specific nuclease
DSSCs	Dye-sensitized solar cells
dUTP-biotin	Biotinylated dutp
ECL	Electrochemiluminescence
EDX	Energy dispersive X-ray
EET	Exciton energy transfer
EIS	Electrochemical impedance spectra
EPI	Exciton-plasmon interaction
ESR	Equivalent series resistance
FESEM	Field emission scanning electron microscope
FIA	Flow injection analysis
FT-IR	Fourier transform infrared
FTO	Fluorine-doped tin oxide
GCE	Glassy carbon electrode
$g-C_3N_4$	Graphitic carbon nitride
G-CdS	Graphene-cds
GDH	Glucose dehydrogenase
GNP	Graphene nanoplatelet
GO	Graphene oxide
GO_x	Glucose oxidase
GQDs	Graphene quantum dots
GSH	Glutathione
GSs	Graphene sheets
hDNA	Hairpin DNA
IAA	Indole-3-acetic acid
ITO	Indium-doped tin oxide

$K_3[Fe(CN)_6]$	Potassium ferricyanide
$K_4[Fe(CN)_6]$	Potassium ferrocyanide
KCl	Potassium chloride
LEDs	Light-emitting diodes
LDH	Lactase dehydrogenase
LoD	Limit of detection
LSV	Linear sweep voltammograms
MB	Methylene blue
miRNAs	Microrna-21
$Na_3C_6H_5O_7$	Trisodium citrate
NADH	Dihydronicotinamide adenine dinucleotide
NCs	Nanoclusters
NiCd	Nickel-cadmium
NRs	Nanorods
PBA	1-pyrene butyric acid
PEC	Photoelectrochemical
PL	Photoluminescence
PPD	P-phenylenediamine
PSA	Prostate-specific antigen
PVDF	Poly(vinylidene fluoride)
QD	Quantum dot
Rh123	Rhodamine 123
Rct	Charge transfer resistance
RCA	Rolling circle amplification
RET	Resonance energy transfer
rGO	Reduced graphene oxide
RO	Reverse osmosis
RSD	Relative standard deviations
SAED	Selected area in the electron diffraction
SCCA	Squamous cell carcinoma antigen
sDNA	Short DNA
SPR	Surface plasmon resonance
ssDNA	Single-stranded DNA

TEA	Triethanolamine
TET	Tetracycline
T-Hg^{2+}-T	Thymine-Hg^{2+}-thymine
TC	Thiocholine
TSEs	Transmissible spongiform encephalopathies
TEM	Transmission electron microscopy
tDNA	Target DNA
VB	Valance band
V_{oc}	Open-circuit photovoltage
WHO	World health organization
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction

CHAPTER 1

INTRODUCTION

1.1 Copper Contamination

Copper is a precious metal which is exceptional in conducting electricity and heat. It is found naturally in metallic form and it has been in use since millennia ago in alloys, tools, coins, food and beverage containers, automobile brake pads, electrical wiring and electroplating. Copper also appears as one of the essential elements that are required by the human and living organism. Only a tiny amount of copper is needed for living organism included human beings, which is around 20 µg/L for the formation of haemoglobin and haemocyanin, the oxygen-transporting pigments in the blood of vertebrates and shellfish (Solomon, 2009). However, excessive doses of copper (over 20 µg/L) can be toxic and affect the environment and humans unfavourably.

As one of the advanced metals of commerce, it is not surprising that copper found significantly in the environment is due to human activity, over than what might be found naturally. The copper mining can produce copper-rich dust which is then spread by the wind in the vicinity of the copper mine site. The ores obtained from the excavation activities are sulfide minerals, then oxidize in the air to form sulfates, thus producing sulfuric acid, which renders the highly soluble copper in the minerals (Baba *et al.*, 2012). Besides, the manufacturing companies which involve extensively with copper such as in the production of metal, electrical appliances, pesticide and other products consisting copper, normally released the contaminated water into the drainage system, which hence flows out to the rivers, streams and other water bodies. The pesticide called “Bordeaux mix” which is rich with the copper compound is usually sprayed on the fruits or vegetables to control various pests. Hence, more copper has been introduced to the environment and may badly affect human health upon consuming unwashed fruits and vegetables sprayed with such pesticide. Moreover, in the plumbing system, the pipe made up of copper metal will be dissolved gradually and thus generating the metal into the water supply.

All of the aforementioned source of copper pollution in the water will definitely give a negative impact on the environment and human health. In human, low level of copper is needed to maintain good health, and upon exceeding the permitted level, health will be affected by various chronic diseases such as diarrhea, chest pains, nausea, and irritation of the respiratory tract(Jaishankar *et al.*, 2014). The use of copper cookware in boiling milk will cause liver cirrhosis in children due to the toxicity of copper. Additionally, the illness such as Wilson’s disease might occur upon superfluous copper consumption, which then leads to damage to the brain. This illness is an inherited disease that hinders the excretion of copper into the bile by the liver (Purchase, 2013). Moreover, a very high copper level can be harmful to the kidney and liver and may lead to death.

The adverse health effects due to long-term exposure to copper have become world concerns, and many different strategies have been proposed to prevent copper pollution in the industries. However, the prevention strategy to substitute the copper with other metals is found to be irrelevant in the copper metal manufacturing industries. This is because this metal is comparably lower in price, excellent conductivity behaviour, and naturally and readily available compared to other metals. In regards to this matter, monitoring of copper in water bodies is necessary to ensure that copper released into the drainage system is within the acceptable level. Therefore, it is an urgent need to develop a monitoring or sensor device that is highly sensitive to copper ions.

1.2 Photoelectrochemical (PEC) Sensor

The evolution of photoelectrochemistry from the electrochemical method has grown vigorously since the past few years, involving investigation on the effect of light on the photoelectrode, as well as the interaction between solar energy and electrical energy. There are two basic principles involved in PEC process: (1) the oxidation and reduction reactions between the electrochemically active species in the electrolyte, and (2) the excitation of photoactive materials in the electrode/electrolyte interface during irradiation of light. These principles have broadly been utilized in the research for photocatalyst, photovoltaic and solar cell. By applying the same principle, the PEC sensing for detecting chemical or biological analytes has evolved actively over time by combining the PEC technique with biochemical analysis, in which light served as an excitation source for photosensitive materials, and subsequently generating the current as signal readout (Ibrahim *et al.*, 2018).

Unlike the optical detection technique which suffers from signal attenuation, complex and high-cost detection system, very temperature-sensitive, as well as require precise installation procedures (Ahuja & Parande, 2012), the execution of electronic detection makes PEC measurement and instrument simpler, cost-effective, strong signal and enabling detection in the complex samples. In addition, the total separation of the excitation source (light) and the detection signal (electric) in the PEC system have led to the advantages of a negligible background signal and ultra-sensitive sensor. Nevertheless, the regular photoactive species used in the existing research of PEC sensor is found to be restricted due to low photo-conversion efficiency which is related to the wide band gap energy of semiconductor, susceptibilities to photo-bleaching and unclear signalling mechanism. The aforementioned drawback of the PEC sensor has created a huge obstacle to realizing these principles in the practical and real-world application. Therefore, the selection of photoactive semiconductor and the thorough reorganization of complex PEC strategies is compulsory to attract reader interest in understanding the inherent principle more systematically, and thus exploit a new detecting mechanism (Zang *et al.*, 2017).

1.3 Cadmium Sulfide (CdS)

The simple composition of CdS by pairing one cadmium atom with one sulfur atom was emerged naturally both as a pure mineral. The cadmium and sulfur atom are frequently present as an impurity in zinc ores. In 1817, Stromeyer and Hermann, a German chemist, was credited for extracting CdS from zinc carbonate (calamine), which then heated to produce pure cadmium (Zang *et al.*, 2017). When first CdS is found in the relatively rare pure form in nature, it consists of two different crystal structures. The hexagonal crystal structure of CdS named Greenockite was found on Lord Greenock's land in 1840, which reflects the internal mineral composition of CdS. Meanwhile, the second structure called a Hawleyite was named after the Canadian Mineralogist, James Hawley, who was the first person discovered this crystal structure. The Hawleyite is a cubic form of CdS with a similar structure to zinc sulfide, which sometimes called as a zinc blende structure. Soon after the discovery of CdS, it has drawn much attention among the artists due to the strong yellowish color of CdS. The vibrant yellow pigment of CdS is usually used in painting as the earlier colors for the range of yellow to red pigment.

To date, cadmium sulfide still becomes a main source of metal, which commonly used in batteries such as rechargeable nickel-cadmium (NiCd) batteries to helps power out our mobile lifestyle (Lankey & McMichael, 2000). Moreover, CdS is widely been utilized as semiconductor materials for numerous form of application. CdS have a bandgap, which is the intrinsic properties for all the semiconductor materials. The bandgap of CdS enables the absorbed photon to promote the electron across this gap, under light illumination and thus allow flowing or moving of electron to produce conductivity. Exposing CdS to the light source will make use of this abundant energy (light energy) to facilitate the electron to cross to the conduction layer, then making CdS as an effective photoresistor. Furthermore, it can also be utilized as photovoltaic cells for solar panels by doping it with other semiconductor or noble metals.

1.4 Cadmium Sulfide- based Nanomaterials

Many endeavours have been focused on enhancing the PEC performance of CdS by modifying its structure (Xing *et al.*, 2013b) and hybridizing it with other metal or semiconductor materials (Huo *et al.*, 2015), carbon-based materials (Ibahim *et al.*, 2016; Wang *et al.*, 2015), and noble metals (Ibrahim *et al.*, 2016a). A three-dimensional hexagonal CdS nanostructure had been fabricated by (Li *et al.*, 2013b), and a notable enhanced PEC performance of hexagonal CdS was obviously found upon illumination if compared to the cubic CdS NPs. This was due to the large surface area and high charge transportability that resulted from the large band gap of hexagonal CdS compared to that of cubic CdS (Li *et al.*, 2013b), which revealed its predominant advantages in PEC applications. Additionally, much research has been devoted to comparing hexagonal and cubic CdS (Khatamian *et al.*, 2014; Li *et al.*, 2016; Matsumura *et al.*, 1985), and the results have suggested that the high crystallinity of hexagonal CdS will lead to fewer surface defects in the crystals, thus efficiently enhancing the charge transport and separation. The hydrophilic CdS nanorods prepared by (Bao *et al.*,

2015) displayed a broader light absorption and evident photocurrent intensity amplification at -0.2 V vs. Ag/AgCl, as compared to CdS nanoparticles, which used a spherical structure. Hence, the great PEC performance illustrated by CdS nanorods has become a potential candidate for PEC application.

1.5 Problem Statements

The agglomeration of NPs has become a common problem faced by nanomaterials researchers. Despite its excellent physical and chemical properties manifested by these nano-sized materials, the formation of cluster NPs becoming a major challenge involves in synthesizing of these nanomaterials due to its higher surface area to volume ratio. The large surface area of NPs may result in large surface energy. To diminish or minimize the surface energy, the particles tend to agglomerate uncontrollably due to van der Waals interaction between the particles (Saravanan *et al.*, 2011). As a consequence, the agglomeration of NPs inhibits the real potential of NPs and subsequently tend to create problems such as poor electrochemical performance due to the difficulty for electron mobility and the existence of charge recombination. In this regards, the agglomerations of the NPs can be avoided by stabilizing them electrostatically, modifying with capping agents, and covering with inorganic shell or organic ligand. Nevertheless, encapsulating the surface of NPs to hinder the growing rate of NPs will lead to the presence of foreign species within it. Therefore, the additional fabrication process is needed, and it will consume much time for synthesis and post-treatment of NPs especially for the removal of by-product.

Additionally, there are two mechanisms involved in PEC sensor which are based on the reductive property of the photoelectron or oxidative capacity of the photogenerated hole (Ibrahim *et al.*, 2018). Yet, it is still a great challenge for PEC sensors to distinguish the individual targeted species without the auxiliary. In order to achieve more accurate determination of target species, certain biomolecules such as DNA, enzyme, antibody, and aptamer were incorporated as auxillary means on the PEC platform. Even though it manifested a good selectivity with the assistance of those biomarkers, the sophisticated design process, harsh storage conditions and high cost have remarkably restricted its practical applications. Hence, it is substantial to execute the selective detections with label-free PEC sensors.

CdS are II and IV semiconductor with a bandgap of ~2.4 eV, promoting an excellent visible light absorption under solar irradiation. Unfortunately, the problem of photocorrosion and rapid charge recombination of CdS in aqueous media have restricted its practical application in light-driven reaction. Therefore, to protect the CdS from photocorrosion, coupling CdS with other materials such as metal or semiconductor materials, carbon-based materials, and noble metals is an effective method to inhibit the photocorrosion and recombination of charge carries of CdS (Ibrahim *et al.*, 2018). Besides, the addition of hole scavenger to the electrolyte such as triethanolamine (TEA) and ascorbic acid is another practical approach that can suppress the aforementioned limiting factor through kinetic competition (Dotan *et al.*, 2011).

Besides, there are numerous researches have reported on the detection of Cu^{2+} ions in contaminated water, but seldom was reported on simultaneous detection and removal of that hazardous metal. Vulnerable from superfluous Cu^{2+} ions in drinking water (guidelines value of copper ions in drinking water standardized by the World Health Organization (WHO) was limited to ~20 μM) or other environmental sources can cause detrimental effect to health and ecosystem. Therefore, there was a pressing need to come out with new materials for its simultaneous detection and removal from the water sources. The dual-functional material is not only able to monitor the level of Cu^{2+} ions, but it also can diminish the number of metal ions found in the water samples.

Despite the fact that the utilization of CdS with other nanomaterials reveals promising results in promoting their overall physiochemical properties, but the understanding in the structural morphology of CdS and mechanism in PEC reaction is crucial. Therefore, it is important to investigate the correlation between the PEC performances with the structural evolution of CdS changing from zero-dimensional (0D, nanosperical) to one-dimensional (1D, nanorod) architecture.

1.6 Scope of Research

In this research, we aim to investigate the effect of manipulating the shape of CdS nanocrystals due to the importance of the morphology and texture of the materials in determining the PEC properties of CdS. It has remained as an important goal of modern materials science to conduct a comprehensive study on the influence of structural morphology towards the PEC performance such as the sensitivity, selectivity and limit of detection (LoD). Moreover, the CdS synthesized via aerosol-assisted chemical vapor deposition (AACVD), hydro- and solvothermal approaches will be analysed to fabricate different CdS nanomaterials such as cadmium sulfide/reduce graphene oxide (CdS-rGO) nanocomposite, cadmium sulfide/gold (CdS-Au) nanocomposite, cadmium sulfide/gold/graphene quantum dots (CdS/Au/GQDs) nanocomposite and cellulose acetate/cadmium sulfide/methylene blue (CA/CdS/MB) nanocomposite, which will be employed as photosensitive species in visible-light induced PEC sensors of copper ions. The nanocomposite materials based on CdS will enable the inhibition of photo-corrosion as compared with pure CdS nanoparticles (NPs), and subsequently displayed an enhanced photo-to-current conversion efficiency.

In order to investigate the workability of the as-prepared CdS nanomaterials, the study on this precious materials will not be restricted to monitoring the copper level by the proposed PEC sensor technique but also removing this harmful metal from the environment especially in water bodies. The dual-function CdS nanocomposite for PEC detection and removal of copper will be incorporated with CA, which act as adsorbent materials for copper ions (Cu^{2+}). Additionally, the performance of the as-synthesized photoactive and bioadsorbent materials will also be evaluated by parameter optimization, performance comparison with other electrochemical detection methods and real-sample study for clarification and justification of the proof-of-concept study.

1.7 Research Objectives

This research is to fabricate a functional PEC sensor platform that relies on the nanocomposite of CdS. The primary objective of this thesis is to investigate the practicality of CdS-based nanomaterials in PEC detection as well as to remove Cu^{2+} ions from water bodies. Remarkable attention was devoted to addressing the current problems related to agglomeration of NPs, label-free sensor, enhancement in the PEC performance, and simultaneous detection and removal of Cu^{2+} ions device. The specific objectives of the study are outlined below:

i. To construct a multi-functional hybrid nanomaterial with graphene derivatives and Au for enhancement in PEC performance of CdS modified electrode
ii. To investigate the practical applicability and sensitivity of the proposed CdS-based photoelectrode in the real sample application.
iii. To investigate the structural evolution of CdS from nanospherical to nanorod morphology in the overall performance of PEC study.
iv. To analyse the performance of CdS with cellulose acetate as bioadsorbent materials and methylene blue for dual-functional based sensor-adsorbent materials.

1.8 Thesis Outline

In Chapter 1, brief introduction on copper contamination, PEC sensor and CdS nanomaterials are given, problem statements and the main objective of the thesis. A comprehensive literature review on visible-light induced photoelectrochemical sensors based on CdS nanoparticles is explained in Chapter 2.

Chapter 3 covers the experimental works used for the synthesis of CdS-rGO photoelectrode via AACVD and dip-coated approach. The nanospherical structured CdS was adhered securely to the glass substrate by a continuous network of rGO that also acted as an avenue to intensify the transfer of electrons from the conduction band of CdS. A study on the stability of photocurrent performance between the presence and absence of TEA for scavenging the photogenerated hole was carried out in this chapter.

In Chapter 4, the modification of spherical CdS decorated with Au QDs was conducted. The influence of the amount of Au QDs loaded on the CdS NPs on the PEC performance was evaluated. The femtosecond transient absorption dynamics of the modified photoelectrode was also conducted to investigate the rate transfer of photoexcited electrons.

In Chapter 5, the incorporation of graphene quantum dots and gold on the CdS nanorods was developed to attain an ultra-sensitive PEC sensor device. The simplistic fabrication of CdS/Au/GQDs photoelectrode achieve a remarkable PEC response due to the assembly of precious carbon family of GQDs, which holds a role similar like semiconductor, and also the good distribution of plasmonic Au on the CdS NRs surface, thus contributing to excellent light scattering ability producing hot electron on the CdS NRs. The synergistically interaction of CdS/Au/GQDs enabling smooth transportation of charge carrier to the charge collector and providing a channel to inhibit the charge recombination reaction. To obtain a firm resolution on the selectivity and sensitivity of CdS/Au/GQDs photoelectrode, simultaneous determination of all ions mixture of Cu^{2+}, Ba^{2+}, Co^{2+}, Li^{+}, Ni^{2+}, Mn^{2+}, K^{+}, Zn^{2+}, Na^{2+}, Mg^{2+}, Ag^{+} and Fe^{2+} was feasible via differential pulse voltammetry (DPV).

In Chapter 6, a novel approach in fabricating multi-functional hybrid of CA/CdS/MB in the beads composition was synthesized and investigated as a photosensor-adsorbent for rapid, facile, selective and sensitive detection and adsorption of Cu^{2+} ions. A study of different precursor ratio of CdS and the difference in the morphology obtained was evaluated. The successful application of CA/CdS/MB in this research has provided new insight into the selection as excellent photoactive materials in PEC sensor as well as adsorbent materials for Cu^{2+} ions.

Lastly, Chapter 7 contains the general conclusion and several future recommendations. The list of references cited in this thesis, appendices, biodata of students and a list of publications is listed in the post of Chapter 7.

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BIODATA OF STUDENT

Izwaharyanie Ibrahim is a Ph. D. candidate in the laboratory of Associate Professor Dr Hong Ngee Lim at Universiti Putra Malaysia. Her research interests involve in the advances of photoelectrochemical (PEC) sensor based on nanocomposite of CdS semiconductors for the development of detection systems for chemical and biological analytes. She is interested in the synthesis and development of CdS based nanomaterials, and other functional nanomaterials such as noble metal, graphene derivatives, polymers and dyes. She is also interested in the development of a photo-conversion system such as PEC water splitting, dye-sensitized solar cell (DSSC), photocatalysis and photo-supercapacitor. Before starting her Ph. D., Ibrahim I. has participated in the internship at the Worldwide Water Technologies Sdn Bhd, which involve with the project based on wastewater treatment and purification. She has collaborated with the National Hydraulic Research Institute of Malaysia (NAHRIM) and continue the research on water treatment based on photocatalysis method. She holds a Bachelor of Science degree in industrial chemistry from UPM and actively involved in various co-curricular activities such as ‘Rakan Muda’ and Al-Biruni Club.

LIST OF PUBLICATIONS

Publications

Ibrahim, I.; Lim, H. N.; Zawawi, R. M.; Tajudin, A. A.; Ng, Y. H.; Guo, H.; Huang, N. M., A review on visible-light induced photoelectrochemical sensors based on CdS nanoparticles. *Journal of Materials Chemistry B* **2018**, 6(28), 4551-4568. (Impact Factor: 4.776, Q1 journal)

Lee, S. X.; Lim, H. N.; **Ibrahim, I.;** Jamil, A.; Pandikumar, A.; Huang, N. M., Horseradish peroxidase-labeled silver/reduced graphene oxide thin film-modified screen-printed electrode for detection of carcinoembryonic antigen. *Biosensors and Bioelectronics* **2017**, *89*, 673-680. (Impact Factor: 8.173, Q1 journal)

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Shams, N.; Lim, H.N.; Hajian, R.; Yusof, N.A.; Abdullah, J.; Sulaiman, Y.; **Ibrahim, I.**; Huang, N.M., Electrochemical sensor based on gold nanoparticles/ethylenediamine-reduced graphene oxide for trace determination of fenitrothion in water. *RSC Advances* **2016**, *6*(92), 89430-89439. (Impact Factor: 2.936, Q1 journal)

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Ibrahim, I.; Lim, H. N.; Sharifuddin, S. S.; Yusof, M. A. M,; Huang, N. M.; Pandikumar, A., Preparation of Polypropylene Filter Incorporated with Titanium Dioxide and Reduced Graphene Oxide for Real Water Treatment. *Science of Advanced Materials* **2015**, *7*(8), 1556-1566. (Scopus index, Q2 journal)

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Conferences Attended

Ibrahim. I.; Lim, H. N., Visible-Light-Prompt Photoelectrochemical Sensor of Copper(II) Ions based on CdS Nanorods Modified with Au Nanoparticles and Graphene Quantum Dots. *Symposium on Advanced Materials and Nanotechnology (SAMN2018), Malaysia (15th – 16th August 2018).* Oral presentation.

Ibrahim. I.; Lim, H. N., Photoelectrochemical Sensing Platform of Copper(II) Ions Based Cadmium Sulfide-Reduced Graphene Oxide Modified Photoelectrode. *The 28th Regional Symposium of Malaysian Analytical Sciences (SKAM28), Malaysia (17th – 20th August 2016).* Poster presentation.

Ibrahim. I.; Lim, H. N., Ultrasensitive Photoelectrochemical Sensor Based on Nafion Polymer-Modified Cadmium Sulfide-Gold/Indium Tin Oxide Electrode for Selective Detection of Copper Ions. *International Symposium on Advanced Polymeric Materials 2016 (ISAPM 2016), Malaysia (16th – 19th May 2016).* Oral presentation.

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Ibrahim. I.; Lim, H. N., Reduced graphene oxide/titanium dioxide incorporated polypropylene filter for effective water treatment. *International Symposium on Advanced Polymeric Materials 2014 (ISAPM 2014), Malaysia (14th – 15th May 2014).* Poster presentation.

Patent Pending

1) Lim, H. N. and **Ibrahim. I.** (2018). *Visible Light-Driven Photoelectrochemical Sensor based on CdS-Au Photosensitive Semiconductor.*

2) Lim, H. N. and **Ibrahim. I.** (2018). *Microbeads of Cellulose Acetate Modified Cadmium Sulfide and Graphene Bioadsorbent for Effective Removal And Photoelectrochemical Detection of Cu(II) Ions.*

UNIVERSITI PUTRA MALAYSIA

STATUS CONFIRMATION FOR THESIS / PROJECT REPORT AND COPYRIGHT

ACADEMIC SESSION : ____________________

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