

UNIVERSITI PUTRA MALAYSIA

STRUCTURAL AND OPTICAL PROPERTIES OF HYDROXYLFUNCTIONALIZED GRAPHENE QUANTUM DOTS-BASED THIN FILM AND ITS POTENTIAL SENSING FOR FERRIC ION USING SURFACE PLASMON RESONANCE

NUR AIN ASYIQIN BINTI ANAS

ITMA 2020 17



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By

NUR AIN ASYIQIN BINTI ANAS

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

July 2020

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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July 2020

Chair: Yap Wing Fen, PhDInstitute: Advanced Technology

As a new promising carbonic nanomaterial with a lot of outstanding advantages, graphene quantum dots (GQDs) opened up a new field for the development of excellent sensors. Herein, the preparation of hydroxyl-functionalized graphene quantum dots (HGQDs) based thin film with different materials that are chitosan and cetyltrimethylammonium bromide (CTAB) have been described namelv chitosan/hydroxyl-functionalized graphene quantum dots (Cs/HGQDs) thin film and cetyltrimethylammonium bromide/hydroxyl-functionalized graphene quantum dots (CTAB/HGQDs) thin film. The Cs/HGQDs and CTAB/HGQDs were deposited homogenously using the spin coating technique. The synthesized thin films were then characterized using Fourier transform infrared spectroscopy (FTIR) to confirm the existence of functional groups in the composites such as hydroxyl, carboxyl, and carboxylic acid. From the atomic force microscope (AFM) analysis, the addition of chitosan and CTAB increased the roughness of the thin films. Meanwhile, the optical properties of the thin films were studied using UV-Vis absorption spectroscopy and photoluminescence (PL) spectroscopy. The absorbance peaks of Cs/HGQDs and CTAB/HGQDs thin films can be observed around the wavelength of 270 nm to 300 nm with optical band gap values of 3.80 eV and 4.16 eV, respectively. Moreover, the intensity of PL spectra for both thin films were noticed around the wavelength of 420 nm to 450 nm. The development of optical sensors for heavy metal ions detection has been rapidly growing. However, the current methods suffer limitations which then led to the emergence of an outstanding technique called surface plasmon resonance (SPR) spectroscopy. In this study, the as developed thin films have been incorporated with SPR for the detection of ferric ion (Fe^{3+}) . The sensors produce positive responses upon exposure to Fe³⁺ of various concentration. At lower Fe³⁺ concentration, the CTAB/HGQDs thin film showed higher sensitivity equals to 29.886° ppm⁻¹ compared to Cs/HGQDs with value of 0.114° ppm⁻¹. Subsequently, the Langmuir isotherm model yielded higher binding affinity constant, K for CTAB/HGQDs thin film than Cs/HGQDs

thin film with values of 221.729 ppm⁻¹ and 5.79 ppm⁻¹, respectively. Thus, both thin films show potential for the detection of Fe^{3+} .



Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

SIFAT STRUKTUR DAN OPTIK FILEM NIPIS BERASASKAN TITIK KUANTUM GRAFIN BERFUNGSI HIDROKSIL DAN POTENSINYA BAGI MENGESAN ION FERIK MENGGUNAKAN RESONANS PLASMON PERMUKAAN

Oleh

NUR AIN ASYIQN BINTI ANAS

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Sebagai nanomaterial karbonik baru yang mempunyai harapan dengan banyak kelebihan luar biasa, titik kuantum grafin (GQDs) membuka bidang baru untuk kemajuan sensor yang sangat baik. Di sini, penyediaan filem tipis berasaskan titik kuantum grafin berfungsi hidroksil (HGQDs) dengan bahan berbeza iaitu kitosan dan setiltrimetilammonium bromida (CTAB) telah dinyatakan jaitu filem tipis kitosan/titik kuantum grafin berfungsi hidroksil (Cs/HGQDs) dan filem tipis setiltrimetilammonium bromida/titik kuantum grafin berfungsi hidroksil (CTAB/HGQDs). Cs/HGQDs dan CTAB/HGQDs telah diletakkan secara seragam menggunakan teknik lapisan spin. Filem-filem tipis yang telah disintesis kemudiannya dicirikan menggunakan spektroskopi transformasi Fourier inframerah (FTIR) untuk mengesahkan kewujudan kumpulan berfungsi dalam komposit seperti hidroksil, karboksil, dan asid karbosilik. Dari mikroskopi daya atom (AFM), penambahan kitosan dan CTAB meningkatkan kekasaran filem tipis. Sifat optik filem tipis dikaji menggunakan spektroskopi penyerapan UV-Vis dan spektroskopi fotoluminesens (PL). Puncak penyerapan bagi filem nipis Cs/HGODs dan CTAB/HGODs boleh diperhatikan sekitar panjang gelombang 270 nm hingga 300 nm dengan jurang jalur optik masing-masing bernilai 3.797 eV dan 4.162 eV. Tambahan lagi, keamatan spektrum PL untuk kedua-dua filem tipis dapat diperhatikan sekitar panjang gelombang 420 nm hingga 450 nm. Selanjutnya, filem tipis juga digabungkan dengan sensor optik resonans plasmon permukaan (SPR) bagi pengesanan ion ferik (Fe³⁺). Sensor tersebut menghasilkan tindak balas positif setelah didedahkan kepada Fe³⁺. Pada kepekatan Fe³⁺ yang lebih rendah, filem tipis CTAB/HGQDs menunjukkan kepekaan yang lebih tinggi bersamaan dengan 29.886° ppm⁻¹ berbanding Cs/HGQDs dengan nilai 0.114° ppm⁻¹. Seterusnya, model isoterma Langmuir menghasilkan pemalar tarikan ikatan yang lebih tinggi, K untuk filem tipis CTAB/HGQDs berbanding Cs/HGQDs masing-masing bernilai 221.729 ppm⁻¹ dan 5.79 ppm⁻¹. Oleh itu, kedua-dua filem tipis menunjukkan potensi tinggi untuk mengesan Fe³⁺.

ACKNOWLEDGEMENTS

First and foremost, alhamdulillah, all praises to Allah the Almighty for bestowing me with wellness, persistence, and understanding to accomplish this master project. I am also using this chance to convey my appreciation to everybody who backed me throughout this journey.

I would like to thank my project supervisor, Assoc. Prof. Dr. Yap Wing Fen for his keen interest and dedication including all overwhelming support, guidance, and both direct and indirect lessons that he gave since the very first day of this project. His patience in guiding all his students has motivate us to be better each day. Not to forget, I would like to give my thanks to my co-supervisor, Prof. Dr. Nor Azah binti Yusof for her generous help in my research.

Moreover, I am highly obliged in taking the opportunity to sincerely thanks all my lab mates from Applied Optic Laboratory for their valuable ideas, comments, sharing, support, and helps during my study. A special word to my best friends here, Nurul, Nabilah, Amalini, and Nadiah for their support and time spent with me for these 6 years. I hope that this friendship will lasts forever until Jannah.

I would also like to offer my heartfelt thanks to my husband, Naqi for his great encouragement in my study. Finally, I would like to thank my family especially my parents, Anas bin Osman and Nor Aswa binti Abu. Without their love, prayers, care, and support over the years, none of this would have been possible. To my loveliest siblings, Along, Kak Pija, and Baby, thank you. My family have always been there for me and I am very thankful for everything they have helped me achieve. This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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TABLE OF CONTENTS

	Page
ABSTRACT	i
ABSTRAK	111
ACKNOWLEDGEMENTS	iv
APPROVAL	V
DECLARATION	vii
LIST OF TABLES	ix
LIST OF FIGURES	xiii
LIST OF ABBREVIATIONS	xvi

CHAPTER

G

1	INT	RODUC	CTION	
	1.1	Graphe	ene Quantum Dots	1
	1.2	Chitosa	an	2
	1.3	Cetyltr	imethylammonium Bromide	3
	1.4	Ferric 1	Ions	3
	1.5	Surface	e Plasmon Resonance	4
	1.6	Problei	m Statements	6
	1.7	Resear	ch Objectives	6
	1.8	Thesis	Outlines	7
2	LIT	ERATU	RE REVIEW	
	2.1	Introdu	action	8
	2.2	Structu	ral and Optical Properties of Composite Materials	8
		2.2.1	Chitosan-based Materials	8
		2.2.2	Cetyltrimethylammonium Bromide-based Materials	10
		2.2.3	Graphene Quantum Dots-based Materials	11
	2.3	Sensing	g Properties of Chitosan-based Materials towards Metal	l 13
	2.4	Sensing	g Properties of Cetyltrimethylammonium Bromide-base	ed 16
		Materia	als towards Metal Ions	
	2.5	Sensing	g Properties of Graphene Quantum Dots-based Materia	ls 19
		toward	s Metal Ions	
		2.5.1	Incorporation of Graphene Quantum Dots-based	19
			Materials with Various Optical Methods	
		2.5.2	Incorporation of Graphene Quantum Dots-based	24
			Materials with Surface Plasmon Resonance	
			specification	
3	ME	THODO	LOGY	
	3.1	Introdu	iction	27
	3.2	Prepara	ation of Chemical	27
		3.2.1	Chitosan/Hydroxyl-functionalized Graphene Quantum Dots Composite	. 27
		3.2.2	Cetyltrimethylammonium Bromide/Hydroxyl-	27
			functionalized Graphene Quantum Dots Composite	
		3.2.3	Fe ³⁺ Ion Solution	28

	3.3 Pr	eparation of Thin Film	28
	3.4 Cł	aracterization	29
	3.4	1 Fourier Transform Infrared Spectroscopy	29
	3.4	Atomic Force Microscope	30
	3.4	UV-Visible Spectroscopy	30
		3.4.3.1 Absorbance Coefficient	31
		3.4.3.2 Energy Band Gap	32
	3.4	4 Photoluminescence	32
	3.4	5 Surface Plasmon Resonance	33
4	RESUL	FS AND DISCUSSION	
	4.1 In	roduction	35
	4.2 Cł	itosan/Hydroxyl-functionalized Graphene Quantum Dots	35
	Tł	in Film	
	4.2	2.1 Structural Properties	35
		4.2.1.1 FTIR Analysis	35
		4.2.1.2 AFM Analysis	37
	4.2	2.2 Optical Properties	39
		4.2.2.1 UV-Vis Absorption	39
		4.2.2.2 Energy Band Gap	40
		4.2.2.3 Photoluminescence Emission	42
	4.2	2.3 Sensing Properties using SPR Spectroscopy	42
		4.2.3.1 SPR Signal for Fe^{3+} on Gold Single Layer	42
		Thin Film	
		4.2.3.2 SPR Signal for Fe ³⁺ using Cs/HGQDs Thin	44
		Film	
		4.2.3.3 Sensitivity of Cs/HGQDs Thin Film	46
		4.2.3.4 Binding Affinity Constant, <i>K</i>	47
		4.2.3.5 Performance Parameter of the SPR Signal	49
	4.3 Ce	tyltrimethylammonium Bromide/Hydroxyl-functionalized	52
	Gı	aphene Quantum Dots Thin Film	
	4.	3.1 Structural Properties	52
		4.3.1.1 FTIR Analysis	52
		4.3.1.2 AFM Analysis	54
	4	3.2 Optical Properties	56
		4.3.2.1 UV-Vis Absorption	56
		4.3.2.2 Energy Band Gap	57
		4.3.2.3 Photoluminescence Emission	59
	4.	S.3 Sensing Properties using SPR Spectroscopy	60
		4.3.3.1 SPR Signal for Fe ³⁺ on Gold Single Layer	60
		Thin Film	<i>(</i> 1
		4.3.3.2 SPR Signal for Fe^{3+} using CTAB/HGQDs	61
			<i>(</i>)
		4.3.3.3 Sensitivity of CTAB/HGQDs Thin Film	64
		4.3.3.4 Binding Affinity Constant, K	65
	4.4 0	4.3.3.5 Performance Parameter of the SPR Signal	66
	4.4 Se	nsiuvity Ennancement	68
5	CONCI	USIONS	
2	5.1 Co	nclusion	71
	5.2 Re	commendations for Future Work	72

REFERENCES BIODATA OF STUDENT LIST OF PUBLICATIONS

73 90 91

LIST OF TABLES

Table		Page
1.1	The advantages and disadvantages of optical sensors.	5
2.1	The GQDs-based optical sensor for ferric ion detection.	25
4.1	FTIR band assignment of chitosan, HGQDs, and Cs/HGQDs thin films.	37
4.2	RMS roughness for each sample.	39
4.3	The SPR resonance angle and shift of resonance angle for Cs/HGQDs in contact with different concentration of Fe^{3+} solution.	47
4.4	Values of FWHM, DA, and SNR of Cs/HGQDs thin film in contact with Fe^{3+} of different concentration.	51
4.5	FTIR band assignment of CTAB, HGQDs, and CTAB/HGQDs thin films.	53
4.6	RMS roughness for each sample.	56
4.7	The SPR resonance angle and shift of resonance angle for CTAB/HGQDs in contact with different concentration of Fe^{3+} solution.	64
4.8	Values of FWHM, DA, and SNR of CTAB/HGQDs active layer in contact with different concentration of Fe ³⁺ ion.	67
4.9	Comparison of SPR-based sensor for Fe ³⁺ detection	70

LIST OF FIGURES

Figure		Page
1.1	Chemical structure of GQDs.	2
1.2	Chemical structure of (a) chitin and (b) chitosan.	3
1.3	Chemical structure of CTAB.	3
1.4	Kretschmann configuration of SPR sensor.	5
3.1	Flow chart for the preparation of the composite solution.	28
3.2	Coater machine: (a) sputter coater and (b) spin coater.	29
3.3	Optical setup of surface plasmon resonance spectroscopy.	34
4.1	FTIR spectrum of the chitosan, HGQDs, and Cs/HGQDs thin films.	36
4.2	AFM images of chitosan thin film.	37
4.3	AFM images of HGQDs thin film.	38
4.4	AFM images of Cs/HGQDs thin film.	38
4.5	AFM images of Cs/HGQDs thin film after in contact with Fe ³⁺ solution.	38
4.6	Absorbance spectrum of chitosan, HGQDs, and Cs/HGQDs thin films.	40
4.7	Optical band gap for chitosan thin film.	40
4.8	Optical band gap of HGQDs thin film.	41
4.9	Optical band gap of Cs/HGQDs thin film.	41
4.10	PL spectra of chitosan, HGQDs, and Cs/HGQDs thin films.	42
4.11	The SPR curve of gold layer in contact with deionized water.	43
4.12	The SPR curves for Fe^{3+} (20-100 ppm) in contact with gold layer.	43
4.13	The resonance angle shift of gold surface in contact with different Fe^{3+} concentration.	44
4.14	SPR curve for Cs/HGQDs thin film in contact with deionized water.	45

4.15	SPR reflectivity curves of Cs/HGQDs thin film in contact with Fe^{3+} solution of different concentration ranged from (a) 0.5-20 ppm and (b) 20-100 ppm.	45
4.16	The comparison of the shift of the resonance angle for concentration of Fe^{3+} in contact with Au and Au/Cs/HGQDs thin films.	47
4.17	The shift of the resonance angle for different concentrations of Fe^{3+} (0-10 ppm) in contact with Au/Cs/HGQDs thin film and Au thin film.	48
4.18	Full width at half maximum of SPR curve (for deionized water) correspond to half from its maximum value.	49
4.19	Detection accuracy of Cs/HGQDs SPR sensor in contact with Fe ³⁺ (0-100 ppm).	50
4.20	Signal-to-Noise-Ratio of the Cs/HGQDs SPR sensor in contact with Fe^{3+} (0-100 ppm).	51
4.21	FTIR spectrum of the CTAB, HGQDs, and CTAB/HGQDs thin films.	52
4.22	AFM images of CTAB thin film.	54
4.23	AFM images of HGQDs thin film.	55
4.24	AFM images of CTAB/HGQDs thin film.	55
4.25	The AFM images of CTAB/HGQDs thin film after in contact with Fe^{3+} solution.	55
4.26	Absorbance spectrum of CTAB, HGQDs, and CTAB/HGQDs thin films.	56
4.27	Optical band gap of CTAB thin film.	57
4.28	Optical band gap of HGQDs thin film.	58
4.29	Optical band gap of CTAB/HGQDs thin film.	58
4.30	PL spectra of CTAB, HGQDs, and CTAB/HGQDs thin films.	59
4.31	PL spectra of CTAB/HGQDs thin film at different excitation wavelength.	59
4.32	The SPR curve of gold layer in contact with deionized water.	60
4.33	The SPR curves for Fe^{3+} (0.001-10 ppm) in contact with gold layer.	61

xiv

4.34	The resonance angle shift of gold surface in contact with different Fe^{3+} concentration.	61
4.35	The SPR curve of CTAB/HGQDs thin film in contact with deionized water.	62
4.36	SPR reflectivity curves for CTAB/HGQDs thin film in contact with different concentration of Fe^{3+} solution ranged from (a) 0.001-0.1 ppm and (b) 0.1-10 ppm.	63
4.37	The comparison of the resonance angle for different Fe ³⁻ concentration in contact with Au thin film and Au/CTAB/HGQDs thin film.	64
4.38	Langmuir isotherm model of the SPR angle shift for different Fe ³⁺ concentration in contact with Au thin film and Au/CTAB/HGQDs thin film.	66
4.39	Full width at half maximum of SPR curve (for deionized water) correspond to half from its maximum value.	67
4.40	Detection accuracy of CTAB/HGQDs SPR sensor in contact with Fe^{3+} (0-10 ppm).	68
4.41	Signal-to-Noise-Ration of the CTAB/HGQDs SPR sensor in contact with Fe^{3+} (0-10 ppm).	68
4.42	Comparison of Langmuir isotherm model between gold and the modified gold thin films in contact with different concentration of Fe^{3+} (0- 10 ppm).	69
4.43	Possible mechanism for Fe ³⁺ detection using CTAB/HGQDs thin film.	70

C

LIST OF ABBREVIATIONS

A	absorbance
α	absorbance coefficient
AFM	Atomic Force Microscopy
Cs	chitosan
CTAB	cetyltrimethylammonium bromide
DW	deionized water
E_g	energy band gap
FTIR	Fourier Transform Infrared
GO	graphene oxide
GQDs	graphene quantum dots
hv	photon energy
HGQDs	hydroxyl-functionalized graphene quantum dots
Io	intensities of incident light
I_t	intensities of transmitted light
K	constant
MMW	medium molecular weight
nm	nano meter
PL	Photoluminescence
ppb	parts per billion
ppm	parts per million
rms	root-mean-square
rpm	revolution per minute
SPR	Surface Plasmon Resonance
Т	transmittance
t	thickness
UV-Vis	Ultraviolet-visible

CHAPTER 1

INTRODUCTION

1.1 Graphene Quantum Dots

Graphene quantum dots (GQDs) are zero-dimensional, nanometer-sized graphene fragments that possess the properties of both graphene and carbon dots (Bacon *et al.*, 2014; Mueller *et al.*, 2010). Figure 1.1 shows the basic chemical structure of GQDs. The introduction of GQDs has overcome the limitation of its two-dimensional counterparts, graphene sheets that have zero band gap thus not feasible to be applied in optical and photonics field (Wang *et al.*, 2018). Briefly, the band gap of graphene can be increased by quantum confinement effect when reducing the lateral dimensions of graphene into quantum dots. One of the remarkable properties of GQDs is the ability to tune its band gap and therefore to control the light absorbance and emission frequency. In this way, chemical and optical properties of GQDs can be adjusted depending on the desired applications (Das *et al.*, 2016; Kim *et al.*, 2012).

There are basically two ways for the synthesis of GQDs which are often called as topdown and bottom-up methods. In one hand, top-down can be explained as cutting, breaking, or splitting of carbon materials such as graphene oxide, graphite rods, graphite powder, and carbon black. The examples of process in this method includes hydrothermal cutting, solvothermal cutting, microwave-assisted cutting, and so forth (Li *et al.*, 2011; Peng *et al.*, 2012; Pan *et al.*, 2012). On the other hand, graphene moieties (i.e. citric acid, polycyclic aromatic hydrocarbon, etc.) that undergo organic process such as pyrolysis, carbonization, thermolysis, and reduction are called bottom-up method (Dong *et al.*, 2012; Tang *et al.*, 2012).

The most beneficial and unique properties of GQDs are that they are abundantly available since they are carbon material, low toxicity, highly soluble in various solvents, and can be modified with functional groups at their edges (Sun *et al.*, 2013). Due to the outstanding optical, electrical, mechanical, and thermal properties of GQDs, it offers some unique merits for new applications and has been one of the popular choices to incorporate with various applications. Comparisons between different research studies for various semiconductor quantum dots have been made to highlight GQDs' great potential in the field of photovoltaics, electronics, bio-imaging and optical sensors (Fan *et al.*, 2015; Sun *et al.* 2013; Wang *et al.*, 2015).

In order to increase the hydrophilicity and biocompatibility of a GQDs, it can be functionalized with various functional groups that contain oxygen such as hydroxyl, carboxyl, and epoxy groups (Hasanzadeh *et al.*, 2016). Focusing on hydroxyl groups, some studies found that it helps to stabilize the surface of the molecule, enhance fluorescent yield, and helps in radiative recombination (Geethalakshmi *et al.*, 2016). The hydroxyl groups tend to form a bond with carbon atoms and contribute to make other molecules soluble in water. The functionalization of GQDs with hydroxyl group will

form hydroxyl-functionalized GQDs or named as HGQDs. Lately, GQDs-based materials were modified with nanoparticles, polymer, and other materials in order to enhance the conductivity, optical properties, and also to improve the deposition of GQDs on any surface forming a uniform and regular thin layer (Mirzaie *et al.*, 2019; Ou *et al.*, 2015; Sadrolhosseini *et al.*, 2018). In another words, GQDs could be modified with other composites for better sensing performance.



Figure 1.1: Chemical structure of GQDs. (Hasanzadeh et al., 2016)

1.2 Chitosan

Chitosan (Cs) is the derivative of chitin where chitin is the second most important naturally occurring polysaccharide after cellulose. Chitosan is also defined as a copolymer of glucosamine and N-acetyl glucosamine which linked by β -1, 4 glucosamine. The main sources of production of chitin are from the treatment of marine crustaceans' shell such as shrimp and crab with the alkali sodium hydroxide. Moreover, it can also be found in the exoskeleton of insects and fungal cell wall (Thanou & Junginger, 2004). In order to modify chitin into chitosan, the acetyl group that attached to the nitrogen atom was replaced with hydrogen through hydrolysis, yielding a primary amine group. The comparison of chemical structure between chitin and chitosan is shown in Figure 1.2.

Additionally, chitosan is widely used in drug delivery systems, separation of membranes, wastewater treatment, and biosensors due to its biocompatibility and biodegradability. Because of the presence of reactive hydroxyl and amino functional groups in chitosan, it displays good susceptibility to chemical modifications with a variety of nanomaterials. As a natural polysaccharide, chitosan has good adhesion, high water permeability, high solubility, abundantly available, low cost, high mechanical strength, and excellent filmforming ability (Hasanzadeh *et al.*, 2016; Suginta *et al.*, 2013; Arena *et al.*, 2017; Jiang & Wu, 2019).

Due to the remarkable afore-mentioned advantages of chitosan, it has been selected as the most suitable biopolymer to combine with other materials in order to cater specific applications. Here, chitosan acted as a stabilizing and reducing agent and expected to chelate with the ferric ions. This combination of chemical and electrical properties of the element materials (i.e. chitosan and HGQDs) could positively lead to the development of new sensor.



Figure 1.2: Chemical structure of (a) chitin and (b) chitosan. (Thanou & Junginger, 2004)

1.3 Cetyltrimethylammonium Bromide

Surfactant is a substance that helps to reduce the surface tension of a liquid in which it dissolves. Cetyltrimethylammonium bromide (CTAB) is an important positively charged surfactant that has a long tail of 16-carbon atoms and a head of ammonium group with three methyl groups attached. CTAB that is soluble in water, commonly used as a coating agent, stabilizing agent, passivating agent, structure-directing agent in the synthesis of inorganic materials, and also helps in the accumulation of target materials (Bi *et al.*, 2012; Liu *et al.*, 2018; Mao *et al.*, 2014; Selvi *et al.*, 2018; Yao *et al.*, 2013). Besides, CTAB could also enhance the absorption of pollutants that is why it is commonly used in the wastewater treatment to facilitate the absorption and reaction with the pollutants (Jin *et al.*, 2012).

Above all afore-mentioned special properties of CTAB, the addition of CTAB to a material will enhance it sensing performance such as improve the sensitivity and the limit of detection. This is because CTAB improves the hyperchromicity and sensitization to the probe (Leng *et al.*, 2016). Since CTAB is positively charged, it is highly attracted to negatively charged GQDs. Figure 1.3 shows the chemical structure of CTAB.



Figure 1.3: Chemical structure of CTAB. (Lezaic et al., 2014)

1.4 Ferric Ions

Iron is known as the cheapest and most-used metal in our lives. There are several oxidation states of iron existed in the environment such as +2, +3, +4, and +6. When losing 3 electrons, iron will form ferric ion (Fe³⁺). Fe³⁺ are mostly found in the environment, industrial, clinical, and biological fields while playing remarkable and versatile roles in many physiological and pathological processes. Among them are oxygen transport, enzyme catalysis, electron transport, and DNA and RNA synthesis (Abbaspour *et al.*, 2014).

Even though Fe^{3+} is important for living things, both too much or insufficient amount of it can bring disadvantages and very harmful to the consumers. Lack of Fe^{3+} ions can cause anemia, affect the synthesis of hemoglobin, and restricting the delivery of oxygen to cells which resulting in lethargy, low work performance, and decrease immunity (Zimmermann & Hurrell, 2007). On the other hand, excess amounts of Fe^{3+} ions in a living cell can cause severe disease such as hepatitis, organ disfunction, hemochromatosis, and even cancers (Chen *et al.*, 2017; Zhou *et al.*, 2013). With a vast development of the world in many various human activities, the possibility for improper disposal of the pollutants which include toxic metal ions into the environment also increases.

Due to the awareness of this issues, the activities and efforts in the field of toxic metal ions sensing that include Fe^{3+} have been attracting researchers' attention up until today (Li *et al.*, 2013b). Moreover, sensitive and selective detection of Fe^{3+} is also highly due to the impact upon exposure mentioned above. Many modern technologies have been developed for the detection of Fe^{3+} and in this work, a method that allows rapid and simple detection of Fe^{3+} in solution was further investigated.

1.5 Surface Plasmon Resonance

Optical sensor is a device that able to convert light rays into a form that is readable by a measuring device. When there is interaction between materials and metal ions, optical sensors will give optical information for instance the absorbance, reflectance, fluorescent emission, change in the intensity, and quenching efficiency. The properties are often measured in the ultraviolet, visible, or near infrared ranges. The examples of commonly sensor are fluorescence sensor, photoluminescence used optical sensor. electrochemiluminescence sensor, colorimetric sensor, and surface plasmon resonance (SPR) sensor (Gao et al., 2018; Liu & Kim, 2015; Tang et al., 2019; Zhou et al., 2013). SPR is one of the well-known and versatile emerging optical methods that has been used over the past years in various biosensing and chemical sensing that also covers metal ions. The very first SPR phenomenon was observed by Wood in 1902 (Wood, 1902). SPR has been diligently studied and made vast advances in the development of technology and its application since the first demonstration of surface plasmon resonance for the learning of processes at the surfaces of metals and sensing of gases (Homola, 2008).

There are few approaches in SPR devices such as grating coupled systems, optical waveguide systems, optical fibres, and prism coupled system. Prism-based SPR is the most widely used approach in current SPR systems due to its sensitivity and simple usage (Löfås *et al.* 1991; Matsubara *et al.*, 1988). Prism-based approach can be divided into two arrangements which are Otto configuration and Kretschmann configuration. Comparing both configurations, Kretschmann configuration is normally used in most SPR applications where a metal that carries a large number of electrons like silver, copper, gold, or aluminum is placed at the interface of two dielectric media. Gold is favorably used as metal film since it is the most stable and sensitive compared to others. When plane-polarized light hits the gold-coatekd film prism under total internal

reflection conditions, SPR will occur resulting in reflected beam that will be detected for processing. The diagram for Kretschmann configuration is presented in Figure 1.4. One of the most crucial parts in SPR sensor is the development of active layers or recognition element. The active layer is sandwiched between metal layer and cell. A lot of studies have been made to improve the sensitivity of SPR sensor for toxic metal ions detections and one of them is by introducing new active layer of different materials such as semiconductors, polymers, dyes, and so forth (Chen *et al.*, 2008; Fen *et al.*, 2012; Pelossof *et al.*, 2012; Yu *et al.*, 2004). SPR sensors are widely used because it allows label-free, real time detection and have a sensitive property besides facile preparation of sample, quick measurement capability, and cost-effective (Fen *et al.*, 2012). The comparison of the advantages and disadvantages between several optical sensors for heavy metal ions are summarized in Table 1.1.



Figure 1.4: Kretschmann configuration of SPR sensor.

Optical sensors	Advantages	Disadvantages
Fluorescent	High sensitivity and	High sensitivity and
	selectivity; real-time	selectivity; real-time
	measurement; good	measurement; good
	reproducibility	reproducibility
Electrochemiluminescence	Good sensitivity and	High cost; low
	selectivity; stable;	compatibility;
	strong anti-	complicated preparation;
	interference ability;	frequent electrode fouling
	wide detection range	
Photoluminescent	High sensitivity and	Low precision and
	selectivity; real-time	accuracy; time
	measurement; good	consuming; limited
	reproducibility	application (small
		molecules)
Colorimetric	Good sensitivity; fast	Low reproducibility; low
	detection; inexpensive	stability; low selectivity
SPR	Very high sensitivity;	Low selectivity
	simple; low cost;	(improving)
	label-free	

Tab	e 1.1:	The	advant	ages	and	disad	vant	tages	of	op	tical	sensors.
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1.6 Problem Statements

Nowadays, as a new promising carbonic material with plenty of excellent advantages, GQDs-based materials have been receiving researchers' attention from the scientific community. A lot of studies have been conducted related to the preparation of various GQDs-based material via different methods to enhance the properties of the materials. This was done to cater desired and particular properties for specific applications such as for solar cells (Li et al., 2011; Tsai et al., 2016), photodetectors (Chiang et al., 2016; Haider et al., 2016), batteries (Park et al., 2016), bioimaging (Wu et al., 2018; Zhu et al., 2011), and sensors (Benítez-Martínez & Valcárcel, 2014; Raeyani et al., 2018). The incorporation of GQDs with different composite materials is believed to give positive effect on the properties of the GQDs (Chinnusamy et al., 2018; Gobi et al., 2017; Jiang et al., 2019; Kausar, 2019). Although there are many works on the combination of GQDs-based material with other materials, there were still no study on the incorporation of GQDs with chitosan and CTAB. Therefore, in this study, the optical and structural properties of chitosan/HGQDs (Cs/HGQDs) and CTAB/HGQDs are explored, since it is believed that the properties of composite materials are better compared to the independent material.

Prolonged exposure to toxic metal ions such as ferric ion can cause deleterious health effects in human. Thus, the increasing awareness on toxic metal ions pollution has led to the vast development and construction of sensing strategies using different materials to detect heavy metal ions. Up to now, many conventional methods have been widely used for the detection of metal ions such as anodic stripping voltammetry (Rosolina et al., 2015), plasma atomic emission spectroscopy (Ochsenkuhn-Petropoulou & Ochsenkuhn, 2001), and atomic absorption spectroscopy (Tarley et al., 2011). The methods are accurate and sensitive but they demand complicated sample preparation, expensive instruments, highly destructive, and time-consuming. Other modern methods than commonly used for toxic metal ions detection nowadays are electrochemical (Ting et al., 2015), photoluminescence (Huang et al., 2013), fluorescent (Xia et al., 2017), electrochemiluminescence (Chen et al., 2016), and colorimetric (Gao et al., 2018). However, the main drawbacks of these methods are slow detection, low compatibility, low sensitivity and selectivity. In order to overcome the circumstances, surface plasmon resonance (SPR) sensor is the alternative to be used in this work because it is well-known as one of the best emerging sensor technology where it has a highly sensitive property, allows the label-free and real-time detection (Homola, 2003). Besides that, the advantages of SPR sensor are cost-effective, easy preparation of sample, quick measurement capability and no compulsion of reference solution (Fen & Yunus, 2011). To the best of our knowledge, there is no report on the incorporation of SPR sensor with GQDs-based material. Hence, in this study, SPR will be attempted to incorporate with Cs/HGQDs and CTAB/HGQDs thin films for the detection of Fe³⁺.

1.7 Research Objectives

The objectives of this study are summarized as follows:

1. To study the optical and structural properties of HGQDs-based thin films after the addition of chitosan and CTAB.

2. To determine the potential sensing of Fe^{3+} using Cs/HGQDs and CTAB/HGQDs thin films using surface plasmon resonance spectroscopy.

1.8 Thesis Outlines

Chapter 1 consists of the introduction of the study. This chapter introduces and explains each important element in this study which are graphene quantum dots, chitosan, cetyltrimethylammonium bromide, ferric ion, and surface plasmon resonance optical sensor. Besides, this chapter contains the idea for the development of active layers using HGQDs-based composite material for Fe^{3+} sensing. The problems that are currently happening are also discussed in this chapter which proceed to the list of objectives of this research.

Chapter 2 contains previous and present researches mostly related to the study. The findings of all the works were discussed further throughout this chapter and they were used as a guidance to develop active layers using HGQDs-based composite material. This chapter contain two section i.e. optical and structural properties of composite materials and sensing properties of composite materials.

Chapter 3 introduces the methodology of sample preparation and characterization techniques used to observe and study the optical, structural, and sensing properties of all the samples in this study. The rigorous explanation on method starts from the preparation of chemical, preparation of thin film, and ends with the characterization of samples including sensing potential using surface plasmon resonance optical sensor.

Chapter 4 demonstrates the experimental results obtained on the optical, structural, and sensing properties of the thin films. This chapter also covers and discusses the analysis of results acquired from all characterization thoroughly.

Chapter 5 is the final chapter of the thesis which presents the conclusion of this study. Besides, all the findings are briefly summarized in this chapter in order to form a conclusion. The conclusion covers all the optical, structural, and sensing properties of the samples. The recommendation for improvements that can be done in the future work were also included in this chapter.

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Nur Ain Asyiqin binti Anas was born on December 21, 1995 in Muar, Johor. She took her primary school education at SK Parit Bunga and continued her study for secondary school in Sultan Abu Bakar Girls School, Muar until 2012. In 2013, she entered Johor Matriculation College for a year before entering UPM for her degree of Bachelor of Science with Education (Honours) Major in Physics and successfully graduated in the year 2018. She hopes to become someone who has a lot of knowledge and experience that can be shared with others in the future.

LIST OF PUBLICATIONS

Papers

- Nur Ain Asyiqin Anas, Yap Wing Fen, Nur Alia Sheh Omar, Nur Syahira Md Ramdzan, Wan Mohd Ebtisyam Mustaqim Mohd Daniyal, Silvan Saleviter, Afiq Azri Zainudin. (2019). Optical properties of chitosan/hydroxyl-functionalized graphene quantum dots thin film for potential optical detection of ferric (III) ion. *Optics & Laser Technology*, 120: 105724.
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Conferences

As oral presenter:

1. Nur Ain Asyiqin Anas (2019, March). Optical studies of functionalized graphene quantum dots and their implementation into an optical system for potential sensing application. Poster presented at Materials Technology Challenges 2019 (MTC 2019), Dewan Sri Harmoni, UPM Serdang, Selangor

Awards

1. Gold Medal for the invention of "Optical studies of functionalized graphene quantum dots and their implementation into an optical system for potential sensing application" at Materials Technology Challenges 2019 (MTC 2019): Awarded by Faculty of Science, Universiti Putra Malaysia.

2. Best Poster for the invention of "Optical studies of functionalized graphene quantum dots and their implementation into an optical system for potential sensing application" at Materials Technology Challenges 2019 (MTC 2019): Awarded by Faculty of Science, Universiti Putra Malaysia.



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