UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT OF CHITOSAN-GRAPHENE OXIDE ACTIVE LAYERS FOR DETECTION OF COBALT ION USING SURFACE PLASMON RESONANCE OPTICAL SENSOR

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By

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

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September 2019

Chairman : Yap Wing Fen, PhD
Faculty : Institute of Advanced Technology

The development of chitosan-graphene oxide based active layer namely chitosan-graphene oxide-cadmium sulphide quantum dots (chitosan-GO-CdS QDs) thin film and chitosan-graphene oxide-4−(2−pyridylazo)resorcinol (chitosan-GO-PAR) thin film for the detection of cobalt ion (Co^{2+}) by using surface plasmon resonance optical sensor has been studied. The synthesized thin films were confirmed by Fourier transform infrared spectrum by showing the functional groups of the composites. The optical properties of the composite thin films were characterized by using UV-Vis-NIR absorption spectroscopy where the absorbance peaks of the thin films can be observed in the range of 220 nm to 300 nm, and the band gaps are 4.033 eV and 4.066 eV for chitosan-GO-CdS QDs and chitosan-GO-PAR thin film respectively. From the Atomic Force Microscope (AFM) images, the addition of graphene oxide increased the roughness of the composite thin films and the presence of cadmium sulphide quantum dot was observed using Transmission Electron Microscope (TEM). Then, the prepared thin films were successfully used as the active layer for the detection of Co^{2+} in solution. The sensor showed good results and produced some linear responses as the concentration of the Co^{2+} were increased. At lower concentration, the chitosan-GO-PAR active layer showed higher sensitivity that is 0.2370° ppm^{-1}, compared to the chitosan-GO-CdS QDs active layer with sensitivity of 0.1188° ppm^{-1}. In addition, the calibration curve of the angle shift data using Sips model shows that the active layers can attract the cobalt which is proven by the high binding affinity of 1.649 ppm^{-1} for the gold (Au) modified with chitosan-GO-PAR active layer and 0.939 ppm^{-1} for the gold modified with chitosan-GO-CdS QDs active layer.

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

PEMBANGUNAN LAPISAN AKTIF BERDASARKAN KITOSAN-GRAFIN OKSIDA PENENTUAN ION KOBALT MENGGUNAKAN SENSOR OPTIK RESONANS PLASMON PERMUKAAN

Oleh

SILVAN SALEVITER

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Pengerusi : Yap Wing Fen, PhD
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Pembangunan lapisan aktif berasaskan kitosan-grafin oksida (kitosan-GO) iaitu filem nipis kitosan-grafin oksida-titik kuantum kadmium sulfida (kitosan-GO-TK CdS) dan filem nipis kitosan-grafin oksida-4−(2−pyridylazo) resorcinol (kiitosan-GO-PAR) untuk mengesan ion kobalt (Co^{2+}) dengan menggunakan sensor optikal resonans plasmon permukaan telah dikaji. Filem nipis yang disintesis telah disahkan oleh Fourier Transform Infrared spectroscopy (FTIR) dengan menunjukkan kumpulan berfungsi komposit tersebut. Sifat-sifat optik filem nipis komposit dicirikan dengan menggunakan spektroskopi penyerapan UV-Vis-NIR di mana puncak-puncak penyerapan filem-filem nipis dapat diperhatikan dalam lingkungan 220 nm hingga 300 nm, dan jurang tenaga untuk kitosan-GO-TK CdS dan chitosan-GO-PAR adalah masing-masing 4.033 eV dan 4.066 eV. Dari imej Atomic Force Microscope (AFM), penambahan GO meningkatkan kekasaran filem nipis komposit dan kehadiran titik kuantum kadmium sulfida diperhatikan menggunakan Transmission Electron microscope (TEM). Seterusnya, filem nipis yang telah siap digunakan sebagai lapisan aktif untuk mengesan Co^{2+} dalam larutan. Sensor ini telah menunjukkan keputusan yang baik dan menghasilkan beberapa respon linear apabila kepekatan Co^{2+} ditingkatkan. Walaubagaimanapun, lapisan aktif kitosan-GO-PAR menunjukkan kepekaan yang lebih tinggi iaitu 0.2370° ppm^{-1} berbanding lapisan aktif kitosan-GO-TK CdS dengan kepekaan 0.1188° ppm^{-1}. Sebagai tambahan, lengkung penentukuran data peralihan sudut menggunakan model Sips menunjukkan bahawa kedua-dua lapisan aktif mampu menarik Co^{2+} dan terbukti dengan pertalian mengikat yang tinggi iaitu 1.649 ppm^{-1} untuk emas yang diubahsuai dengan lapisan aktif kitosan-GO-PAR dan 0.939 ppm^{-1} untuk emas yang diubah suai dengan lapisan aktif kitosan-GO-TK CdS.

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LIST OF ABBREVIATIONS

AFM	Atomic force microscopy
Co^{2+}	Cobalt ion
FTIR	Fourier transform infrared
SPR	Surface plasmon resonance
TEM	Transmission electron microscope
GO	Graphene oxide
rGO	Reduced graphene oxide
PAR	4−(2−pyridylazo)resorcinol
CdS QDs	Cadmium sulphide quantum dots
EM	Electromagnetic
QDs	Quantum dots
Au	Gold
chitosan-GO-PAR	Chitosan-graphene oxide-4−(2−pyridylazo)resorcinol
chitosan-GO-CdS QDs	Chitosan-graphene oxide-cadmium sulphide quantum dots

CHAPTER 1

INTRODUCTION

1.1 Cobalt ion

Cobalt is an element with atomic number of 27 that is located in the first transition series of Group 9 in the periodic table. Cobalt occurs at three valence states which are 0, +2 and +3 with one stable isotope which is ^{59}Co. In its metalic state, cobalt (Co) is a hard and silvery gray solid at room temperature with a relative molecular mass of 58.93. This metal is soluble in diluted acid and it also soluble with water in ultrafine metal powder form at 1.1 mg/L. On the other hand, cobalt in compound forms are mainly occurs as cobaltous (Co^{2+}) and cobaltic (Co^{3+}) which are commercially and environmentally obtainable (Barałkiewicz & Siepak, 1999). According to the Concise International Chemical Assessment Document (CICAD), cobalt is the 33rd most abundant element that can be found in almost every media including water (ground and surface), air, soil, sediment and many more (Kim et al., 2006).

Sources of cobalt compounds exposure are from both natural and anthropogenic sources. Natural sources include by wind blowing dust, volcanoes eruptions, seawater spray, forest fire and marine biogenic emission. Anthropogenic sources include mining and smelting of cobalt ore, burning of fossil fuels, phosphate fertilizer and cobalt processing industries (Ministry of the Environment programs and initiatives, 2001). Cobalt that was released into the atmosphere by these sources may be deposited on soil and also absorbed by water. The main sources of cobalt exposure to the general population came from inhalation of ambient air and consumption of food and drinking water containing Co compounds. In addition, occupational exposure is another way of cobalt exposure to human as there are numerous industrial application such as paint, cobalt alloys production, mining, tabacco industries and so much more (Kim et al., 2006).

Trace amounts of cobalt are essential for human body as it is a component of vitamin B_{12} which therefore found in most tissues (Yamagata et al., 1962). Moreover, some radioactive isotopes of cobalt such as ^{60}Co are used in the nuclear medical field to treat patients. On the other hand, excessive exposure of inorganic cobalt compounds can be toxic for the environment and to the human body (Laura et al., 2017). In addition, cobalt is a metal with marked allergenic potential, asthma, interstitial lung disease and combined asthma and alveolitis have been described as occupational health hazards (Sheikh, 2016). Therefore, to reduce the chances of cobalt poisoning, there is a need to develop or improve a convenient tool for the detection of cobalt.

1.2 Optical sensor

Nowadays, optical sensor is known as one of the most versatile sensing tools that have the ability to detect wide range of target by different optical sensors such as temperature, pressure, radiation level, forces, electric field, pH values, strain, chemical concentration, displacement, liquid level, displacement, humidity, magnetic field, Acoustic field and many more (Safavi & Abdollahi, 1998; Ramakrishnan et al., 2016; Javanshir et al., 2018; Yan et al., 2018; Chen et al., 2019).

In general, optical sensor device consist of three basic parts which are light sources, light detector and transmitting medium as shown in Figure 1.1. But most of the time, some additional parts are required for the device to function such as lenses, mirrors, chopper, filters, polarizers, diaphragm depending on the need of the sensor (Ahuja & Parande, 2012).

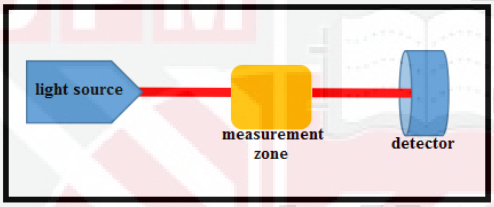

Figure 1.1: General components of optical sensors.

An optical sensor has the ability to measure the changes of the light beam which due to the alteration of the intensity of the light and that may change in its optical properties which is intensity, phase, wavelength, polarization and spectral distribution (Regtien & Dertien, 2018). In short, it is a device that measure the quantity of light and translate it into some form that is readable by the instrument (Peixoto & Silva, 2017). There are three general types of optical sensor which is through beam, reflective and retro reflective. Through beam sensor is a type of sensor where the transmitter and receiver are pointing directly at each other. It is a type of sensor that is suitable for the detection of solid object where the sensing method depend on the intensity of light received when the object passed through the light transmission path. Reflective sensor on the other hand is a type of sensor that have the transmitter and receiver parallel to each other.

The method of sensor detection is depending on the reflection of light emitted by the receiver. Retro-reflective sensor is similar to reflective sensor but the transmitter and receiver are in the same housing (Jorgenson, 2001). Some advantages of optical sensors compared to non-optical sensors are greater sensitivity, electrical passiveness, no electromagnetic interference, available in both point and distributed configuration and wide dynamic range of measurands (Regtien & Dertien, 2018). Due to these advantages, there are greater interest from scientists to research whether on the development of new optical sensor or the improvement of the existing optical sensors.

Surface plasmon resonance (SPR) is a versatile optical sensor that has been developed for the detection of variety of samples such as gas, biomolecule, and chemical which includes metal ions. The SPR phenomenon was first observed by Wood in 1902, when he observed a pattern of white and dark band as he shone a monochromatic polarized light on a mirror with a diffraction grating on its surface (Wood, 1902). A complete physical interpretation of this phenomenon was not available until 1968 when Otto and Kretschmann individually reported the excitation of surface plasmon in their works (citation). Since then, SPR has slowly made its way into the limelight with its practical applications in sensitive sensors where it first emerged as gas sensing and biosensing in 1983 by Liedberg et al. SPR is a phenomena where energy from electromagnetic (EM) radiation of p-polarized monochromatic light interact with free electron on the metal-dielectric interface that will induce a wave-like oscillation on the free electrons which then reducing the intensity of the reflected light that will occur only at a specific angle (Liedberg et al., 1983). The angle at which maximum dip of the light intensity is called SPR angle.

The basic principle of SPR sensor works by observing the changes of the resonance angle when there is a change in the refractive index near the metal surface. Over the year, SPR spectroscopy has been developed as novel and high potential optical sensing technique for sensing varieties of analytes including heavy metals in biomedical and environment field (Lokman et al., 2014; Fen et al., 2015a; Singh, 2016; Sadrolhosseini et al., 2017; Omar et al., 2018a). Because of that, there is a huge call for scientists to study various materials as active layer adjacent to the metal surface to create a greater change in the refractive index and hence improve the sensitivity of this technique.

1.3 Active layer material

Active layer or recognition element is one of the most essential part for the enhancement of the SPR sensor. For the past few years, the development of SPR sensor has been growing rapidly by the introduction of different materials active layer detecting some specific target molecules (Fen & Yunus, 2013a). In this study, chitosan-graphene oxide (GO) based material were applied to enhance the SPR sensor for the determination of cobalt ion.

Chitosan is a semicrystalline polymer material that is derived from chitin by deacetylation under alkaline condition. The chemical structure of chitosan is shown in Figure 1.2. Chitosan is said to be the most important chitin derivative, much easier to process has a good mechanical and optical properties. It is used to produce films, hydrogels, fibers where it is mostly used in the biomedical field because this material is non-toxic, biodegradable, biocompatible and possesses excellent film-forming ability. (Elsabee & Abdou, 2013). However, the stability of chitosan is low because of their hydrophilic character and also pH sensitivity (Rinaudo, 2006). Therefore, number of technique has been used to improve the mechanical and chemical properties of chitosan. One of the technique is by crosslinking with some reagent such as ionophore, glutaraldehyde and formaldehyde (Maitra & Shukla, 2014). Other than that, chitosan can be reinforced by blending it with other novel material such as graphene based material.

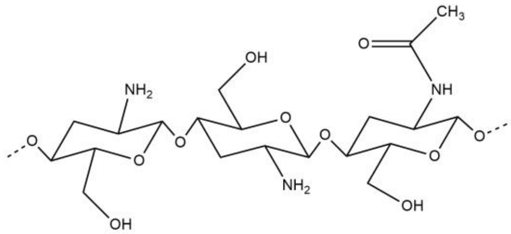

Figure 1.2: Chemical structure of chitosan. (Rinaudo, 2006)

Graphene is the most interesting allotrope of carbon given by its one-atom-thick layer of sp^2-bonded carbons atom arranged into 2D honeycomb lattice (Chakraborty & Hashim, 2018). Due to its unique 2D crystal structure, graphene can be controlled to have unlimited dimension of carbon materials with 0, 1, 2 and 3 dimensions (Geim & Novoselov, 2007). In addition, there are lots of studies that proven graphene to have remarkable strength, excellent electrical and thermal conductivity, large surface area and biocompatibility (Geim, 2009; Avouris, 2010).

Graphene can be chemically derived into graphene oxide (GO) or reduced graphene oxide (rGO). These graphene derivatives can be easily obtained from inexpensive graphite therefore are cost effective and also highly hydrophilic thus are stable in aqueous solution makes it easy to facilitate for the assembly of macroscopic structure (Ray, 2014). Moreover, these materials is known to have a potential binding sites as shown in Figure 1.3 that drove to its reputation for the past decades (Yang et al., 2010; Zhu et al., 2010; Hunt et al., 2014; Raidongia et al., 2014; Zainudin et al., 2017).

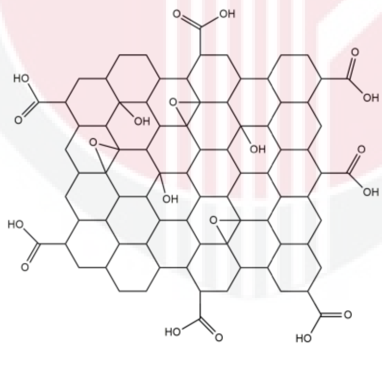

Figure 1.3: Chemical structure of graphene oxide. (Chakraboty & Hashim, 2018)

Quantum dots (QDs) are nano-sized crystals which compose of semiconducting materials or in other words are actually some tiny semiconductors that have the ability to convert incoming energy (Minti et al., 1991). As a semiconductor nanostructure material, a quantum dot confines the motion of conduction band electrons, valance band holes, or excitons which means it contain a small finite numbers of conduction band electrons, valance band holes, or excitons (Bogue, 2014). The size of this nano-particle is usually aroud 2 nm to 20 nm. Due to this confinment-effects, QDs exhibit unique and remarkable physical and chemical properties compared to bulky materials. Some of the advantages of QDs over other bulky materials is that, it have broader absorption spectra, narrow emission spectra, better photostability and longer fluorescent lifetime after excitation (Sobhana et al., 2011).

QDs are made up from atoms of groups II-VI, III-V or IV-VI elements in the periodic table. In the past few decades QDs have attracted considerable attention due to their special properties and cadmium sulphide quantum dots (CdS QDs) is one of the most studied QDs which composed from II-VI groups of semiconductors (Tyagi et al., 2014). There are many research information can be searched and obtained from varies sources regarding the preparation, properties and applications of CdS QDs (Li et al., 2003; Koneswaran & Narayanaswamy, 2009; Liu et al., 2014; Uppa et al., 2016; Tshemese et al., 2017; Wang et al., 2018). Cadmium sulphide based QDs are promising materials for optics, optoelectronics, medicine, and sensor technology development (Li et al., 2017; Veerathangam et al., 2017; J. Wang et al., 2017; Choi et al., 2018).

4−(2−pyridylazo)resorcinol (PAR) is a well-known chromogenic reagent, a type of reagent that gives or changed colour in a reaction (Florence & Farrar, 1963). This compound is a popular reagent applied in spectrophotometric, chelatometric, and cholorimetric analyis due to its abbility to correlate with many different metals (Starosta et al., 2014). The versatility of this compound may be contributed by its several reactive sites such as a pyridyl nitrogen atom, azo group and o-hydroxyl group as shown in Figure 1.4 and from the name itself (Karmakar & Singh, 2017). Pyridyl is a group that was derived from pyridine (C_5H_5N) by removal of a hydrogen atom from a ring carbon atom. By the removal of the hydrogen atom opens a room for a bonding with the azo group (−N=N−) at the second carbon. Resorcinol ($C_6H_6O_2$) on the other hand is an organic compound synthesized from the destructive distillation of a natural resin (Schmiedel et al. 2012). In its compound form, it appears as a white crystalline compound with a weak odor and a bittersweet taste (Schmiedel et al., 2012).

The reaction of resorcinol with 2-pyridylazo has led to the synthesis of 4−(2−pyridylazo)resorcinol (PAR) for the first time in 1959 (Pollard et al., 1959). Since then, PAR have been widely used as a chromogenic reagent for the detection of mainly metal ions.

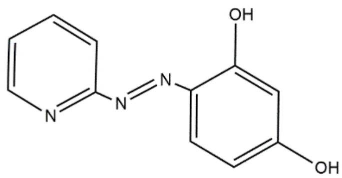

Figure 1.4: Chemical structure of 4−(2−pyridylazo)resorcinol. (Ghasemi et al., 2007)

1.4 Problem Statement

Nowadays, SPR sensor has been known as one of the versatile optical sensor for chemical sensing including metal ion sensing. One of the essential part in SPR metal ion sensor is the modified gold layer thin film and each thin film was fabricated to have its own sensitivity and selectivity towards specific metal ion. In order to further improve the reliability and versatility of this sensor, some novel material is needed to be incorporated in the development of the SPR sensor (Pattnaik, 2005; Fen et al., 2013b; Lokman et al., 2014; Paliwal et al., 2016; Qiu et al., 2018). The development of surface plasmon resonance as an optical sensor for metal ion sensing have been receiving growing attention from the scientific community including the detection of mercury, lead, arsenic, cadmium, copper, nickel and zinc (Abdi et al., 2011; Moksin et al., 2011; Fen et al., 2015b; Kamaruddin et al., 2017; Sadrolhosseini et al., 2017; Daniyal et al., 2018a). Excessive exposure of all metal ion, including cobalt ion can be harmful to living organisms. However, to the best of our knowledge, there is still no report on the detection of cobalt via SPR technique. Cobalt is a trace element that are widely found in nature. Although it has a biological need as a metal constituent of vitamin B_{12}, excessive intake of it has been shown to cause bad effects on human health (Laura et al., 2017). Therefore, it is of interest to develop active layer for the detection of cobalt via SPR technique.

Over recent years, chitosan-graphene oxide based material have been receiving continuous attention from the scientific community. Significant amount of researches and methods had been done for the development of this material matching with the intended application (Pan et al., 2011; Anandhavelu & Thambidurai, 2013; Yu et al., 2013; Kamaruddin et al., 2016; Kamaruddin et al., 2017; Zainudin et al., 2018). In this project, 4-(2-pyridilazo)resorcinol (PAR) and cadmium sulfide quantum dot were incorporated with chitosan-graphene oxide composite material for the detection of cobalt ion. However, the development of chitosan-graphene oxide based active layer thin film via spin coating technique are still lacking in study. Therefore, it is significant to study the characteristics and properties of the developed active layer thin film.

1.5 Objectives

The objectives of this research are summarized as follows:

1. To prepare chitosan-graphene oxide based thin films using spin coating technique.
2. To study the structural and optical properties of the prepared chitosan-graphene oxide based thin films.
3. To investigate the sensing properties of the chitosan-graphene oxide based thin film as an active layer for the determination of cobalt ion using surface plasmon resonance optical sensor.

1.6 Thesis outline

This thesis started with Chapter 1 which in begins with the introduction of cobalt ion, optical sensor, surface plasmon resonance optical sensor and also the composite materials of the active layer. In Chapter 2, the past and also the present researches from other researchers were reviewed. Then, the methodology for the preparation of the active layer and the instrument and experimental setup used for the analysis were explained in Chapter 3. In the next chapter, the experimental results were presented and discussed. Finally, the conclusion of the study and the recommendation for future work were written in Chapter 5.

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BIODATA OF STUDENT

Silvan Saleviter was born on the 31 of October 1994 in Ranau, Sabah. He began his early education in Pai Wen kindergarten and Pai Wen Chinese primary school. Then, he had his lower secondary in SMK Dato Paduka Mat Salleh, Ranau and upper secondary in SM St. Peter, Kuala Penyu. In 2012, he enrol into Labuan Matriculation College and a year later pursued his higher education in Universiti Putra Malaysia. He obtained his degree in Bachelor of Science with Honors Major in Physics from Universiti Putra Malaysia (UPM) in the year 2017. After graduating he pursued his study at Institute of Advanced Technology Universiti Putra Malaysia (UPM) in the field of Sensor Technology.

LIST OF PUBLICATION

MAIN AUTHOR

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Competitions

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Yap Wing Fen, Wan Mohd Ebtisyam Mustaquim Mohd Daniyal, **Silvan Saleviter**, Jaafar Abdullah. “Kretchmann configuration of surface plasmon resonance: From fundamental study to potential sensing application*.” Invention, Innovation & Design Exposition 2018 (iidex2018)*, held from 24-28 September 2018 at Dewan Agung Tuanku Canselor (DTAC), Universiti Teknologi Mara Shah Alam, Selangor. (Silver Medal)

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