UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT OF BIOPOLYMER-POLY(3,4-ETHYLENEDIOXYTHIOPHENE) BASED THIN FILMS AND THE POTENTIAL FOR MERCURY ION DETECTION USING SURFACE PLASMON RESONANCE

NUR SYAHIRA BINTI MD RAMDZAN

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By

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October 2021

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The global issue caused by agricultural and industrial waste products has driven researchers to develop heavy metal ion sensors for the detection of water pollution. There are numerous works on the development of sensing layers involving different types of materials. Apart from the novelty of material, the effectiveness and efficiency of materials as sensing layers also should be emphasized. Therefore, this works aims to incorporate the biopolymer and conducting polymer materials with surface plasmon resonance sensors for better sensing properties toward heavy metal ions. This thesis suggests two different sensing thin films, chitosan/poly(3,4-ethylenethiophene) (Cs/PEDOT) and nanocrystalline cellulose/poly(3,4-ethylenethiophene) (NCC/PEDOT) for the detection of mercury ions. The structural properties of both thin films were confirmed by Fourier transform infrared (FTIR) spectroscopy by the presence of functional groups of the composites. Moreover, the atomic force microscopy (AFM) shows that the roughness of thin films increased for Cs/PEDOT and NCC/PEDOT thin films, which shows that the surface roughness is influenced by the presence of chitosan and nanocrystalline cellulose. Meanwhile, the optical properties of synthesized thin films were investigated using ultraviolet-visible (UV-Vis) spectroscopy where the absorbance peaks for the thin films can be observed at a wavelength around 220–700 nm. Based on the band gap energy analysis, the values obtained for both thin films are 4.093 eV and 4.082 eV for Cs/PEDOT and NCC/PEDOT thin films, respectively. Next, the proposed thin films have been incorporated with a surface plasmon resonance sensor (SPR) to evaluate the effectiveness and efficiency of sensing mercury ions in an aqueous solution. For Cs/PEDOT thin film, the detection performance

showed the sensitivity of 21.9607° ppm^{-1} and the binding affinity constant value of 204.4990 ppm^{-1}. Meanwhile, the NCC/PEDOT thin film showed higher sensitivity of 48.5193° ppm^{-1} and the Langmuir isotherm model yielded a higher binding affinity constant with values of 211.8644 ppm^{-1}. The results indicate NCC/PEDOT thin film has a better sensing property compared to Cs/PEDOT thin film in detecting mercury ions. In conclusion, this work has successfully developed a new sensing layer in fabricating an effective and potential heavy metal ions sensor.

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

PEMBANGUNAN FILEM NIPIS BERASASKAN BIOPOLIMER-POLI(3,4-ETHYLENEDIOXYTHIOPHENE) DAN POTENSI UNTUK PENGESANAN ION MERKURI MENGGUNAKAN RESONANS PLASMON PERMUKAAN

Oleh

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Isu global yang berpunca dari bahan buangan pertanian dan industri telah mendorong para penyelidik untuk menghasilkan sensor ion logam berat untuk mengesan pencemaran air. Terdapat banyak kajian mengenai penghasilan lapisan penderiaan yang melibatkan pelbagai jenis bahan. Selain dari kebaharuan sesuatu bahan, keberkesanan dan kecekapan bahan sebagai lapisan penderiaan juga harus dititikberatkan. Oleh itu, kajian ini bertujuan untuk menggabungkan bahan biopolimer dan polimer pengalir dengan sensor resonans plasmon permukaan untuk menjamin sifat penderiaan yang lebih baik terhadap ion logam berat. Tesis ini mencadangkan dua filem nipis penderiaan yang berbeza, *chitosan/poly(3,4-ethylenethiophene)* (Cs/PEDOT) dan *nanocrystalline cellulose/poly(3,4-ethylenethiophene)* (NCC/PEDOT) untuk pengesanan ion merkuri. Sifat struktur bagi kedua-dua filem nipis disahkan oleh spektroskopi transformasi Fourier inframerah (FTIR) dengan kehadiran kumpulan berfungsi dalam komposit. Tambahan pula, mikroskopi daya atom (AFM) menunjukkan bahawa kekasaran filem nipis meningkat untuk filem nipis Cs/PEDOT dan NCC/PEDOT, yang membuktikan bahawa kekasaran permukaan dipengaruhi oleh kehadiran *chitosan* dan *nanocrystalline cellulose*. Sementara itu, sifat optik filem nipis yang telah disintesis dikaji menggunakan *ultraviolet-visible* (UV-Vis) spektroskopi, puncak penyerapan untuk filem nipis dapat diperhatikan pada panjang gelombang sekitar 220–700 nm. Berdasarkan analisis jurang tenaga jalur, nilai yang diperolehi untuk kedua-dua filem nipis adalah 4.093 eV dan 4.082 eV untuk filem nipis Cs/PEDOT dan NCC/PEDOT masing-masing. Kemudian, filem nipis yang dicadang telah digabungkan dengan sensor resonans plasmon permukaan (SPR) untuk menilai keberkesanan dan kecekapan penderiaan ion merkuri dalam larutan berair.

Bagi filem nipis Cs/PEDOT, prestasi pengesanan menunjukkan sensitiviti, 21.9607° ppm^{-1} dan nilai untuk pemalar tarikan ikatan, 204.4990 ppm^{-1}. Sementara itu, filem nipis NCC/PEDOT menunjukkan sensitiviti yang lebih tinggi iaitu 48.5193° ppm^{-1} dan model isoterma Langmuir menghasilkan pemalar tarikan ikatan yang lebih tinggi dengan nilai 211.8644 ppm^{-1}. Hasil kajian menunjukkan filem nipis NCC/PEDOT mempunyai sifat penderiaan yang lebih baik berbanding filem nipis Cs/PEDOT dalam mengesan ion merkuri. Kesimpulannya, kajian ini telah berjaya menemukan lapisan penderaian baru dalam menghasilkan sensor ion logam berat yang berkesan dan berpotensi.

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I certify that a Thesis Examination Committee has met on 7 October 2021 to conduct the final examination of Nur Syahira binti Md Ramdzan on her thesis entitled "Development of Biopolymer-Poly(3,4-Ethylenedioxythiophene) Based Thin Films and the Potential for Mercury Ion Detection using Surface Plasmon Resonance" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

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Name of Member of Supervisory Committee: Dr. Josephine Ying Chyi Liew

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LIST OF ABBREVIATIONS

Symbols	Description
A	Absorbance
α	Absorption coefficient
AFM	Atomic Force Microscopy
Cs	Chitosan
DA	Detection accuracy
E_g	Energy band gap
FTIR	Fourier Transform Infrared
FWHM	Full width at half maximum
hv	Photon energy
I_o	Intensities of incident light
I_t	Intensities of transmitted light
k	Plank’s constant
NCC	Nanocrystalline cellulose
PEDOT	Poly(3,4-ethylenedioxythiophene)
ppm	Parts per million
PSS	Polystyrene sulfonate
rms	Root-mean-square
rpm	Revolution per minute
SNR	Signal-to-noise-ratio
SPR	Surface Plasmon Resonance
T	Transmittance
t	Thickness
UV-Vis	Ultraviolet-Visible

CHAPTER 1

INTRODUCTION

1.1 Biopolymer Materials

Owing to naturally-based characteristics, biopolymer materials have been attracted huge attention from worldwide researchers in the last decades. Biopolymers are natural polymers such as chitosan, cellulose and nanocrystalline cellulose that can easily find and synthesize from organic materials on the Earth. Because of that, biopolymer has been regarded as one of the most used materials and had been applied in various beneficial applications. Other than that, these natural materials also have common properties that have been highlighted in previous studies which are low cost, biodegradable, and renewable material (Faham et al., 2019; Jia et al., 2017).

Biopolymers consist of monomeric units that are covalently bonded to form larger molecules. As chitosan, it is a linear amino polysaccharide of glucosamine and an N-acetyl glucosamine unit that can be obtained by alkaline deacetylation of chitin. The main ingredients of chitosan are chitin that is available in surrounding resources, for example, the shells of crab, prawn, shrimps, fish scales and plant-based material (Badawy et al., 2011). It is an excellent stabilizer of metal nanoparticles, has good biocompatibility and is a low cytotoxicity material (Bodnar et al., 2005; Mathew et al., 2012). With hydrophilic properties and a large number of amino and hydroxyl groups, this polymer is also able to undergo several chemical modifications; hence, it governs the properties of the material (Klemm et al., 2005). These properties of chitosan make this biopolymer one of the promising materials for many applications especially related to environmental monitoring.

Apart from chitosan, cellulose is another natural-based material that has various applications. This colourless and odourless polymer consists of linear chains of several hundred to ten thousand of β-1,4 linked to D-glucose units with the formula of $(C_6H_{10}O_5)_n$. This polymer is one of the most common polysaccharides and an unlimited organic material on Earth that can be synthesized from many sources. Over the past decade, this biopolymer material had widespread use in applications such as the food industry, pharmaceutical industry and drug-delivery systems (Gupta et al., 2019). To enhance the properties of cellulose, researchers had done some modifications using the acid hydrolysis process. Hence, nanometer-sized single crystal cellulose which is commonly referred to as nanocrystalline cellulose (NCC) can be obtained. NCC is a derivative of cellulose that possesses hydroxyl groups on its skeleton. This nano-sized polymer had attracted huge of researchers' attention, because of its biodegradable, biocompatible material, low toxicity and low cost. Extensive studies showed that NCC had great potential application in fields, such as regenerative medicine, optics, drug delivery systems and composited materials (Jia et al., 2017; Golmohammadi et al., 2017; Listyanda et al., 2020).

Compared to these two types of biopolymers, chitosan has numerous reactive amino acid and hydroxyl groups. Meanwhile, only hydroxyl groups existed on the NCC surface. Thus, these functional groups allow surface and chemical modification of the material and resulting in producing a compatible and stable matrix (Manan et al., 2016). As a result, biopolymer materials had offered promising and novel properties in many applications, especially sensors. Hence, biopolymer materials had proved an important role in fabricating an effective and good sensing layer. Biopolymer materials are an excellent matrix to develop a high selectivity and sensitivity sensor in detecting heavy metal ions.

1.2 Conducting Polymers Materials

In this modern globalization, world technologies had improved and there is also the rapid development of conducting polymers in previous research. Generally, conducting polymer is an organic material that can conduct electricity and has the properties of conductors or semiconductors material. This beneficial group also known as synthetic metals consists of polyanilines, polypyrroles and polythiophene as polymers-based. The advantages that had been proposed by this polymer are low-cost synthesis, good environmental stability, biocompatibility, and low oxidation potential (Tavoli et al., 2016; Lo et al., 2019). Hence, these advantages can be employed for many types of applications, e.g., batteries, transistors, light-emitting diodes (LED) and optical sensors (Sun et al., 2015; Saikia et al., 2018; McFarlane et al., 2010).

Polythiophene and its derivatives have been at the centre of considerable scientific interest for their attractive and excellent properties. However, a derivative of polythiophene called poly(3,4-ethylenedioxythiophene) (PEDOT) is the most promising material by the fact of its physical and chemical stability. Since PEDOT was first synthesized, many works of properties PEDOT have been carried out. With high transparency, high conductivity, good chemical and electrochemical properties, this polymer possesses excellent properties compared to other conducting polymers (Rahimzadeh et al., 2020).

On the other side, PEDOT has limitations on hydrophobicity and low stability. However, researchers had figured out this problem, by adding an acid dopant, polystyrene sulfonate (PSS) to improve the stability and the optical properties of PEDOT (Lee et al., 2019). The dopant plays an important role in the stability of conducting polymer which is influenced by the mobility of anions (Gade et al., 2007). As accounted by the previous study, the backbone of PEDOT:PSS exhibits better electron transfer and proved the enhancement in this polymer (Mahakul et al., 2017). The acid treatment of PEDOT also dissolves the problem of PEDOT insolubility whereas the dispersibility of this material is increased. Since that, this conducting polymer can be incorporated with other aqueous solutions and deposited on the metal surface. In this context, the combination of conducting polymer and other materials is expected to come up with a better and high sensitivity of sensing material for future application.

1.3 Surface Plasmon Resonance

Over the past decade, there have been increasing interest by researchers in the development of an optical sensor for heavy metal ions detection. Although, various types of optical sensors have been emerged for detecting heavy metal ions in aqueous solution, such as colorimetric, electrochemiluminescence, fluorescence and photoluminescence (Jiang et al., 2018; Xu et al., 2018; Lee et al., 2017). Surface plasmon resonance (SPR) has provided more advantages compared to other mentioned optical sensors, because of the low detection limit and the sensitivity towards targeted analytes.

SPR is a surface-sensitive technique that involves the interaction between dielectric medium and metal thin film. A sharp shadow called surface plasmon resonance is observed by the reduction of the intensity of the reflected light at a specific incident angle, it is due to the resonance energy that happens by the incident beam and surface plasmon wave. One of the important works in 1983, Liedberg et al. have reported the first chemical sensing based on the SPR technique. The successful research on gas and bio-sensing using this technique had offered worldwide attention for sensing potential in the field of sensor application. Hence, the SPR technique has been extensively studied and developed as a powerful technique for a variety of sensor applications, especially in biological and chemical sensing. Other than that, SPR has also been used in real sample detection, such as dengue viruses, water pollution and many more (Omar et al., 2019).

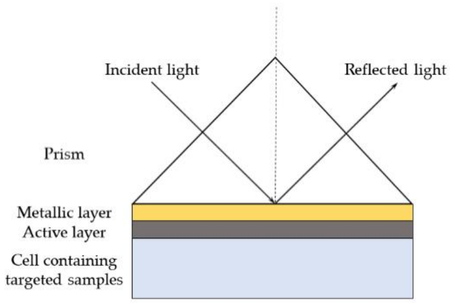

Figure 1.1: Kretschmann configuration of prism coupler.

The basic principle of this technique is by observing the changes of resonance angle by adding the interface between media and a metal thin film, which will cause the change in the refractive index of the metal surface. However, to ensure the effectiveness of SPR analysis, a few components should be considered before the experiment started, which are the configuration of the prism coupler and the active layer coated on metal thin film. There are two types of configurations for a prism coupler in SPR setup, Otto configuration and Kretschmann configuration. The prism coupler is important to verify the excitation of the plasmon can generate in two semi-infinite media, and the Kretschmann configuration is a preferable and practical setup, because of the unnecessary sensing medium gap between the prism and metal thin film as shown in Figure 1.1.

Another important component is the active layer coated on metal thin film. For the metal thin film, the gold layer more tends to be selected due to its high stability and sensitivity compared to silver or aluminum. Moving to the active layer or also known as the sensing layer, the researchers have studied and developed various types of sensing layers to enhance the sensitivity of SPR sensor in detecting heavy metal ions. Biopolymer, conducting polymer, graphene oxide and quantum dots are examples of novel materials that extensive studies to develop a high sensitivity active layer in SPR sensor (Kumari et al., 2014; Sadrolhosseini et al., 2012; Lokman et al., 2014; Anas et al., 2019). The incorporation of two novel materials also showed the increase of sensitivity towards analytes. Accordingly, the development of an active layer always motivates worldwide researchers to study the variation of materials and so on to apply it as a better and highly sensitive active layer in a future application.

1.4 Problem Statement

Researchers have investigated the potential applications of biopolymer materials in diverse platforms. Among the biopolymers that had drawn attention are chitosan and nanocrystalline cellulose, which have been applied in the food industry (Fortunati et al., 2019), pharmaceutical industry (Ou et al., 2013), drug-delivery systems, cosmetics (Aranaz et al., 2018) and environmental monitoring (Yun-Fei et al., 2017). Numerous studies have been carried out on the integration of biopolymer material and conducting material (Sadrolhosseini et al., 2011; Abdi et al., 2011; Verma et al., 2015). Interestingly, a significant potential had shown when both polymers were combined. Recently, Ravit et al. had reported on the electrochemical properties of PEDOT/NCC films for supercapacitors application (Ravit et al., 2019). In addition, there is research has been studied on the PEDOT/Chitosan electrodes for electrochemical bio-sensing (Sui et al., 2017). Although there were extensive researches has been carried out on biopolymer materials incorporated with conducting polymer, it is only focusing on the applications of the composite material. Hence, there is still a lack of studies on the structural and optical properties of these incorporated materials, chitosan/ poly(3,4-ethylenedioxythiophene) (Cs/PEDOT) and nanocrystalline cellulose/ poly(3,4-ethylenedioxythiophene) (NCC/PEDOT). Consequently, this limitation gives the inspiration to explore the structural and optical properties of Cs/PEDOT and NCC/PEDOT thin films. The incorporation between two types of polymers is predicted to enhance the properties of composite material throughout this experiment.

As water pollution had become the world's major problem, great efforts have been recently devoted to the development of environmental sensors, in which various approaches and sensing strategies have been practiced using different types of materials and sensors. Inspired by previous research, glassy carbon electrodes for linear sweep anodic stripping voltammetry (LSASV) (Anandhakumar et al., 2011) and differential pulse stripping voltammetry (Yasri et al., 2014) are the examples of methods been applied to determine the potential of poly(3,4-ethylenedioxythiophene) (PEDOT) in sensing application. But these methods have disadvantages such as complex procedures involved, expert operators are needed and time-consuming (Nawaz et al., 2018). For overcoming these disadvantages, surface plasmon resonance has been recently applied as a valuable optical sensor, which has its advantages such as higher sensitivity, stability and effectiveness on metal ion detection. However, there are no previous study has investigated mercury ion detection using PEDOT-based material. Therefore, it provides important insights into the development of PEDOT-based sensors for detecting mercury ions using surface plasmon resonance spectroscopy. As a matter of fact, surface plasmon resonance is a highly feasible method to use in this study to determine the sensing potential of Cs/PEDOT and NCC/PEDOT thin films toward mercury ions.

1.5 Objectives of Study

From the problem statement stated above, the interest of this research is to explore the properties of composite thin films. Thus, this research embarks on the following objectives:

1. To determine the structural and optical properties of Cs/PEDOT and NCC/PEDOT thin films.
2. To investigate the potential sensing of PEDOT-based thin films for mercury ion detection using surface plasmon resonance.

1.6 Outlines of Thesis

This thesis consists of five chapters. The first chapter (Chapter 1) provides an introduction of about two types of material that have been discovered throughout this research, which are biopolymer and conducting polymer materials. Surface plasmon resonance has also been explained throughout this chapter, where this optical sensor has been involved to evaluate the potential sensing of the sensing layer in detecting mercury ions. The motivation and objectives of the study are also outlined. Next, Chapter 2 reviews some literature related to structural and optical properties of biopolymer and conducting polymer materials. Other than that, the previous and current research on the potential sensing application of both polymers using surface plasmon resonance techniques have been highlighted.

Moving on, Chapter 3 covers the methodology of preparation composite thin film and characterization techniques involved for structural, optical and sensing properties analysis. This chapter starts with the preparation of chemicals, preparation of the thin films, and ends with the characterization of the thin films including sensing potential using surface plasmon resonance optical sensor. In Chapter 4, the experimental results obtained from characterization have been analyzed and its comprehensive explanations have also been elaborated. The results were discussed on the structural, optical properties and sensing potential of the thin film in detecting mercury ions (Hg^{2+}). Finally, Chapter 5 summarizes all findings to form a conclusion based on structural, optical and sensing properties of the thin films. Besides, there always has room for improvement, therefore some recommendations and suggestions for future work were also included.

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