

UNIVERSITI PUTRA MALAYSIA

INTERDIGITATED CAPACITOR INCORPORATING CHITOSAN FILM AS MERCURY METAL ION SENSOR AT MICROWAVE FREQUENCY

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By

RASHIDAH BINTI RADZALI

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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DEDICATION

To my beloved parents Radzali bin Bong & Siti Sawibah Jasin Brothers and Sisters

Thank You for Everything



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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May 2016

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Various techniques have been used to determine the presence of heavy metal ion. This thesis propose a new finding in a detection of heavy metal ion focusing on mercury metal ion which is by using Interdigitated Capacitor (IDC) and chitosan film as sensor at microwave frequency. This method was chosen because the existing methods to determine the presence of heavy metal ion are complicated and time consuming. IDC sensor incorporating chitosan film will be a novel method as it has never been used to determine the presence of heavy metal ion. The simplicity and cost effective of this project will be a good finding to detect mercury metal ion as well as iron and lead ions.

Resistivity measurement of chitosan film dipped into mercury solution with different temperatures, concentrations and contact time were carried out by using Four-Point Probe. Resistivity of chitosan film increased with increasing concentration and contact time of mercury solution and decreased with increasing temperature. This measurement shows the capability of chitosan to detect the presence of mercury ion by changing in value of resistivity.

IDC sensor was designed and fabricated at frequency range between 130 MHz to 3 GHz. For the microwave measurement, in order to make sure IDC sensor functioning well, the test result for the IDC sensor had been carried out. It shows good agreement with the simulation result and the best sensor has been chosen for microwave measurement.

IDC sensor was then combine with chitosan film and tested for capacitance and transmission (S_{21}) measurement. Chitosan film was dipped into different concentrations of heavy metal ions which are mercury (Hg), lead (Pb) and Iron (Fe). Results show the capacitance increased with increasing concentration for mercury, iron and lead

solution. Transmission (S_{21}) measurement was only carried out for mercury solution and it increased with concentration of mercury solution. Based on the result, this research has proved the capability of IDC sensor incorporating chitosan film to detect the presence of heavy metal ion at 1 ppm to 10 ppm by changing in value of capacitance as well as transmission (S_{21}) .



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

PENGGABUNGAN KAPASITOR INTERDIGITAL DAN FILEM CHITOSAN SEBAGAI SENSOR ION LOGAM MERKURI PADA FREKUENSI MIKROGELOMBANG

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Pelbagai teknik telah dijalankan untuk mengesan kehadiran ion logam berat. Kajian ini mencadangkan pengesanan ion logam berat terutama ion logam merkuri menggunakan sensor IDC dan filem chitosan pada frekuensi mikrogelombang. Kaedah ini dipilih kerana kaedah yang sedia ada untuk mengesan kehadiran ion logam merupakan proses yang rumit dan mengambil masa. Penggabungan sensor IDC dan filem chitosan merupakan kaedah yang baru dimana kaedah ini tidak pernah digunakan untuk mengesan kehadiran ion logam berat. Kaedah ini yang mudah dan ekonomi akan menjadi kaedah yang bagus untuk mengkaji kehadiran ion logam berat merkuri serta ion ferum dan ion plumbum.

Kerintangan filem chitosan yang di celup kedalam larutan merkuri dengan kepekatan, suhu dan masa yang berbeza telah dikaji menggunakan Four-Point Probe. Nilai kerintangan filem chitosan menaik dengan naiknya kepekatan dan masa dan menurun dengan naiknya suhu larutan merkuri. Pengukuran ini menunjukkan keupayaan filem chitosan mengesan kehadiran ion logam merkuri.

Sensor IDC telah direka dan di fabrikasi pada frekuensi mikrogelombang di antara 130 MHz hingga 3 GHz. Bagi pengukuran mikrogelombang, untuk memastikan sensor IDC berfungsi dengan baik, IDC sensor telah dikaji dan hasil kajian sensor IDC dipersetujui dengan keputusan simulasi. Sensor yang paling bagus telah dipilih untuk pengukuran mikrogelombang.

Seterusnya, sensor IDC digabungkan bersama filem chitosan dan nilai kapasitor dan penghantaran (S_{21}) telah dikaji. Filem chitosan dicelup kedalam tiga larutan ion logam berat yang berbeza iaitu merkuri (Hg), plumbum (Pb), dan ferum (Fe). Kajian menunjukkan nilai kapasitor menaik apabila kepekatan larutan tinggi. Pengukuran bagi

penghantaran (S_{21}) telah dijalankan bagi ion logam merkuri sahaja dan nilai penghantaran (S_{21}) menaik dengan kepekatan larutan. Kajian ini membuktikan keupayaan sensor IDC bersama filem chitosan untuk mengesan kehadiran ion logam berat pada 1 ppm hingga 10 ppm dengan perbezaan diantara nilai kapasitor dan penghantaran (S_{21}) .



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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 Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

IDC Interdigitated Capacitor MUT Material Under Test GHz Giga Hertz dB Decibel Mercury Hg Pb Lead Iron Fe °C Degree Celcius Transmission S_{21} Part Per Million ppm Minutes min Relative Permittivity $\epsilon_{\boldsymbol{r}}$ AAS Atomic Adsorption Spectroscopy XRF X-Ray Fluorescence Spectrometry SPR Surface Plasmon Resonance **CVAFS** Cold Vapour Atomic Fluorescence Spectrometry Micrometer μm

CHAPTER 1

INTRODUCTION

Water pollution is recognised as one of the universal problem and has globally drawn the attention of scientist due to the need of clean and vital environment. Cases of heavy metal pollution in water system have been increasingly reported in recent years. A lot of activities such as improper disposal of electric goods, agricultural as well as industrial activities are the main reasons that lead to water pollution.

One of the effects of water pollution is metal ion poisoning. Metal ion poisoning is a deadly problem which may contribute to the neurological dysfunction and cancer. In the United States, the second most abundant water contaminant is mercury as cites by the Environmental Protection Agency (US EPA, n.d.). Based on survey conducted by the Department of Environment Malaysia in 2014, from 876415.44 metric tonnes of schedule waste generated, 12.81 % of it contained heavy metal sludge where it proved that heavy metal pollution has grown to a dangerous level (Malaysia Quality Report, 2014).

Mercury is the 67th most abundant element in the earth and often released into the atmosphere by degassing of the earth's crust. 170, 000 tons of mercury contaminated waste was produced per year by fossil fuel combustion and other activities such as improper waste disposal (Ozuah, 2000). Exposure to mercury may causes loss of myelinated nerve fibers, abnormal central nervous system cell division and autonomic dysfunction. Mercury poisoning also leads to autism and neurodevelopmental syndrome. Several symptoms which are associated with autism are found in mercury poisoning cases such as immune, sensory, neurological motor and behavioural dysfunctions (Bernard *et al.*, 2002).

Although mercury is cited by Environmental Protection Agency in USA as the leading water contaminant, other heavy metals ions such as lead, iron, chromium, arsenic and cadmium can be found abundantly in water supply. Some of these heavy metals are essential to the human body but some of them can cause harm.

Lead poisoning is also one of the most persistent metal contamination issue as it can damaged reproductive system and harmful to the foetus and young children. People who have been exposed to lead for a long time may suffer memory deterioration, prolonged reaction time and reduced ability to understand. In less serious cases, the most obvious sign of lead poisoning is disturbance of haemoglobin synthesis and long-term lead exposure may lead to anaemia (Järup, 2003).

In humans, iron is an essential component but excess of iron can cause toxicity even death. In human poisonings, symptoms of iron intoxication include vomiting, cirrhosis of the liver, coma and abdominal pain (Ahmed & Roy, 2009).

Due to these harmful heavy metals presence in the water system, it is important to implement a sensor system to detect mercury ion as well as any other heavy metals ion in drinking water as heavy metal pollution has been a serious concern to human health.

1.1 Chitosan

Chitin is the second most abundant carbohydrate on earth after cellulose and synthesized by a number of living organisms such as crab and fungi. As chitin is only dissolved in certain solvents, it is often converted into chitosan by alkali treatment which replace the acetyl side group with an amine groups along its backbone (Murray & Dutcher, 2006).

Chitosan is a β -(1 \rightarrow 4)-linked biopolymer of 2-amino-2-deoxy-D-glucose (glucosamine) and it is a linear cationic polysaccharide. Chitosan is non-toxic, biodegradable and occurs naturally in the cell walls of most fungi and some alga (Wan et al., 2003). Chitosan is a material which is soluble in dilute acidic solutions when the percentage of free amine groups along its backbone that is responsible in chelating heavy metal ions is 60 % or higher. The percentage free amine group is called degree of deacetylation (Mcilwee, 2008).

Ability of chitosan to form complexes with transition metals has given rise to the interest from researchers (Guibal *et al.*,1999). Chitosan gained recognition in water treatment and particularly to recover metal as traces in water system better than any natural compounds due to the free amine groups along its backbone (Rhazi *et al.*, 2002). Chitosan is used in a wide range of applications such as in biotechnology, pharmacists, drugs, food and biosensors (Harish & Tharanathan, 2007).

1.2 Microwave Technique

The applications of microwave technology can be found in various fields from telecommunications, broadcasting, military, environmental remote sensing, medical system, ground positioning system and many more.

Microwave methods for material characterization have two main types which are resonant and non-resonant method. Non resonant methods are often used to investigate the electromagnetic properties over a wide range of frequency while resonant methods are used to get more accurate dielectric parameter at single or several discrete frequencies.

The fundamental principal behind the two methods is the interaction between propagation waves and medium of which the waves is propagating in materials. There are several forms of such interactions such as reflection, refraction, scattering, emission and adsorption. Microwave sensors are used for various applications. Some of the examples of the microwave sensors are transmission sensors, reflection sensors, resonators and radar sensors.

IDC had been used widely at microwave frequencies due to the fact that it is more effective to measure the capacitance value. Interdigitated capacitors sensor (IDC) is used for the evaluation of near-surface properties such as permeability, conductivity and permittivity of the materials. IDC has been used for measurement of dielectric material for dairy products (Mukhopadhyay et al.,2006), gas and humidity sensor, biosensor application and also detection of dangerous toxins in contaminated seafood (Radke & Alocilja 2005a; Syaifudin et al., 2009). Application of IDC depends on the characteristic of the sensor itself and also the characteristics of the material under test (MUT). Therefore, there is a possibility to develop IDC sensor for a novel low-cost sensing system for several heavy metal ion which are mercury (Hg), lead (Pb), and iron (Fe).

1.3 Problem Statement

Due to the severe threats that heavy metal pollution poses to human health and environment, it is important to provide a detection system which are low cost, portable, reliable and fast. Several conventional techniques with high sensitivity have been used for heavy metal detection such as atomic adsorption spectroscopy (AAS), x-ray fluorescence spectrometry (XRF), and surface plasmon resonance (SPR) and cold vapour atomic fluorescence spectrometry (CVAFS). All these methods are time consuming, expensive and normally involve complicated processes in order to identify the heavy metal ions from the samples. Hence, sensor which is economical, rapid and has the ability to characterize heavy metal should be prioritized.

This project presents a new technique to detect heavy metal ion by developing a low cost sensor incorporating IDC sensor with chitosan film. Even though chitosan film have been used in conjunction with other detection techniques for heavy metal detection but there is no report on the use of chitosan film with IDC sensor as part of a sensor element. This project will focus on design and characterization of IDC sensor coated with chitosan film for detection of heavy metal ion which are mercury, lead and iron at frequency from 130 MHz to 3 GHz.

1.4 Objectives of the Project

The objectives of this project are

- To design, fabricate and test IDC sensor at microwave frequency
- To determine the presence of heavy metal (mercury, iron, lead) ion focusing on mercury metal ion by using IDC sensor incorporating chitosan film
- To study the effect of different concentration of mercury, lead and iron (1 ppm, 3 ppm, 5 ppm, 7 ppm, 10 ppm) on the capacitance and transmission (S₂₁)

1.5 Thesis Outlines

This thesis is structured as follows. Chapter 1 gives a brief introduction on the water pollution and the main problem behind it, followed by chitosan as metal ion binding agent, detection strategies and the problem statement of this project. The previous and current works on chitosan and IDC sensor were covered in Chapter 2. Theory of IDC sensor was explained later in Chapter 3. The methods to prepare chitosan film, technique of detection and sensor fabrication were explained in Chapter 4. The results on the electrical characterization of the chitosan film and the detection of heavy metal ion by using IDC sensor were analysed and discussed in Chapter 5. Lastly, Chapter 6 gives the conclusion and suggestions for future works.

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