UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT OF MULTIMODE FIBER OPTIC-BASED ETHANOL SENSOR USING NANOCOMPOSITE SENSITIVE LAYER

ALI MOHAMMED ALI

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By

ALI MOHAMMED ALI

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ALI MOHAMMED ALI

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Chairman : Associate Professor Ahmad Shukri b. Muhammad Noor, PhD
Faculty : Engineering

Optical fiber sensors have been gaining popularity on a wide scale over the past two decades as the call for highly efficient, miniaturised and versatile sensing devices are rapidly becoming a necessity. This research focused on the design and analysis of a standard multi-mode fiber optic to be used as a sensor for the detection of aqueous ethanol. Using multi-mode fiber optic (MMF) sensor in the process of detecting liquid required a process to amend the fiber by mode of tapering in order to increase the effectiveness of the sensor for sensing. The focus of this project is to increase the evanescent waves of the sensor and to increase the absorption rate and sensitivity of the sensor. It was done by using Au (gold) and Pd (palladium) as the nanocomposite coating layer. In this thesis, the effect of Au layer on the fiber, Pd layer on the fiber and the combination of both of Au and Pd as a nanocomposite are investigated for the sensor performance. Au and Pd have emerged as leading materials in a wide variety of applications, including chemical sensors, due to their exceptional thermal, optical and mechanicalproperties. The combination of Au and Pd are able to enhance the chemical reaction with ethanol, resulting in sensing capability of the sensor. Moreover, increments of the sensitivity were observed by coating a nanocomposite layer of the Au and Pd on the tapered fiber. The study dealt with five sensors with different coating ratios. The first sensor was coated by nanocomposite layer of Au and Pd with ratio 2 to 1 respectively. The second sensor was coated with a compound of 1 to 0.7 ratio of Au to Pd. The third sensor was coated with 1 to 1 ratio of the compound Au and Pd respectively. The fourth sensor is coated by 1 to 2 ratio of Au to Pd. The last sensor was coated with Au and Pd ratio of 1 to 3 respectively. All the nanoparticle layer were deposited on the tapering region using drop casting technique, after the sensors were annealed at 70°C. The developed sensors demonstrated high sensitivity at 0.074/vol % and response and recovery times of approximately 13 and 9 seconds, respectively. The achievements of this study are to use a large diameter tapered as a sensitive sensor toward ethanol and to apply Au and Pd mixture to tapered fiber toward ethanol detection.

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

PEMBANGUNAN SENSOR ETANOL BERASASKAN OPTIK GENTIAN PELBAGAI-MOD MENGGUNAKAN LAPISAN SENSITIF NANOKOMPOSIT

Oleh

ALI MOHAMMED ALI

Ogos 2017

Pengerusi : Profesor Madya Ahmad Shukri b. Muhammad Noor, PhD
Fakulti : Kejuruteraan

Penderia gentian optik telah mencapai popularity dalam skala yang besar sejak dua dekad yang lalu disebabkan permintaan yang semakin meningkat untuk peranti penderia yang sangat cekap, kecil dan serbaguna. Kajian ini memberi tumpuan kepada rekabentuk dan analisis gentian optik pelbagai mod yang biasa, diubahsuai sebagai penderia untuk pengesanan cecair etanol. Penggunaan gentian optik pelbagai mod dalam proses pengesanan cecair memerlukan proses pengubahsuaian dalam bentuk penirusan untuk meningkatkan keberkesanan penderia. Tumpuan projek ini adalah untuk menguatkan gelombang evanesen penderia dan meningkatkan kadar penyerapan dan kepekaan penderia. Ini dilakukan dengan menggunakan lapisan emas (Au) dan palladium (Pd) sebagai lapisan nanokomposit. Dalam tesis ini, kesan lapisan Au, Pd dan kombinasi Au-Pd disiasat terhadap prestasi penderia. Au dan Pd telah muncul sebagai bahan utama dalam pelbagai aplikasi, termasuk penderia kimia, kerana mereka mempunyai ciri-ciri termal, optik dan mekanikal yang luarbiasa. Gabungan Au dan Pd dapat meningkatkan tindakbalas kimia dengan etanol, mengakibatkan peningkatan keupayaan pengesanan penderia. Selain itu, peningkatan kepekaan dikaji dengan lapisan lapisan nanokomposit Au dan Pd yang berlainan pada gentian optic tirus. Kajian ini menyiasat lima penderia dengan nisbah bahan salutan Au-Pd yang berlainan. Penderia pertama disalut dengan lapisan nanokomposit Au dan Pd dengan nisbah 2:1. Penderia kedua dilapisi dengan nisbah Au-Pd 1:0.7. Penderia ketiga diselaputi lapisan Au Pd bernisbah 1:1 diikuti penderia ke empat di salut Au Pd bernisbah 1:2. Penderia terakhir disalut dengan nisbah Au dan Pd 1:3. Setiap lapisan nanokomposit didepositkan pada kawasan penirusan menggunakan teknik penyalutan jatuh, selepas itu gentian optik di tempatkan di ketuhar pada 70°C untuk proses penyepuh lindapan. Penderia yang direkabentukkan ini menampilkan kepekaan yang tinggi iaitu 0.074 / vol% serta masa tindakbalas dan pemulihan kira-kira 13 dan 9 saat masing-masing. Pencapaian kajian ini adalah menggunakan penderia gentian optik diameter besar sebagai penderia etanol yang peka untuk menerapkan kombinasi nanokomposit Pd dan Au kepada gentian optik tirus terhadap pengesanan etanol.

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The members of the Supervisory Committee were as follows:

Ahmad Shukri b. Muhammad Noor, PhD
Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Nizam Tamchek ,PhD
Senior Lecturer
Faculty of Sciences
Universiti Putra Malaysia
(Member)

ROBIAH BINTI YUNUS, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

Declaration by Member 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.

Signature: ______________________________

Name of Chairman of Supervisory Committee: Associate Professor Dr. Ahmad Shukri b. Muhammad Noor

Signature: ______________________________

Name of Member of Supervisory Committee: Dr. Nizam Tamchek

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LIST OF ABBREVIATIONS

AFM	Atomic Force Microscopy
Ag/rGo	Sliver Nanoparticles/Reduce Graphene Oxide
Au	Gold
BTU B	British Terminal Unit
C_2H_5OH/ CH_3-CH_2-OH	Ethanol
CNT	Carbon Nanotubes
CVD	Chemical Vapour Deposition
DC	Directional Coupler
DS-DNA	Double-Stranded Deoxyribonucleic Acid
EDX	Energy Dispersive X-Ray Spectroscopy
EIA	Environmental Impact Association
EWA	Evanescent wave absorption
FBG	Fiber Bragg Grating
FESEM	Field Emission Scanning Electron Microscopy
FITC	Fluoresce in Isothiocyanate
FWHM	Full width Half Maximum
GO	Graphene Oxide
G-TMMF	Graphene- Tapered Multi-Mode Fiber
HCl	Hydrochloric Acid
HF	Hydrofluoric Acid
IR	Infrared
LEL	Lower Explosive Limit
MMF	Multimode Fibers
MWCNT	Multi Walled Carbon Nanotube

NON-OECD	NON- Organization for Economic Cooperation and Development
OECD	Organization for Economic Cooperation and Development
Pd	Palladium
POF	Polymer Optical Fiber
rGO	ReducedGraphene Oxide
RI	Refractive Index
SMF	Single Mode Fiber
SNR	Signal to Noise Ratio
SPP	Surface Plasmon Polariton
SPR	Surface Plasmon Resonance
TIR	Total Internal Reflection
TMMF	Tapered Multi-Mode Fiber
UEL	Upper Explosive Limit
USA	United States America
UV	Ultraviolet
VIS	Visible
VOC	Volatile Organic Compound
WDM	Wavelength Division Multiplexing
XRD	X-Ray Diffraction
ZnO	Zinc Oxide

CHAPTER 1

INTRODUCTION

1.1 Overview

Over the past few decades, optical fibers have been established as the medium of choice for the transmission of data for telecommunications[1], [2]. Yet, it is exhilarating to note that lately optical fibers have been used in various applications such as in the areas of medical treatment, diagnostics and illumination[3]–[5]. Moreover, the development and research of biological, chemical and physical optical sensors have been reported to have numerous points of interest. The wide diversity of optical sensing technologies and their improvement are based on a number of driving factors. One example is in the field of environmental monitoring, where the aim is to monitor the compositions of high risk chemicals as well as their concentrations in real time[6]–[8]. However, it would be beneficial if this monitoring could be performed by using an appliance that is secure in an environment that is possibly flammable or volatile. Therefore, an essentially harmless technology is required. Optical fiber sensors are becoming more popular in view of their ability to be used in such harmful environments. Furthermore, optical fiber sensors are easily integrated into optical telecommunication and network systems because of their interaction origin coupled with the additional ability of remote and distributed sensing.

Volatile organic compounds (VOCs) are categorized as a carbon based chemical group with a tendency to evaporate at room temperature[9]. VOCs are commonly used in countless industrial and commercial settings as disinfecting and cleaning agents, as well as solvents. Even though most VOCs are not highly toxic, prolonged exposure can lead to numerous health problems in the long term[9].

The organic compound are ethanol a colourless, volatile and flammable liquid[9]. This compound also known as Ethan-1-ol or ethyl alcohol, which goes by the chemical formula C_2H_5OH or CH_3-CH_2-OH. Alcoholic beverages are general sources ofethanol and, when consumed, they may cause insobriety. Ethanol is also gaining popularity as an alternative, clean fuel for use in motor vehicles as it burns cleaner than fossil fuels[10].

The need for clean and renewable energy is continuously growing worldwide. Scientists are seeking other renewable energy sources to meet these multitudinous requirements. As was referred to online in 2013 the Environmental Impact Association (EIA), that includes the Organization for Economic Cooperation and Development (OECD), along with Non-OECD nations,released its latest projection, as shown in Figure 1.1, which showed that the energy expenditure of the world would grow by 56% from 2007 to 2035[10].

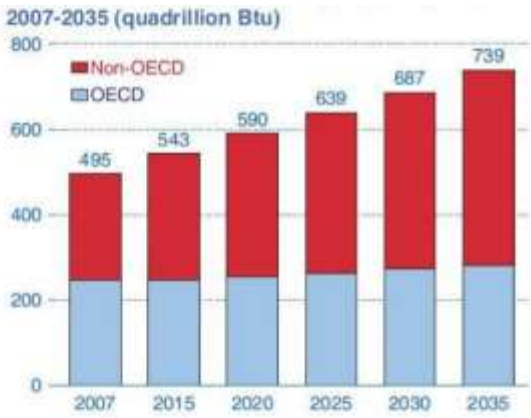

Figure 1.1 : World energy consumption projection[10]

Over the past decade, the production of ethanol has risen significantly due to increasing requests for ethanol fuels. A study in 2013 reported that 13 billion gallons of ethanol were transported, either by rail or barges, from production facilities in the USA alone. At present, the biggest volume of risky materials being transported by rail is ethanol[10]. The rise in ethanol production in the USA between 2000 and 2012 is shown in Figure 1.2.

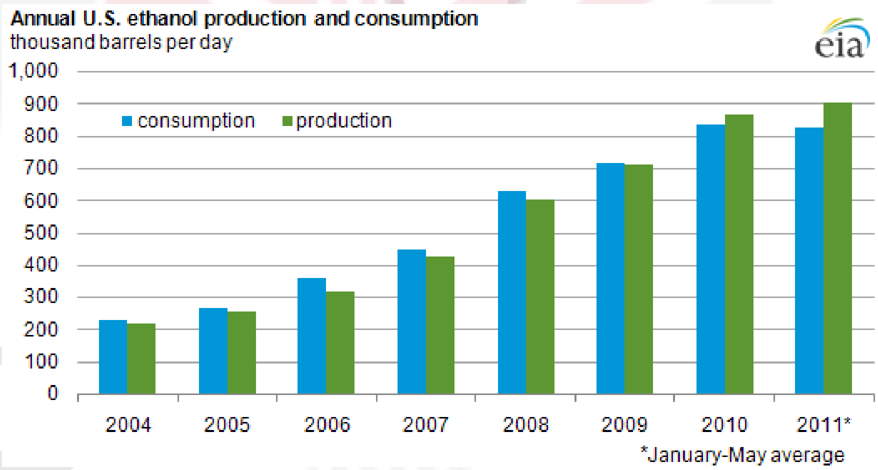

Figure 1.2 : Ethanol production from 2000 to 2012[10]

One of the major challenges is spills or leakage during storage or transportation. As mentioned earlier, liquid ethanol is flammable, colourless and dissolves completely in water. The combustible range of gasoline is much lower than that of ethanol. However, it has a mass that is heavier than air. The range of ethanol from the Lower Explosive Limit (LEL) to the Upper Explosive Limit (UEL) is from 3.3% up to 19%; however, the flash point of pureethanol is very low at 13°C[9].

The flammability of ethanol is affected when it is mixed with water. Nevertheless, even if only 20% ethanol is present in water, it remains flammable. At this level of concentration, an indication of 36°C was stated as the flash point, at which it is considered to be a highly flammable and volatile type of liquid[9]. The risk to humans during ethanol leaks or spills usually involves ingestion, inhalation or direct skin contact in certain circumstances, where ethanol has seeped into water catchment areas (groundwater or exterior water intakes)[11], [12].

Thus, the level of ethanol in water should be continuously monitored to ensure that the water quality is not advancing towards toxic or anoxic levels. Sensors to meticulously analyse the concentration of VOCs in liquid field mediums are extremely vital to further avoid possible health as well as environmental complications. Typical experimental methods with aqueous ethanol include purification, followed by refractometry or hygrometry, gas chromatography [13], [14] and, last but not least, infrared spectroscopy [15].

1.2 Problem Statement

The risk of ethanol to humans during leaks or spills usually involves unambiguous contact with the skin, inhalation, or consumption in certain circumstances, where ethanol has slipped into water catchment intakes such as from groundwater or outer water sources[9]. The most common thing happen usually is associated with the spill over of ethanol to exterior parts of water. The inevitability of marine species expiring several days after the discharge of ethanol due to the decrease of oxygen in the water has been disclosed. This occurs regularly at some distance from the initial location of the spill. Once ethanol has spilled into a larger body of water, the options for responding are very limited. In the United States of America (USA), it was reported that from 2006 till 2010 there were 11 major spills, which mostly resulted in the contamination of water[9]. Even though ethanol is very commonly used, it is a dangerous chemical, it is highly flammable; as such, it has exact flash points which are important to know when using it. While ethanol is consumed when drinking alcoholic beverages, consuming ethanol alone can cause coma and death.

Thus, to ensure that the quality of water is not advancing towards toxic or anoxic levels, continuous monitoring of ethanol levels in water should be executed. It is highly important to have sensors to accurately monitor the concentrations of VOCs in liquefied mediums to avoid probable health and environmental consequences. Typical tests for liquefied ethanol include purification, followed by hygrometry or refractometry, gas chromatography and, last but not least, infrared spectroscopy. On the whole, these methods involve the accumulation of samples that will later be brought back to the lab. The results can normally be obtained after a few days.

1.3 Objectives

The objectives of this research work are:

1- To design ethanol sensor using tapered multimode fiber (MMF) coated with Pd/Au nanocomposite layer
2- To characterize the developed sensors.
3- To evaluate the performance of the developed sensors.

1.4 Scope

This thesis presented experimental approaches in developing the sensor. First by usinga multi-mode fiber optic (MMF) sensor coated with a mixture of Pd and Au. This study focused on a tapered multi-mode fiber optic sensor and tried to enhance its sensitivity towards the concentration of ethanol in water. This study investigated the effects of Au/Pd nanocomposite on a tapered MMF sensor and measures its responses towards ethanol concentrations.

1.5 Thesis Organization

Firstly, this section provides the information needed to understand the rest of the thesis. In Chapter 2, some theoretical background on optical fiber sensing systems, ethanol sensors and nanostructure materials will be discussed. A review of previous works in this area will also is presented here. Chapter 3 (Methodology) explains the methods and technique used in this work to achieve the specified objectives. These consist of the techniques that were used by previous researches, as explained in Chapter 2. The research methodology, which includes the experimental works, is explained in Chapter 3. Chapter 4 (Results and Discussion) explains the results in detail, and presents a complete discussion and validation of the experimental results obtained in this study. The results are illustrated in tables. Chapter 5 (Conclusion and Recommendations) lists the final achievements and verifies them with the set objectives, as highlighted in the first chapter, to ensure that all the objectives have been fulfilled. Some of the limitations of the present work will be identified and recommendations will be made to improve on the current research in the future.

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