UNIVERSITI PUTRA MALAYSIA

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

ALI MOHAMMED ALI

FK 2017 71

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

By

ALI MOHAMMED ALI

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

August 2017

© COPYRIGHT UPM

COPYRIGHT

All material contained within the thesis, including without limitation text, logos, icons, photographs and all other artwork, is copyright material of Universiti Putra Malaysia unless otherwise stated. Use may be made of any material contained within the thesis for non-commercial purposes from the copyright holder. Commercial use of material may only be made with the express, prior, written permission of Universiti Putra Malaysia.

Copyright © Universiti Putra Malaysia

© COPYRIGHT UPM

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

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

By

ALI MOHAMMED ALI

August 2017

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.

© COPYRIGHT UPM

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.

© COPYRIGHT UPM

ACKNOWLEDGEMENTS

First and foremost, I would love to express my heartfelt gratitude to those that has made all of this possible. It is vital that, my appreciation goes to the Almighty Allah, my creator for made me alive and made my journey in life memorable. Secondly, my parents, Mohammed Ali Jasem and Hadiyh Khalaf, for the continuous unconditional love and support even though from distances away. Not forgetting my sister, Enas Mohammed Ali, one of the contributor for making my experience and education here possible. Furthermore, without any level of gratitude or whatsoever, I would like to extend my appreciation to my supervisor, Associate Professor Dr. Ahmad Shukri bin Muhammad Noor; for he was the one who initiated such challenge and giving me this opportunity to undergo with my experiments within his supervision. His valuable suggestions and guidance helped me grow educationally as well as mentally. Along with Dr. Puntithavathi Thirunavakkarasu; for her time for me to help guide and support me thoroughly with my thesis and experiments. Also to Mohd Anwarul Arif bin Md Rosli, for helping me and teaching me the skills to use the spectroscopy equipment and tools to make my experiments a doable and possible task. In addition, valuable support from my friend Abdullahi M. Yusufu and shared experiences by giving me advice. Last but not least, Raeesah Bt. Anuar for she has motivated me and supported me I would also like to extent my honest gratitude to those indirectly and directly made my journey educational and myself mentally, physically as well spiritually intact, which resulted in a complete my journey.

© COPYRIGHT UPM

I certify that a Thesis Examination Committee has met on 4 August 2017 to conduct the final examination of Ali Mohammed Ali on his thesis entitled "Development of Multimode Fiber Optic-Based Ethanol Sensor using Nanocomposite Sensitive Layer" 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.

Members of the Thesis Examination Committee were as follows:

Muhammad Hafiz bin Abu Bakar, PhD
Senior Lecturer
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Mohd Hanif bin Yaacob, PhD
Senior Lecturer
Faculty of Engineering
Universiti Putra Malaysia
(Internal Examiner)

Fairuz bin Abdullah, PhD
Associate Professor
Universiti Tenaga Nasional
Malaysia
(External Examiner)

NOR AINI AB. SHUKOR, PhD
Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 26 October 2017

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science
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:

© COPYRIGHT UPM

Declaration by graduate student

I hereby confirm that:

- this thesis is my original work;
- quotations, illustrations and citations have been duly referenced;
- this thesis has not been submitted previously or concurrently for any other degree at any other institutions;
- intellectual property from the thesis and copyright of thesis are fully owned by Universiti Putra Malaysia, as according to the Universiti Putra Malaysia (Research) Rules 2012;
- written permission must be obtained from supervisor and the office of Deputy Vice-Chancellor (Research and Innovation) before thesis is published (in the form of written, printed or in electronic form) including books, journals, modules, proceedings, popular writings, seminar papers, manuscripts, posters, reports, lecture notes, learning modules or any other materials as stated in the Universiti Putra Malaysia (Research) Rules 2012;
- There is no plagiarism or data falsification/fabrication in the thesis, and scholarly integrity is upheld as according to the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) and the Universiti Putra Malaysia (Research) Rules 2012. The thesis has undergone plagiarism detection software.

Signature: _____ Date: _____

Name and Matric No.: Ali Mohammed Ali (GS44269)

© COPYRIGHT UPM

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

© COPYRIGHT UPM

TABLE OF CONTENTS

		Page
ABSTRACT		i
ABSTRAK		ii
ACKNOWLEDGEMENTS		iii
APPROVAL		iv
DECLARATION		vi
LIST OF TABLES		x
LIST OF FIGURES		xi
LIST OF ABBREVIATIONS		xiv
CHAPTER		
1	**INTRODUCTION**	1
	1.1 Overview	1
	1.2 Problem Statement	3
	1.3 Objectives	4
	1.4 Scope	4
	1.5 Thesis orgnazation	4
2	**LITERATURE REVIEW**	5
	2.1 Introduction	5
	2.2 Optical fibers	5
	2.3 Optical fibers sensors	6
	2.4 Tapered optical fibers sensors	7
	2.5 Evanescent wave	16
	2.6 Absorbance-Based Sensors	16
	2.7 Sensitivity calculation	17
	2.8 Gold (An) metal	18
	2.9 Palladium (Pd) metal	19
	2.10 Ethanol sensor	21
	2.11 Summary	22
3	**RESEARCH METHODOLOGY**	23
	3.1 Outline	23
	3.2 Research workflow chart	23
	3.3 Fabrication tapered optical fiber	25
	3.4 Au/Pd Mixture preparation	26
	3.5 Pd deposition on MMF	26
	3.6 Au deposition on MMF	26
	3.7 Mixture of Au and Pd deposition on MMF	27
	3.8 Experimental setup	27
	3.9 Sample Preparation and Chemical Testing Procedures	28
	3.10 Results of Au-Pd coating characterization	29

© COPYRIGHT UPM

3.11 Results of second concentration of Pd and Au mixture coating characterization (2:1) Au toPd 30
3.12 Results of Pd and Au mixture with three different concentration coating characterization by EDX technique 31
3.13 Summary 33

4 RESULTS AND DISCUSSION ON SENSING PERFORMANCE OF MMF SENSOR TOWARDS AQUEOUS ETHANOL 34
4.1 Introduction 34
4.2 Results of the comparison betweenthe three diameters of the uncoated tapered MMF sensor 34
4.3 Results of Tapered MMF Sensor (20µm) 35
4.4 Results of Tapered MMF Sensor (30µm) 37
4.5 Results of Tapered MMF Sensor (40µm) 38
4.6 Results of comparison offive MMF sensors with coating 40
4.7 Results of Au-coated MMF Sensor with diameter of 40 µm 42
4.8 Results of Pd-coated MMF Sensor with diameter of 40 µm 44
4.9 Result of the mixture with the ratio of (1:1) Au to Pd 45
4.10 Result of the mixture with the ratio of (1:2) Au to Pd 47
4.11 Result of the mixture with the ratio of (1:3) Au to Pd 49
4.12 Result of the mixture with the ratio of (2:0.7) Au to Pd 51
4.13 Result of the mixture with the ratio of (2:1) Au to Pd 53

5 CONCLUSION AND FUTURE WORK 57
5.1 Conclusion 57
5.2 Future work 58

REFERENCES 59
BIODATA OF STUDENT 64
LIST OF PUBLICATIONS 65

© COPYRIGHT UPM

LIST OF TABLES

Table		Page
2.1	Summary previous study used to detect the ethanol	15
2.2	Physical and chemical properties of ethanol	21
4.1	Response and recovery time of tapered fiber optic ethanol sensor	55

© COPYRIGHT UPM

LIST OF FIGURES

Figure		Page
1.1	World energy consumption projection	2
1.2	Ethanol production from 2000 to 2012	2
2.1	Refraction and reflection of light	6
2.2	(a) Intrinsic and (b) Extrinsic optical fiber sensor	7
2.3	Dynamic response time of coated the fiber optic with 1.0 mg/mol of GO	8
2.4	Response of tapered fiber sensor with various concentrations of ethanol	8
2.5	Dynamic Response time	10
2.6	(a) Dynamic response with uncoated fiber, (b) Dynamic response with coated fiber	11
2.7	(a) Dynamic response with coated fiber, (b) Dynamic response with uncoated fiber	12
2.8	Dynamic response of a 365-nm CNT layer-coated fiber tip sensor towards various ethanol concentrations	13
2.9	Various thicknesses of Au for diverse ethanol concentrations in water	14
2.10	A 2D image of the electromagnetic field distribution inside a waveguide	18
2.11	Field behaviour as gold nanoparticle is attached to the waveguide surface	19
2.12	The applications of Pd	20
3.1	Workflow chart	24
3.2	Standard multi-mode fiber	25
3.3	Illustration of the dimensions of the tapered fiber	25
3.4	A Simplified diagram of the sensing setup	28

© COPYRIGHT UPM

3.5	SEM of Au-coating on MMF	29
3.6	SEM of Pd-coating on MMF	30
3.7	SEM of Pd and Au drop casted onto glass substrate	31
3.8	SEM of Pd and Au drop casted on to fiber surface	31
3.9	The EDX spectrum of the mixture of the gold and palladium with ratio of (1:1)	32
3.10	The EDX spectrum of the mixture of the gold and palladium with ratio of (1:2)	32
3.11	The EDX spectrum of the mixture of the gold and palladium with ratio of (1:3)	33
4.1	Comparison of absorbance levels of uncoated MMF sensor	35
4.2	Decrease in intensity with regard to different concentrations of ethanol	35
4.3	Absorbance with regard to five concentrations of ethanol	36
4.4	Dynamic response time for 20μm diameter uncoated	36
4.5	The spectra of the uncoated MMF sensors with a diameter of 30 μm	37
4.6	Absorbance with regard to different concentrations of ethanol	38
4.7	Dynamic response time for 30μm diameter uncoated	38
4.8	Intensity of the uncoated MMF sensors with a diameter of 40 μm	39
4.9	Absorbance with regard to different concentrations of ethanol	39
4.10	Dynamic response time for 40μm diameter uncoated	40
4.11	(A)Comparison of absorbance level of coated MMF sensor (B) plot of ethanol concentration against intensity	41
4.12	Optical spectra of absorbance with the Au film coating	42
4.13	Intensity spectra of Au-coated MMF sensor	43
4.14	Dynamic response of Au-coated MMF sensor towards various ethanol concentrations.	43
4.15	Absorbance spectra of Pd-coated MMF sensor	44

© COPYRIGHT UPM

4.16	Intensity spectra of Pd-coated MMF sensor	45
4.17	Dynamic response of Pd-coated MMF sensor with ethanol concentrations	45
4.18	Intensity spectrum for the mixture of (1:1) ratio	46
4.19	Absorbance spectrum for the mixture of (1:1) ratio	46
4.20	Dynamic response for the first concentration of a mixture of Pd and Au(1:1)	47
4.21	Intensity spectrum for the mixture with (1:2) ratio	48
4.22	Absorbance spectrum of the mixture with (1:2) ratio	48
4.23	Dynamic response for the first concentration of a mixture of Pd and Au(1:2)	49
4.24	(A) intensity spectrum for the mixture with (1:3) ratio (B) Absorbance spectrum of the mixture with (1:3) ratio	50
4.24	(C) Dynamic response for mixture with ratio of (1:3)	51
4.25	Absorbance spectrum for each concentration	52
4.26	Intensity spectrum of tapered fiber sensor towards ethanol concentrations	52
4.27	Dynamic response for the first concentration layer of a mixture of Pd and Au	53
4.28	Absorbance spectra of tapered fiber sensor towards ethanol concentrations	54
4.29	Intensity spectra of the tapered fiber sensor towards concentrations of ethanol	54
4.30	Dynamic reactions of tapered optical fiber sensor to ethanolconcentrations	55
4.31	Dynamic response of the mixture-coated MMF sensor with ratio (2:1) Gold to palladium	56

© COPYRIGHT UPM

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

© COPYRIGHT UPM

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

© COPYRIGHT UPM

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].

© COPYRIGHT UPM

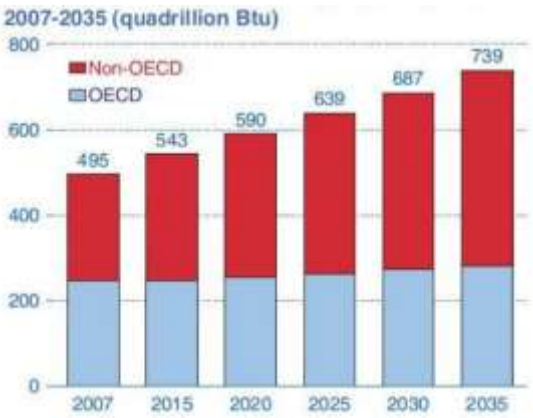

Figure 1.1 : World energy consumption projection[10]

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.

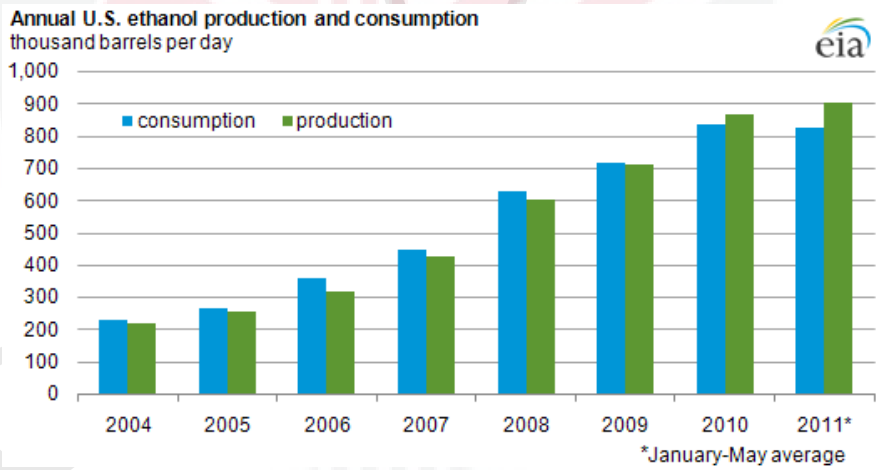

Figure 1.2 : Ethanol production from 2000 to 2012[10]

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.

© COPYRIGHT UPM

REFERENCES

[1] A. Ghatak, *Optics*, Fourth Edi. McGraw-Hill, 2009.

[2] G. Keiser, *Optical Fiber Communications*, Fourth. McGraw-Hill, 2008.

[3] R. Bharadwaj, V. V. R. Sai, K. Thakare, A. Dhawangale, T. Kundu, S. Titus, P. K. Verma, and S. Mukherji, "Evanescent wave absorbance based fiber optic biosensor for label-free detection of E. coli at 280nm wavelength," *Biosens. Bioelectron.*, vol. 26, no. 7, pp. 3367–3370, 2011.

[4] M. I. Zibaii, H. Latifi, Z. Saeedian, and Z. Chenari, "Nonadiabatic tapered optical fiber sensor for measurement of antimicrobial activity of silver nanoparticles against Escherichia coli," *J. Photochem. Photobiol. B Biol.*, vol. 135, pp. 55–64, 2014.

[5] H. J. Han, S. B. Riffat, S. H. Lim, and S. J. Oh, "Fiber optic solar lighting: Functional competitiveness and potential," *Sol. Energy*, vol. 94, pp. 86–101, Aug. 2013.

[6] D. Engles, S. Prashar, A. Singh, and M. T. Student, "Etched FBG as Chemical Sensor for Fuel Adultration," *Int. J. Eng. Res. Technol.*, vol. 1, no. 4, pp. 1–5, 2012.

[7] A. Shabaneh, S. Girei, P. T. Arasu, M. Mahdi, S. Rashid, S. Paiman, and M. Yaacob, "Dynamic Response of Tapered Optical Multimode Fiber Coated with Carbon Nanotubes for Ethanol Sensing Application," *Sensors*, vol. 15, no. 5, pp. 10452–10464, 2015.

[8] A. Aziz, H. N. Lim, S. H. Girei, M. H. Yaacob, M. A. Mahdi, N. M. Huang, and A. Pandikumar, "Chemical Silver / graphene nanocomposite-modified optical fiber sensor platform for ethanol detection in water medium," *Sensors Actuators B. Chem.*, vol. 206, pp. 119–125, 2015.

[9] Shaws Infrastructure and Environmental Group, "Large Volume Ethanol Spills-Environmental Impacts and Response Options," *MassDEP*, no. July, 2011.

[10] "Today In Energy," *US Energy Information Administration*, 2013. [Online]. Available: http://www.eia.gov/todayinenergy/detail.cfm?id=12251.

[11] P. . Arasu, A.S.M.Noor, and A. A. Shabaneh, "Absorbance properties of gold coated fiber Bragg grating sensor for aqueous ethanol," *J. Eur. Opt. Soc.*, vol. 9, p. 14018, 2014.

[12] D. L. McCorkle, R. J. Warmack, S. V. Patel, T. Mlsna, S. R. Hunter, and T. L. Ferrell, "Ethanol vapor detection in aqueous environments using micro-capacitors and dielectric polymers," *Sensors Actuators, B Chem.*, vol. 107, no. 2, pp. 892–903, 2005.

© COPYRIGHT UPM

[13] F. James, P. Breuil, C. Pijolat, M. Camara, D. Briand, a. Bart, and R. Cozic, "Development of a MEMS Preconcentrator for Micro-gas Chromatography Analyses," *Procedia Eng.*, vol. 87, pp. 500–503, 2014.

[14] S. Zampolli, I. Elmi, F. Mancarella, P. Betti, E. Dalcanale, G. C. Cardinali, and M. Severi, "Real-time monitoring of sub-ppb concentrations of aromatic volatiles with a MEMS-enabled miniaturized gas-chromatograph," *Sensors Actuators, B Chem.*, vol. 141, no. 1, pp. 322–328, 2009.

[15] J. L. Noel, R. Udayabhaskar, B. Renganathan, S. Muthu Mariappan, D. Sastikumar, and B. Karthikeyan, "Spectroscopic and fiber optic ethanol sensing properties Gd doped ZnO nanoparticles," *Spectrochim. Acta Part A Mol. Biomol. Spectrosc.*, vol. 132, pp. 634–638, 2014.

[16] P. . T. Arasu, "Development of Fiber Bragg Grating Based Ethanol Sensor Using Nanostructured Sensitive Layers," Universiti Putra Malaysia, 2016.

[17] T. Bora, H. Fallah, M. Chaudhari, T. Apiwattanadej, S. W. Harun, W. S. Mohammed, and J. Dutta, "Controlled side coupling of light to cladding mode of ZnO nanorod coated optical fibers and its implications for chemical vapor sensing," *Sensors Actuators, B Chem.*, vol. 202, pp. 543–550, 2014.

[18] Y. C. Tan, W. B. Ji, V. Mamidala, K. K. Chow, and S. C. Tjin, "Carbon-nanotube-deposited long period fiber grating for continuous refractive index sensor applications," *Sensors Actuators, B Chem.*, vol. 196, pp. 260–264, 2014.

[19] A. L. Chaudhari and A. D. Shaligram, "Simulation and experimental studies of an extrinsic fiber optic sensor for liquid refractometry," *Opt. - Int. J. Light Electron Opt.*, vol. 124, no. 17, pp. 3134–3137, Sep. 2013.

[20] M. A. A. Rosli, P. T. Arasu, H. N. Lim, and A. S. M. Noor, "Dynamic Response of Tapered Optical Fiber Coated with Graphene Oxide for Detecting Aqueous Ethanol," in *IEEE 6th International Conference on Photonics*, 2016, pp. 16–18.

[21] M. A. A. Rosli, P. T. Arasu, A. S. M. Noor, H. N. Lim, and N. M. Huang, "Reduced Graphene Oxide nano-composites layer on fiber optic tip sensor reflectance response for sensing of aqueous ethanol," *J. Eur. Opt. Soc. Publ.*, 2016.

[22] S. H. Girei, A. A. Shabaneh, H. N. Lim, N. M. Huang, M. A. Mahdi, and M. H. Yaacob, "Absorbance response of graphene oxide coated on tapered multimode optical fiber towards liquid ethanol," *J. Eur. Opt. Soc.*, vol. 15019, no. 10, pp. 1–5, 2015.

[23] H. Saad, M. K. Abd, Rahman, and M. T. Ali, "Tapered Plastic Optical Fiber Sensor for Detection of Ethanol Concentration in H2O," in *Seventh International Conference in Sensing Technology*, 2013, pp. 559–564.

© COPYRIGHT UPM

[24] R. K. Verma, A. K. Sharma, and B. D. Gupta, "Surface Plasmon Resonance Based Tapered Fiber Optic Sensor with Different Taper Profiles," *Opt. Commun.*, vol. 281, no. 6, pp. 1486–1491, 2008.

[25] H. Qiu, S. Gao, P. Chen, Z. Li, X. Liu, C. Zhang, Y. Xu, S. Jiang, C. Yang, Y. Huo, and W. Yue, "Evanescent wave absorption sensor based on tapered multimode fiber coated with monolayer graphene film," *Opt. Commun.*, vol. 366, pp. 275–281, 2016.

[26] Y. Hang Zhou, Q. Xue Guang, I. Md Rajibul, L. Kok-Sing, and H. Ahmad, "Simultaneous measurement of aliphatic alcohol concentration and temperature based on etched taper FBG," *Sensors Actuators, B Chem.*, vol. 202, pp. 959–963, 2014.

[27] S. Hu, Y. Zhao, and H. Hu, "Modeling and simulation of tapered fiber-optic oil concentration sensor using negative dielectrophoresis," *Sensors Actuators, B Chem.*, vol. 199, pp. 70–75, 2014.

[28] Y. Tian, W. Wang, N. Wu, X. Zou, and X. Wang, "Tapered optical fiber sensor for label-free detection of biomolecules.," *Sensors (Basel).*, vol. 11, no. 4, pp. 3780–90, Jan. 2011.

[29] N. Zhao, H. Fu, M. Shao, X. Yan, H. Li, Q. Liu, H. Gao, Y. Liu, and X. Qiao, "High temperature probe sensor with high sensitivity based on Michelson interferometer," *Opt. Commun.*, vol. 343, pp. 131–134, 2015.

[30] S. H. Girei, A. A. Shabaneh, P. T. Arasu, and M. H. Yaacob, "Tapered Multimode Fiber Sensor for Ethanol Sensing Application," in *International Conference on Photonics*, 2014, no. 2, pp. 2–4.

[31] F. B. Xiong and D. Sisler, "Determination of low-level water content in ethanol by fiber-optic evanescent absorption sensor," *Opt. Commun.*, vol. 283, no. 7, pp. 1326–1330, Apr. 2010.

[32] W. Cao and Y. Duan, "Optical fiber-based evanescent ammonia sensor," *Sensors Actuators, B Chem.*, vol. 110, no. 2, pp. 252–259, 2005.

[33] B. Schyrr, S. Pasche, E. Scolan, R. Ischer, D. Ferrario, J. A. Porchet, and G. Voirin, "Development of a polymer optical fiber pH sensor for on-body monitoring application," *Sensors Actuators, B Chem.*, vol. 194, pp. 238–248, 2014.

[34] A. Shalabney, A. Lakhtakia, I. Abdulhalim, A. Lahav, C. Patzig, I. Hazek, A. Karabchevsky, B. Rauschenbach, F. Zhang, and J. Xu, "Surface plasmon resonance from metallic columnar thin films," *Photonics Nanostructures - Fundam. Appl.*, vol. 7, pp. 176–185, 2009.

© COPYRIGHT UPM

[35] J. M. Corres, F. J. Arregui, and I. R. Matías, "Sensitivity optimization of tapered optical fiber humidity sensors by means of tuning the thickness of nanostructured sensitive coatings," *Sensors Actuators, B Chem.*, vol. 122, no. 2, pp. 442–449, 2007.

[36] R. Gravina, G. Testa, and R. Bernini, "Perfluorinated plastic optical fiber tapers for evanescent wave sensing.," *Sensors (Basel).*, vol. 9, no. 12, pp. 10423–33, Jan. 2009.

[37] J. Zhou, Y. Wang, C. Liao, B. Sun, J. He, G. Yin, S. Liu, Z. Li, G. Wang, X. Zhong, and J. Zhao, "Intensity modulated refractive index sensor based on optical fiber Michelson interferometer," *Sensors Actuators B Chem.*, vol. 208, pp. 315–319, 2015.

[38] J. Dai, M. Yang, Y. Chen, K. Cao, H. Liao, and P. Zhang, "Side-polished fiber Bragg grating hydrogen sensor with WO3-Pd composite film as sensing materials.," *Opt. Express*, vol. 19, no. 7, pp. 6141–8, Mar. 2011.

[39] H. A. Rahman, S. W. Harun, M. Yasin, S. W. Phang, S. S. A. Damanhuri, H. Arof, and H. Ahmad, "Tapered plastic multimode fiber sensor for salinity detection," *Sensors Actuators, A Phys.*, vol. 171, no. 2, pp. 219–222, 2011.

[40] M. H. Chiu and C. H. Shih, "Searching for optimal sensitivity of single-mode D-type optical fiber sensor in the phase measurement," *Sensors Actuators, B Chem.*, vol. 131, no. 2, pp. 596–601, 2008.

[41] Y. C. Tan, Z. Q. Tou, V. Mamidala, K. K. Chow, and C. C. Chan, "Continuous refractive index sensing based on carbon-nanotube-deposited photonic crystal fibers," *Sensors Actuators, B Chem.*, vol. 202, pp. 1097–1102, 2014.

[42] T. Kundu, V. V. R. Sai, R. Dutta, S. Titas, P. Kumar, and S. Mukherjee, "Development of evanescent wave absorbance-based fibre-optic biosensor," *Pramana*, vol. 75, no. 6, pp. 1099–1113, Jul. 2011.

[43] M. Holzki, H. Fouckhardt, and T. Klotzbücher, "Evanescent-field fiber sensor for the water content in lubricating oils with sensitivity increase by dielectrophoresis," *Sensors Actuators, A Phys.*, vol. 184, pp. 93–97, 2012.

[44] F. B. Xiong, W. Z. Zhu, H. F. Lin, and X. G. Meng, "Fiber-optic sensor based on evanescent wave absorbance around 2.7 μm for determining water content in polar organic solvents," *Appl. Phys. B*, Jul. 2013.

[45] D. R. Raj, S. Prasanth, T. V Vineeshkumar, and C. Sudarsanakumar, "Surface Plasmon Resonance based fi ber optic sensor for mercury detection using gold nanoparticles PVA hybrid," *Opt. Commun.*, vol. 367, pp. 102–107, 2016.

[46] B. a. Sexton, B. N. Feltis, and T. J. Davis, "Characterisation of gold surface plasmon resonance sensor substrates," *Sensors Actuators A Phys.*, vol. 141, no. 2, pp. 471–475, Feb. 2008.

© COPYRIGHT UPM

[47] M. Karlberg, “Scattering of gold nanoparticles in an evanescent waveguide field,” *Thesis, Dep. Chem. Univ. Technol.*, 2013.

[48] M. F. Bin Alam, D.-T. Phan, and G.-S. Chung, “Palladium nanocubes decorated on a one-dimensional ZnO nanorods array for use as a hydrogen gas sensor,” *Mater. Lett.*, vol. 156, pp. 113–117, 2015.

[49] K. Schroeder, W. Ecke, and R. Willsch, “Optical fiber Bragg grating hydrogen sensor based on evanescent-field interaction with palladium thin-film transducer,” *Opt. Lasers Eng.*, vol. 47, no. 10, pp. 1018–1022, 2009.

[50] C. L. Tien, H. W. Chen, W. F. Liu, S. S. Jyu, S. W. Lin, and Y. S. Lin, “Hydrogen sensor based on side-polished fiber Bragg gratings coated with thin palladium film,” *Thin Solid Films*, vol. 516, no. 16, pp. 5360–5363, 2008.

[51] C. Liewhiran and S. Phanichphant, “Effects of Palladium Loading on the Response of Thick Film Flame-made ZnO Gas Sensor for Detection of Ethanol Vapor,” *Sensors*, vol. 7, no. 7, pp. 1159–1184, 2007.

[52] R. Chen, X. Ruan, W. Liu, and C. Stefanini, “A reliable and fast hydrogen gas leakage detector based on irreversible cracking of decorated palladium nanolayer upon aligned polymer fibers,” *Int. J. Hydrogen Energy*, vol. 40, no. 1, pp. 746–751, 2014.

[53] M. Konstantaki, A. Klini, D. Anglos, and S. Pissadakis, “An ethanol vapor detection probe based on a ZnO nanorod coated optical fiber long period grating.,” *Opt. Express*, vol. 20, no. 8, pp. 8472–84, Apr. 2012.

[54] M. a L. dos Reis, F. Thomazi, J. del Nero, and L. S. Roman, “Development of a chemiresistor sensor based on polymers-dye blend for detection of ethanol vapor,” *Sensors*, vol. 10, no. 4, pp. 2812–2820, 2010.

[55] P. T. Arasu, A. S. M. Noor, A. A. Shabaneh, M. A. Mahdi, M. H. Yaacob, S. H. Girei, H. N. Lim, and H. A. Abdul Rashid, “Absorbance properties of gold coated fiber Bragg grating sensor for aqueous ethanol,” *J. Eur. Opt. Soc.*, vol. 14018, pp. 1–5, 2014.

[56] C. Bianchini and P. K. Shen, “Palladium-based electrocatalysts for alcohol oxidation in half cells and in direct alcohol fuel cells,” *Chem. Rev.*, vol. 109, no. 9, pp. 4183–4206, 2009.

[57] O. Deparis, P. Megret, M. Decreton, and M. Blondel, “The Radiation-Induced Absorption Band at 600 mn and its Impact in Fibroscopy 0.,” *IEEE Trans. Nucl. Sci.*, vol. 47, no. 3, pp. 693–698, 2000.

[58] W. Serrano, A. Melendez, I. Ramos, and N. J. Pinto, “Sensor response of electrospun poly(lactic acid)/polyaniline nanofibers to aliphatic alcohol vapors of varying sizes,” *2014 IEEE 9th Iberoam. Congr. Sensors, IBERSENSOR 2014 - Conf. Proc.*, pp. 1–4, 2014.