

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

SENSITIVITY ENHANCEMENT OF OPTICAL FIBER SENSOR FOR LIMONENE SENSING APPLICATIONS BY USING TAPERED OPTICAL



MASTER OF SCIENCE IN ELECTRONIC ENGINEERING



Faculty of Electronics and Computer Technology and Engineering

SENSITIVITY ENHANCEMENT OF OPTICAL FIBER SENSOR FOR LIMONENE SENSING APPLICATIONS BY USING TAPERED OPTICAL FIBER

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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Master of Science in Electronic Engineering

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DECLARATION

I declare that this thesis entitled "Sensitivity Enhancement of Optical Fiber Sensor for Limonene Sensing Applications by Using Tapered Optical Fiber" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Master of Science in Electronic Engineering.



DEDICATION

This thesis is dedicated to my loving and supportive parents, siblings and supervisor who helped me through this journey. Thanks for making me see this adventure journey to the end.



ABSTRACT

Evanescent wave sensing has led to significant advancements in various fields, including the development of optical fiber sensors (OFS). A simple and low-cost optical fiber sensor based on evanescent wave coupling of light into the core modes of plastic optical fiber (POF) coated with Zinc Oxide (ZnO) nanorods is reported here. The main objective of this research is to study the potential of optical sensing characteristics in the characterization of limonene concentration. Current detection methods for limonene sensing, which is one of a liver disease biomarker, lack sensitivity, and there is a need for a more accurate biomarker for early diagnosis. Study on the limonene concentration characterisation by using a fiber-optic sensor has not been conducted and published before. In this work, the sensor's performance was evaluated through changes in refractive index and light intensity. The study involved optimization of the sensor model, optical and electrical characterizations, and performance validations through mathematical analysis. The POF sensor's sensing region was successfully developed using chemical etching and hydrothermal synthesis. Two POF design parameters were varied: tapered waist diameter and sensing region length. The sensor's performance was evaluated using three different lengths: 1 cm, 2 cm, and 3 cm. The effect of sensor diameter differences was also investigated, which included a study of sensor diameters of 0.70 mm, 0.65 mm, 0.60 mm, 0.55 mm, 0.50 mm, and 0.45 mm. The experiment was also conducted using three different LEDs: blue, green, and red together with five different limonene concentrations of 20%, 40%, 60%, 80% and 100%. ZnO nanorods' growth which is horizontal and vertical oriented nanoparticles with time of 10 hours leads to the best sensitivity of the fiber sensors. The best sensor design was discovered to be a vertically oriented ZnO coated sensor with a 0.45 mm tapered waist diameter, a 3 cm sensing region length, and a red LED light source. The sensitivity obtained was 6.85V/RIU, with the linearity of 99%. Limit of detection and standard deviation achieved by 0.03% and 0.06V respectively. This research lays the foundation for further comprehensive assessments and advancements in the field of optical sensing for limonene sensing.

PENINGKATAN KEPEKAAN PENDERIA GENTIAN OPTIK UNTUK APLIKASI PENDERIAAN LIMONENA DENGAN MENGGUNAKAN GENTIAN OPTIK TIRUS

ABSTRAK

Penderiaan gelombang fana telah membawa kepada perkembangan di dalam pelbagai bidang, termasuk pembangunan penderia gentian optik. Penderia gentian optik yang mudah dan berkos rendah berasaskan gandingan cahaya gelombang fana ke dalam teras gentian optik plastik (POF) yang disalut dengan nanorod Zink Oxida (ZnO) dilaporkan di dalam kajian ini. Objektif utama kajian ini adalah bagi mengkaji potensi ciri-ciri penderia optik di dalam pencirian kepekatan limonen. Kaedah pengesanan sedia ada untuk mengesan limonen, salah satu penanda- bio bagi penyakit hati kekurangan ciri kepekaan, dan memerlukan penanda-bio yang lebih tepat bagi pengesanan awal. Kajian tentang pencirian kepekatan limonene dengan menggunakan sensor gentian optik belum pernah dijalankan dan diterbitkan sebelum ini. Di dalam kajian ini, prestasi penderia dinilai melalui perubahan indeks biasan dan keamatan cahaya. Kajian ini melibatkan pengoptimasian model penderia, pencirian optik dan elektrik, dan pengesahan prestasi melalui analisis matematik. Kawasan penderiaan penderia POF telah berjaya dibangunkan menggunakan punaran kimia dan sintesis hidrotermal. Dua parameter rekabentuk divariasi: lebar tirus dan panjang kawasan penderiaan. Prestasi penderia dikaji menggunakan tiga panjang yang berbeza: 1 cm, 2 cm dan 3 cm. Kesan perbezaan diameter penderia juga dinilai, dengan mengambilkira diameter 0.70 mm, 0.65 mm, 0.60 mm, 0.55 mm, 0.50 mm, dan 0.45 mm. Experimen ini juga dijalankan menggunakan tiga LED yang berbeza: biru, hijau dan merah dengan lima variasi limonen konsentrasi 20%, 40%, 60%, 80% dan 100%. Masa pertumbuhan 10 jam nanorod ZnO yang berorientasi menegak dan mendatar membawa kepada sensitiviti terbaik penderia gentian ini. Rekabentuk penderia terbaik adalah penderia bersalut ZnO berorientasi menegak dengan diameter lebar 0.45 mm, kawasan penderia 3 cm dan menggunakan sumber cahaya LED merah. Kepekaan yang diperolehi ialah 6.85V/RIU, dengan lineariti 99%. Had pengesanan dan sisihan piawai dicapai sebanyak 0.03% dan 0.06V. Penyelidikan ini meletakkan asas untuk penilaian dan kemajuan komprehensif lebih lanjut dalam bidang penderiaan optik bagi pengesanan limonen.

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TABLE OF CONTENTS

DE AP DE AB AC TA LIS LIS LIS LIS LIS LIS	CLAH PROV DICA STRA STRA KNO' BLE (5T OF 5T OF 5T OF 5T OF 5T OF	RATION VAL TION ACT AK WLEDGEMENTS OF CONTENTS OF CONTENTS 'TABLES 'TABLES 'FIGURES 'ABBREVIATIONS 'SYMBOLS 'APPENDICES 'PUBLICATIONS	i ii iii iv vii ix xiii xv xvi xvi xvii
CH	APTE	ER	4
1.		TRODUCTION	
	1.1	Background Study	1
	1.2	Objectives	
	1.3 1A	Scope of Work	4
	1.4	Thesis Organization	6
	1.5		0
2.	LIT	ERATURE REVIEW	7
	2.1	Introduction	7
	2.2	Fundemental concept of fiber optic	7
	2.3	Polymer Optical Fiber (POF)	10
	2.4	Optical fiber sensor configurations	11
		2.4.1 Cladding removed OFS	12
		2.4.2 Tapered Fiber	14
		2.4.3 Fiber Bragg grating	15
		2.4.4 Interferometers	18
	2.5	Fiber optic sensor	19
		2.5.1 Light scattering in OFS	20
	26	2.5.2 Refractive index change measurement	21
	2.0	Eiber entic costing	23 25
	2.1	7.1 Zinc Oxide as sensitive coating material	25 26
		2.7.1 Zine Oxide as sensitive coating indicital 2.7.2 ZnO growth techniques	20 27
	28	Tapered optical fiber for bio-sensing applications	30
	2.0	2.8.1 Enzyme detection	30
		2.8.2 Pathogens and pesticides detection	32
		2.8.3 Glucose level detection	34
		2.8.4 Protein detection	36
	2.9	Tapered optical fiber for limonene characterization	37
		2.9.1 Introduction to limonene	37
		2.9.2 Limonene applications	38

iv

	2.10	2.9.3 Summ	Limonen ary	he characterization for liver disease detection	40 42
3.	RES	EARC	Н МЕТН	IODOLOGY	43
	3.1	Introdu	uction		43
	3.2	Experi	mental M	aterial and Equipment	43
	3.3	Sensor	probe fal	brication	46
	3.4	ZnO co	oating by	hydrothermal technique	48
		3.4.1	Fiber pro	bbe preparation	50
		3.4.2	Seeding	process	51
		3.4.3	Growth	process	55
	3.5	Experi	mental se	tup for the sensor	56
	3.6	Receiv	ver circuit	design	58
	3.7	Result	analysis r	performance	61
	3.8	Summ	ary		64
4.	RES	ULT A	ND DIS(CUSSION	65
	4.1	Introdu	uction		65
	4.2	ZnO G	browth Per	rformance Analysis	65
	4.3	Perform	mance ana	alysis of tapered fiber sensor	69
		4.3.1	Performa	ance analysis of tapered fiber sensor using blue	
		S	LED		69
		N.	4.3.1.1	Performance analysis of horizontally oriented	
		F		ZnO growth fiber sensor using blue LED	69
		E	4.3.1.2	Performance analysis of vertically oriented	
		2		ZnO growth fiber sensor using blue LED	74
			4.3.1.3	Performance summary of a tapered fiber sensor	
		1.	1 (with blue LED	77
		4.3.2	Performa	ance analysis of tapered fiber sensor using green	
			LED.		78
		UNI	4.3.2.1 VERSI	Performance analysis of horizontally oriented ZnO growth fiber sensor using green LED	78
			4.3.2.2	Performance analysis of vertically oriented	
				ZnO growth fiber sensor using green LED	81
			4.3.2.3	Performance summary of a tapered fiber sensor	
				with green LED	83
		4.3.3	Performa	ance analysis of tapered fiber sensor using red	
			LED		85
			4.3.3.1	Performance analysis of horizontally oriented	
				ZnO growth fiber sensor using red LED	85
			4.3.3.2	Performance analysis of vertically oriented	
				ZnO growth fiber sensor using red LED	86
			4.3.3.3	Performance summary of a tapered fiber sensor	
				with red LED	87
	4.4	Compa	arison of s	sensor's sensitivity for 0.45mm tapered waist	88
	4.5	Summ	ary		92
5.	CON	ICLUS	ION ANI	D FUTURE WORK	93
	5.1	Conclu	ision		93
	5.2	Future	works an	d recommendations	94
				V	

REFERENCES APPENDICES

96 110



LIST OF TABLES

TABLE	TITLE	PAGE
2.1	Comparison of tapered optical fiber results in enzyme application	32
2.2	Comparison of tapered optical fiber results in glucose application	35
2.3	Chemical and physical properties of limonene	38
2.4	Limonene Level in healthy, Cirrhosis, and HCC patients	42
3.1	Experimental materials	43
3.2	List of Equipment	44
4.1	1 cm sensing region with horizontal oriented ZnO coated fiber sensor	73
	performance towards limonene concentration using blue LED	
4.2	2 cm and 3 cm sensing region with horizontal oriented ZnO coated fiber	74
	sensor performance towards limonene concentration using blue LED	
4.3	1 cm, 2 cm and 3 cm sensing region with vertical oriented ZnO coated	76
	fiber sensor performance towards limonene concentration using blue LED)
4.4	1 cm, 2 cm and 3 cm sensing region with horizontal oriented ZnO coated	81
	fiber sensor performance towards limonene concentration using green	
	LED	
4.5	1 cm, 2 cm and 3 cm tapered with vertical oriented ZnO fiber sensor	83

performance towards limonene concentration using green LED

- 4.6 1 cm, 2 cm and 3 cm sensing region with horizontal oriented ZnO coated 86 fiber sensor performance towards limonene concentration using red LED
- 4.7 1 cm, 2 cm and 3 cm sensing region with vertical oriented ZnO coated 87 fiber sensor performance towards limonene concentration using red LED

92

4.8 Comparison of previous works



LIST OF FIGURES

FIGURE TITLE

PAGE

1.1	Research Focus	6
2.1	(a) optical fiber layers and (b) cross-section of optical fiber	8
2.2	Light propagation in fiber optic	9
2.3	General construction of optical fiber	11
2.4	Illustration of OFS with removed cladding	12
2.5	Illustration of variation OFS (a) modified partially	13
	cladding removal OFS (b) U-shape OFS	
2.6	Schematic illustration of the flame approach used for the	
	fabrication of tapered OFS	15
2.7	FBG structure and their corresponding output spectra	17
2.8	Schematic illustration of an optical fiber modal Mach-Zehnder	19
	interferometer setup	
2.9	Schematic diagram of basic OFS	21
2.10	Refractive index based on modified sensitive material	22
2.11	SEM images of Zinc Oxide nanoparticles (a) Nanoflowers	29
	(b) Nanowires (c) Nanorods	
2.12	Chemical structure of Limonene	37
2.13	Limonene therapeutic effects	40

3.1	Diagram of (a) POF cladding removal, (b) tapered POF and (c)	46
	ZnO coating deposition	
3.2	Flowchart of probe fabrication optimization	47
3.3	Image from microscope (a) Un-tapered POF (b) Tapered POF	48
3.4	Flowchart of hydrothermal method for ZnO synthesis process	49
3.5	Fiber preparation process (a) Fiber with jacket (b) Fiber exposed	51
	with 2cm length (c) tapered POF with micrometer	
3.6	The steps involved in the seeding of ZnO nanoparticles	52
3.7	Process of 2mM of Zinc Acetate Dihydrate	52
3.8	Alkaline process of ZnO nanoparticles solution by NaOH	53
3.9	(a) Tween 80 preparation (b) POF surface treatment	54
3.10	Dip and Dry techniques for seeding solution	54
3.11	The process ZnO growth on tapered POF (vertical)	55
3.12	The process ZnO growth on tapered POF (horizontal)	56
3.13	The real setup between PD and LED	57
3.14	A block diagram of the experimental setup for limonene	57
	concentration characterization MALAYSIA MELAKA	
3.15	The experimental setup for electrical characterization	58
3.16	Circuit diagram of Transimpedance Amplifier	60
3.17	Illustration of straight-line equation	62
4.1	(a) SEM image of horizontally oriented ZnO NPS deposited	66
	POF (b) magnified SEM view of coated sensing region (c) EDS	
	image of ZnO NPs, for 10 hours growth time	
4.2	EDS element mapping of the 10 hours horizontally oriented ZnO	67
	growth for POF coating	
4.3	(a) SEM image of vertically oriented ZnO NPS deposited POF	68
	(b) magnified SEM view of coated sensing region (c) EDS image	
	Х	

of ZnO NPs, for 10 hours growth time

4.4	EDS element mapping of the 10 hours vertically oriented ZnO	68
	growth for POF coating	
4.5	Response of voltage output against limonene concentration	71
	percentage using blue LED with horizontal oriented ZnO growth	
	for 1 cm tapered sensing region	
4.6	Response of voltage output against refractive index using blue	72
	LED with horizontal oriented ZnO growth for 1 cm tapered	
	sensing region	
4.7	Response of voltage output against limonene concentration	75
	percentage using blue LED with vertical oriented ZnO growth	
	for 1 cm tapered sensing region	
4.8	Response of voltage output against refractive index using blue	75
	LED with vertical oriented ZnO for 1cm tapered sensing region	
4.9	Performance of blue LED-powered horizontally oriented ZnO	77
	اونيوبرسيني تيڪنيڪ coated fiber sensor	
4.10	Performance of blue LED-powered vertically oriented ZnO	78
	coated fiber sensor	
4.11	Response of voltage output against limonene concentration	79
	percentage using green LED with horizontal oriented ZnO	
	growth for 1 cm tapered sensing region	
4.12	Response of voltage output against refractive index using green	80
	LED with horizontal oriented ZnO growth for 1 cm tapered	
	sensing region	
4.13	Response of voltage output against limonene concentration	82
	percentage using green LED with vertical oriented ZnO growth	

for 1 cm tapered sensing region

4.14	Response of voltage output against refractive index using green LED	82
	with vertical oriented ZnO growth for 1 cm tapered sensing region	
4.15	Performance of green LED-powered horizontally oriented ZnO	84
	coated fiber sensor	
4.16	Performance of green LED-powered vertically oriented ZnO	84
	coated fiber sensor	
4.17	Performance of red LED-powered horizontally oriented ZnO	88
	coated fiber sensor	
4.18	Performance of red LED-powered vertically oriented ZnO	88
	coated fiber sensor	
4.19	The sensitivity for 0.45 mm tapered waist sensor for uncoated,	90
	horizontal oriented ZnO growth and vertical oriented ZnO growth	
4.20	The sensitivity of 0.45mm tapered waist diameter with 3 cm	91
	sensing region optical sensor using three different LEDs	
	"SAINO	
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LIST OF ABBREVIATION

Au	-	Gold
BW	-	Bandwidth
CYTOP	-	Amorphous fluorinated polymer
DI		De-ionized
EW	-3	Evanescent Wave
FBG	TER	Fiber Bragg Grating
GBP	E	Gain Bandwidth
GO	- 33	Graphene Oxide
LCD	-sh	Liquid Crystal Display
LED	-	Light Emitting Diode
LOD	UNI	Limit of DetectionNIKAL MALAYSIA MELAKA
LPSR	-	Localized Surface Plasmon Resonance
MI	-	Michelson Interferometer
MMF	-	Multimode Fiber
MZI	-	Mach Zehnder Interferometer
NA	-	Numerical Aperture
OFS	-	Optical Fiber Sensor
OSA	-	Optical Spectrum Analyzer
OTDR	-	Optical Time Domain Reflectometry
PANI-ZnC)-	Polyaniline-Zinc Oxide
PD	-	Photodetector
PMMA	-	Polymethyl Methacrylate
		XIII

POF	-	Polymer Optical Fiber
PS	-	Polystyrene
RI	-	Refractive Index
SD	-	Standard Deviation Error
SEM	-	Surface Electron Microscope
SI	-	Saganc Interferometer
SMF	-	Single Mode Fiber
Sn	-	Stannum
SUT	-	Sample under Test
TIA	-	Transimpedance Amplifier
UA	-	Uric Acid
UV	-	Ultraviolet
VLS	- 8	Vapor–Liquid–Solid
ZIF-8	K	Zeolitic Imidazolate Framework-8
ZnO	TT ITTES	Zinc Oxide
	لك	اونيومرسيتي تيكنيكل مليسيا مل
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LIST OF SYMBOLS

η	-	Efficiency
λ	-	Wavelength
θ	-	Angle of Incidence
n	- 3	Refractive Index
C_{f}	TEKN	Feedback Capacitor
E_x	FIG	Evanescent Field
d_p	-	Penetration depth
δn _{eff}	2	ويورسيني Different between refractive indices
	UNI	VERSITI TEKNIKAL MALAYSIA MELAKA

LIST OF APPENDICES

APPENDI	X TITLE	PAGE
А	Fiber Optic Photodiode Datasheet	110
В	Limonene Datasheet	111
С	Acetone Datasheet	112
D	Acetate Dihydrate Datasheet	113
E	Sodium Datasheet	114
F	Zinc Nitrate Hexahydrate Datasheet	115
G	Hexamethylenetetramine Datasheet	116
Н	Ethyl Alcohol Datasheet	117
Ι	Hexane Datasheet	118
J	Tween 80 Datasheet	119

LIST OF PUBLICATIONS

- 1. Thanigai Anbalagan, Hazura Haroon, Hazli Rafis Abdul Rahim, Siti Halma Johari, Siti Khadijah Idris, Hanim Abdul Razak, Maisara Othman. 2023. A novel examination of limonene detection using plastic fiber optic sensors and the tapered approach. *Bulletin of Electrical Engineering and Informatics*, 12 (2), pp. 807-814.
- Thanigai Anbalagan, Hazura Haroon, Huda Adnan Zain, Hazli Rafis and S.W. Harun. 2022. Recent Progress of Tapered Optical Fiber for Biosensing Applications. *Journal* of Engineering Science and Technology Review, 15 (5), pp. 204 – 209.
- Hazura Haroon, Siti Noraminah Nordin, Hazli Rafis Abdul Rahim, Thanigai Anbalagan, Maisara Othman. 2022. Examination Of Adulterated Coconut Oil By Fiber Optics Displacement Sensor Using Lateral Offset Approach. *Jurnal Teknologi*, 84:5, pp. 185–190.
- 4. H Haroon, SN Nordin, T Anbalagan, M Othman. 2022. Edible oils adulteration analysis by fiber optic multimode displacement sensor. *Optoelectronics and Advanced Materials Rapid Communications*, 16, 1-2, January-February, pp.36-40 (2022).
- 5. Thanigai Anbalagan, Hazura Haroon, Hazli Rafis Abdul Rahim and Siti Halma Johari. 2022. Examination for limonene concentration using tapered plastic optical fiber sensor. *Journal of Physics: Conference Series 2411*, 012010 Photonic Meeting 2022.

CHAPTER 1

INTRODUCTION

1.1 Background Study

Evanescent wave study has brought tremendous advancements in many fields, which has opened the door to new research lines and possibilities. There has been a lot of research into optical fiber sensor (OFS) in recent times, due to its excellent performance and cost reduction. The trend has been driven by many telecommunications fields that resulted in a firmer penetration of fiber-optic sensors into various industrial, medical, environmental, power, and protection applications.

The primary goal of this study is to evaluate the potential of evanescent wave sensing as a non-invasive technique for limonene concentration characterisation, which is one of the biomarkers for liver disease detection. As liver disease is difficult to detect, researchers have found that the limonene compound in individuals diagnosed with liver disease is higher than in healthy individuals. Recently, typical biomarkers, such as isoprene, acetone, and ethanol were employed for liver disease detection. However, the technique was found to be insufficiently specific since they can be biomarkers for other types of disorder or produced by a variety of normal metabolic processes. Therefore, there is a need for an extensive research to establish an accurate, precise, specific, and sensitive non-invasive biomarker for early liver disease diagnosis.

This project proposed a development of fiber-optic sensor as an alternative to the bio-sensor, and subsequently explored the potentials of fiber-optic sensor for limonene characterisation. The fiber-optic sensing mechanism is the basis for the sensor development.