# CHARACTERIZATION OF ZINC OXIDE COATED POLYMER OPTICAL FIBER FOR LIMONENE CONCENTRATION MEASUREMENT

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### NOR ASYIQIN BINTI AZHAR



2023

# DECLARATION

I declare that this report entitled "Characterization of Zinc Oxide Coated Polymer Optical Fiber for Limonene Concentration Measurement" is the result of my own work except for quotes as cited in the references.



Date : 23 JANUARY 2023

# 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 Bachelor of Electronic Engineering with



Date : 23 JANUARY 2023

# **DEDICATION**



### ABSTRACT

Limonene concentration detection based on ZnO coated polymer optical fiber (POF) by using the intensity modulation technique was proposed. Zinc oxide was prepared by using a hydrothermal method and to prove the success of ZnO coating, the POF surface morphology was observed by using Scanning Electron Microscope (SEM). The POF was tapered to a certain diameter by using the chemical etching method. The tapered waist diameters of the POF are 0.45 mm, 0.50 mm, 0.55 mm, 0.60 mm, 0.65 mm, and 0.70 mm at 10 cm fiber length, and the unclad length of 2 cm was coated with ZnO using the dip-coating method. The refractive index for different concentrations of limonene solution was studied in this project. It is observed that as the limonene concentration increase from 20% to 100%, the output voltage will decrease. The higher sensor's sensitivity was recorded at 0.205 V/%, and the slope has more than 99%, linearity for the 0.55 mm tapered POFs, respectively. Furthermore, the refractive index varied as the concentration of limonene changed.

## ABSTRAK

Pengesanan kepekatan limonene berdasarkan gentian optik polimer bersalut ZnO (POF) dengan menggunakan teknik modulasi intensiti telah dicadangkan. Zink oksida telah disediakan dengan menggunakan kaedah hidroterma dan untuk membuktikan kejayaan salutan ZnO, morfologi permukaan POF telah diperhatikan dengan menggunakan Scanning Electron Microscope (SEM). POF ditiruskan kepada diameter tertentu dengan menggunakan kaedah etsa kimia. Diameter pinggang tirus POF ialah 0.45 mm, 0.50 mm, 0.55 mm, 0.60 mm, 0.65 mm, dan 0.70 mm pada panjang gentian 10 cm, dan panjang 2 cm yang tidak bersalut disalut dengan ZnO menggunakan kaedah salutan celup. Indeks biasan untuk kepekatan larutan limonene yang berbeza telah dikaji dalam projek ini. Adalah diperhatikan bahawa apabila kepekatan limonene meningkat daripada 20% kepada 100%, voltan keluaran akan berkurangan. Kepekaan sensor yang lebih tinggi telah direkodkan pada 0.205 V/%, dan cerun mempunyai lebih daripada 99%, lineariti untuk POF tirus 0.55 mm, masing-masing. Tambahan pula, indeks biasan berubah apabila kepekatan limonene berubah.

## ACKNOWLEDGEMENTS

I would like to praise and appreciate Allah Subhanahuwata'ala for giving me the time and strength during completing this Final Year Project.

I want to express my high appreciation and deepest gratitude to my supervisor, Dr. Hazura Binti Haroon for giving me the opportunity and trust, and also, I would like to thank her for her invaluable guidance, encouragement, and idea during completing this project.

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Other than that, I would like to acknowledge with much appreciation to all my friends for their help all time during the experiment and for giving full support and recommendations throughout the process of completing it. Without her guidance and kindness, this project might not have been done on time.

Lastly, many thanks to my family for their support and faith in me by spending more time and money on this project. Same goes for my friends who help to assist me by giving me some ideas and supporting each other weaknesses during completing this project.

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# LIST OF SYMBOLS AND ABBREVIATIONS



## **CHAPTER 1**

## **INTRODUCTION**



# Plastic Optical Fiber, also known as POF is very useful, particularly for data transmission from one location to another. POF has been widely used because of its advantages such as high flexibility in bending, low cost, and high sensitivity[1]. For this project, POF was used as a sensor to detect the limonene concentration. At high doses, limonene can cause human diseases such as liver disease, breast cancer, and others. The use of a fiber as a sensor is a non-invasive method for detecting limonene inside the human body

The performance of the POF sensor can be increased by tapered the fiber at a certain length[2]. The waist diameter of the fiber was reduced so that a high portion of the evanescent field can travel inside the fiber[3]. ZnO is frequently used in sensing applications to enhance the sensing capability of the sensor. This is due to the high

sensitivity of ZnO after the manual dip-coating technique ZnO [4]. ZnO has a direct wide bandgap of 3.37 eV, making it appealing for short-wavelength light-emitting devices [5]. There are many advantages of using ZnO as a sensing material such as high transmittance, good electrical conductivity, non-toxic and low cost[6]. As a result, ZnO has a wide range of applications, including biosensors, photodetectors, gas sensors, and many others [7]. Limonene or also known as d-limonene, l-limonene, and dl-limonene has been widely used as a flavor or fragrance for food and perfumes. At room temperature, limonene is a clear, colorless liquid [8]. This limonene can also be absorbed by humans and other mammals. It will quickly disperse in various organs and be converted into active metabolites [9].

### **1.2 Problem Statement**

Limonene has been used for many years as a flavor in foods and beverages. In previous studies, it was reported that limonene at high doses caused several effects on human beings. Some experiments have been done on volunteers to dissolve gallstones by infusing limonene directly into the bile system, and pain in the upper abdomen, nausea, vomiting, and diarrhea was reported associated with increases in serum aminotransferase and alkaline phosphatase. To detect the limonene inside a human body, the equipment that has been used is quite expensive. To address this issue, this project employs a low-cost material called fiber optics. The goal of this study is to create a low-cost, high-sensitivity, and simple optical sensing technology for limonene concentration characterization, which is a bio-marker for the early detection of liver disease.

### 1.3 **Objectives**

- a) To design a polymer optical fiber (POF) based sensor for limonene concentration characterization.
- b) To enhance sensing capability of ZnO coated POF by hydrothermal method.
- c) To analyze the sensor's performance for limonene detections in terms of sensitivity and linearity.

### **1.4** Scope of Work

For this project, the type of fiber that can be used is Polymer Optical Fiber (POF). Zinc oxide (ZnO) acts as a sensitive solution that used to be coated with POF. The method used to prepared ZnO is a hydrothermal method and to prove the successful coating of ZnO into the POF by using Scanning Electron Microscope (SEM). Some of the components used to estimate the output value for the limonene concentration from the photodetector are red LED, POF sensor, receiver circuit, and LCD to display the output. The tapered waist diameter of the POF is 0.45 mm, 0.50 mm, 0.55 mm, 0.60 mm, 0.65 mm, and 0.70 mm at the fiber length of 10 cm, and the unclad length of 2 cm was coated with ZnO.

### 1.5 Significant and Important

- a) One of the benefits of using fiber as a based sensor for concentration characterization is low cost, easy to handle, and also good flexibility.
- b) Many applications can be done using POF as a sensor-based that is coated with specific materials which are used in humidity measurement, detection of gas, and liquid levels.

### **1.6** Thesis Organization

Chapter 1 explained about introduction which includes the project description, problem statement, objective, scope of work, and significance and importance of this project. Chapter 2 explained the research results from a previous study that is relevant to this project. Some theoretical explanations related to this project have also been discussed in this chapter. Chapter 3 explained the whole process of the implementation to achieve the objective. All the equipment and the material used were discussed in detail. The experiment process of the tapered POF, ZnO coating by using a hydrothermal method, and the experimental setup for the sensor were explained in detail. All the result and the analysis of this project was explained in chapter 4. Chapter 5 was a summary of the whole project and suggestions for future work.

### 1.7 Summary

This chapter provides a description of the project, the main objectives, the scope of work for the whole process of the project, the thesis organization, and the significance and importance of this project.

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# **CHAPTER 2**

# LITERATURE REVIEW



This chapter contains the research results from a previous study that is relevant to this project. These studies also serve as the primary source of information, with the theoretical, methodology, and interpretation of the studies assisting in the support of the project's material.

### 2.1 Limonene Characterization

Limonene is normally found in orange peels, lemon, and mint. It is colorless and clear and be classified as cyclic monoterpene that occurs naturally in nature. It contains 90-95% orange peel oil and 75% lemon peel oil [10]. Limonene can be absorbed quickly into the human body. It will transform it into an active metabolite that can be present in serum, liver, lung, and various parts of the human body[9].



Figure 2.1 Chemical Structure of limonene.

In past research, some experiment has been done to determine the limonene in adipose tissue by using a gas chromatography-mass spectrometer (GC-MS). GC-MS was chosen for the experiment because it can measure the trace level of volatile, organic molecules, and this molecule will be considered a "gold standard." This equipment is sensitive, accurate, and precise for the quantification of limonene in adipose tissue. The sample of adipose tissue will be spiked with 30  $\mu$ L of d-limonene calibration working standard. It will be incubated in the water bath at a temperature of 37°C for about 2 hours and 30 minutes with 200  $\mu$ L of 30% potassium hydroxide. Then the sample will be analyzed using GC-MS at 300°C and held for about 5 minutes[11]. The result of the mass spectra of d-limonene from past research is shown in Figure 2.2.



Figure 2.2 Mass spectra of d-limonene.

### 2.2 Polymer Optical Fiber (POF)

POF is made of polymers including polymethylmethacrylate (PMMA) and the core diameter and step-index of the commercial POF with typical core and cladding are 1.49 and 1.41, respectively[12]. Plastic Optical Fiber also known as POF have a lot of benefits such as flexibility, high fracture, and high sensitivity[1]. POF also can make a sensor to measure the distance from one place to another, location, color, brightness, and many more[13]. Some basic structures of POF with core, cladding, and coating (buffer), as shown in Figure 2.3. The process of tapering the plastic optical fiber is at the core.



Figure 2.3 Basic structure of POF.

POF fiber must be tapered at a certain diameter to improve its performance. Fabrication of the fiber can be accomplished using acetone, de-ionized water, and sandpaper. Because the fiber is tapered, the power of the evanescent wave (EW) inside the POF cladding is increased when the diameter of the tapered POF is decreasing. Tapered POF is good to make as a sensing application especially when it is coated with appropriate material that can make it enhance at a correct thickness. Some of the optical parameters for this optical sensor to detect are refractive index (RI), absorbance, reflectance, and fluorescence depending on the application of the fiber[14]. When the tapered fiber is tapered at a good diameter, it can produce good sensitivity. From the last research, a diameter of 0.50 mm and below shows good performance compared to a diameter above 0.50 mm[2].

### 2.3 Zinc Oxide (ZnO)

Zinc oxide (ZnO) has unique characterization for physical and chemical properties. ZnO has good absorption of radiation and also high chemical stability. For use in short-wavelength optoelectronic applications, ZnO is a desirable material because has a direct bandgap of 3.3 eV at 300K, high transmittance, and good electrical conductivity which make ZnO widely used for sensing applications. Gas sensors, biosensors, photodetectors, and many other applications have been developed[15]. According to the previous article, many techniques such as thermal evaporation, laser ablation, sol-gel, hydrothermal, mechanical milling, and others can be used to synthesize ZnO [7]. There is various type of method to produce a ZnO which is, the hydrothermal method. Sol-gel method, vapor deposition, mechanochemical process, and many more. This method can produce different types of particles, sizes, and shapes [16]. The physical properties of ZnO was shown in Table 2.1 below.

Property	Value
Molecular formula	ZnO
Molar mass	81.4084 g/mo1
Appearance	Amorphous white or yellowish white powder.
Odour	Odourless
Density	$5.606 \text{ g/cm}^3$
Melting point	1975 °C
Boiling point U	2360 °C
Solubility in water SITI TEKNIKAL N	0.16 mg/100 mL MELAKA
Refractive index	2.0041
Lattice Constants	$a_0 = 0.32469 \text{ Å}$ $c_0 = 0.52069 \text{ Å}$
Relative Dielectric Constant	8.66
Energy Gap	3.4 eV Direct
Intrinsic Carrier Concentration	< 10 <sup>6</sup> /cc
Exciton Binding Energy	60 meV
Electron effective mass	0.24
Electron mobility (at 300 K)	200 cm <sup>2</sup> /V.sec.
Hole Effective mass	0.59
Hole mobility (at 300 K)	5-50 cm <sup>2</sup> /V.sec

Table 2.1: Physical properties of ZnO

Most of the techniques before are not used on a large scale. From the previous research, there are a few chemical methods and characterization of ZnO by using X-ray diffraction, scanning electron microscopy (SEM), transmission electron microscopy (TEM), selected area electron diffraction (SAED), UV-Vis absorbance, and photoluminescence spectra[17]. In this experiment, ZnO will act as a sensitive solution that has been coated to the fiber to enhance the sensor's sensitivity for the limonene concentration detection.

### 2.4 Previous Work

A comparison between the previous work related to the project was shown in Table 2.2 below. Most of the previous work used ZnO as a sensitive solution coated with POF to act as a sensing device and the type of fiber used is a plastic optical fiber. From the comparison table, there are many types of ZnO-coated techniques have been studied and explored.

No	Author	I TEKTILEKAL M	IAL	AYSMethodELAKA	N	Main Finding
	A.R.A.	ZnO Coated	•	POF coated with	•	The
	Rashid,	Optical Fiber for		ZnO.		absorbance
	A.N.A. Latiff,	Alcohol Sensing	•	ZnO is synthesized		and
	W.M.	Applications.		sonochemically by		transmittance
	Mukhtar,			using the bath-type		spectra for
1	N.A.M. Taib,			sonicator.		ZnO
1	S. Suhaimi		•	The optical		nanoparticles.
	and K.A.			properties of ZnO	•	The
	Dasuki[4]			are determined by		reflectance
				using an		and refractive
				ultraviolet-visible		index of ZnO
				(UV- Vis)		nanoparticle.

Table 2.2: Comparison of previous work.

			spectrophotometer.	٠	Output power
					reading for 20
					mm and 25
					mm length for
					ZnO coated
					POF under
					ethanol and
					methanol
					solution.
	Zuraidah	ZnO nanorod	• POF coated with	٠	Output
	Harith,	coated tapered	ZnO.		voltage
	Malathy	plastic optical	• ZnO nanorods		against
	Batumalay,	fiber sensors for	synthesized using		relative
	Ninik Irawati,	relative humidity.	hydrothermal		humidity for
	Sulaiman	AKA	method.		the proposed
	Wadi Harun,		• The		tapered POF
	Harith Ahmad,		characterization of		with ZnO
	Taoping		the fiber was done		nanorods.
2	Hu[18]	1.15:5	using Field	•	The
	2)*****		Emission		comparison of
	UNIVERSI	I TEKNIKAL N	Scanning Electron		performances
			Microscope		for sensitivity
			(FESEM)		and linearity
					between sol-
					gel and
					hydrothermal
					method for
					growing ZnO.
	A. R. A.	Zinc oxide coated	• POF coated with	•	UV-Vis the
	Rashid, N. A.	polymer optical	ZnO.		absorbance
3	F. Shamsuri,	fiber for	• Zinc oxide was		spectrum of
	A. H. Surani,	measuring uric	prepared using sol-		ZnO
	A. A. N.	acid	gel method.		nanoparticles.

	Hakim, K.	concentrations	• ZnO characterized	٠	Transmittance
	Ismail[19]		using UV-Vis		spectra of
			spectrometer in		undoped ZnO.
			order to determine	•	Refractive
			its optical properties		index against
			such as		wavelength
			transmittance,		for undoped
			absorbance and		ZnO.
			refractive index.	•	Power output
					ratio against
					concentration
					ofuric acid for
	1 AVO				ZnO coated
	at MACATO	A NO.			and uncoated
		Let a			POF at 0.02 m
		×			and 0.04 m
	Ella III				length.
	M. Winn	Tapered plastic	• POF coated with	٠	Refractive
	Batumalay,	optical fiber	graphene.		index of the
	S. W. Harun,	coated with	• Produce graphene		uric acid
	F. Ahmad,	graphene for uric	flakes using the		solution.
	R.M. Nor,	acid detection	electrochemical	•	The output
	N.R.		exfoliation process.		voltage
	Zulkepely				against uric
4	and H.				acid
	Ahmad[6]				concentrations
					for the
					proposed
					tapered POF
					based sensor
					without and
					with graphene
					polymer

						composite of
						2:20, 4:20 and
						6:20.
					•	The
						performance
						of the
						proposed uric
						acid detection
						sensor for
						sensitivity,
						linearity,
						standard
	ALAYS					deviation and
	At MARCING	A 140				limit of
		A.K.				detection.
	Malathy	Tapered plastic	•	POF coated with	٠	Output
	Batumalay,	optical fibe		HEC/PVDF.		voltage
	Asiah	coated with	•	HEC/PVDF is		against RH
	Lokman,	HEC/PVDF fo		prepared by mixed		for the
	Fauzan	measurement o		the PVDF powder		proposed
	Ahmad, ERSI	relative humidity	MAL	with DMF at 90°C		tapered POF
	Hamzah Arof,			in water bath then		based sensor
	Harith			cooled down to		with and
5	Ahmad, and			room temperature.		without
5	Sulaiman			Then, mix the		HEC/PVDF
	Wadi			solution with HEC		composite.
	Harun[20]			for 10 hours.	٠	Performance
			•	HEC/PVDF		of Humidity
				composite solution		Sensor such
				slowly dropped		as
				onto the tapered		sensitivity,
				region of the POF		linearity, std
				using syringe		deviation,

					and limit of
					detection
					with and
					without
					HEC/PVDF
					within the
					relative
					humidity
					range from
					55% to 80%
					composite.
	M.	Tapered plastic	• POF coated with	٠	Output
	Batumalay, Z.	optical fiber	ZnO.		voltage
	Harith, H.A.	coated with ZnO	• Zinc oxide was		against uric
	Rafaie, F.	nanostructures for	prepared using sol-		acid
	Ahmad, M.	the measurement	gel method.		concentratio
	Khasanah,	of uric acid	• The		ns for the
	S.W. Harun,	concentrations and	characterization of		proposed
	R.M. Nor, H.	changes in relative	the fiber was done		tapered POF
	Ahmad[14]	humidity	using Field		with seeded
	UNIVERSI	TI TEKNIKAL IV	Emission Scanning		and non-
6			Electron		seeded ZnO
			Microscope		nanostructur
			(FESEM)		e.
				•	The
					performance
					of the
					proposed
					uric acid
					detection
					sensor such
					as
					sensitivity,

				linearity, std
				deviation
				and limit of
				detection.
	B Mulyanti1,	Optical Sensing	• Multimode POF	• Absorption
	A B	Performance of	coated with ZnO.	spectra of
	Pantjawati, L	Multimode	• ZnO was synthesized	methanol at
	Hasanah, F	Polymer Optical	with sol-gel method.	different
	Abdurrahman,	Fiber (POF)	• ZnO characterized	concentratio
	R E	Coated with ZnO	using SEM and XRD	n from 5% to
	Pawinanto, H	towards Methanol		50%.
7	Ramza and G	Vapour		• Response of
	Sugandi[21]			the intrinsic
	AT MACATO	A 410		sensor of
		N.		POF to
				methanol
	ELS.			vapor inside
	SAINO .			the testing
	ship	1.15:0	the second	chamber
	Masayuki	Plastic Optical	• POF sensor head	Alcohol
	Morisawa and	Fiber Sensing of	coated with a a	concentratio
	Shinzo	Alcohol	mixture polymer of	n properties.
	Muto[22]	Concentration in	novolac resin and	• The change
		Liquors	polyvinylidenefluori	in output
0			de (PVDF)	light
8				intensity
				against a
				various
				ethanol
				concentratio
				n
	Thanigai	A novel	• using POF fiber to	• Relationship
9	1			

	Hazura	limonene	without any coating		output
	Haroon, Hazli	detection using	process.		voltage and
	Rafis Abdul	plastic fiber optic	• Tapered fiber using		limonene
	Rahim, Siti	sensors and the	chemical etching		concentratio
	Halma Johari,	tapered approach	enemieur etennig.		n percentage
	Siti Khadijah			•	Relationship
	Idris@				between the
	Othman,				
	Hanim Abdul				output voltage
	Razak,				and refractive
	Maisara				index of
	Othman.[23]				1.00
	ALAYS				different
	AT MAN	10			limonene
	KMIN	AKA			concentration
	List III				8
	P	Taparad Optical	• Toporod DOE		Output
	K. Siyacoumar	Fiber Bio Sensor	• Tapeled FOF	•	intensity of
	M Vinoth	for	atshing method by		the optical
	Zachariah	TEKNIKAL N	IALAYSIA MELAKA		the optical
		Detection		_	Lineen
	Alex	Detection	• This research using	•	Linear
10			tapered POF to		relationship
			detect Testosterone.		between
			• Using UV-visible		optical
			spectrometer to		intensity
			analyze the		variation and
			absorption of		concentration
			testosterone.		of TES

### 2.5 Summary

This chapter provides the theory related to this project. All explanations were explained in detail. A comparison between the previous work that was related to this project was also provided in this chapter.



## **CHAPTER 3**

## **METHODOLOGY**



This chapter covers the whole process of implementation and decisions to achieve the goal. The equipment and materials utilized and its tasks for this project were discussed specifically. This chapter also includes the software and hardware design of the project. The mechanism of the experiment was clarified in this chapter. The experiment for limonene concentration measurement on six different depths of a tapered plastic optical fiber coated with ZnO was also discussed in detail in this chapter.

### **3.1** Experimental Material and Equipment

Some of the experimental materials for this project are listed in Table 3.1. All equipment is available at PERG/ASEC Lab, FKEKK, UTeM. Many of the materials were employed in the fiber probe fabrication and hydrothermal method for fiber coating. The list and the explanation of the tools that have been used for this project was shown in Table 3.2.

No	Material	Linear Formula	Brand
1	Zinc Acetate Dihydrate	Zn(CH <sub>3</sub> COO)2H <sub>2</sub> O	او نيو.
2	Sodium Hydroxide	L MALAYSIA ME NaOH	EMSURE
3	Ethanol	CH <sub>3</sub> CH <sub>2</sub> OH	SYSTERM

 Table 3.1 Experimental Materials.




No	Equipment Name	Function
1	Eco-cell Laboratory Oven	This Eco-cell Laboratory is a line
		of economic ovens with a large
		temperature range, accurate
		measurement, and dependable
	E E	operation for simple material
		heating and drying procedures.
	ALAYSIA 40	The operation of this oven runs
		very smoothly without noise and
	Elenne U	gentle airflow during the process.
		The temperature can be
		controlled accurately depending
		on users' preferences.
2	Ultrasonic Cleaner	This ultrasonic cleaner used for
		this assignment is to do some
		cleaning of the equipment used
		such as beakers, spatula, and
		many more. This ultrasonic
		cleaning can work at high-
		frequency sound waves and
		produce cavitation bubbles. For
		this project, this equipment was

 Table 3.2 List and Explanation of the Equipment.

		used for the water bath process
		and cleaning.
3	Laboratory Magnetic Stirrer and Hot Plate	This laboratory magnetic stirrer
		and the hot plate were used to mix
		any solution for this project and
		also can heat the solution if
	Extension MALTING M	needed. For this project, this
	MACINETIC STORING	equipment was used during the
		hydrothermal method.
4	Digital Micrometer	This digital micrometer was used
		to measure the most accurate
		information for an object that is
		small. The unit measurement for
		this equipment is in millimeters.
	يكنيكل مليسيا ملاك	It also can measure thickness and
	UNIVERSITI TEKNIKAL MALA	length. During this project, this
		equipment was used to measure
		the waist diameter of the tapered
		fiber.
5	Scanning Electron Microscope (SEM)	This scanning electron
		microscopy (SEM) was used to
		scan over the surface of an object
		and focused on the electron. This
		equipment will also produce an
		image after doing some scanning.

		For this assignment, this equipment was used to check the POF surface of the ZnO growth process after completing the hydrothermal method.
6	Digital Refractometer	This digital refractometer was used to measure the refractive index of any type of solution. The solution was placed on the surface of the prism to display the digital readout of the RI value. In this project, this equipment is used to display the reading of the refractive index for different
		limonene concentrations.

#### **3.2** Sensor Probe Fabrication

According to previous research, the cladding of the fiber must be removed to trigger the probe's sensitivity for the analysis of the refractive index. Chemical etching is used in this project to remove a portion of the fiber's plastic cladding. This method makes it more sensitive, which increases the power of the evanescent wave (EW) inside the POF cladding. [24]. The sensing region length for this project is 2 cm, and the tapered diameters are 0.45 mm, 0.50 mm, 0.55 mm, 0.60 mm, 0.65 mm, and 0.70 mm. The first step is to use an acetone solution to etch the area of the sensing probe. The surface will turn milky white. The sandpaper will then be used to etch the sensing area until it reaches the previously specified diameter. The flowchart of the tapered fiber process



Figure 3.1: Tapered Fiber Process.

The process of the etching took about 5-7 minutes depending on the diameter of the desired parameter. One disadvantage of this process is that it must be handled with caution because the lower the diameter, the more brittle the fiber. The microscope image of the tapered core diameter of plastic optical fiber for 0.60 mm, 0.55 mm, and 0.50 mm, respectively, were shown in Figure 3.2(a), Figure 3.2(b), and Figure 3.2(c), respectively using an image analyzer (Axioskop 2 MAT) with 100X magnification lens.



**(b)** 



(c)

Figure 3.2: Image from the microscope (a) 0.60 mm (b) 0.55 mm (c) 0.50 mm.

## 3.3 ZnO coating by hydrothermal technique

The hydrothermal method has been widely used because of easy and tolerable growth conditions. This method is applied to increase the evanescent wave sensor's sensing capacity. The flowchart of the ZnO growth using the hydrothermal method was shown in Figure 3.3 below.

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The three main stages for the growth of ZnO nanoparticles on plastic optical fiber

are fiber preparation, seeding process, and growth process as shown in Figure 3.4.



Figure 3.4: Procedure for ZnO nanoparticles on POF by using hydrothermal method.

#### **3.3.1** Fiber Preparation

The preparation of fiber was prepared for the ZnO growth on plastic optical fiber. The POF jacket was removed manually to expose the core of the POF as shown in Figure 3.5(a). For this project, the length of the sensing region is about 2 cm at the center of the POF as shown in Figure 3.5(b). The outer layer of the POF is a fluorinated polymer jacket with a diameter of 1880 to 2120 nm. The fiber will be tapered using acetone, de-ionized water, and sandpaper. When the acetone solution is applied to the core fiber, the area will turn into a milky white surface. The core of the POF was tapered into six different diameters by using sandpaper which is 0.45 mm, 0.50 mm, 0.55 mm, 0.60 mm, 0,65 mm, and 0.70 mm. The diameter of the POF was measured using a digital micrometer as shown in Figure 3.5(c).



**(a)** 





(c)

#### Figure 3.5: Fiber Preparation Process (a) Fiber with jacket (b) Fiber exposed in 2cm length (c) Measurement using micrometer

#### 3.3.2 Seeding Process

After the fiber process, the seeding process is an important step before proceeding into the growth of the ZnO process where it can determine the ZnO nanorod's growth such as diameter, length, uniformity, and density. For this process, there are four important steps in the seeding process which consist of preparing the seeding solution, POF core surface treatment, dipping the POF in the seeding solution, and annealing, as shown in Figure 3.6 below.



Figure 3.6: Step Seeding Process

For the first step, two types of solutions need to be prepared. For the first solution is ZnO nanoparticles, 0.088g of Zinc Acetate Dihydrate [Zn(CH<sub>3</sub>COO)2H<sub>2</sub>O] by Merck KGaA, Germany was dissolved in 80 ml of ethanol (Merck KGaA, Germany) for about 30 minutes under slow stirring at a temperature of 50°C to produce 5mM solution. After that, the solution is cooled down at ambient temperature for a while before adding another 80 ml of ethanol into the solution. The illustration process of zinc acetate dihydrate with the final amount of 160 ml was shown in Figure 3.7 below.



**UNIVER Figure 3.7: Process of Zinc Acetate Dihydrate.** 

The second solution is a pH control solution, 0.016g Sodium Hydroxide (NaOH) was dissolved in 80 ml ethanol for about 30 minutes under slow stirring at a temperature of 50°C to produce a 5mM solution. The process of pH control solution was shown in Figure 3.8 below.



Figure 3.8: Process of pH Control Solution using NaOH

For the next process, the pH control solution is added to the ZnO nanoparticles solution by using a 1 ml pipet after 10 minutes to produce a final amount of 240 ml seeding solution as shown in Figure 3.9 below. For every drop of pH control solution, the zinc acetate dihydrate was stirred slowly for every 1 minute able to increase the amount of hydroxyl ions (OH-) in the seeding solution. The process is repeated about 50 times until the pH control solution is finished. The seeding solution was put in a water bath at 60°C for about 3 hours. This process significantly changes the color of the solution from plain to milky.



Figure 3.9: Alkaline process of ZnO nanoparticles solution by NaOH

The next step was to make a good uniformity of ZnO growth on POF by treated a core surface of the POF using polysorbate 80 (tween 80). The process of this method was shown in Figure 3.10 below. 50 ml of Polysorbate 80 (tween 80) was dissolved in 500 ml deionized (DI) water under slow stirring at a temperature of 45°C. The fiber is submerged in the solution vertically for 10 minutes and let dry in the open air for about two hours.



The last step of the seeding process is to dip the POF into the seeding solution. This method is called a dip and dry method where it is to develop a nucleation site on the POF as shown in Figure 3.11. All the POF samples were submerged into the seeding solution for 1 minute and dried for 1 minute at a temperature of 70°C inside the oven. This process is repeated 10 times and then annealed for three hours at 70°C.



Figure 3.11: Process of Dip and Dry method for the seeding process

#### **3.3.3** Growth process

The last process of the hydrothermal method was the growth process. The process started with 2.97g of Zinc Nitrate Hexahydrate [Zn (NO<sub>3</sub>)<sub>2</sub> 6H<sub>2</sub>O] and 1.40g of Hexamethylenetetramine ( $C_6H_{12}N_4$ ) was dissolved in 1000 ml of deionized (DI) water under slow stirring to produce 10mM solution. Then, all the seeded POF are put vertically into the synthesis solution and heated at a temperature of 90°C inside the oven as shown in Figure 3.12 below. After 5 hours, the solution needs to change to a new solution because wants to maintain the condition of the growth process. The total growth time for this process ranged from 8 to 10 hours. After synthesis, the POF was removed and rinsed with DI water several times. The color of the solution will significantly change from clear to milky.



Figure 3.12: ZnO Growth Process

#### **3.4** Experimental Setup for Sensor

For the experimental setup, the block diagram that consists of a red LED, POF sensor, receiver circuit, and LCD display for this project was shown in Figure 3.13. POF sensor Receiver circuit RED LED (630-700 nm) UNVERSITI TEKNIKAL MALAYSIA MELAKA

#### Figure 3.13: Block Diagram for Experimental Setup

This experimental setup was used to measure the output voltage for the limonene concentration using a POF sensor. The red LED will act as a light source with a wavelength of 660 nm and the average power dissipation is 105mW. During an experiment on testing the limonene solution, the POF fiber needs to be in a straight line to prevent any loss occurred and needs to be handled carefully to prevent from break. The sensing region of the POF was dipped into the solution. The limonene solution was put into the petri dish during the whole process of measurement. The refractive index of each different concentration was measured by using a digital

refractometer. During the measurement of the POF sensor toward limonene concentration, the LCD displays the voltage value. The real setup of the experiment was shown in Figure 3.14.



Figure 3.14: The Real Setup of The Experiment

For the limonene concentration, 20/80, 40/60, 60/40, 80/20,100/0 of limonene oil/hexane solution was measured using the volume ratio (ml/ml), produced a value of refractive index 1.4094, 1.4136, 1.4374, 1.4481, and 1.4882 respectively. This limonene concentration needs to be stored tightly in a glass compartment container to prevent it to be evaporated. This solution also needs to be kept at an ambient temperature of 25°C to entirely remove the influence of temperature on the measurement.

#### 3.5 Summary

In this chapter, the analysis of the sensor device was been tested towards a different type of limonene concentration, various typed of tapered POF, sensitivity, linearity, and the output voltage of the sensor performance were also been discussed in this paper. All parameters for performance characterization were also discussed.



# **CHAPTER 4**

# **RESULTS AND DISCUSSION**

This chapter includes all results related to this project and the project implementation. The key parameter observed and reported were the different depths of tapered POF coated with ZnO affected by limonene concentration to determine the performance of the fiber has been discussed in this chapter. All the data collected were recorded and analyzed in detail.

#### 4.1 Result Analysis

#### 4.1.1 Tapered fiber

The core of the POF must be removed to enhance the sensibility of the fiber. The analysis begins with the six different diameters of tapered POF which were 0.70 mm, 0.65 mm, 0.60 mm, 0.55 mm, 0.50 mm, and 0.45 mm. All the tapered fibers were coated with ZnO to determine the performance of each fiber sensor. The diameter of the POF for 0.70 mm, 0.65 mm, and 0.60 mm was measured using a digital micrometer as shown in Figure 4.1(a), Figure 4.1(b), and Figure 4.1(c), respectively. The output voltage was observed and recorded for the sensor development by the parameters variation in the experiment. The six different diameters of the tapered fiber were shown in Figure 4.2 below.



**(a)** 





**(b)** 



Figure 4.1: Measurement of tapered fiber using a digital micrometer.



Figure 4.2: Six different diameters of tapered fiber

#### 4.1.2 Limonene Concentration

Different values of limonene concentration were used to determine the different parameters for the sensor to study the sensor's performance. To obtain each concentration, a limonene oil/hexane volume (ml/ml) ratio of 20/80, 40/60, 60/40, 80/20, and 100/0 was used. Figure 4.3 depicts the results of each RI value of limonene concentration measured with a digital refractor meter, with the solution placed on the surface of the prism to display the digital readout of the RI value. As the concentration of limonene increases gradually from 20% to 100%, the RI of the limonene solution increases from 1.4094 to 1.4882. Summarises of the RI value for the limonene concentration was shown in Table 4.1.

<b>Table 4.1:</b>	Refractive	index of	t the limoner	ne concentrations
	6			

0.13 34

Limonene concentration	Refractive Index (RI)
20%	1.4094
كنيكل مايسيا ملاك	او يوم سيخ ش
60%	1.4374
UNIVERSITI TEKNIKAL N	IALAYSIA MELAKA
80%	1.4481
100%	1.4882





Figure 4.3: Measurement of refractive index for limonene concentrations

# 4.2 UNIVERSITI TEKNIKAL MALAYSIA MELAKA Experiment Analysis

## 4.2.1 Output Voltage

The output voltage of the different limonene concentrations from 20% to 100% for different tapered POF was shown in Table 4.2 below.

Limonene	Tapered POF (mm)					
Concentration, %	0.70 mm	0.65 mm	0.60 mm	0.55 mm	0.50 mm	0.45 mm
20%	1.17 V	1.29 V	1.41 V	1.57 V	1.66 V	1.72 V

 Table 4.2: Output voltage result of different limonene concentrations

40%	1.16 V	1.28 V	1.38 V	1.55 V	1.61 V	1.7 V
60%	1.14 V	1.27 V	1.37 V	1.51 V	1.58 V	1.66 V
80%	1.13 V	1.27 V	1.35 V	1.49 V	1.54 V	1.62 V
100%	1.10 V	1.25 V	1.33 V	1.46 V	1.49 V	1.56 V



Figure 4.4: Average output voltage of limonene concentrations

For the first analysis result, the average output voltage from different tapered POF against the concentration of limonene solution was shown in Figure 4.4 above. The sensor system has been observed to respond effectively to changes in limonene concentration when the limonene concentration increases from 20% to 100%. To clarify, the output voltage of 0.45 mm tapered POF with 20% limonene concentration is 1.72 V compared to 1.56V with 100% limonene concentration is significantly lower as the limonene concentration increases. The same goes for the other tapered POF

where from 1.66 V to 1.49 V for 0.50 mm tapered POF, 1.57 V to 1.46 V for 0.55 mm tapered POF, 1.41 V to 1.33V for 0.60 mm tapered POF, 1.29 V to 1.25 V for 0.65 mm tapered POF, and 1.17 V to 1.10 V for 0.7 mm tapered POF.

Refractive	Tapered POF (mm)					
Index (RI)	0.70 mm	0.65 mm	0.60 mm	0.55 mm	0.50 mm	0.45 mm
1.4094	1.17 V	1.29 V	1.41 V	1.57 V	1.66 V	1.72 V
1.4136	1.16 V	1.28 V	1.38 V	1.55 V	1.61 V	1.7 V
1.4374 MAL	1.14 V	1.27 V	1.37 V	1.51 V	1.58 V	1.66 V
1.4481	1.13 V	1.27 V	1.35 V	1.49 V	1.54 V	1.62 V
1.4882	1.10 V	1.25 V	1.33 V	1.46 V	1.49 V	1.56 V
E.						

 Table 4.3: Output voltage result for a refractive index of limonene concentrations



Figure 4.5: Average output voltage for a refractive index of limonene concentrations

For the next analysis was conducted using the same different diameters of tapered POF. The output voltage result for a refractive index of limonene concentrations was shown in Table 4.3. It can be observed that as the refractive index of limonene concentration increase, the output voltage will decrease where the output voltage for 0.45 mm tapered POF decrease from 1.72V to 1.56V, for 0.50 mm tapered POF decrease from 1.66V to 1.49V, for 0.55 mm tapered POF decrease from 1.57V to 1.46V, for 0.60 mm tapered POF decrease from 1.41V to 1.33V, for 0.65 mm tapered POF decrease from 1.29V to 1.25V and for 0.70 mm tapered POF decrease from 1.17V to 1.10V as shown in Figure 4.5 above.

#### 4.3 Sensitivity and Linearity

The graph of linearity and sensitivity of the sensor performance for the limonene concentration and refractive index of the limonene concentrations was shown in Figure 4.6 and Figure 4.7. From the graph, the tapered POF at 0.45 mm, 0.50 mm and 0.55 mm have the highest sensitivity and linearity compared to the 0.60 mm, 0.65 mm, and 0.70 mm tapered POF. The sensitivity is recorded at 0.205 V/%, 0.14 V/%, and 0.20 V/% and the slope shows good linearity of more than 99%, 98%, and 97% for the 0.55 mm, 0.50 mm, and 0.45 mm tapered POF respectively as shown in Table 4.4. The lowest sensitivity was recorded at 0.65 mm tapered POF which is 0.045 V/% with a linearity of 92%. The data for a refractive index of limonene concentrations was shown in Figure 4.6 below. The sensitivity of the tapered POF was higher at 0.55 mm, 0.50 mm, and 0.45 mm which are 1.3288 V/%, 1.9334 V/%, and 1.9545 V/% where the linearity of tapered POF is 95%, 94%, and 99% respectively. From both results, it can be concluded as the tapered region diameter decreases, the sensitivity and the linearity of limonene detection increase. The best performance of the tapered POF was when the diameter of the tapered is in the range of 0.40 mm to 0.50 mm. When the

diameter of the POF was above 0.55 mm and below 0.40 mm, it did not exhibit the substantial sensitivity of the fiber.



Figure 4.6: Average output voltage of limonene concentrations



Figure 4.7: Average output voltage for a refractive index of limonene concentrations

Tapered POF (mm)	Linearity, %	Sensitivity, V/%
0.7	96.33	0.085
0.65	92.05	0.045
0.6	98.1	0.095
0.55	99.35	0.205
0.5	98.99	0.14
0.45	97.09	0.20

 Table 4.4: Linearity and sensitivity for an output voltage of limonene concentrations



 Table 4.5: Linearity and sensitivity for output voltage for a refractive index of limonene concentrations

Tapered POF (mm)	Linearity, %	Sensitivity, V/%
×0,7	99.34	0.8327
مليسيا0.65	ىيتى ئېر23.82 يىكى	0.4383
0.6 UNIVERSITI TE	90.44 ** KNIKAL MALAYSIA	0.8799 MELAKA
0.55	95.82	1.3288
0.5	94.96	1.9334
0.45	99.63	1.9545

#### 4.4 Sensitivity and Linearity Result Comparison with Previous Study

The result from the past research related to the project was made as a comparison with the result that has been measured for this project was shown in Figure 4.8 below[25]. From past research, the experiment was based on the effect of relative humidity percentage on the optical fiber sensor that is coated with ZnO. From the graph, the tapered POF for 0.50 mm shows good sensitivity and linearity compared to other diameters of tapered POF which are 0.0129 V/RH% and 99.53% respectively. It can be concluded that different waist diameters of the tapered POF give different results on sensitivity and linearity. The sensitivity for tapered POF below 0.55 mm gives the highest sensitivity value and shows good linearity. Both results did not produce almost the same value because both experiments do not measure the same thing which is the past research was using different relative humidity percentages and this PSM project was using different concentrations of limonene solution. To make it more clear, a table comparison between past research and the PSM project was shown in Table 4.6.



#### **Figure 4.8: Result from the past research**

	Previous I	Result [25]	This I	Result	
Tapered POF	(Relative ]	Humidity)	(Limonene Concentration)		
(mm)	T	Sensitivity,	T	Sensitivity,	
	Linearity, %	V/RH%		V/%	
0.7	-	-	96.33	0.085	
0.65	-	-	92.05	0.045	
0.6	99.58	0.0079	98.1	0.095	
0.55 MAI	99.27	0.005	99.35	0.205	
0.5	99.53	0.0129	98.99	0.14	
0.45	99.13	0.0092	97.09	0.20	
0.4	65.09	0.0021		-	

#### Table 4.6: Comparison results of past research and PSM project

# 4.5 Comparison of SEM Result With Previous Study

A scanning Electron Microscope (SEM) was used to identify the morphology of the ZnO coated tapered POF. The comparison result between the two methods which are non-coated and coated ZnO-coated POF was shown in Table 4.7 below. The result proved that the ZnO was growth on the tapered POF. The seeded approach verified that ZnO's structure consists of many superfine POF nanorods compared with the noncoated method. The element analysis content for the coated method was shown in Figure 4.9 below. From the analysis, the top layer of POF contain Zinc (Zn) was 10.84%, Carbon (C) was 15.01%, Oxygen (O) was 8.04%, and Platinum (Pt) was 61.51%, which verified the sensing material is ZnO as shown in Figure 4.10.





Figure 4.9: Element Analysis of Tapered POF



performance of the tapered POF sensor was compared with the limonene concentration and the refractive index of limonene concentration. The sensitivity and linearity of the fiber were also provided in this chapter. in addition, the comparison result with the previous study was also discussed.

# **CHAPTER 5**

# **CONCLUSION AND FUTURE WORKS**



In conclusion, this project shows the expansion of a low-cost and simple ZnO based sensor device for limonene concentration measurement. After all of the experiments, the different waist diameters of a tapered POF have a significant impact on the sensor's performance. The analysis shows that the smallest diameter of a tapered POF can produce higher sensitivity than the larger diameter. The result for 0.45 mm, 0.50mm, and 0.55 mm gives good result which are 0.20 V/%, 0.14 V/%, and 0.205% respectively. When compared to the other diameters which are 0.60 mm, 0.65 mm, and 0.70 mm produce the least sensitivity which are 0.95 V/%, 0.045 V/%, and 0.085 V/% respectively. Besides that, the output voltage also shows a result when the limonene concentration increases which also corresponds to the refractive index of the limonene solution, the output voltage will be decreased. This is consistent with the

fundamental concept that as a solution concentration increases, more light was absorbed by the solution, resulting in a reduction in the intensity of light received and a consequent drop in output voltage. From the analysis of the project, the output voltage of 0.45 mm tapered POF with 20% limonene concentration is 1.72 V compared with 100% limonene concentration is 1.56V was significantly dropped as the limonene concentration increased. As the waist diameter of the tapered POF decrease from 0.70 mm to 0.45 mm, the output voltage for the limonene concentration will increase. To sum up, all three objectives of this project have been achieved which were able to design of a polymer optical fiber (POF) based sensor for limonene concentration, the sensing capability of the ZnO coated POF by using the hydrothermal method achieved, and also able to study the relationship of limonene concentration to the optical output voltage, sensitivity, and linearity.

### 5.2 Future Work

For future work, some of the improvements that can be made are to enhance more synthesis of ZnO nanostructures. All the synthesizing methods can give an impact on the output and can be explored more about the outcome. Besides that, rather than using a ZnO as a sensitive solution, different materials also can be used. Some of the materials are graphene and HEC/PVDF. The performance of the tapered POF can be compared by using different types of material Other than that, despite using just using LCD to display an output, the IoT platforms can be used to display the data. Cayenne, Blynk, ThingWorx, and others are examples of IoT platforms that can be used.

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