UNIVERSITI TEKNIKAL MALAYSIA MELAKA

TAPERED-LOOP PLASTIC OPTICAL FIBRE COATED WITH ZNO NANORODS USING LED SOURCE FOR FORMALDEHYDE SENSING APPLICATION

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DEDICATION

Gratitude towards the past myself for all the time and effort that had been spent;

Thanks for the support from my wife and my parents;

Thank you, my supervisor, for the guideline and chance to do this master’s;

Thanks, God, for granting me the opportunity to collect data from the experiments;

Along the journey of completing this thesis.

ABSTRACT

The development of plastic optical fibre (POF) as an optical sensor presents a promising solution for low-cost sensor production. POF demonstrates commercial potential across various sensing applications, notably in humidity and chemical vapour sensing. Enhancing the sensor's performance involves coating a semiconductor material on a tapered POF. Furthermore, the shape and diameter of the optical sensor significantly influence its sensitivity; a larger diameter introduces more sensing region. Formaldehyde, a hazardous volatile organic compound commonly found in adhesives used in furniture production, building materials, and interior decoration, poses significant health risks, including cancer, when its concentration exceeds safe levels. This study aims to develop a formaldehyde vapour sensor utilising a POF coated with zinc oxide (ZnO) nanorods. The sensor's construction involves tapering the fibre to remove the outer cladding layer, exposing the core layer, and subsequently looping and coating it with ZnO nanorods using the hydrothermal method. Various loop diameters ranging from 3.5 cm to 6.11 cm were prepared with consistent waist diameter. Humidity sensing tests were conducted to determine the optimal diameter. The sensor's sensitivity to humidity levels ranging from 30% RH to 90% RH was observed across different diameter lengths. Results indicate that the sensor with a 6.11 cm diameter exhibited superior performance, with a sensitivity of 0.0285 V/RH and 98.13% linearity. A comparison between coated ZnO and uncoated POF sensors revealed higher sensitivity (0.0285 V/RH) for the former compared to the latter (0.0115 V/RH). Additionally, when subjected to different LED colours—red, blue, and green—the sensor exhibited heightened sensitivity to blue light. The optimised diameter was further tested with varying concentrations of formaldehyde vapour to evaluate the sensor's response, yielding a sensitivity of -0.0062 V/%, indicative of successful formaldehyde sensing capabilities.

FIBER OPTIK PLASTIK GELUNG TIRUS BERSALUT NANOROD ZNO MENGGUNAKAN SUMBER LED BAGI APLIKASI PENGESANAN FORMALDEHID

ABSTRAK

Pembangunan serat optik plastik (POF) sebagai sensor optik telah menjadi penyelesaian yang menjanjikan dalam pengeluaran sensor kos rendah. POF mempunyai potensi komersial dalam banyak jenis aplikasi pengesan, termasuk mengesan kelembapan dan wap kimia. Dengan menyalut bahan semikonduktor pada POF tirus, prestasi pengesan sensor boleh ditingkatkan. Bentuk dan diameter sensor optik juga boleh menjejaskan kepekaan sensor. Sensor yang mepunyai diameter yang besar akan memiliki kawasan pengesan yang lebih besar. Formaldehid boleh dijumpai pada pelekat yang digunakan didalam pengeluaran perabot, bahan bangunan dan hiasan dalaman. Formaldehid merupakan sebatian organik yang berbahaya. Apabila kepekatan formaldehid melebihi tahap tertentu, ia akan menimbulkan ancaman kepada manusia. Formaldehid boleh menyebabkan kerosakan kepada sistem tubuh manusia termasuk menyebabkan kanser. Kajian ini bertujuan untuk membangunkan sensor wap formaldehid menggunakan POF dilapisi dengan Zink Oksida (ZnO) nanorod. Sensor ini telah dibangunkan dengan mendedahkan serat untuk mengeluarkan lapisan luar POF, yang dipanggil penutup. POF yang dipotong kemudian dibentuk gelung dan dilapisi dengan ZnO nanorod menggunakan kaedah hidrotermal. POF sensor disediakan dalam diameter loop yang berbeza dari 3.5cm hingga 6.11cm dengan ketebalan diameter yang sama. Ujian pengesan kelembapan dijalankan untuk mendapatkan diameter yang optima. Sensor diuji dalam ruang kelembapan daripada 30%RH-90%RH untuk menguji kesan panjang diameter yang berbeza pada kepekaan sensor. Hasil ujian menunjukkan bahawa sensor dengan diameter 6.11cm memperoleh prestasi pengesan terbaik dengan kepekaan 0.0285V/RH dan lineariti 98.13%. Prestasi sensor untuk ZnO disalut dan tidak disalut terhadap tahap kelembapan yang berbeza menunjukkan bahawa sensor POF disalut ZnO mendapat kepekaan yang lebih tinggi 0.0285 V/RH berbanding dengan 0.0115 V / RH tanpa lapisan ZnO. Apabila diuji dalam 3 warna LED yang berbeza, merah, biru dan hijau, hasilnya menunjukkan kepekaan yang tinggi untuk biru berbanding gelombang lain. Diameter yang optima kemudian digunakan untuk diuji dalam kepekatan berbeza uap formaldehid untuk memerhati respons sensor. Sensitiviti yang diperolehi dalam kajian ini, ialah -0.0062V/% menunjukkan bahawa reka bentuk mempunyai hasil yang baik dalam mengesan formaldehid.

ACKNOWLEDGEMENT

In the Name of Allah, the Most Gracious, the Most Merciful. First and foremost, I would like to take this opportunity to express my sincere acknowledgement to my supervisor, Dr. Hazli Rafis bin Abdul Rahim for his knowledge shared throughout my study journey. I would like to thank my family and friends for their constant support. Finally, I would like to thank my wife Siti Nurhidayah for helping me to go through the challenges I face throughout my study journey.

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LIST OF ABBREVIATIONS

$UTeM$	-	Universiti Teknikal Malaysia Melaka
POF	-	Plastic Optical Fibre
LED	-	Light Emitting Diode
SnO_2	-	Tin Oxide
In_2O_3	-	Indium Oxide
ZnO	-	Zinc Oxide
EDS	-	Energy-dispersive X-ray Spectroscopy
SEM	-	Scanning Electron Microscope
SOF	-	Silica Optical Fibre
OFS	-	Optical Fibre Sensor
PMMA	-	Polymethyl Methacrylate
GI-POF	-	Graded Index Plastic Optical Fibre
Ppb	-	Parts per billion
OH^-	-	Hydroxyl groups
SIPOF	-	Step Index Plastic Optical Fibre
CYTOP	-	Fluoropolymer With Special Structure
EW	-	Evanescent Wave
HEC/PVDF	-	Hydroxyethyl- cellulose/Polyvinylidenefluoride
pH	-	Potential of Hydrogen
CO	-	Carbon Monoxide
NH_3	-	Ammonia

TiO_2	-	Titanium Oxide
PCS	-	Plastic-clad silica
$Zn(NO_3)_2.6H_2O$	-	Zinc nitrate hexahydrate
HMT	-	Hexamethylenetetramine
RH	-	Relative humidity
UV	-	Ultraviolet
NA	-	Numerical aperture
DI	-	Deionized water
TIA	-	Transimpedance amplifier
RI	-	Refractive index

LIST OF SYMBOLS

A	-	Ampere
°C	-	Degree celcius
V	-	Volt
%RH	-	Relative humidity
pm	-	Per million
db	-	Decibel
Hz	-	Hertz

LIST OF APPENDICES

LIST OF PUBLICATIONS

The following is the list of publications related to the work on this thesis:

Mohamad Noriszakiy Hisam, Hazli Rafis Abdul Rahim, Hanim Abdul Razak, Siti Nurhidayah Azmi, Siti Halma Johari, Mohd Hafiz Jali, Siti Amaniah Mohd Chachuli, Siddharth Thokchome, Ahmad Razif Muhammd, Sulaiman Wadi Harun, 2022. Tapered Plastic Optical Fibre Loop Coated With Zno Nanorods Using Multiple Channels For Relative Humidity Sensing. *Przegląd Elektrotechniczny,* ISSN 0033-2097, pp. 72-76.

CHAPTER 1

INTRODUCTION

1.1 Background

Research on optical technologies has been widely conducted by various researchers around the globe. By leveraging lighting technology, research has discovered advancements that could enhance industries while promoting environmental sustainability. Recent advancements in photonics studies have utilised the benefits of plastic optical fibre (POF). The development of POF has led to fascinating applications that could enhance various sectors, such as the medical and educational industries. POF has demonstrated its advantages in numerous previous studies conducted by researchers. Its properties offer benefits in terms of cost, flexibility, weight, and more. POF is cost-effective, providing great flexibility and lightweight properties. Additionally, it exhibits resilience to bending, shock, and vibration. Conducting research using POF does not necessitate specialised tools or techniques for handling the fibre.

Moisture detection and control are emphasised across almost every industry today. Applications such as agriculture, healthcare, food processing, and meteorology require accurate, high-speed, and cost-effective moisture measurement (Gomez et al., 2018; Lin Bo, et al., 2014; Ndoye et al., 2017). Relative humidity (RH) describes the amount of water vapour in the air. It is defined as the ratio between the actual pressure of water vapour and the saturated vapour pressure at a given temperature (Xia et al., 2013). In recent decades, the development of optical fibre humidity sensors has exhibited better performance compared to traditional electrical sensors. Optical fibres are unaffected by electromagnetic fields and harsh environments (Chen et al., 2019).

Formaldehyde is a colourless gas with a pungent odour. It is utilised in the manufacture of building materials and household products such as particle board, plywood, and fibreboard (Mirzaei et al., 2016). Additionally, formaldehyde serves as an intermediate in chemical manufacturing and as a preservative for certain foods. It may also be present in products such as preservatives, drugs, and cosmetics (Lefebvre et al., 2012). However, the ubiquitous presence of formaldehyde in various facets of life poses a danger to human health. The safety threshold for exposure, as established by the World Health Organisation (WHO), is less than 8 hours for outdoor exposure at 2 ppm and less than 30 minutes for indoor exposure at 0.08 ppm (Choi et al., 2013; Chung et al., 2013; Jiang et al., 2010; J. Wang et al., 2008). Formaldehyde can have harmful effects on human organs, including the liver, eyes, kidneys, and skin. Exposure to formaldehyde gas may lead to burning sensations, coughing, wheezing, and skin irritation (Zaven Adamyan et al., 2017). In severe cases, prolonged exposure to relatively large amounts of formaldehyde increases the risk of developing leukemia and certain types of cancer.

In this research, a plastic optical fibre (POF) is employed to leverage its advantageous properties, as discussed earlier. A visible light emitting diode (LED) serves as the transmitter, while a photodiode functions as the receiver. The resulting output voltage from the photodiode is manipulated and observed during the data collection process. The research commenced with a comprehensive review of related literature, aiming to examine previous studies and compare their research designs, methods, and findings.

1.2 Problem Statement

Formaldehyde can be found in adhesives used in the production of furniture, building materials, and interior decoration. It is a hazardous volatile organic compound (VOC) (Ishihara et al., 2017). When its concentration exceeds a certain level, it poses a threat to human health, potentially causing damage to the human body system, including the development of cancer.

Previous research on sensing formaldehyde vapour using optical fibre has shown promising results in terms of sensing performance. Methods such as spectroscopy (Werle et al., 2002), chemiresistor (Lee et al., 2006; Li et al., 2006), bio-sniffer (Mitsubayashi et al., 2008) and cataluminescence (Zhou et al., 2006) have demonstrated their ability to detect formaldehyde. However, these methods have drawbacks, as they require extensive equipment and high operating temperatures, resulting in high operational costs. The drawbacks of each method are listed as follows:

- Spectroscopy: Difficult and complex setup, require laser light as source (Dyroff et al., 2007)
- Chemiresistor: Complex fabrication process (Mondal et al., 2018)
- Bio-sniffer: Development of a bio-sniffer require complex process (Toma et al., 2016)
- Cataluminesscence:System require high temperature to operate (Zhou et al., 2017)

Tapering a silica optical fibre for sensor development is a complex process. Focusing on a small specific coating area rather than the entire coating area makes the process more challenging and complicated. Silica, in general, requires laser light as the input source, and mechanical support is needed to align the beam into the fibre. Moreover, lasers themselves are costly and introduce heat, which is not suitable for sensing chemical vapour.

1.3 Research Objectives

The primary aim of this research is to investigate the utilisation of an affordable sensing device using POF. The following sub-objectives must be achieved to ensure the success of the research. Specifically, the objectives are as follows:

1. To fabricate sensitive material coatings based on ZnO nanorods using the hydrothermal method on POF.
2. To optically characterise and optimise the diameter of the POF loop coated with ZnO nanorods.
3. To experimentally validate an optical sensor-based loop POF for formaldehyde vapour sensing.

1.4 Scope of Research

For this research, a multimode POF of the ESKA brand, type GH-4001-P, was utilised for the sensing device. The specifications of the POF are presented in Table 1.1.

Table 1.1 ESKA GH-4001-P specifications

Property	**Value**
Core material	Polymethyl-Methacrylate Resin
Cladding material	Fluorinated Polymer
Core Refractive Index	1.49
Numerical Aperture	0.5
Core Diameter	920-1040µm
Cladding Diameter	940-1060µm

Given that POF served as the primary material, all processes conducted for this research must operate within the limitations of its plastic nature. The POF employed in this study could withstand a maximum operating temperature of 85°C.

The light source suitable for POF falls within the wavelength range of 400 to 700 nm, which corresponds to the visible spectrum. While some products can withstand laser light as a source, which is expensive and complex to set up, this research prefers the use of LEDs as the light source due to their affordability and lower operating temperature. In this research, three channels of LEDs were used: red (630nm), green (520nm), and blue (466nm).

To optimise the sensing region, humidity sensing was conducted, while formaldehyde vapour sensing was proposed as an application of this sensor. In the relative humidity test, a UNI-T brand humidity meter, model UT333, was employed as the reference. The humidity sensing range was set at 30-90% RH inside a closed chamber. For the formaldehyde vapour sensing test, a formalin solution from the Sigma brand, model HT501128, was used, with concentrations varying from 0-20%.

1.5 Research Hypotheses

The hypotheses under investigation in the current study are as follows:

1. Different loop diameters will demonstrate varying sensing effects.
2. Loop POF coated with sensitive materials will increase sensing sensitivity.
3. A simple and efficient device will serve as an optical transducer for sensing applications.

1.6 Significance of the Research

At the conclusion of this research, a tapered looped POF coated with ZnO was optimised, and the experimental results were analysed, thus leading to a better understanding of the effects of the tapered looped POF design on environmental changes. This understanding is crucial as the tapered looped design has not been extensively studied in previous research utilising POF as the primary material. The impact of ZnO as the coating material was also observed and documented. In summary, this research work contributes to providing experimental validation of the designed and optimised tapered looped POF coated with ZnO for sensing formaldehyde vapour as an application. While previous research has been conducted, only a limited number of studies have utilised POF. The experimental results presented herein demonstrate the potential of the design to effectively sense formaldehyde vapour using POF.

1.7 Thesis Outline

Based on the objectives previously stated and the proposed approach, this thesis comprises five (5) chapters, outlined as follows:

- Chapter 1: Introduction. This chapter presents the background of the study, research problems, objectives, scopes, contributions, and significance of the research.
- Chapter 2: Literature Review. This chapter discusses the theoretical review of the research, incorporating evidence from related works by previous researchers. It explores optical fibre technology, the fabrication method of POF, and provides an overview of humidity and formaldehyde vapour sensing using optical fibre.
- Chapter 3: Methodology. This chapter presents the procedure for designing the POF sensor in detail. The method for growing a ZnO nanorod on the tapered POF is discussed. Finally, the preparation for the sensing procedure is clearly outlined in this chapter.
- Chapter 4: Results and Discussion. In this chapter, the outcomes of the experiments regarding humidity and formaldehyde vapour sensing are discussed. The results are presented graphically and analysed in detail.
- Chapter 5: Conclusion and Recommendations for Future Research. This chapter summarises the main conclusions and achievements of the work undertaken in this research and suggests areas for future research.