

RESEARCH ARTICLE

Formaldehyde Sensing Using Tapered U-Shape Plastic Optical Fiber Coated With Zinc Oxide Nanorods

SITI H. JOHARI^{1,2}, TIU ZIAN CHEAK³, HAZLI RAFIS ABDUL RAHIM⁴, MOHD HAFIZ JALI⁵, HAZIEZOL HELMI MOHD YUSOF⁴, MD ASHADI M. JOHARI², AND SULAIMAN WADI HARUN¹

¹Department of Electrical Engineering, Faculty of Engineering, University of Malaya, Kuala Lumpur 50603, Malaysia

²Faculty of Electrical and Electronic Engineering Technology, Universiti Teknikal Malaysia Melaka, Malacca 76100, Malaysia

³Faculty of Engineering and Quantity Surveying, INTI International University, Nilai, Negeri Sembilan 71800, Malaysia

⁴Faculty of Electronic and Computer Engineering, Universiti Teknikal Malaysia Melaka, Malacca 76100, Malaysia

⁵Faculty of Electrical Engineering, Universiti Teknikal Malaysia Melaka, Malacca 76100, Malaysia

Corresponding author: Tiu Zian Cheak (ziancheak.tiu@newinti.edu.my)

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ABSTRACT Continuous exposure to formaldehyde may cause injury to the central nervous, respiratory, blood, and immunological systems. Tapered U-shape plastic optical fiber (POF) coated with zinc oxide nanorods was evaluated at wavelength of 645 nm for formaldehyde vapor sensing within a concentration range from 5% to 20%. The tapered POF with 500 μm waist diameter was prepared using chemical etching technique. Zinc oxide nanorods were synthesized using hydrothermal method and growth for 12 hours on the tapered POF. The proposed sensor exhibited a good response to formaldehyde concentration ranging from 5% to 20% with sensitivity and linearity measured to be 0.00543V/% and 98.58%, respectively. Excellent measurement stability was observed when the concentrations from 5% and above are maintain over a 600 second period. Highest difference voltage was produced by 0.0958V due to the more scattering effect of ZnO nanorods at 20% of formaldehyde concentration. This proposed sensor might be also used to detect air pollution produced not just by formaldehyde vapor, but also by other dangerous or poisonous vapors or gases.

INDEX TERMS Formaldehyde sensing, zinc oxide nanorods, tapered, U-shape, plastic optical fiber.

I. INTRODUCTION

Formaldehyde (HCHO) is a colourless, rapidly polymerised gas at ambient temperature that pollutes both indoor and outdoor air [1]. The majority of us use formaldehyde-containing products on a daily basis. It is found in a broad number of items, including fabrics, foods, disinfectants, photo processing chemicals, preservatives, and even beauty products [2], [3], [4]. Formaldehyde is a highly toxic volatile organic compound (VOC) that becomes dangerous to humans when concentrations are above a certain level. Considering this issue, the World Health Organization (WHO) published a recommendation for safe exposure settings, specifying that a concentration of 0.08 ppm should not be exposed for

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more than 30 minutes indoors [5]. The World Health Organization has determined that formaldehyde is teratogenic, and the International Agency for Research on Cancer has classified it as a human carcinogen (IARC) [6], [7], [8]. Dizziness, fatigue, headaches, and irritation of the skin, nose, eyes, and throat have all been reported as temporary symptoms.

Several formaldehyde detection methods have been proposed and demonstrated in the previous studies, including cataluminescence [9], spectroscopy [10], bio-sniffer [11], and chemiresistor [12]. However, some of the methods utilized need high temperatures to work, which uses more electricity. Due to this disadvantage, fiber-optic sensor is preferred in recent sensing applications because it can be operated in room temperature and immune to electromagnetic interference (EMI) [13], [14]. In addition, due to low cost sensor

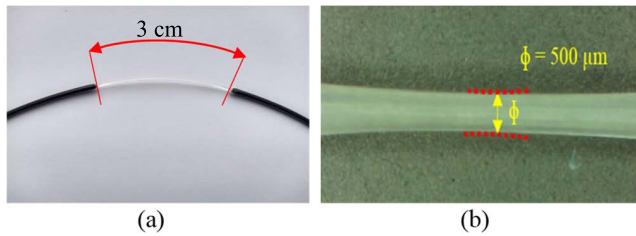


FIGURE 1. Waist diameter of 500 μm tapered POF (a) Overview and (b) Center.

fabrication [15], [16] and strength in mechanical [17], [18], many works have also been focused on the use of plastic optical fiber (POF).

To date, many researchers are interested in nanomaterial based sensor devices because of its mechanical properties and electrical [19], unique optical [20], where it is suitable for a wide range of applications including gas sensors, photocatalysis, superconductors, and optoelectronic devices [21], [22], [23]. Owing to this, a variety of nanomaterials from semiconductor metal oxides, including zinc oxide (ZnO) [24], [25], [26], tin oxide (SnO₂) [27], titanium dioxide (TiO₂) [28], indium oxide (In₂O₃) [29], and nickel oxide (NiO) [30] have been used to fabricate gas sensors. However, zinc oxide (ZnO) has higher electrical compatibility and biocompatibility [31], as well as its great chemical stability [32]. In addition, ZnO nanorods are a practical material among researchers due to huge advantages such as simple preparation [33] and low-cost [34]. Furthermore, due to its excellent optical transparency and ability to work within the visible range, ZnO is suited for optoelectronic applications [23].

This paper reports a tapered U-shape POF coated with ZnO nanorods for formaldehyde vapor sensing at room temperature. The hydrothermal technique was utilized to grow ZnO nanorods on the 500 μm waist diameter of tapered POF before the fiber was bent into a U-shape. The variations of output voltage were recorded on the microcontroller as the changes in light intensity were guided in the POF. The proposed sensor was exposed to formaldehyde vapor concentrations ranging from 5 to 20% at room temperature utilizing a working wavelength of 645 nm.

II. EXPERIMENTAL WORK

A. PREPARATION OF TAPERED POF

The standard POF model SH4001 Super Eska was used in this work, with 980 μm core and 1000 μm cladding diameters, respectively. The jacket of the fiber was removed using a cutter blade for 3 cm length from the center in order to expose the cladding layer. The overview of the polished area was measured using a micrometer during the chemical etching process until desire waist diameters of 500 μm of fiber were obtained as shown in Figure 1 (a). Figure 1(b) shows a microscope view of a 500 μm waist diameter at the center POF after tapering.

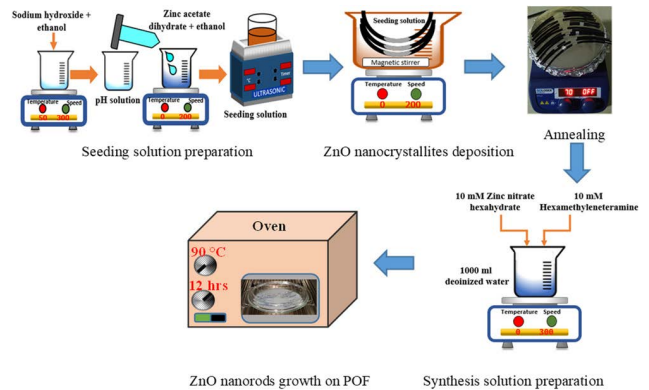


FIGURE 2. Hydrothermal process.

B. PREPARATION OF TAPERED POF COATED WITH ZnO

A hydrothermal technique was used to grow ZnO nanorods on a tapered POF. However, prior to initiating the growth process, the tapered region was treated with a ZnO nanocrystallites deposition method during the seeding process. The seeding solution was made by mixing zinc acetate dihydrate ($\text{Zn}(\text{CH}_3\text{COO})_2$) and ethanol. Then, the solution was mixed with pH control solution, which is formed by mixing sodium hydroxide and ethanol. Thus, the deposition of ZnO nanocrystallites begins by immersing the fiber sample in the seeding solution and stirring the solution at a 200 rpm speed. Finally, the seeded fiber was anneal for 3 hours at 70 $^\circ\text{C}$ on the heater before initiating growing activity.

Throughout the growth phase, a synthesis solution of 10 mM zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) and hexamethylenetetramine ($(\text{CH}_2)_6\text{N}_4$) was utilized. The seeded fiber was immersed in the synthesis solution and dried for 12 hours at 90 $^\circ\text{C}$ in the oven. The synthesis solution was replaced every 5 hours with a new solution to maintain a uniform rate of ZnO growth. The seeding and growth processes were followed according to the previous methods reported in [35] and [36]. Figure 2 show the hydrothermal process of ZnO nanorods coated around the whole surface of the tapered POF.

C. PREPARATION OF TAPERED U-SHAPE POF COATED WITH ZnO

After the process was completed, the both of fiber tips were inserted into a plastic holder to form a U-shape structure with a radius of 3 cm. Figure 3 show the tapered U-shape fiber coated with ZnO nanorods fabrication with diameter of 6 cm.

D. PREPARATION OF FORMALIN

The formalin concentration was measured by diluting the formaldehyde with DI water. The required concentration was calculated using the formula from Equation 1 [24].

$$M1V1 = M2V2$$

where $M1$ specifies the molarity concentration of the concentrated solution, $V1$ specifies the volume of the concentrated solution, $M2$ specifies the molarity concentration of the dilute

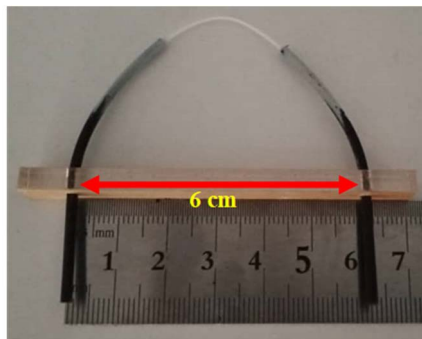


FIGURE 3. Tapered U-shape fiber coated with ZnO nanorods.

TABLE 1. Formalin concentration refractive index.

Concentration (%)	Refractive index
0	1.3326
5	1.3382
10	1.3444
15	1.3502
20	1.3558

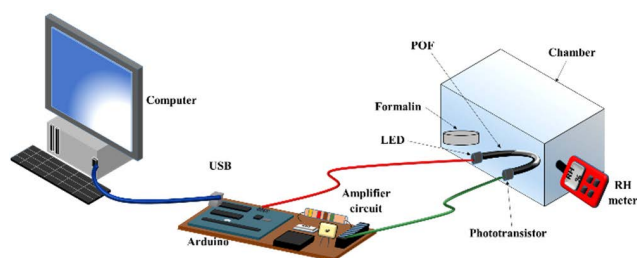


FIGURE 4. Experimental setup for formaldehyde sensing.

solution, and V_2 specifies the volume of the dilute solution. The total concentration volume (100 ml) was fixed for each concentration at 5, 10, 15, and 20% to preserve reliability throughout the test. Then, to confirm the final mixture of DI water and formaldehyde, the refractive index of each concentration were measured using a refractometer (PAL-RI, Atago, Japan). Table 1 shows the refractive index of each formalin concentration. Since formaldehyde has a slightly higher RI number, the substance can only be detected when it is fully evaporated at a high humidity level, assuming the environment is fully covered by the humidity and formalin vapor.

E. EXPERIMENTAL SETUP OF FORMALDEHYDE VAPOR SENSING

Figure 4 presents the experiment setup for formaldehyde vapor sensing. The tapered fiber waist diameter of $500 \mu\text{m}$ was placed inside the controlled chamber with a size of $0.13 \text{ m} \times 0.9 \text{ m} \times 0.6 \text{ m}$. Then, the light source of POF LED (Industrial Fiber Optics, USA) with 645 nm wavelength was used to investigate the output voltage of the sensor when it was exposed to different formalin concentrations.

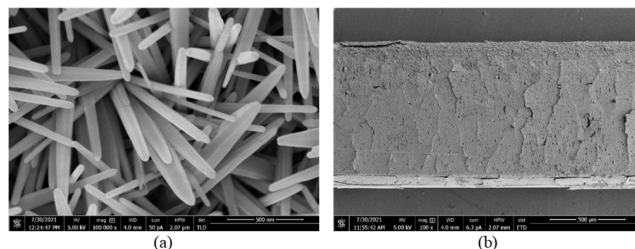


FIGURE 5. Morphology of ZnO nanorods on POF (a) 100kx (b) 100x.

A temperature and humidity meter (Model UT333, Uni-Trend Technology, China) were mounted within the chamber to provide a reference of formalin spread level. Firstly, the controlled chamber was set at 55 % at a 26 °C of humidity level in order to duplicate the real user environment. Following that, 30 ml of formalin was poured into the beaker and allowed it to naturally evaporate and spread throughout the chamber. Then, the mixture of formalin and water will cause environment is filled with humidity and formalin concentration when it is fully evaporated. In this case, we use humidity with 0% formalin concentration as a reference to distinguish between formalin concentrations. Thus, the humidity level will increase regularly throughout this period because water from the solution is readily available. The output voltage was measured when the humidity meter display at 80, 85, and 90%. This procedure was conducted for formalin concentrations of 5, 10, 15, and 20%, and the results for each concentration were compared to 0% (DI water). A phototransistor (Model IF-D92, Industrial Fiber Optics, USA) was used to detect the voltage changes and record in the computer using Arduino platform. During formalin exposure, adsorption and desorption of formalin vapor on the ZnO surface alters the refractive index of ZnO, which modifies the light scattering pattern inside the fiber and changes the output voltage [37], [38]. The strategy for detecting with ZnO nanorods was based on how the refractive index and electrical conductivity of the area around the rods changed during the absorption process [39].

III. RESULTS AND DISCUSSION

Figure 5 illustrates field emission scanning electron microscopy (FESEM) images of hydrothermally grown ZnO on tapered U-shape POF. The morphologies validated the hexagonal wurtzite of ZnO nanorods and were comprised of a large number of superfine nanorods on the fiber. This structure also has been observed in [40]. The height and diameter of ZnO nanorods were in the range of 700 to 1000 nm and 40 to 70 nm respectively.

The chemical components in the POFs were determined using energy dispersive X-ray (EDX) analysis with a 10 keV working voltage. The topcoat layer of the tapering U-shaped POF had 79.02% zinc and 20.98% oxygen as shown in Figure 6. This showed that ZnO was used as the material for sensing formaldehyde vapor.

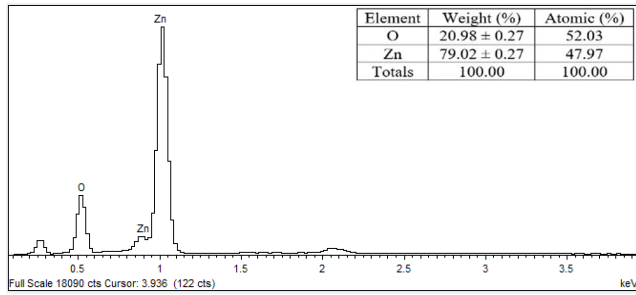


FIGURE 6. EDX elemental analysis of ZnO nanorods on tapered U-shape POF.

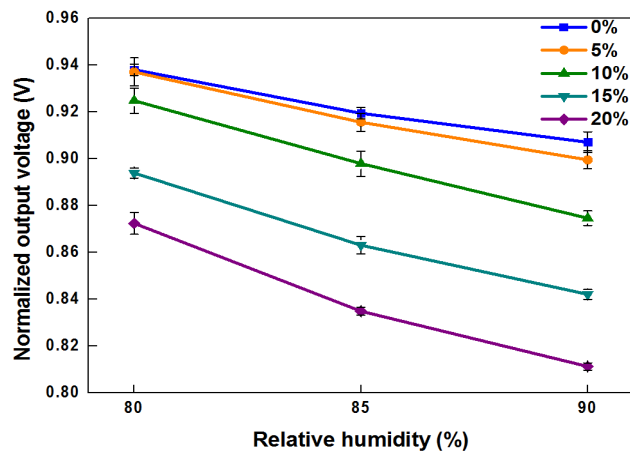


FIGURE 7. Formaldehyde vapor sensing response.

The formaldehyde vapor sensing response against humidity for five different formalin concentrations is shown in Figure 7. RH values ranging from 80% to 90% were used to examine the distribution of formaldehyde vapor throughout the chamber. According to the graph, the proposed sensor responded linearly to change in formaldehyde vapor concentration caused by the ZnO light scattering effect inside the fiber. This is attributed to ZnO, which has a good electrical conductivity. It attracts the formalin molecules to increase the effective refractive index at ZnO coating around the fiber [41]. Therefore, higher light loss leakage at the sensor area and a drop in output voltage was observed when higher formalin concentration exposed to the sensor.

Figure 8 shows the voltage difference observed for various formalin concentrations ranging from 0% to 20% at three different humidities; 80, 85 and 90% RH. According to the bar graph, the voltage variations do not significantly affect the 0% to 10% formalin concentration at 80% and 85% RH, respectively. This might be because the formalin content did not entirely change to formaldehyde vapor. The gas concentration reflected by the change in conductivity is significantly affected by the subsequent desorption of reaction products and the catalytic dopant [42]. As a result, the voltage difference for 0% to 20% of formalin concentration demonstrated a significant increment when the RH level reach at 90%. At this

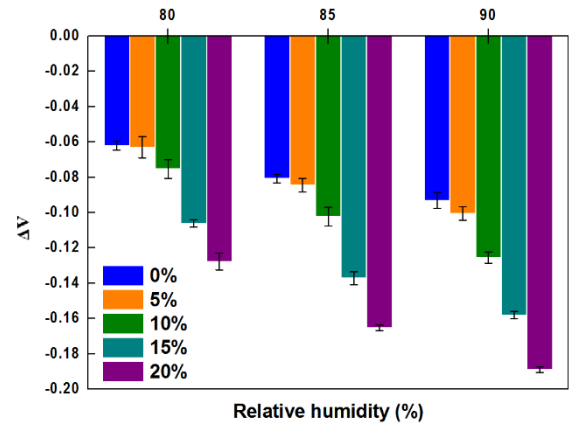


FIGURE 8. Voltage difference of 0% to 20% formalin concentration at 80% to 90% RH.

TABLE 2. Reduction of output voltage at 90 % RH.

Formalin concentration (%)	ΔV (V)
5	-0.0075
10	-0.0324
15	-0.0650
20	-0.0958

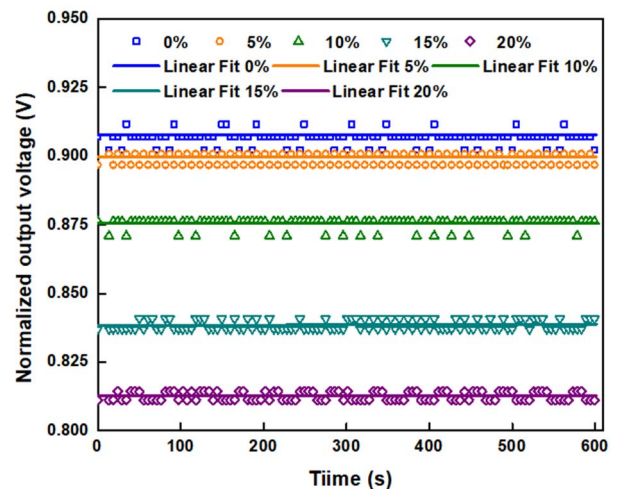


FIGURE 9. Stability performance of 0% to 20% formalin concentration in 600 s at 90% RH.

time, the chamber might be considered completely saturated with vapor. Furthermore, natural evaporation of formalin takes a long time, so the occurrence of saturated vapor was validated by measuring the drop in output voltage at 90% RH compared to DI water. Table 2 shows the significant drop in output voltage for formalin concentrations ranging from 5% to 20%. The total drop was detected at 0.0958 V when the sensor was exposed to a higher concentration of 20%.

Figure 9 illustrates the stability performance of 0% to 20% formalin concentration in 600 s at 90% RH. In this

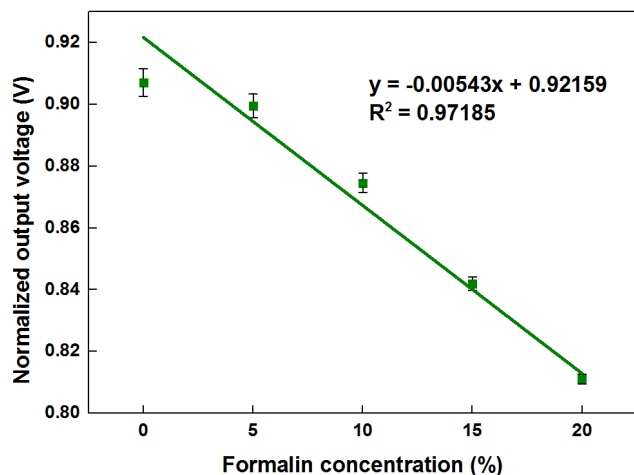


FIGURE 10. Sensor performance at 90% RH.

TABLE 3. Characteristics of sensor.

Parameters	Performance
Linearity (%)	98.58
Sensitivity (V/%)	-0.00543
Average Standard deviation (V)	0.00746
Resolution (%)	1.37385

experiment, all the formalin concentrations produce stable output voltage with slope range between $8.53975e-9$ to $9.50776e-7$. These numbers are very small and very reasonable for such a sensor system. As a result, the proposed sensor shows the ability of the optical fiber sensor to maintain its performance characteristic for a certain period of time [43].

Figure 10 presents the proposed sensor performance at 90% of the RH level. According to the graph, the output voltage decreases as the formalin concentration increases. This behaviour is accordance with the finding reported in [36]. The plot of formalin with 0% concentration was distant from the linear fit line because it operates as a zero reference that is unaffected by formalin. The proposed sensor was determined to have an average linearity of 98.06% as shown in Table 3. Furthermore, the sensor has a sensitivity of 0.00543 V/% and can detect formaldehyde vapor concentrations above 5% with a standard deviation of 0.00746 V.

Due to their high surface-to-volume ratio, zinc oxide nanorods are capable of increasing the adsorption of vapor molecules on their surface [44]. According to Table 1, when the formalin concentration increases, the refractive index value also increases. Therefore, during the adsorption process, the vapor modulates the refractive index around the ZnO nanorods, hence altering their electrical conductivity. The refractive index of ZnO was changed as the electrical conductivity changed, resulting in changes in light scattering patterns on ZnO nanorods. Here, the effects of higher light leakage will be observed when the vapor concentration is increased.

IV. CONCLUSION

A tapered U-shape plastic optical fiber coated with ZnO nanorods using a hydrothermal technique was demonstrated to detect formaldehyde vapor. The fabricated sensor exhibited a substantial sensitivity to concentrations ranging from 5% to 20%. The drop in output voltage was determined to be 0.0958 V for the concentration at 20% concentrations with comparison to 0% which is DI water. The sensor's response demonstrated an excellent linearity of 98.58% across all vapor concentrations. The sensor's sensitivity was measured to be -0.00543 V/%. Finally, when tested continuously for 600 seconds at a 5% concentration variation, the sensor displayed exceptional stability.

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SITI H. JOHARI received the bachelor's degree in electronic engineering (industrial electronics) from Universiti Teknikal Malaysia Melaka (UTeM), in 2005, and the M.Eng. degree in control and automation from the University of Malaya (UM), Malaysia, in 2014, where she is currently pursuing the Ph.D. degree in photonic research with the Photonics Engineering Laboratory, Faculty of Engineering. She was a Test Engineer with the Agilent Technologies Department, Venture Electronic Services, from 2005 to 2010. From 2010 to 2014, she was a Teaching Engineer with UTeM and then, she was appointed as a Lecturer, in 2015.



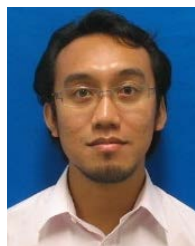
TIU ZIAN CHEAK received the Ph.D. degree in engineering from the University of Malaya (UM), Malaysia. He has registered as a Chartered Engineer, under Institution of Engineering and Technology (IET), and a Professional Engineer, under the Board of Engineering Malaysia (BEM). He is currently a Professor with the Faculty of Engineering and Quantity Surveying, INTI International University, Malaysia.



HAZIEZOL HELMI MOHD YUSOF received the Bachelor of Electronic Engineering degree (Hons.) in industrial electronics from Kolej Universiti Teknikal Malaysia Melaka (KUTKM), in 2005, and the M.Eng. and Ph.D. degrees from the University of Malaya, in 2015 and 2019, respectively. He worked as an Electrical Validation Engineer with Intel Microelectronics (M) Sdn. Bhd., from 2005 to 2010. He is currently working as a Senior Lecturer with the Faculty of Electronic and Computer Engineering, Universiti Teknikal Malaysia Melaka (UTeM), Malaysia.



HAZLI RAFIS ABDUL RAHIM received the Ph.D. degree in optical fiber sensor from the University of Malaya, Malaysia, in March 2017. His Ph.D. research focused on the structured growth of zinc oxide nanorods on optical fiber and light side coupling towards sensing applications. He is currently a Senior Lecturer with the Faculty of Electronic and Computer Engineering, Universiti Teknikal Malaysia Melaka, Malaysia. His research interest includes optical fiber sensor for various applications.



MD ASHADI M. JOHARI received the degree in electronic engineering (industry) from Universiti Teknikal Malaysia Melaka, Malaysia, in 2006, the master's degree in telecommunication engineering from the University of Malaya, Malaysia, in 2011, and the Ph.D. degree from the Photonic Engineering Laboratory, University of Malaya, in 2021. He was worked as an QA Engineer with NCMC, Malaysia, from 2006 to 2009, and a Test Engineer with AEX System, from 2009 to 2011. Since 2011, he has been working as a Lecturer with Universiti Teknikal Malaysia Melaka.



MOHD HAFIZ JALI received the B.Sc. degree (Hons.) in electrical and electronic engineering from the MARA University of Technology (UiTM), Malaysia, in 2007, the M.Eng. degree (Hons.) in industrial electronics and control from the University of Malaya (UM), Malaysia, in 2012, and the Ph.D. degree from the Photonic Engineering Laboratory, University of Malaya, in 2020. He was a Test Engineer with Automotive Company, Proton Sdn. Bhd., from 2007 to 2010, under the Complete Vehicle Testing (CVT) Department. In 2012, he was appointed as a Lecturer with Universiti Teknikal Malaysia Melaka (UTeM), Malaysia.



SULAIMAN WADI HARUN received the B.Eng. degree in electrical and electronics system engineering from the Nagaoka University of Technology, Japan, in 1996, and the M.Sc. and Ph.D. degrees in photonics from the University of Malaya, in 2001 and 2004, respectively. He is currently a Senior Professor with the Faculty of Engineering, University of Malaya. His research interests include fiber optic active and passive devices. He is a fellow of the Academy of Science Malaysia. He is also the Founder and an Advisor of the Optical Society of Malaysia.

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