Effects of cladding modification on the sensing potential of intrinsic polymer optical fiber as a VOCs sensor

F. ABDURRAHMAN[a], N. ARSAD[a,b*], S. SHAARI[b], A. ASHRIF A. BAKAR[a], P. SUSTHITHA MENON[b]
[a]*Department of Electrical, Electronic and System Engineering, The National University of Malaysia, Selangor, Malaysia*
[b]*Photonics Laboratory, Institute of Microengineering and Nanoelectronics, The National University of Malaysia, Selangor, Malaysia*

The effects of cladding modification on polymer optical fiber (POF) are investigated theoretically and experimentally as a preliminary step in developing a fiber optic intrinsic sensor. Etching technique critically affects the geometrical result of the fiber during the initial stage of fabricating the intrinsic fiber probe. In order to create intrinsic fiber sensor, the cladding must be entirely removed as to generate thorough evanescent wave dispersion into the sensing target. In this research we demonstrate the investigation on two cladding modification methods; chemical etching and mechanical etching. Fiber optics with different etched cladding's length and diameter are fabricated and later tested on volatile organic compound inside the testing chamber. The modified sensor probe exhibits good response and is able to effectively detect the presence of methanol vapor ranging from 95% to 20% in the chamber.

(Received January 29, 2015; accepted May 7, 2015)

Keyword: Fiber intrinsic sensor, Chemical etching, Evanescent, Sensor probe

1. Introduction

Polymer optical fiber has been employed expansively as a sensor, and this is confirmed by numerous researches being performed and reported since the 1970's. This device possesses significant advantages over electronics sensors, in which it has remote measuring capability, electromagnetic field immunity, lower weight, and most importantly does not require bias for its utilization [1-3]. Utilization of fiber as a sensor can be classified as either intrinsic or extrinsic. Evanescent method falls under the intrinsic category; the unclad section of the fiber is used as the sensor probe. This unclad section is where the interaction between the exposed light and the sample will take place. In the case of using optical fiber for the purpose of detecting gas, liquid, chemical concentration level, and biological mechanisms, a perfect geometric fiber core is essential [4].

A Fiber optic intrinsic sensor offers the benefit of in line measurement that occurs at the interaction area of the fiber sensor. Based on total internal reflection (TIR) phenomenon, the transparent fiber core carries the propagating light throughout its overall length. The sensing process is realized when the light comes into contact with, and is absorbed by the sample. This process takes place at one section of the fiber; that is the "interaction area" of the sensor. Creation of the interaction area requires the removal of cladding layer, so that the light inside the fiber's core will "leak" into the surrounding sample, inducing a phenomenon known as evanescent field interaction. In order to remove the cladding, the fiber needs to be immersed in a specific chemical solvent for a certain amount of time with constant monitoring of the etching rate. Incomplete etching result can lead to failure of evanescent field intensity to cover the entire absorbing medium in the sensing area [5].

The etching process is the preliminary step in fabricating the fiber as an intrinsic sensor. The etching rate of the fiber can be monitored manually by measuring the cladding's diameter size using a digital vernier caliper after each time it undergone acetone chemical etching procedure. Scanning Electron Microscopy (SEM) provides the magnified image of the etched cladding and accurate measurements of all the perimeters involved while power meter gives out the output loss results. Understanding of the diameter size is important in order to identify the transformation that will occur to the fiber during the etching process [6] . The purpose of this research is to produce a fiber core diameter with a better geometry that will be employed as sensing probe for the intrinsic evanescent fiber optic sensor to detect the presence of volatile organic compounds.

Apart from chemical etching method, there exists another method being practiced in a multitude of researches called the mechanical etching method. Both methods are proven to be capable of achieving uniform removal rate albeit having different qualities. In this study, we uses both methods in order to compare the advantages and disadvantages of each, and to find out which method is the most compatible with our setup.

2. Physical identifying of polymer optical fibre

In this research the type of polymer optical fiber being used is the cylindrical wave guide type having fluorinated polymer as the cladding material and polymethylmethacrylate (PMMA) as the core. Number of modes of the POF is explicitly defined by its refractive index, core diameter, and wavelength interrogation. The light inside the fiber core that remains at the surface after the cladding has been etched away possesses a dominant number of modes [7]. The penetration depth of the evanescent field decays exponentially, and the proportion of power rises significantly in the waist area of the etched fiber section[8]. V number can be determined by the source of light that is being transmitted in the fiber, as expressed in the following equation:

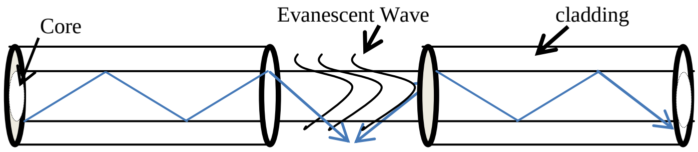


Fig.1. Evanescent wave of the etched polymer optical fiber

$$V = \frac{2\pi a_0}{\lambda}\sqrt{{n_{co}}^2 + {n_{cl}}^2} \qquad (1)$$

a_o is the radius of the core, n_{co} is the core refractive index, and n_{cl} is the fiber cladding refractive index. Once the POF cladding is removed, the refractive index of the cladding will be replaced by the refractive index value of air or absorptive media that surrounds the core during the etching process.

$$\alpha(t_{etch}) = -\frac{1}{2\pi r(t_{etch}).L}\, log_{10}\left(\frac{P(t_{etch})}{P_0(t_{etch})}\right) \qquad (2)$$

Absorption coefficient of fiber is symbolized by α, while L is the length of the etched portion, r is radius of fiber cross section based on etching time, P is the optical power of the light source that propagates inside the fiber, and P_0 is the optical output power based on fiber's etching time. Equation above will yield $\alpha \approx 2k$ [9], if the propagation constants are complex numbers, and the refractive index can be clearly expressed as follows:

$$k = \frac{2\pi}{\lambda} = \frac{2\pi f}{v} = \left(\frac{\omega}{c}\right) n_t \qquad (3)$$

If the complex refractive index is identified by its real and imaginary parts we will get:

$$n_{comp} = n_x + in_l \qquad (4)$$

Where n_x is the general refractive index and n_l is called the extinction coefficient of the polymer optical fiber. n_l value will be 1.4894 for Lambda = 650 nm. The POF's refractive index is 1.49.

3. Procedures and Materials

3.1. Strain - Free Etching Technique with Flat Vessel

For this procedure, pure acetone was utilized as the chemical solvent to remove a portion of the fiber's cladding to obtain a smooth surface, together with a 30 cm long flat vessel [6, 10]. 3 identical optical fibers of the multimode type, model Mitsubishi Eska 4001 with core refractive index of 1.49 and numerical aperture of 0.5 was used. The POF core is made from a pure polymethylmethacrylate (PMMA) with fluoroplymer cladding. The POF core and cladding diameter is 980 µm and 20 µm, respectively. The fibers were etched at the middle of their bodies, with each fibers received different etched cladding's length. Besides ensuring the POFs are in a strain-free state, special care must be put on the temperature of the experimental setup.

During the etching process, the temperature must be carefully controlled as high temperature will cause fiber breakage due to accelerate evaporation of acetone that leads to uncontrolled etching process. Fibers taken from the POF roll should be maintained in a bend condition to avoid strain during the etching process inside the acetone-filled flat vessel. The experimental setup for this procedure is depicted in Fig. 2.

Loss measurement was performed during the chemical etching progression by coupling the fiber with a power meter and introducing light into the fiber. The expected loss within the fiber was indicated by the propagation mode radiating outside the core while the layer of cladding was gradually stripped away.

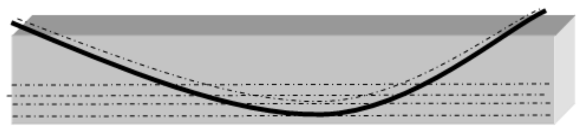


Fig.2. Experimental setup for the strain-free POF chemical etching procedure

3.2. Mechanical Etching

The mechanical etching procedure made use of sandpaper and abrasive aluminum oxide polishing film of various sizes to etch the fiber's cladding. This technique was done at a very low speed to guarantee the production of a scratch-free surface. Mechanical etching claims the favors of being reproducible, delivers great end result in the form of a smooth fiber surface, and is efficient at avoiding fiber breakage [11, 12]. This method is preferred for the creation of intrinsic sensor probe in many researches.

3.3. Propagation Loss Measurement

Power output measurement was executed to monitor power change in each phase of etching process. Figure 3 illustrates the schematic diagram for measuring the mode of propagation loss caused by the reduction of cladding layer in fiber. A broadband light source was employed to transmit the light through the etched fiber from one end towards the S-120 photodiode coupled to the other end of the fiber. The output data were measured using Thorlab power meter model PM100 and the measured data of power loss are shown in fig. 7. The graphs indicate that there exists a proportional relationship between the thickness of the cladding layer and the output measurement; as the thickness decreases so does the measured output. Also, length of the etched cladding influences the decrement of the propagation loss output.

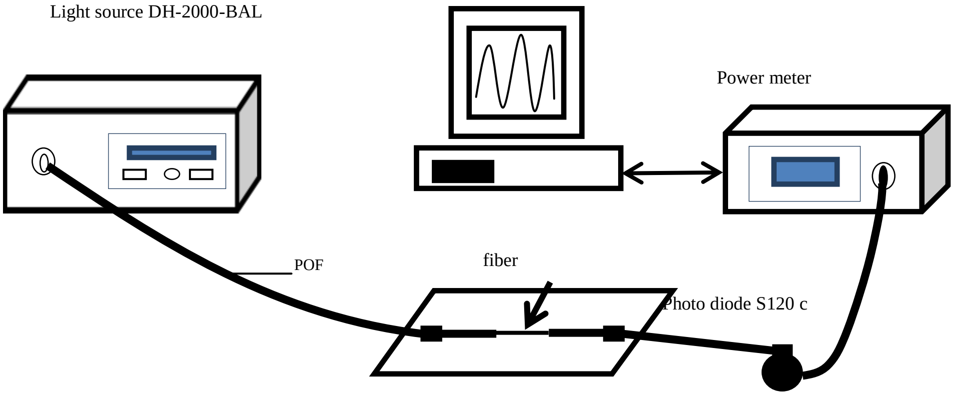


Fig.3. Schematic diagram for measuring propagation loss in POF

3.3 Light Intensity Measurement

Light that is guided through an optical fiber will undergo a decline in intensity whenever there is a reduction in the cladding layer's thickness. In this research the light intensity measurement was done by channeling a DH-200BAL broadband light source through the etched fiber and measuring the output intensity using a HR spectrometer, as illustrated in Fig. 4. The broadband light source has a wavelength response length of between 200 nm to 1100 nm. The etching process produced a tapered section in the middle of the polymer optical fiber which shown a constant decrement in light intensity, proving the existence of evanescent field. This tapered section is where the interaction between the light and the targeted sample will occur.

4. Characteristic and discussion

4.1 Characteristic of Chemical Etching Method

The plastic optical fibers were used in the experiment with each fiber subjected to different length of unjacketed section at the middle of its body; 7 cm, 8 cm, and 9 cm respectively. The cladding was etched horizontally to avoid stress because once the fiber is immersed in the acetone it will be extremely susceptible to breakage. Chemical etching process removed the thick layer of cladding, leaving only the bare fiber core.

The result of the experiment is depicted in the form of a graph in Fig. 5 to describe the behaviour of the cladding's etching rate. As time elapsed the layer of cladding was gradually etched away, however the diameter of the removed cladding cannot be ascertained.

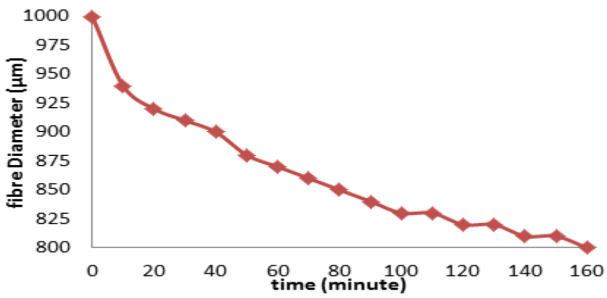


Fig. 4. Chemical etching rate of POF

This experiment was done in a low temperature room to avoid uncontrolled etching rate as a result of high temperature. The diameter of the fiber was periodically measured every 10 minutes using vernier caliper and microscope. Before measurements were made, the fiber was immersed in deionized water to neutralize the acetone on the fiber's surface, thus halting the etching process. This step is necessary in order to avoid cloudy or fractured surface caused by acetone residue.

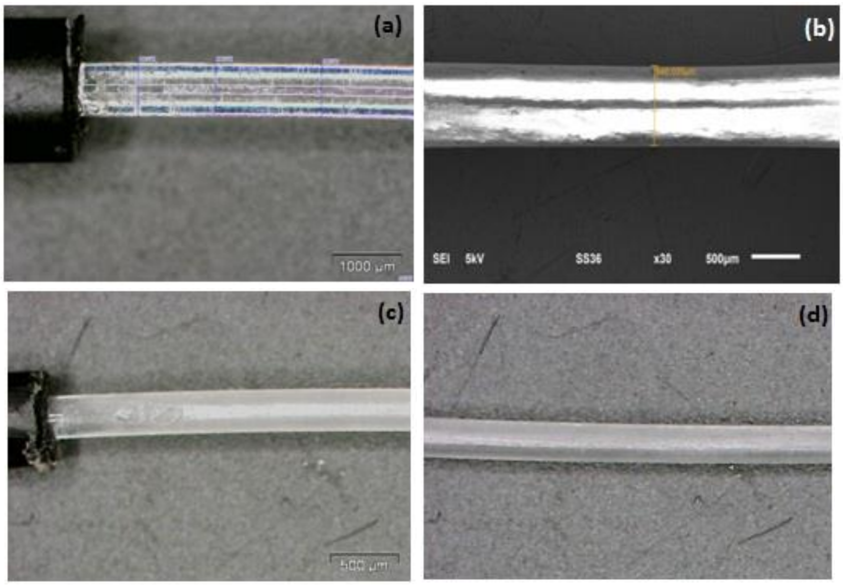


Fig. 5. POF etching result (a) Microscopic picture of chemically etched POF (b) SEM Image of 0.8 mm chemically etched POF (c) Microscopic picture of mechanically etched POF (d) Microscopic picture of 0.8 mm POF 0.8

4.2 Characteristic of POF's propagation loss measurement

Changes induced to the tapered waist of the fiber's unclad section will influence the output power of the transmitted light. Every diameter of cladding layer removed will further restrict the propagation of light beyond the waist portion. Same approach was used to measure propagation loss in both chemically and mechanically etched POFs, and both methods showed similar power transmittance behaviour. Higher mode order of the multimode fiber was transferred, whereas some lower modes were redistributed. As the diameter of the cladding decreases, the evanescent field and the amount of power within the field increase. Hence, smaller radius of the fiber with lower numerical aperture will lead to higher absorbance of the sample, directly translating into improved sensitivity of the fiber sensor [13]. Reduction of the numerical aperture occurs within the etching process in accordance to the number of modes. Approximation of the guided modes can be calculated using the following equation [6]:

$$M \approx (Kr_c N)^2 = (Kr_c\sqrt{n_0^2 - n_1^2}\ )^2 \qquad (2)$$

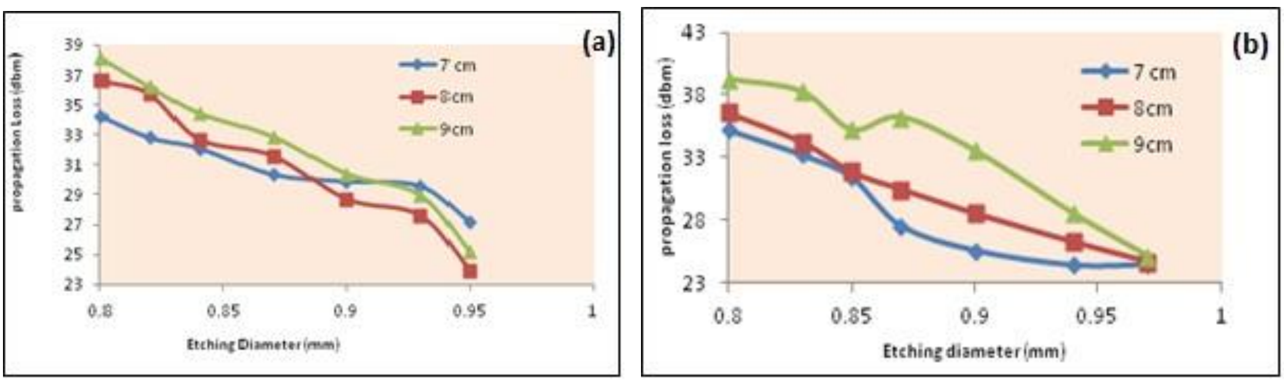
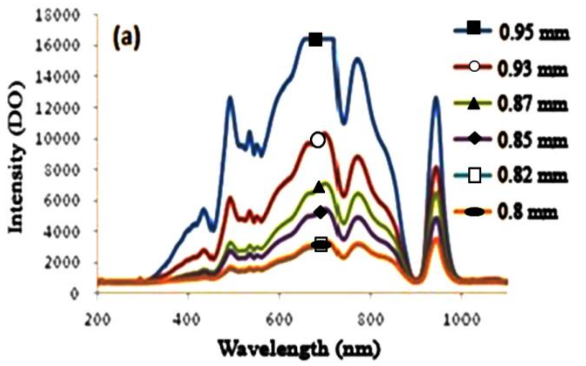


Fig 6. Propagation loss measurement (a) Propagation loss of mechanically etched POF (b) Propagation loss of chemically etched POF

4.3 Characteristic of Light Intensity Measurement

The change in light intensity of the POF output is portrayed in the form of an intensity difference graph as shown in Fig. 8. Removal of the cladding caused a decline in the output intensity. This condition was expected to occur as it indicates effective penetration depth of evanescent field from the fiber's surface into the targeted sample surrounding the core, and thus successful interaction between the two components within the experimental approach [14].



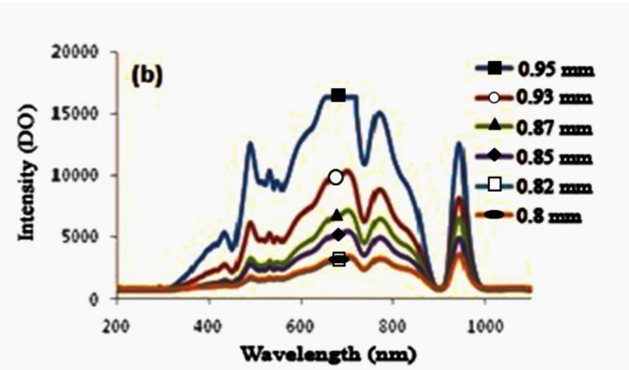


Fig 7. Light intensity measurement (a) Light intensity result of mechanically etched POF (b) Light intensity result of chemically etched POF

5. Fibre optic intrinsic sensor detection of methanol vapour

5.1 Experiment

The experimental setup to measure the absorbance spectrum of the previously prepared fibers is as shown in Fig. 9. A white light source (model: DH-2000-BAL Ocean Optics) and a spectrometer (model: HR4000CG: UV-NIR) with full spectral output compromising a wavelength range of 200 nm to 1100 nm were used in this experiment. A personal computer was utilized as an interface to analyze the signal generated from the spectrometer.

As for the gas piping flow, it consisted of a vacuum pump, a handheld digital pressure meter (model: HHP-90), a testing chamber, and a modified condenser. Air in the testing chamber was first pumped out using the vacuum pump. The pressure in the testing chamber was monitored using the digital pressure meter. Methanol vapor was then slowly allowed to enter the testing chamber to interact with the fibers. As stated earlier in the theoretical section, the evanescence absorbance is affected by the fiber's parameters. Therefore, in this experiment the prepared fibers were prepared in three different unclad section's length; 7cm, 8cm, and 9cm. Comparison were made on the absorbance spectrum between two etching methods used on the fibers namely mechanical etching and chemical etching.

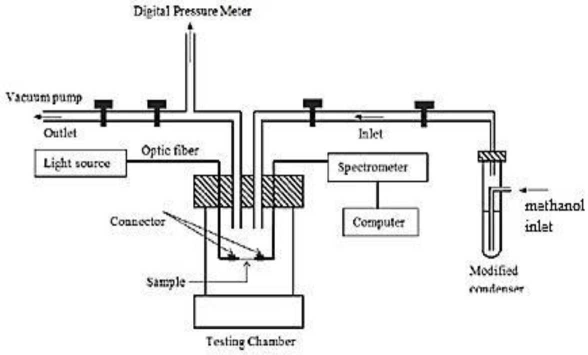


Fig. 8. Schematic diagram of the fiber optic VOCs sensor experimental set up.

5.2 Result and Discussion

The light that radiates outside the core as a result of evanescent field wave is expected to react to the methanol vapour, and when the reaction occurs at the surface of the etched POF there will be changes to the internal reflection and intensity of the propagating light inside the fiber due to changes in the refractive index of the air around the core. Optical probes from both cladding modification method (chemical and mechanical) were exposed to the methanol vapour and the results obtained were compared to see the difference in their performance. The absorption spectrum coefficients graph showing the absorption of methanol on chemically and mechanically modified cladding is presented in Fig. 9. Absorption of light having a spectral characteristic ranging from 350 nm to 900 nm took place inside the testing chamber when the etched fiber was exposed to the methanol vapor. This condition implies that the modified cladding possesses good reaction as it is able to effectively detect the presence of the volatile organic compound. We were able to obtain excellent sensing probe response on the presence of the methanol vapor by enhancing the process accuracy for both cladding modification methods.[15]

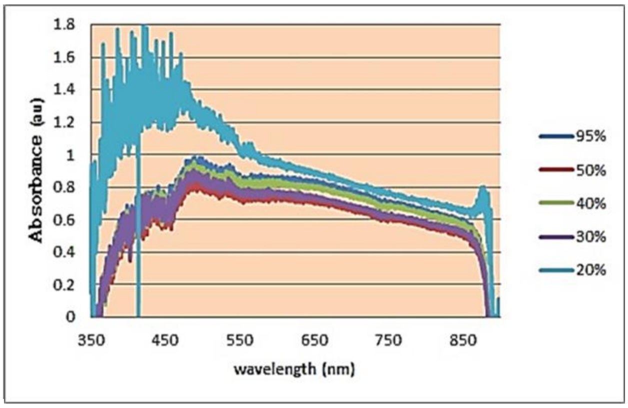


Fig.9. Absorption spectra of the methanol vapour inside the testing chamber.

Fig. 10 shows the recording of the change in absorbance optical measurement during interaction of gas and etched POF inside the chamber test. When the gas pump in and interact with the fiber it will show the increment of absorbance and when the gas pump out interaction gas and fiber will decrease. The concentration of methanol expose is similar, to observe the response time of the sensor during the presence of methanol

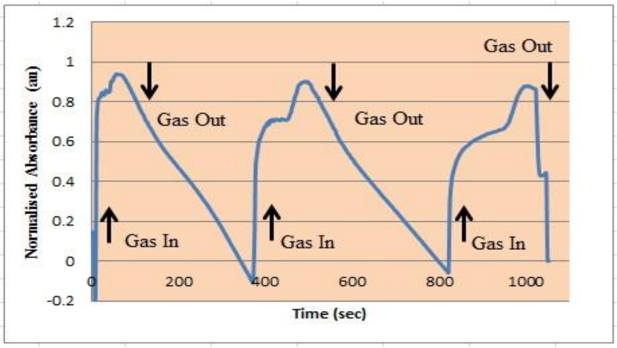


Fig.10. Response of The polymer optical fiber to methanol vapour inside the testing chamber

5. Conclusion

A systematic and easy-to-handle fabrication technique for the fiber optic intrinsic sensor using multimode optical fiber with comparison on two cladding modification methods namely chemical etching and mechanical etching was successfully developed. The working principal of this research involved the initial process of fabricating the POF as an intrinsic sensor probe, followed by characterizing the phenomena that occurred during both chemical and mechanical etching processes in order to produce an intrinsic sensor with the best performance. Precise control is the main key in obtaining an evanescent wave intrinsic sensor having high sensitivity.

Acknowledgement

The main author (Faizar Abdurrahman) is grateful to the project leader of GUP-2013-041 and ERGS/1/2012/TK02/UKM.02/6 for the financial support. All authors are grateful to Photonics Lab for providing the laboratory facilities to carry out the research.

Reference

[1] M. El-Sherif, L. Bansal, J. Yuan, Sensors **7**(12), 3100 (2007).
[2] F. Abdurrahman, N. Arsad, H. Ramza, Chinese Optics Letters,. **12**(4), 043002 (2014).
[3] X. Cheng, et al., Chinese Optics Letters, **9**(2), 020602 (2011).
[4] A. Kersey, A. Dandridge, IEEE Transactions on, **13**(1), 137 (1990).
[5] C.R. Taitt, G.P. Anderson, F.S. Ligler, Biosensors and Bioelectronics, **20**(12), 2470 (2005).
[6] D.F., Merchant, P.J. Scully, N.F. Schmitt, Sensors and Actuators A: Physical, **76**(1–3), 365 (1999).
[7] A. Leung, et al., Biosensors and Bioelectronics, **21**(12), 2202 (2006).
[8] Y. Xu, A. Cottenden, N.B. Jones, Optics and lasers in engineering, **44**(2), 93 (2006).
[9] F.L. Pedrotti, L.S. Pedrotti, Introduction to optics. Vol. 2. 1993: Prentice-Hall Englewood Cliffs.
[10] M.S. Ab-Rahman, L. Supian, N. Arsad, Optik-International Journal for Light and Electron Optics, **125**(2), 893 (2014).
[11] M. Im, et al., Modification of Plastic Optical Fiber for Side-Illumination, in Human-Computer Interaction. Interaction Platforms and Techniques, J. Jacko, Editor. 2007, Springer Berlin Heidelberg. p. 1123-1129.
[12] B. Renganathan, et al., Sensors and Actuators B: Chemical, **156**(1), 263 (2011).
[13] V. Ruddy, B. MacCraith, J. Murphy, Journal of Applied Physics, **67**(10), 6070 (1990).
[14] V. Sai, et al., Sensors and Actuators B: Chemical, **143**(2), 724 (2010).
[15] F.V.B., de Nazare, et al. Geometric aspects in a novel plastic optical fiber biosensor system. in Instrumentation and Measurement Technology Conference (I2MTC), 2011 IEEE. 2011.

*Corresponding author: norhana@eng.ukm.my
fhayzar@gmail.com