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Enhancement of Sensing Performance for Alcohol in Aqueous Solution using Tapered Optical Fiber Coated with Polyaniline via Air-Brushing Technique

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Abstract

Tapered fibers with superior properties can be used in sensing applications such as humidity sensors, temperature sensors, and refractive index sensors. The main objective of this work is to investigate the influence of Polyaniline (PANI) as a coating in the fabrication of a polymer microfiber for the detection of different concentrations of methanol, ethanol, and propanol. In this study, a high-quality tapered optical fiber is fabricated using the flame-brushing technique and the microfiber is then coated with the polymer Polyaniline (PANI) to detect different types of alcohol at various concentrations. It was found that the sensor using PANI as the coating material on the tapered optical fiber exhibited higher sensitivity to concentration changes of aliphatic alcohol solutions than the bare tapered optical fiber. The improvement in sensitivity for methanol, ethanol, and propanol is 80.17%, 106.43%, and 42.92% respectively. A sensitivity of 0.9755 dBm/%, 1.06 dBm/%, and 1.039 dBm/% for methanol, ethanol, and propanol respectively was achieved by using a tapered PANI-coated optical fiber with a diameter and length of 4 µm and 5 mm respectively. The digital microscope (DM) confirmed the successful coating of PANI on the tapered microfiber which helped to enhance the performance of the sensor. Overall, this work has effectively demonstrated a conductive polymer-coated optical microfiber sensor for alcohol detection that is inexpensive, effective, and easy to set up.

1. Introduction

Optical fibers are considered a feasible sensor because the sensing and measurement have been in existence for over 40 years. Photonic sensors, which were first patented in the mid-1960s, were based on bifurcated fiber bundles, with half of the bundle used to illuminate a surface and the other half to receive the reflected signal [1-2]. With proper calibration, the received signal can provide a highly accurate approximation of the relative position between the end of the fiber and the reflecting surface [3]. A decade later, the introduction of single-mode optical fibers and the idea of incorporating them into interferometers marked a significant advancement, offering substantial engineering benefits over their free-space predecessors fixed to optical tables. However, the primary application of optical fiber technology was in communication [4].

Due to its properties such as compact size, low weight, electromagnetic shielding, excellent stability, and the ability to analyze sensor arrays remotely, optical sensors have long been a popular research topic. Light pulses are transmitted from their source to the destination via fiber optic waveguides [2,5]. A fiber optic is made up of cladding, buffering, and a core that transmits light through total internal reflection while providing protection. Because of their small size, flexibility, and minimal losses, silica fiber optics are widely employed. Although they are widely used in telecommunications, they are also being used more and more in the creation of sensors [6]. Fiber optic sensors can be employed in potentially dangerous conditions and are immune to electromagnetic interference, unlike typical electronic sensors.

Various methods, such as U-bends, tapers, and D-fibers, have been employed to construct refractive index sensors, including fiber-optic displacement sensors. Among these, fiber tapering is the most fundamental method for sensor applications [7]. Recently, tapered fibers have garnered significant interest from researchers worldwide due to their superior performance and versatility in a wide range of sensing applications [8]. The immunity of fiber optic sensors to electromagnetic interference makes them particularly attractive for sensing applications.

Fiber optic sensing has been in high demand for quite some time, paralleling the remarkable advancements in micro and nanotechnology. When the diameter of the tapered fiber is in the micrometer (μm) range, it is referred to as a microfiber, and when it is smaller than 1 nm, it is called nanofiber [9]. Fiber optics with smaller diameters exhibit higher sensitivity and responsiveness, offering a wide dynamic range, minimal attenuation loss, tight optical confinement, and strong evanescent fields [10].

Tapered optical fibers possess these qualities, making them well-suited for remote sensing applications such as humidity, refractive index sensing, vapor sensing, biomedicine, chemical analysis, environmental engineering, and the automotive industry [7]. The enhanced performance of these fibers in various environmental conditions underlines their importance in the continued development of advanced sensing technologies.

Tapered optical fibers are known for their strong evanescent fields, allowing a substantial portion of the transmitted light to interact with the surrounding environment, significantly enhancing their sensing capabilities. This characteristic makes them highly attractive as sensor components, enabling the creation of devices that are extremely small, inexpensive, and lightweight [11].

Microfibers are typically fabricated by heating and stretching standard-diameter glass fibers. Among the various methods used for microfiber production, the flame brushing technique is the most commonly employed due to its ability to maintain a uniform heat source, regulate flame movement, and efficiently extend the fiber length [12]. This method is particularly advantageous because it meets adiabatic criteria, ensuring minimal thermal loss during the tapering process, which is critical for producing high-quality conical fibers.

The precision and control offered by the flame brushing technique are essential for tailoring the physical and optical properties of the tapered fibers, making them suitable for a wide range of sensing applications. These applications include environmental monitoring, biomedical diagnostics, and chemical detection, where the enhanced sensitivity provided by the strong evanescent fields of tapered fibers is crucial. The method's efficiency and ability to produce fibers with consistent quality further underscore its importance in fabricating advanced optical sensors.

The integration of functional materials, such as Polyaniline (PANI), into optical sensors offers a significant enhancement in their performance. PANI has emerged as a promising material for smart sensor development due to its numerous advantages, including ease of synthesis, low cost, good environmental stability, and the unique ability to switch electrically between conductive and resistive states [13-15]. These properties make PANI particularly attractive for improving the sensitivity and selectivity of optical sensors, enabling them to effectively detect a wide range of analytes.

While PANI does have some limitations, such as low solubility and poor mechanical properties, these challenges can be mitigated through various approaches. For example, blending PANI with other polymers or forming composites can enhance its solubility and mechanical strength without compromising its electrical properties [13, 16]. This adaptability further underscores PANI's potential in sensor applications.

Conductive polymer-coated optical fibers, particularly those coated with Polyaniline (PANI), have demonstrated rapid and substantial responses to various chemical solutions. This responsiveness, coupled with the ease of fabrication and stability under varying humidity and temperature conditions, makes PANI a more suitable material for sensor applications compared to traditional materials like metal oxides. Notably, Chiam et al. [17] successfully detected alcohols using PANI-coated optical microfibers, observing a wavelength shift in the output spectrum as steric influences increased. The presence of PANI was found to increase the dihedral angle and band gap energy, further enhancing the sensor's sensitivity [17].

Recent advancements have focused on improving the mechanical properties and solubility of PANI by blending it with other polymers or doping it with various substances, which can significantly enhance its performance in sensor applications [18]. PANI-coated optical fibers have been effectively employed for ammonia detection, demonstrating high sensitivity and rapid response times [19]. Additionally, the development of flexible

PANI-coated optical fiber sensors for gas detection has opened possibilities for wearable sensor applications, highlighting the versatility and potential of PANI in advanced sensing technologies [20].

Building on these previous studies, this research explores the use of PANI-coated tapered optical microfibers for the detection of methanol, ethanol, and propanol at various concentrations. The tapered optical microfiber was fabricated using the flame brush method, which allows for precise control over the fiber's dimensions. PANI was dissolved in N-methyl-2-pyrrolidone (NMP), chosen for its strong hydrogen bond acceptance and significant solvation influence, and was then applied to the tapered fiber using the airbrush coating [21-22]. This coating method ensures that the fiber is responsive to specific chemical or biological species, enhancing its functionality as a sensor.

The primary goal of this study is to measure the presence of methanol, ethanol, and propanol at different concentrations using the PANI-coated tapered optical microfiber. This approach offers a promising solution for the development of inexpensive, effective, and easy-to-implement alcohol sensors. This study intends to enhance the field of optical fiber sensors by offering new insights into the design and implementation of functionalized optical fibers for chemical detection. It does this by utilizing the special qualities of PANI and the accuracy of the tapered microfiber fabrication technique.

2. Methodology

2.1 Preparation of Tapered Fiber

The process of removing cladding from a fiber optic cable consists of several detailed steps, each essential for maintaining the fiber's reliability. It begins with thoroughly cleaning the fiber to remove any dust, dirt, or contaminants that could interfere with the removal process. This step is essential to prevent any particles from affecting the subsequent operations.

Next, a fiber optic scoring tool is used to carefully create a shallow groove around the circumference of the cladding. This step requires precision to avoid damaging the core of the fiber, which could compromise its functionality. The groove marks the section of the cladding to be removed, which typically measures about 5 cm in length.

Once the groove is in place, the fiber is securely held with a pair of fiber optic holders. The cladding is then gently twisted and removed from the core using a stripper tool, which must be done carefully to ensure the core remains intact. After the cladding is removed, the newly exposed core is meticulously cleaned using a tissue soaked in acetone to eliminate any remaining residue that could affect the fiber's performance.

Following the cleaning, a cleaver tool is used to precisely cleave the fiber, ensuring a clean and smooth end face, which is vital for efficient splicing. After the cleaving operation, the buffer layer surrounding the fiber is removed, and the strand is terminated, preparing it for the final splicing process with the splicer.

In addition to the technical steps, it is essential to adhere to safety protocols throughout the process. This includes wearing protective attire such as gloves and boots to prevent injury when handling the fiber, as well as using the proper tools and equipment to avoid accidents or damage to the fiber. These precautions are crucial to ensuring both the safety of the operator and the success of the operation.

The fiber optic used in this experiment is a single-mode fiber (SMF). The flame brushing technique was used to trim the SMF fiber. This method enables the production of biconical tapered fiber [8, 23]. It offers a great degree of control over flame movement, fiber stretching length, and speed. The fiber may be manufactured with high precision and repeatability. Furthermore, this approach allows for the creation of low-loss microfibers.

Fig. 1 illustrates flame-brushing techniques to fabricate the tapered optical fiber. Two fiber holders fused and stretched a tiny segment of bare single-mode fiber while it was burned with an oxy-butane torch. When the target ratio was reached, which in this investigation was approximately 5 mm in length with a diameter of 4µm, the heating and pulling procedures were terminated.

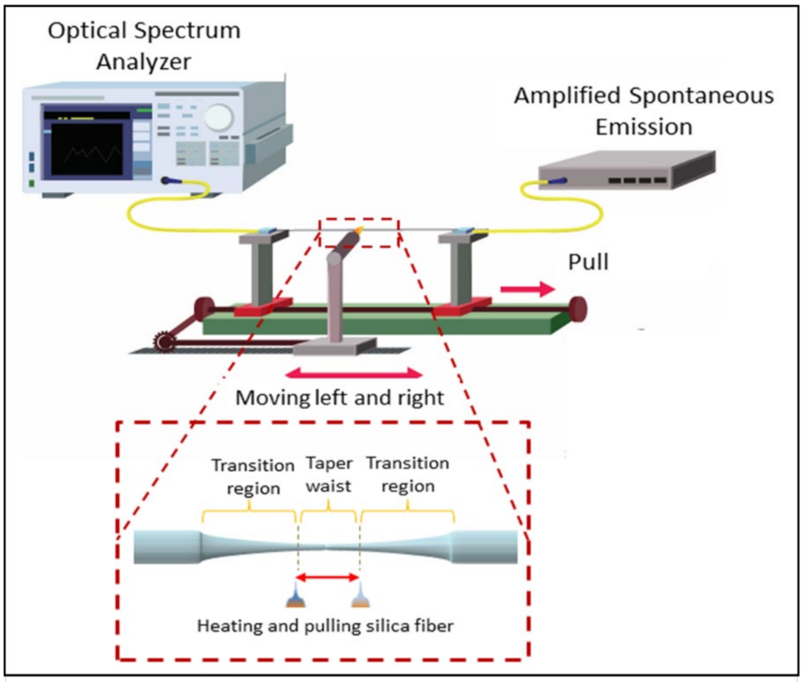


Fig. 1 *Flame brushing technique for tapering optical fiber*

Fig. 2 shows the Oxygen and Butane Control Valve to control the flame. The flame plays a significant function in the tapering process where a good flame can be achieved by controlling the convection airflow from the oxy and butane valve.

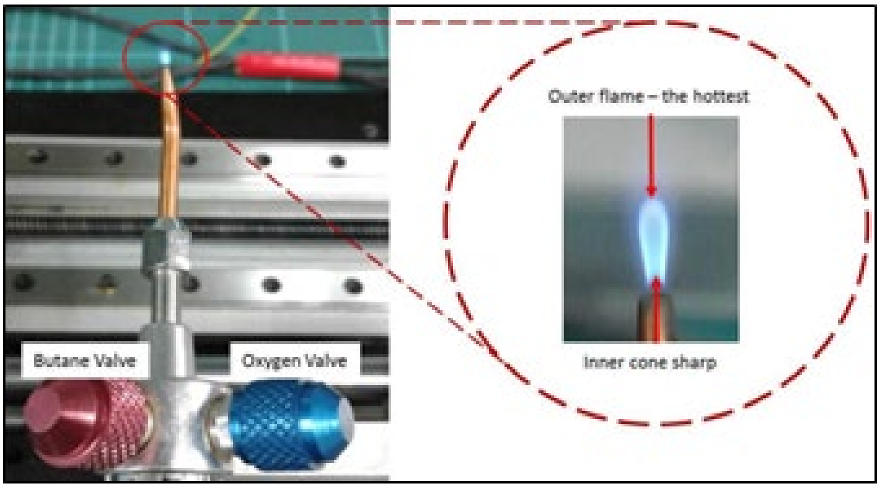


Fig. 2 *Oxygen and butane control valve*

2.2 Preparation of Polyaniline

Mechanical stirring and heating were conducted to dissolve Polyaniline (PANI) in N-methyl-2-pyrrolidone (NMP). The PANI emeraldine salt was synthesized in a chemical laboratory in the School of Chemical Engineering UiTM. The first step in preparing the PANI solution for coating the tapered fiber is to combine PANI with NMP in a beaker. Fig. 3(a) shows the 5 ml of NMP being dropped into a 0.5 g PANI powder. To prepare the PANI solution, the mixture of PANI and NMP was dropped into the beaker and placed on a hot plate with a controlled temperature at around 35°C as visualized in Fig. 3(b). As shown in Fig. 3(c), the mechanical stirring is performed with the magnetic stirring bar in the beaker. Stirring was conducted for approximately 60 minutes at a speed of 500 rpm until the Polyaniline was entirely dissolved in the NMP. Once the Polyaniline was dissolved and then cooled at room temperature for another 60 minutes, the solution was stored in an anti-UV container to maintain the bonding.

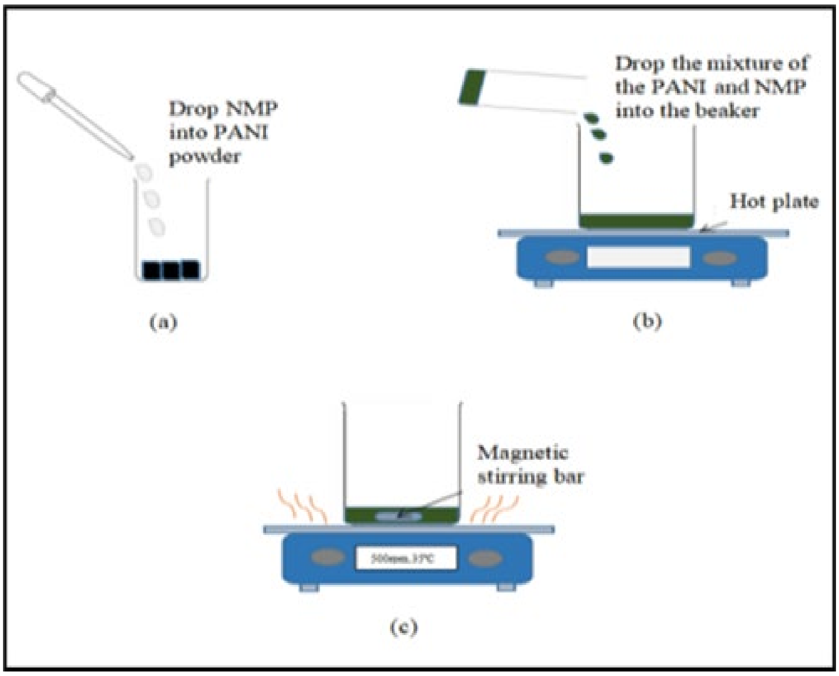


Fig. 3 *Schematic illustration for the preparation of PANI solution (a) NMP solution dropped into the PANI powder; (b) The mixture is dropped into the beaker placed on a hot plate; (c) The solution was stirred using a magnetic stirring bar*

2.3 Coating of the Tapered Fiber

After successfully preparing the solution of PANI, the tapered fiber was then coated with the PANI solution using an airbrushing technique. This technique required great handling of the pressure and hand movement to coat the tapered fiber optics finely. The airbrushing process is normally carried out in a controlled environment, with temperature, solution viscosity, and immersion duration carefully managed to produce the required coating thickness and uniformity. Typically, the coated fiber is cured or dried to solidify the coating substance and increase its adherence to the fiber. The process is extensively used to create optical fiber coatings that protect against environmental threats and mechanical damage. In this research, the coated fiber was left dried out at the room temperature of 25˚C for 24 hours. On a side note, coatings can also be utilized to improve the fiber's optical qualities, such as numerical aperture or reflection loss [24]. It is vital to note that the airbrushing process may be rather sensitive and necessitates the use of specialist equipment and skills to obtain high-quality results.

2.4 Preparation of Alcohol in Aqueous Solution

In this research, the primary focus is on the preparation of aqueous aliphatic alcohols, specifically methanol, ethanol, and propanol. The process of reducing the concentration of these alcohols by mixing them with water or another solvent is known as dilution. By adding a solvent, such as water, as used in this study, the concentration of methanol, ethanol, and propanol is decreased. The dilution equation can be employed to calculate the amount of solvent added and the resulting concentration of the alcohols. The dilution equation may be used to calculate the amount of solvent supplied and the resulting concentration:

$$C_1V_1 = C_2 V_2 \tag{1}$$

where C_1 and V_1 are the initial alcohol concentration and volume, and C_2 and V_2 are the final alcohol concentration and volume following dilution. Finally, methanol, ethanol and propanol were diluted in 5 stages of concentration which are 2%, 4%, 6%, 8% and 10%.

2.5 Experiment Set Up

Fig. 4 shows the experimental setup of the proposed aliphatic alcohol sensor. The amplified spontaneous emission (ASE) with a wavelength of 1550 nm provides light to the detector while the output light was captured and recorded using an Optical Spectrum Analyzer (OSA, Yokogawa AQ6370B). Firstly, the bare tapered fiber on a glass slide was tested to sense all three aliphatic alcohols in 5 different concentrations. The test was conducted and repeated for five times, and data was collected for each type of alcohol and concentration ranging from 2% to 10% individually to get more accurate data.

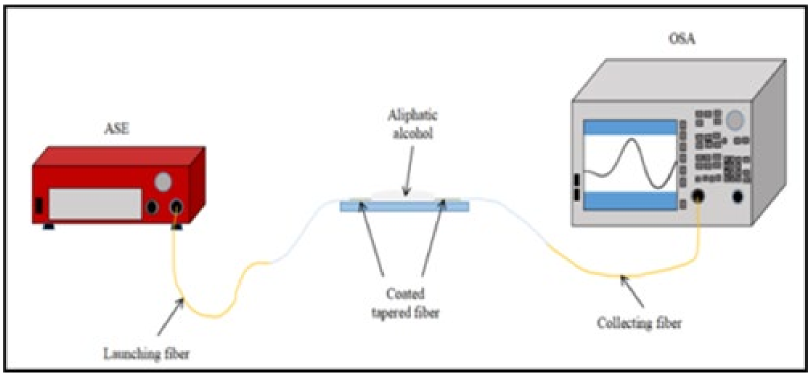


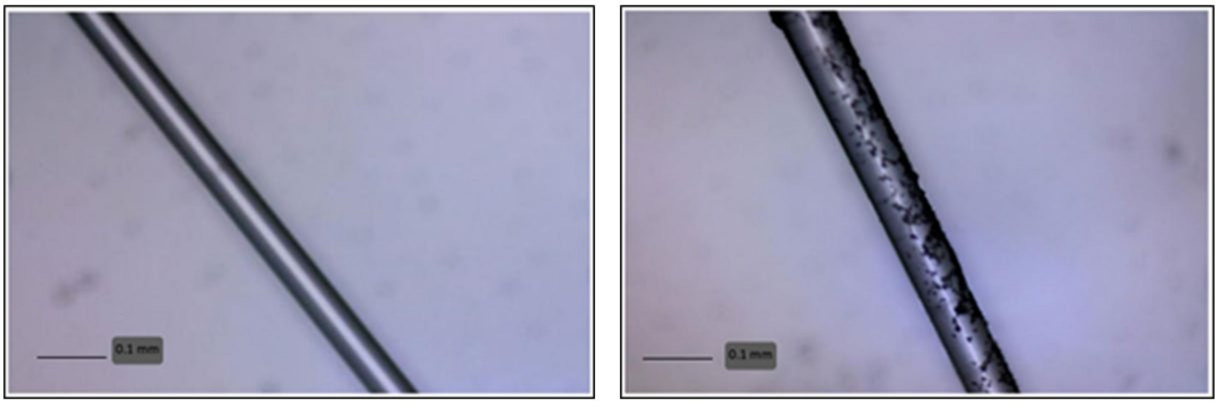
Fig. 4 *Experimental setup of the tapered fiber coated with PANI for methanol, ethanol and propanol concentration measurements*

Once done with the alcohol sensing on the bare tapered optical fiber, the methanol, ethanol and propanol sensitivity measurements were carried out by dropping 2 mol alcohol like in the previous test onto the tapered optical microfiber coated with PANI. The glass slide was used to place the tapered microfiber and placed between the ASE laser source and the OSA. The OSA was used to display the output spectrum and the output power. The process was repeated, but this time the fiber used is the tapered microfiber coated with PANI. Both results of bare and coated tapered microfiber were recorded in the external source to be compared and analysed.

3. Results

3.1 Surface Morphology

The surface morphology of the bare tapered optical fiber and the tapered optical fiber coated with PANI was examined using a Digital Microscope (DM). Fig. 5 shows the DM image of both conditions of the tapered optical fiber with a waist diameter of 4 µm. As seen in Fig. 5(a), the image shown is the surface of the bare tapered optical fiber. While in Fig. 5(b) shows the image of the coated tapered optical fiber where the is a presence of PANI particles identified on the surface.



(a) **(b)**

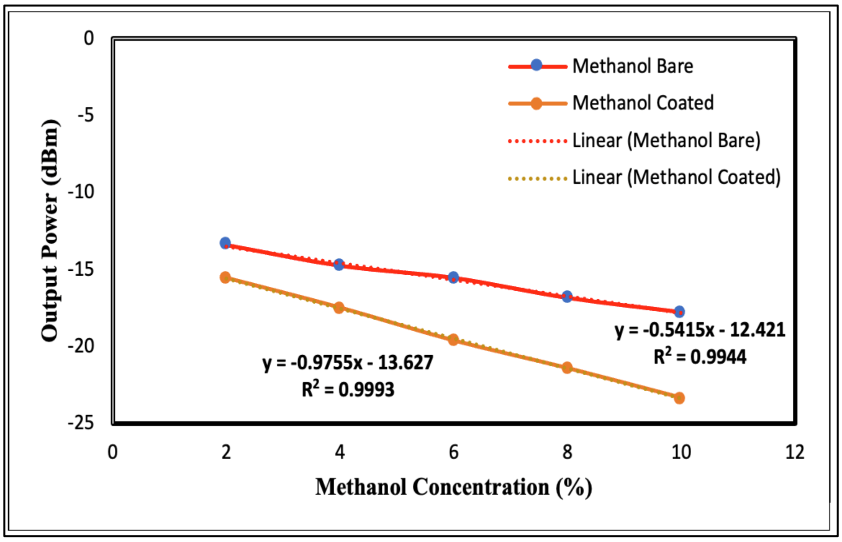
Fig. 5 *Digital Microscope (DM) image of the tapered fiber (a) DM image of the bare tapered optical fiber; (b) DM image of the surface for the PANI-coated tapered optical fiber. (Note: 0.1 mm indicates the measurement scale)*

Fig 6(a), (b) and (c) illustrate the linear function of the transmission power output signal against the aliphatic alcohol concentrations, respectively, for the bare tapered fiber and tapered fiber coated with PANI. In the experiment, the diameters of the microfibers were maintained at 4µm for both the bare and coated tapered fibers. Based on Fig. 6(a), the tested alcohol was methanol, where the sensitivity and linearity of the bare tapered fiber were obtained at 0.5415 dBm/% and 99.44% while the sensitivity and linearity of the PANI-coated tapered fiber achieved 0.9755 dBm/% and 99.93% respectively. It is observed that the output power reduces as the concentration is increased from 2% to 10%.

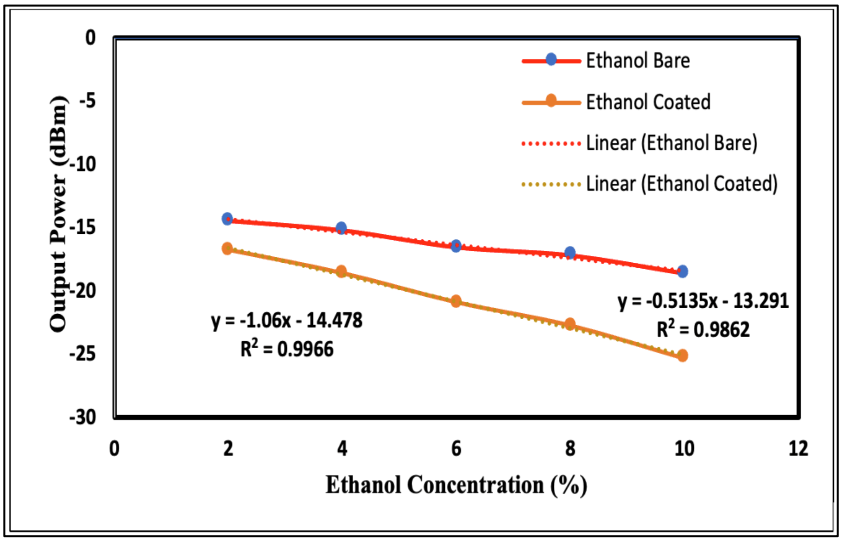
A similar pattern was observed when the various concentrations of ethanol were tested on the bare and tapered fiber coated with PANI. Fig. 6(b) shows that as the ethanol concentration increases, the output power from the OSA decreases.

The sensitivity and linearity of the bare tapered fiber were obtained at 0.5135 dBm/% and 98.62% while the sensitivity and linearity of PANI-coated tapered fiber were obtained at 1.06 dBm/% and 99.66% respectively. Based on Fig. 6(c), where the propanol was observed, the sensitivity and linearity of the bare tapered fiber were obtained at 0.727 dBm/% and 99.00. On the other hand, the performance of PANI-coated tapered fiber increased when it produced 1.039 dBm/% sensitivity with 99.25% linearity. The changes in the cladding of the tapered fiber have affected the dispersion of light when sensing the region exposed to alcohol substances [25-26].

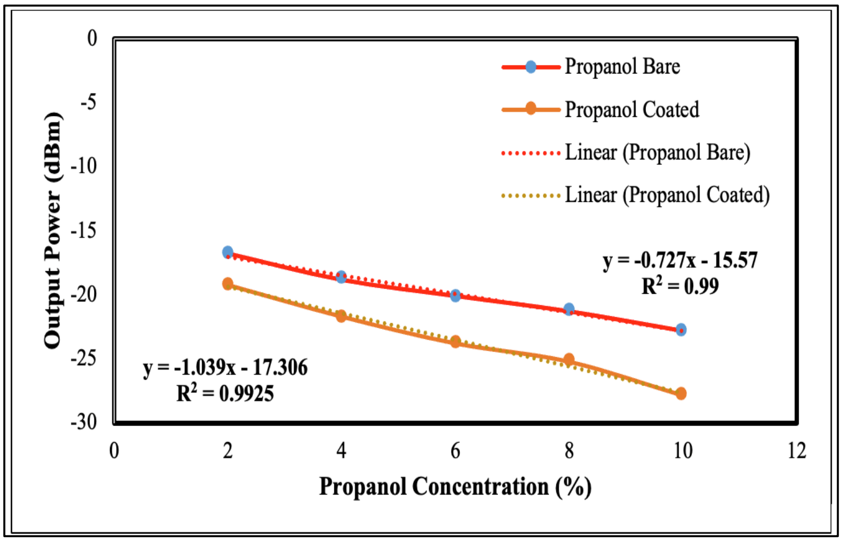
The sensor's sensitivity is increased after being coated with PANI. Joulazadeh & Navarchian [27] previously published a PANI-based sensor for alcohol vapor. The sensitivity of the constructed sensor increases due to H-bonding interactions between the -OH groups of alcohol and the nitrogen atoms in the PANI structure. This is understood as the chemisorption process, in which the chemical linkages formed resulted in variations in light intensity when exposed to different quantities of alcohol molecules in the sample material.



(a)



(b)



(c)

Fig. 6 *Linear function of the output signal against various concentrations for the bare and PANI-coated tapered fiber of (a) Methanol; (b) Ethanol; and (c) Propanol sensing*

Table 1 shows the summaries of the performance characteristics for methanol, ethanol, and propanol sensors utilizing the PANI as a coating substance. The sensitivity of the sensor is obtained from the slope of the output response graph and the standard deviation values were averaged over the five distinct runs [28]. To calculate the sensor's resolution, the average standard deviation was divided by the sensor's sensitivity.

Overall, the sensor is observed to be more sensitive for the coated tapered fiber compared with bare tapered optical fiber for methanol, ethanol and propanol as shown in Table 1.

Table 1 *The performance of the bare tapered optic fiber and tapered optic fiber coated with PANI for aliphatic sensor*

Parameter	Methanol		Ethanol		Propanol	
	Bare	PANI-coated	Bare	PANI-coated	Bare	PANI-coated
Linear Range (%)	2-10	2-10	2-10	2-10	2-10	2-10
Standard Deviation (dBm/%)	0.0551	0.1935	0.479	0.3355	0.1947	0.4136
Linearity (%)	0.9944	0.9993	0.9862	0.9966	0.99	0.9925
Sensitivity (dBm/%)	0.5415	0.9755	0.5135	1.06	0.727	1.039
Resolution (%)	0.1018	0.2002	0.9328	0.3165	0.2678	0.3981

As for methanol, the sensitivity increased by 80.17% when the tapered optical fiber was coated with PANI. The same case goes for ethanol and propanol where the sensitivity increases by 106.43% and 42.92% respectively. The linearity for methanol, ethanol and propanol also increased from 0.9944% to 0.9993%, 0.9862% to 0.9966% and 0.99% to 0.9925% respectively, when the alcohol tested on the tapered optical fiber coated with PANI On the side note, the sensor's resolution can be increased by lowering the standard deviation number or raising the sensor's sensitivity [29].

An optical microfiber's tiny diameter and strong index contrast between the fiber core and its surroundings allow it to display remarkable wave-guiding features such as tight optical confinement, huge evanescent fields, and waveguide dispersion [9-10].

Conclusion

An alcohol sensor is proposed and demonstrated using tapered optic fiber coated with Polyaniline (PANI) for the measurement of different concentration of methanol, ethanol and propanol. The tapered optic fiber is fabricated using the flame brushing technique and has a waist of 4 µm and a length of 5 mm. As the solution concentration of the methanol, ethanol and propanol solution increasing from 2% to 10%, the linearity and sensitivity of the sensor is increase. Furthermore, it has been shown that a higher sensitivity can be achieved by coating the tapered fiber with PANI and an improvement of 80.17% in terms of its sensitivity for the methanol sensor. As for the ethanol and propanol, the sensitivity shows an improvement of 106.43% and 42.92% respectively. To the best of our knowledge, this research has proven to enhance the sensitivity as the tapered fiber coated with PANI tested on methanol, ethanol and propanol. In comparison to uncoated fibre, PANI-coated fibre is preferred for alcohol sensing. To conclude, PANI-coated fiber has improved tapered optical fiber performance for alcohol sensing. Moreover, fiber coated with PANI performs better for methanol sensing than other types of alcohol.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception, methodology, and design:*** *Noor Aliah Kahlet, Naimah Mat Isa;* ***data collection:*** *Noor Aliah Kahlet, Naimah Mat Isa, Sakinah Mohd Alauddin, Norhafizah Burham;* ***analysis and interpretation of results:*** *Noor Aliah Kahlet, Naimah Mat Isa, Sakinah Mohd Alauddin, Norhafizah Burham;* ***draft manuscript preparation:*** *Noor Aliah Kahlet, Naimah Mat Isa;* ***Manuscript review, intellectual content and supervision:*** *Naimah Mat Isa, Hasnida Saad,* ***reviewed the results and approved the final version of the manuscript.***

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