

## A novel examination of limonene detection using plastic fiber optic sensors and the tapered approach

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### ABSTRACT

A novel tapered plastic optic fiber (POF) biosensor is proposed and demonstrated for monitoring limonene in different concentrations. The mechanism of this device is based on an increase in the light transmission of a sensor submerged in a higher-concentration limonene solution, which also reflects an increase in the refractive index of the sensor. The tapered POF was fabricated using the chemical etching method to accomplish different waist diameters of 0.6 mm, 0.55 mm, and 0.5 mm, with a fiber length of 10 cm and a 2 cm sensing region. An Arduino integrated development environment (IDE) program was used to drive the voltage values from the photodetectors to obtain the measurements. As the limonene concentration solution varied from 20% to 100%, the output voltage of the sensor increased linearly, showing a sensitivity of 0.295 V/%, 0.33 V/%, and 0.46 V/% for tapered waist diameters of 0.6 mm, 0.55 mm, and 0.5 mm, respectively. The proposed sensor is a low-cost solution measurement option with high sensitivity, while it also involves a simple and easy fabrication technique.

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## 1. INTRODUCTION

Limonene is an essential oil obtained from citrus fruits such as lemons, oranges, and grapefruit and it is one of the most common natural terpenes. Limonene acquires an attractive lemon-like odor and can be made into a colorless liquid [1]. Moreover, limonene exists as two optical isomers, labelled as d-limonene and L-limonene, as well as racemic combination [2]. Limonene is generally used as a fragrance and flavor enhancer in food items such as juice, candy, chewing gum, and ice cream. Limonene is also frequently used in cosmetic formulations and can be found in soaps, perfumes, shampoos, and shower gels [3]. Limonene causes chronic liver disease due to excessive alcohol consumption and metabolism disorders. The early detection of liver diseases or cirrhosis is treatable with the existing technologies. However, the volatile organic compound (VOC) is a general technique used with human breath, which has been employed to determine limonene levels in the human body and also to detect liver disease [4]–[6]. Therefore, non-invasive, high-sensitivity, compact, and low-cost fiber optic sensor approaches are preferred as an alternative.

To determine the limonene in a solution, optical methods typically employ a refractometer to measure the optical refractive index.

Plastic optical fibers (POF) have benefits when used as intensity-based optical fiber sensors with a large 1 mm core diameter and a high numerical aperture of 0.47. POF sensors have been used to develop and demonstrate a variety of current biosensor applications which are based on utilizing these multimode fibers for intensity modulation [7]–[11]. Polymethylmethacrylate (PMMA), polycarbonate (PC), polystyrene (PS) [12], and, more recently, cyclic transparent optical polymer (CYTOP), have all been used to manufacture the fiber's core. CYTOP has the lowest attenuation—50 dB/km at 650 nm—compared to PMMA-based POF, the value of which is 160 dB/km. Fluorinated polymers are typically used to create the cladding layer of the fibers. Despite their wider availability and lower price, step-index PMMA-based POF sensors currently make up the majority of sensors [13], [14]. To increase the bandwidth and decrease the bending sensitivity, other POF variations have also been introduced, such as multicore fibers, double-step-index fibers, multi-step-index fibers, and graded-index fibers [12], [15], [16]. The ability to modify the hole shape to optimize the sensitivities of various loss characteristics for a variety of measurands and the use of asymmetric microstructures for directional bend sensitivity are the advantages of POFs [17]–[20].

Fiber optic sensors have recently been the subject of much attention, compared to electrical transducer sensors. This is because fiber optic sensors have a natural immunity to electromagnetic interference, as well as being low-cost, small, highly sensitive, low-noise, and highly durable [21]–[25]. In the main, to determine limonene in a solution, the working procedure of the optical sensor is based on two measurements, which are optical refractive index measuring and light intensity. Furthermore, an inherent attribute of the solution is the optical refractive index. Many researchers have undertaken studies based on using optical refractive index measurements to detect different liquids. For example, Rahman *et al.* [26] reported using a tapered multimode POF sensor to measure sodium chloride (NaCl) in de-ionized water with different refractive indexes and varying concentrations. Teng *et al.* [27] demonstrated a twisted tapered POF sensor which was used to detect different refractive indexes of glycerine solution. Banerjee *et al.* [28] experimented with a low-cost POF sensor system for sucrose solution detection using the changes in the refractive index. However, light intensity measurement techniques have also been used by researchers with optical fiber sensors [29]–[31].

In this work, a tapered POF sensor developed for limonene detection is proposed. This limonene detection approach was based on using various concentrations of limonene oil in hexane, which differed in terms of the refractive index of the solutions. The suggested sensor monitored the detector's output voltage, which was affected by the interaction between the evanescent wave generated in the tapering cladding and the solution that surrounded it. This method was low-cost and reliable, and it demonstrated continuous measurement capabilities.

## 2. EXPERIMENTAL METHOD

This paper introduced a sensor system involving a POF with a core diameter size of 1 mm and a numerical aperture of 0.5. It was used for designing a tapered sensor region, which functions as a light-transmitting element and was purchased from Mitsubishi Chemical Corporation. The refractive index values of the core and cladding were 1.49 and 1.41, respectively. Acetone, ethanol, limonene, and hexane were purchased from Polyscientific, Malaysia. A red light emitting diode (LED) and an industrial fiber optics (IF-D91) photodetector (PD) were purchased from Digikey Inc. Table 1 shows the chemical properties of the limonene solution. The tapered sensor was made by first stripping off 2 cm from the 10 cm long POF jacket. Then, the cladding of the POF was removed using small amounts of acetone and cotton. Excessive acetone may damage or break the POF core. The POF cladding reacted to the acetone and formed milky white foam, while the remaining cladding was removed using sandpaper. This process was repeated until the tapered region was formed. Lastly, the tapered region was neutralized with de-ionized water and ethanol.

Table 1. The chemical properties of limonene

Properties	Quantity
Molecular weight	136.23 g
Density at 20.85 °C	0.8402 g/ml
Boiling point	176.0 °C
Solubility at 25 °C in water	7.57 mg/L
Vapour pressure at 25 °C	1.55 mmHg

The electronic circuit, also known as the receiver circuit, was used to control the LED and PD. The circuit consisted of the PD IF-D91 and a dual rail-to-rail operational amplifier LM358P, which was used to

amplify and convert the optical signal to an electrical signal. The dual rail-to-rail operational amplifier (op-amp) LM358P was chosen for the receiver circuit design due to the high input impedance and the wide range of supply voltage provided by the Arduino Uno microcontroller. The inverting input of the LM358P was attached to the photodiode and the non-inverting input was attached to the ground. The output from the receiver was in voltage values. A 9 V rechargeable was used as the power input for the circuit. An Arduino was used to drive the circuit at 5 V. The LED was powered by a direct current power supply based on bipolar junction transistor (BJT) semiconductors. With the use of resistors in series with the PDs, voltage signals corresponding to the photocurrents across the two PDs were acquired. The voltage signals were fed to the Arduino, where the signals from the PDs were displayed using a liquid crystal display (LCD). An Arduino integrated development environment (IDE) program was used to drive the voltage values from the PDs to obtain the measurements. Figure 1 shows the schematic diagram for the receiver circuit designed for this sensor system.

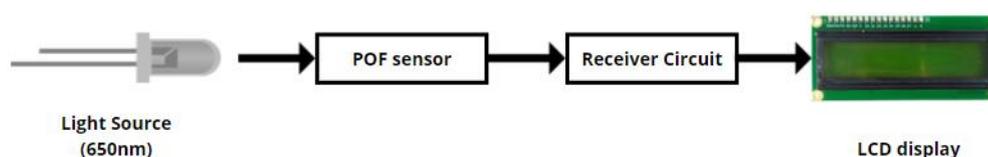


Figure 1. The schematic diagram of the receiver circuit

The experimental setup shown in Figure 2 was used to carry out the limonene concentration detection with the POF sensing probe. This mainly consisted of a petri dish, the optoelectronic circuit, the PD, the LED, and a limonene liquid dropper. A 3D printer plastic block was customized to hold the LED and PD for testing the sensing probes. The block was sealed after the LED and PDs had been inserted to prevent cross-coupling between the LED and the PDs, as well as from the ambience. The light source operated at a wavelength of 650 nm (red region) and an average power dissipation of 105 mW. The fiber probe was aligned in a straight line to minimize any bending losses that may have occurred in the POF and increase the accuracy of the measurement. It was handled carefully to avoid breakages when testing the limonene and hexane solutions. To assess the concentration-sensing performance of the fabricated sensors, the sensing part was dropped onto the limonene, which had been diluted with hexane. Different limonene oil/hexane volume concentrations (ml/ml) were used: 20/80, 40/60, 60/40, 80/20, and 100/0, which had refractive index values of 1.3976, 1.4134, 1.4363, 1.4496, and 1.4711, respectively. To eliminate any influence of temperature on our measurement, the solution container was tightly covered to prevent evaporation and placed in a temperature-controlled area with an ambient temperature of 25 °C.

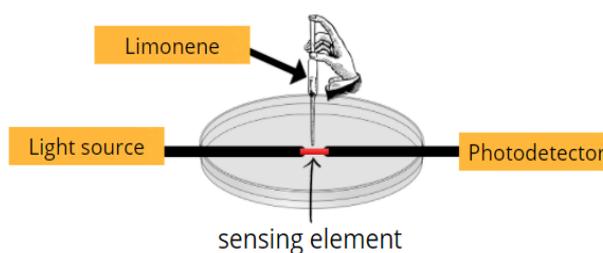


Figure 2. Experimental setup for the sensing region

### 3. RESULTS AND DISCUSSION

Figure 3 shows a microscope image of the tapered POF samples with four different diameters, which are 3(a) 0.99 mm, 3(b) 0.6 mm, 3(c) 0.55 mm and (d) 0.5 mm. Figure 4 shows the performance of the tapered POF against the concentrations of limonene solution. As shown in Figure 4, the output voltage from the photodetector, which was directly proportional to the light transmission, linearly increased as the limonene concentration increased for three different tapered POF diameters. The sensitivity was obtained at 0.295 V/%, 0.33 V/%, and 0.46 V/% and the slope shows good linearity of more than 94%, 97%, and 97% for the 0.6 mm, 0.55 mm, and 0.5 mm tapered POFs, respectively. The refractive index of the external medium acted as passive cladding and was able to affect the amount of power lost when the signal passed

through the tapered region since the diameter of the cladding in the tapered region had decreased. The refractive index values of the plastic multimode fiber core and cladding (1.492 and 1.406, respectively) used in this experiment and compared to the silica multimode fiber index were relatively larger than the POF refractive index. When immersing the tapered fiber sensing region into the limonene solutions with different concentrations from 20% to 100%, the index differences increased since the refractive index (RI) of the limonene solutions was higher than that of water RI (1.3333). As the limonene solution concentration increased, the solution RI also increased. The output voltage value increased with the differences in RI, reducing the power leakage from the tapered region to the surroundings.

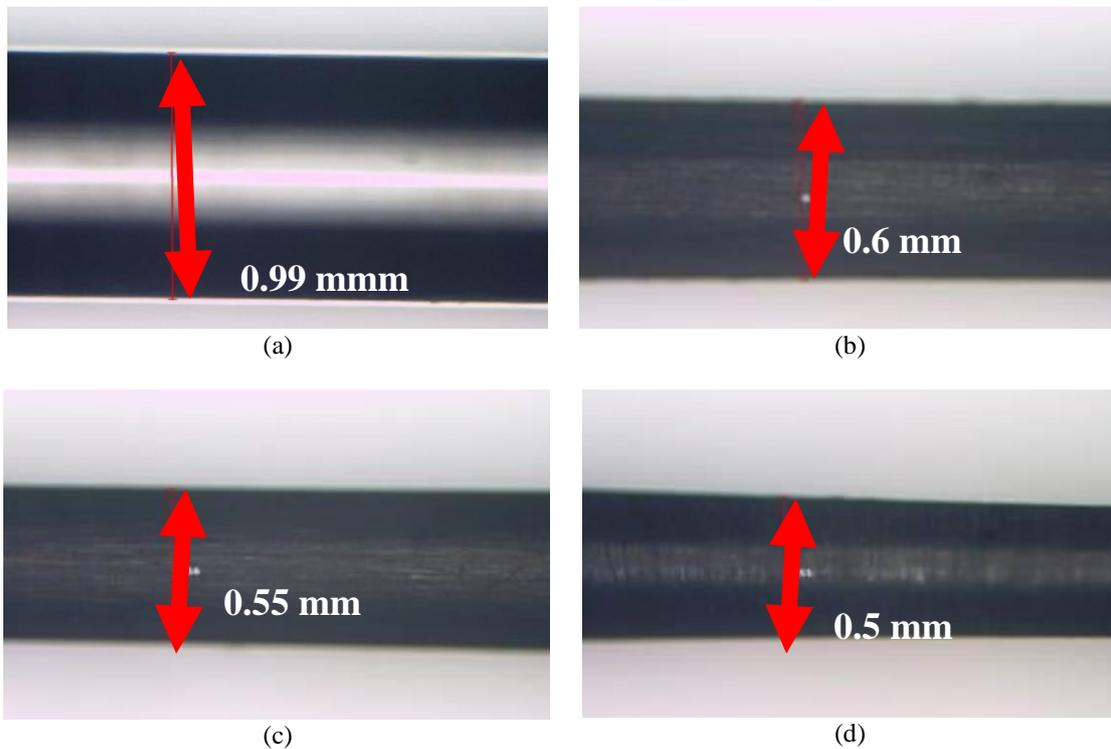


Figure 3. Microscope pictures of the different sensing diameters of the tapered plastic optical fiber (POF) (a) untapered POF, (b) 0.6 mm tapered POF, (c) 0.55 mm tapered POF, and (d) 0.5 mm tapered POF

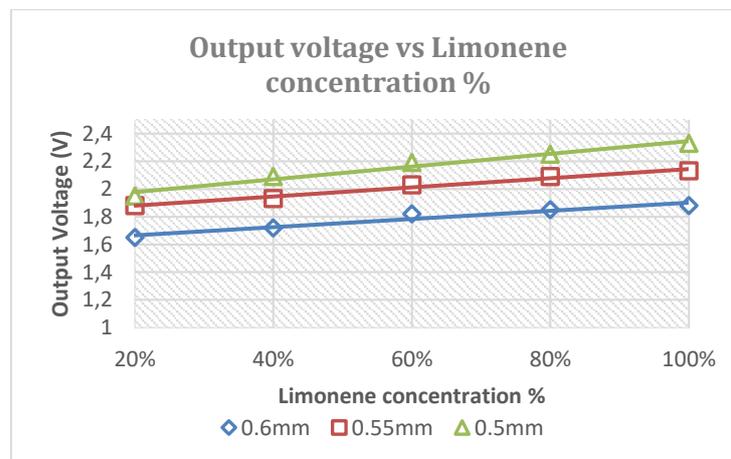


Figure 4. Relationship between the output voltage and limonene concentration percentage

Figure 5 shows the differences in output voltage along with the refractive index of the limonene solution. As the concentration of limonene increased gradually from 20% to 100%, the RI of the limonene

solution increased from 1.3976 to 1.4711. The value of RI was obtained using a refractometer, whereby different concentrations of limonene solution were placed on the surface of the prism to display the digital readout and the output voltage changes were recorded from the designed amplifier optoelectronic circuit. The sensor characteristic curve was established based on the output voltage of these liquids, and refractive indices of the 20%-100% limonene concentrations were produced. As shown in Figure 5, the output voltage for the 0.6 mm tapered POF increased from 1.65 V to 1.88 V; for the 0.55 mm tapered POF, it increased from 1.88 V to 2.13 V; and for the 0.5 mm, the voltage recorded rose from 1.95 V to 2.33 as the RI values of the limonene concentration increased from 1.3976 to 1.4711. Meanwhile, the slope shows good linearity of more than 94%, 97%, and 97% for the 0.6 mm, 0.55 mm, and 0.5 mm tapered POFs, respectively. The highest sensitivity was obtained with the 0.5 mm tapered POF, which was 0.46 V/%, and good linearity of more than 97% was recorded. The lowest sensitivity was obtained with the 0.6 mm tapered POF, which was 0.295 V/%, but good linearity was observed (more than 94%). It was concluded that as the tapered region diameter decreased, the sensitivity of the limonene detection increased.

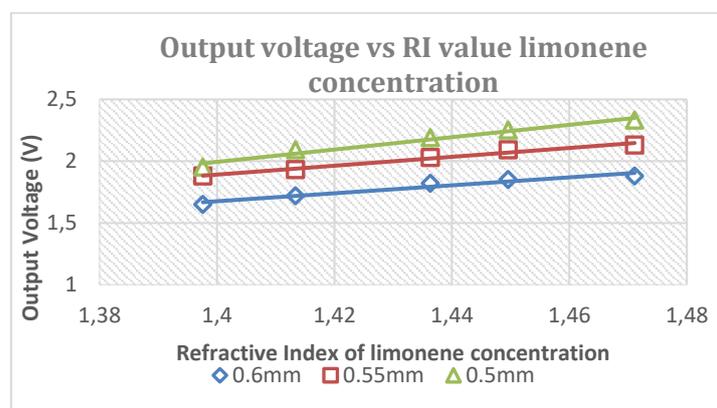


Figure 5. Relationship between the output voltage and refractive index of different limonene concentrations

Table 2 shows a summary of the sensor performance for the three different tapered POF diameters, along with the sensitivity, linear range, linearity, standard deviation, and limit of detection. During the whole experiment, the same amount of limonene solution was dropped on the tapered region of the POF, and the corresponding output voltage was measured at the LCD of the amplifier optoelectronic circuit. Due to the sensor's capacity to offer real-time limonene detection and continuous control of diverse mixes, these results suggest that the proposed sensor is relevant and beneficial for limonene detection, particularly in biomedical applications. Table 3 shows the performance analysis of different liquid sensors compared with their limonene liquid sensor performance. Table 3 shows a comparison of this novel approach to limonene solution detection with different types of solutions, such as glycerine, milk fat, and sucrose, which used the same tapered POF method.

Table 2. The performance of the limonene sensor with different tapered waists

Parameters/tapered variation	0.6 mm	0.55 mm	0.5 mm
Sensitivity	0.295 V/%	0.33 V/%	0.46 V/%
Linear range (RI)	1.3976-1.4711	1.3976-1.4711	1.3976-1.4711
Linearity	>94%	>97%	>97%
Standard deviation	0.0621 V	0.0411 V	0.0641 V
Limit of detection	0.6958%	0.4107%	0.4597%

Table 3. The performance analysis of different liquid sensors compared to the sensor used in this work

Sensor structure	Solution type	Sensitivity	RI detection range	Ref
Twisted tapered POF	Glycerine-water	1700%/RIU and 3496%/RIU	1.37-1.41 and 1.41-1.44	[27]
Twisted tapered POF	Glycerine-water	0.65%/mm	1.36	[32]
Double side polish U-shape POF	Glycerinum solution	1541%/RIU	1.33-1.39	[33]
U-bent POF	Milk fat content	0.15A/%	1.341-1.359	[34]
Stripped POF	Sucrose	-	1.33-1.56	[28]
U-bent POF	Sulfate-reducing bacteria	103 MPN/mL	1.334- 1.394	[35]
Tapered POF	Limonene	0.46 V/%	1.3976-1.4711	This work

#### 4. CONCLUSION

In this study, we developed and tested a simple, small, and highly sensitive fiber sensor, based on the tapered technique. Three different tapered diameters (0.60 mm, 0.55 mm, and 0.50 mm) were fabricated to test the sensor device's response to five different concentrations of limonene solutions with a refractive index range of 1.3976 to 1.4711. The smallest tapered optic fiber diameter produced higher sensitivity than the larger tapered diameters. It was also observed from the sensor's performance that the detection with the 0.50 mm tapered fiber produced 0.46 V/% sensitivity, compared to the 0.6 mm tapered diameter fiber, which obtained lower sensitivity of 0.29 V/%. The proposed sensor is a low-cost option for solution measurement, as well as a highly sensitive, simple, and easily fabricated technique.

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