

Zinc oxide-coated fiber-optic sensors for monitoring of edible oil adulteration with internet of things integration

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Article Info

Article history:

Received Sep 29, 2023

Revised May 7, 2024

Accepted May 17, 2024

Keywords:

Biosensor

Fiber optic sensor

Internet of things

Plastic optical fiber

Tapered plastic optical fiber

ABSTRACT

The study proposes a novel approach for detecting adulteration in edible oils utilising a zinc oxide (ZnO)-coated optical sensor. The procedure included the development of a sensor probe using a plastic optical fiber (POF) with a ZnO nanolayer deposition. The ZnO nanorods were applied to the surface of the POF via a hydrothermal process. The sensitivity and accuracy of uncoated and ZnO-coated POFs were compared, and it was discovered that the ZnO-coated POF was more sensitive to changes in the refractive index of the samples under testing. The study ascertained a correlation between the optical power and voltage of the sensor and the refractive index of the medium. As the adulterant concentration in the oil mixture increased, the refractive index of the medium altered. As a result, both the sensor's output voltage and optical power decreased. Upon completion, it was discovered that the uncoated POF had a sensitivity of 0.073 V/%, whereas the ZnO-coated POF had a sensitivity of 0.085 V/%. These findings highlight the effectiveness of ZnO-coated optical sensors, as well as their potential integration into internet of things (IoT) platforms for monitoring adulteration in edible oils.

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1. INTRODUCTION

The study encompassed the development of optical fiber sensors based on plastic optical fiber (POF), a well-known technology renowned for its broad range of applications [1]–[3]. POF has been used in numerous fields, including telecommunications networking, medicine, safety, and the automotive industries [4]–[6]. Due to their ease of handling, adaptability, and affordability, POFs have also replaced glass fibers in short-distance communication links [7]. These advantages light-emitting diode (LED) to POF's widespread use in sensor applications [8], [9], including food adulteration detection. The detection of contaminated oil, particularly edible oil, is one of the main areas of focus.

Improving the functional efficiency of a POF sensor can be accomplished by modifying the fiber's geometric properties, either in terms of length or diameter [10], [11]. One effective approach, for example, is to reduce the fiber's waist diameter, allowing for greater transmission of the evanescent field along the fiber length. Furthermore, the sensor's performance can be improved by incorporating nanostructures onto the sensing probe. Because of its high sensitivity, zinc oxide (ZnO) is a popular nanostructure in a variety of

sensing applications. ZnO has a direct wide bandgap of 3.37 eV, thus making it ideal for applications involving short-wavelength light-emitting devices. Furthermore, as a sensing material, ZnO has numerous advantages, including exceptional light transmittance, excellent electrical conductivity properties, non-toxicity, and cost-effectiveness [12], [13].

Edible oils are an essential component of daily life, with extensive application in gastronomic uses and the production of various food products. The purity of edible oils has long been a source of concern, despite their importance in terms of nutrition. Due to its high demand, the issue of adulterants has grown to be a major concern [14], [15]. It is undeniable that adulteration of edible oils has a negative impact on people's health. Paraffin oil is one of the main contaminants found in edible oil. The fine texture makes it challenging for the consumer to determine the level of adulteration [16]. Coconut oil, for example, could be combined with paraffin oil without leading to any discernible changes in flavour, consistency, or visual characteristics [17].

Recent research has concentrated on improving the capabilities of POF-based optical sensors for monitoring the various aspects of edible oil quality. While previous studies investigated the detection of adulterants by analysing changes in refractive indices and other chemical variations, they rarely explicitly addressed the influence of ZnO coating on POF in improving the capabilities of optical sensors. Furthermore, the integration of POF sensors with internet of things (IoT) technology is relatively new and underexplored. It is known that the integration of POF sensors with IoT technology has enabled real-time monitoring, remote data acquisition, and timely corrective actions. The integration has opened up new opportunities for data-driven decision-making and better connectivity. It allows for the rapid transmission of data from sensors to cloud-based analytics systems, allowing for faster responses to changing conditions and overall system efficiency [18]–[21]. As IoT applications continue to expand across industries [22], [23], understanding the contributions and possibilities of POF technology within these platforms becomes increasingly relevant. Therefore, in this project, we propose a fiber-optic sensor for estimating the level of adulterant in coconut oil as a result of contamination with paraffin oil, with integration to an IoT platform.

2. METHOD

The method includes sensor probe fabrication, oil sample preparation, ZnO coating by hydrothermal process, and experimental setup for sensor characterization, all of which will be explained in this section.

2.1. Sensor probe fabrication

To prepare the POF probe as the sensing region, the POF jackets were stripped using a knife to expose the fiber. A 1.5 cm length of the POF was uncladded in the middle of the POF to serve as a sensing probe. The core was then exposed by rubbing acetone to the unclad area. The optical microscope is used to confirm that the POF's cladding has been fully removed. The unclad POF was subsequently covered with a ZnO nanolayer, which acted as the main buffer, as the final component.

2.2. Oil sample preparation

To investigate the effects of adding adulterants to coconut oil, paraffin oil as adulterants were gradually added to coconut oils, as shown in Table 1. To prepare the solution, the beakers were sterilised with ethanol. The magnetic capsule was placed in the beaker and stirred with a magnetic stirrer. The stirring process was carried out for three seconds on all samples. The initial refractive indices of all samples had been taken using the refractometer before the fiber probe was coated with ZnO nanoparticles.

Table 1. Oil sample volume ratio

Sample	Coconut volume (ml)	Paraffin oil volume (ml)	Volume (%) of adulterant
1	50	0	0
2	40	10	20
3	30	20	40
4	20	30	60
5	10	40	80

2.3. Hydrothermal process

The ZnO coating on the POF was deposited using a hydrothermal process. ZnO nanorod development on POF depends on the seeding procedure. The seeding process consists of four steps: preparing the seeding solution, treating the surface of the POF core, producing nucleation sites on the POF, and annealing. To produce the seeding solution, two separate solutions—a pH-controlled solution and a ZnO nanoparticle solution—had to be prepared. For the ZnO nanoparticle solution, 20 ml of ethanol (Merck KGaA,

Germany) were slowly dissolved in approximately 0.0044g of zinc acetate dihydrate $[\text{Zn}(\text{O}_2\text{CCH}_3)_2(\text{H}_2\text{O})_2]$ and stirred for 30 minutes at a temperature of 50 °C to create a 1 mM solution. After the solution had cooled to room temperature, another 20 ml of ethanol was added to treat the ZnO solution, which eventually contained 40 ml mixture. A pH-controlled solution was prepared by dissolving sodium hydroxide (NaOH) in 20 ml of ethanol at a temperature of 50 °C while slowly stirring the mixture. A 1 ml pipet was used to mix the ZnO nanoparticle solution with 20 ml of pH control solution after 10 minutes. For each drop of the 1 ml pH control solution, the ZnO nanoparticle solution was gently swirled for 1 minute. The process was repeated 20 times. The seeding solution was then submerged for three hours in a water bath set at 60 °C. A slight change in the solution's colour from clear to milky indicated that this technique was successful.

The nucleation site on the POF was successfully formed using the dip and dry method, resulting in a uniform profile of the ZnO nanorods. The POF samples were dipped for 1 minute in the seeding solution and then dried for 1 minute on a hot plate at 90 °C. The procedure was repeated ten times. As the last stage of the dipping procedure, the POFs were annealed at 90 °C for three hours. Lastly, 1.40 g of hexamethylenetetramine (HMT), and 2.97 g of zinc nitrate hexahydrate, both from Ajax Finechem Pty Ltd., were dissolved in 1000 ml of deionized (DI) water to produce 10 mM solutions of each substance. After being positioned vertically in 200cc of the synthesis solution, the seeded POFs were cooked in a 90 °C oven.

2.4. Experimental setup

The schematic representation of the sensor system is depicted in Figure 1. The NodeMCU ESP8266 microcontroller is connected to a receiver circuit that includes a phototransistor. This receiver circuit is augmented by an operational amplifier (Op-Amp) which amplifies the received signal, facilitating the transmission of an analog voltage spanning the range of 0-3.3 V to the microcontroller's analog port. The voltage reading and the adulteration level will be shown on a 16×2 liquid crystal display (LCD). The Blynk platform was used to link the system to the IoT platform. The system parameter and output values, such as output voltage and adulteration level, are then displayed on the smartphone.

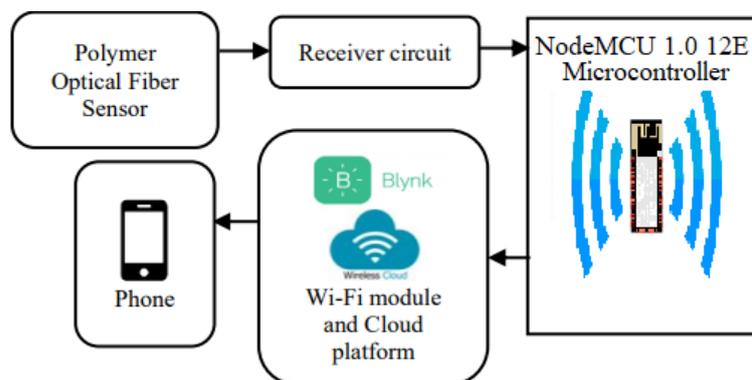


Figure 1. Block diagram of IoT integration to POF sensor

3. RESULTS AND DISCUSSION

This section will discuss the performance of the proposed sensor in terms of refractive index analysis, output optical power, output voltage for electrical characterization, and sensitivity.

3.1. Refractive index analysis

The variation in the refractive index with the volume percentage of adulterants in coconut oil for the uncoated POF probe is shown in Table 2. We found that, when more paraffin oil is added to coconut oil, the refractive index of the oil samples increases. Our findings indicate that, the refractive index of pure coconut oil is 1.4511, and the refractive index increases with the amount of adulterant added. For example, the refractive index increases by up to 0.9% with 80% adulteration. Paraffin oil has a different intensity due to its refractive index, which is roughly 1.4300 compared to 1.4511 for pure coconut oil. Therefore, given that paraffin oil and coconut oil have different chemical properties, the refractive index of the oil sample will change as more paraffin oil is added to the coconut oil.

Table 2. Volume % of adulterant oil in coconut oil vs refractive index

Sample	Volume % of adulterant oils	Refractive
1	0	1.4511
2	20	1.4531
3	40	1.4593
4	60	1.4620
5	80	1.4640

3.2. Output optical power

As the percentage of paraffin oil in the oil mixture increases, the output power, measured in dBm, decreases significantly for both uncoated and ZnO-coated sensor shown in Figures 2(a) and (b), respectively. As previously stated, the introduction of adulterants into pure coconut oil causes the chemical properties to change. As a result, the molecular structure of pure coconut oil changes dramatically, leading to the formation of new molecular entities within the composite medium. According to Figure 2, the output power of pure coconut oil is -21.81 dBm, while 80% adulteration produces -23.33 dBm. Meanwhile, the output power for pure coconut oil for ZnO-coated POF is -21.65 dBm, compared to -23.56 for an 80% adulteration level. When the POF sensor is immersed in different oil concentrations, the rate at which the light source passes through the POF sensor varies, resulting in a variation in output power [24]. Referring to both figures, the output power produced by the ZnO-coated POF shows a higher output power compared to the uncoated POF.

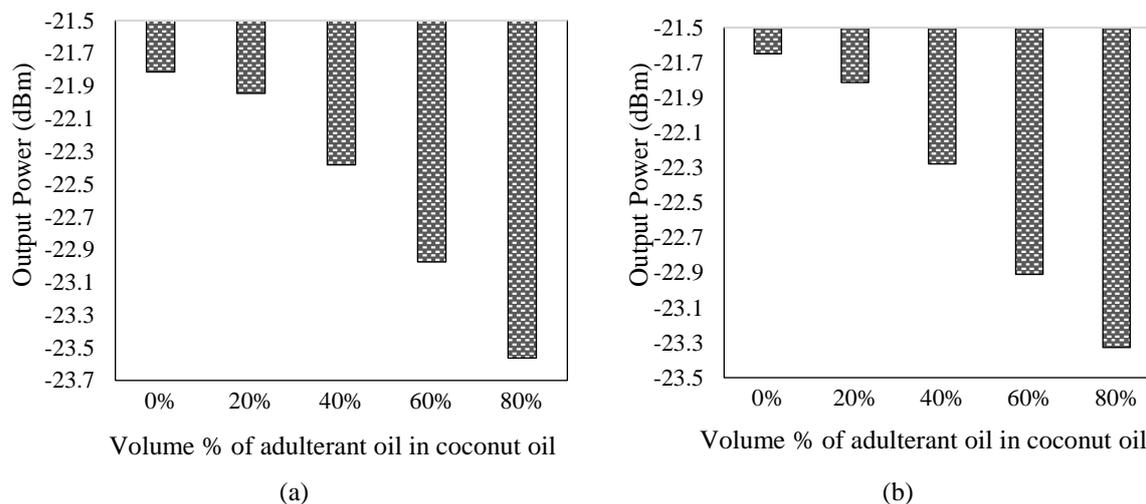


Figure 2. Response of output power against adulteration percentage for; (a) uncoated POF and (b) ZnO-coated POF

3.3. Output voltage

The output optical power is directly related to the output voltage [25]. Figures 3(a) and (b) show that the output voltage declines as the percentage of paraffin oil in the oil mixture increases for both uncoated and ZnO coated POF, respectively. It implies that the responsiveness of the output voltage is closely related to the output optical power. Previous research work, as discussed in reference [26], can be used to validate the findings presented in the graph. The researcher investigated the use of optical fiber sensors in salinity measurement. It was discovered that the output voltage had an inversely proportional relationship with the concentration of sodium chloride (NaCl) solutions. The research yielded similar results to the current study's findings. From Figure 3, it can also be concluded that the uncoated POF (Figure 3(a)) has a lower voltage sensing capability than the ZnO coated POF (Figure 3(b)).

3.4. Sensitivity and accuracy

The sensitivity and accuracy of the proposed sensor can be determined using a linear fitting curve for the voltage response against the level of adulteration shown in Figure 3. The slope of the line graph represents the sensitivity of the fiber when interacting with different adulteration level [26]. A steeper slope indicates that the fiber sensor is more sensitive to variations in the solution's composition. Hence, it can be

seen that the ZnO-coated POF has a much steeper slope than the uncoated POF. The sensitivity of the uncoated POF is 0.073 V/%, whereas the sensitivity of the ZnO-coated POF is 0.085 V/%. This disparity emphasises the coating's effectiveness in improving the sensor's responsiveness to changes in the percentage of adulteration, enhancing its potential for use in the precise detection and monitoring of edible oil adulteration. It can be concluded that coating the POF with a sensitive material such as ZnO improves the optical sensor's sensitivity.

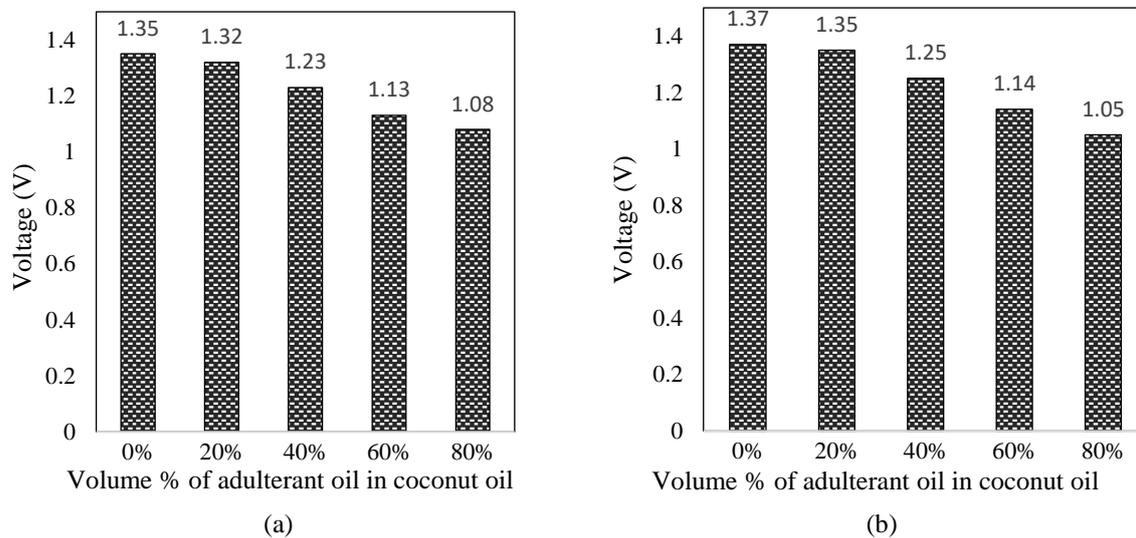


Figure 3. Graph of output voltage against oil samples concentration for; (a) uncoated POF and (b) ZnO coated POF

In order to validate the results, the adjusted coefficient of determination (R^2) was calculated from the linear curve in Figure 3. R^2 , which measures the strength of the correlation between the sensor and the response variable, is widely used to assess model accuracy, with the R^2 value determining the degree of fit. The R^2 value of the uncoated POF was 97.46%, while the R^2 value of the coated POF was 96.49%. A R^2 value of more than 90% indicates that the model is accurate [27]. Table 3 summarises the sensor's performance in terms of sensitivity and R^2 value.

Table 3. Sensitivity and R^2 for coconut oil adulteration sensor

Sample	Sensitivity (V/%)	R^2
Uncoated POF	0.073	0.9746
Coated POF	0.085	0.9649

3.5. Internet of thing monitoring

The final stage of the detection system involved the establishment of a linkage between the sensor and the IoT platform. Figure 4 shows the voltage display on the smartphone by the Blynk application. The ZnO-coated sensor was chosen for the final characterization stage due to its higher sensing sensitivity. The integration facilitated the visualisation of recorded output voltage data on the interface of the real-time monitoring system, ensuring users are aware of any potential contamination of the oil sample via the IoT platform.

Aside from displaying the voltage of the oil sample, the device also reported if the oil was safe for human consumption. This functionality was demonstrated by the illumination of either red or green LEDs in response to the oil samples current conditions and voltage. The red LED was set to light up when the level of oil adulteration reached 50%, which is around 1.2 V, while the green LED was set to light up when the adulteration rate was less than 50%.

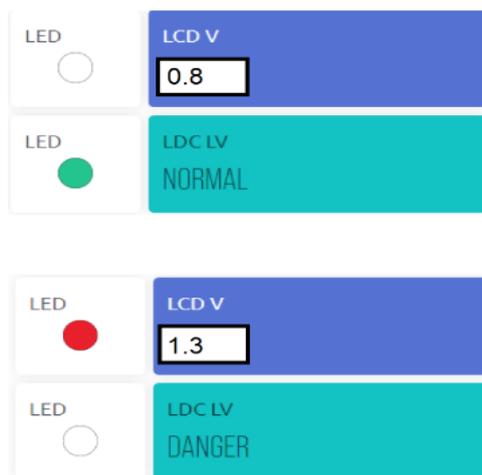


Figure 4. An example of the voltage displays on the smartphone by the Blynk application

4. CONCLUSION

In conclusion, the study presented in this paper evaluated the performance of a proposed sensor for detecting adulteration in edible oil, specifically coconut oil with paraffin oil as an adulterant. The performance of the sensor was assessed through refractive index analysis, output optical power, output voltage, and sensitivity. The results showed that the ZnO-coated POF had a higher voltage sensing capability and sensitivity than the uncoated POF, confirming that coating the POF with a sensitive material such as ZnO improves the optical sensor's sensitivity. The study also established an integration between the sensor and the IoT platform, allowing for the visual representation of recorded output voltage data on the real-time monitoring system's interface. The study's findings indicate that the proposed sensor has the potential to be utilised for the precise detection and monitoring of edible oil adulteration, providing a reliable and accurate method for ensuring the safety and quality of edible oil products. However, additional and in-depth research may be required to better comprehend the sensor POF's respond as an adulterant detector, involving more design parameter variations to be investigated.

ACKNOWLEDGEMENTS

We would like to thank Universiti Teknikal Malaysia Melaka (UTeM) and the Ministry of Higher Education (MOHE). This research is supported by funding from UTeM.

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