

Graphene- and Multi-Walled Carbon Nanotubes-Coated Tapered Plastic Optical Fiber for Detection of Lard Adulteration in Olive Oil

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Abstract: A rapid and high-performance sensor for lard adulterant in edible oil was developed using the tapered plastic optical fiber (POF) coated with graphene and multi-walled carbon nanotubes. The coating material was deposited onto a tapered POF with a taper waist diameter and a taper length of 0.45 mm and 1 cm, respectively. The addition of the coating material was used to increase the sensitivity and selectivity coefficient of the tapered POF toward the lard substance. The sensing mechanism is based on a simultaneous interaction of lard substance and an evanescent wave of tapered POF with the coating layers. The results showed that graphene coating on the tapered POF increased the selectivity coefficient of the tapered POF towards lard substance from 33.54 to 324.19, and it gave a sensitivity of 0.427 dBm/%. In comparison, multi-walled carbon nanotubes coating increased the selectivity coefficient to 71.65 and increased its sensitivity to 1.189 dBm/%. Thus, the proposed configuration of the tapered POF with the coating material offered a simple configuration for a rapid, high sensitivity and selectivity detection of lard adulterant in edible oils.

Keywords: Lard adulteration; tapered optical fiber sensor; graphene; carbon nanotubes; food safety

Citation: Ika PUSPITA, Ninik IRAWATI, Kartika Anoraga MADURANI, Fredy KURNIAWAN, Sekartedjo KOENTJORO, and Agus Muhamad HATTA, "Graphene- and Multi-Walled Carbon Nanotubes-Coated Tapered Plastic Optical Fiber for Detection of Lard Adulteration in Olive Oil," *Photonic Sensors*, 2022, 12(4): 220411.

1. Introduction

Animal-based oils and fats are immensely used as a flavor enhancer and reduce food production costs [1]. In some countries, lard is one of the cheap animal fats. Consequently, lard is the typical animal fat adulterated in edible oils to produce low-cost margarine, butter, and shortenings [2, 3]. Besides its

taste, lard is added to vegetable oils on margarine or butter production to increase its appearance since lard will maintain its texture at room temperature [4].

The addition of lard in edible oil and fat-based products has mostly not been listed on the product label. This action is categorized as food forgery that was reported by the Food and Drug Administration

Received: 22 June 2021 / Revised: 30 December 2021

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DOI: 10.1007/s13320-022-0652-y

Article type: Regular

(FDA) in the last two decades [5–8]. Mislabeling of product composition leads to an imbalance of fatty acid consumption by the consumers. It can cause harmful diseases, including coronary heart and neurodegenerative diseases, and become a religious matter [9–11]. Therefore, it is necessary to identify lard substances in oil and fat products.

Several methods have been developed to identify and detect lard in food products [12–16]. In our previous work [12], a tapered plastic optical fiber (POF) demonstrated an excellent performance to detect lard addition in olive oil since the POF has several advantages compared with the silica optical fiber. It has better compatibility with organic materials, flexibility, and durability [17]. Besides, the POF is cheaper than the silica optical fiber. The tapered structure is a simple modified structure, ease of implementation and fabrication on the POF, allowing the guided modes to be excited and interact with the surrounding medium [18]. However, increasing its selectivity to a specific lard substance is essential. Therefore, a nanoparticle layer is commonly added to an optical fiber biosensor to be bounded with a specific target compound since it can improve the sensor's sensitivity, selectivity, and response time [19–22]. Graphene and carbon nanotubes (CNTs) are nanomaterials that have currently become limelight in the physical and chemical sensing technology. They have extraordinary optical properties, including optical and molecular adsorption, surface plasmon, and photoluminescence [23–25]. Recent works demonstrated the tremendous performance of graphene and CNTs for glucose, ethanol, and gas detections [26–28].

On the other hand, a gold-graphene quantum dots (Au-GQDs) layer was coated onto a tapered POF for lard detection. The photoluminescence properties of Au-GQDs were investigated as the change of lard concentration [29]. This work showed a promising lard detection by applying nanomaterial layers on optical fiber sensors.

This paper reported the excellent performance of lard adulteration in olive oil sensors based on the tapered POF coated with graphene and multi-walled carbon nanotubes (MWCNTs). This study investigated the proposed sensor's sensitivity and selectivity to adulterants indicated by the oil refractive index variation. The achieved results were carried out experimentally based on the output intensity measurement of the tapered POF. It gave advantages, including a compact design and rapid detection method compared with conventional lard detection methods [13, 29].

2. Materials and methods

2.1 Sample preparation

The adipose tissue of pig was rendered from subcutaneous fat of pig belly obtained from a local market. It was cut into small pieces and melted on a pan heated on the stove. The pig fat (lard) was filtered using a cloth filter from an unwanted solid phase of fat. Meanwhile, the olive oils were obtained from the local supermarket. Lard was mixed into the olive oil with varied volume ratios ranging from 0 – 5% (v/v) with a 1% difference since it was challenging for detecting adulterants in low concentrations [13, 15, 16]. In order to obtain a homogenous mixture, the adulterated oil samples were mixed in ultrasonic bathing for 30 minutes. All the samples were kept at room temperature of 20 °C during the measurement. The temperature variation of the sample would lead to the optical properties' variation, including refractive index and absorption of samples and refractive index of the optical fiber sensor [30]. Refractive indices of the olive oils were measured as well by using an Abbe-Wellington refractometer.

2.2 Synthesis of graphene and MWCNTs

Graphene and MWCNTs were developed as presented in [31, 32]. The graphene was obtained from graphene-poly(lactic acid) (PLA) filament in the previous study [31] and characterized by the field

emission scanning electron microscopy (FESEM) and Raman spectroscopy. The FESEM image showed the morphology of the graphene slurry solution. Raman spectroscopy measurement indicated that the obtained graphene was multi-layer with some graphene layers (nGL) of around 25 layers [31]. MWCNTs were obtained from MWCNTs-acrylonitrile butadiene styrene (ABS) filament [32]. Characterization of the obtained MWCNTs was conducted by Raman spectroscopy in acetone suspension.

2.3 Fabrication of graphene and MWCNTs-coated tapered POF sensor

The tapered POF was fabricated by a chemical process as reported in [33]. A multimode POF of Super ESKA™, SH4001 with a length of 15 cm, was utilized to fabricate the proposed tapered POF lard sensor. The POF had a polymethyl-methacrylate resin core with a refractive index of 1.49 and a numerical aperture of 0.50. The core diameter and the fluorinated polymer cladding thickness were 0.98 mm and 0.02 mm, respectively. Firstly, the POF jacket was removed using a stripper for 5 cm on the middle of the length. The cladding of 1 cm length was polished after being immersed in the acetone solution to soften the cladding layer. Then, de-ionized water was used to remove the residuals. After the cladding was removed, the tapering process was started. The tapering process was the same as the cladding removal process. The process was carried out until it reached the desired diameter of the waist of 0.45 mm since, in our previous work, the tapered POF with a taper length of 1 cm and a waist diameter of 0.45 mm demonstrated the excellent performance on the detection lard substance in olive oil [12]. The microscopic images of tapered POF parts are depicted in Fig. 1. 2 mg of synthesized graphene and 2 mg of MWCNTs were diluted in 2 ml tetrahydrofuran (THF) and 2 ml acetone, respectively, as described in [31, 32]. The dilution was drop-casted onto a clean tapered POF

surface. The coating layer covered the transition region up to the other transition region since the coating layer would act as the new cladding layer for the tapered POF and interact with the sample molecules [31, 32]. The coated tapered POF was dried at room temperature for 24 hours before it was used for lard sensing. Figure 2 exhibits the microscopic images of the tapered POF coated with graphene and MWCNTs. The thickness of the coating layer obtained was around 0.02 mm.

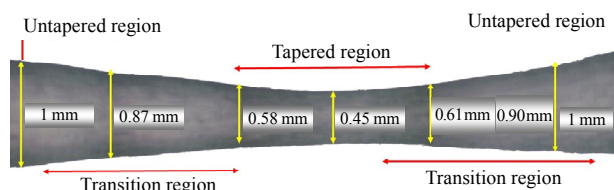


Fig. 1 Microscopic image of the tapered plastic optical fiber and its regions.

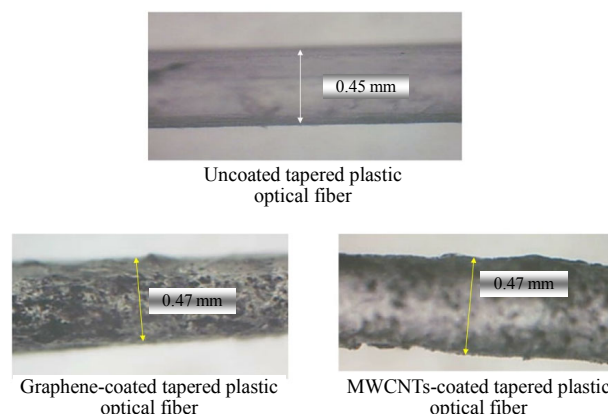


Fig. 2 Microscopic images of the tapered POF coated with graphene and MWCNTs in comparison with the tapered POF without the coating layer.

2.4 Lard sensing experimental setup

Figure 3 shows a schematic experimental setup. A light-emitting diode (LED) with an operational wavelength of 930 nm and a photodetector, Thorlabs S140C optical power meter, were connected to both ends of the coated tapered POF. The optical power meter was connected to a universal serial bus (USB) computer cable to record the measured output optical power fluctuation. The coated tapered POF was fixed in a customized petri dish. The LED source, the coated tapered POF in the petri dish, and

the photodetector were aligned in a straight line to eliminate the bending losses that might occur. The Arduino was utilized to give the stable voltage to the LED. The LED was turned on 15 minutes at a minimum before it was used for measurements to reach a stable emission. Then, a certain amount of the oil sample was dropped to the Petri dish, and the measured output optical power was recorded for 100 seconds – 200 seconds. After the measurement was recorded, the coated tapered POF was cleaned using distilled water to break the polar bonding of the lard molecules and the coating materials. The process was repeated until the measured output power of the coated tapered POF back to the level when there was no sample. Then, the cleaned coated tapered POF could be used to measure the other oil samples.

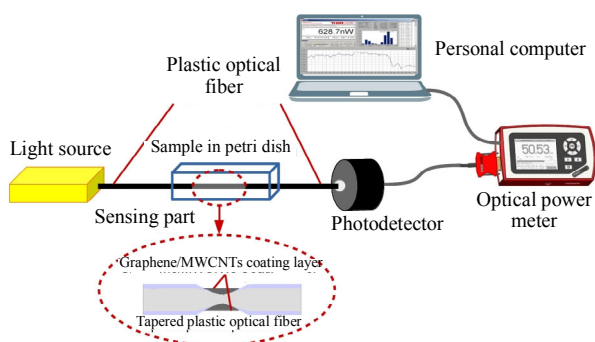


Fig. 3 Experimental setup for performance evaluation of the proposed fiber sensor.

3. Results and discussion

3.1 Refractive index of the samples

The refractive index changes of olive oil due to the addition of the lard substance are presented in Table 1. It shows that with an increase in the lard concentration in olive oil, the refractive index of olive oil increases. It can be seen that the refractive index measurement has an average error of 0.0001 RIU.

3.2 Selectivity coefficient of the proposed sensors

The absorbances of lard and olive oil at 930 nm are depicted in Fig. 4(a). They were calculated based

Table 1 Refractive index of olive oil as the variation of lard concentration in olive oil.

Lard concentration (%)	Refractive index
0	1.468 0
1	1.468 4
2	1.468 6
3	1.468 9
4	1.469 0
5	1.469 1
100	1.472 5

on the measured transmission power of the tapered POF with different coatings by using the equation below [27]:

$$A = -\lg\left(\frac{P-D}{R-D}\right) \quad (1)$$

where P is the measured optical power, D is the measured dark optical power, and R is fiber's optical power reference value without the sample. The proposed tapered POF should have high selectivity as a sensor for a specific lard substance. The selectivity coefficient of lard (S) can be calculated as in [34]:

$$S = \frac{A_{\text{lard}}}{A_{\text{oil}}} \quad (2)$$

where A_{lard} and A_{oil} are the absorbances of 100% lard and 100% olive oil at the corresponding wavelength, respectively. Lard's selectivity coefficients on untapered, non-coated, graphene-coated, and MWCNTs-coated tapered POF were 1.63, 33.54, 324.19, and 71.65, respectively, using (2), as shown in Fig. 4(b). The addition of the coating material on the tapered POF increased the selectivity coefficient. Note that graphene-coated tapered POF was more selective than lard substance.

3.3 Static performance of the proposed sensors

When it was immersed in the samples, graphene-coated tapered POF sensor's transmission power was measured repeatedly five times from varied lard concentrations in olive oil (0% – 5%) and refractive indices (1.468 0 – 1.469 1). The measured transmission powers are presented in Fig. 5. The measured transmission power decreased

as the lard concentration in olive oil and refractive index increased. It showed that the transmission power of the graphene-coated tapered POF and lard

concentration in olive oil had a polynomial relationship as well as the transmission power and the refractive index of the olive oil samples.

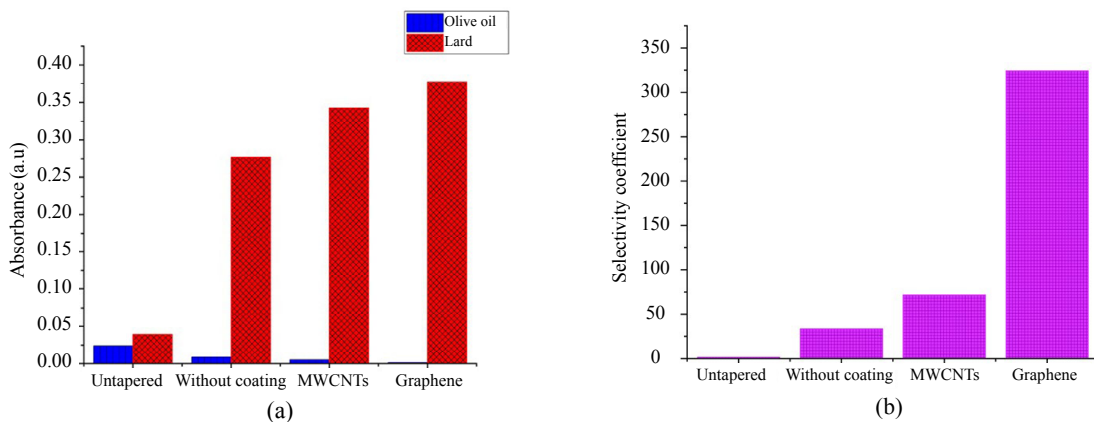


Fig. 4 Properties of tapered POF at 930 nm with different coatings: (a) absorbance of lard and olive oil and (b) selectivity coefficient on the lard substance.

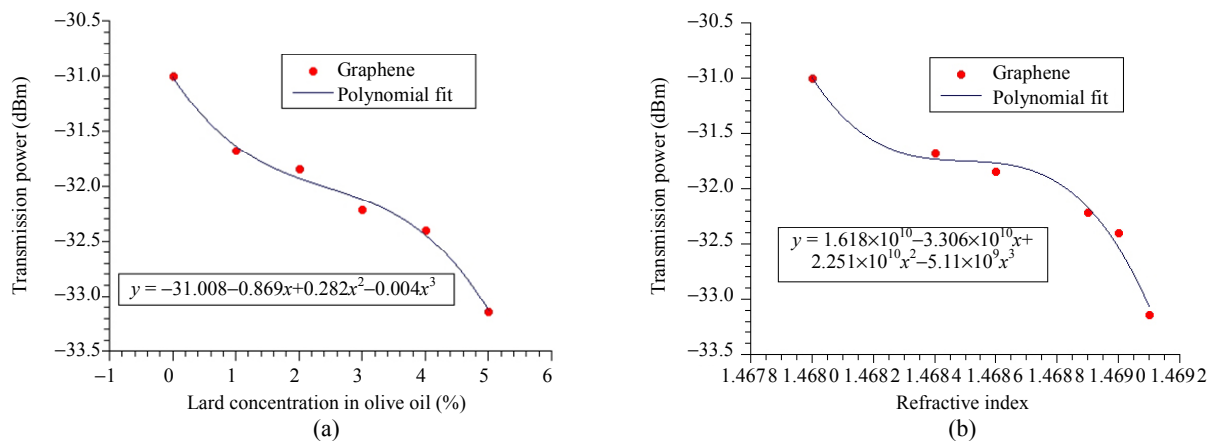


Fig. 5 Transmission power of the graphene-coated tapered POF as a function of (a) lard concentration in olive oil and (b) refractive index.

In the tapered POF, the constriction of the core diameter from the transition region to the tapered region allowed the confined modes to travel through the fiber and became evanescent modes [35]. The evanescent modes would interact with the surrounding materials of the tapered POF. It led to the change in the measured transmission power of the developed optical fiber sensor. In the graphene-coated tapered POF, oil sample's fatty acid molecules could not penetrate and were absorbed by the graphene layer since graphene is impermeable to any atoms [26, 36, 37]. Hence, it contributed to the high selectivity coefficient of the graphene layer to the lard substance. The existence

of the fatty acid molecules in the graphene structure changed the optical characteristic of the graphene. Small changes in the refractive index of a graphene layer on the tapered POF led to the mode propagation variation in the tapered POF, as illustrated in Fig. 6. The real part of the graphene refractive index affected the depth of penetration of each mode. Simultaneously, the imaginary part of the graphene refractive index affected the ability of the graphene to absorb the TE mode [38, 39]. Therefore, the graphene-coated tapered POF had a high degree nonlinear response toward different lard concentrations in olive oil and refractive index.

A different coating material was applied on the

tapered POF to investigate the optimum performance (high sensitivity and selectivity) of the tapered POF to detect the lard substance in olive oil. The investigation was carried out for the same number of repetitions and oil samples on the MWCNTs-coated tapered POF. Figure 7 shows the measured transmission power of the MWCNTs-coated tapered POF as a function of the lard concentration in olive oil and oil’s refractive index.

As the opposite of the graphene-coated tapered POF, the measured transmission power of the MWCNTs-coated tapered POF increased nonlinearly as the lard concentration, and the refractive index increased. Unlike the response on the graphene-coated tapered POF, the MWCNTs-coated tapered POF response had a lower degree of nonlinearity. The response was almost linear towards the oil’s refractive index variation, as shown in Fig. 7(b).

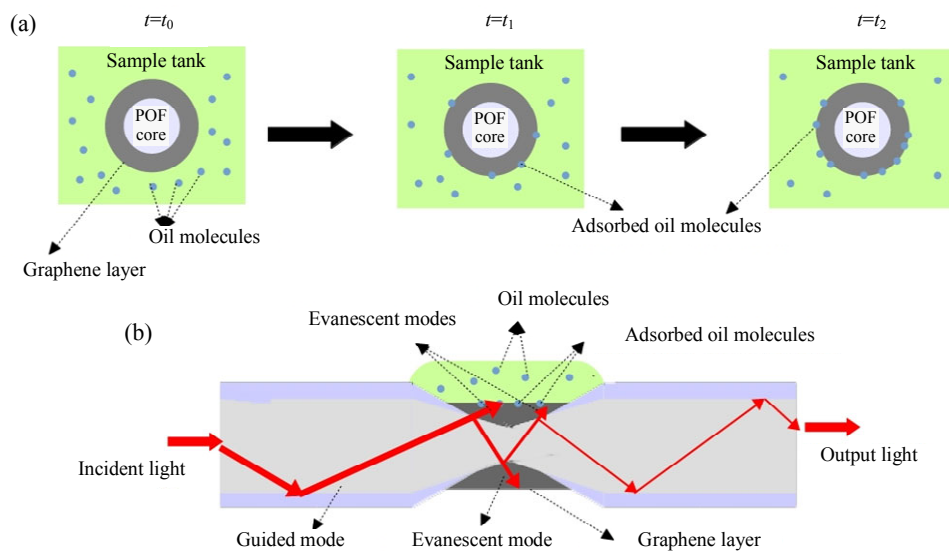


Fig. 6 Illustration of (a) time evolution of oil molecules adsorption by the graphene layer on tapered POF and (b) schematic diagram of the mode propagation in the graphene-coated tapered POF.

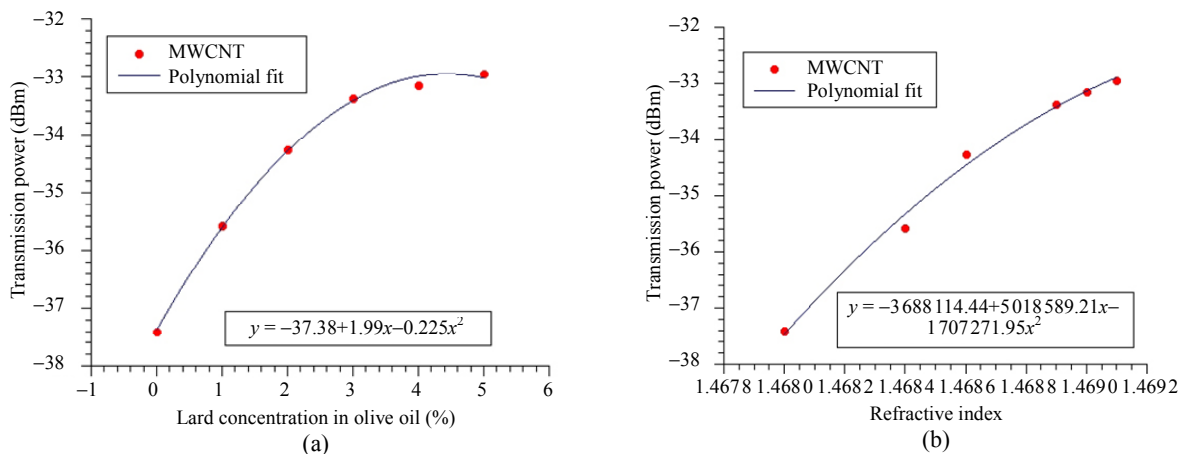


Fig. 7 Transmission power of the MWCNTs-coated tapered POF as a function of (a) lard concentration in olive oil and (b) refractive index.

The porous structure of MWCNTs allowed the fatty acid molecules to penetrate deeply and interact

with the evanescent wave, as shown in Fig. 8. It was consistent with the result in Fig. 9(b). It showed that there was no slow diffusion process, and it could be seen that there was no rise/decay time on the

dynamic response. The surface roughness of the MWCNTs coating was higher than that of graphene coating [40]. Hence, it gave a significant contribution to increasing the sensitivity.

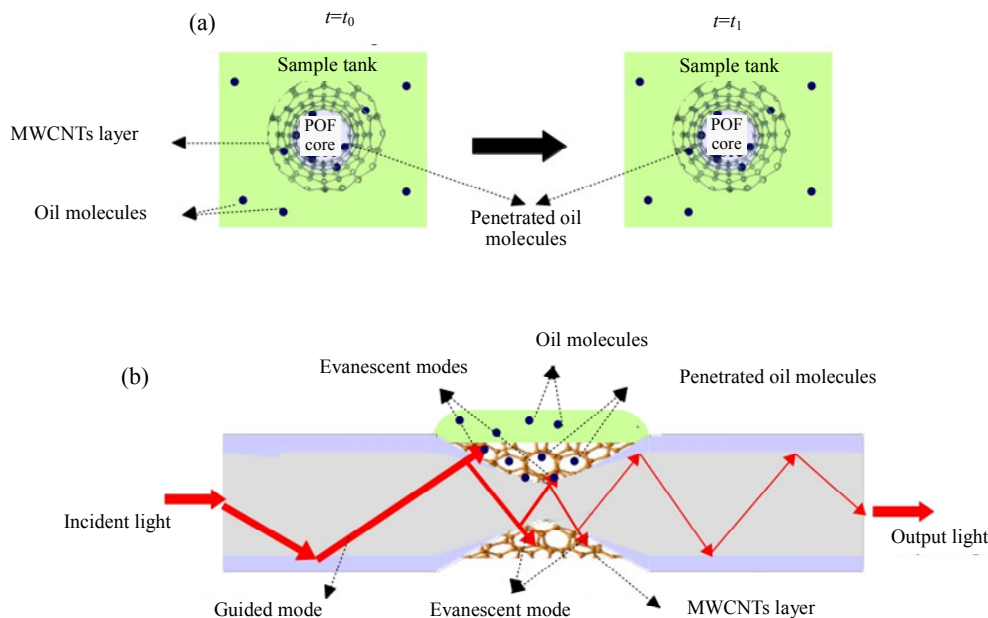


Fig. 8 Illustration of (a) time evolution of oil molecules adsorption by the MWCNTs layer on the tapered POF and (b) schematic diagram of the mode propagation in the MWCNTs coated tapered POF.

3.4 Dynamic performance of the proposed sensors

The dynamic measurements were also carried out to evaluate the sensor performance. The measurement stabilities of the graphene-coated and MWCNTs-coated tapered POFs are presented in Figs. 9(a) and 9(b), respectively. The output optical power from the graphene tapered POF was measured and recorded for 200 seconds. It can be seen in Fig. 9(a) that the measured output decayed before reaching the steady-state measurement. It showed that the molecule adsorption process occurred nonlinearly since the molecules diffused gradually into the graphene layer. For the 1% lard concentration, the measured optical power dropped rapidly within 40 seconds. The decay time increased as the lard concentration in olive oil increased, while the dynamic response of the MWCNTs-coated

tapered POF was measured and recorded for 100 seconds, as shown in Fig. 9(a). Unlike the dynamic response of the graphene-coated tapered POF, it did not decay or rise to reach the stable measurement. Therefore, it showed the stable measurement since $t = 0$.

Table 2 shows the performance comparison of the untapered POF and tapered POF without the coating and with the graphene coating and MWCNTs coating. The addition of the graphene coated on the tapered POF increased the selectivity coefficient towards the lard substance. However, it decreased the sensitivity of the tapered POF. It also had a long decay time. Meanwhile, adding the MWCNTs layer on the tapered POF enhanced the sensitivity and the selectivity of the tapered POF towards the lard substance. With an advantage, it did not have a rise time or decay time to give the actual value of the measurement.

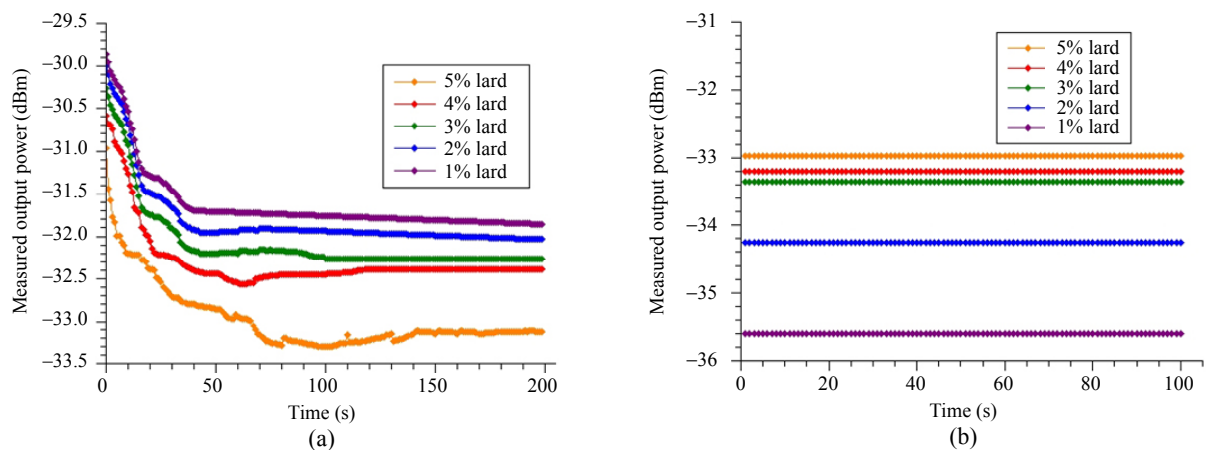


Fig. 9 Measurement of stability of the tapered POF with the (a) graphene coating and (b) MWCNTs coating.

Table 2 Performance evaluation of the tapered POF with various coating layers.

Parameters	Untapered [12]	Without coating [12]	Graphene-coated	MWCNTs-coated
Sensitivity (dBm/%)	0.03	0.858	0.427	1.189
Standard deviation (dBm)	0.001	0.003	0.002	0.07
Selectivity coefficient	1.63	33.54	324.19	71.65
Rise time/decay time (min)	0	0	2.5	0

Table 3 summarizes the performance comparison of the proposed graphene- and MWCNTs-coated tapered POF, Au-GQD-coated tapered optical fiber [28], and Fourier transform infrared spectroscopy [13] for detecting lard adulteration in fat-based products. The proposed fiber sensor showed the highest sensitivity and the smallest limit of detection (LOD). The LOD was obtained by using the equation [41]:

$$\text{LOD} = \frac{3S_D}{a} \quad (3)$$

where S_D and a are the standard deviation and sensitivity of the developed sensor, respectively. MWCNTs-coated tapered POF's superior performance gives a promising potency of rapidity, low cost, and ease of implementation for lard adulteration in olive oil detection.

Table 3 Performance comparison with existing lard detection sensors.

Parameters	Our proposed fiber sensor (MWCNTs-coated tapered POF)	Our proposed fiber sensor (graphene-coated tapered POF)	Au-GQD-coated tapered optical fiber [29]	Fourier transform infrared spectroscopy [13]
Sensitivity	1.189 dBm/%	0.427 dBm/%	0.022 a.u./%	0.0243 a.u./gram
Linearity	0.90	0.86	0.80	0.98
LOD	0.18%	0.01%	0.46%	-
Standard deviation	0.07	0.002	0.0034	1.75

4. Conclusions

Graphene-coated and MWCNTs-coated tapered POF sensors for detecting lard mixed in olive oil based on the refractive index variation were fabricated and investigated experimentally. The tapered POF was fabricated with a waist diameter of 0.45 mm and a length of 1 cm. The graphene and MWCNTs were deposited onto the tapered POF by cast dropping the graphene-THF and MWCNTs-acetone suspension. The static and dynamic responses of the proposed fiber sensors were also evaluated. A graphene coating layer on the tapered POF increased tapered POF's selectivity by ten times. However, it decreased the sensitivity by half. On the other hand, the MWCNTs coating layer on the tapered POF increased the selectivity of the tapered POF by two times and increased the

sensitivity. Thus, the proposed sensor offered the high sensitivity, simple configuration, low cost, and easy implementation to detect lard adulteration in olive oil to contribute to the food safety technology.

Acknowledgment

The research has been funded by the Indonesian Government, KEMENRISTEKDIKTI (Ministry of Research, Technology, and Higher Education of the Republic of Indonesia) for supporting this work under project scheme University's Fundamental Research Program (Grant No. 918/PKS/ITS/ 2021).

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