Taper parameters effect on tapered POF for lard adulteration in olive oil detection

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Lard adulteration in edible oil products gives the concern to develop a method for the detection of lard substance in oil products. In this paper, a tapered plastic optical fiber (POF) was investigated experimentally to detect lard in olive oil. Tapered POF is a sturcture made by reducing the fiber diameter gradually. Taper length and taper waist diameter were varied to evaluate the performance of the tapered POF for detection of lard adulteration in olive oil. A maximum sensitivity of 0.858 dBm/%(v/v) was obtained by using tapered POF with a taper length of 1 cm and taper waist diameter of 0.45 mm.

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1. Introduction

Edible oils are everyday food products made of fat extracts either plant-based fats or animal-based fats. The plant-based edible oils mostly made of palm, olive, coconut, canola, soya bean, sunflower, sesame, and linseed [1] while, the animal-based oils made of livestock, poultry, and fish [2]. Edible oils are widely consumed for baking, frying, as salad dressing, etc. [3].

Recently, food adulteration became a significant issue in food manufacturing in some countries [4-5]. Edible oils are the most common food product to be adulterated [6]. Oil fraud is aimed to increase the apparent value and lowering production cost [6-7]. In general cases, the oil manufacturer adulterated the oils using other cheaper substances. Along in 1993 - 2015, the Food and Drug Administration (FDA) found some olive oil fraud cases. The olive oil products adulterated by cheaper oil products such as soybean oil or refined oil [8-11].

Meanwhile, animal-based oils nowadays are widely used for frying due to favorite flavors [12]. The most consumed animal-based oils are made of pigs (lard), tallow, and grease [13]. Lard is animal-based oil, which commonly blended to vegetable oils or added to cooking oils to produce low-cost margarine, butter, shortenings, or other oil-based products [14].

The oil fraud gives harm to the consumer in respect of allergic and cholesterol diet. Consuming excess saturated fat leads to coronary heart disease [15]. Therefore, it is essential to do authentication for oil products or oil-based products and to detect oil adulteration.

Some techniques, either optical or non-optical based techniques, were utilized to detect lard adulteration in oil products. Non-optical based techniques such as dielectric spectroscopy [16] and electrical nose [17] were used to differentiate animal fats. These techniques were utilized to differentiate lard adulterated in other animal fats. It showed a high performance to detect lard in beef and tallow fats. However, it cannot detect lard adulterated in plant-based oils while optical-based technique such as Fourier Transform Infrared Spectroscopy (FTIR) is commonly used to detect lard adulteration in animal fat [18-20]. FTIR shows great performance to detect adulteration of animal fat in other animal fat, but it is powerless for detecting animal fat in plant fats and has a complex system.

In the other hand, tapered fiber optic structure has been developed and utilized for many sensing applications, such as refractive index sensor and strain sensor [21], label-free biomolecules sensor [22], and salinity detection [23]. The tapered optical fiber is fabricated by reducing the optical fiber diameter. Reducing the diameter of optical fiber induces the guided mode in the core region to be evanescent mode [24]. Tapered optical fiber structure was utilized for some sensing principle such as evanescent absorption [25], evanescent fluorescence [26], and surface plasmon resonance (SPR) [27]. Tapered fiber structure has some superiorities such as ease and low cost of fabrication, compactness, and immunity of electromagnetic interference.

In our previous study [28], tapered plastic optical fiber was investigated to detect lard adulteration in an olive oil based on the spectroscopic method. It showed that there were some wavelength shifting due to the addition of lard substance. It gives the advantage to design a compact and rapid detection method based on intensity change on a specific wavelength. In this paper, the effect of taper parameters such as taper length and taper waist diameter on tapered plastic optical fiber structure for detecting lard adulteration in olive oil is investigated experimentally. The taper parameters are varied in a fixed operating wavelength.

2. Theory

Initially, an optical fiber is designed to be low loss optical signal waveguide which confines all modes in the core region. The mode properties in the fiber optics determined by the V-number, which given as [29],

$$\mathrm{V} = \frac{2\pi}{\lambda} r \sqrt{n_{\mathrm{co}}^2 - n_{\mathrm{cl}}^2} \tag{1}$$

where r is the core radius, λ is the wavelength of the light coupled to the fiber optic. n_{cl} and n_{co} are the refractive indexes of cladding and core, respectively. Since it is designed to be a low loss, hence only a small portion of optical power distribution is unconfined in the core region as an evanescent field. The evanescent field is part of guided mode which penetrates to the cladding layer. The evanescent field has penetration depth, which is much smaller than the cladding thickness. The penetration depth of the evanescent field in the POF is given as [24, 29-31],

$$d_p = \frac{\lambda}{2\pi\sqrt{n_{\mathrm{co}}^2 \sin^2(\theta) - n_{\mathrm{cl}}^2}} \tag{2}$$

where d_p is the penetration depth of the mode, θ is the incident angle at the interface of core and cladding. Each mode propagating in fiber optic has a different evanescent field. The higher mode orders the broader distribution of optical power in evanescent mode [24, 29-31].

Evanescence phenomenon plays a role immensely in the tapered optical fiber structure. The change of the fiber diameter and the surrounding refractive index affect the measured output power. As a chemical sensor, the samples act as the new cladding layer for the tapered optical fiber. The changing of the cladding refractive index varies the V-number of the optical fiber. Hence, the measured optical power will be changed.

The tapered optical fiber structure is made by reducing the diameter of the fiber, as shown in Fig. 1. The tapered optical fiber divided into three parts, untapered region, transition region, and taper region. In the untapered region, the optical fiber has V-number as given in Eq. (1). In the transition region, there are two conditions: adiabatic and non-adiabatic conditions. The adiabatic transition condition is the condition which the all modes are carried out with the efficiency as high as 99.5% [29]. While the non-adiabatic condition is the condition which some of higher order mode can be excited. In the tapered region, the V-number of optical fiber change as the change of the core radius. The new V-number gave as [31],

$$\mathrm{V} = \frac{2\pi}{\lambda} r_{\mathrm{tot}}(z)\sqrt{n_{\mathrm{co}}^2 - n_{\mathrm{sample}}^2} \tag{3}$$

where z is the coordinate along the fiber. r_{tot} is the radius of the tapered fiber and a function of z, n_{sample} is the refractive index of the target sample. In the multimode optical fiber, each mode has the same portion of light energy. In the region of taper waist, some part of the higher order mode is not guided in the thin waist. Therefore, part of the light energy carried by the higher order mode is not confined in the core region. Hence it determines the transmission loss [29].

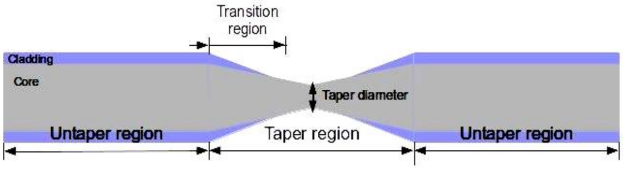


Fig. 1. A schematic of a tapered plastic optical fiber

3. Experiment

Lard was extracted from commercial pork fats. The pork fats were heated up and melted. The liquid phase of pork fats was filtered by using a paper filter, to separate with unwanted particles. Lard was adulterated into commercial olive oil with a concentration of 0-5% (v/v). The lard and olive oil mixed by using ultrasonic bathing for 30 minutes to ensure that the samples are adulterated homogeneously. Before the samples used for measurement, the temperature of all samples were fixed at the room temperature i.e 20 ^{o}C. Since the variation of the temperature of the samples will vary the optical properties of the samples [32].

The tapered structure was made by reducing the core diameter of the plastic optical fiber (POF). The POF type of Super EskaTM, SH-4001 was used, and it has the core diameter and cladding thickness of 980 µm and 20 µm respectively. It has a refractive index of the core of 1.49 and the numerical aperture of 0.50. The tapered structure was fabricated by using the chemical process, as described in [33]. It was fabricated several tapered POF with different taper length from 1 to 3 cm, and the taper waist diameter of 0.76, 0.56, and 0.45 mm. Fig. 2 shows a fabricated tapered plastic optical fiber. The untapered and tapered regions are shown in Fig. 2(a), 2(e), and 2(b), 2(c), 2(d), respectively. One can see the tapered structure with a waist region diameter of 0.45 mm, and the transition region of 0.75(0.7) mm in left (right) side.

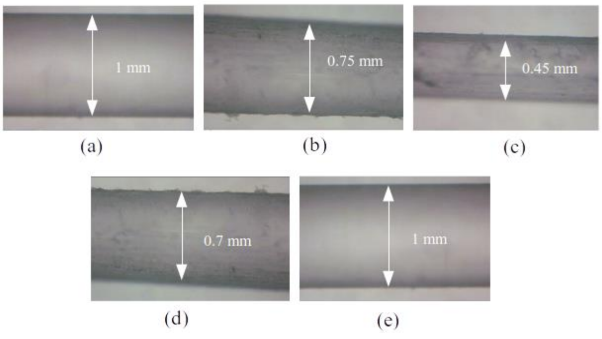


Fig. 2. Microscope view of (a) Untapered region (left side) (b) Transition region (left side) (c) Waist region (d) Transition region (right side) (e) Untapered region (right side)

The absorption characteristics of pure lard, pure olive oil, and lard adulterated in olive oil were investigated. The absorbance of pure lard, pure olive oil and 5 % of lard adulterated in olive oil were measured by using Ocean Optic Spectrometer in the range of 400 - 1000 nm of wavelength (Fig. 3a). Based on the absorbance characteristic, a suitable operational wavelength for an intensity-based measurement can be determined.

In Fig. 3(b), a light emitting diode (LED) was connected to the tapered fiber structure. The output power of the tapered optical fiber was connected to a detector Thorlabs S140C. The data was recorded using a personal computer. It is also noted that the LED source, the tapered POF in the petri dish, and the photodetector were aligned in a straight line to eliminate the bending losses that may occur. The LED source was driven by Arduino to give stable voltage. The measurement was conducted at fixed room temperature of 20^{o}C. Since the tapered plastic optical fiber performance has a dependency on the environmental temperature [34].

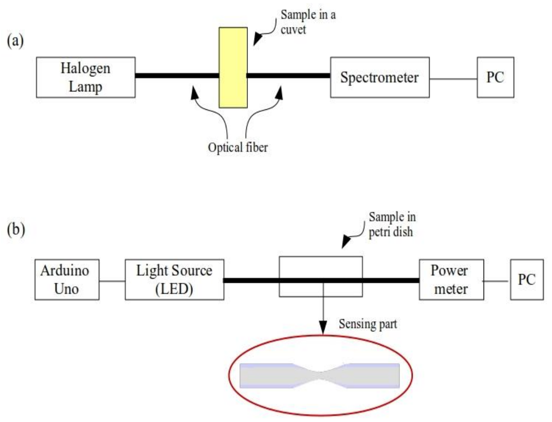


Fig. 3. Experimental setup for (a) absorbance measurement (b) intensity-based detection of lard adulteration in olive oil

4. Result and discussion

Before the effect of the taper parameters on tapered POF for detection of lard adulteration in olive oil were investigated, it is essential to select operating wavelength and to get the refractive index information of the samples. Fig. 4 shows the absorption characteristic of pure lard, pure olive oil, and 5% of lard adulterated in olive oil. One can see, pure lard, pure olive oil and 5% of lard adulterated in olive oil have an absorbance peak at the wavelength of 930 nm. The addition of lard substance to olive oil increased the absorbance of the light at the wavelength of 930 nm. It occurred because lard and olive oil contains fatty acids and some other hydrocarbons compound with a complex chemical structure. The interaction of light and the oil chemical compounds is in the vibrational state. Hence, they absorb light energy in the region of infrared wavelength, as described in [35]. Therefore, the wavelength of 930 nm was used as the operational wavelength for the intensity-based measurement of tapered POF, as presented in Fig. 3(b). Since the evanescent field of tapered POF will be mostly absorbed by the samples which act as the cladding and the small change in the sample's content will give significant change on the measured output power of the tapered optical fiber.

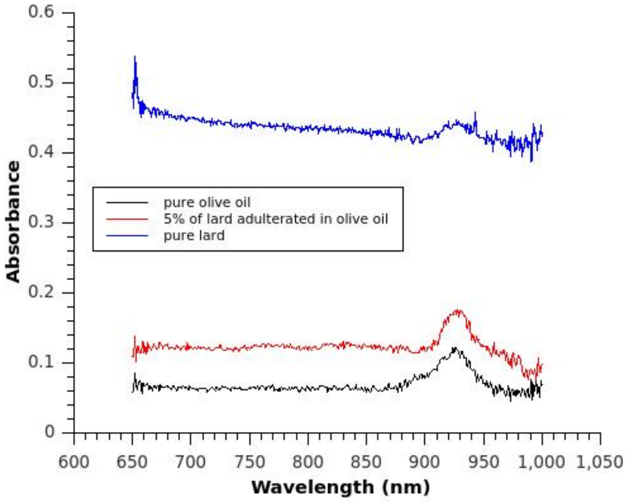


Fig. 4. Absorbance spectra of pure lard, pure olive oil and 5% of lard adulterated in olive oil (color online)

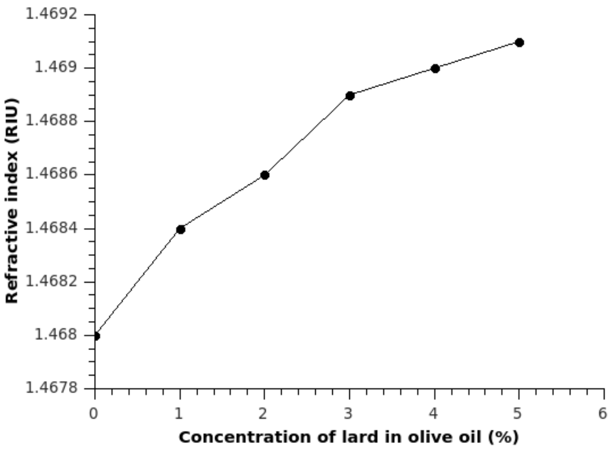


Fig. 5. Refractive index of olive oil caused by the addition of lard in olive oil

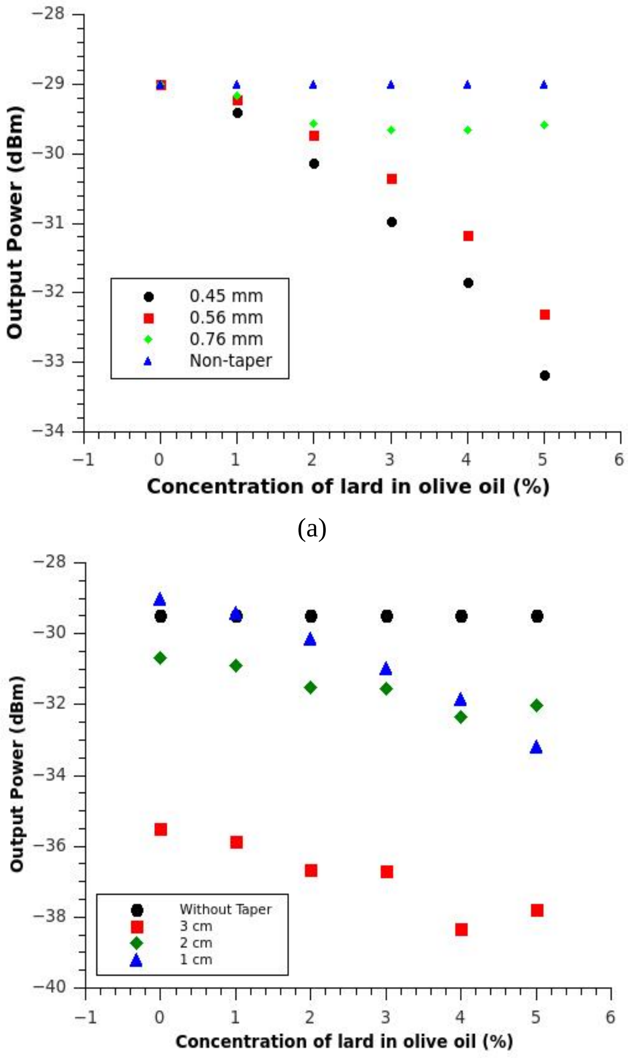
It is necessary to measure the refractive index of olive oil caused by the addition of lard. In Fig. 5, it is presented the change of olive oil's refractive index due to the addition of lard in olive oil. The measurement was carried out using an Abbe refractometer. One can see, the refractive index of the olive oil will be increased as the lard concentration increases since lard has a higher refractive index than olive oil. Based on the Eq. (2), the higher the sample's refractive index, the greater the penetration depth of the guided mode in tapered POF. The deeper the penetration depth, the larger the mode absorbed by the sample. Hence, the measured output power of tapered POF will be significantly dropped.

Fig. 6 shows the intensity-based measurement due to the variation of adulterated oil concentrations (0 to 5%) for the tapered POF with a variation of tapered length and tapered waist diameter. Fig. 6 (a) shows the measured output power for various tapered POF sensors with a tapered waist diameter of 0.45, 0.56, and 0.76 mm at a fixed tapered length of 1 cm in adulterated oils. As the decreasing the tapered waist diameter, the sensitivity of the tapered POF sensor incresased. Since in non-tapered POF, the mode mostly guided in a core region. Only a small distribution of the higher order mode is evanescent mode. Although it has significant penetration depth, the power distribution carried by this mode is small. Hence, it leads to the small change in output power of POF. In the other hand, tapered POF shows the significant effect of the evanescent phenomenon to the output power of POF. The tapered structure plays a role in leading to more higher order modes become evanescent mode. The V-number will significantly change due to the change of the core radius and the sample's refractive index. The smaller the taper waist diameter, the higher order modes become evanescent mode. The evanescent modes carried more power distribution. Hence, it caused the larger change in measured output power.

In Fig. 6 (b), it is presented the measured output power of various sensors with a tapered length of 1 to 3 cm at a fixed tapered waist diameter of 0.45 mm in adulterated oils. The measured output power in the tapered optical fiber is linearly changed due to lard concentration change. While the measured losses in non-tapered optical fiber sensor have almost no change, although the concentration of lard in olive oil is changed. The change of the V-number may cause it. The small change on the V-number does not give a significant change to the number of the guided mode. Hence, the measured optical power does not change much.

Meanwhile, in the tapered POF, the variation of the tapered length cause the change on the angle of the tapered. The shorter the taper length, the larger the angle of the tapered region. The wide angle of the tapered region leads the guided modes to become the evanescent mode. Hence, it can cause a significant change in the measured output power of the tapered POF.

The result shows that an agreement to the theory. The smaller the diameter and the shorter the taper length of the tapered plastic optical fiber, the sensitivity of the tapered optical fiber increased. It occurred because the transition region of the tapered plastic optical fiber is in non-adiabatic condition as described in [23, 28-30]. The smaller the taper waist diameter and the shorter the taper length, the guided mode region decrease steeply. This condition leads to an increase of the evanescent mode. Since the refractive index of the sample increased, the evanescent mode of the tapered POF has great penetration depth. The sample will also absorb this evanescent mode since the sample absorbs light at the wavelength of 930 nm. In this paper, the minimum taper length and diameter, which can be achieved is 1 cm and 0.45 mm, respectively. The shorter taper's length and the smaller taper's waist diameter may be achieved for a greater sensitivity, however the taper structure may be easily broken.



(a)

(b)

Fig. 6. Sensitivity evaluation of (a) different taper lengths (b) different taper waist diameters (color online)

Table 1 shows the performance comparison of the tapered POF for detection of lard adulteration in olive oil with different taper lengths. The tapered POF length of 1 cm and tapered waist diameter of 0.45 mm has the highest sensitivity and good linearity and low limit of detection (LOD). However, the tapered plastic optical fiber with the taper length of 2 cm has the lowest sensitivity of 0.4175 dBm/% and the highest limit of detection.

Table 1. Performance evaluation of tapered plastic optical fiber to detect lard adulterated in olive oil

Parameters	**1 cm**	**2 cm**	**3 cm**
Measurement range (%)	0 - 5	0 - 5	0 – 5
Linearity	>97%	>85%	>86%
Sensitivity (dBm/%)	0.858	0.418	0.832
Limit of Detection (%)	0.00265	0.00141	0.04876
Standard Deviation	0.01	0.01	0.20

The performance of tapered POF presented on Table 1 is only valid at temperature 20 °C. The investigation of temperature variation (10–60 °C) effect on tapered optical fiber performance for the measurement of a glycerine solution with the refractive index range of 1.33–1.41 showed a great dependency of measurement accuracy to a temperature change [34]. Since the temperature variation will lead to thermo-optic effect (TOE) on sample and the tapered POF and thermal expansion (TE) on the tapered POF. TOE causes deviation on sample and tapered POF refractive indexes from their actual values. While TE causes some changes on tapered POF dimension [34]. Hence, it is important to do further investigation on the effect of temperature to tapered POF performance to detect lard adulteration in olive oil.

From all experiments were conducted, tapered plastic optical fiber sensor shows great potential to detect for lard adulterated in olive oil. It gives the best option for quick detection with high sensitivity and low limit of detection. However, it needs further development to be selective at specific lard substance.

5. Conclusion

Lard adulteration detection in olive oil by using the tapered POF sensor was proposed and demonstrated experimentally. The tapered POF was fabricated by reducing the POF diameter gradually on a certain length. It is shown that measured output power of the tapered POF varied by the change of lard concentration in the olive oil. The taper's parameters of length and core waist diameter played an essential role in the sensor's performance. It is found that the tapered POF sensor with a tapered length of 1 cm and tapered waist diameter of 0.45 mm has a maximum sensitivity of 0.858 dBm/% and the linearity of >97%. As the taper's length and core waist diameter decreased, the sensor's sensitivity increased. However, the minimum taper length and core waist diameter, which can be achieved in sensor fabrication were 1 cm and 0.45 mm, respectively. The proposed sensor offers high sensitivity, simple configuration, low-cost and easy for implementation to detect lard adulteration in olive oil. For future development, this configuration will be developed to be selective to the specific lard substance.

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