

Identification of Recycled Cooking Oil by Varying Number of Au/GO Coated Fiber Optic's Loop Ring

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This study investigates the effect gold nanoparticles (Au)/graphene oxide (GO) coated fibre optics with various number of loop ring in identifying recycled cooking oil based on their frying time. Various layers of gold nanoparticles (AuNP) suspension were deposited onto the substrate via drop casting technique. Few material characterizations were performed such as Fourier Transform Infrared (FTIR) spectroscopy, Ultraviolet Visible (UV-Vis) spectroscopy and refractometer analysis. Three macrobending structures of Au/GO coated fiber optic's loop ring such as single, double and triple loops with diameter of 1.5cm were formed to generate large area of evanescent field around the fibre and to excite surface plasmon polaritons due to the interaction between AuNPs and analytes. Cooking oil samples with different frying time such as 14 minutes, 28 minutes and 42 minutes were prepared as analytes. The FTIR analysis revealed some changes in the transmittance percent of some bands as well as some slight shifts in the exact position of the bands due to the different of fatty acid composition. As the Au/GO coated fibre optics were immersed into the samples, the penetration depths were obtained as 142.75nm and 168.91nm at operating wavelengths of $\lambda=1310\text{nm}$ and $\lambda=1550\text{nm}$, respectively. By using 1550nm operating wavelength due to its greater penetration depth than 1310nm, triple loop rings fibre optics coated with two layers of AuNP and one layer of GO exhibits a linear consistency with maximum sensitivity up to 69.17% due to the formation of large evanescent field around the loop area and intense SPP excitation.

Keywords: SPR; Au/GO; fibre optic sensor; recycled cooking oil; loop ring

I. INTRODUCTION

The usage of cooking oil in food production has effects to human's health if consumed regularly. The degree of saturation of fatty acids is the most crucial factor in deciding the oil's resistance to oxidation and staleness. Oxidation causes the form of free radicals that can damage cells and lead to cancer. Free radical levels that exceeds the produced antioxidant will cause oxidative stress (Bogoriani & Sudiarta, 2016). Oxidative stress is an imbalance between the production and detoxification of reactive oxygen species (Münzel *et al.*, 2017). Reusing cooking oil induces a change of physical and chemical properties such as the formation of toxic compound such as 4-hydroxy-trans-2-nonenal (HNE).

Consuming cooking oil containing HNE can contribute to cardiovascular disease, stroke, Alzheimer's disease, Parkinson's disease, various liver complications as well as cancer diseases (Ishak *et al.*, 2016).

Plant extraction produces oils or fats that are commonly known as vegetable oils. Most of the oils can be used for cooking oil or for fuel production. There are various types of vegetable oils such as palm oil, sunflower seed oil, canola oil, soybean oil and many more. Vegetable oils are commonly used in food production as they contain more unsaturated fatty acids compared to animal fats. Vegetable oils or cooking oils are used in preparing food especially frying. The quality assurance of cooking oil is important as to prevent the

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formation of toxic compounds and the loss of nutritional components (Hosseini *et al.*, 2016). At high temperature, lipid reaction such as hydrolysis, oxidation and thermal alteration are occurred during heating of cooking oil. Dobarganes and Marquez-Ruiz (2015) stated that there are many resulting compounds of hydrolysis, oxidation and thermal alteration that will increase the rate of degradation of cooking oil.

Recently, various types of sensors have been rapidly developed for food security applications. Surtono *et. al* (2018) designed a measuring instrument for cassava starch content using a strain gauge sensor as a weight gauge. Electrochemical sensor is one of the alternative methods for food safety inspection (Jingna & Yang 2017; Maduraiveeran *et. al.*, 2017; Mishra *et al.*, 2018). Lately, better sensitivity and less complicated structure of optical sensor attracts researcher to employ this light based sensor in various applications including food safety area (Cocola *et. al.*, 2019; Pechprasarn *et. al.*, 2019; Semeano *et al.*, 2018). The optical sensor can be categorised into two types, such as fibre optics based sensor (Ascorbe *et. al.*, 2017; Kamarulzaman *et. al.*, 2020; Mukhtar *et. al.*, 2012; Ascorbe *et al.*, 2017) and free space optics based sensor (Gan *et. al.*, 2019; Mukhtar *et. al.*, 2019; Smith *et al.*, 2017). Surface plasmon resonance (SPR) were found able to enhance the sensitivity of the sensor by excited surface plasmon polaritons to enhance the strength of evanescent field. This phenomenon can be created with the interaction between TM polarized light and noble metal. Gold (Au) and silver (Ag) have been proved as the best materials to generate maximum plasmonic effect (Mukhtar *et. al.*, 2017a; Mukhtar *et. al.*, 2018b; Wang *et. al.*, 2018; Zhao *et al.*, 2018). There are few factors that affect the optimization of SPR signal namely light wavelength, metal thin film's thickness, physical structure of prism, size and structure of nanoparticles and the introduction of materials (Mukhtar *et. al.*, 2013; Mukhtar *et. al.*, 2017b; Murat *et. al.*, 2016; Usha *et al.*, 2016).

The penetration depth of the fibre can be calculated as shown in the following Equation (1) where λ is the vacuum wavelength of the light launched into the fibre, n_1 and n_2 are the refractive index of the core and analytes, respectively. θ_c is critical angle in the sensing region with respect with the normal to the core-cladding interface, θ is the angle of the

wave with the normal to the core-cladding interface, θ_0 and is the skewness angle (Rashid *et al.*, 2020).

$$d_p = \frac{\lambda}{2\pi n_1 (\cos^2 \theta_c - \cos^2 \theta \sin^2 \theta_0)^{1/2}} \quad (1)$$

This study presents the utilization of SPR phenomenon to detect the quality of cooking oil by using a macrobend fibre optics. The loop ring fibre optics has been coated with gold nanoparticles (AuNP) to excite surface plasmon polaritons (SPP). Various stages of recycled oil samples have been prepared by immersed the sensor into the samples that had been heated with different frying time. The output of this work exhibits the potential of SPR based macrobend fibre optic sensor to identify the recycled cooking oil. We believe this research outcome will benefit to the society in food safety area.

II. MATERIALS AND METHOD

The jacket and polymer coating of standard optical communication single mode fibres (SMF) were removed by using optical fibre stripper. Three loop ring structures of fibre optics with various number of loops, such as single loop, double loops and triple loops were formed by bending the SMF into 1.5 cm in diameter. The bending fibre structure is believed will generate the evanescent waves propagation. Figure 1 shows the schematic diagram of macrobent SMF with various number of loop rings. The proposed sensor utilizes the undesirable loss, e.g. bending loss as sensing mechanism by allowing the perturbation (acoustic vibration) to induce optical power losses through the bend fibres. Such losses are associated with the random change in the radius of bending

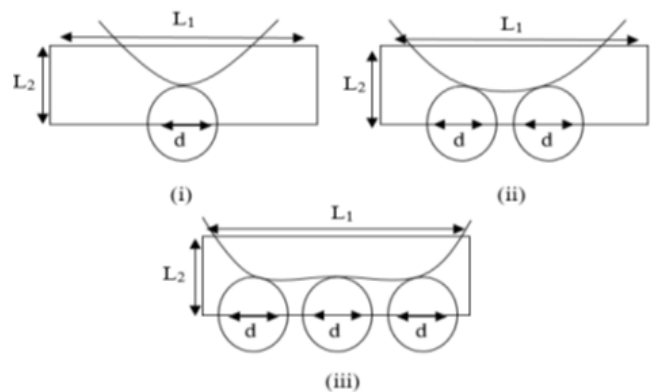


Figure 1. Schematic diagram of macrobent fiber optics with various number of loop rings (i) single loop, (ii) double loops, (iii) triple loops, where $d=1.5$ cm, $L_1=7.5$ cm and $L_2=2.5$ cm.

due to applied force or displacement (Ong *et al.*, 2017). When the single-mode fiber is bent, transition loss will arise due to the mode conversion from fundamental mode to leaky clad mode where light transmission at the outer part of the curvature is travel faster than the inner part which has smaller inner radius (Keiser 2008; Kamarulzaman *et al.* 2020). The unjacketed area was coated with gold nanoparticles, AuNP (diameter, $d=50\text{nm}$) and graphene oxide (GO) by using a drop casting technique. This technique was performed by deposited $50\ \mu\text{L}$ of AuNP in which was dispersed in H_2O onto the bent region by using a micropipette. The coated area was left dry for approximately 3 hours at room temperature. This step was repeated by adding the number of AuNP layer from one to three layers. Next, $50\ \mu\text{L}$ of GO (Brand: Graphenea) with 2.5 wt% concentration was coated onto the AuNP layer and let dry for approximately 12 hours at room temperature. In this study, the GO layer was fixed at one layer only. Note that, too thick of GO layer will result the absorption of SPP which will decrease the sensing ability of our proposed sensor (Mukhtar *et al.*, 2018a; Murat *et al.*, 2020). The optical properties of Au and GO were characterized by using UV-Vis spectrometer. The loop ring Au/GO coated fibre optics was glued onto the glass slide. The glass slide acted as a sensor holder.

The recycled cooking oil samples were prepared by deep fried 100g of potato fries in a cooking palm oil (Brand: Saji, manufacturer: Delima Oil Products Sdn. Bhd) with a different number of cooking cycles. About 20 ml of cooking oil samples were collected for each frying time, approximately about 14 minutes per cycle. The digital refractometer was used to determine the refractive index of the samples. Each sample was characterized by using Fourier Transform Infrared (FTIR) spectroscopy to determine the composition and existence of specific functional groups. All spectra were measured at region from 4000cm^{-1} to $650\ \text{cm}^{-1}$. Four samples of recycled cooking oil were prepared as listed in Table 1.

Table 1. Description of recycled cooking oil's samples

Sample	Description
1	New cooking oil
2	14 minutes of frying time
3	28 minutes of frying time
4	42 minutes of frying time

Figure 2 illustrates an experimental setup of loop ring Au/GO coated fibre optics for recycled cooking oil identification. The evanescent field and excitation of surface plasmon polaritons (SPP) were generated by connecting the loop ring Au/GO coated fibre optics with infrared (IR) laser source with wavelength of $\lambda=1310\text{nm}$ and $\lambda=1550\text{nm}$ (Brand: Noyes). The output signal was measured by using an optical power meter (Brand: Noyes). The Au/GO coated sensing region was then immersed in the cooking oil samples. The identification of recycled cooking oil was studied by varying the number of loop ring and Au layer of the Au/GO macrobent SMF. Throughout this work, the optimized structure of our proposed sensor had been investigated based on percentage of optical power changes as the sensor exposed to the samples.

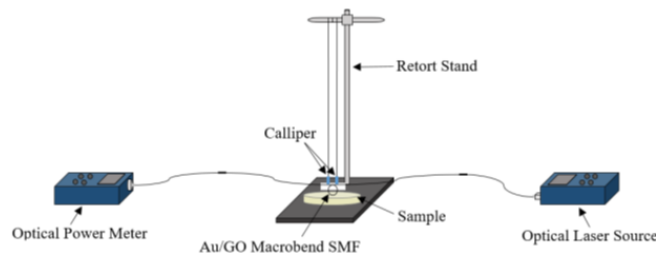


Figure 2. Schematic diagram of loop ring Au/GO coated fiber optics to identify recycled cooking oil

III. RESULTS AND DISCUSSIONS

All samples showed similar colour based on naked-eye visual observation. Refractive indices for each sample were slightly difference due to oxidization as recorded by the digital refractometer, as shown in Table 2. As the loop ring of the Au/GO coated fibre optics were immersed into the samples, the penetration depths were obtained as 142.75nm and 168.91nm at operating wavelengths of $\lambda=1310\text{nm}$ and $\lambda=1550\text{nm}$, respectively. As stated in Equation (1), the penetration depth is influenced by the refractive index of core

and medium. In this study, the penetration depth values for each wavelength at different refractive indices were similar since no distinct differences had been observed on the value of refractive index's samples.

Table 2. Refractive index of recycled cooking oil and the penetration depth of proposed sensor

Sample	Refractive index	Penetration depth, d_p (nm)	
		$\lambda=1310\text{nm}$	$\lambda=1550\text{nm}$
1	1.4653		
2	1.4654		
3	1.4655	142.75310	168.90634
4	1.4655		

According to the FTIR analysis, the cooking oil samples displayed indistinguishable patterns without distinct differences, except for intensities or peak areas as shown in Figure 3. A close interpretation of the spectra revealed that there are some changes in the transmittance percent of some bands as well as some slight shifts in the exact position of the bands. This change proposes that the oil composition has effects on the exact position of the bands and shifts in the bands when the fatty acid composition is changed.

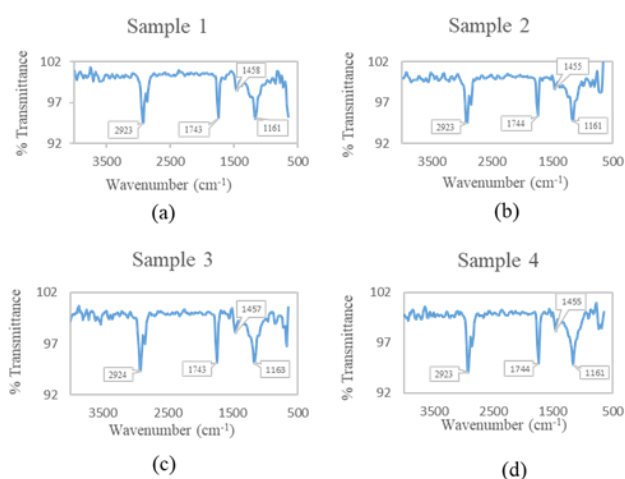


Figure 3. FTIR spectra for sample 1, 2, 3 and 4

Small changes of peak for each oil samples were resulted due to the changes of cooking oil's composition resulted from frying time (Table 3). The absorption bands at 2923-2924 cm^{-1} were due to the stretching vibration of the hydroxyl group ($\nu\text{O-H}$). The absorption bands at 1743-1744 cm^{-1} because of the stretching vibration of $\nu\text{C}=\text{O}$ and $\nu\text{C}=\text{C}$, or by the in-plane bending vibration of $\delta\text{N-H}$, showing the presence of carboxylic acid, fatty ketone, or amino groups functional groups. The peaks at 1161-1163 cm^{-1} were recognized to the $\nu\text{C-O}$ stretching vibrations, which are

Table 3. Major peaks observed from FTIR spectra of cooking oil samples

Functional group	Sample 1	Sample 2	Sample 3	Sample 4
C-H stretching band (hydroxyl group ($\nu\text{O-H}$))	2923 cm^{-1}	2923 cm^{-1}	2924 cm^{-1}	2923 cm^{-1}
C=O stretching band (carboxylic acid, fatty ketone, or amino groups functional groups)	1743 cm^{-1}	1744 cm^{-1}	1743 cm^{-1}	1744 cm^{-1}
Bending vibrations of CH_2 and CH_3 aliphatic groups	1458 cm^{-1}	1455 cm^{-1}	1457 cm^{-1}	1455 cm^{-1}
C-O stretching vibrations (hydroxyl, ester, and ether functional groups)	1161 cm^{-1}	1161 cm^{-1}	1163 cm^{-1}	1161 cm^{-1}

resulted from the hydroxyl, ester, and ether functional groups (Alshuiael & Al-Ghouti, 2020). AuNPs demonstrate a discrete optical characteristic usually known as localized surface plasmon resonance (LSPR), that is, the cumulative oscillation of electrons in the conduction band of AuNPs in resonance with a particular wavelength of incident light. Figure 4 shows the optical absorbance properties of AuNPs as the nanoparticles' suspension were deposited at 1 layer (single drop), 2 layers (two drops) and 3 layers (three drops) on the

glass slide against the wavelength ranges between 300 nm to 800 nm. Obviously, LSPR of AuNPs resulted in a strong absorbance band in the visible region around 500 nm to 600 nm in which indirectly verified the presence of AuNPs on the substrate via drop-casting deposition technique. The maximum optical absorbance peak for GO were found around 286 nm to 296 nm as illustrated in Figure 5, in which shows an excellent agreement with previous studies (Samsuri *et al.*, 2017).

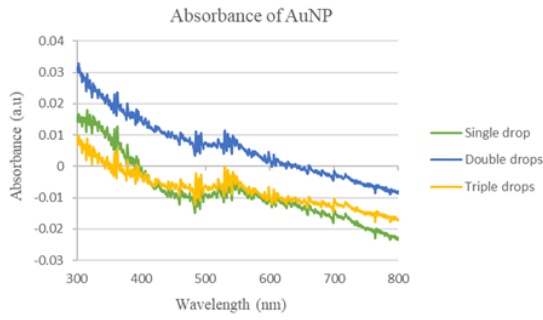


Figure 4. The optical absorbance properties of one, two and trilayers of AuNPs against the wavelength ranges between 300 nm to 800 nm

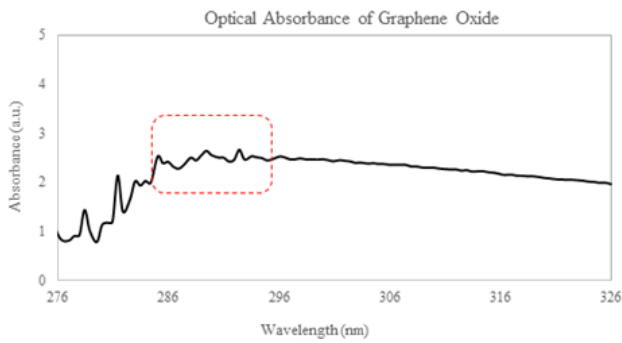


Figure 5. The optical absorbance properties of GO against with maximum absorption between wavelength of 286nm to 296nm

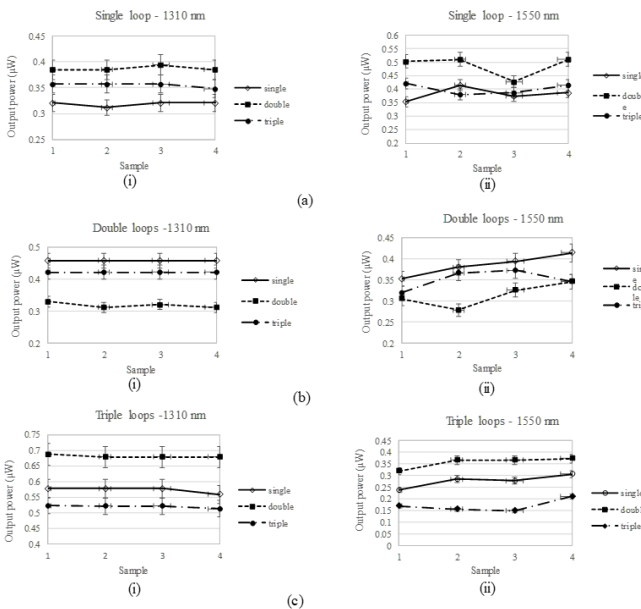


Figure 6. Optical output power against various samples of recycled cooking oil detected by loop rings Au/GO coated fibre optics (a) single loop, (b) double loops and (c) triple loops

Figure 6 presents the characteristics of optical power output against various types of cooking oil detected by Au/GO coated fibre optics sensor with numerous number of loop ring such as single loop, double loops and triple loops. The number of Au layers and excitation wavelength's values were also manipulated to determine the optimum structure to produce high sensitivity sensor. The deployment of sensor with operating wavelength at 1550nm resulted better sensitivity than 1310nm since less attenuation occurred at 1550nm (Kamarulzaman & Mukhtar, 2020). This condition could be apparently noticed by unobvious differences of output power when the operating wavelength was set at 1310nm. The output of this analysis indicates that 1310nm is unsuitable to be appointed as light source since the sensor cannot differentiate various types of analytes (Figure 6(a)(i), (b)(i) and c(i)). As lights source was changed to 1550nm, the value of optical output power shows a good response as the sensor exposed to the various stage of recycled cooking oil represented by the apparent changes of output power (Figure 6(a)(ii), (b)(ii) and c(ii)).

Sensitivity analyses were performed by determined normalised percentage difference of optical power changes before and after exposed to the samples as illustrated in Figure 7. The greater the changes, the better the sensitivity of sensor (Mukhtar *et al.*, 2020). The employment of single and triple loop rings of Au/GO coated fibre optics shows an excellent sensitivity of sensor by using 1550nm light source (Figure 7(a) and (c)). Based on the analysis, we found that the sensor's sensitivity can be optimized by using triple loop rings fiber optics coated with two layers of 50nm in diameter of AuNPs. This condition can be explained by considering the enhancement of evanescent field's strength due to the surface plasmon resonance and the macrobending effect as few numbers of loop ring were introduced in this study (Mukhtar *et al.*, 2020; Murat *et al.*, 2020). Note that the intensity enhancement of evanescent field resulted from the strong excitation of evanescent waves due to the triple macrobending of three loops SMF increased the penetration depth area of the sensor (Kamarulzaman & Mukhtar, 2020).

Figure 8 describes the sensing characteristics of triple loop rings Au/GO coated fibre optics with two layers of AuNP using 1550nm wavelength of laser source. Note that, the optical power differences increased linearly with the

increment of frying time. Recycled cooking oil contains more saturated fatty acids than new cooking oils. Heated cooking oil has poor quality due to saturated fatty acid content (Sugito & Firdausi 2018). The relationship can be expressed as $\Delta P=18.57x$. When the sensor was exposed to the new cooking oil, the optical power changed about 20.53%. The sensor detected about 25.56% power change as it was immersed into the recycled cooking oil that was used to deep fry food for 14 minutes. After 28 minutes of frying time, the percentage of power change resulted 68.77%. About 69.17% of power change was observed as the triple loop rings of Au/GO coated fibre optics was dipped into sample 4 (42 minutes of frying time). This analysis proves the selectivity capability of the

produced when duration of frying time increased. This condition occurred due to the presence more saturated fatty acids in recycled cooking oil. Small portion of light was radiated away from the fibre due to light leakage. This condition caused part of light was absorbed by the cooking oils resulted changes in output power. Excellent sensing properties were observed with the deployment of triple loop rings of Au/GO fibre optics due to the stronger evanescent field produced around the bent area. Larger coverage region of evanescent field resulted intense sensing area. The excitation of SPP were successfully enhanced by employed two layers of AuNPs. SPR with collective oscillation of electrons on the interface of metal/dielectric, detected the change of refractive index near the sensing surface, which is cooking oil. The recycled cooking oil interacted with the AuNPs, which caused the change of refractive index near the sensing surface. Since we used AuNP suspension, it was difficult to ensure that all macrobent region of fibre optics were uniformly coated by these metal nanoparticles. By adding another layer of AuNPs, the number of particles coated on the fibre increased. However, one layer of GO was sufficient to amplify the SPR signal and also acted as resistance layer to avoid oxidization of noble metal.

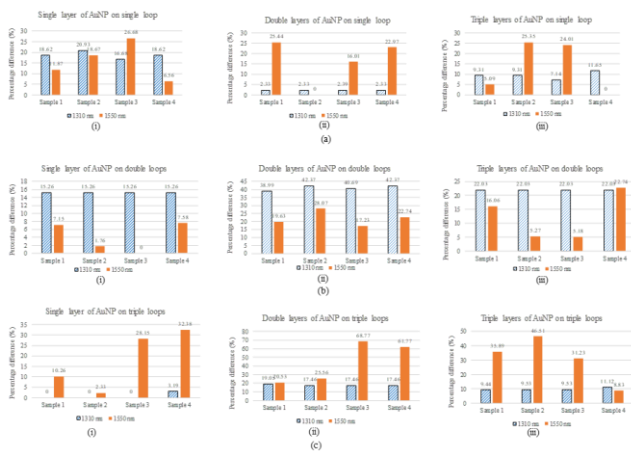


Figure 7. Percentage difference of optical power output with various number of loop (a) single, (b) double and (c) triple

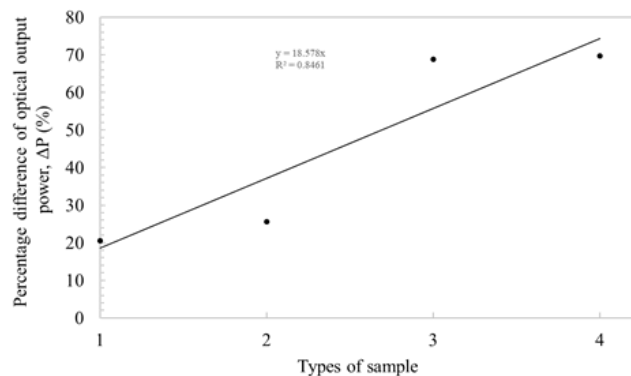


Figure 8. Percentage difference of optical power output for optimized Au/GO triple loops macrobend SMF sensor using two AuNP layers for quality assessment of used cooking oil samples

proposed sensor in detecting different samples of recycled cooking oils based on the amount of percentage power changes. Note that, higher refractive index of sample was

IV. CONCLUSION

This study proposed an alternative approach to identify the recycled cooking oil. A high sensitive Au/GO coated fibre optics sensor was successfully developed by using triple loop rings of fiber, which had been coated with two layers of Au (50nm in diameter) and one layer of GO. The linear relationship between the increment of frying time and optical output power indicates the excellent consistency and selectivity of the sensor with maximum sensitivity of 69.17%. Combination of strong excitation of SPR due to light interaction between AuNPs and analytes and wide evanescence field region around fibre optics produced by the triple loop rings resulted excellent sensitivity sensor. The output of this study portrays the potential of loop ring Au/GO coated fibre optics in identifying recycled cooking oil samples based on optical technique by employing low cost and simple fabrication technique.

V. ACKNOWLEDGEMENT

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