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ORIGINAL ARTICLE

Real time detection of milk's spoilage using Au/GO bend SMF sensor based on localized surface plasmon resonance effect

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ABSTRACT – Potential of hybrid gold nanoparticles/Graphene Oxide (Au/GO) coated single mode fiber (SMF) sensor by exploiting localized surface plasmon resonance (LSPR) effect for detection of spoiled milk is studied. Various diameters of SMF's cladding are prepared ranging from its original size, d=0.1250mm to d=0.1215mm. A mechanical polishing technique using sandpaper is applied to etch the cladding area in which results the SMF with diameter less than 0.1250mm. Hybrid layers of Au/GO are deposited onto the SMF via drop-casting technique by varies the number of layers of GO between one to five layers, meanwhile Au nanoparticles are kept constant at one layer. To generate LSPR, light with excitation wavelength of 1310nm and 1550nm are transmitted at the first end of Au/GO coated SMF resulting in strong scattering and extinction spectra. The fiber is bend about 8cm in diameter to produce evanescent waves around it. To investigate the sensitivity of sensor in detecting milk's spoilage condition, the Au/GO bend SMF sensor is immersed into the milk's solution which has been exposed to the environment for 24 hours, 48 hours and 72 hours. The maximum optical power response, ΔP%=6.85% is obtained when the one layer of Au/GO coated bend SMF had been immersed into the milk solution with exposure time of 24 hours. The values of ΔP% decrease about 3.50% and 2.00% respectively with the increment of exposure time at 48 hours and 72 hours. These results indicate the sensing ability of our proposed sensor to detect different levels of spoiled milk as the exposure time increased. The output of this study validates the main role of LSPR effect in enhancing the sensitivity of Au/GO bend SMF sensor for real time detection of milk's spoilage.

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Single mode fiber (SMF); gold; graphene oxide; Au/GO; LSPR; plasmonic; milk spoilage; food security; bend sensor

INTRODUCTION

Dairy product such as milk is a very famous routine food and can be considered as an ideal food especially for human because it provides many types of nutrients that human needs. The sterilized and pasteurized milk can maintain its quality only up to 24 hours in room temperature [1]. When it is left beyond 24 hours left in room temperature, milk become spoiled due to the presence of bacteria [2]. Microbial milk spoilage can severely affect industrial quality process and commercial success of dairy. Common spoilage bacteria species includes Psuedomonas fluorescens, Pseudomonas fragi and Bacillus cereus [3].

Recently, the development of optical sensors for various detection applications indicates their potential to be used in food safety [4-6]. The operating principle of optical sensors rely to the detection of refractive index of surrounding medium. They can be categorized into two types, namely fiber-based sensor and free space-based sensor. The fiber-based sensor utilizes optical fibers such as silica single mode fiber (SMF) [7,8] and multimode fiber (polymer optical fiber) [9] to be used as a substrate and light coupler. Meanwhile, the free space-based sensor usually deploys prism for light coupling [10]. SMF is commonly used in optical communication for networking purpose. It is easily commercially available in comparison with polymer optical fiber. Its small cladding area about 9µm in diameter make it an excellent candidate for optical sensing application which can enhance the propagation of evanescent waves around it [11].

Localized surface plasmon resonance (LSPR) happens when electromagnetic radiation interacts with the electrons in metal-based nanoparticles, such as gold (Au), silver (Ag) and platimum (Pt) resulting in strong scattering and extinction spectra that can then be exploited for various critical applications [12,13]. Au is the best noble metal to generate LSPR due to its high resistivity from oxidization and able to excite large amount of plasmon polaritons [14]. The LSPR effect can be generated through the use of various materials, including nanoparticles, nanoporous films, nanorods and also nanohole arrays [15]. All these structures show LSPR peaks at different locations, as the LSPR frequency depends primarily on the nanoparticle shape, size and properties of its material as well as the properties of the surrounding medium.

Today, optical sensors incorporated with 50nm diameter of Au nanoparticles are found able to enhance their sensing properties due to high ratio from surface to volume in comparison with Au thin film [16,17]. Graphene is an exciting material because it has large specific surface area, high intrinsic mobility, excellent thermal conductivity, and its optical transmittance and good electrical conductivity merit attention for optical sensing applications. Graphene oxide (GO), one of the most important derivatives of graphene, is characterized by a layered structure with oxygen functional

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groups bearing on the basal planes and edges. Previous research shows that the employment hybrid Au/GO in optical sensors resulted better sensitivity than the single layer Au [18]. Perfect light coupling techniques such as prism coupling or by using the optical fiber are required to ensure 100% of light is successfully converted into LSPR signal.

This paper discusses the potential of Au/GO bend SMF sensor by exploiting the LSPR effect for detection of spoiled milk. A 0.125mm diameter of SMF is prepared by using original SMF (9/125µm), meanwhile fibers with diameter less than 0.125mm are fabricated by using mechanical polishing technique. Various layers of Au/GO are coated onto the bend SMF to amplify the LSPR signal. The output of this work indicates a simple operating principle of high sensitivity and selectivity sensor as it was immersed into the spoiled milk with various environment exposure time. This real time monitoring detection exhibits an excellent potential of Au/GO bend SMF sensor in food safety area.

METHODS AND MATERIALS

Preparation of SMF with Various Diameters for Sensing Application

A single mode fiber (SMF28, 9/125µm) was unjacketed about 2cm long to expose its cladding part by using a jacket stripper. The exposed SMF was wiped with alcohol (ethyl alcohol) to ensure the debris was totally removed. Five SMFs with different diameters were prepared with maximum size of d=0.1250mm. Note that the 0.125mm diameter is the original size of SMF without experienced any etching process. To fabricate other sizes of fiber (smaller than 0.125mm), part of cladding area was etched via low cost mechanical polishing technique by using a P2000-grit sandpaper. The effect of cladding's diameter on the optical power response of our proposed sensor was investigated. The diameters were measured by using an optical microscope (Brand: Mitutoyo). Table 1 tabulates the list of polishing frequencies and their descriptions.

Table 1. List of polishing frequencies, *n* and their descriptions

Frequency of polishing, n	Description
0	• Requires no polishing • No part of cladding is etched. The diameter remains, d=0.125mm.
1	• Requires one time polishing on SMF • Part of cladding is etched.
2	• Requires two times polishing on SMF • Part of cladding is etched.
3	• Requires three times polishing on SMF • Part of cladding is etched.
4	• Requires four times polishing on SMF • Part of cladding is etched.

Deposition of Au/GO onto the SMF using Drop Casting Technique

Hybrid Au/GO films were deposited onto the SMF by using a drop casting technique [19]. Commercially available Au nanoparticles were used in this study (Brand: Sigma Aldrich). First, one droplet (volume=10µL) of Au nanoparticles in a form of aqueous solution was deposited onto the SMF. The application of one droplet of Au nanoparticles solution produced one layer of Au. This procedure was conducted inside the petri dish (refer Figure 1(a)). To ensure the Au nanoparticles were completely coated on the SMF, it was dried within four hours at room temperature. This step was repeated to produce two layers and three layers of Au nanoparticles. UV-Visible characterization was performed to determine the optimum layer of Au nanoparticles based on the maximum absorbance peak. Once the optimum layer of Au recognized, the experiment was continued by adding one to five layers of GO solution on top of Au layer to amplify the SPR signal because of the property of GO that has large specific surface area (Figure 1(b)) [20]. The Au/GO coated SMF was then left for 24 hours at room temperature. Figure 2 displays an illustration of Au/GO which had been coated on the substrate.

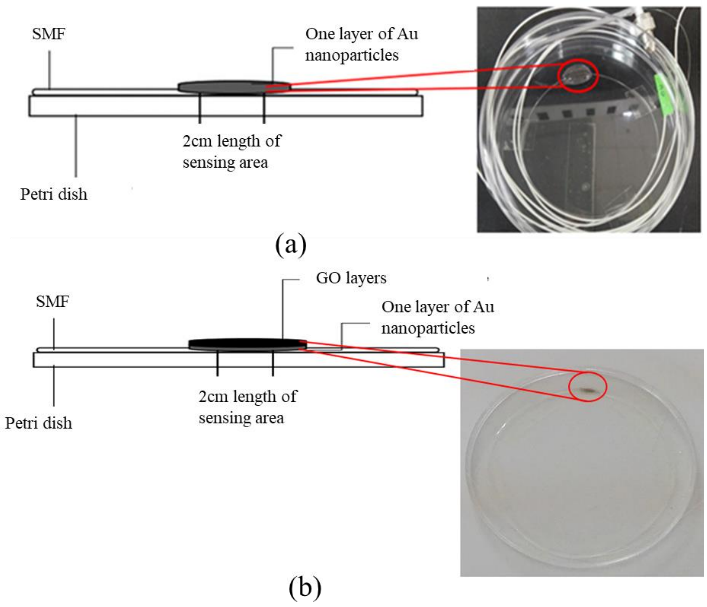

Figure 1. A drop-casting technique to deposit: (a) one layer of Au nanoparticles and (b) hybrid Au/GO layers; on SMF inside the petri dish

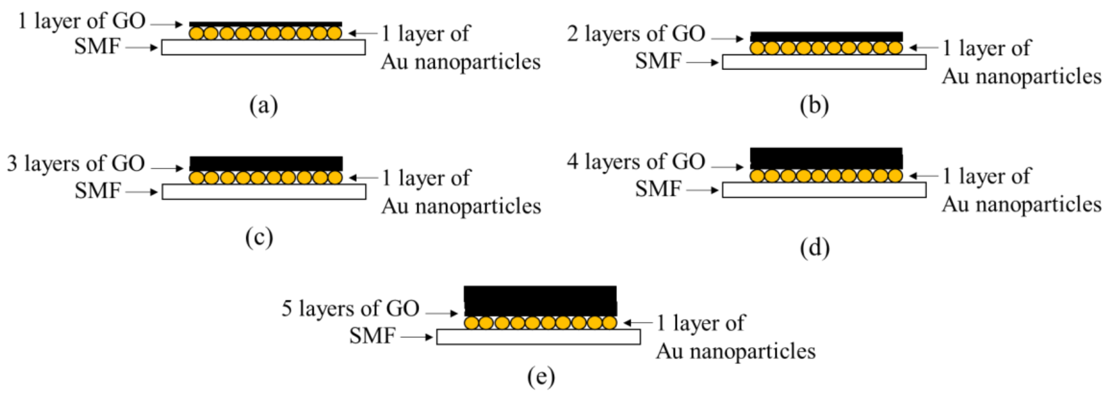

Figure 2. Deposition of Au/GO on the substrate using drop-casting technique by varied the number of GO layers, meanwhile the layer of Au nanoparticles was kept constant as one layer: (a) 1 layer of Au, 1 layer of GO (b) 1 layer of Au, 2 layers of GO (c) 1 layer of Au, 3 layers of GO (d) 1 layer of Au, 4 layers of GO and (e) 1 layer of Au, 5 layers of GO

Experimental Setup of Au/GO Bend SMF to Detect Spoiled Milk via Plasmonic Effect

Figure 3 shows an experimental setup of the Au/GO bend SMF sensor to study the quality of milk's freshness as it was exposed to the environment based on the real time optical output power reading. The SMF was cut about 65 cm in length where 2cm of it was unjacketed. The SMF was inserted into a petri dish by looped it about 8cm in diameter. The bending curve due to the fiber's looping is believed able to increase the evanescent waves to propagate around the bending area [21,22]. The looped SMF was fixed with the sticky tape to avoid its movement. The fibers with various cladding's diameters were coated with Au and GO to excite SPR and to enhance the sensing properties respectively. Before connected the pigtail SMF to the laser source (Brand: Noyes) and optical power meter (Brand: Noyes), both ends of pigtails were cleaned by using Neoclean Connector Cleaner to remove dusts and particles.

Full-cream milk (pasteurized) was purchased off-the-shelf. The Au/GO coated bend SMF was immersed into the 20µL of milk which had been exposed to the environment about 24 hours, 48 hours and 72 hours at room temperature. The effect of light excitation wavelength (λ=1310nm and λ=1550nm) on the optical power output, P_{output} with various exposure time duration was investigated. The reference values of the optical power output, P_A for λ_1=1310nm and λ_2=1550nm were set as 31.50dBm and 29.80dBm respectively. The optical power response was determined by calculating the percentage difference of P_{output} (ΔP%) between the P_{output} of the reference value (P_A) and P_{output} as the coated bend SMF was immersed into the milk's sample (P_B) as expressed in Eq. (1):

$$\%P = \frac{P_{(A)} - P_{(B)}}{P_{(A)}} x100\% \quad (1)$$

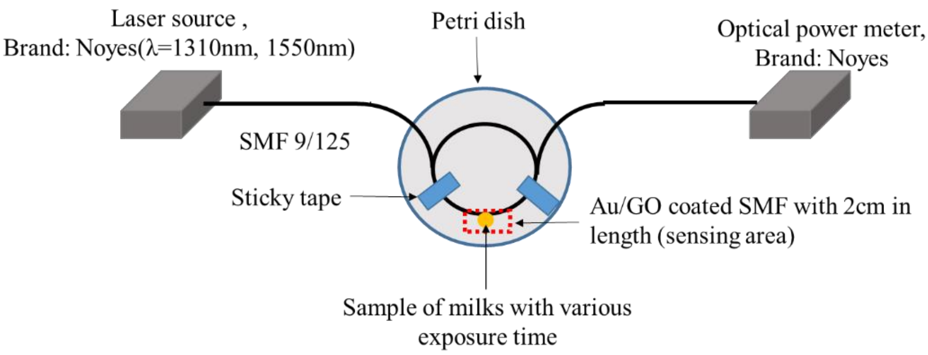

Figure 3. Experimental setup of Au/GO bend SMF sensor via LSPR effect to detect level of milk's spoilage

RESULTS AND DISCUSSIONS

Figure 4 displays the UV-Vis characterization of Au nanoparticles ranging from one to three layers. The Au nanoparticles were successfully coated on the substrate representing by the shallow peaks at λ=575nm. Note that the absorption wavelength for gold nanoparticles with d=50nm was within the range of 520nm-580nm [18]. Solutions of colloidal Au have a distinctive red colour, which arises from the tiny dimensions of the Au nanoparticles. At nanometre dimensions, the electron cloud can oscillate on the particle surface and absorb electromagnetic radiation at a particular energy. The increasing layer of Au nanoparticles resulting in the decrement of absorbance value. About 21% of plasmon absorption occurred as one layer of Au was deposited on the substrate. Values of absorbance decreased to 13.8% when light incident on both two and three layers of Au nanoparticles. Weak plasmon absorption happens maybe due to the presence of repulsive forces experienced by both nanoparticles as the additional layers of Au was drop-casted on the layer. According to this analysis, we found that one droplet of Au nanoparticles solution is sufficient enough to produce LSPR. Additional layers of Au will reduce the plasmonic effect due to the possibilities of nanoparticles' removable from the substrate as the drop casting method was performed. By considering this result, the potential of Au/GO bend SMF as sensing device was further investigated by maintaining one layer of Au and varying the thicknesses of GO from one to five layers.

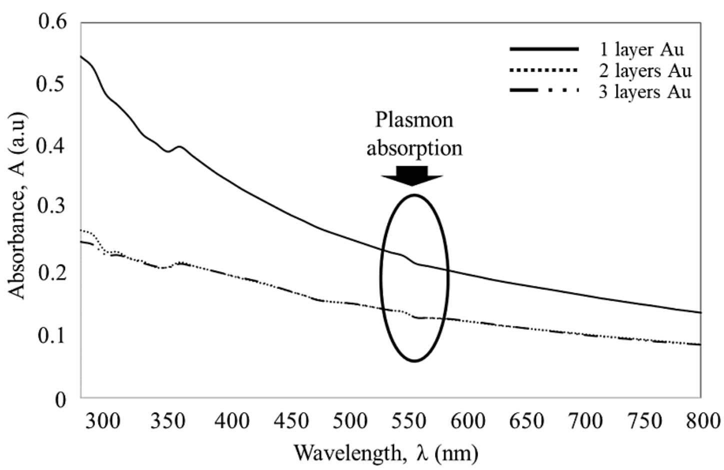

Figure 4. UV-Vis spectroscopy characterization of Au nanoparticles with various layers

Figure 5 displays the microscope images of SMF with various diameters. The frequency of polishing was increased from n=1 until n=4. About 0.40% of the cladding area was successfully etched as the SMF was polished with n=1 producing 0.1245mm of cladding's diameter (Figure 5(b)). Figure 5(c) shows the image of etched fiber as n=2 was applied on the SMF resulting in d=0.1235mm. The smallest diameter that can be achieved by using this technique was d=0.1215mm as n=4 as depicted in Figure 5(e). In general, the greater the frequency of polishing, *n* results the decreased of cladding's diameters with linear relationship as depicted in Figure 6. The advantages of this technique are due to its low cost and uncomplexed procedure. In contrary, the mechanical polishing technique are less repeatability and only small part of cladding about 3% of the cladding area can be removed which make it less significant to produce strong evanescent field.

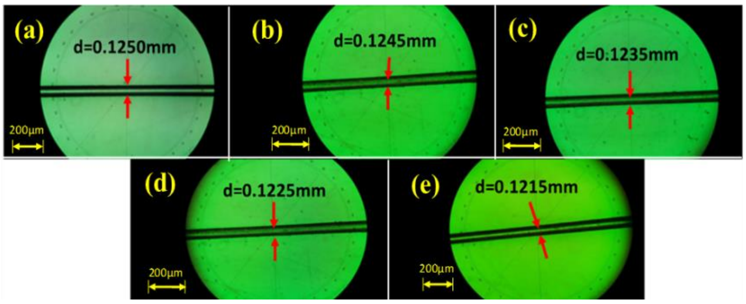

Figure 5. Microscope images of cladding with various diameters: (a) d=0.1250mm (no etching, n=0), (b) d=0.1245mm (n=1), (c) d=0.1235mm (n=2), (d) d=0.1225mm (n=3) and (e) d=0.1215mm (n=4)

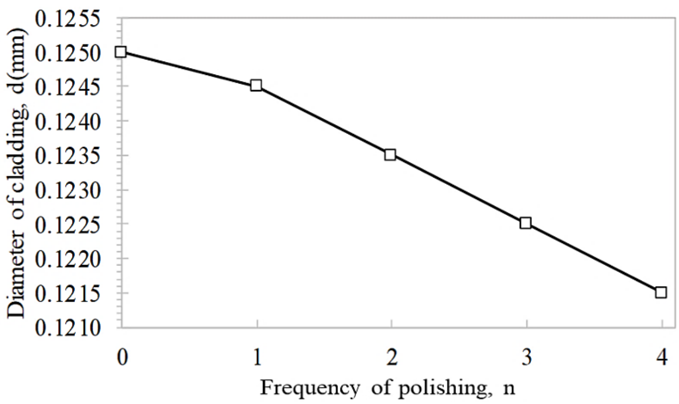

Figure 6. Inverse linear relationship between diameter of cladding,d and frequency of polishing, n

Figure 7 displays the values of optical output power as light source with λ=1310nm was transmitted through the bend SMF coated with one layer of Au nanoparticles and various layers of GO. In general, the sensor shows a good response as the diameter changed. Nonetheless, its selectivity performance became weaker with the increment of GO layers' thicknesses where the output power differences between three exposure time have become increasingly difficult to distinguish. The best sensitivity of the sensor was obviously obtained with the employment of one layer of GO as illustrated in Figure 7(a). The sensor's performance was evaluated based on the significant different values of the output power reading as the exposure time, GO thicknesses and cladding diameters varied. Greater value of ΔP indicates better sensor's performance due to its ability to detect larger output power difference. We found that the application of one layer GO exhibited outstanding sensing properties. Note that although GO has been proven able to enhance the sensitivity of the surface plasmon resonance (SPR) optical sensor, its thickness mainly influenced this sensing properties. Too thick of GO layer will weaken its sensitivity because the surface plasmon waves have been absorbed by the GO itself [18]. An excellent selectivity performance was obtained as the cladding's diameter was set at d=0.1250mm. Bear in mind that this diameter belongs to the original size of SMF without experience any polishing process.

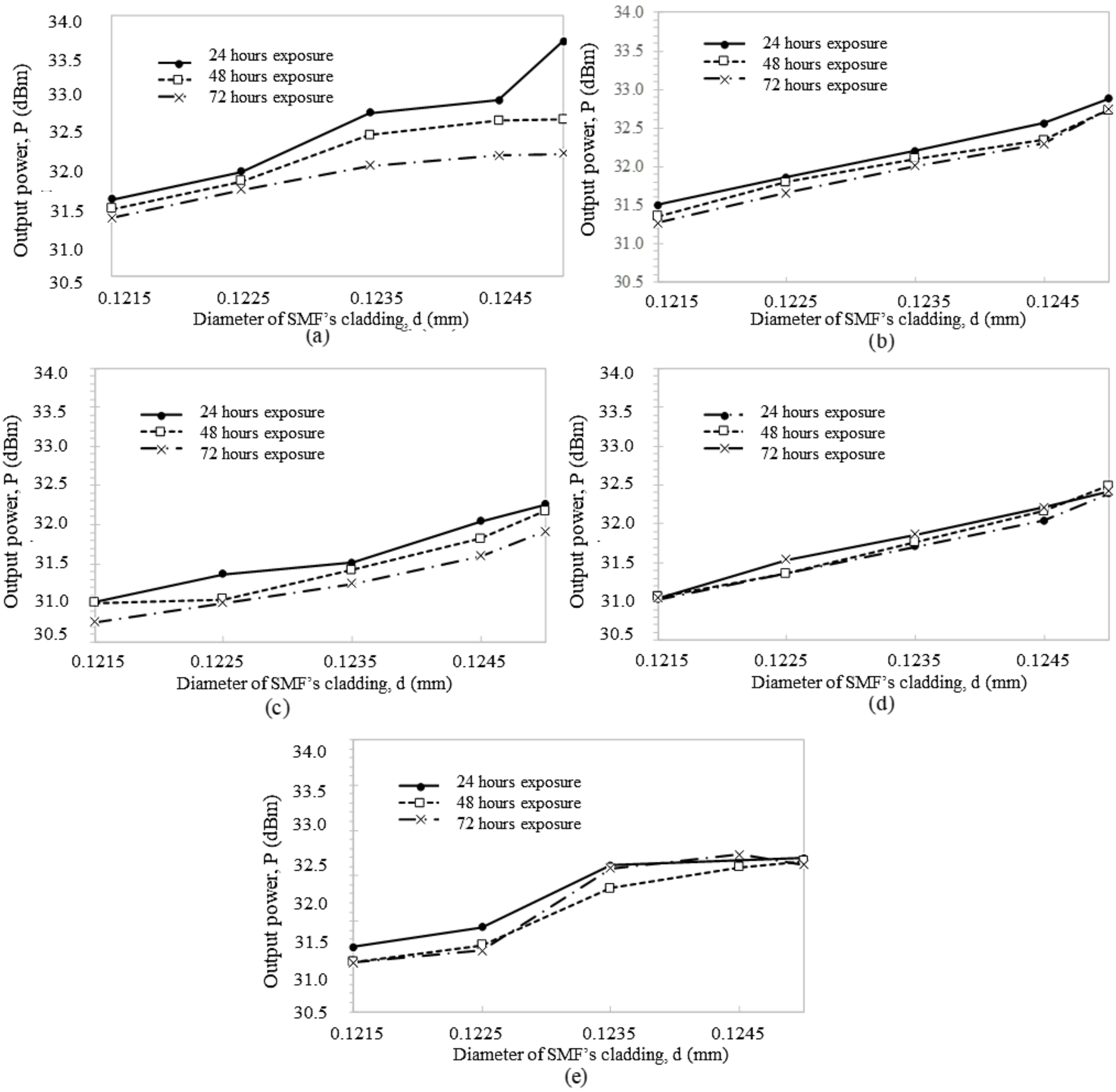

Figure 7. Various optical power output, P (dBm) using different size of cladding's diameters with light source, λ=1310nm as milk was exposed to the environment for 24 hours, 48 hours and 72 hours: (a) Au=1 layer, GO=1 layer, (b) Au=1 layer, GO=2 layers (c) Au=1 layer, GO=3 layers, (d) Au=1 layer, GO=4 layers and (e) Au=1 layer, GO=5 layers

Similar patterns were obtained as the wavelength's excitation changed from λ=1310nm to λ=1550nm as portrayed in Figure 8. However, in comparison between both excitation wavelengths, the λ=1550nm exhibits poor performance due to its less sensitivity and selectivity abilities. It is notewothy to mention that the occurrence of slightly inconsistent values of output power most probably due to the non-uniform structural condition of the etched fiber. Approximately, maximum output power was obtained when the exposure time was set at 24 hours. The value of output power became smaller as the exposure time increased to 48 and 72 hours respectively. This condition happened maybe because of the factor that most evanescent waves had been absorbed by the bacteria or microorganism in the spoiled milk resulted low output power [22].

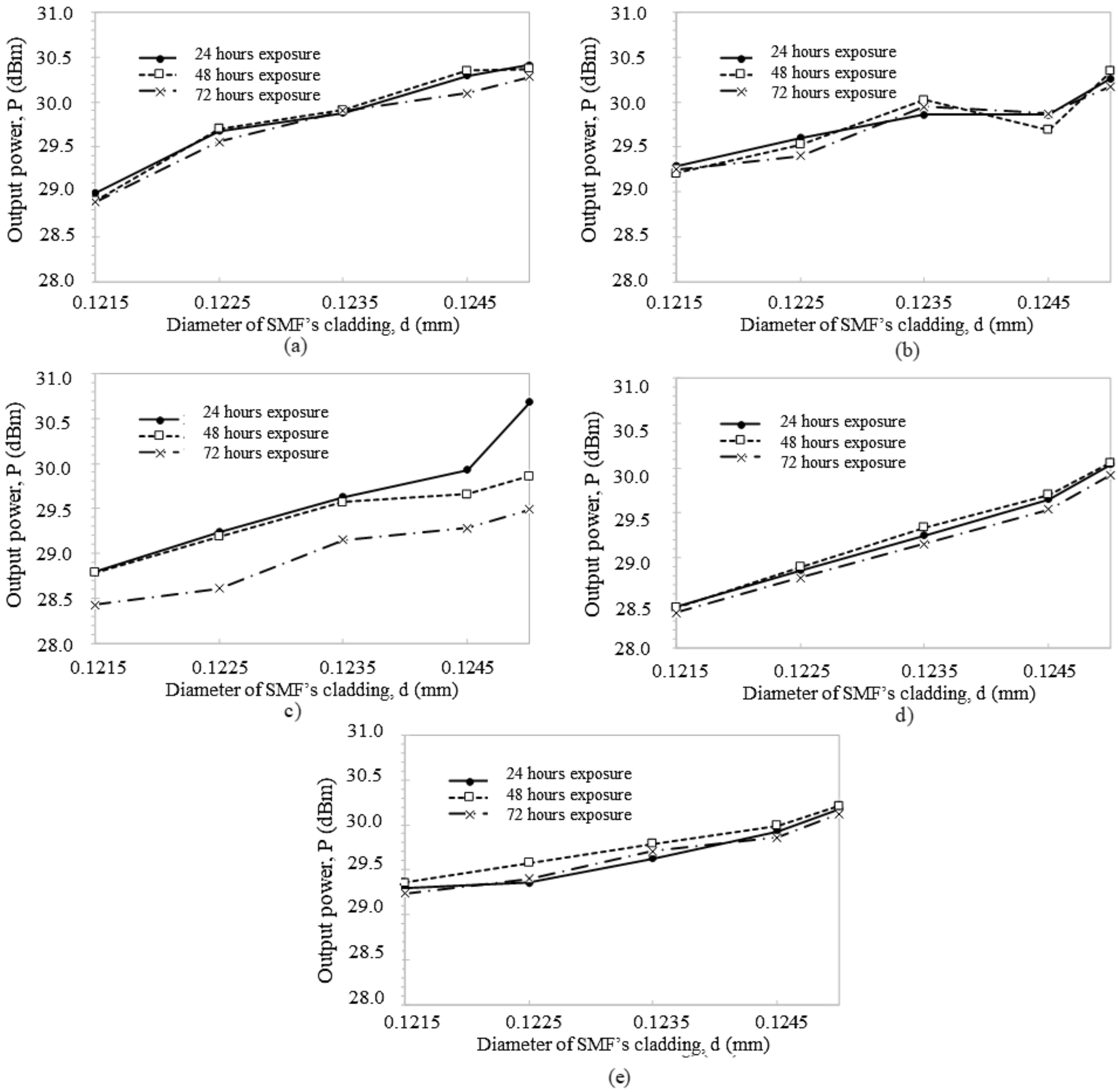

Figure 8. Various optical power output, P (dBm) using different size of cladding's diameters with light source, λ=1550nm as milk was exposed to the environment for 24 hours, 48 hours and 72 hour: (a) Au=1 layer, GO=1 layer, (b) Au=1 layer, GO=2 layers, (c) Au=1 layer, GO=3 layers, (d) Au=1 layer, GO=4 layers and (e) Au=1 layer, GO=5 layers

For accurate analyses, the optical power response based on the percentage differences of the optical powers (using Eq. (1)) for various sizes of cladding and numerous number of GO layers when λ=1310nm are studied as illustrated in Figure 9. Obviously, the greatest percentage difference was obtained using the unetched original SMF with d=0.125mm as shown in Figure 9(a) until Figure 9(e). By focusing at d=0.125mm, the sensor exhibited an impressive response when both Au nanoparticles and GO were set as one layer with maximum value of ΔP%=6.85% at 24 hours of exposure time (Figure 9(a)). As the exposure time increased to 48 hours and 72 hours, the values of ΔP% decreased to 3.5% and 2.0% respectively. The same patterns of ΔP% were resulted when the cladding's diameters varied between d=0.1215mm until 0.1245mm in which their values became smaller as the cladding sizes reduced. As two layers of GO coated onto the cladding, the values of ΔP% decreased indicate the less responsitivity of this sensor (Figure 9(b)). Note that the sensor's optical power response became less significant as Au/GO bend SMF consisted of two layers of GO was employed where the values of ΔP% remain constant at 48 and 72 hours. By considering this output, we conclude that the optimum performance of sensor can be developed by coating the original unetched SMF with one layer of Au nanoparticles (d=50nm) and one layer of GO. Figure 10 displays the optical power response analyses of Au/GO coated SMF with the employment of laser, λ=1550nm. The inconsistency of ΔP% was observed as number of GO layers increased (Figure 10(a) until Figure 10(e)) validates the unsuitability of light wavelength, λ=1550nm to be used as a laser source due to the instability factor.

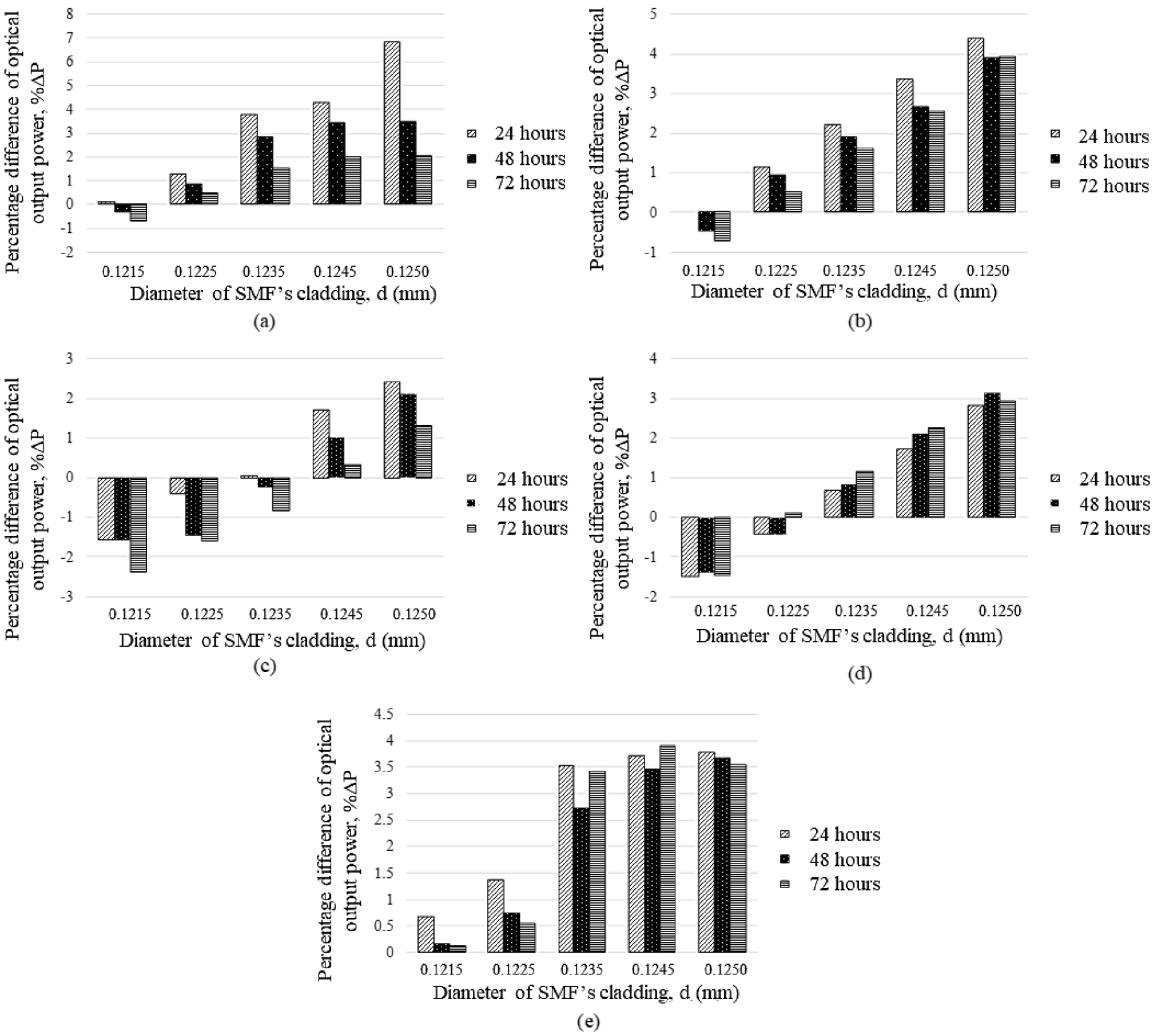

Figure 9. Percentage difference of optical power output, ΔP% as diameter of SMF's cladding increases using light source, λ=1310nm: (a) Au=1 layer, GO=1 layer, (b) Au=1 layer, GO=2 layers, (c) Au=1 layer, GO=3 layers , (d) Au=1 layer, GO=4 layers and (e) Au=1 layer, GO=5 layers

The sensor's optical power response is determined based the percentage of ΔP. We found that the ΔP% decreased as the milk's exposure time increased. It is noteworthy to mention that the working principle of optical sensor is based to the detection of refractive index of the surrounding medium. In this study, milk acts a surrounding medium for detection. The pH of a milk ranges from 6.4 to 6.8. This value changes over time. As milk goes sour due to longer exposure time, it becomes more acidic and the pH gets reduced. This occurs as bacteria in milk convert the sugar lactose into lactic acid [23]. The decrease in the pH value of the solution resulting in the increase in the refractive index of the solution [24]. This condition causes the changes in optical power output as the exposure time increased. This study also found that the mechanical polishing technique using sandpaper is not suitable to etch silica SMF due to many factors such as unrepeatability, non-uniform etched surface and fragility. This technique is more convenient for multimode fiber such as POF because of its larger size and made by plastic materials [25]. Previous works found that smaller diameter of cladding results better sensitivity of the sensor. It is noteworthy to highlight that their fabrication techniques are chemical etching and tapering [26,27]. Our results are contradicted with theirs due to the nonuniform surface of the etched SMF using sandpaper. On the bright side, the application of original SMF without experience any etching process incorporated with the fiber's bend structure exhibits a good sensing ability of the sensor to detect milk's spoilage conditions. The combination of localized plasmonic effect excited by Au nanoparticles resulting in strong scattering and extinction spectra, together with the signal enhancement by GO successfully produce an excellent sensitivity of the sensor.

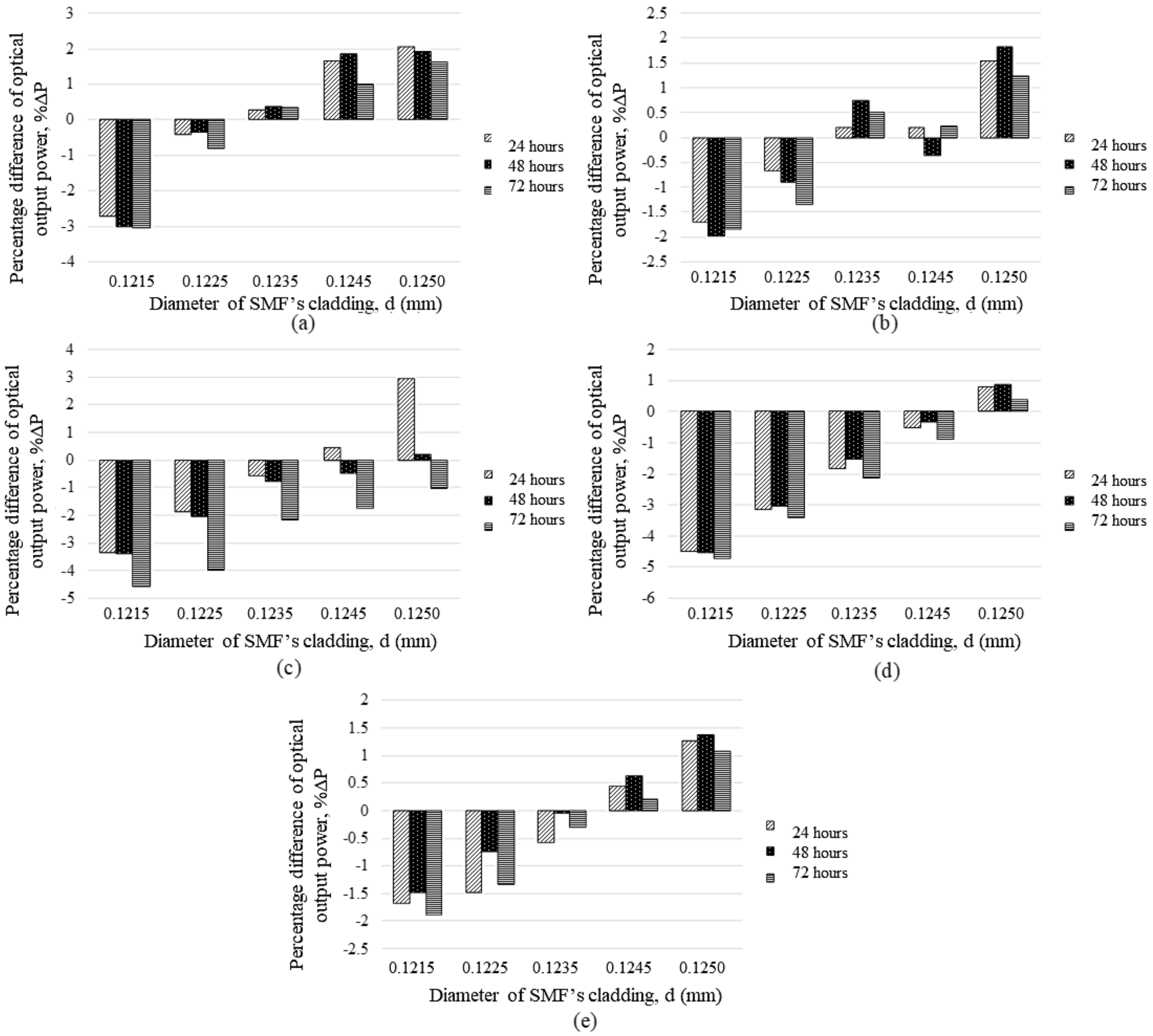

Figure 10. Percentage difference of optical power output, ΔP% as diameter of SMF's cladding increases using light source, λ=1550nm: (a) Au=1 layer, GO=1 layer, (b) Au=1 layer, GO=2 layers, (c) Au=1 layer, GO=3 layers, (d) Au=1 layer, GO=4 layers and (e) Au=1 layer, GO=5 layers

CONCLUSIONS

In conclusions, the physical structure of sensor affects its performance in detecting milk's spoilage condition. Few parts of sensor's had been optimized to amplify its sensitivity such as number of material's layers, LSPR effect, light excitation wavelength and fiber's structure. By excites laser with λ=1310nm and bends the SMF in a form of loop structure with diameter of 8cm, the usage of one layer for both Au nanoparticles (diameter=50nm) and GO exhibits excellent sensitivity with maximum optical power response of ΔP%=6.85%. This real time monitoring detection of milk spoilage condition shows an impressive potential of Au/GO bend SMF sensor for food safety application.

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