

# Detection of Milk Spoilage Using Au D-Shaped Fiber Sensors Based on Localized Surface Plasmon Resonance

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## Abstract

The effectiveness of gold nanoparticle coated D-shaped fiber sensor by utilizing localized surface plasmon resonance (LSPR) effect for detection of spoiled milk is studied. Milk is a highly perishable food product that is susceptible to spoilage due to microbial activity and improper storage conditions. Conventional methods for milk spoilage detection are often time-consuming, subjective, and require laboratory facilities. Therefore, this study investigates the feasibility of using an optical fiber-based sensor for the detection of milk spoilage through changes in optical response. An Au-coated D-shaped optical fiber sensor was developed based on the localized surface plasmon resonance (LSPR) effect to enhance sensitivity to refractive index variations in milk samples. Different types of milk, namely fresh milk, low-fat milk, full cream milk, and condensed milk, were prepared and allowed to undergo natural spoilage at room temperature over exposure periods of 0, 24, 48, and 72 hours. A thin gold (Au) layer was deposited using a sputter coater, and UV-Vis spectroscopy was employed to confirm the plasmonic characteristics of the coating. The sensor's surface morphology was examined using field emission scanning electron microscopy (FESEM). Optical measurements were carried out using an Optical Spectrum Analyzer (OSA) and an Optical Power Meter (OPM) to monitor spectral changes and variations in transmitted optical power. The results demonstrated clear time-dependent changes in both spectral response and optical power for all milk samples, with more pronounced variations observed in full cream and condensed milk. These changes indicate sensitivity to compositional and refractive index variations associated with spoilage.

## 1. Introduction

Milk is a widely consumed beverage across the world due to its high nutritional value, including calcium, proteins, and essential vitamins. Despite these benefits, milk is highly perishable and vulnerable to microbial contamination if it is not handled or stored under appropriate conditions. Milk naturally contains bacteria, and when exposed to unsuitable temperatures or prolonged storage, these microorganisms can multiply rapidly and accelerate the spoilage process. Spoilage causes undesirable changes such as sour taste, unpleasant odour, texture alteration, nutrient degradation, and reduced consumer acceptability. In addition, the consumption of spoiled milk may pose health risks, making effective freshness monitoring an important aspect of food safety and quality control in the dairy industry [1].

Traditional techniques for detecting milk spoilage include sensory evaluation, pH measurement, and microbiological analysis. Although these methods are widely used, they are often time-consuming, require laboratory facilities, and depend on trained personnel. Sensory evaluation is subjective, while microbiological tests may not provide immediate results. As a result, these approaches are not suitable for rapid or on-site quality assessment, especially in large-scale dairy operations or remote locations. The limitations of conventional methods have encouraged the development of alternative technologies that can offer faster, more reliable, and real-time monitoring of milk quality [2].

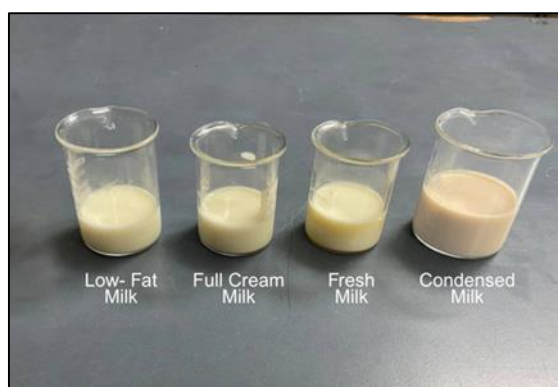
Recent advancements in optical sensing technology have introduced optical fiber-based sensors as a promising tool for food quality monitoring. These sensors operate by detecting changes in the refractive index of a surrounding medium, which varies as milk undergoes chemical and biological changes during spoilage [3]. In this study, a D-shaped optical fiber coated with a thin layer of gold (Au) is used to enable localized surface plasmon resonance (LSPR). The flat surface of the fiber enhances the interaction between guided light and the milk sample. As spoilage progresses, variations in milk composition influence this interaction, resulting in measurable changes in transmitted optical power or spectral response. This approach offers a sensitive, rapid, and non-invasive method for detecting milk spoilage, supporting improved quality assurance and consumer safety [4].

## 2. Methodology

This section outlines the methodology used to evaluate the performance of an Au-coated D-shaped optical fiber sensor for milk spoilage detection based on the localized surface plasmon resonance principle. The study involved the preparation of milk samples, preparation of Au-coated D-shaped fiber sensor, and optical measurements using a laser source together with an optical spectrum analyzer (OSA) and optical power meter (OPM). The D-shaped optical fiber was coated with a thin gold layer using a sputter coater to enhance light-matter interaction at the sensor surface. Milk samples were stored under controlled conditions to allow spoilage to occur over time. As the milk spoiled, changes in its optical properties affected the LSPR response of the sensor, resulting in variations in the transmitted optical signal. These changes were recorded and analysed to determine the effectiveness of the sensor in detecting different levels of milk spoilage.

### 2.1 Sample Preparation

Four types of milk were used to observe their spoilage progression over several days. The milk types included full cream, low-fat, fresh milk and condensed milk, all of which were purchased from a local supermarket. In the laboratory, approximately 20 mL of each milk type was dispensed into separate clean, beakers and clearly labelled. These beakers were stored at room temperature with no coverings to allow natural spoilage through exposure as shows in Fig. 1. For each measurement, a small volume of 20  $\mu$ L of milk was carefully extracted using a syringe and dropped into a sterile Petri dish containing the Au-coated region of the D-shaped optical fiber sensor. The same milk batch was used repeatedly over several days, with measurements taken at 24, 48, and 72 hours to monitor the progression of spoilage.

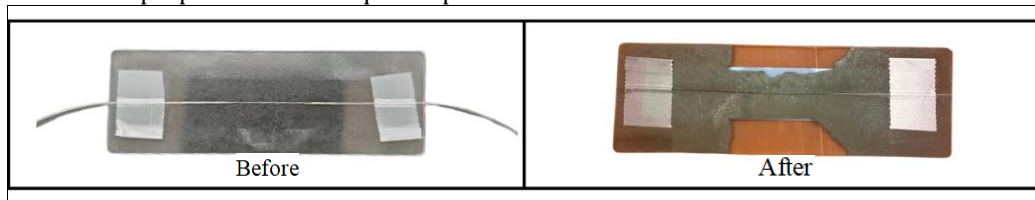


**Fig. 1** Milk Samples

### 2.2 Preparation of Au Coated D-Shaped Fiber Sensor

D-shaped fiber sensor was coated with gold (Au) by using sputter coater. Prior to the coating process, the optical fiber was carefully cleaned using alcohol wipes to eliminate dust and any surface impurities. A sputter coating technique was then applied to deposit a thin and uniform gold layer onto the sensing region of the fiber. The coating parameters were adjusted to obtain an estimated gold thickness of 50 nm. The condition of the D-shaped optical fiber before and after the gold coating process is illustrated in Figure 2. Before coating, the polished surface of the fiber appeared smooth and exposed, allowing direct interaction with the surrounding medium but without

plasmonic activity. After the sputter coating process, a thin gold layer was successfully formed on the sensing region, resulting in a visibly darker and more uniform surface. This gold-coated layer is essential for the excitation of localized surface plasmon resonance (LSPR), as it enhances the interaction between the guided light and the external medium. The comparison shown in Fig. 2 confirms that the coating process was successfully carried out and that the fiber was prepared for subsequent optical measurements.



**Fig. 2** Before and After Au Coating on D-Shaped Optical Fiber Sensor

## 2.3 Experimental Setup

The experimental setup for this study is shown in Fig. 3 and was designed to evaluate the performance of the Au-coated D-shaped optical fiber sensor in detecting milk spoilage. The setup consisted of a 1550 nm laser source, the D-shaped fiber sensor, an Optical Spectrum Analyzer (OSA), and an Optical Power Meter (OPM) to monitor changes in light transmission over time. During the experiment, light from the laser source was carefully aligned and launched into one end of the D-shaped fiber, while the output end was connected to either the OSA or OPM for measurement. The flat region of the fiber, coated with a thin layer of gold, served as the sensing area and was exposed to a small volume of milk (20  $\mu$ L) placed in a Petri dish. As the milk underwent spoilage, the biochemical changes in the sample influenced the refractive index near the gold-coated surface, which in turn affected the localized surface plasmon resonance (LSPR) response. These variations were recorded in real time using the OSA and OPM, allowing for continuous monitoring of the sensor's response to milk spoilage. This setup ensured precise control of the light input and reliable measurement of the sensor's optical response throughout the experiment.



**Fig. 3** Experimental Setup

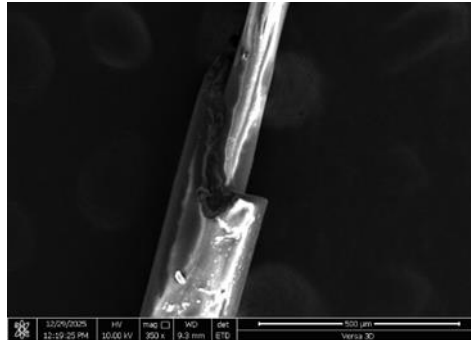
## 3. Results and Discussion

This section presents the results and discussion of detection milk spoilage using an Au-coated D-shaped optical fiber sensor based on the localized surface plasmon resonance (LSPR) effect. The sensor performance was assessed by observing changes in its optical response as different types of milk experienced spoilage over time. To characterize the sensor, ultraviolet-visible (UV-Vis) spectroscopy was first employed to verify the plasmonic absorption behaviour of the gold-coated layer. In addition, field emission scanning electron microscopy (FESEM) was used to study the surface structure and coating quality of the Au-coated D-shaped optical fiber. The sensing behaviour was further evaluated using an Optical Spectrum Analyzer (OSA) to monitor spectral changes and an Optical Power Meter (OPM) to record variations in transmitted optical power. Experiments were conducted on four different milk samples at exposure durations of 0 h, 24 h, 48 h, and 72 h. These measurements were performed to evaluate the sensitivity and overall effectiveness of the proposed sensor in detecting milk spoilage over time.

### 3.1 FESEM Analysis

FESEM analysis was conducted to examine the surface structure of the D-shaped optical fiber sensor used in this study. The typical D-shaped profile, in which one side of the fiber has been trimmed to reveal the core region, was

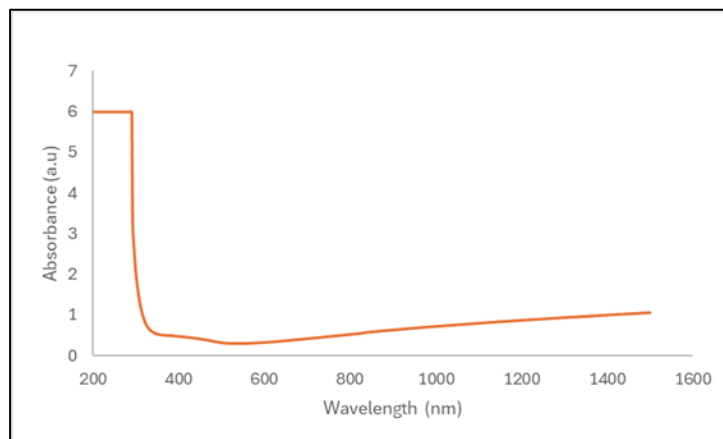
clearly visible in the image shown in Fig. 4. This discovery confirmed the D-shaped fiber's potential for sensing applications by confirming its given shape. The fiber condition was suitable for coating and experimental use, as seen by the exposed flat surface's smooth, well-defined appearance and lack of obvious surface flaws. To have a high sensing response, the interaction between the guided optical field and the surrounding medium must be improved, which is why the flat region is important. Overall, the FESEM results verified that the D-shaped fiber structure was suitable for sensing based on localized surface plasmon resonance.



**Fig. 4** FESEM of D-shaped Fiber

### 3.2 Absorbance Analysis using UV-Vis

When evaluating a sensor's applicability for environmental and monitoring applications, optical characterisation is a very important step. In this study, the optical response of the Au-coated D-shaped optical fiber sensor was examined using UV-Visible (UV-Vis) spectroscopy. A strong absorption peak is shown in the visible wavelength range, about between 520 and 580 nm, in Fig. 5, which displays the coated fiber's UV-Vis absorbance spectrum. This wavelength range is typical of gold's localized surface plasmon resonance (LSPR) phenomena. The appearance of this absorption peak shows that the gold layer has been successfully deposited on the surface of the D-shaped optical fiber. LSPR takes place because of the resonance of free electrons in the gold layer when it interacts with the incident light. The incident light leads to the intensification of the optical field at the sensor surface, which is a major aspect of the sensor using the concept of plasmons. Hence, the UV-Vis spectral analysis shows that the Au-coated D-shaped optical fiber has the desired optical properties to be used for sensing and monitoring experiments.



**Fig. 5** UV-Vis absorbance spectrum of the Au-coated D-shaped optical fiber

### 3.3 Optical Spectrum Response Analysis

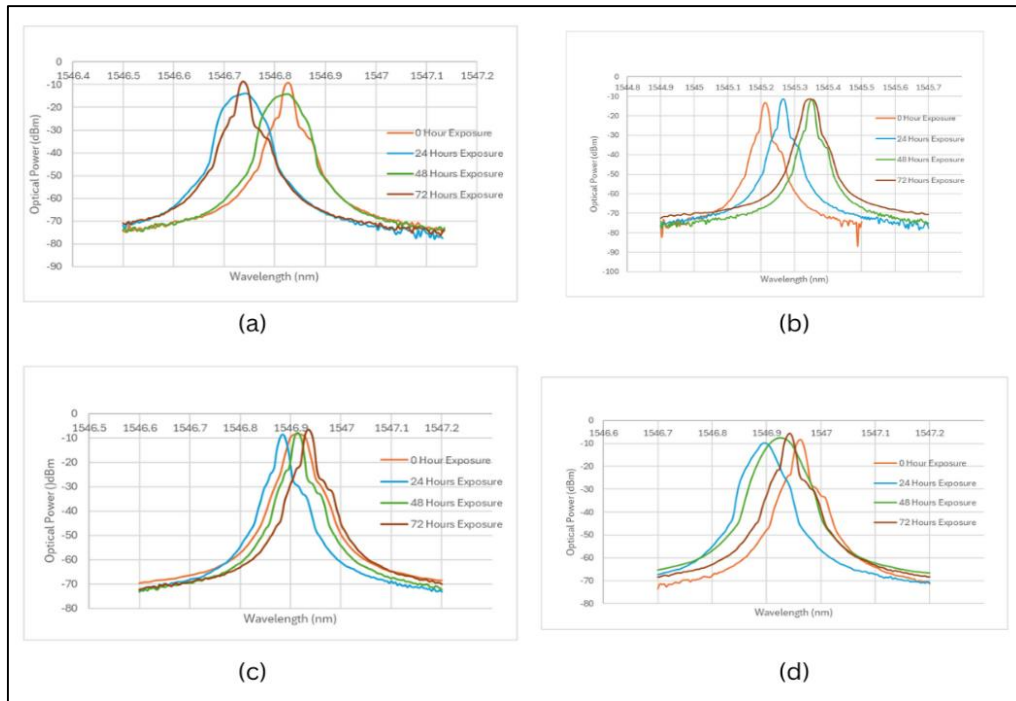
The Optical Spectrum Analyzer (OSA) is used in this study to observe changes in wavelength and optical power as milk samples undergo spoilage over time. Variations in the OSA spectra indicate changes in light transmission caused by alterations in the refractive index near the gold-coated D-shaped fiber surface and results as Fig. 6 are obtained. These changes are closely related to biochemical processes occurring in milk during spoilage, such as bacterial growth, fermentation, protein degradation, and pH reduction.

For low-fat milk, the OSA spectra show a gradual increase in transmission loss as exposure time increases from 0 to 72 hours. Fresh samples exhibit relatively shallow loss dips, indicating stable optical properties. As spoilage progresses, the resonance dip becomes deeper and broader, suggesting enhanced localized surface

plasmon resonance (LSPR) interaction due to changes in milk composition. This trend reflects increasing refractive index variation caused by microbial activity and metabolite accumulation.

A similar pattern is observed for full cream and fresh milk samples, but with more pronounced loss depths at longer exposure times. Full cream milk shows stronger attenuation due to its higher fat content, which enhances evanescent field interaction at the sensing region. Fresh milk also demonstrates clear time-dependent spectral changes, confirming the sensor's sensitivity to spoilage development.

For condensed milk, noticeable resonance peak shifts and intensity changes are recorded across exposure times. Its high sugar content and viscosity strongly influence plasmonic interaction, resulting in distinct spectral behaviour compared to other milk types. Overall, the OSA results confirm that the sensor effectively tracks spoilage progression through measurable spectral changes.



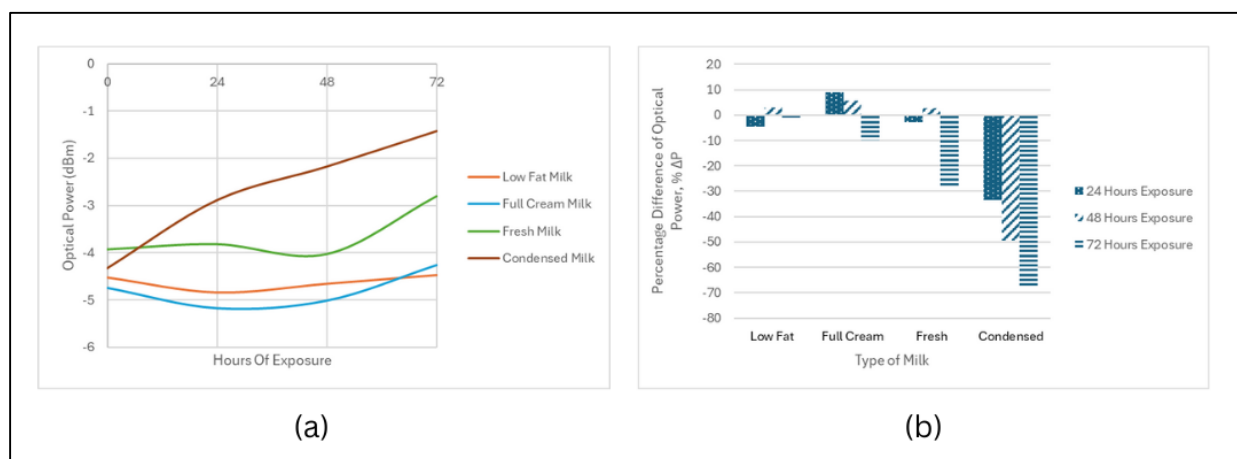
**Fig. 6** Various optical spectrum responses as milk were exposed to the environment for 24, 48 and 72 hours:

(a) Low Fat Milk, (b) Full Cream Milk, (c) Fresh Milk, (d) Condensed Milk

### 3.4 Optical Power Output Analysis

Although the optical spectrum analyzer (OSA) and optical power meter (OPM) are used in the same setup, they measure different light responses, which explains the differences seen in some trends, particularly for condensed milk. The OSA measures wavelength-dependent transmission, where deeper resonance dips indicate stronger localized surface plasmon resonance (LSPR) interaction caused by refractive index changes near the gold-coated fiber surface. As milk spoils, changes such as bacterial growth and chemical breakdown modify the surrounding optical environment, leading to increased spectral loss.

In contrast, the OPM measures the total transmitted optical power over a range of wavelengths. Therefore, stronger resonance dips do not always result in lower overall power. For condensed milk, changes in composition during spoilage can redistribute optical energy, causing an increase in total power. The percentage difference ( $\Delta P\%$ ) reflects deviation from the initial state rather than contradicting this trend.



**Fig. 7** Optical Power Analysis of Different Milk Types Over Time: (a) Various optical power output for different milk samples at 24, 48, and 72 hours of exposure, (b) Percentage difference in optical output power,  $\Delta P\%$  for different milk samples at 24, 48, and 72 hours of exposure.

#### 4. Conclusion

This study was conducted to evaluate the capability of an optical fiber-based sensing method for detecting milk spoilage through changes in optical output power and refractive index behaviour. Three main objectives were set at the beginning of the project, and the experimental results confirmed that all objectives were successfully achieved. The first objective, which focused on developing an optical fiber sensing system for monitoring milk quality over time, was accomplished using a D-shaped optical fiber sensor together with a light source and an optical spectrum analyzer (OSA). The system produced stable and consistent output power readings, indicating reliable performance. The second objective involved analysing the optical response of different milk types at exposure times of 24, 48, and 72 hours. The results showed clear time-dependent changes, with full cream and condensed milk exhibiting larger variations compared to low-fat and fresh milk due to differences in composition. The third objective was to evaluate optical output power variation and percentage difference ( $\Delta P\%$ ) as spoilage indicators. The observed trends confirmed that this sensing approach is sensitive to spoilage-related changes and suitable for milk quality monitoring.

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#### Conflict of Interest

Authors declare that there is no conflict of interest regarding the publication of the paper.

#### Author Contribution

The authors confirm contribution to the paper as follows: **study conception and design:** Nur Farzana Nazurah Rosli, Noor Azura Awang; **data collection:** Nur Farzana Nazurah Rosli; **analysis and interpretation of results:** Nur Farzana Nazurah Rosli; **draft manuscript preparation:** Nur Farzana Nazurah Rosli, Noor Azura Awang. All authors reviewed the results and approved the final version of the manuscript.

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