

# Graphene - Coated Surface Plasmon Resonance (SPR) Sensor for Detection of Preservatives in Milk : A Theoretical Investigation

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## ABSTRACT

Milk is an essential dietary staple for human sustenance. However, the presence of various contaminants, notably preservatives, within milk can potentially pose significant health risks and impact overall well-being. Consequently, the meticulous surveillance of pollutants and impurities, particularly preservatives, in natural and everyday food products has emerged as a pivotal endeavor to ensure food safety and enhance its overall quality. In this research study, the Surface Plasmon Resonance (SPR) technique is strategically employed for a theoretical exploration of preservatives, namely hydrogen peroxide, formaldehyde, and sodium carbonate, within milk. The investigation encompasses varying concentrations of these preservatives, ranging from 0% to 14.3%. Concurrently, the refractive indices span from 1.34550 to 1.35093, representing the concentration between the minimum (0%) and maximum (14.286%) concentrations of hydrogen peroxide, formaldehyde, and sodium carbonate. To authenticate the efficacy of the proposed sensor, an extensive assessment of performance parameters is conducted. These parameters encompass Sensitivity, Full Width at Half Maximum (FWHM), Figure of Merit (FOM), and Detection Accuracy (DA), with calculations performed for each specific case.

Keywords: Surface Plasmon Resonance, Copper, Barium Titanate (BaTiO<sub>3</sub>), Graphene, Sensitivity.

## I. INTRODUCTION

Milk serves as a rich reservoir of essential nutrients such as calcium, protein, and vitamin D, making it a

cornerstone of a well-rounded dietary regimen for numerous individuals. Nonetheless, there exists a subset of the population who opt to abstain from its consumption, often influenced by diverse factors and

personal preferences[1-2]. Milk and milk-derived products originate from a spectrum of animal sources, encompassing cattle, sheep, camels, goats, and various others. Alternatively, for those seeking dairy alternatives, a plethora of options is available, including soy milk, almond milk, flax milk, coconut milk, and hemp milk. These alternatives cater to varying dietary requirements and preferences, offering a diverse range of choices beyond conventional dairy products. In essence, milk stands as a robust nutritional resource, while the diverse origins and alternatives in the realm of dairy products ensure dietary inclusivity and flexibility [3-5]. Different types of pollutants and impurities, such as preservatives that exist in milk may cause serious health issues, and affect the body. Preservatives are commonly employed within the dairy industry to address the inherent challenge of the short shelf life of raw milk[11-13]. These substances are introduced into milk and dairy products to prolong their viability and prevent spoilage. Their primary function is to inhibit the growth of microorganisms, including bacteria and fungi, which can lead to rapid deterioration. The use of preservatives in the dairy sector offers several notable advantages. Firstly, it significantly extends the shelf life of dairy products, thereby reducing waste and increasing product availability. Secondly, it ensures the maintenance of product quality by preventing undesirable changes in flavor, odor, and texture over time. Moreover, the incorporation of preservatives enhances the safety of dairy products by mitigating the risk of contamination, which could potentially lead to foodborne illnesses [14-16]. Lastly, it promotes convenience by facilitating the distribution of dairy products to a broader consumer base. However, it is essential to consider the potential effects on human health [17-19]. While preservatives are generally regarded as safe when used within recommended limits, individuals with sensitivities or allergies to certain additives should exercise caution. Additionally, the choice between natural and artificial

preservatives can impact the overall health implications of consuming dairy products. Natural preservatives are generally perceived as a healthier alternative to their synthetic counterparts. Monitoring pollutants and impurities, such as preservatives, in natural and everyday foods has been a key focus to assure safety and thus boost food quality [20-24]. Preservatives in milk can be detected using various technologies, one of which is surface plasmon resonance. The Surface Plasmon Resonance (SPR) phenomenon represents a cutting-edge and highly sensitive technology designed for the precise detection of variations in the refractive index of biological or chemical analytes upon direct interaction with the metal layer of the sensor [25-26]. Presently, scientific inquiry revolves around the utilization of the Kretschmann configuration, employing an angular interrogation approach, as the prevailing theoretical framework for SPR investigations [27]. In SPR sensors, plasmonic materials, notably metals such as Gold (Au) and Silver (Ag), have historically dominated sensor construction. However, it has been demonstrated that both metals exhibit suboptimal adhesion to prisms. Consequently, a strategic approach has been adopted, involving the application of thin coatings comprising Chromium (Cr), Teflon, and indium phosphate, with the aim of enhancing adhesion, sensitivity, and various other critical properties associated with SPR biosensors. While Silver remains a popular choice, Copper (Cu) emerges as a cost-effective alternative, albeit one that has historically received less attention as a plasmonic material due to its tendency to oxidize rapidly. Nevertheless, a recent study by Singh et al. [28] proposes that the judicious application of oxide coatings may mitigate the oxidation concern associated with Copper. Notably, the paramount attribute sought in SPR technology is sensitivity, with a paramount emphasis on achieving a high degree of it. The addition of two-dimensional (2D) material to traditional sensors can increase their sensitivity [29]. Graphene serves as a Biomolecular Recognition

Element (BRE) layer in Surface Plasmon Resonance (SPR) sensors for several compelling reasons: Graphene exhibits exceptional electrical conductivity and a substantial surface area, rendering it exquisitely responsive to even minute changes in the refractive index caused by biomolecule binding. This heightened sensitivity allows for precise detection, greatly amplifying the sensor's responsiveness [30]. Graphene's biocompatibility is a pivotal attribute, enabling its application in biological sensing scenarios without eliciting adverse reactions with biomolecules. This quality is vital in SPR sensors tailored for interactions with biological samples, ensuring the accuracy and safety of detection processes [31]. Graphene's expansive surface area facilitates the immobilization of a substantial quantity of biomolecules or receptors. This feature augments the sensor's capability to capture target analytes, fostering specificity and selectivity in detection processes [32]. The sensitivity of graphene-based SPR sensors can be finely tuned by manipulating the incident angle. This adaptability is advantageous for optimizing sensor performance across diverse applications and analyte concentrations, offering versatility in sensing tasks [33]. Graphene has gained widespread recognition as a detection layer in SPR sensor implementations due to its exceptional properties, encompassing sensitivity and biocompatibility. It is commonly integrated with metals like gold (Au) in sensor construction, underscoring its prevalence in SPR sensor technology. In summary, graphene's amalgamation of heightened sensitivity, biocompatibility, functionalization capacity, and tunability renders it a valuable choice as the BRE layer in SPR sensors. This is especially pertinent in applications geared towards the detection of complex analytes, notably biomolecules.

In this study, we have suggested a sensor based on SPR for detecting preservatives in milk. The reflectance curve, resonance angle, and corresponding sensitivity are analyzed theoretically by using the Fresnel equation and transfer matrix method using MATLAB software environment.

## II. METHODOLOGY AND SIMULATION STUDY

### 2.1 Theoretical Modelling and SPR Sensor Configuration

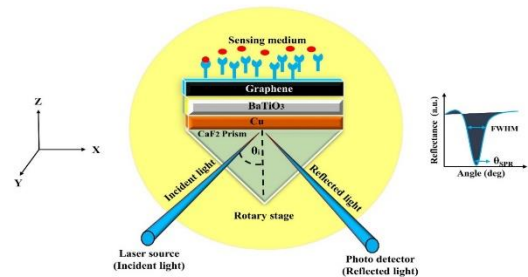


Fig.1 Proposed SPR Sensor Structure

The proposed Surface Plasmon Resonance (SPR) sensor adopts a Kretschmann configuration, featuring a composition that includes a CaF<sub>2</sub> prism, Copper (Cu), Barium Titanate (BaTiO<sub>3</sub>), Graphene, and a sensing medium, as elucidated in Figure 1. The Kretschmann configuration structure is founded on the principle of prism coupling, employing an angular interrogation approach as its operational mechanism. Within this methodology, the analysis is focused on the reflectance (R) concerning the incident angle, maintaining a fixed wavelength of 633 nm. To acquire the empirical data related to the performance parameters of the described SPR sensor, several essential components are indispensable. These components encompass a monochromatic laser source, specifically a He-Ne laser, along with the inclusion of a polarizer. Furthermore, the setup incorporates a multilayer stacking configuration that integrates a CaF<sub>2</sub> glass prism. The system also features a photodetector or charge-coupled camera (CCD) and a computer screen, as illustrated in Figure 1. For the prism coupling mode investigation, a Cadmium Fluoride (CaF<sub>2</sub>) glass prism with a low refractive index (RI) is employed. It assumes the crucial role of a light coupler, facilitating the determination of the proposed SPR sensor's shift in resonance angle and angular sensitivity.

The refractive index of prism CaF<sub>2</sub>[34] can be calculated by using the dispersion formula

$$n_{CaF_2} = \left( 1 + \frac{0.5675888\lambda^2}{\lambda^2 - 0.050263605} + \frac{0.4710914\lambda^2}{\lambda^2 - 0.1003909} + \frac{3.8484723\lambda^2}{\lambda^2 - 34.649040} \right)^{1/2} \tag{1}$$

Here, the incident wavelength ( $\lambda$ ) of the laser (633 nm) has been employed in this study. The optimized RI of the CaF<sub>2</sub> glass prism is 1.4329 at 633 nm wavelength.

The complex refractive index of Cu is calculated by the Drude model [35]

$$n^2 = 1 - \frac{\lambda^2 \lambda_c}{\lambda_p^2 (\lambda_c + i\lambda)} \tag{2}$$

For Cu, Plasma Wavelengths ( $\lambda_p$ ) =  $1.3617 \times 10^{-7}m$  and Collision Wavelength( $\lambda_c$ ) =  $4.0853 \times 10^{-7}m$  respectively.

The optimized thickness and RI of various materials at 633 nm wavelength have been tabulated in Table 1. The RI of sensing medium lies between 1.34550 and 1.35093.

Table 1.

Layer Structure	Material Used	Optimized Thickness	RI at 633nm	Reference
Layer-01	CaF <sub>2</sub> Prism	100.00nm	1.4329	[31]
Layer-02	Copper (Cu)	55.00nm	0.5840+i3.3466	[32]
Layer-03	BaTiO <sub>3</sub>	05.00nm	2.4043	[37]
Layer-04	Graphene	00.34nm	3.0+i1.1490	[24]

### 1.2 Mathematical analysis

The Surface Plasmon Resonance (SPR) sensor is an optical device that relies on a multi-layer thin-film configuration, featuring key optical parameters such as reflectance and transmittance. These critical parameters are determinable through the utilization of three distinct mathematical methodologies: The Transfer Matrix Method (TMM), the Field Tracing Technique, and the Resultant Wave Method. The current study predominantly employs the Transfer Matrix Method within a multi-layered framework. Notably, the technical application of the Transfer Matrix Method is exceptionally advantageous due to its exceptional precision and simplicity when contrasted with alternative techniques. By applying the boundary condition, the tangential field at  $Z=Z_1=0$  are presented in terms of tangential field at  $Z=Z_{N-1}=0$  as follows.

$$\begin{bmatrix} H_1 \\ P_1 \end{bmatrix} = A_{ij} \begin{bmatrix} H_{N-1} \\ P_{N-1} \end{bmatrix} \tag{3}$$

Where  $H_1, H_{N-1}$  and  $P_1, P_{N-1}$  are the tangential components of magnetic and electric fields of the 1<sup>st</sup> and N<sup>th</sup>, A is the characteristics matrix for the combined structure which is obtained by the following equation;

$$A = \prod_{k=2}^N A_k = \begin{vmatrix} A_{11} & A_{12} \\ A_{21} & A_{22} \end{vmatrix} \tag{4}$$

$$A_k = \begin{vmatrix} \cos \beta_k & -i \sin \beta_k / q_k \\ -i \sin \beta_k & \cos \beta_k \end{vmatrix} \tag{5}$$

Here  $\beta_k$  and  $q_k$  are

$$q_k = \frac{(\epsilon_k - n^2 \sin^2 \theta_1)^{1/2}}{\epsilon_k}$$

$$\beta_k = \frac{2\pi d_k}{\lambda} (\epsilon_k - n^2 \sin^2 \theta_1)^{1/2}$$

Where  $\theta_1$  and  $\lambda$  are incident angle and wavelength of incident light,  $d_k$  and  $\epsilon_k$  are the kth layer's thickness and dielectric constant

The total reflection coefficient of p-polarized light( $r_p$ ) is determined as follows.

$$r_p = \frac{(A_{11} + A_{12}X_N)X_1 - (A_{21} + A_{22}X_N)}{(A_{11} + A_{12}X_N)X_1 + (A_{21} + A_{22}X_N)} \quad (6)$$

The reflectivity of the given multilayer configuration is given by

$$R_p = |r_p|^2 \quad (7)$$

### Performance Parameters

The sensitivity (S), detection accuracy (DA), and quality factor (QF) are the principal parameters governing the performance of a Surface Plasmon Resonance (SPR) sensor. In addition to these, the full width half maximum (FWHM) represents a critical component in assessing sensor efficacy. It is imperative that these metrics attain the highest possible values to ensure the excellence of the sensor's performance.

Sensitivity is formally defined as the proportion between the alteration in resonance angle ( $\Delta\theta_{res}$ ) and the variation in refractive index ( $\Delta n_a$ ) and given as:

$$S = \frac{\Delta\theta_{res}}{(\Delta n_a)} \quad (8)$$

"Angular sensitivity" denotes the alteration in angle resulting from fluctuations in refractive index and is quantified in degrees per Refractive Index Unit (deg/RIU). Furthermore, the Full Width Half Maximum (FWHM) of the reflectance curve is intricately linked to the sensor's Detection Accuracy (DA) and given as:

$$DA = \frac{1}{FWHM} \quad (9)$$

DA is expressed as 1/deg. Meanwhile, the Figure of Merit (FOM) is formally defined as the ratio between sensitivity and the Full Width Half Maximum (FWHM) and given as.

$$FOM = \frac{Sensitivity}{FWHM} \quad (10)$$

## III. RESULTS AND ANALYSES

The first phase of this study focused on optimizing the deposition of copper (Cu). Figure 2 illustrates a graphical representation of the optimization process for the Cu layer. This optimization procedure involved a methodical investigation of layer thickness, spanning from 40 nanometers (nm) to 60 nm. Remarkably, the analysis revealed that the minimum reflectance value is achieved at a layer thickness of 55 nm. As a result, it is determined that a thickness of 55 nm represents the most favorable choice for the copper layer in this context.

The influence of BaTiO<sub>3</sub> (L) and graphene (G) layers when placed above the metal layer is illustrated in Figure 3. The refractive index (RI) of the sensing medium is maintained within the range of 1.33 to 1.34. The corresponding sensitivity values are documented in Table 2. Consequently, the analysis conducted here demonstrates a clear enhancement in sensitivity after introducing monolayers of both L and G atop the conventional SPR structure, denoted as L=0, G=0 (Prism+ Metal). This enhancement results in an increase in sensitivity from 146 deg./RIU to 147 deg./RIU when L=0 and G=1 (indicating the absence of the L layer and the presence of the G layer). Furthermore, when a single L layer is present while G is absent (represented as L=1, G=0), sensitivity is further augmented to 176 degrees per RIU. Lastly, in the proposed configuration with both L=1 and G=1, sensitivity reaches 179 degrees per RIU.

Regarding the variation of graphene layers while keeping BaTiO<sub>3</sub> constant, it is observed that this variation impacts the width of the SPR curves. Specifically, as the number of graphene layers increases, the SPR curves shift to higher incidence angles. A similar effect is noted when varying the BaTiO<sub>3</sub> layers,

affecting the broadness of the SPR curves. It's important to note that the refractive index (RI) is held constant at RI=1.33.

**Table 2** Sensitivity Parameter for various combination of L and G

Figure No.	Layers	Sensitivity
3(a)	L=0, G=0	146 deg./RIU
3(b)	L=0, G=1	147 deg./RIU
3(c)	L=1, G=0	176 deg./RIU
3(d)	L=1, G=1	179 deg./RI.

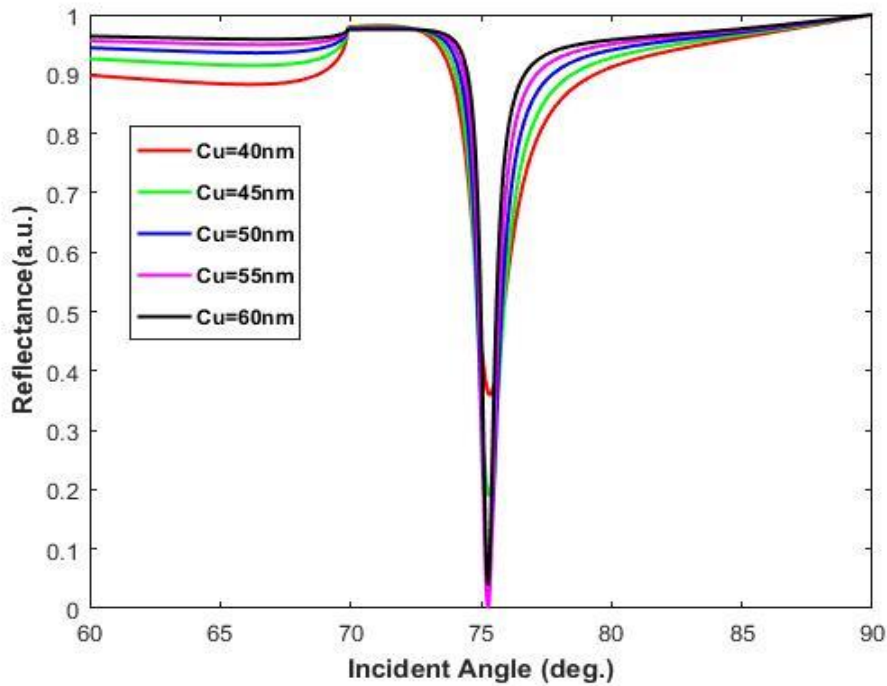
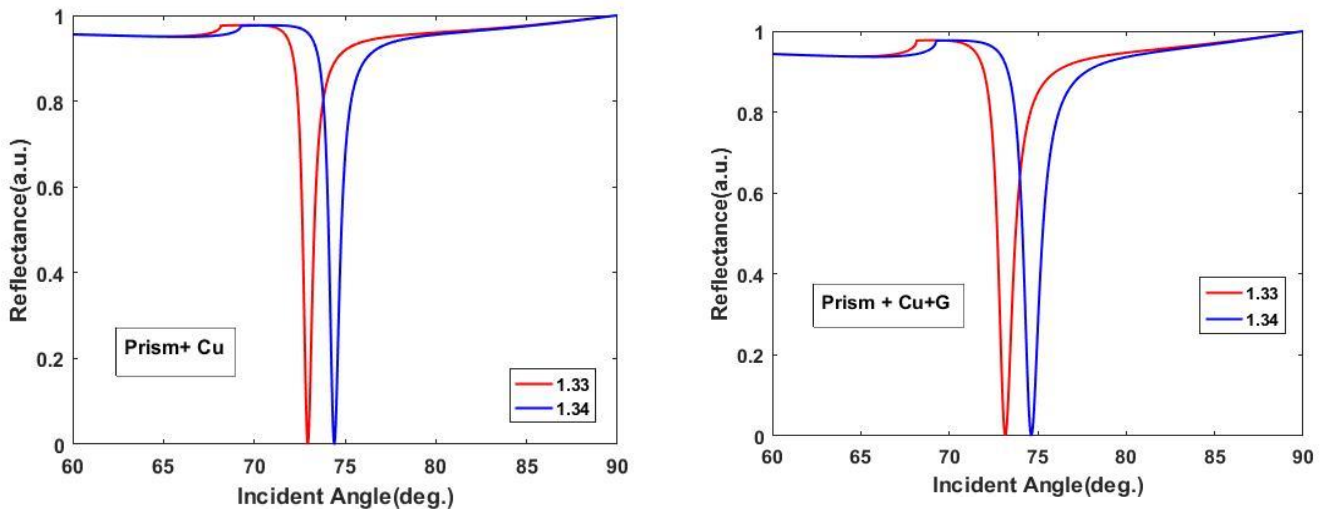


Fig.2 Thickness optimization curve for Cu layer



(a)

(b)

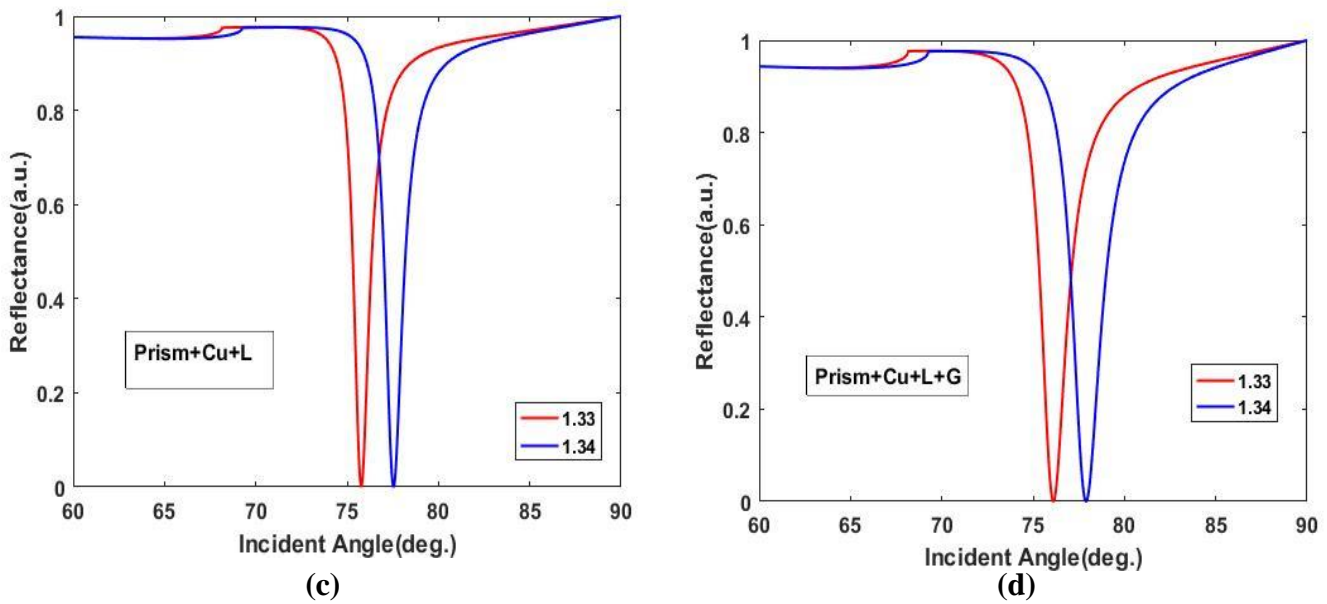


Fig.3 Alteration of reflectance concerning the incidence angle for two RI of sensing medium (1.33 and 1.34), the layer taken as a L = 0, G = 0, b L =0, G =1 c L =1, G = 0, d L =1, G = 1

### 3.1. Detection of Preservatives

To prevent milk from undergoing acidification, various preservatives, namely formaldehyde, hydrogen peroxide, and sodium carbonate, are introduced. This research endeavor involves a comprehensive numerical simulation and analysis of specific preservatives: Hydrogen Peroxide-39, Formaldehyde-37, Sodium Carbonate-12.5, and Sodium Carbonate-25, within the milk matrix. All pertinent parameters have been meticulously computed utilizing transfer matrix methodologies. Furthermore, MATLAB software has been employed to generate graphical representations. The value of concentration with its RI is taken from reference [38].

#### 3.1.1 Detection of Hydrogen Peroxide-39

To quantify the H<sub>2</sub>O<sub>2</sub>-39 content within milk samples, a crucial step involves the placement of these samples onto a graphene substrate. The detection mechanism relies on the alteration of the Surface Plasmon Resonance (SPR) angle, shifting to the right as the refractive index increases due to elevated H<sub>2</sub>O<sub>2</sub>-39 concentration levels in the milk samples.

This shift is attributed to the rising refractive index with increasing H<sub>2</sub>O<sub>2</sub>-39 content in the milk samples. Comprehensive performance parameters associated with changes in refractive index were determined using Equations -8, 9, and 10. The analysis yielded the following results: Maximum Sensitivity (S) 205 deg./RIU, FWHM is 2.14 deg., and DA is 0.47 deg<sup>-1</sup>.

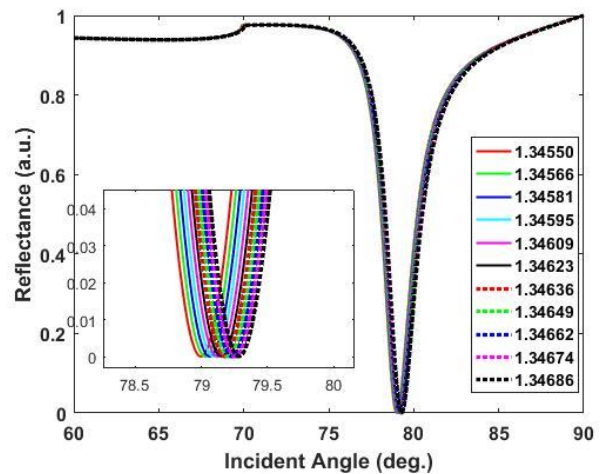


Fig.4 Effect of sample refractive index on reflectance and angle of incidence

#### 3.1.2 Detection of Formaldehyde-37

To quantify the Formaldehyde-37 content within milk samples, a crucial step involves the placement of

these samples onto a graphene substrate. The detection mechanism relies on the alteration of the Surface Plasmon Resonance (SPR) angle, shifting to the right as the refractive index increases due to elevated Formaldehyde-37 concentration levels in the milk samples. This shift is attributed to the rising refractive index with increasing Formaldehyde-37 content in the milk samples. Comprehensive performance parameters associated with changes in refractive index were determined using Equations -8, 9, and 10. The analysis yielded the following results: Maximum Sensitivity (S) 216 deg./RIU, FWHM is 2.14 deg., and DA is 0.47 deg<sup>-1</sup>.

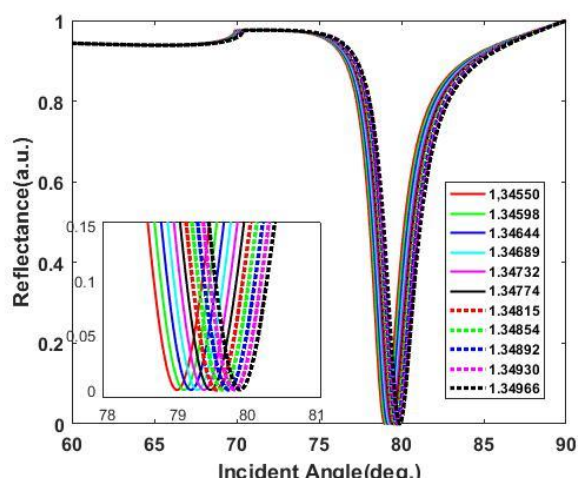


Fig.5 Effect of sample refractive index on reflectance and angle of incidence

### 3.1.3 Detection of Sodium Carbonate -12.5

To quantify the Sodium Carbonate -12.5 content within milk samples, a crucial step involves the placement of these samples onto a graphene substrate. The detection mechanism relies on the alteration of the Surface Plasmon Resonance (SPR) angle, shifting to the right as the refractive index increases due to elevated Sodium Carbonate -12.5 concentration levels in the milk samples. This shift is attributed to the rising refractive index with increasing Sodium Carbonate -12.5 content in the milk samples. Comprehensive performance parameters associated with changes in refractive index were determined using Equations -8, and 9. The analysis yielded the following results: Maximum Sensitivity (S) 213 deg./RIU, FWHM is 2.14 deg., and DA is 0.47 deg<sup>-1</sup>.

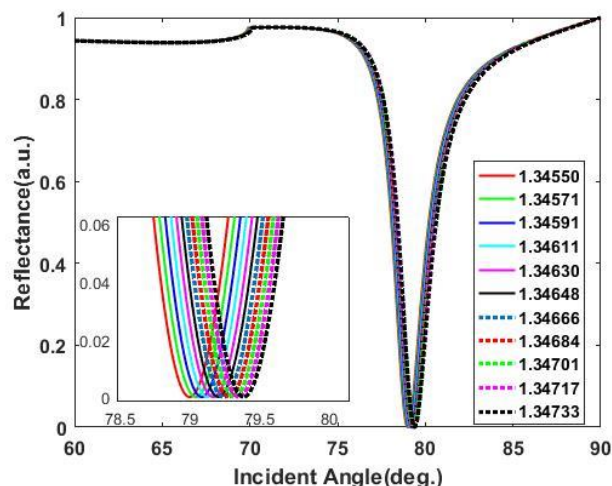


Fig.6 Effect of sample refractive index on reflectance and angle of incidence

### 3.1.4 Detection of Sodium Carbonate -25

To quantify the Sodium Carbonate -25 content within milk samples, a crucial step involves the placement of these samples onto a graphene substrate. The detection mechanism relies on the alteration of the Surface Plasmon Resonance (SPR) angle, shifting to the right as the refractive index increases due to elevated Sodium Carbonate -25 concentration levels in the milk samples. This shift is attributed to the rising refractive index with increasing Sodium Carbonate -25 content in the milk samples. Comprehensive performance parameters associated with changes in refractive index were determined using Equations -8, 9, and 10. The analysis yielded the following results: Maximum Sensitivity (S) 219 deg./RIU, FWHM is 2.14 deg., and DA is 0.47 deg<sup>-1</sup>,

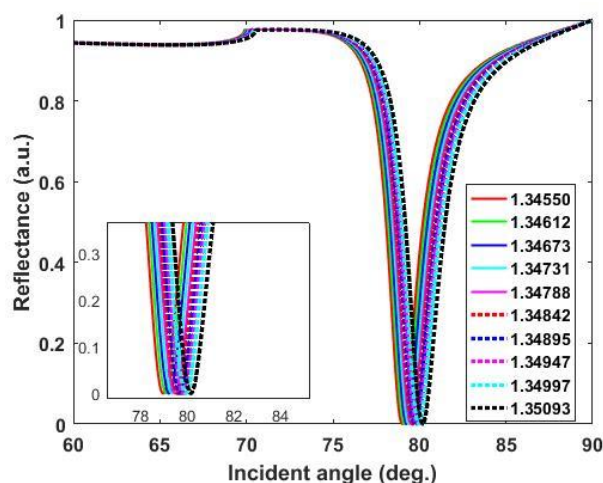




Fig.7 Effect of sample refractive index on reflectance and angle of incidence

#### IV. Conclusions

The study introduces a graphene-coated Surface Plasmon Resonance (SPR) sensor design and simulation, emphasizing its remarkable sensitivity to changes in refractive index. This sensitivity is contingent upon alterations in the refractive index of the surrounding medium, exemplified here using low-fat milk. The refractive index modulation is achieved through the introduction of preservatives such as formaldehyde, hydrogen peroxide, and sodium carbonate. These additives induce refractive index shifts ranging from 1.34550 (0% concentration of sodium carbonate-25) to 1.35093 (14.43% concentration). To rigorously assess the sensor's performance, key metrics including sensitivity, Full Width at Half Maximum (FWHM), and detection accuracy were computed using a numerical approach for each scenario. This comprehensive analysis validates the sensor's efficacy.

Consequently, the SPR-based sensor proposed in this research holds promise for applications in liquid food processing, particularly for monitoring target parameters susceptible to changes in refractive index.

**Conflict of Interest:** The author has no conflict of interest.

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