Acetic acid concentration estimation using plastic optical fiber sensor based surface plasmon resonance

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Abstract
Optical fiber chemical sensor based on surface plasmon resonance for sensing and measuring the refractive index and concentration for Acetic acid is designed and implemented during this work. Optical grade plastic optical fibers with a diameter of 1000 μm were used with a diameter core of 980 μm and a cladding of 20 μm, where the sensor is fabricated by a small part (10 mm) of optical fiber in the middle is embedded in a resin block and then the polishing process is done, after that it is deposited with about (40 nm) thickness of gold metal and the Acetic acid is placed on the sensing probe. It was noted from the results that the refractive index increasing as the magnitude of shifting of the dip position increase of wavelength thus concentration increasing.

Introduction
Optical fiber sensors based on SPR have been widely studied over the past 20 years with many configurations that often use multi-mode optical fibers with large cores and plastic claddings [1]. Optical fiber sensors field has increased in its research lines and possibilities with the use of nanocoating deposition techniques. Nanostructured thin films and nanocoatings have been applied to the diverse optical fiber configurations for the fabrication of new sensors [2]. Several optical systems based (SPR) has been developed to act as chemical sensors, refractometers or even to measure the thickness for metals and thin films. These developments provided solutions to several of the problems related to sensors. The problems which can confront the sensors associated with the sensor size, its ability to sense small variations, the cost, the amount of the sample to be sensitive, and its performance parameters like resolution and sensitivity etc [3, 4]. SPR has achieved

Key words
Optical fiber sensor, surface plasmon resonance, chemical sensor, Acetic acid.

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many achievements to provide developed sensors especially after their participation in optical fiber technology. This type of sensors, optical fiber sensor based SPR, is useful in many areas because of its important features such as label-free samples, very small amount of sample, monitoring in hard-to-reach environments, extremely fast response, miniature components, remote sensing, low coast as well as its good performance parameters [5, 6]. The coupling conditions are realized when the optical wave is reflected in the metal film because of the high sensitivity to change of the refractive index in the surrounding medium, and because of the energy transferred from the photons incident to the surface Plasmon’s observed a sharp dip in the resonance angle. Thus, the energy of reflected light is reduced. There are angle interrogations and spectral interrogations for this dip. Spectral interrogation is mostly used in optical fibers where the angle is maintained and the resonance length is tracked. A shift occurs in the position of the wavelength resonance when it changes in the refractive index. The fiber optic based on total reflection is generally used for exciting of surface plasmon [7]. The SPR technique was used in 1982 for gas sensing for the first time, where optical excitation of Surface Plasmon Waves (SPW’s) has been utilized to develop many sensors [8-10]. In this work, an optical fiber chemical sensor relies on SPR was designed and implemented for sensing the changes in the refractive index for the Acetic acid.

Theoretical basis

The sensing of the SPR sensors is based on optical fiber. This type of sensors mainly consists of three layers: optical fiber core, thin layer of metal, and sensing medium as shown in Fig.1. Small portion of optical fiber unclad firstly and then a metal layer deposit directly on the core. According to spectral interrogation method a polychromatic light is launched into one of the ends of the optical fiber and the transmitted light at the other end is detected using optical spectrum analyzer (OSA). At a specific wavelength a resonance occurs and at this point a sharp dip in the transmitted power appears. Any change of the refractive index of the sensing medium, which is adjacent to the metal surface, leads to shifting in the resonance wavelength [9].

![Fig.1: Illustration diagram of three layers system: the core of fiber, the sensitive metal layer and the medium to be analyzed](image)

The experimental work and devices

1. Optical sensor systems

Optical grade plastic optical fiber with a diameter of 1000 μm was used with a diameter core of 980 μm and a cladding of 20 μm. The length of fiber used was 30 cm and numerical aperture (0.51) without a jacket and a small part (10 mm) of optical fiber in the middle is embedded in a resin block and then the polishing process is done. The removed clad part is cleaned with
distilled water, and then it is deposited with about (40 nm) thickness of gold metal using (ION-COATER). The machine of Model KIC-1A used was from COXEM Company, Korea. The optical grade plastic optical fiber is shown as a photograph in Fig.2.

![Image](image1.png)

**Fig.2:** The photograph of the optical grade plastic optical fiber.

2. Experimental work

The experimental work for measuring the transferred light spectrum consists of the light source (halogen lamp), optical – grade plastic optical fiber from Thorlabs and finally the optical spectrum analyzer (OSA) from Thorlab, as shown as a schematic in Fig.3.

![Image](image2.png)

**Fig.3:** The Outline of the experimental setup of SPR based on plastic optical fiber.

3. Preparations a solution of different refractive indices

The sensitive region of the sensor is covered in various sucrose / water solutions with various concentrations and then different $n_s$ refractive indices. We measured the refractive indices of the solutions using (Abbe) refractometer. The relation between the solution concentrations and the refractive index are shown in the Fig.4. Regression lines are be used as a way of visually depicting the relationship between the independent (x) and dependent (y) variables in the Fig.4. A straight line depicts a linear trend in the data (i.e., the equation describing the line is of first order. and $R^2$ is expressed as a correlation coefficient. The closer $R^2$ is to 1.00, the better the
This too we calculated and displayed in the Fig.4, where the relation between the solution concentrations and the refractive index is linear relationship.

**Fig.4: Refractive index of sucrose /water solutions as a function of the solution concentration.**

**Results and discussion**

The values of the parameters used in the numerical calculations and also in the experimental study were: fiber optic numerical aperture (NA) =0.51, fiber core diameter (D) =980 µm, sensing length (L) =10 mm, metal layer thickness (d) =40 nm and different values (1.346, 1.359, 1.364, 1.392, 1.417 and 1.429) of refractive index from sucrose /water solutions.

The spectra are obtained by recording the transmission curves (T) of the light through the optical fiber. The transmission (T) is calculated from the ratio of the intensity (I) measured in the presence of a sample (sensing medium) and the intensity of the optical signal reference (I₀) that measured in the lack of the sample as in Fig.5.

**Fig.5: Spectrum of the polychromatic light through the sensor measured using the spectrum analyzer; blue line for sensitive region of optical fiber in air, red line for sensitive region dipped in a solution of refractive index n = 1.364.**
Normalized transmitted power (T), calculated as a percentage is from the ratio of the intensity (I) measured in the presence of a sample (sensing medium) and the intensity of the optical signal reference (I₀) that measured in the lack of the sample, is plotted in Fig.6 as a function of the wavelength expressed in nm, this Figure called SPR curve.

![Normalized transmitted power obtained through a sensor fiber, metallized with gold of thickness (d=40 nm, L= 10 mm, D= 980 μm) immersed in a solution having an index n = 1.364. This curve is calculated directly from the spectra of Fig.5.](image1)

**Fig.6:** Normalized transmitted power obtained through a sensor fiber, metallized with gold of thickness (d=40 nm, L= 10 mm, D= 980 μm) immersed in a solution having an index n = 1.364. This curve is calculated directly from the spectra of Fig.5.

The transmission (T) is a function of the wavelength in (nm). The T-wavelength curve is called the SPR curve and at a particular wavelength named resonance wavelength, a sharp dip happens in T because of the energy of incident light transfer to the electrons of the metal and thus lessens the reflected light intensity. The location of this dip based on the refractive index (n) to the sensing medium. As the refractive index of the sensor medium increases the resonance wavelength increases thus the sharp dip of the resonance wavelength will be shifted to along with longer wavelengths (redshift) as shown in Fig.7.

![Refractive index as a function of resonance wavelength for the sensor with gold layer.](image2)

**Fig.7:** Refractive index as a function of resonance wavelength for the sensor with gold layer.
Fig.8 explains the surface Plasmon resonance for the fabricated sensor with a gold layer at a various refractive indexes of the Acetic acid samples (sensing medium). The width and dip position of each (SPR) response curve is changed to the sensor with each sample having a different refractive index and also the refractive index increasing as the magnitude of shifting of the dip position increase. These variations make the performance parameters, which depend on the SPR curve width, the value of the shifting and the position of dip changing with the changing of resonance wavelength and refractive index of the sensing medium because it depends on the change of the resonance wavelength, the width of the spectral curve and the change of refractive index.

![SPR curves](image)

Fig.8: SPR curve of the optical fiber sensor with a gold metal for different samples of Acetic acid.

Table 1 explains the values of the refractive index and concentration for each sample of chemical at different resonance wavelengths. The resonance wavelengths have been determined from SPR curves for various samples of Acetic acid in Fig.8 while concentration and refractive index values were calculated from slope equations in Figs. 4 and 7 respectively. The concentration of the samples increases as the refractive index increases and hence the resonance wavelengths increase this happens because the sharp dip shifting to the red wavelength.
Table 1: Values of the refractive index and concentration for various resonance wavelengths of Acetic acid.

<table>
<thead>
<tr>
<th>Samples of Acetic acid</th>
<th>Resonance Wavelength ($\lambda_{res}$)</th>
<th>Refractive Index (n)</th>
<th>Experimental Concentration (C) %</th>
<th>Theoretical Concentration (C) %</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>490</td>
<td>1.3439</td>
<td>11.72</td>
<td>10</td>
</tr>
<tr>
<td>b</td>
<td>504</td>
<td>1.3494</td>
<td>14.83</td>
<td>15</td>
</tr>
<tr>
<td>c</td>
<td>522</td>
<td>1.3567</td>
<td>18.83</td>
<td>18</td>
</tr>
<tr>
<td>d</td>
<td>536</td>
<td>1.3623</td>
<td>21.94</td>
<td>20</td>
</tr>
<tr>
<td>e</td>
<td>550</td>
<td>1.3679</td>
<td>25</td>
<td>25</td>
</tr>
</tbody>
</table>

Conclusions
This search presents the using of optical grade plastic optical fibers as a sensor of concentration of the Acetic acid as a chemical sample. Surface Plasmon Resonance response curve for different samples of the Acetic acid was recorded in this work and exhibited a dip in the position of resonance. A change in the value of the resonance wavelength occurs for each change in the refractive index and hence for each change in the concentration of Acetic acid. From the results that the refractive index increasing as the magnitude of shifting of the dip position increase of wavelength thus concentration increasing.

References