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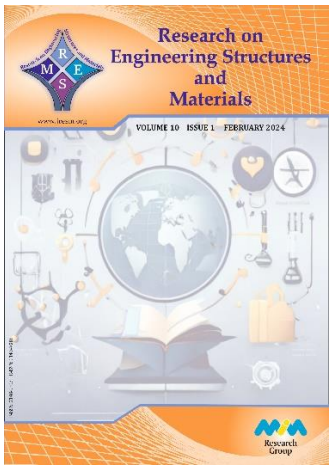

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Research Article

Surface plasmon resonance chemical sensor modified with α-naphthylmethacrylate nano thin film for chloroform detection

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Abstract

The research on the rapid and sensitive detection of pollution caused by the products and technologies used has recently attracted attention in the literature. In the detection of volatile organic compounds (VOCs), polymers/monomers are used in the production of sensitive, fast-responding, reversible thin-film gas sensor elements. In this work, the monomer α-Naphthylmethacrylate was selected as thin film sensor element. The α-Naphthylmethacrylate-based nano thin films were prepared with technique of LB. Monomer LB thin films fabricated onto gold-coated glass substrate to investigate their vapour sensing properties by using Surface Plasmon Resonance (SPR) optical technique. This prepared monomer-based thin film sensor exposed to five different concentrations of chloroform vapors varying between $13.98x10^{3}$-$69.9x10^{3}$ ppm and the sensor response values were recorded. Swelling dynamics' of this monomer thin film sensor was also illuminated by using Fick's early-time diffusion law. Diffusion coefficients of α-Naphthylmethacrylate LB thin film sensor materials exposed to the five different concentrations of chloroform vapor were calculated with the help of reflected light intensity graph data and Fick's Law as a function of time. It was determined that the diffusion coefficient values of the first and second slope regions as varying between $5.72x10^{-17}$-$21.97x10^{-17}$ cm^2s^{-1} and $3.06x10^{-17}$-$6.25x10^{-17}$ cm^2s^{-1}, respectively. SPR kinetic measurement results showed that α-Naphthylmethacrylate material is promising for the detection of chloroform vapor.

1. Introduction

Volatile organic compounds (VOCs) are the leading chemicals causing environmental pollution [1,2]. These compounds, which have low boiling points and high vapour pressures and can pose very serious health risks, have great negative effects on people's respiratory, circulatory, nervous, excretory and even immune systems [3,4]. It is of great importance that these compounds remain within acceptable limits in the indoor or outdoor environment, and various sensors are being developed for their detection. One of these compounds, chloroform, is harmful to the environment and human health. Chloroform with chemical formula $CHCl_3$ is colourless, having a boiling point of 61 °C and it has a density of 1.48 g cm^{-3}. Chloroform has very high volatility and very low solubility in water. It is a chemical that can be found at very low levels in some cleaning products and medicines. While short-term exposure can cause central nervous system disorders, long-term exposure can lead to hepatitis, lung disorders, depression and irritability. Exposure to chloroform above 40000 ppm can result in death. Chloroform, which used to be used for anaesthesia, is no longer used for this purpose.

These vapors can be easily detected due to the favourable cavity and host-guest interaction. With their assistance of Quartz Crystal Microbalance (QCM), Surface Plasmon

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Resonance (SPR) and other gas measurement systems, the response of the thin film sensor can be recorded as a function of time when organic vapor is injected into a thin film sensor. Among these techniques, SPR technology provides both the elucidation of the optical properties of thin films and the analysis of the prepared thin film sensors at a low concentration.

Many sensors with different operating mechanisms, fast and sensitive detection of organic volatile compounds in the indoor or outdoor environment can be carried out [5,6]. A thin film layer is usually preferred as the basic gas sensor element in the detection of volatile organic compounds in this field [7-9]. These thin films are designed to have a controlled, homogeneous structure and effective detection capability; researchers are making great efforts to develop gas sensor elements with increased sensitivity, low cost and reversibility [10-12]. Many thin film fabrication techniques such as LB thin film technique [13-15] and spin coating [16-18] are in the focus of researchers' attention in this sense; design, modelling and fabrication of thin films are carried out. These techniques, in which an unlimited number of raw materials can be used for different application areas, contribute to the emergence of thin films suitable for the purpose with their flexibility. There is a limited number of research on the synthesis and characterisation of new raw materials that can offer many of the desired properties at the same time at low cost. The majority of these synthesised raw materials are monomers and polymers.

Polymers are considered as versatile materials that are suitable for an uncountable number of applications due to their controllable properties. The low cost of polymers, which can be produced in various forms and properties, makes them indispensable for many fields [19-21]. In the detection of volatile organic compounds (VOCs), these unique polymers are utilized in the production of sensitive, fast responding, reversible thin film chemicals sensor elements. The main objective of this study is to investigate monomers that can best interact with the volatile organic compounds to be detected and enable the production of efficient chemical sensors [22-24]. It is important to synthesize raw materials that are suitable for the molar volume of the gas to be detected and that can form dipole moments or other secondary interactions with the gas.

Increasing environmental pollution and harmful organic vapors, which are increasingly emitted into the indoor and outdoor environment, can reduce the quality of life of people and cause serious health problems in long-term exposures. Considering the increase in these harmful chemical compounds, the importance of the design and application of reversible gas sensors that can perform fast, sensitive and repetitive detection is understood. For these reasons, this study aims to produce thin films from monomer materials, to elucidate their reactions and interaction mechanisms against harmful organic vapors.

In this study, α-Naphthylmethacrylate based thin films were prepared by utilizing LB thin film technique onto the gold-coated glass substrates. These LB thin films were expose to chloroform vapour at different concentrations to investigate their gas sensing abilities. The performance of α-Naphthylmethacrylate based LB thin film sensor was measured by calculating the values of sensitivity, LOD and diffusion coefficients. The diffusion coefficients of chloroform for α-Naphthylmethacrylate LB thin films were illuminated with this study.

2. Theory

2.1. Method of Surface Plasmon Resonance (SPR)

SPR is one of the spectroscopic methods used to measure the thickness and refractive index of thin films on metal surfaces. Fig. 1 shows a symbolic presentation of the SPR

measurements. The thickness of LB films and their weakly bound molecular interactions with organic vapors can be studied with the sensitivity of SPR [25,26]. That is, the layer-by-layer production of thin films is controlled in relation to the angle shift in the SPR curve of the molecules attached to the substrate. On the contrary, the thickness of the thin film is kept constant and exposed to organic vapors and the molecular interactions are analysed in relation to the reflected light changes recorded in the photodetector.

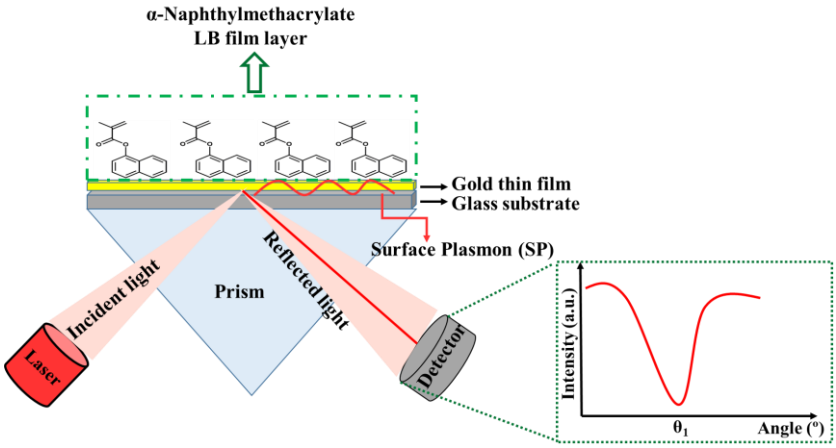

Fig. 1. A symbolic presentation of the SPR measurements

The light intensity reflected of the metal-thin film structure is recorded in the form of an SPR curve given in Fig.1. Surface plasmons (SPs) occurs during interaction between incident light and thin film [27]. The setup of SPR experimental system known as the Kretschmann configuration is shown in Fig. 1. Molecular interactions that cause a change in the refractive index, or an increase in molecular mass, change the intensity of the sensor material and hence the angle of incident light, which translates into a response signal in the detector [28]. This change causes a shift in the minimum of the resonance angle in the SPR graph. For thin films with linearly increasing thickness, the change in the resonance angle increases in direct proportion.

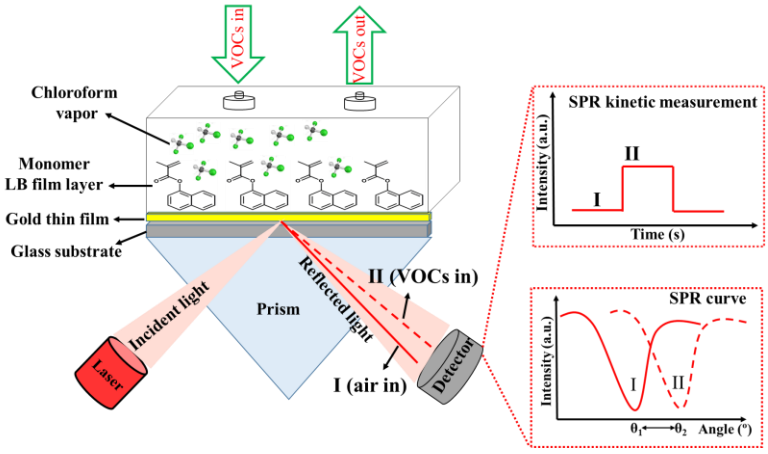

Fig. 2. A schematic demonstration of SPR curves and SPR kinetic measurements

From this data, the thickness of the transferred layers can be determined. Since SPR curve can display the distribution and amount of molecules transferred to a metal surface in real time, its use for the determination of the manufacturability of thin film monolayers is quite common [29]. A schematic representation of the gas cell for SPR kinetic measurements is given in Fig. 2. When the sensor surface is organized to contain selective receptors for specific chemicals or biomolecules, the structure of the newly formed material can be applied in chemical and biochemical sensor fields. SPR spectroscopy is one of the main optical techniques used today for the development of low-cost and high-resolution chemical and biochemical optical sensors [30]. SPR sensing has been receiving increasing attention in the scientific community due to its advantages of real-time monitoring and remarkable sensitivity.

2.2. Fick's Law and its Application

The diffusion coefficient (Ds) is calculated with the equations of diffusion described in Fick's 2. law. The data of SPR kinetic results obtained in real-time measurements was used during this calculations [31]. In this study, this diffusions' law was applied to calculate the value of diffusion coefficient during chloroform molecule diffuse into the monomer film. The early-time approximation presented in Eq (1) for plane layers [31]. a_0 is the initial thickness of the surface layer, D is the diffusion coefficient, M_t and M_∞ represent the amount of material diffusing into the surface layer at times t and infinity, respectively.

$$\frac{M_t}{M_\infty} = 4\sqrt{\frac{D}{\pi a_0^2}}t^{1/2} \tag{1}$$

This behavior suggests that the number of saturated vapor molecules M_t diffusing into the LB thin film should be inversely proportional to I_r and Eq (1) can be expressed as Eq (2).

$$\frac{M_t}{M_\infty} \cong \left(\frac{I_r(t)}{I_r(\infty)}\right)^{-1} = 4\sqrt{\frac{D}{\pi a_0^2}}t^{1/2} \tag{2}$$

This new equation states that the ratio of reflected luminous intensity varies in direct proportion to the $t^{1/2}$. Using Eq (2) and the plot of the normalized irradiance as a function of the $t^{1/2}$, the diffusion coefficients can be calculated.

3. Experimental Details

The monomer α-Naphthylmethacrylate (given in Fig. 3) preferred in preparation of chemical sensor thin films was obtained with the procedure given in previous studies [32,33].

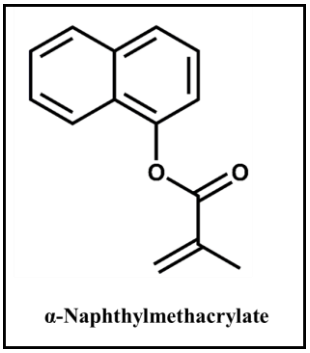

Fig. 3. The chemical structure of α-Naphthylmethacrylate monomer

In this study, the α-Naphthylmethacrylate was chosen as thin film element to research its VOCs sensing characteristics. The optimum surface pressure value for the production of α-Naphthylmethacrylate LB thin film was determined as 13mN m^{-1} from isotherm graph of this monomer [25] and 10 layers of LB thin film were produced on a gold-coated glass surface for investigating their gas sensing properties.

4. Results and Discussion

Generally, the interaction mechanism between the thin film and organic vapor molecules is known to occur in three steps: surface adsorption interaction, interlayer diffusion and desorption [34,35]. A schematic representation of these steps is given in Fig. 4. When organic vapor is injected into the gas cell, the reason for the rapid increase in reflected light intensity is due to the surface adsorption interaction between the LB thin film and organic vapor molecules. This is followed by the diffusion effect where the organic vapor molecules begin to penetrate into the LB thin film layers. The diffusion process is related to many important physical properties of vapor molecules. Desorption is the removal of organic vapor by injecting fresh air into the gas cell.

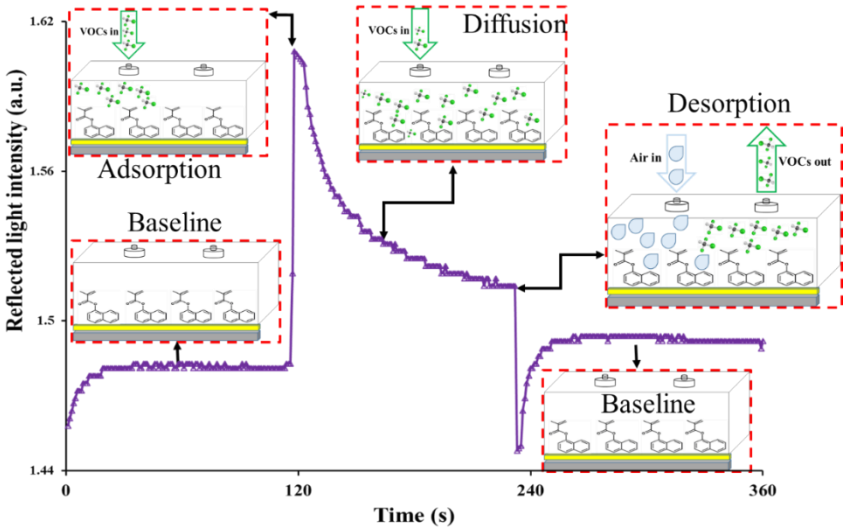

Fig. 4. A schematic representation of the interaction mechanism between monomer thin film and chloroform vapour

From the graph given in Fig. 4, there is fresh air in the environment between 0 and 120 seconds. At 120. Second, when chloroform vapor was introduced into the gas cell, the reflected light intensity increases rapidly and reaches a maximum value. It is noteworthy that the intensity changes rapidly as soon as organic vapor is introduced, indicating that the thin film reacts quickly to chloroform vapor. After a certain change in the reflected light intensity, approximately takes a constant value. The fact that the reflected light intensity remains stable in this period indicates that the thin film interacts with chloroform vapor. After allowing the chloroform vapor to remain in the environment for 2 minutes, air was introduced into the environment at 240. second and the reflected light intensity was observed to return to its previous value. After air introduced to cell, if the value of the reflected light intensity is not return to its previous value, the sensor coated with monomer thin film is not reversible property. Similarly, three times chloroform vapor was sent to cell and the reflected light intensity change was recorded. Fig. 5 displays the responses of monomer-coated SPR sensor to chloroform vapors nearly same.

The interactions of monomer LB thin film sensor with saturated and different concentrations of chloroform organic vapour is shown in Fig. 6. Between 0 - 120 seconds there is air in the environment. At 120 seconds, 13.98x10^3 ppm chloroform was introduced into the environment where the monomer LB thin film sensor was located. After the chloroform vapor was kept in the environment for 2 minutes, air was introduced into the environment to remove it from the environment. Then, at 360 seconds, 27.96x10^3 ppm chloroform vapor was introduced into the environment and kept in the environment for 2 minutes, and then air was again introduced into the environment and removed from the environment. This process was repeated with 41.94x10^3, 55.92x10^3 and 69.9x10^3 ppm chloroform vapour. Fig. 7 shows that the shift in the reflected light intensity increases as the concentration ratio increases. The LB thin film sensor fabricated with monomer was found to be sensitive to harmful chloroform vapor.

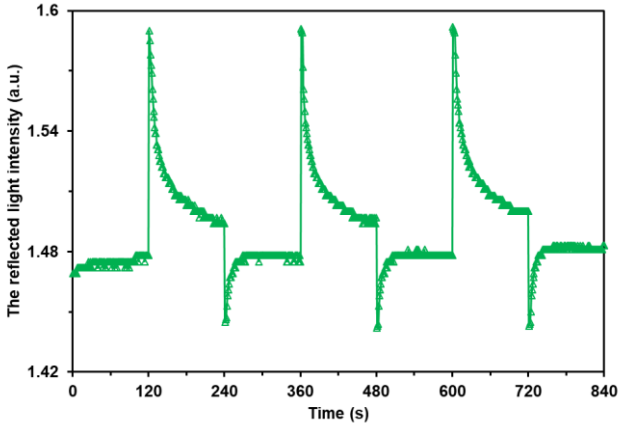

Fig. 5. The response of monomer LB thin film sensor to the saturated chloroform vapour for three cycles

The sensitivity value of the monomer-based SPR optical sensor was obtained from the slope of Fig. 7. This figure explains the relationship between the sensor response and chloroform vapor at distinct concentration. As a result, the sensitivity value of the monomer-based SPR optical sensor is 1.297 × 10^{-3} ppm^{-1} with LOD value of 9.02 ppm.

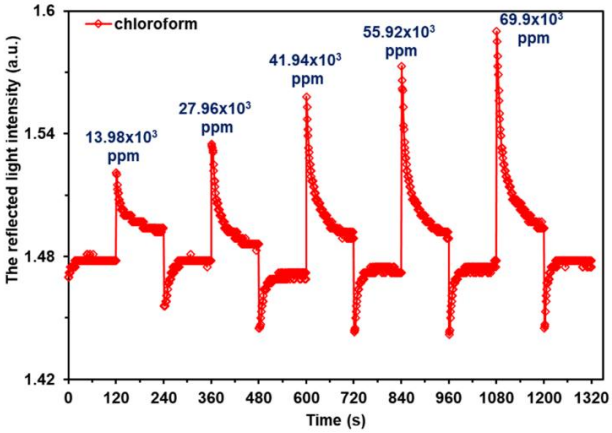

Fig. 6. The response of monomer LB thin film sensor to chloroform vapour at different concentrations

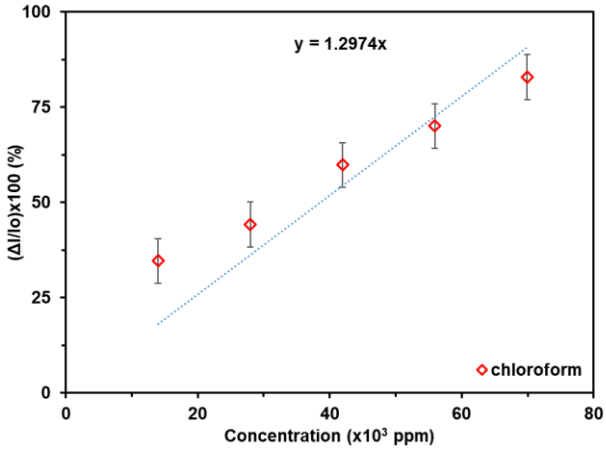

Fig. 7. The linearship relation between chloroform vapour and the change in the reflected light intensity

The chloroform vapour may be interacted with the sensor films by the emergence of hydrogen bonds during interaction between VOC and sensor film. The SPR kinetic result can be also explained in terms of molecular weight of chloroform vapour. Such physical parameters have an impact on the adsorption behavior when vapor molecules bind to sensing films. A larger vapor molecular weight such as chloroform leads to higher sensitivity as reported in previous findings in the literature [36,37]. The diffusion data between 120-240 second obtained from Fig. 4. Using these data, the change in reflected light intensity of the monomer LB thin film for chloroform vapor is obtained as a function of diffusion time shown in Fig. 8. In order to compare the experimental data with each other, the y-axis is arranged as normalized irradiance. The variation of the normalized irradiance value versus time decreases as an exponential function.

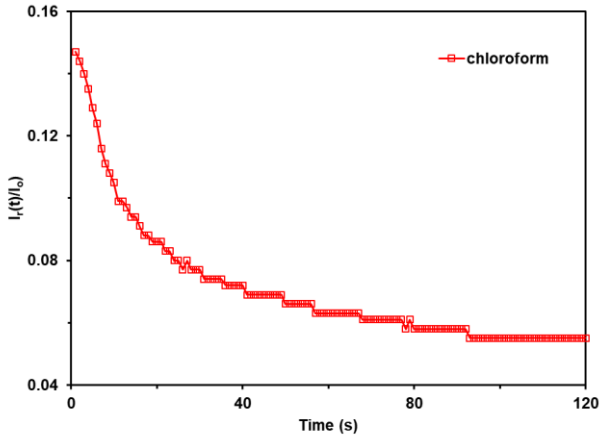

Fig. 8. The normalised responses as a function of time for the interaction of monomer LB thin film and chloroform vapour

In the diffusion interaction process, two different slopes were observed. During the first slope period, rapid surface adsorption interaction occurs as the organic vapor makes initial contact with the monomer LB thin film surface when it is introduced into the gas cell. In the second slope region, the chloroform vapor molecules enter the monomer LB thin film

layers, where they diffuse into the monomer LB thin film structure through physical and/or chemical interactions. This diffusion process can be reversible or irreversible depending on the physical and chemical interactions between the sensitive LB thin film material and the chloroform organic molecules. These interactions are not only dependent on physical properties such as vapor pressure, molar volume, viscosity, etc. [7,15], but also chemical interactions such as host-guest, hydrogen bonding, Van der Waals, etc. [38].

Fick's law is one of the most effective approaches to analyze the diffusion process and calculate diffusion coefficients. When the saturated chloroform vapour (for $69.90x10^3$ ppm concentration) molecules in the gas cell increases, the amount of material penetrating the monomer LB thin film will increase in both slope regions.

Table 1. The values of diffusion coefficient for the first and second regions at different chloroform concentrations.

Concentration (ppm)	First region $D(cm2s^{-1})x10^{-17}$	Second region $D(cm2s^{-1})x10^{-17}$
$13.98x10^3$	8.23	4.05
$27.96x10^3$	5.72	6.89
$41.94x10^3$	14.64	3.06
$55.92x10^3$	16.14	4.01
$69.90x10^3$	21.97	6.25

From this linear relationship given in Fig. 9, diffusion coefficients for both slope regions were obtained using Eq (2). As shown in the inset in Fig. 9, the values of diffusion coefficient for the first and second regions were determined as $21.97x10^{-17}$ cm^2s^{-1} and $6.25x10^{-17}$ cm^2s^1, respectively. The diffusion coefficient values of the first and second slope regions for other concentrations were presented in Table 1.

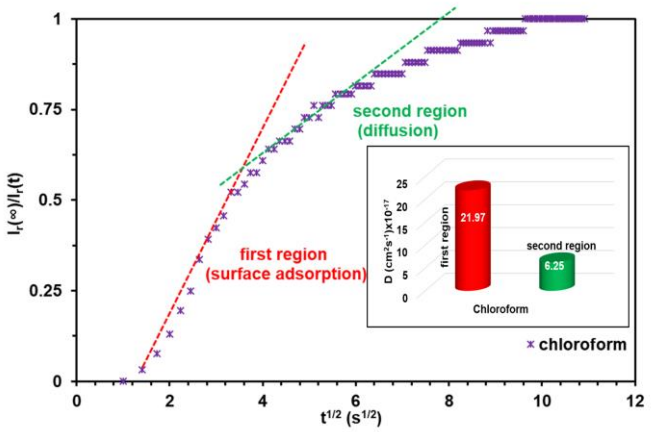

Fig. 9. Plot of $t^{1/2}$ versus normalized value of $I_r(\infty)/I_r(t)$ for monomer thin film-chloroform vapour interaction

The pyrene-based (PS) polymer chains, having three different molecular weights (PS1, PS2 and PS3) were prepared via LB thin film technique by Erdogan and his group [22]. The response of PS-based sensor for chloroform vapor at distinct five concentrations by using

QCM technique. The obtained values of diffusion coefficients vary among 0.2-3.0x10^{-16}, 5.0-13x10^{-16}, and 1.0-1.6x10^{-15} cm^2s^{-1} for the sensor of PS1, PS2, and PS3, respectively. The values of diffusion coefficients for PS1 LB thin film nearly the values for α-Naphthylmethacrylate LB thin film against to chloroform vapour.

5. Conclusions

SPR LB thin film sensors prepared using the monomer α-Naphthylmethacrylate were exposed to different concentrations of chloroform organic vapors. An increase in reflected light intensity was observed with increasing vapor concentrations. After the SPR LB thin film sensor materials were exposed to saturated chloroform organic vapors, time-dependent measurements were repeated three times and the repeatability capacity of the monomer-based sensor material was measured.

The results obtained for monomer LB thin film sensors indicate that this sensor is self-renewing and has the capacity for multi-use potential. The response of monomer thin film materials was analyzed from SPR kinetic data. The fact that monomer-based SPR sensor material reacted to the chloroform VOC and that their responses to this vapor differed showed that this material have selectivity against chloroform organic vapors.

The illumination of the diffusion process, which is a factor in the sensor interaction mechanism for the selected material, was also studied for the first time in this study. Diffusion coefficients of α-Naphthylmethacrylate LB thin film sensor materials exposed to the saturated concentration of chloroform vapor were calculated with the help of reflected light intensity graph data and Fick's Law as a function of time. It was determined that the diffusion coefficient values of the first and second slope regions as 21.97x10^{-17} cm^2s^{-1} and 6.25x10^{-17} cm^2s^{-1}, respectively. All diffusion coefficient values of the first and second slope regions as varying between 5.72x10^{-17}-21.97x10^{-17} cm^2s^{-1} and 3.06x10^{-17}-6.25x10^{-17} cm^2s^{-1}, respectively. It has been revealed that monomer LB thin films have sensor properties such as sensitive, selective, fast, recyclable and reusable as well as being suitable for use as sensor materials and can be used in this field.

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