Gas-sensing performance enhancement in ZnS/polymer films by homogenous morphology surface

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Abstract: The ZnS/polymer films have been successfully prepared by casting technique with different thickness (10, 12, 13.5 and 14) and to carry out a comprehensive study of their gas detection performance. All the different ZnS polymer films thickness demonstrates excellent selectivity and accuracy and stability. ZnS-doped PMMA films show higher sensitivity to ethanol vapors. Further, the films show fast response and recovery to methanol and methanol vapors at higher operating temperatures. The methanol-ethanol sensing mechanism of the film has been explained.

Keyword: Gas sensor; PMMA polymer; Surface Morphology; Methanol; ZnS.

1. Introduction
One of the most important sensors that have a field of engineering research is the gas sensors, which generate an electrical signal when they are sensitive to certain gas emissions. There are gas sensors that sense a specific gas such as methane or carbon dioxide sensors and others [1]. Accurate identification and early warning of flammable, explosive and toxic gases become major challenges in industrial processes due to industrial safety concerns [2]. Gas sensors based on metallic solid-state oxides, such as ZnO, SnO2, TiO2, WO3, and ZnS, etc., have attention to detect a low gas concentrations because of their distinction. Because of their simplicity benefits in the structure of the device and circuits, great sensitivity, versatility, robustness and low manufacturing cost [3-7]. Gas sensors based on polymers are important due to the ease of availability of polymers today and their cheapness in addition to the ease of designing these sensors. Sensors built from polymers are also quick to respond to changes in the amount of gas, as well as controlling their response to the change in the amount of gas. Zinc sulfide is a promising material for gas sensing applications due to its chemical sensitivity to volatile gases and other radical gases, its great chemical stability, doping capability, non-toxicity, natural abundance and low cost. This material was used for gas collectors in the form of simple crystals, sintered pellets, thick and thin layers, etc. [8–12]. However, thin films are better
suited to these sensors because the gas sensing properties are related to the surface of the material where the gases are adsorbed and surface reactions occur. This reaction alters the concentration of the load bearers in the material, giving rise to a change in its electrical resistance, which is used for gas detection [13].

Many researchers have investigated ZnS (doped) thin films to detect toxic gaseous pollutants, combustible gases and organic vapors, [14], ZnS nanoparticles were fabricated using a new route chemical reaction [15,16] which is an increasingly important topic in electrochemical and industrial environments. Nanto et al [17] investigated ZnS thin-film ammonia gas sensors, while the LPG detection properties of ZnS films were investigated by Mitra and Maiti [18]. Sahay et al.[19,20] studied thin-film ZnS sprays for detecting acetone and ethanol vapors. To the best of our knowledge, very little work has been reported on the ZnS. Since methanol and ethanol are very toxic and often deadly to people, developing reliable and selective methanol and ethanol sensor is necessary. Thus, the study of methanol-ethanol detection properties of ZnS polymer films has been carried out and presented in this paper. Nearly all major polymer film preparation methods can be used to make ZnS polymer films. Also, by discussing the effect of temperature on the conductivity of polymeric films in different thickness and examining the relationship of sensitivity with the time of exposure of the membrane to the gas.

2. Sensors Sample preparation

Glass substrate with dimensions of (0.0 x 2.55 x 7.55) cm were used as bases for sedimentation of films on them, and because the presence of impurities or materials suspended on the surface of these slides affects the properties of the prepared films and leads to changing their physical properties, so these bases have been carefully selected and cleaned to obtain a suitable basis for sedimentation films, and this process was carried out in several steps, here the slices were chosen sure that there were no scratches or cracks on their outer surface, which the slides were washed well with distilled water and washing powder, then these slices were immersed in distilled water for a period of 30 minutes, After that, the slides were placed in pure acetone for a period of 15 minutes, then they were dried using filter paper and placed in a thermal oven at a temperature of 373K for a period of 15 minutes to complete the drying process, thereby placed in closed plastic containers until they used for the purpose of depositing the films for electrical measurements. A group of glass slides was chosen after cleaned and dried as previously passed, then learned and weighed each slice, after which a metal wire 0.15 mm wrapped on each slide after it was fixed on a piece of plastic to ensure the applicability of the metal wire to the surface of the glass slide, and the slide was placed inside the evaporator to evaporate a pure aluminum film on it, and after the evaporation process, the metal wire and piece of plastic removed from the glass slide, where we obtained a glass slide with a thin film of aluminum except the areas that with the metal wire, which appeared in the form of voids inside the aluminum film, then used masks in the form of aluminum foil to cover the sides of these bases to prevent the material from being deposited on them during the spraying process, where they will be used as connection points with an external circuit intended for electrical measurements. These stages can be illustrated in several steps, as shown in Figure 1.
Figure 1. The stages of preparing the film for electrical measurements.

3. Sensor Surface Morphology

The topography of the ZnS polymer films was studied, where the choice was made in the thickness of 10 μm and the thickness of 13.5 μm. Where the measured shape of the two thicknesses shows the homogeneity of the casting at the two different thicknesses in the middle, and thus provided us with knowledge of the optimal area to build measurements around, and figure 2 illustrates that.

Figure 2: scan homogeneity of 10 μm and 13.5 μm ZnS films

The characterization of the surface topography or the so-called topography of the surface is very important in the manufacture of optical devices [21,22]. In general, from the observation of figures 2 and 3, that the spread of the surface increases with the average roughness, as the roughness variables are among the important variables in linear and nonlinear optical applications[23,24], such as the linear photoelectric effect [25-27], optical filters[28-30], thermal lens technique [31-34], optical limiting device [35-37],self diffraction ring[38-43] and optical storage devices[44].
Figure 3. Surface graph of 10 μm and 13.5 μm ZnS films
The topography of the surface morphology of one ZnS polymer film is clear and understandable when processing the microscopic of the films. A visual simulation process was performed to measure the surface roughness of the two films (two samples) 10μm and 13μm as shown in Fig. 3. It was found that the ZnS polymer material from which the polymer film was made is distributed almost evenly in some bands. In addition to the feature of the material in some ranges, it has regular and homogeneous shapes when casting and the sizes at the end of the ranges are almost the same, and there are no cracks and separations (scratches) that have been observed in figures 2 and 3. From the two figures, the homogeneity of the distribution in three dimensions is evident, and no clear aggregation was observed in the sample (see Fig. 4). Each sample was also tested separately, which was also done by examining the optical quality of the films bypassing a low-power laser beam of 5mW. The other ZnS samples were also prepared similarly.

Figure 4. 3D image of 10 μm and 13.5 μm ZnS films.

4. Experimental study
4.1 Gas sensor Design
A gas sensor system was designed and built to operate in normal weather conditions and at different temperatures, and the response data was recorded with time and with the amount of inert gas flow (N₂ Nitrogen Gas), and the sensor gas used in this study: ethanol, methanol. The sensor system consists of a 500 ml glass beaker placed on a thermal heater (Heater) whose temperature can be controlled through a voltage divider. The liquid to be vaporized is placed in the glass beaker and its temperature is gradually raised until it reaches the evaporation temperature. The glass beaker is connected to two glass tubes containing each tube includes a valve that controls the flow of gas, one of them is connected to a cylinder filled with nitrogen gas N₂ of Spanish origin, its purity is 99%. The amount of nitrogen gas can be controlled by the gas flow meter, nitrogen gas is characterized as an inert gas that does not affect the measurement process, and it pushes the vapor placed in the glass beaker into the sample to be measured.
The other glass tube is connected to a room that was built from transparent polymer sheets (Pyrex -Perse). The sample is connected to the electrical measurement circuit as shown in Figure 5. Figure 5 shows the gas sensor setup that was built in the physics laboratory and used in this study.

![Gas sensor setup](image)

**Fig. 5:** Gas sensor setup.

The gas sensor system works after placing the sample in the place designated for it and connecting the electrodes of the sample in the electrical properties measurement circuit, which consists of a power supply of the Farnell instruments type, and a voltmeter to measure the potential difference at the two ends of the sample of the Ley Bold type, and a meter to measure the current of the Philips PM 2421 type. Valves 1 and 2 are closed, which are the N₂-carrying gas valve and the liquid-vapor valve placed in the beaker, the amount of current passing in the measuring circuit is measured after a certain voltage is applied, the N₂ gas carrier valve is opened and the current is recorded again at the same recorded voltage difference in the first stage. In practice, it has been observed that the reading of the current is the same in the case before and after opening the nitrogen gas, the temperature of the liquid placed in the glass flask is raised until it reaches the boiling point and turns into vapor, after which both valves are opened at the same time to pushing the gas whose effect on the sample is to be studied and recorded. Current passing through the sample. The ratio between the current values in this case with one of the previous two cases is defined by the response (sensitivity). As for the process of studying the change of the current passing through the circuit with time, it is done in the same way as the previous method, but the measurement process is done with time and at a constant voltage at different flow velocities, 1, 2, 3, 4 (L / min) for ethanol and methanol gas.

### 4.2 Methanol Gas Sensor

Gas sensor properties in case of exposure of polymer films (ZnS doped PMMA polymer film), to ethanol and methanol gases were studied separately, as the sensitivity of these polymer films to ethanol and methanol gases was calculated. By measuring the feature of (current-voltage) in the case of the presence of the two gases and the gas flow rate is constant (1 L/min), the ability of the sensor to respond by changing (current - time) was tested, and a change was also studied (Sensitivity - time) with a constant potential difference of (10V) for
polymer films with different thicknesses (10, 12, 13.5 and 14 μm). By using nitrogen gas as an inert gas at a pumping rate of (2,3L/min) in laboratory conditions of temperature and humidity. Figure 6 shows the relationship between (Log I-Log V) for polymer films measured under the influence of ethanol and methanol both separately. Nitrogen gas (N₂) was used to push these vapors through glass tubes designed for this purpose. It is noticed from all these figures that the current is at its highest value in the case of exposure of the polymer film to methanol gas and all concentrations, while in the case of ethanol gas it is less than it is, but it is greater compared to the case of normal conditions (room temperature),

Fig. 6: Voltage as a function of current for ethanol and methanol vapor.

The process of determining the conduction mechanism depends mainly on studying the (current – voltage) feature. This feature has been studied for polymeric films of different thicknesses. Figure 7 shows the feature (current - voltage) of the polymeric films measured at different temperatures (303-343 ° K). It is evident from these figures that the increase in the voltage applied to these samples is followed by an increase in the current. Also, it is noticed that an increase in the temperature results in an increase in the values of the current and for all the aforementioned samples that the increase in the current is due to the increase in the movement of the charge carriers and their freedom of movement due to the temperature. This behavior is quite similar to studies precedent.
Fig. 7: Voltages as a function of current with different temperature degrees.

The electrical conductivity of all the aforementioned samples was calculated from the slope of the straight line in the ohmic region ($V < 10$) using the relationship [45]:

$$\sigma = \frac{\Delta I}{\Delta V} \times \frac{A}{d}$$  \hspace{1cm} (1)

Where $d$ represents the thickness of the membrane and $A$ represents the area of the electrode, Figure 7 shows that the values of electrical conductivity increase with increasing temperatures for each polymer film, and Table (2) shows that the values of electrical conductivity as a function of the temperature of the polymeric films increase with the increase in temperature, and this indicates that these Models behave like a semiconductor.

The effect of ethanol and methanol gas on the properties of the gas sensor was studied by changing (current - time) at a constant voltage (10V), where the sensitivity was calculated using the following equation [46]:

$$S = \frac{(I_g - I_a)}{I_a} \times 100\%$$  \hspace{1cm} (2)

Table (2) electrical conductivity as a function of temperature for different polymer films thickness.

| $T(K)$ | $10\mu m$ | $12\mu m$ | $13.5\mu m$ | $14\mu m$ |
|--------|-----------|-----------|-------------|-----------|
|        | $\sigma(S.cm^{-1})$ | $\sigma(S.cm^{-1})$ | $\sigma(S.cm^{-1})$ | $\sigma(S.cm^{-1})$ |
| 303    | 0.041     | 0.066     | 0.158       | 0.468     |
| 313    | 0.073     | 0.325     | 0.488       | 0.844     |
| 323    | 0.332     | 0.977     | 0.879       | 2.07      |
| 333    | 0.655     | 1.48      | 1.63        | 3.04      |
| 343    | 2.17      | 2.99      | 3.02        | 3.79      |
4.4 Activation Energy \((E_a)\)

The activation energy is defined as the least energy required to penetrate the electrons or ions into the voltage barriers. The activation energies of the polymeric films were calculated according to the thickness by using the following relationship [47,48]:

\[
\sigma = \sigma_0 \exp \left( - \frac{E_a}{K_B T} \right)
\]  

(3)

Whereas, \(\sigma_0\) is a logarithmic inverse coefficient, which is not affected by temperature often and depends on the concentration of the impurity material and the movement of current carriers, as it represents the minimum electrical conductivity at \(0^\circ K\) and that the unit of measurement is the reciprocal of the unit of measurement of resistivity, which is \((\Omega^{-1} \text{cm}^{-1})\) or Siemens by the reciprocal of a centimeter \((S \text{ cm}^{-1})\), as the Siemens is equivalent to \(\Omega^{-1}\). Where the relationship between the natural logarithm of electrical conductivity \((\ln \sigma)\) was drawn. The y-axis and the reciprocal of the product of the Boltzmann constant at the absolute temperature of the membrane \((1/T) \text{ K}^{-1}\) on the x-axis and for different temperature ranges, as shown in Figure 8.

![Fig. 8](image)

**Fig. 8:** Electrical conductivity as a function of temperature with different thicknesses.

The sensitivity of the polymer films was calculated according to the thicknesses using the following equation [49]:

\[
S = \frac{\sigma_{\text{gas}}}{\sigma_{\text{air}}}
\]

(4)
As $\sigma_{\text{gas}}$ the conductivity when the sample is exposed to the gas, $\sigma_{\text{air}}$ the conductivity if the sample is exposed to the air surrounding the experiment. The values of sensitivity (response) to ethanol gas and to the polymeric films are listed in Table 1.

**Table 1.** Activation energy, Conductivity and Sensitivity of polymer films

| Thickness ($\mu$m) | $E_{\text{act, gas}}$ (eV) | $\sigma_{\text{gas}}$ ($S.m^{-1}$) x10^{-7} | $\sigma_{\text{air}}$ ($S.m^{-1}$) x10^{-9} | Sensitivity ($S$) |
|-------------------|-----------------|-----------------------------------|-----------------------------------|-----------------|
| 10                | 0.77            | 1.998                             | 1.622                             | 123.18          |
| 12                | 0.7             | 1.683                             | 1.511                             | 111.38          |
| 13.5              | 0.53            | 1.399                             | 1.466                             | 95.42           |
| 14                | 0.49            | 1.092                             | 1.288                             | 84.78           |

The table shows that the activation energy values decrease when the polymeric membrane is exposed to ethanol vapor, with different thicknesses, and increases with the thickness of the films. The decrease in activation energy values when exposed to ethanol vapor means a decrease in voltage barriers and an increase in conductivity, which helped increase the sensitivity of these films when exposed to ethanol vapor. Figure 9 shows the relationship between the sensitivity of the polymer films with the change in thickness in the presence of ethanol gas, where it is noticed from the figure that the sensitivity is at its maximum value when the thickness is 10 micrometers, then it drops to its minimum value at a thickness of 14 micrometers. The electrical resistance increases as a result of increasing the thickness of the ZnS films, which reduces the conductivity and this is consistent with previous studies, while the activation energy increases with the increase in thickness to form high barriers and unlike the above-mentioned reasons.

![Fig.9. Relationship between thickness and sensitivity of ZnS-doped polymer films.](chart)

**Fig.9.** Relationship between thickness and sensitivity of ZnS-doped polymer films.

### 4.5 Response of PMMA films to gas flow

Figure 10 shows the relationship between sensitivity and time for PMMA polymer film when exposed to ethanol and methanol separately, in the case of opening and closing the $N_2$ gas flow valve with a flow velocity of (2, 3 L/min) for 60 seconds, The gas valve was opened and closed at regular and equal periods and under a constant voltage of (10 V). The film response begins to increase gradually from the opening of the gas valve until the current reaches its highest value at a time of 60 seconds. The sensitivity values begin to drop the moment the gas valve is closed and...
for the same period when the gas valve is opened for 60 seconds that the response of the membranes increases with time, as well as with the increase in the flow of both gases (ethanol and methanol). Methanol gas for the same membrane, the reason for the sensitivity values of ethanol gas is greater than that of methanol gas is because ethanol gas acts at a higher ionization speed than methanol gas, this speed makes the PMMA film faster in response to ethanol gas than it is in methanol gas. The efficiency of the sensor is measured by the response speed, so the polymeric films are highly efficient when exposed to ethanol gas.

![Fig. 10: PMAA Sensitivity film as a function of time for ethanol and methanol flow.](image)

Figure 11 shows the relationship between the sensitivity and exposure time of the pure PMMA polymer film and dye-doped polymer films with different thicknesses when exposed to both ethanol and methanol gases separately at a flow velocity of 2.3 L/min. It was observed that the pure polymer PMMA (5μm thick) is more sensitive to ethanol and methanol at two gas flow velocities (2.3L/min), while the polymeric membrane with a thickness of 14μm is less sensitive to the two gases at the same flow velocity. This means that increasing the thickness reduces the response of these polymeric films to ethanol and methanol gases and the velocity of flow (2.3 L/min). In other words, as the thickness of the membrane increases, the resistance of the membrane increases due to the steric irregularity that increases the number of traps in the polymer membrane, which impedes the movement of charge carriers within the conducting medium, which leads to an increase in resistance and a decrease in electrical conductivity that causes a decrease in the response to the ethanol and methanol gases with the increase in thickness.

![Figure 11: Sensitivity of PMMA films with different thicknesses as a function of time for ethanol and methanol flow.](image)
Fig. 11: ZnS-doped PMMA films Sensitivity as a function of time for ethanol and methanol flow.

Conclusions
The films of ZnS/PMMA have been successfully deposited by casting technique using a gas sincere. The films deposited at optimum conditions, exhibit a sensitive sensor film in the ethanol and methanol gas region and the values of electrical conductivity increase with increasing temperatures for each polymer film. The ZnS films have shown a smooth polycrystalline surface morphology irrespective of the casting conditions. Through the measurements of the (current - voltage) feature, it was found that all the polymer films recorded an increase in current with an increase in voltage as it was observed that they behave similarly to the normal behavior. The continuous conductivity significantly increased with increasing temperature from 303K to 343K. Also, the calculations of the activation energies of the polymer films showed that their value decreases with the increase in electrical conductivity. It was also observed in practice that they recorded a decrease at higher thicknesses. The response of the prepared films indicates that the films exposed to ethanol gas are more responsive than the other films exposed to methanol gas. The study of the feature of (sensitivity - time), indicated that the prepared film has a high response in the case of opening and closing the valve of ethanol and methanol gas at regular intervals of time.
Where the highest response 25000 was obtained from the PMMA pure polymer films after some time (60 seconds) when these films were exposed to ethanol gas at the pumping speed 2L and the highest response 60000 at the pumping speed 3L, also the highest response was obtained 2000 for pure PMMA films after a lapse of time (60 seconds) when these films were exposed to methanol gas at pumping speed 2L and the highest response 2500 at pumping speed 3L. It is observed that compared to the undoped film (pure PMMA film), ZnS-doped PMMA films are highly sensitive to ethanol and methanol.

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