Edelweiss Applied Science and Technology
ISSN: 2576-8484
Vol. 9, No. 5, 3309-3320
2025
Publisher: Learning Gate
DOI: 10.55214/25768484.v9i5.7691

Chemical bath deposition-produced Ag_2O nanoparticles synthesis and characterization for NO_2 gas sensing applications

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Abstract: Deposition of thin films of silver oxide Ag_2O of a nanoscopic nature, which are highly sensitive to NO_2 gas, on quartz glass bases using the chemical bath deposition technique. Then conduct tests for pH values at the time of deposition at 75°C. In this work, $AgNO_3$ was used. Through XRD examinations, the results showed that all the thin films are polycrystalline. Peaks of silver oxides such as Ag_2O and AgO also appeared, with an average particle size ranging from 31.9 to 46 nm, depending on the sedimentation parameters. Also, through the use of (FESEM) electron microscopy technology, images of the samples used and the arrangement and distribution of the particles are shown. Using an atomic force microscope (AFM), the samples showed that the thin films were homogeneous with different surface roughness, and the particle size ranged from 55.57 to 87.1 nm. It was noted that Ag_2O films have a high sensitivity of approximately 71.58 to NO_2 gas at an operating temperature of 75°C. The study showed that it is possible to manufacture high-quality and efficient gas sensors from silver oxide nanoparticles.

Keywords: *Ag2O, Chemical bath, XRD, SEM, AFM, Gas sensors.*

1. Introduction

Ag_2O is a semiconductor that has an energy gap ranging from 1.3 to 3.6 eV [1]. Then silver oxides were used as gas sensors, as it turned out to be a good sensor for gases, especially NO_2 gas. It is also characterized by its non-toxic properties and is often low in cost and other advantages of Ag_2O [2, 3]. Silver oxides are used in photoelectronic applications [4, 5]. The oxygen vacancies present in silver oxide Ag_2O have a very important role in the conduction mechanism [6]. Therefore, the choice for Ag_2O thin films is a good conductive oxide group that is transparent to P-type thin films. Nitrogen dioxide (NO_2) is a toxic gas present in the atmosphere as well as in laboratories. Due to the danger of this gas, many researchers have sought to find means to protect against this gas [7-9]. Through several techniques, thin films of Ag_2O can be created [10]. Among these techniques are: technique [11] is considered one of the simplest methods for obtaining thin films of silver oxide. The chemical bath deposition technique properties can also be controlled by changing the deposition time, working temperature, and solution pH. In this work, silver oxide was deposited on quartz glass bases at a different pH as well as a different dipping time. Then verify its structural properties and gas sensing.

2. Experimental Work

Quartz glass bases were used before precipitating Ag_2O. Then make scratches on the bases using 25% chromic acid with water for 24 hours. After that, it is cleaned with a detergent solution using ultrasonic waves. Dissolve solid silver nitrate weighing 0.887 g in 5 ml of distilled water. Then add triethanolamine ($C_6H_{15}NO_3$), which is considered a complexing agent. Then add drop by drop and with continuous stirring in order to obtain a colorless solution so that the volume of the solution becomes approximately 41 ml with 0.02 M. The pH also increases after adding nitric acid drip. After that, the

History: Received: 21 March 2025; Revised: 16 May 2025; Accepted: 19 May 2025; Published: 31 May 2025
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samples are placed in a beaker, vertically with a bath, at a constant temperature of 75°C. The immersion times in the bath are different, (30, 40 and 50) min, and the pH values are also different, (10, 11 and 12). Then, the coated samples are taken out of the bath and using distilled water, the samples are rinsed and dried with hot air, then the thickness of the thin films is measured by optical interference. Using XRD technology, the grain size and crystalline structure of the samples were determined (Philips PW 1050 Å Target: Cu-Kα, Current: 20 mA, Voltage: 40 KV, Wavelength: 1.541874 Å). Topography and roughness of the surface of the deposited films were determined by atomic force microscope AFM (SPM-AA3000 Angstrom Advanced Inc). Gas sensing calculations were made for the samples by measuring the change in electrical resistance at about 450 ppm of NO_2 gas, with different working temperatures and also different pH values of the solution.

Table 1.
The sample number with preparation conditions of the prepared nano-silver oxide thin filmsat 75°C

Sample	pH value	Deposition Time (t min)	Thickness (nm)
a	10	40	299.8
b	11	40	250.6
c	12	40	214.7

3. Result and Discussion

Through XRD examination of the samples used in this work, we show that the thin films are polycrystalline based on the appearance of many peaks for (Ag_2O, Ag_3O_4 and AgO). Silver oxide is a cubic structure at level (200), which is compatible with card No. (43-0997). While we note that AgO is a single structure with level (023) according to card No. (43-1038). We believe that the pH of 11 and at the peak has increased significantly for all oxides sample (b) and also the increase in deposition time for sample (c). From Fig (1), it is possible that this is due to the nucleation of Ag_2O, which has an effect on the crystallization process. The average particle size was calculated using the relationship [7].

$$D = \frac{K\lambda}{\beta cos\theta} \qquad (1)$$

Where k is a constant with a value of (0.9), which is usually called the (shape factor). λ is the wavelength of the x-rays. β is the diffraction peak. 2θ represents the diffraction angle in degrees. The grain size ranges from (33.21 to 38.11) nm. as shown in Table (2).

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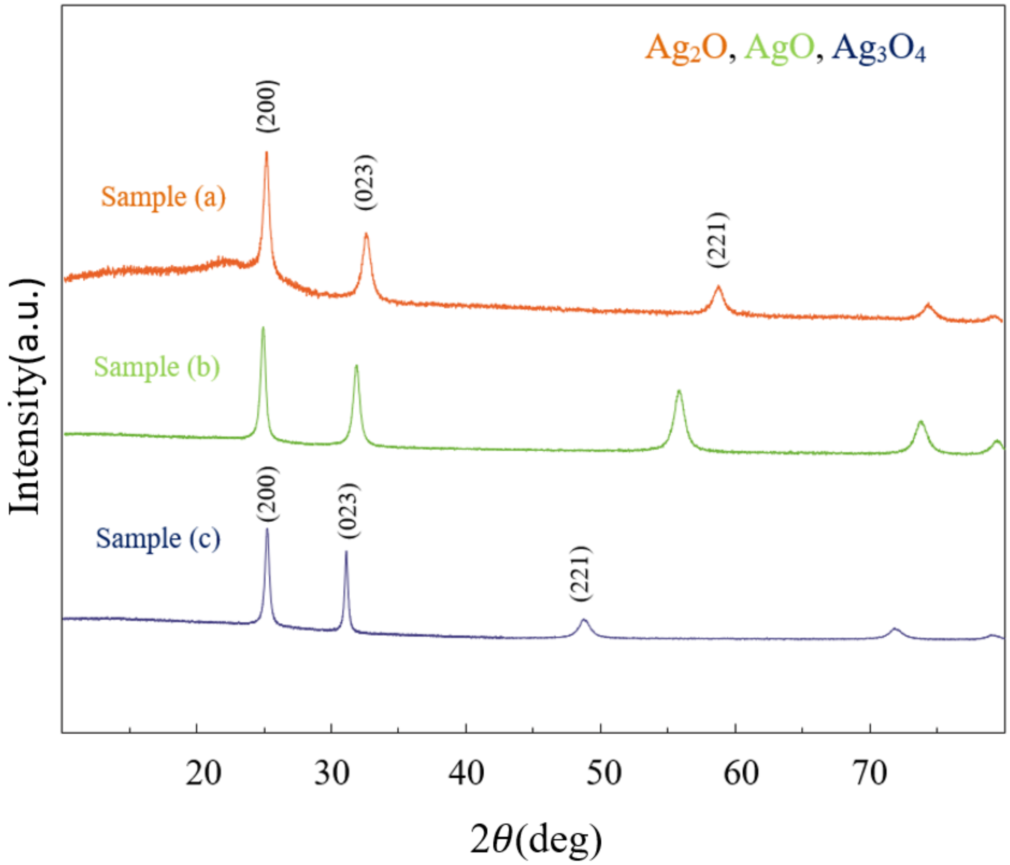
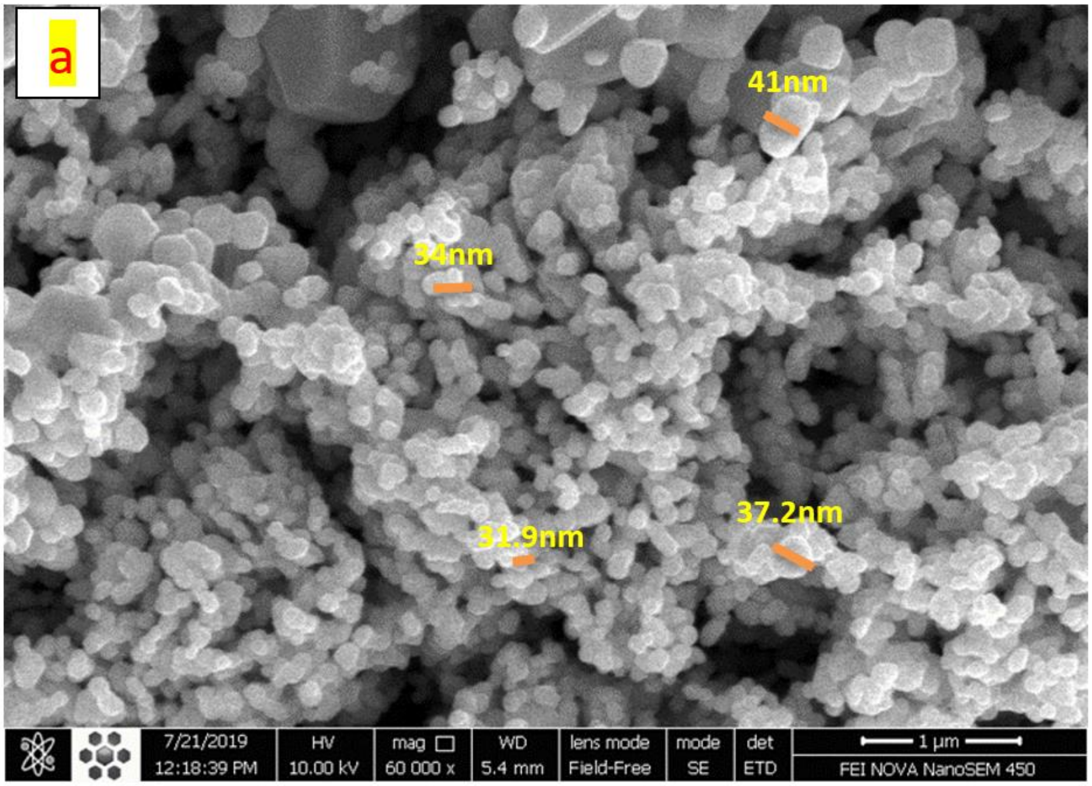


Figure 1.
The XRD patterns of the deposited nano-silver oxide thin films.

Figure 2 is showed the results of FESEM examinations of the surface morphology of the samples used showed that the Ag_2O particles formed a nanostructure arranged in a geometric manner and in the form of layers on top of each other. Upon further magnification, it was observed that the grain size ranged from (31.9 to 41) nm, and this corresponds to the XRD examination of the samples that were examined. Note that there are interstitial distances between the nanoparticles and these distances are very large when conducting a gas sensing test on samples[12].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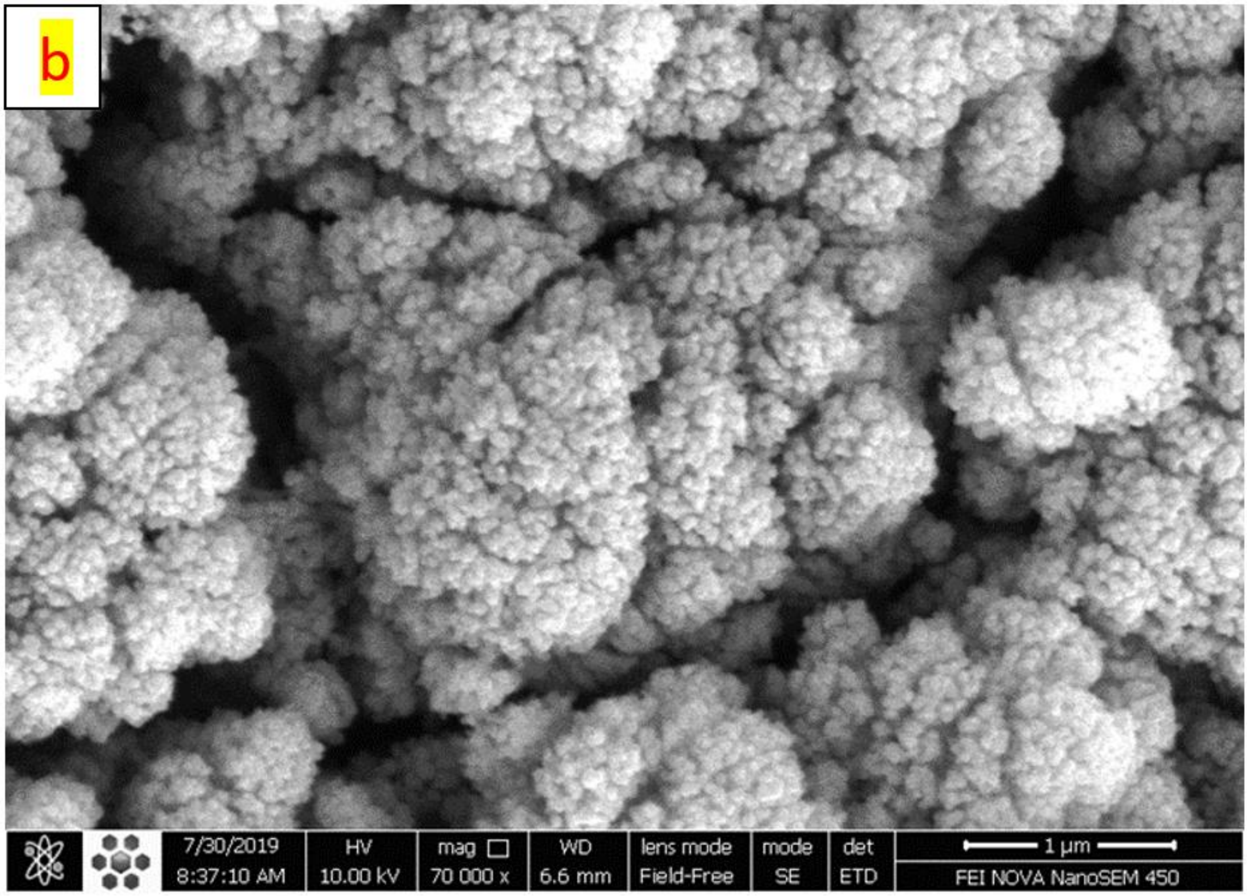

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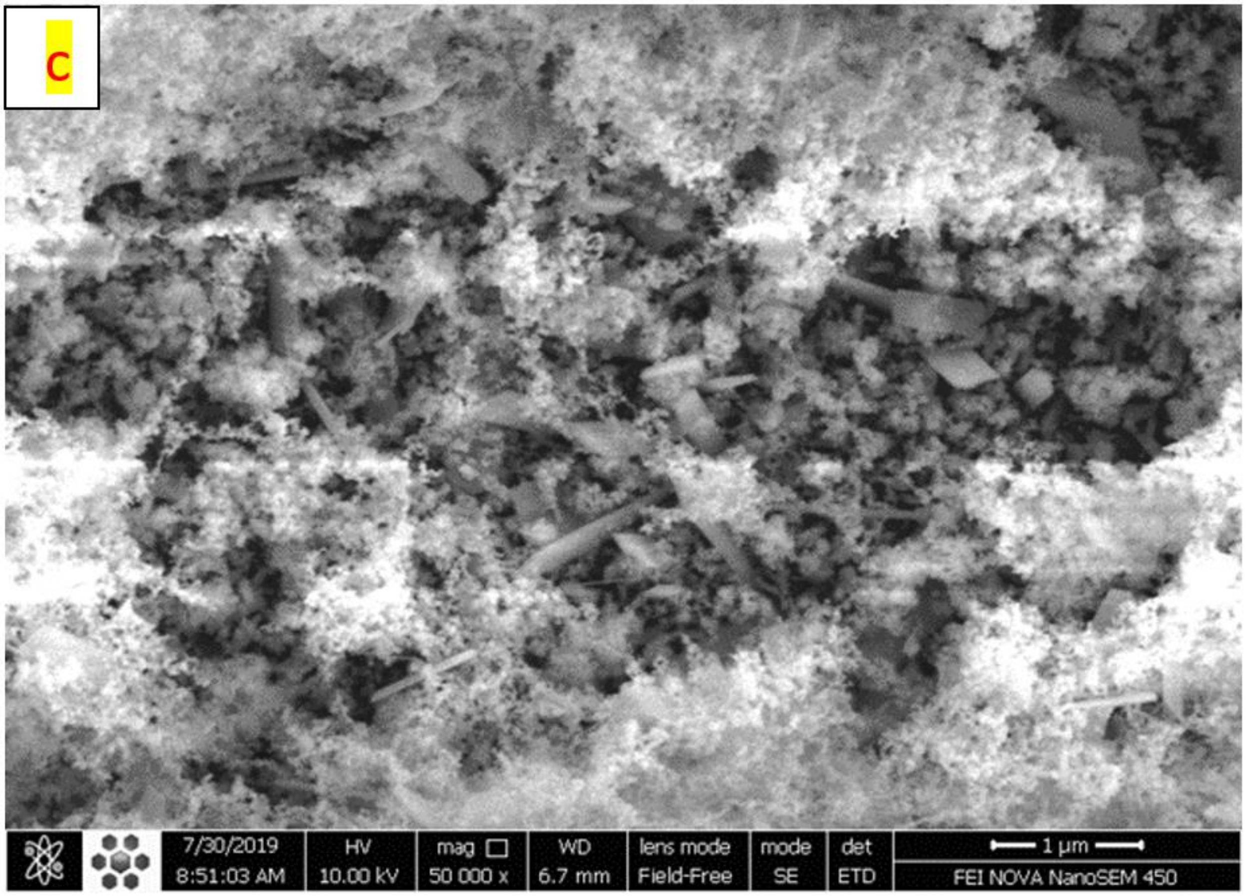

Figure 2.
FESEM images of Ag_2O structures deposited on quartz glass.

When conducting an AFM examination of Ag_2O nano-silver oxide samples and the particle size distribution. It was noted that the surface roughness and particle size that made up the surface of the thin films were primarily dependent on the parameters that were adopted in preparing the samples, which are pH and time t. Through Table 2 which displays the RMS surface roughness as well as the average particle size, it ranged from (57.1 to 87.54) nm. Depending on the deposition parameters, we notice a change in roughness for the thin films of pure Ag_2O nanoparticles between smooth and rough, which is related to the movement and growth of the thin films. Such thin films can be used in many fields, such as gas sensing, for example sample (c), as well as in the field of electronic applications, for example smooth sample (a) [13].

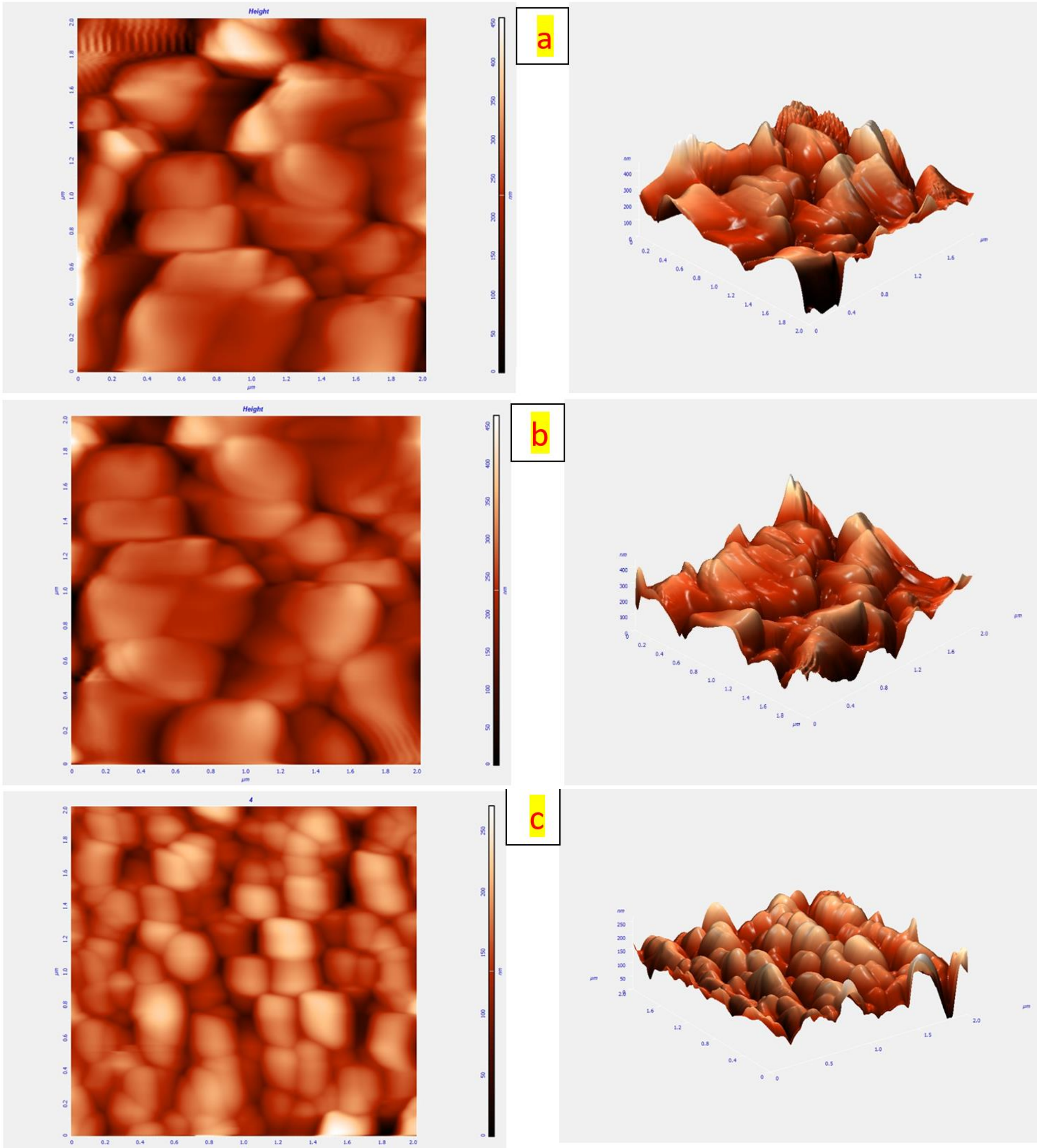

Figure 3.
AFM images of silver oxide thin films at different preparation parameters.

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Table 2.
XRD and AFM measurements of the prepared silver oxide films.

	XRD	AFM		
Sample	**Crystallite size (nm)**	**Ra (nm)**	**RMS (nm)**	**Average grain size (nm)**
a	38.11	0.91	0.94	57.1
b	36.43	4.07	4.71	73.23
c	33.21	3.17	3.55	87.54

3.1. Gas Sensor Characterization

The sensitivity of samples prepared from Ag_2O nanoparticles to 450 ppm of NO_2 gas was measured according to the equation...

$$S = \frac{Ra - Rg}{Ra} x100\% \qquad (2)$$

Where Ra is the air resistance and Rg is the gas resistance, respectively. From Figure (4), we notice the change in sensitivity over time from samples that have a high response due to the change in the pH solution. The sensitivity, response time and recovery time with temperature were recorded and determined for the operating sensor and for all prepared samples. As shown in Table 3 From Figure (4) and Table 3 we can notice that the thin films of Ag_2O did not respond to 450 ppm of NO_2 gas when the temperature was 25°C, that is, at room temperature. But on the other hand, we see that the sensitivity has increased well at 50°C. The explanation for this is that the sensors operate at low temperatures and have good sensitivity values. The change in good sensitivity is due to the change in the pH of the solution, which is related to the surface morphology, as well as the particle size and also the thickness of the thin films. Given that the sensitivity is a function of the particle size. Therefore, a decrease in particle size will lead to an increase in surface sensitivity. This increase is important because it leads to an increase in adsorption. For this reason, the sensors prepared for this work showed very good performance. According to the source [14] the good sensitivity obtained for the Ag_2O gas sensor was due to the grain size, which was rather small. We note that sample (b) had a low response time (0.93) sec and also had a low recovery time (0.97) sec. This may be due to its good compositional properties.

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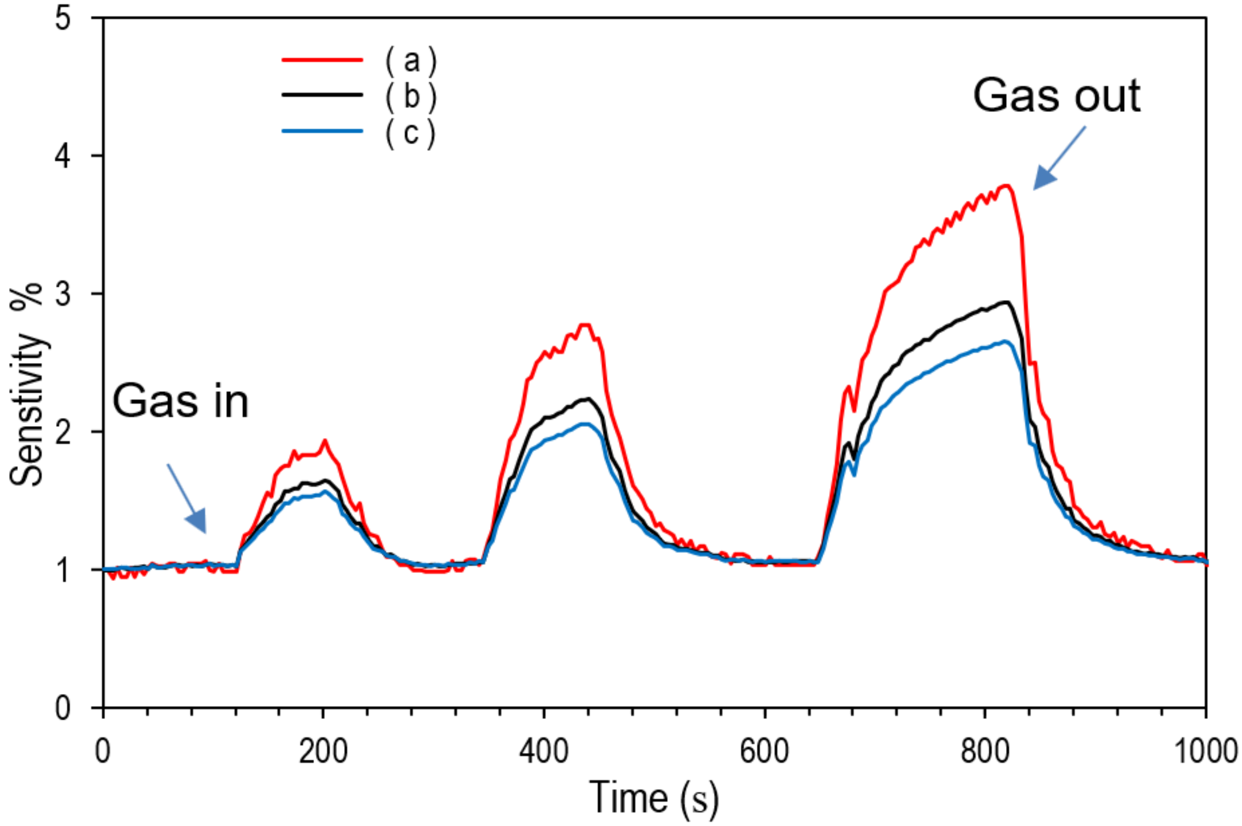

Figure 4.
Sensitivity of the optimal Ag_2O thin film with different operating time.

3.2. Values at450 ppm NO_2 gas.

Table 3.
Values of sensitivity, response and recovery time with respect to temperature at 450ppm NO_2 gas.

Sample	Temp (°C)	Sensitivity	Response Time (sec)	Recovery Time (sec)
	25	0.00	0.00	0.00
	50	7.11	1.70	0.92
a	75	72.20	11.01	1.09
	100	8.11	1.83	1.57
	25	0.00	0.00	0.00
	50	66.70	0.93	0.97
b	75	10.30	11.89	2.09
	100	1.80	1.85	0.88
	25	0.00	0.00	0.00
	50	1200	9.00	1.50
c	75	10.75	11.89	4.44
	100	0.45	2.55	10.01

From Figure 5 we notice the variation of operating temperatures with the sensitivity value. It can be noted that sample (b) works at pH, which is equal to 11, and at a somewhat low temperature, which is equal to 50°C with high sensitivity. While it can be observed that sample (c), which was prepared at a

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pH of 12, works with a low sensitivity value at a temperature of 50°C. For this reason, we believe that the best bathing condition for obtaining the best gas sensor is when the pH is 11, the bathing temperature is 75°C, and the immersion time is 40 min.

The mechanism of sensing the action of silver oxide Ag_2O includes trapping oxygen in the form of O^- or O^{-2} on the roof. This leads to an increase in resistance relative to the oxygen surface. When NO_2 gas interacts with the surface of Ag_2O or with the absorbed oxygen electrons that are released from the oxide surface, we notice that there is an increase as follows [9].

$$4NO_2 + 3O^-_{2\,(ads)} \rightarrow 2N_2 + 6H_2O + 6e^- \quad (3)$$

$$2NO_{2(ads)} + 3O^-_{(ads)} \rightarrow N_2 + 3H_2O + 3e^- \quad (4)$$

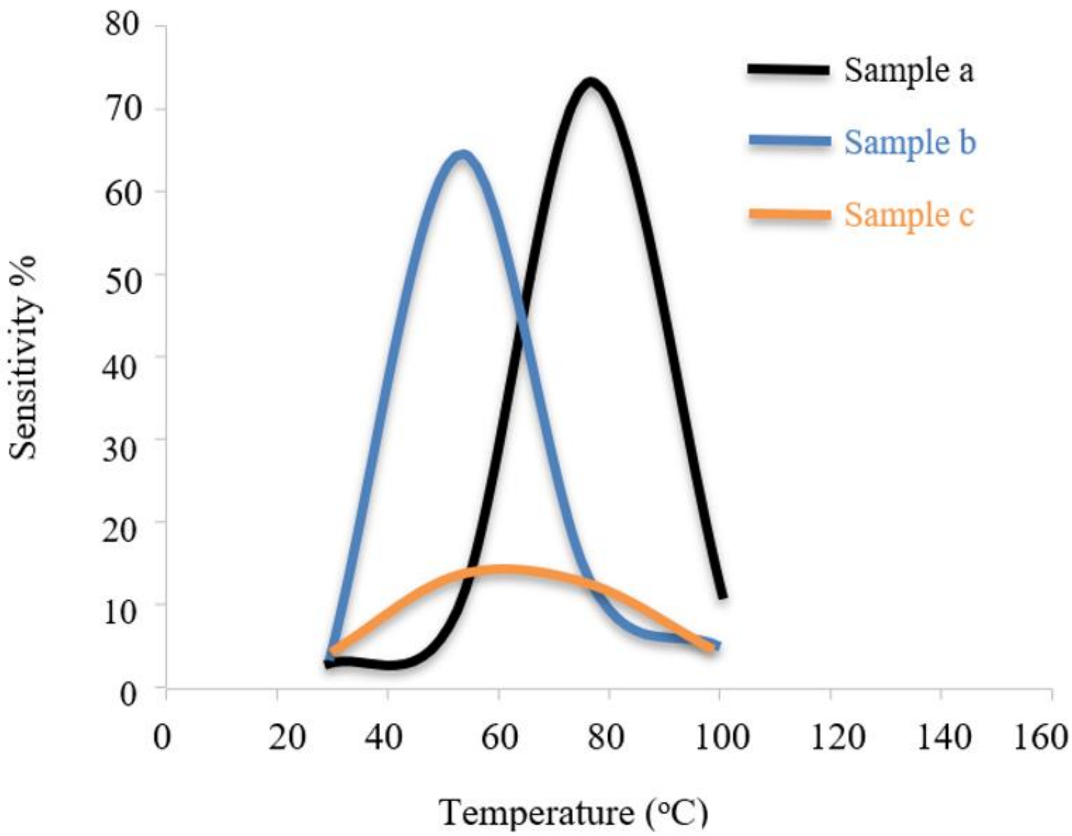

Figure 5.
Changing of the sensitivity with temperature at different pH values at 450 ppm NO_2 gas.

The reaction requires activation energy in order to continue, and therefore thermal energy must be provided. For this reason, we note that silver oxide nanoparticles do not have a response at room temperature. If the temperature is not increased. We note that the maximum degree of response to the gas is achieved when good and actual thermal energy is given to the reaction, and we also note despite this. There is a decrease in responsiveness when there is an increase in operating temperature. There is absorption and adsorption of oxygen on the surface of the sensor [15, 16].

4. Conclusions

In this work, thin films of Ag_2O nanoparticles were prepared using (Chemical bath deposition). XRD examination showed that the thin films had different compositions of (Ag_2O, Ag_3O_4 and Ag_2O). The average grain size as well as the average crystallite size measured by XRD and AFM are within the nanometer range. The Ag_2O nanomembranes had good sensing properties for NO_2 gas at 50°C, which is somewhat close to room temperature. Noting that there is a high and good gas sensing value and a low response time and recovery time. From these results, we can see the possibility of using Ag_2O nano thin films in many applications, including gas sensors as well as electro-optical applications.

Transparency:

The author confirms that the manuscript is an honest, accurate, and transparent account of the study; that no vital features of the study have been omitted; and that any discrepancies from the study as planned have been explained. This study followed all ethical practices during writing.

Acknowledgement:

Thanks, and appreciation to the Department of Physics at the College of Education at (Mustansiriya University), Baghdad, Iraq for supporting this work.

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