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Structural, Optical and Gas Sensor Properties of Zinc Oxide Nanostructured thin films prepared by Chemical Spray Pyrolysis

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Abstract:
ZnO Nanostructured films were formed by chemical spray pyrolysis technique on stander glass (Made in China) substrate at (450°C). The structural and optical properties were studied in this research. The samples are a compound of pure (ZnO) with a hexagonal structure. X-Ray examination showed showing that the lattice parameters a= 3.286 A° and c= 5.311 A°. The average grain the size of crystals is on the order of (55nm), studying the topography of the surface by SEM, it appears that the growth of the thin film is very small nanoparticles, and there are also nanowires in it, which increases the region of the thin film's surface. From the sake of studying the optical properties, Also, the absorbance factor was calculated for sample. energy gap for (ZnO) appeared to be about (3.25 eV), with direct band transitions. NH_3 gas was used to measure the sensitivity of the thin film used in the study and the thin film was shown to have a high sensitivity to the gas.

Keywords: ZnO, XRD, SEM, Gas sensor.

1. Introduction:

Semiconducting oxide nanostructured films can show good properties and are more sensitive than bulk. (ZnO) is one of these important oxides from a technological as well as industrial point of view, because it has a wide range of optical and electrical properties [1]. It has a lot of applications that have to do with sensing gas and toxic chemicals, as well as transparent electrodes, laser diodes, and light-emitting UV devices, Surface acoustic wave (SAW) devices, and so on. Important in our daily life [2,3]. The crystal structure of (ZnO) contains nanowires, nano arcs, nanobelts, and nanocages. In addition to all this, the ZnO has been proven to be environmentally friendly [4,7].

The current study aims to prepare nanofilms of (ZnO) by thermal chemical decomposition, which included the study of structural properties including surface topographic measurements represented by electron microscopy measurements (SEM), in addition to the study of optical properties that included electronic transitions and optical constants and to obtain a nanostructured films with good specifications and improve its properties in the visible spectrum and infrared region because of the practical applications of these two regions in solar cells. and reagents and others.

2. Experimental Work:

To prepare the solution used in the preparation of (ZnO) thin films by chemical spray pyrolysis technique, zinc nitrate and its chemical code $Zn(NO_3)_2$ were used, which is a white powder quickly soluble in water, and the solution was prepared with a molar concentration (0.1 mol / L), by adding (3.5058 g) of it in 100 ml) of distilled water, and to obtain the required weight to be dissolved within the previous standard, the following relationship was used [8]:

$$\mathbf{M = (W_t / M_{Wt}).(1000/V)}$$

Whereas: M: molar concentration. WT: weight to be thawed. MWt: molecular weight of the material. V: the volume of distilled water in which the dissolution was made. The solution is mixed using a magnetic stirrer for a period of (10-15) min, and after completing the dissolution process, a colorless clear solution is obtained.

The crystal structure of each of the prepared samples is determined through an X-Ray device (XR-DIFRACTOMETER/6000) Shimaduz type of Japanese origin with the following specifications (Target : Cu – Kα, Wavelength : 1.5406A°, Voltage : 40 Kv, Current : 30 mA, Range (2Ө) : 20 – 60 deg) .Then the size and shape of the nanoparticles are measured through a scanning electron microscope (FESEM) manufactured by (TEASCAN) and the factory is from a company (UEGA.LM) of Jiki origin and with effort Acceleration is about (5Kv).

Visible and ultraviolet spectrum calculations for (ZnO) films were measured and recorded by a device. To find out the characteristics of the sensor element used for gas detection was placed in a well-sealed hall with a size of (250 ml). A predetermined the introduction of a gas concentration into the hall with a syringe, sensor has been tested for different gases, which are (CO_2, NH_3 and NO).

3. Thickness Measurement of Thin Films:

The gravimetric method was adopted to measure the thickness of the prepared thin film and the thickness is calculated in this way as follows:

The bases are weighed before the thin film is deposited on them, and the weight is re-weighed after the completion of the deposition process has been used for this purpose a sensitive balance of the type (DENVER) with sensitivity (4-10µg) and from knowing the area and density of the deposited material can calculate the thickness of the thin film from the following equation [8]:

$$t = \frac{m}{D.a} \quad \text{.......... (1)}$$

Where t: thin film thickness (cm), m: thin film mass (gm), D: density of thin film material (gm / cm^3), a: thin film area (cm^2) for the thickness of zinc oxide thin film (ZnO) prepared in this study was about (350 nm).

4. Results and Discussion:

Through the X-ray diffraction patterns of (ZnO) films, which we can observe from (Fig. 1), all the information about the crystal structure, particle size, and intensity d of the intended diffraction digit with the crystal shape of (JCPD card no. 036-1451) Hexagonal (ZnO). The presence of diffraction inertia of other contaminants was not observed. The parameters of the lattice pertaining to the number d were calculated as follows: a= 3.286 A° and c = 5.311 A°. It was noted that there are capillary parameters, and the reason for this is due to the sample being infinitely small, of course. The average age of the crystal size was determined by the relationship:

$$D= \frac{0.89\,\lambda}{\beta \cos \theta} \text{ .... } (2)$$

where, D is the crystallite size, β is full width at half maximum (FWHM) measured in radians and h is the Bragg angle. λ is wavelength of X-ray The average crystallite size is (55 nm).

By studying the surface morphology of the (ZnO) thin film surface and at a magnification of (5.00) kx and (5.03) kx in (Fig. 2). We notice that the growth of the thin film occurs in the form of a folded structure. This increases the surface area of the thin film and thus we benefit from the surface area. These surfaces are used in a gas sensing application. It is also observed from the figure that the (ZnO) nanoparticles are evenly distributed, with a grain size distribution of (55.85) nm. The typical particle dimension is about (65) nm, and these results agree with the XRD calculation.

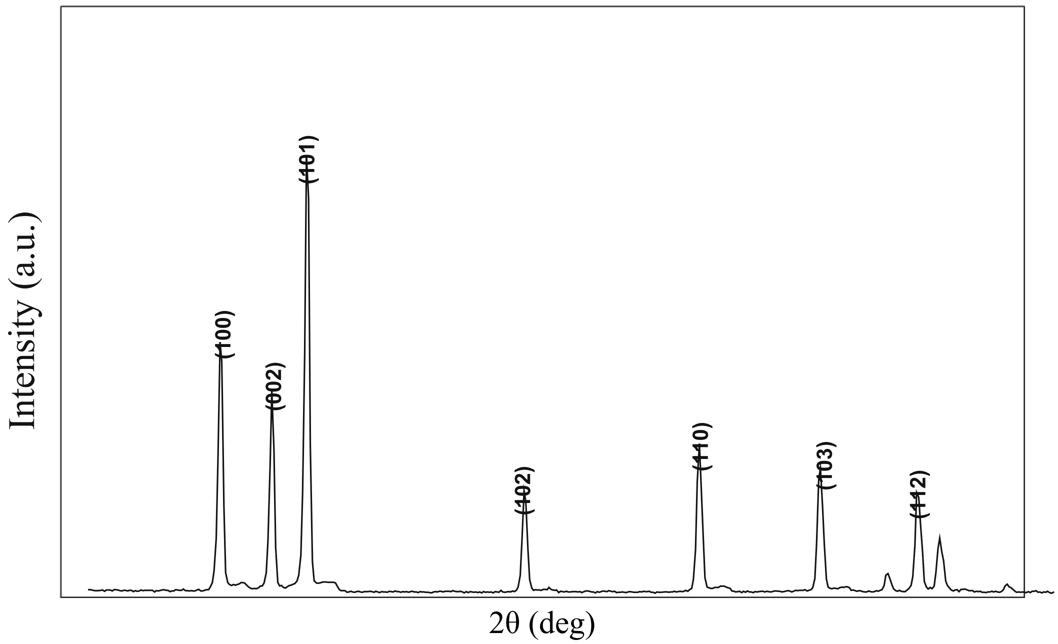


Figure. 1. Thin film (ZnO) X-ray diffractogram.

Spectrum of optical absorption of (ZnO) sheet at wavelengths between 350 and 800 nm at RT. From the observation of (Fig. 3), there is a sharp UV absorption edge at (395 nm). It was investigated to note the absorbency factor a and suddenly the forbidden energy as well as how the change will be made. The absorption coefficient is quite high, it was discovered (106 cm^{-1}) and that each of the absorption coefficient (α) and the energy of the photon (hυ) and energy gap (Eg) are related through the following relationship [9-10]:

$$\alpha h\upsilon = A\,(h\upsilon - Eg)^{n/2}$$

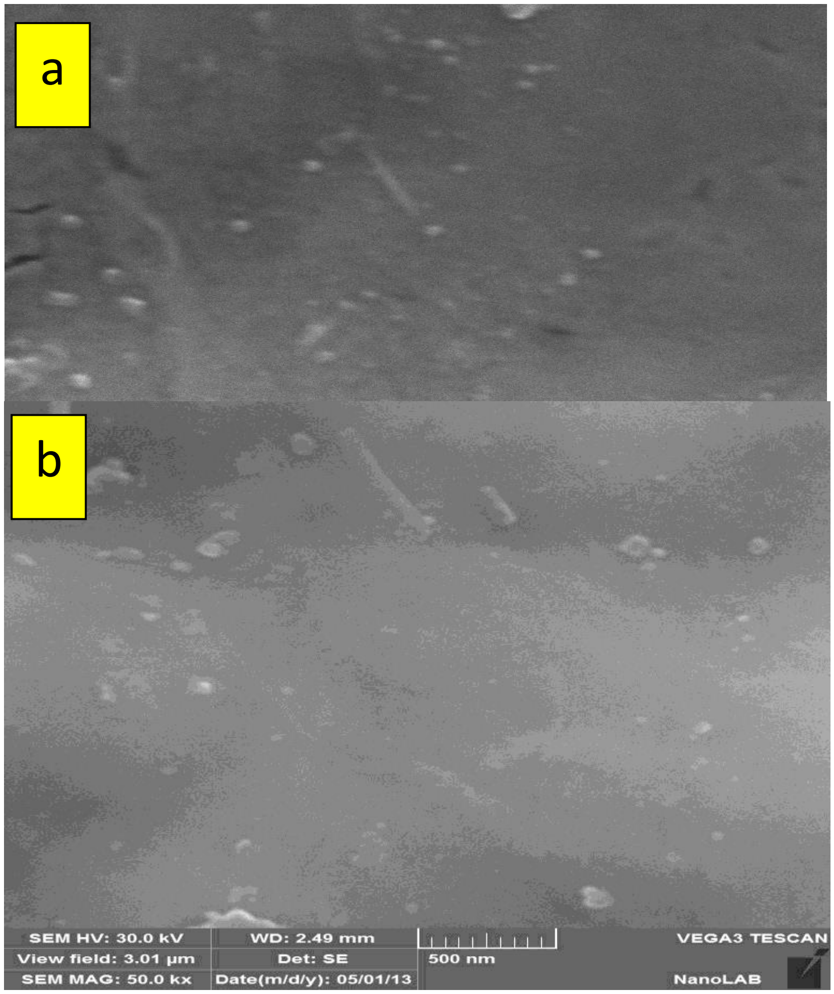


Figure. 2. shows SEM pictures of a thin ZnO sheet at various magnifications. (a) 5,00 kx and (b) 5.03 kx.

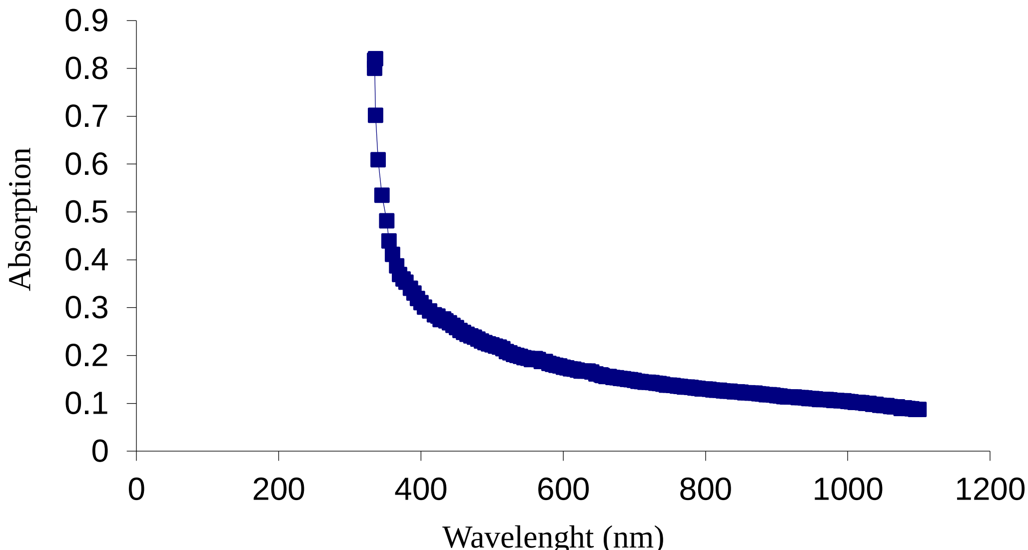


Figure. 3. Optical absorption of (ZnO).

We assume that the allowed direct transmission is (n = 1). Then calculate the energy gap from (αhυ) vs. (hυ) (Fig. 4). It appears that curved (αhυ) vs. (hυ) are on the side of higher energy, straight line behavior which is confirmed to be the type of direct transitions in such films. It was found that the energy gap of the (ZnO) thin film is (3.25 eV) and this value corresponds to the energy gap perfectly with [11-12]. The (ZnO) thin film gas sensor has been tested for each of the following gases: (CO_2, NH_3 and NO) after stabilizing (ZnO) opposition to the surrounding air before exposure to the gas, which is critical to ensuring a stable zero level for gas sensing applications. Before exposure of the surface of the membrane to the gas, which is allowed to be stable with respect to electrical resistance during the course of (25) minutes. find out the characteristics of sensor, the (ZnO) membrane is exposed to a concentration of (125 ppm) of the used gases (CO_2, NH_3 and NO), and its resistance is recorded. These measurements were made at room temperature. It has been a drop in (ZnO) resistivity has been seen. when exposed to (NH_3, NO) gases, on the other hand, it has been observed that its resistance increases when exposed to (CO_2) gas. By observing (Fig. 5), we notice a response to gases (CO_2 ,NH_3 and NO) from the basement good degree.

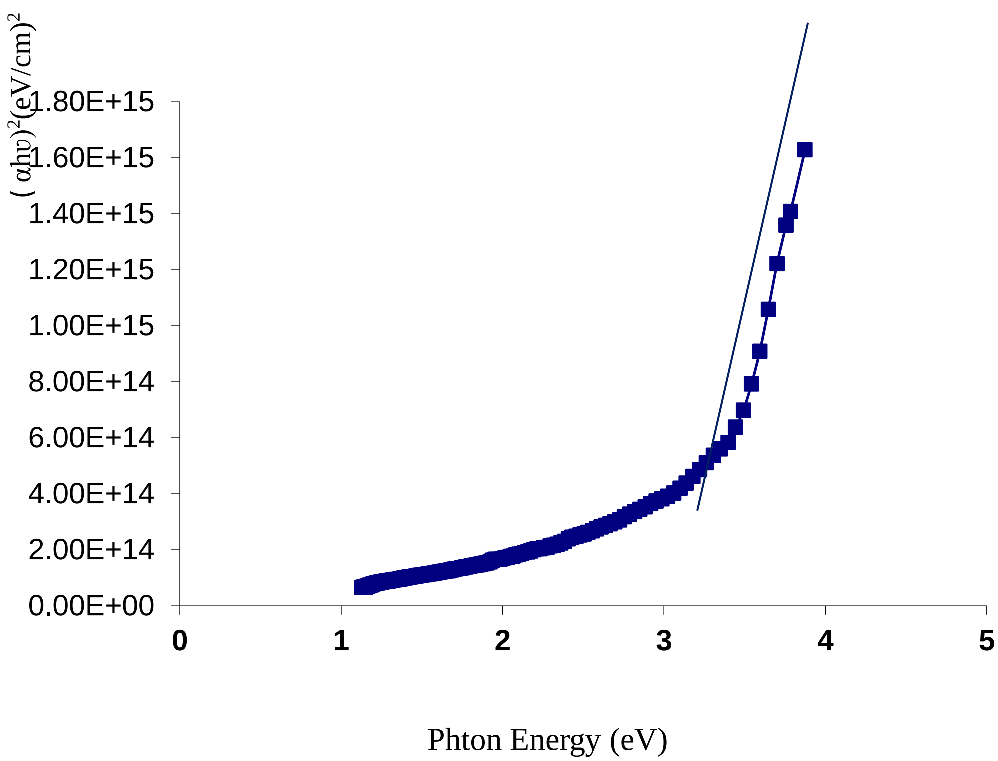


Figure. 4. A diagram representing the energy gap as a function of photon energy for a thin film

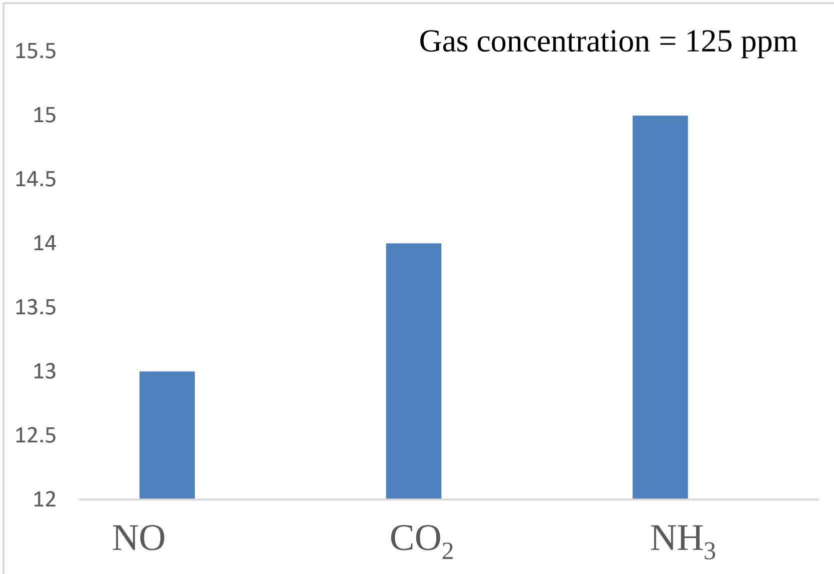


Figure. 5. Response (ZnO) sensor for several gases at RT.

From the excellent sensitivity of (NH_3). it can be seen that the (ZnO) thin film is selective for (NH_3). This article will describe how gas sensing works. by conduction, either through the absorption surface-level atmospheric oxygen, or by the direct the oxygen in the lattice interacts with the used gas, good response to selective (NH_3) gas a sensor's performance it is possible to describe the interactive processes that occur on the surface [13-14]. Where the (O_2) molecules present in the atmosphere are absorbed from the sensor's surface in the shape of (O^-) and ($O2^-$) as these electrons take the molecules in n-type semiconductors' conduction band and are absorbed as O^-_{ZnO} in this case the material shows a resistance that is high in the air that surrounds it and through the following relationship:

$O_2\ (g) + 2e^- \longrightarrow 2\ O^-_{ZnO}$ ………… (3)

When (NH_3) gas molecules interact, the oxygen, which is negatively charged, and through an adsorption process, electrons, which are trapped, are returned to conduction band the (ZnO) membrane. The energy emitted during the decomposition process of (NH_3) gas the probability of electrons entering the conduction band of molecules is low (ZnO). and this leads to an improvement in the sensor's conductivity [15]. There will be a reaction which is:

$2NH3 + 3\ O^-_{ZnO} \longrightarrow 3H_2O + N_2 + 3e^-$ …………… (4)

In Figure (6) here the resistance's current state value. is stable and varies with concentration of (NH_3) gas. For the purpose of knowing the effect of the working temperature, the special element of the sensor is exposed to a concentration of (125 ppm) of (NH_3) gas, and during this time the response is recorded in the temperature range (100-450^{o}C). By observing (Fig. 6) which shows the sensor's response to temperature, we see that the response has increased with an increase in temperature until it reaches a maximum of (300^{o}C). Note that it decreases further. The reason for explaining this behavior is as follows: At temperatures that are low, we can expect a low response, considering that the gas molecules might not be thermally active, which is sufficient to interact by using types of oxygen that are low in the surface. With an increase in temperature, there is a rather high thermal energy to be sufficient to pass the potential challenge. An elevated level of electron concentration results based on the sensor's next response. It can be seen that at high temperatures the response of the sensor is restricted in the diffusion velocity of the gas molecules. Some average temperatures can also be observed. Here we note that the speed value for both processes is equal. Thus, the response of the sensor at this point may reach its maximum [16]. The optimum operating temperature for the (ZnO) membrane is (300^oC) at which it can be observed that the sensor response has reached its highest value.

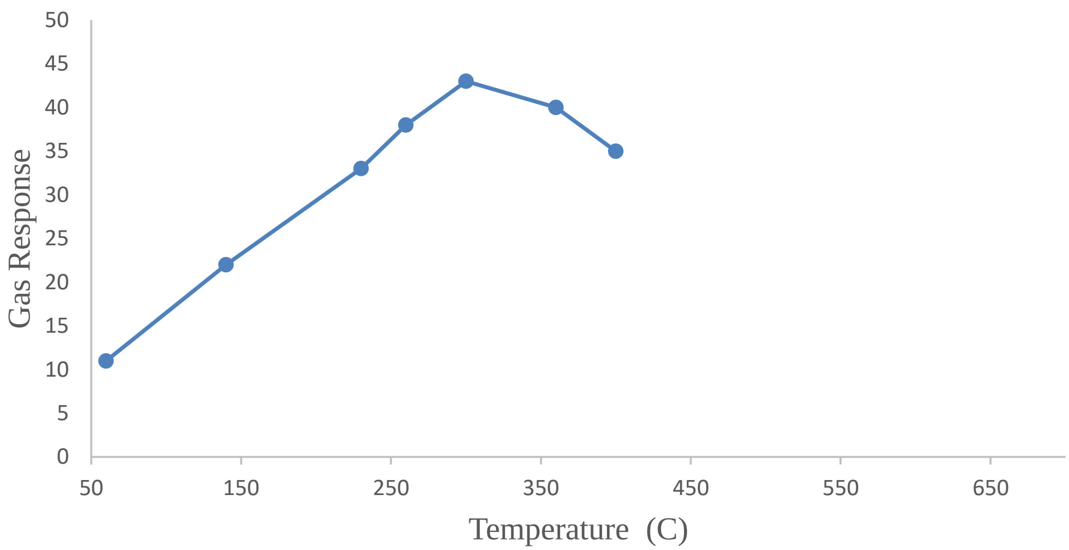
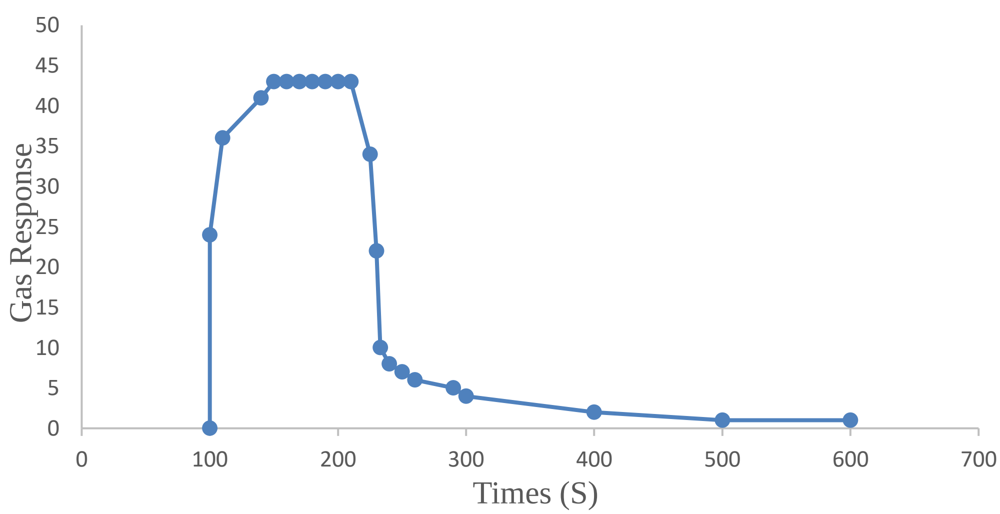


Figure. 6. Impact of temperature on (ZnO) sensor sensitivity to (NH_3).

(Fig. 7), shows the response of the sensor in relation to the time after exposure to (NH_3) gas at a concentration of (125 ppm). We notice increases in conductivity and speed of the sensor to (NH_3) and also note that it recovers towards the original value after the process of introducing pure air. This indicates that the sensors have excellent capabilities, which are metal oxide nanoparticles with high sensitivity. From calculating the sensor's reaction and recovery times were discovered to be (27-82) seconds, respectively.



In (Fig. 8), we notice the sensitivity variation with the concentration of ammonia gas. The gas concentration was in the range of (25-260 ppm). It has been observed that the as gas concentration rises, sensitivity rises practically linearly until (125 ppm) eventually veers off course. There is a monolayer of gas molecules at low gas concentrations. forms ink is present on the sensor's surface, which has a more active interaction, which gives a linear response. When the gas concentrations are high, the many layers relative to the gas molecules are saturated [17].

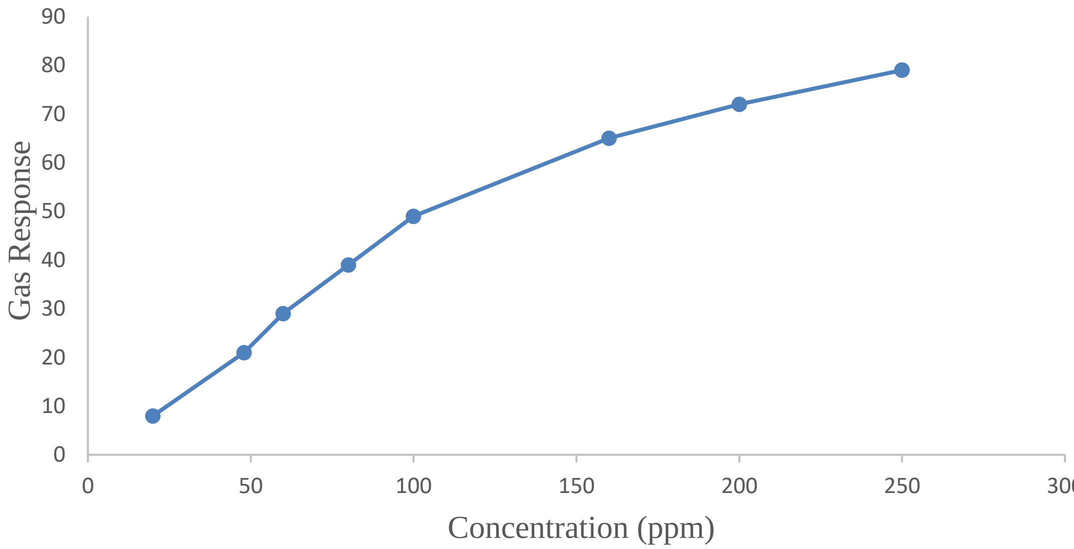


Figure. 8. Response of the (ZnO) sensor at different (NH_3) concentrations.

5. Conclusion:

A thermochemical decomposition technique was used to synthesize nanostructured (ZnO). Thin films. The fitted sample is of pure (ZnO) with hexagonal structure. The parameters of the lattice were a= 3.286 A° and c = 5.311 A°. The average crystallite size was approximately (55 nm). The surface morphology was studied by SEM, and the distributed average particle size was about (65 nm). UV measurements showed us that there is an absorption edge of about (395 nm). When studying the membrane sensor for the used gas, which is (NH_3), the sensing properties showed that (ZnO) is sensitive and also has a fast response to (NH_3) gas. The excellent sensitivity of this gas indicated that (ZnO) is selective for this gas. The response time was (27 s) and the recovery time was (82 s). We can explain this mechanism of sensing depending on the chemical adsorption that occurs for surface-level atmospheric oxygen and the ensuing interaction between the used gas and the oxygen. Thus, obtaining improved conductivity of the sensor.

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Conflicts of Interest Statement…………………………………………………

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Ameer I. Khudadad		**27/2/2025**