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A New Piezo-Amperometric Sensing Method Based on Comb-like Nanostructured Zinc Oxide Thin Films for the Efficient Detection of Na_2SO_4

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Abstract

A chemical vapor deposition (CVD) was employed to deposit ZnO thin films on two different substrates, glass (quartz) and silicon. The characterization of structural properties of the comb-like nanostructure ZnO thin films were carried out through X-ray diffraction (XRD), atomic force microscopy (AFM) and scanning electron microscopy (SEM). Nanostructured thin film sensor has been fabricated to detect the Na_2SO_2 aqueous solution concentrations. The obtained films are strongly c-axis oriented along (100) direction and have other low intensity towards the directions (101), (200) and (211), indicating polycrystalline characteristics. The film exhibits high dense comb-like nanostructured distribution which has been indicated in the SEM images, AFM images depicted the film porosity, the film deposited on the glass substrate has more pinholes and porosity than that deposited on the silicon substrate. A new sensing method has been established depending on the piezoelectrical properties of the ZnO material and the electrochemical interaction with the salt. Furthermore, the Na_2SO_4 sensing properties of the ZnO thin film were investigated for various salt concentrations (100, 200, 300 and 400) mg/L. The nanostructured ZnO thin film exhibits higher response, very fast rise time and acceptable recovery time upon working at room temperature. The relative response of ZnO film linearly behaves with the salt concentrations biomedical sensor was obtained. In this work, the results of detailed

Keywords: *Chemical Vapor Deposition, ZnO thin films, Salt sensing, Amperometric biosensors.*

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1. Introduction

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Recently, different kinds of metal oxides materials as (ZnO, TiO_2, SnO_2, CdO, WO_3) have been used in the many different applications [1-4]. Among them, ZnO is a candidate multifunctional material has a wide and direct band gap (3.37 eV) with a large exciton binding energy (60 meV) [5-8], it is recognized as a promising and interesting in biocompatible [9,10], optoelectronics [11-13], piezoelectric properties [14-17]. ZnO thin film has been synthesized by different type techniques such as chemical bath deposition (CBD) [18,19], chemical vapor deposition (CVD) [20-22] thermal evaporation [23-25], pulse laser deposition (PLD) [26-28], sputtering [29-31], spray pyrolysis [32,33], sol gel coating [34,35], ZnO nanostructured materials has an attractive for biomedical sensing applications due to different types of nanostructured thin film synthetization as; nanoparticles [36], nanorods [37], nanowires [38,39], nanosheets [40, 41]. Nanostructured ZnO thin film has been used in many applications as, transparent conducting electrodes [42], Li batteries [43], optoelectronic devices [44], toxic gas sensors [45-47], heat reflecting mirrors [48], aqueous salts solution [49]. Chemical vapor deposition (CVD) is an attractive technique for the growth of ZnO due to its simplicity and produce high dense nanostructures. Several different metal-oxide systems have been utilized as gas and biomedical sensing materials, such as tin oxide (SnO_2)[50-53] , tungsten trioxide (WO_3)[54,55], titanium oxide (TiO_2)[56,57] and zinc oxide (ZnO)[58,59], ZnO thin films are nanomaterials suitable for gas and biomedical sensors because the sensing properties are related to the material surface where the gases are adsorbed and surface reactions occur, rather than the biomedical sensing depends on the electrochemical and piezo electric properties. Surface reactions change the concentration of charge carriers in the material, creating a depletion layer and surface dipole at the interface, which results in a change in electrical resistance [60].

The aim of our presence work is to construct a new set-up device as salty solution detection. The objective of the work focuses on the prepared nanostructured ZnO thin film via CVD technique through optimum conditions and examined these prepared films to the Na_2SO_4 solution at different concentrations bases on the electrochemical reaction in combined with the piezoelectric properties of ZnO material.

2. Experimental

2.1 Preparation and sensitization

The ZnO thin films prepared by deposited ZnO on the quartz-glass and silicon substrates using CVD. The basis for the CVD system is given by a three zone furnace which can heat up to 1100 °C. The reaction chamber is a quartz glass tube with a diameter of 5 cm and a length of 150 cm. On the left side are three gas inlets, whereas only two were used and equipped with a flow meter. One inlet was for the transport gas argon, where a constant flow of around 100 standard cubic centimeters per minute (sccm) could be adjusted. The unit sccm is representing how the flow rate would be under room temperature condition and atmosphere [61].

The other inlet was for the reactive gas oxygen where the flow was controlled to 0.5 to 10 sccm. On the right side of the tube a pressure control unit was connected to a vacuum pump which assured a gas flow from left to right as shown in the fig.1.

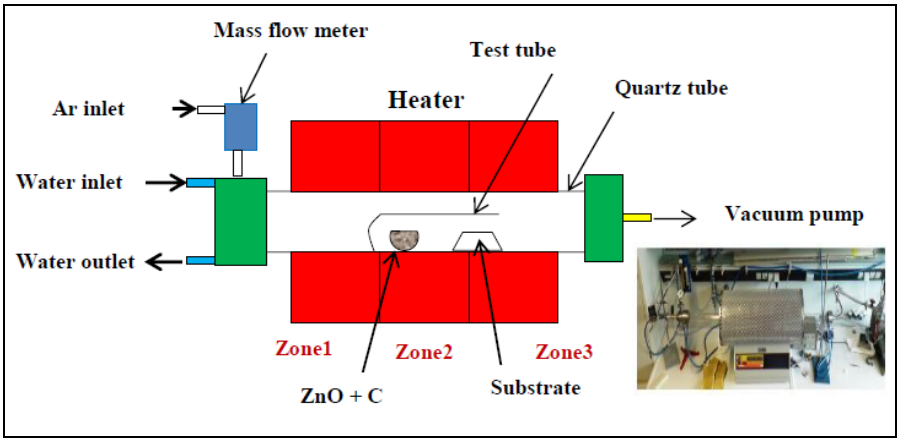


Fig. 1: Three zone tube furnace for chemical vapor deposition of nanostructured ZnO thin films.

So a small test quartz tube with a diameter of 2 cm and a length of 20 cm inserts in the middle region of the reaction chamber. At the closed end of the tube a small quartz boat was placed which was loaded with a molar 1:1 mixture of zinc oxide and carbon nano powders. To obtain the correct molar ratio, the source was prepared by mixing 1 g of carbon nano powder with 6g of zinc oxide nanopowder. The open end faced to the right to decrease the influence of the direct gas flow and thereby the oxygen concentration in the tube evens more.

The electrodes were thermally evaporated on the upper surface of the prepared films employed a finger mask, Figure 2 illustrates the prepared sensor

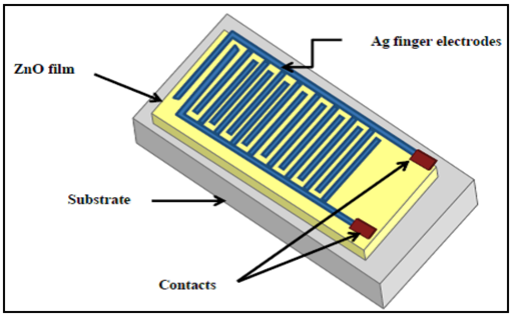


Fig. 2 Prepared ZnO thin film sensor.

The deposition parameters for the nanostructured ZnO thin films by CVD method are describe in the table 1.

Table 1: The Deposition Parameters

Deposition parameter	Data
Reaction chamber (quartz tube) dimension	ϕ(5 cm) x L (150cm)
Zones temperature	900 – 900 – 700 (°C)
ZnO : C (nano powder)	1g : 6g
Ar flow rate	100 sccm
Test tube (quartz) dimension	ϕ(2 cm) x L (20cm)

The sensing response characteristics of ZnO nanostructured thin films were studied using the silver finger electrodes deposited on the upper surface of the sensing layer. Fig. 3.illustrates in details the schematic diagram of the sensing setup.

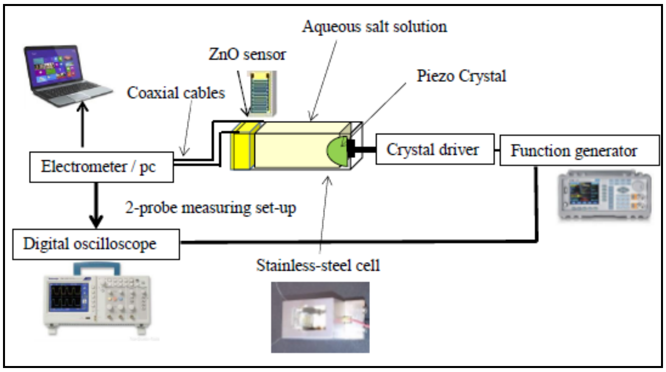


Fig.3. A schematic diagram for measuring the sensing properties of the fabricated ZnO thin film salt sensor.

The main target of our work is to design and construct a new setting for bio medical sensing achievements. The mechanism of our setting basis on the two main topics: A piezo-electric property of the sensors material and the other topic is the electro-chemical interaction of that material with the salt solution. The heart of the sensing setting is the solution cell. It is made of stainless steel filled with the solution from the upper side, the piezo crystal mounted on one side and the sensor could be mounted on the other side by inserted it in the groove. Fig. 4 illustrates the sensing cell.

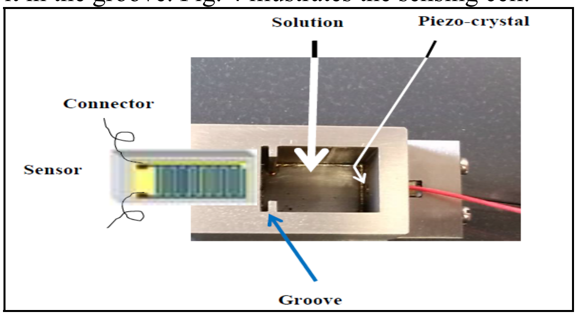


Fig. 4: Piezo–amperometric sensing cell

2.2 Characterization

Crystalline structure and surface morphology of the sensing layer were studied using (2θ) scan of a X-ray Diffractometer using the CuKα1 source (λ = 1.54 Å). In order to investigate the results of the experiments an electron microscope (SEM) and atomic force microscope (AFM) are needed, because the achievable dimensions can go down to several nanometers, which can't be seen with a traditional light microscope. Therefore a scanning electron microscope was used which uses electron instead of light and electromagnetic lenses.

3. Results and discussion

3.1. Structural studies

The crystallinity of the ZnO films was studied by x-ray diffraction (XRD). XRD studies on the nanostructured films revealed that they crystallize as hexagonal ZnO. The films obtained are strongly c-axis oriented along the (002) direction, resulted from the closed-packed hexagonal crystal structure. The diffrogram also presented other peaks of low intensity towards the directions 100, 101, 102 and 103, indicating polycrystalline characteristics. The indexed XRD pattern of the nanostructured films is shown in Fig. 5

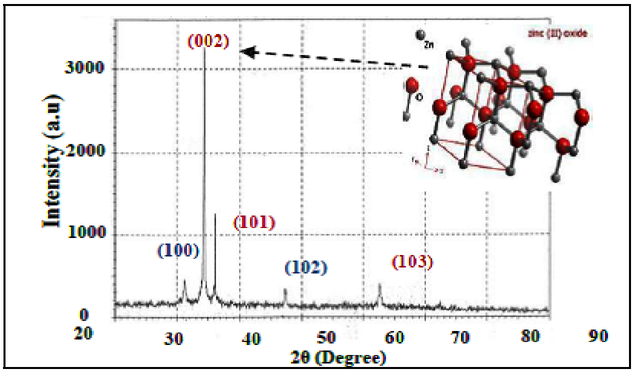


Fig.5. XRD patterns of prepared ZnO thin films.

3.2 Morphological studies

The morphology of the prepared ZnO thin film surface study has been carried out from scanning electron micrographs (SEM). Fig. 6 shows the SEM image of the ZnO film deposited on glass substrate, of high dense nanostructured distribution over the entire surface of the material. The appeared porosity of the ZnO thin film is an important parameter providing larger surface area regarding biomedical sensing point of view.

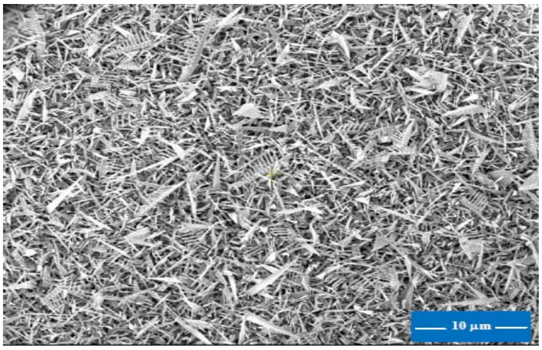
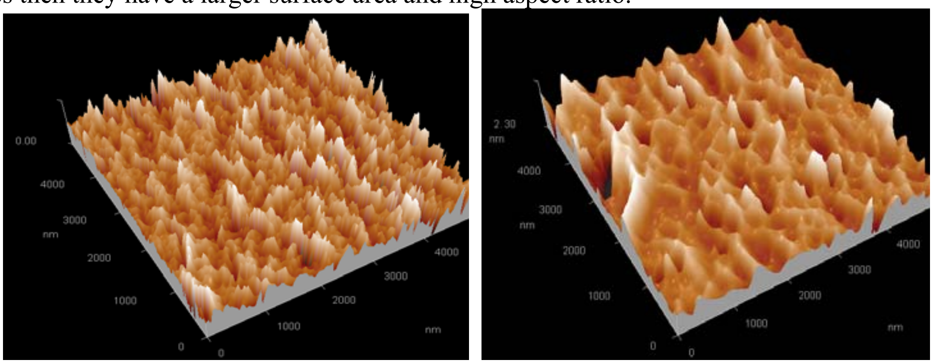


Fig.6. Scanning electron micrographs of ZnO thin film.

The film surface morphology and surface roughness were also studied using an AFM technique. Figure 7 displays AFM images in tapping mode of ZnO thin films deposited from CVD. It depicts that films deposited on the glass substrate are more pinholes and porosity than the films deposited on the silicon substrates then they have a larger surface area and high aspect ratio.



(a) (b)

Fig. 7 AFM images of the prepared ZnO thin film deposited on the a) glass substrate and b) silicon substrate.

3.3. Salt solution sensing performance of the sensor

3.3.1. Measurement of the sensing current

The prepared ZnO thin film was examined for sensing various concentration of the Na_2SO_4 aqueous solution, Fig. 8 shows the resulted sensing current at (100, 200, 300 and 400) mg/L sodium salt concentrations for each glass and silicon substrates, it is clearly observed that the sensing current increased with the salt concentration, it may be due to the high textured film structure and high surface to volume ratio especially for film deposited on the glass substrate, it will provide more probable interaction between the additive solution and the film surface.

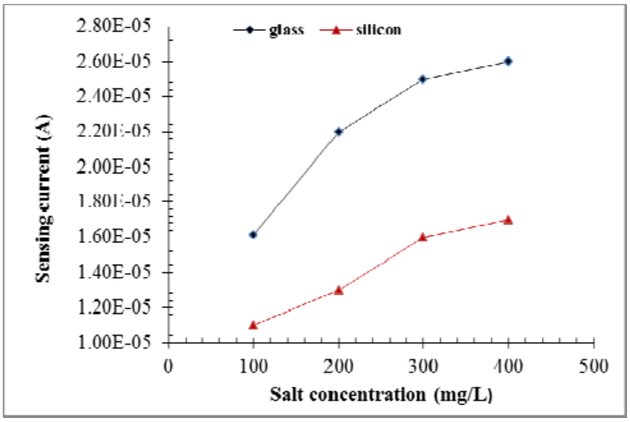
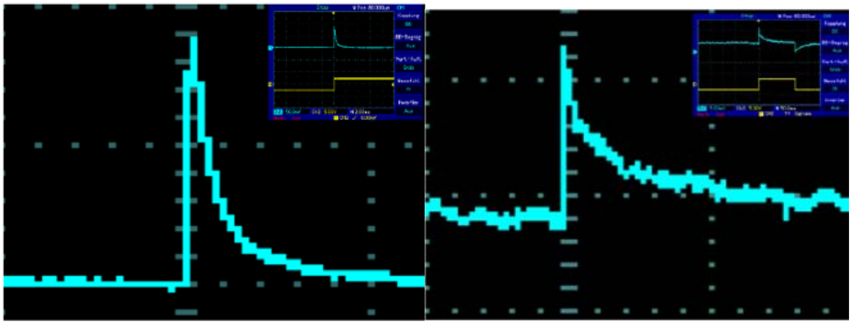


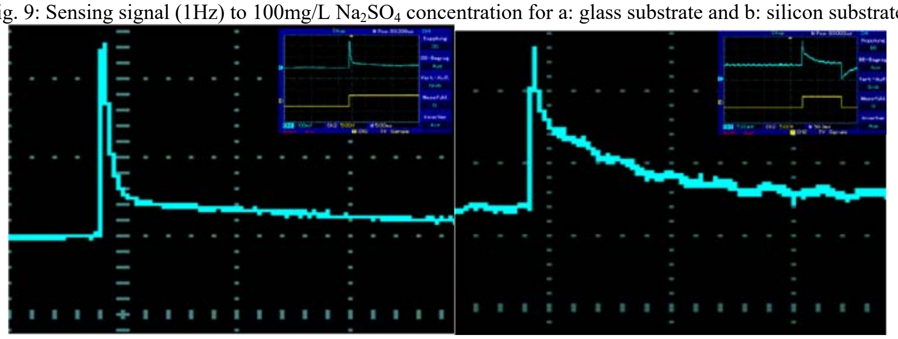
Fig. 8. Sensing current verses the salt concentration for each glass and silicon substrates.

Figures (9-12) show the images of signals for Na_2SO_4 salty solution of concentrations of (100, 200, 300 and 400)mg/L recorded by the oscilloscope. The main images illustrate the sensing signals and the inset of each image describes the incident (yellow) and sensing (blue) signals.



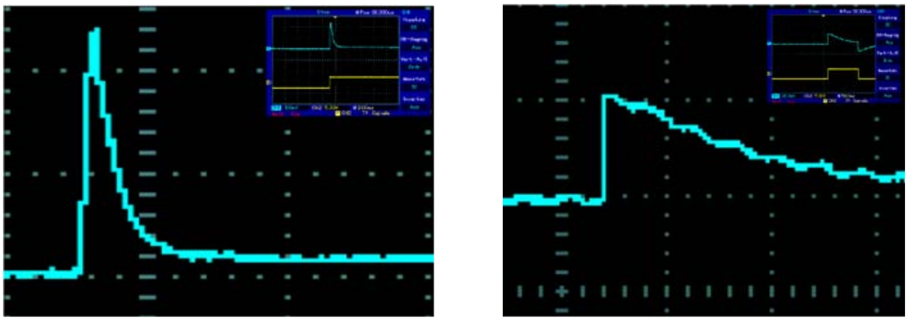
(a) (b)

Fig. 9: Sensing signal (1Hz) to 100mg/L Na_2SO_4 concentration for a: glass substrate and b: silicon substrate.



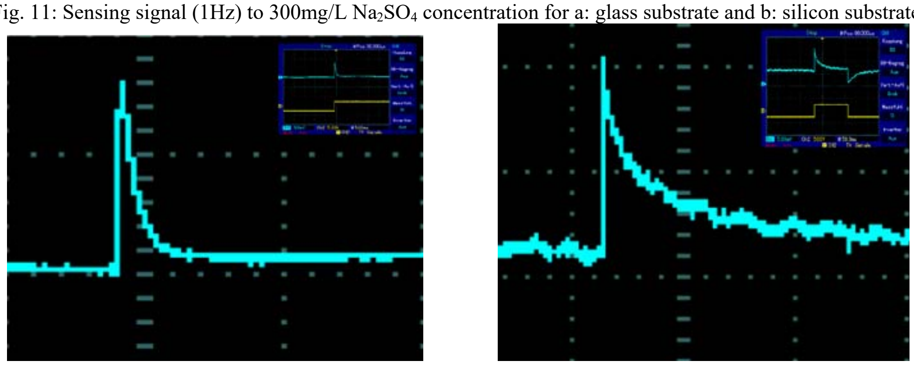
(a) (b)

Fig. 10: Sensing signal (1Hz) to 200mg/L Na_2SO_4 concentration for a: glass substrate and b: silicon substrate.



(a) (b)

Fig. 11: Sensing signal (1Hz) to 300mg/L Na_2SO_4 concentration for a: glass substrate and b: silicon substrate.



(a) (b)

Fig. 12: Sensing signal (1Hz) to 400mg/L Na_2SO_4 concentration for a: glass substrate and b: silicon substrate.

3.3.2. Sensitivity (S)

The sensitivity (S) of the nanostructured ZnO sensor for the sodium salt concentrations can be calculated depending on the measure of current under exposing to the salty solution (exposed current Is), and the aqueous ambient solution current Ia (at same operating conditions). The following relation has been used to calculate the sensitivity of the prepared ZnO film:

$$S = \frac{I_s}{I_a} \times 100\% \quad \text{……………………………..(1)}$$

Were, Is is the current under salt exposure and Ia is the current under ambient aqueous solution.

Fig. 13. shows the sensitivity of ZnO film, it was dramatically increased with the increase of salt concentration especially for concentration higher than 200mg/L. It was clearly appear that the film deposited on the glass substrate more efficient than the film deposited on the silicon substrate.

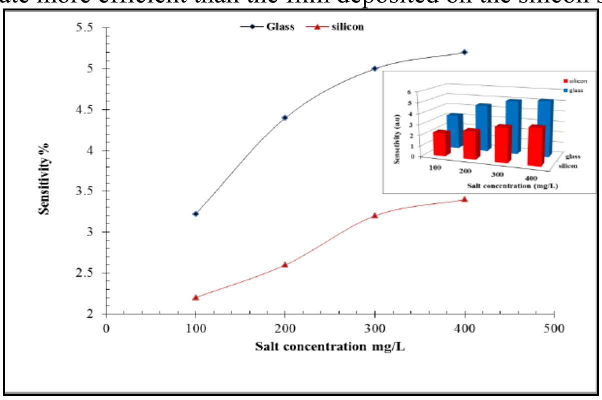


Fig. 13 The sensitivity of the sensor

3.3.3. Rise time and recovery time of the sensor

Rise time is defined as the time taken for the sensor to attain 90% of the maximum increase in sensing current on exposure and recovery time means the time taken for the sensor get back to 90% of the maximum decrease in sensing current for unexposed target. From the previous figures (9 -12), it is strongly observed that each sensor has a very fast response and the sensor has fast rise time which means that the oxygen ions at the film surface may immediate interact and bonded with the salt (Na_2SO_4) molecules, this fast interaction provides a large number of charge carriers which caused the sharp raise of the sensing current [51]. They defiantly have different values of recovery time, the sensor deposited on the silicon substrate slower than that deposited on the glass substrate. These results are shown in Fig. 14.

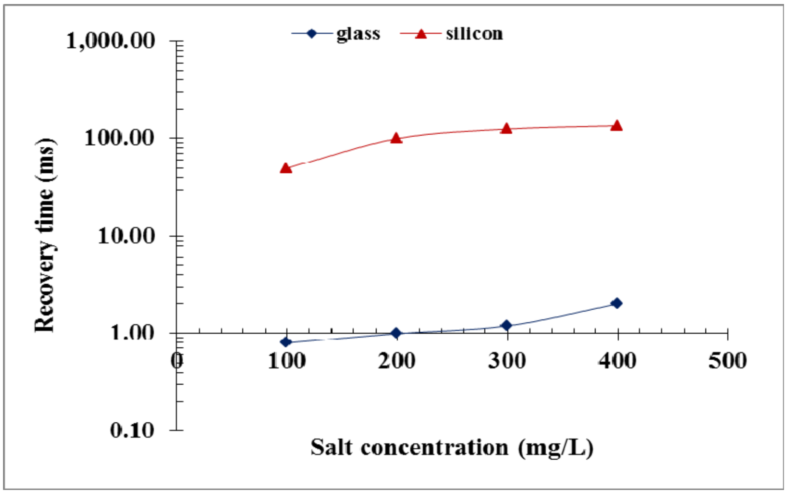


Fig. 14 Recovery time varies with the salt concentration for each glass and silicon substrates.

3.3.4. Relative response of the ZnO thin film

There are linear parts ranging between 100 - 400 mg/L (R^2: 0.8989) and (R^2: 0.9692) for each two kind of thin films. The graph for the calibration was given in Fig. 15. It was shown that the linearity of graph was highly satisfactory and it could be used for the qualitative determination of salty solution.

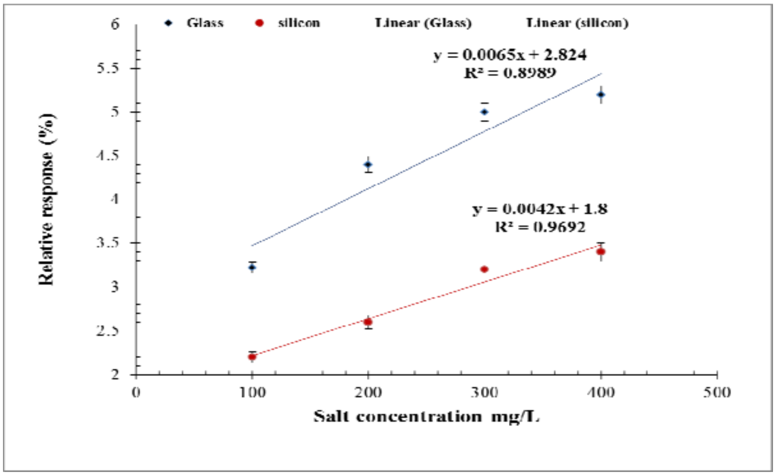


Fig. 15. Effect of Na_2SO_4 concentration on the relative response of ZnO sensor.

4. Conclusion

1- In this study, biomedical Na_2SO_4 salty sensor was made using ZnO comb-like nanostructures thin film. From the results given above, it was demonstrated that ZnO thin film could be provide favorable nanostructured biomedical sensor for salt detection, and we can conclude that: we have demonstrated that nanostructured ZnO thin film has been successfully synthesized by using the chemical vapor deposition (CVD) method. The XRD pattern confirmed the formation of hexagonal wurtzite structure ZnO polycrystalline nature.

2- New method for piezo-amperometric salt detection based on electrochemical interaction and piezoelectric properties employing finger electrodes. The fabricated sensor has very fast rise time for each film deposited on the glass and silicon substrates, and acceptable recovery time (53s). our results showed that the sensor prepared on the glass substrate more efficient and reliable than the sensor deposited on the silicon substrates. However, the high level noise appeared with the silicon substrate signal may be due to mismatch between the ZnO film and Si substrate. All sensing measurements had been made in-vetro, then the repetition is unconquerable. Good sensitivity and response in the range of salt concentration (100-400) mg/L have been obtained.

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