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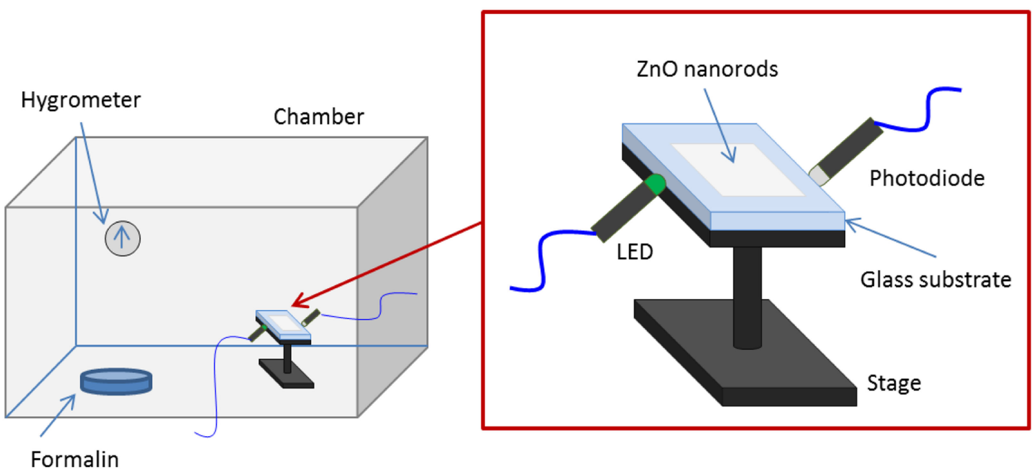

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Detection of Formaldehyde Vapor Using Glass Substrate Coated With Zinc Oxide Nanorods

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Abstract: A glass substrate coated with ZnO nanorods via a hydrothermal method for formaldehyde vapor sensing is reported. The proposed sensor device was characterized against formaldehyde vapor concentrations varying from 1% to 5% with a reference point as 0% (pure water) at room temperature. A significant sensing response was observed where the output voltage reduced by 0.0856 V via scattering effect by ZnO nanorods upon exposure to the maximum tested concentration (5%). The sensitivity and linearity of the sensing response were recorded to be approximate values of −0.0168 V/% and 98.06% correspondingly. The sensor device was found to have good measurement stability in measuring as low as 1% of concentration for a prolonged time of 600 s. This proposed sensor has potential applications in monitoring air pollution caused not only by formaldehyde vapor but also by other harmful and toxic vapors or gases.

Index Terms: Formaldehyde, formalin, vapor, zinc oxide, nanorods, glass substrate.

1. Introduction

Formaldehyde (HCHO) is a colorless and pungent-smelling gas which considered being a toxic air pollutant in both indoor and outdoor environment [1], [2]. It can be found in adhesive such as urea-formaldehyde (UF) and phenol-formaldehyde (PF) resins used for productions of furniture, building materials and interior decorations [1], [3], [4]. The release of formaldehyde in indoor environment also can be found in various daily products such as cleaning agents, disinfectants, textiles, preservatives, photo-processing chemicals and cosmetics [2]–[4]. At room temperature, formaldehyde is a gas and can be converted into other type of gaseous derivatives like trioxane. It can be dissolved easily in water (H_2O) which then converts into a diol ($CH_2(OH)_2$). An aqueous solution of formaldehyde is known as formalin. Pure formalin is a saturated solution of formaldehyde in water (approximately 37% by weight) [5].

Formaldehyde is one of volatile organic compounds (VOCs) which is hazardous and become threat to human when the concentrations exceed a specific level [6]. According to World Health Organization (WHO), the safety threshold limit of exposure should not exceed over 8 h for the concentration of 2 ppm while for indoor a maximum of 30 min on 0.08 ppm. [7]–[10]. It has been confirmed by WHO that formaldehyde is teratogenic and it has been listed as human carcinogen by the International Agency for Research on Cancer (IARC) [7], [11]–[13]. It causes the “sick building syndrome” which results in acute health issues [14]. Several temporary symptoms have been recorded such as dizziness, headache, fatigue and irritation of eyes, nose, throat and skin [3], [15]. The risk of asthma, nausea, allergy and edema become greater when it is used together with protein [16]. Prolonged exposure may cause damage to human body system such as respiratory system, central nervous system, blood and immune systems, pneumonia, bronchospasm and cancer [17], [18].

In previous studies, there are several detection methods of formaldehyde have been presented such as by cataluminescence [19], spectroscopy [20], chemiresistor [21], [22] and bio-sniffer [23]. However, these detection methods require huge equipment and need higher operating temperature which translated into higher operating cost [1]. Nanomaterial based sensor device has gained interest among researchers due to its unique optical, electrical, and mechanical properties which suitable for various applications such in gas sensors, superconductors, photocatalysis and optoelectronic devices [24], [25]. There are numerous nanomaterials from semiconducting metal oxides have been used for the fabrication of gas sensors such as zinc oxide (ZnO) [26], titanium dioxide (TiO_2) [27], tin oxide (SnO_2) [28], indium oxide (In_2O_3) [29] and nickel oxide (NiO) [30]. ZnO is one of the most widely used for gas sensing applications due to its good chemical stability, electrical compatibility and biocompatibility [31]. A simple, low cost and environmental friendly fabrication process of ZnO nanorods make it one of the popular choices among researchers [32]. ZnO has good optical transparency properties and has ability to operate within visible spectrum which make it suitable for optoelectronic applications [33], [34].

In this paper, the detection of formaldehyde vapor at room temperature by employing glass substrate coated with ZnO nanorods via hydrothermal synthesis method is reported. The sensing mechanism via light scattering effect by the ZnO nanorods upon exposure to the formaldehyde vapor was demonstrated which lead to a reduction of light intensity guided in the glass substrate thus reduced the output voltage. The performance of the sensor to detect as low as 1% of formaldehyde vapor concentration is analyzed.

2. Experimental

2.1 The Synthesis of ZnO Nanorods via Hydrothermal

In this fabrication process, the growth of ZnO nanorods on a glass substrate was done via hydrothermal method. All chemical used in this study are of analytical grade and used without any further purification. Firstly, a normal microscopic glass substrate with the dimension of 25 mm X 15 mm X 1 mm was cleaned correspondingly with a series of hydrochloric acid (HCl), sodium hydroxide (NaOH), soap water, acetone, ethanol and deionized (DI) water in ultrasonic bath and dried in atmospheric oven for 1 h. The glass substrate was then masked with polytetrafluoroethylene (PTFE) tape with a square area with the dimension of 10 mm X 10 mm on the surface of the substrate exposed for ZnO nanocrystallites deposition process.

The seeding process was carried out before the growth of ZnO nanorods via hydrothermal takes place [35]–[37]. The seeding solution was prepared by using zinc acetate ($Zn(CH_3COO)_2$) with ethanol for concentration of 1 mM as reported in previous study [38]. The masked sample was placed on a hot plate maintained at 70°C. An amount of 50 μl of zinc acetate solution was drop

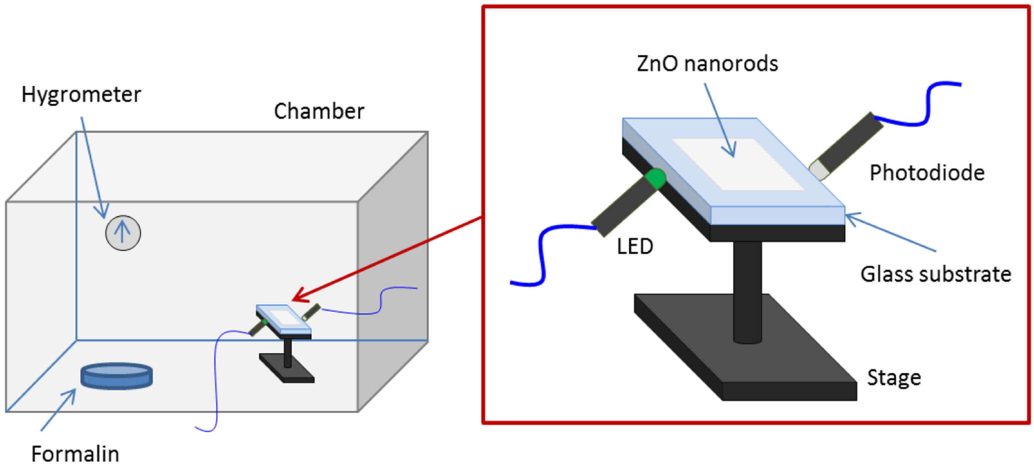

Fig. 1. Schematic representation of the experimental setup for formaldehyde vapor sensing.

casted on the exposed surface of the sample and dried. The dropping and drying process were repeated ten times followed by the annealing process in a furnace at 250°C for 5 h.

The ZnO nanorods were grown on the seeded sample by using the precursor solution containing 10 mM of zinc nitrate hexahydrate ($Zn(NO_3)_2{\cdot}6H_2O$) and hexamethylenetetramine of HMT ($(CH_2)_6N_4$). The seeded sample was dipped in the precursor solution by placing it downwards and kept inside an atmospheric oven for 15 h [39]. The solution was discarded and replenished with a new precursor solution every 5 h to maintain a constant rate of growth [40]. Upon completion of desired growth time, the sample was taken out from the solution and the mask was carefully removed. The sample coated with ZnO nanorods was then rinsed thoroughly with DI water. The sample was placed in the furnace at 350°C for 1 h to remove potential contaminations from the ZnO nanorods surfaces [41]. The ZnO microstructures coated on the glass substrate was characterized by scanning electron microscopy (SEM, Hitachi SU5000 FE-SEM) operating at 20 KV.

2.2 Preparation of Formaldehyde in Aqueous Solution (Formalin)

Formalin was prepared via dilution process of formaldehyde and DI water. The concentrations of the formalin were varied by using dilution formula expressed as

$$M_1 V_1 = M_2 V_2 \tag{1}$$

where M_1 is the concentration in molarity of the concentrated solution, V_1 is the volume of the concentrated solution, M_2 is the concentration in molarity of the dilute solution and V_2 is the volume of the dilute solution. In this experiment, the concentrations of formalin were varied from 1% to 5%. Every concentrations of formalin have similar volume to maintain the respective concentration during the measurement.

2.3 Formaldehyde Vapor Experiment

Fig. 1 shows the experimental setup for the detection of formaldehyde vapor concentrations. A sensing chamber (0.23 m x 0.18 m x 0.15 m) was used in the experiment. A normal green LED (wavelength 495–570 nm) was used as a light source. The green color of LED was chosen based on previous study by the group [42]. A photodiode was used to serve as a light detector which converts the light intensity into voltage. The glass substrate coated with ZnO nanorods was placed on the stage in between the LED and photodiode holder. The LED and photodiode were set as close as possible to both edges of the glass substrate in such a way that the light is guided in the glass from one end to the other. The LED was fixed at the angle of 50° with respective to the edge of glass substrate to achieve 60° of incident angle inside the glass for total internal reflection. In this configuration, the loss mechanism of the light intensity during the sensing experiment was mainly attributed to the scattering effect from the ZnO nanorods upon exposure to different concentrations of formaldehyde vapor. As a reference, a hygrometer was mounted on the chamber throughout the measurements. In this configuration, the detection scheme by the ZnO nanorods is based on two factors. First is the alteration of surrounding refractive index and the second is the change in electrical conductivity of the ZnO nanorods due to adsorption process upon exposure to the vapor concentrations [43], [44].

A desired amount of formalin was poured into a petri dish and placed inside the chamber. The experiment was conducted in room conditions (temperature of 25°C and relative humidity of 60%) to replicate the domestic user environment when handling the substance. Upon closure of the chamber lid, few moments were given to let the solution to naturally evaporate at room temperature through the volume of the chamber while the humidity level of the chamber was monitored. The humidity level of the chamber was increased due to the presence of water from the solution. The output voltage reading from photodiode was recorded at 80%, 85% and 90% of relative humidity (RH). The experiment was repeated for different concentrations of formalin varied from 1% to 5% and the results were analyzed with referenced to 0% (pure water).

The reduction of the output voltage upon the exposure to the formaldehyde vapor was due to the reduction of light intensity measured at the photodiode. In other word, it shows that higher leakage of light via scattering by ZnO nanorods was observed as the vapor concentration increases. The nanostructures of ZnO have high surface to volume ratio which capable to enhance the adsorption of vapor molecules on its surfaces [45]. Water is well known of having refractive index of 1.333 while formaldehyde is 1.3746. This clearly shows that the formaldehyde has higher refractive index and it modifies the refractive index of solution when added with water to form a formalin solution at different concentrations. During the vapor sensing experiment, formaldehyde vapor varies the refractive index of the nanorods surroundings. The adsorption of the vapor molecules by nanorods changed the electrical conductivity of the material [43], [44]. Therefore, the complex refractive index of the ZnO nanorods was altered due to the changing of electrical conductivity thus affects the optical scattering patterns on ZnO nanorods. As a result, changing in light intensity was observed when the sensor was measured against different concentrations of formaldehyde vapor. However, since there is small difference of relative index between water and formaldehyde, therefore small changes of intensity were observed as presented in the result.

3. Results and Discussion

3.1 Physical Characterization of ZnO Nanorods

The scanning electron micrograph (SEM) of the hydrothermally synthesized ZnO nanorods on glass substrate is shown in Fig. 2. Fig 2(a) shows the top view of the ZnO nanorods where the hexagonal wurtzite of nanorods were observed. The inset in Fig. 2(a) shows the layer of ZnO nanorods coating on the glass substrate. The average diameter of the nanorods was found to be about 77.2 nm and the average of the density or number of nanorods per unit area was observed approximately at 5.09×10^{13} nanorods/m^2. Fig. 2(b) shows the cross-sectional of the ZnO nanorods. The average length of the nanorods was found to be around 2.14 μm.

3.2 Formaldehyde Vapor Detection

The response of the fabricated sensor towards formaldehyde vapor concentrations by light scattering effect of ZnO nanorods is shown in Fig. 3. The graph shows the measurement at three different RH levels (80%, 85% and 90%) inside the chamber. It was found that at 80% of RH level the detection of formaldehyde vapor was not significant between the concentrations. The measurement at 85% of RH shows that the sensor starts to response towards the detection formaldehyde vapor concentrations. It can be seen that larger response on measurement at 85% of RH compared to 80%

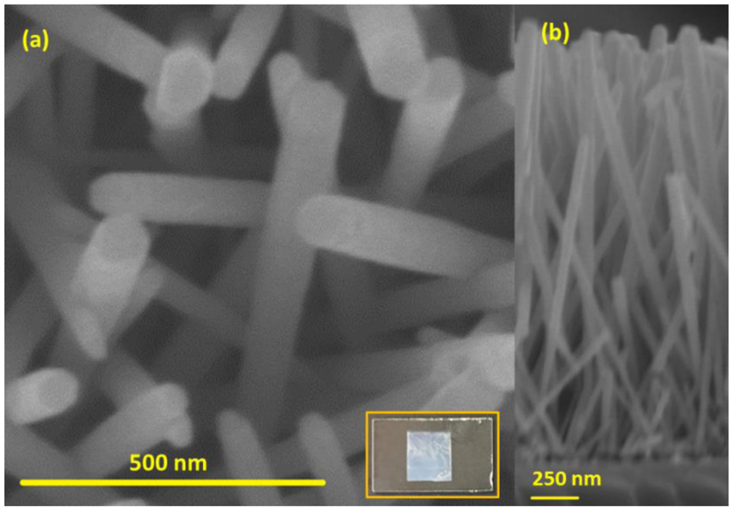

Fig. 2. (a) Top and (b) cross sectional SEM micrographs of ZnO nanorods grown on glass substrate. The inset in (a) show the ZnO layer fabricated on glass substrate.

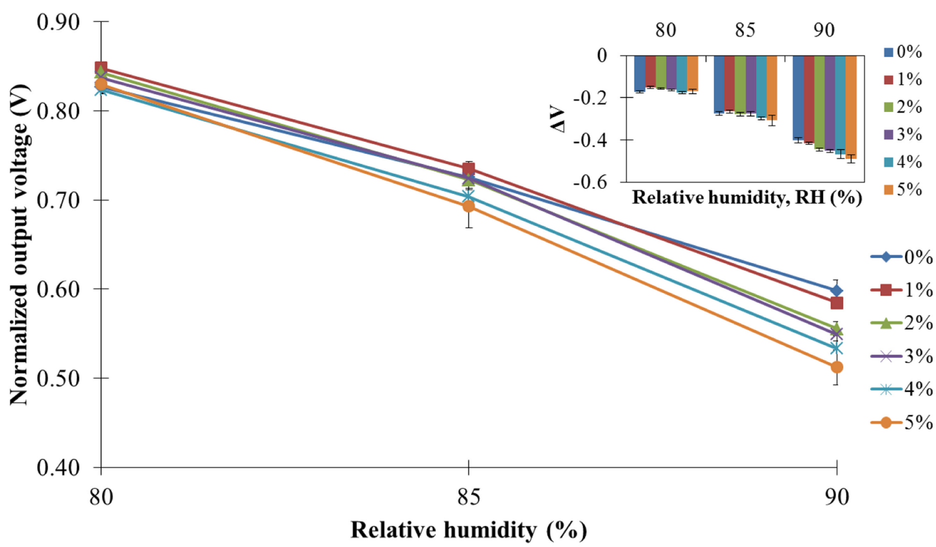

Fig. 3. Formaldehyde vapor sensing at different RH level. The inset shows the reduction of output voltage for the fabricated sensor in response to the vapor concentrations.

for the concentration from 0% to 5%. Although it has inconsistency response at lower concentrations of formaldehyde vapor (0% to 2%), the response eventually starts to have better sensing from the concentrations of 3% towards 5%. This trend shows the detection of the vapor concentrations improved from 80% to 85% of RH levels. At 90% of RH, the response of the fabricated sensor shows significant output voltage differences at every 1% incremental of formaldehyde concentrations. This can be shown by the bar chart as shown in the inset of Fig. 3. It is notable that the reduction of the output voltage (ΔV) at 90% of RH shows consistent incremental of output voltage reduction across the concentrations of formaldehyde vapor as compared to the 80% and 85%.

It was observed that the formaldehyde vapor was not fully evaporated at 80% and 85% of RH level and needs longer time to spread to all over the volume of the chamber for the sensor to response to the changes of low concentrations. This proved by the sensor stability performance as shown in Fig. 4. In this analysis, the measurement at 90% of RH level was prolonged for 600 s to investigate

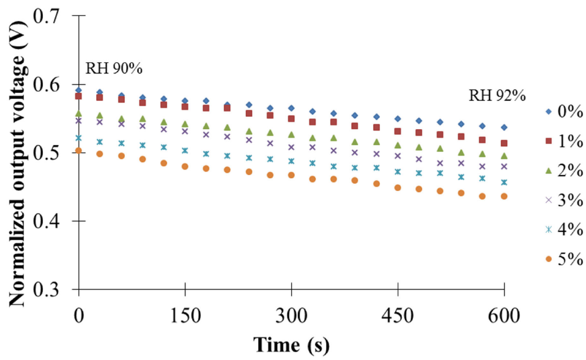

Fig. 4. The stability analysis of the fabricated sensor.

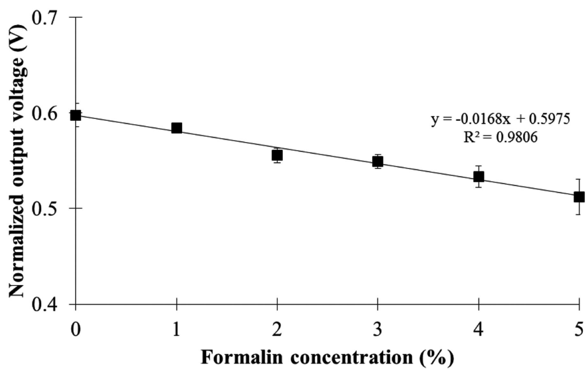

Fig. 5. Formaldehyde Vapor Sensing Performance

the stability of sensor response. The existence of water in formalin causes the humidity in the chamber to continue increased. However, the incremental of the RH level was getting slower as it almost reached the saturation level in the chamber. Therefore during the stability measurement, it was recorded that the RH level only increased from 90% to 92% within 600 s. From the graph, it shows that the measurements were relatively consistent between the concentrations throughout duration. Although there was slight incremental of humidity level, the sensor responded well to the changes and showed relative response between the concentrations of the vapor. This shows that relative humidity is not a critical factor in the measurement of formaldehyde vapor concentrations when the vapor was fully and uniformly spread throughout the volume of the chamber. The result in Fig. 4 also shows that the fabricated sensor has good stability in measuring as low as 1% of vapor concentrations.

Fig. 5 shows the performance of fabricated sensor in detecting formaldehyde vapor at 90% of RH level (considering the vapor was fully spread in the chamber). The sensing response shows that the output voltage reduced as the concentrations of vapor increases. It was found that the response towards concentrations to form a good linear trend line of 98.06%. The sensitivity of the sensor was observed to be approximate 0.0168 V/% which is good considering the capability of the fabricated sensor in detecting as low as 1% of the concentrations with the average standard deviation at 0.0099 V as presented in Table 1. Table 2 summarizes the total reduction of voltage at every tested vapor concentrations with respected to 0%. The data clearly shows that the fabricated sensor responded well to the changes of vapor concentrations. The accumulated reduction of output voltage was consistently increased as the concentration increases. It was found that the total reduction due to the exposure to 5% of vapor concentrations with respected to 0% was 0.0856 V.

The reduction of the output voltage upon the exposure to the formaldehyde vapor was due to the reduction of light intensity measured at the photodiode. In other word, it shows that higher leakage

TABLE 1

Performance of the Fabricated Sensor Device

Parameter	Performance
Linearity (%)	98.06
Sensitivity (V/%)	-0.0168
Average standard deviation (V)	0.0099
Resolution (%)	0.5893

TABLE 2

The Reduction of Output Voltage Upon Exposure to Formaldehyde Vapor

Formalin concentration (%)	ΔV
1	-0.0134
2	-0.0423
3	-0.0487
4	-0.0646
5	-0.0856

of light via scattering by ZnO nanorods was observed as the vapor concentration increases. The nanostructures of ZnO have high surface to volume ratio which capable to enhance the adsorption of vapor molecules on its surfaces [45]. Water is well known of having refractive index of 1.333 while formaldehyde is 1.3746. This clearly shows that the formaldehyde has higher refractive index and it modifies the refractive index of solution when added with water to form a formalin solution at different concentrations. During the vapor sensing experiment, formaldehyde vapor varies the refractive index of the nanorods surroundings. The adsorption of the vapor molecules by nanorods changed the electrical conductivity of the material [43], [44]. Therefore, the complex refractive index of the ZnO nanorods was altered due to the changing of electrical conductivity thus affects the optical scattering patterns on ZnO nanorods. As a result, changing in light intensity was observed when the sensor was measured against different concentrations of formaldehyde vapor. However, since there is small difference of relative index between water and formaldehyde, therefore small changes of intensity were observed as presented in the result.

4. Conclusion

We have demonstrated a simple and low cost sensor device fabricated with ZnO nanorods via hydrothermal method towards formaldehyde vapor sensing application. The fabricated sensor device was observed to have a good response to the tested concentrations as low as 1% up to 5%. The reduction of output voltage was found to be at 0.0856 V for the measurement at 5% of concentrations with referenced to 0% which is pure water. The response of the device throughout the concentrations of vapor showed a good linearity of 98.06%. The sensitivity of the sensor device was found to be at -0.0168 V/%. Finally, the analysis of the sensor throughout the prolonged measurement showed that it has good stability of the measurement at small change of concentration.

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