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Gas Sensitivity Variations of Composites of ZnO/Banana Peel Derived Graphitic Carbon as Measured in Methanol Vapor

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Abstract- ***Gas sensing studies of biomass derived graphitic carbon is hardly found. Films of composites of ZnO and carbon particles derived from dried banana peels were fabricated by doctor blade method. The mass ratio between ZnO and carbon was varied to optimize the gas sensitivity of films measured in 200 ppm of methanol vapor. According to XRD measurements, graphitic carbon has been crystallized. Furthermore, the crystallite size, strain and dislocation density alter with the addition of ZnO. According to XRFS spectrums, metals such as Fe, Cr, Co and Ni were identified in carbon powder. The interactions of these metals with carbon powder at higher activation temperatures are attributed to the formation of graphitic carbon. According to UV-visible spectrums, the optical band gap gradually decreased with the addition of ZnO to carbon particles. The gas sensitivity first increased with the addition of ZnO up to 3% of ZnO, thereafter the gas sensitivity gradually decreased with ZnO concentration. The highest gas sensitivity was 17% at 3% of ZnO. In addition, the response time varies with the addition of ZnO to carbon.***

Index Terms- ***ZnO, Graphitic Carbon, Gas Sensitivity, Methanol***

I. INTRODUCTION

The detection of volatile organic compounds (VOCs) such as methanol has become an important fact for industrial safety, environmental monitoring, and healthcare diagnostics. Methanol is a highly flammable, toxic solvent, yet its colorless and odorless nature makes the leak detection of methanol difficult without high-precision sensors. Although, different sensing technologies exist, the development of composites of carbon and metal oxides for gases and vapors detection remains a significant research focus due to its sustainability.

Metal oxides (MO) have attracted much attention in the field of gas sensing under atmospheric conditions due to their low cost, flexibility in production, simplicity of their use and large number of detectable gases. According to the previous research, the reversible interaction of the gas with the surface of the material is a characteristic of metal oxide gas sensors. This reaction can be influenced by many factors, including internal and external causes, such as natural properties of base materials, surface areas and microstructure of sensing layers, surface additives, temperature and humidity [1]. Metal Oxides can be divided into two groups according to their electronic structure as transition and non-transition metal oxides. Only transition-metal oxides with d^0 and d^{10} electronic configurations find their real gas sensor application. Here, ZnO is used as the metal oxide which has a d^{10} electronic configuration.

Activated carbon derived from biomass enhances the gas sensitivity by increasing the surface area, improving electrical conductivity, and facilitating charge transfer at the interface. The high porosity shown by AC derived from some biomass, which is beneficial for absorption increase the surface area. Also, the higher free electron density led to the remarkable conductivity. Modification of activated carbons' (ACs') surface chemistry with the introduction of basic MO is among the most efficient ways for the promotion of the basic properties of AC and the facilitation of interaction with VOCs. The

bio-mass derived carbon and metal oxides nanocomposites from Persian ironwood by H_3PO_4 activation have shown superior CO_2 capture performance [2]. Composites synthesized from Aloe-Vera-, spent coffee- and corncob-derived activated carbon and ZnO exhibit superior H_2S sorption capacity up to 106 mg_{H2S} / $g_{ads.}$, 66 mg_{H2S} / $g_{ads.}$, and 47 mg_{H2S} / $g_{ads.}$, respectively [3]. The nano particles deposition provides up to 10-fold increase of acetaldehyde amount adsorbed as compared to non-modified activated carbon fibers (ACF). The adsorption capacity increased with the basicity of metal oxides attaining 20 wt.% on La_2O_3/ACF [4]. A review on advances in Metal-Oxide modified activated carbon for H_2S and CO_2 removal underscore the potential of these new composite materials to enhance biogas upgrading technologies, fostering the broader use of biomethane as a sustainable energy source [5].

In this manuscript, we present the gas sensitivity of a composite of carbon derived from banana peels and ZnO at 200 ppm of methanol at room temperature. The gas sensitivity of the composite was optimized by varying the weight ratio of carbon to ZnO. The carbon/ZnO films fabricated by the doctor blade method were characterized using XRD and UV-visible spectra.

II. EXPERIMENTAL

2.1. Preparation of films

First, the carbon powder was prepared from banana peels according to the method described in Weerasooriya et al. [6]. Then, 0.08 g of Polyethylene Glycol (PEG) powder was dissolved in 5 ml of distilled water and stirred at 300 rpm for 15 minutes at a temperature of 45 °C. Next, the mixture of derived carbon powder and ZnO was added to the prepared PEG solution and stirred at 300 rpm for 2 hours at 60 °C. The weight of the carbon content of a sample was 0.42 g and five samples were prepared by changing the weight of ZnO from 0, 2, 3, 4, 6% as the carbon weight. After that, the sample was spread on conductive and non-conductive glass substrates, and the doctor blade method was used to fabricate the samples. The coated samples were annealed at 75°C for 30 minutes. The area of each sample was kept at 1.5 × 1.5 cm^2.

2.2. Characterization of the sample

The optical properties of the samples coated on the nonconductive amorphous glass plates were analyzed using a Shimadzu 1800 UV visible spectroscopy at the wavelength range of 190 nm - 1100 nm. The optical band gap of the samples with 0, 2 and 6% ZnO concentrations were determined. A Rigaku Ultima IV X-ray diffractometer was used to analyze the structural properties of the samples. The samples were coated on the nonconductive glass substrates. For XRD analysis, Cu-Kα radiation (λ = 1.5406 Å) was utilized and scanned over a diffraction angle range from 0° to 100°. X-ray fluorescence spectrums (XRFS) was measured by a Fischeascope X ray XAN XRFS analyzer.

2.3. Gas sensitivity measurement

For measuring gas sensitivity, the samples were coated on the conductive glass plates and placed inside a sealed glass chamber with a volume of 78.25 cm^3. Two gold coated copper wires were used as electrodes to connect the gas sensing setup to a 5V DC power supply. The circuit was given an hour for stabilizing the resistance of the sample (R_o) and it was noted down. The sample, a standard resistance that was nearly equal to the resistance of the sample, and the dc power supply was connected in series. Before introducing the gas, the circuit was stabilized for 1 and half hours. Afterwards, 200 ppm of methanol was applied to the chamber and the voltage between the standard resistor was measured using a Fluke 289 multimeter over the time period of 2 hours. The above procedure was repeated for all other samples and the gas sensitivity of each was calculated based on the readings taken.

III. RESULTS AND DISCUSSION

3.1 XRD analysis

Figure 1 illustrates the X-ray diffraction (XRD) patterns of the films for pure carbon (black line), carbon with 2% (red line) and 6% (blue line) ZnO. According to XRD patterns, the phase of graphitic carbon was found in all the samples [7-9]. The addition of ZnO has contributed few new peaks to XRD patterns. The new peak found at 32.4 degrees

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for 6% of ZnO was identified as a peak of ZnO. A slight shit of peaks was observed with the addition of ZnO. XRD patterns indicate layered hexagonal graphitic structure. Stronger peak at 26.7 degrees shows (002) peak with interlayer spacing 0.342 nm.

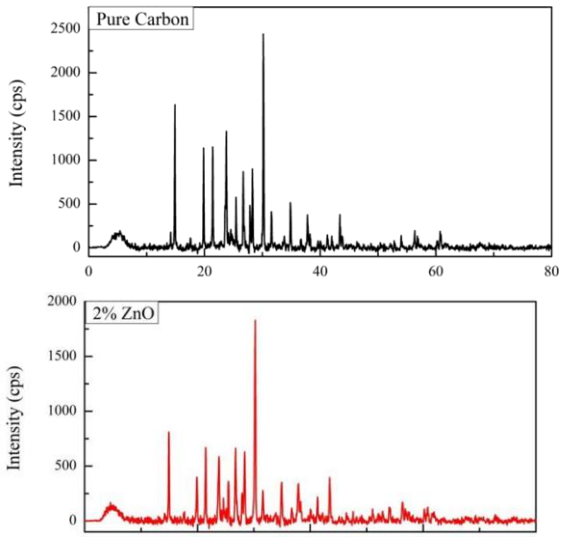
The crystallite size (D), the strain (ε) and dislocation density (δ) are given by [10, 11],

$$D = \frac{0.91\lambda}{\beta \cos(\theta)} \quad (01)$$

$$\varepsilon = \frac{\beta \cos(\theta)}{4} \quad (02)$$

$$\delta = \frac{1}{D^2} \quad (03)$$

Here, λ represents the wavelength of Cu-Kα radiation (λ=1.5406A˚), and β denotes the full width at half maximum (FWHM) of the XRD peak at angle θ. Table 1 represents the estimated values of these parameters for the major peaks of XRD patterns. The crystallite size first decreases and then increases with ZnO concentration. However, the dislocation density and the strain behave in the opposite way.



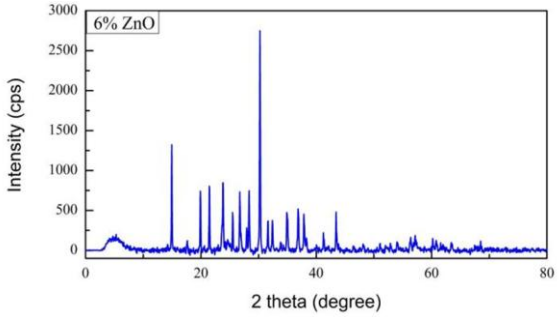


Figure 1: XRD patterns of carbon fabricated from banana peels and carbon/ZnO composites

Table 1: Crystallite size, strain and dislocation density of samples.

ZnO Concentration	angle 2ɵ (deg)	FWHM (deg)	crystallite size (10^{-7}m)	Dislocation density (10^{14} lines/m^2)	Strain
Pure	30.16	0.18	0.461995985	4.685151048	0.000758635
2%	30.25	0.2	0.415884598	5.781683659	0.000842749
6%	30.25	0.14	0.594120855	2.833024993	0.000589925

The graphitization degree (g) can be estimated using Mering-Maire equation as follows.

$$g = \frac{0.3440 - d_{002}}{0.3440 - 0.3354} \quad (04)$$

Where 0.3440 is d_{002} for completely non-graphitized carbon, and 0.3354 is d_{002} for ideal graphite. Calculated d_{002} for XRD of our pure carbon sample is 0.3420 nm. The estimated value of graphitization degree of our pure carbon sample is approximately 0.23.

3.2. XRFS analysis

Figure 2 represents the XRFS spectrum of pure carbon powder prepared from banana peels. Some Fe, Cr, Co and Ni were found in the sample in addition to carbon. The XRFS peaks of carbon are not shown in spectrum. The carbon was heated to 700 ^{0}C in the activation process. Combination of higher

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temperature and availability of metal particles such as Fe, Cr, Co and Ni creates an environment to crystallize graphitic carbon structure. In the preparation of carbon, small sp2 clusters merge into larger graphene sheets. Carbon dissolves into metal at higher temperatures, and carbon precipitates as graphitic layers during cooling. The graphitic formation of biomass carbon is limited.

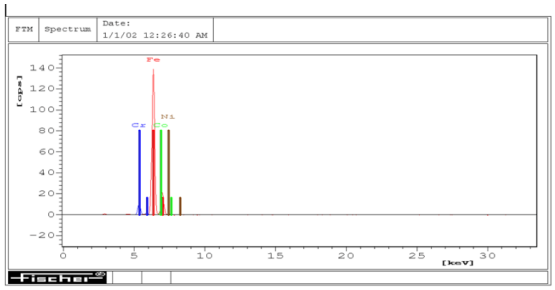

Figure 2: XRFS spectrum of pure carbon sample.

3.3 UV-Visible analysis

Figure 2 illustrates the UV-visible spectra of the films for pure carbon (black line), carbon with 2% (red line) and 6% (blue line) ZnO. The UV visible spectrum indicates information about optical properties and electronic transition in the materials within the ultraviolet and visible regions. All samples show strong absorbance in the UV region due to $\pi \rightarrow \pi^*$ electronic transitions in carbonaceous structures. The ZnO–carbon composite samples exhibit higher absorbance compared to the pure carbon thin film due to charge-transfer transitions involving $O^{2-} \rightarrow Zn^{2+}$ electronic excitation, and the absorption features may shift when ZnO interacts with carbonaceous components. Addition of extra materials introduces new energy levels to the band gap. As a result, the effective band gas decreases [12]. The band gap of activated carbon is generally higher than that of metal oxides. Therefore, the addition of a metal oxide to activated carbon leads to reduce the band gap.

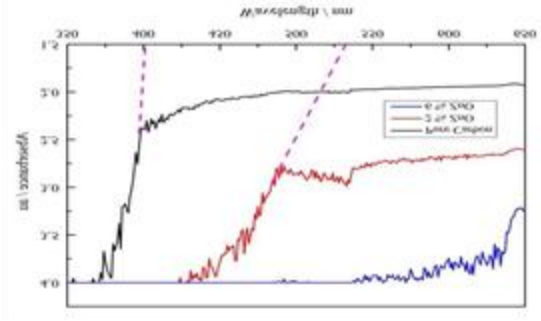

Figure 3: UV-visible spectra of pure carbon and carbon-ZnO composite samples.

Table 2: Optical band gaps of different composite samples

Sample	Wavelength (nm)	Bandgap (eV)
Pure Carbon	400.48	3.10
2% ZnO	531.50	2.33
6% ZnO	679.98	1.82

According to Table 2, the pure carbon sample shows the highest band gap of 3.10 eV. In contrast, the sample with 2% ZnO exhibits a band gap of 2.33 eV, while the 6% ZnO sample shows 1.82 eV. This confirms that increasing ZnO concentration significantly decreases the band gap of the ZnO-carbon composite samples.

3.4 Gas sensitivity analysis

Gas sensitivity is defined as the percentage change in resistance of thin film in the presence and absence of gas. The gas sensitivity of the sample can be calculated using equation (05).

$$Gas\ sensitivity = \frac{|R_{gas} - R_0|}{R_0} \times 100\% \quad (05)$$

Where, R_{gas} is the saturated resistance of the thin film after exposure to 200 ppm methanol vapor and R_o is the resistance of the sample in air before exposure to the gas. In the experiment, the voltage across the standard resistor (V) was noted down with time for 2 hours. The resistance of the thin film (R) and the current through the sample (I) were calculated using the equations below. These equations are derived by applying Ohm's law to the circuit.

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$$R = \left(\frac{5}{v} - 1\right) S \quad (06)$$

$$v = IS \quad (07)$$

Where S is the value of the standard resistor used for each case.

These measurements enabled a detailed evaluation of the gas-sensing performance of the films, highlighting their sensitivity to vapor molecules and the associated interaction mechanisms. The voltage variation was recorded over several cycles to confirm the repeatability of the measurements.

Table 3: Gas Sensitivity and response time of different samples.

Sample	Sensitivity	Response time /min
Pure Carbon	8%	28
2% ZnO	8%	35
3% ZnO	17%	21
4% ZnO	9%	22
6% ZnO	3%	34

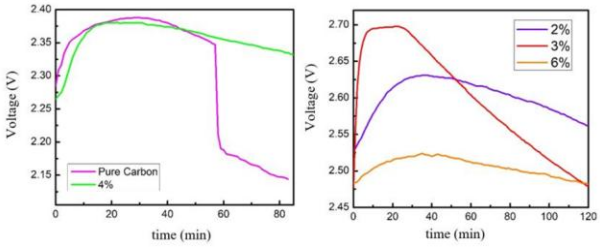

Figure 4: Graph of the Voltage (across standard resistor) vs time for the ZnO and activated carbon composite films measured in 200 ppm of methanol

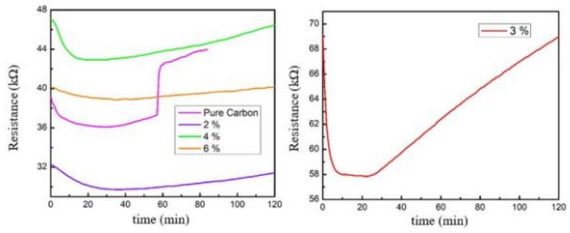

Figure 5: Graph of the Resistance vs time for the ZnO and activated carbon composite films measured in 200 ppm of methanol

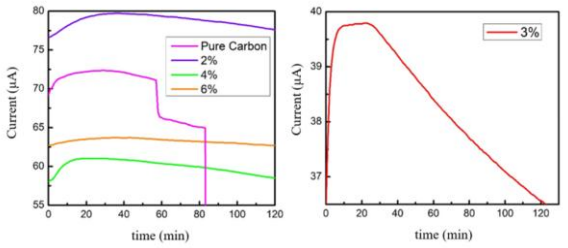

Figure 6: Graph of the Current vs time for the ZnO and activated carbon composite films measured in 200 ppm of methanol

According to figure 4 the voltage between the standard resistor initially increased for each sample after exposure to methanol vapor. Since the film was connected in series with the standard resistor, this indicates a decrease in thin film's resistance and an increase in current. This is due to a change in electrical properties of the sensing layer caused by the adsorption of methanol molecules on the film surface. In pure carbon films, methanol molecules interact with surface defects and oxygen-containing functional groups, resulting in moderate charge transfer that alters the electrical properties of the sensing layer. ZnO–carbon composite films show a greater decrease in resistance compared with the pure carbon film, while the sample with 6% ZnO concentration shows the least decrease. Oxygen molecules adsorbed on the ZnO surface capture electrons from the conduction band, forming ionized species such as O^- and O_2^-. When methanol gas is introduced, it reacts with these adsorbed oxygen ions, releasing the trapped electrons back into the conduction band and increasing the charge carrier concentration. The increased electron concentration lowers the resistance of the sensing layer and results in a stronger change in the measured voltage across the series resistor [13].

The adsorbed methanol reacts with ionized oxygen species and release electron as follows.

$CH_3OH + O^-$ (ads) → $CO_2 + H_2O + e^-$

These released electrons enhance the electrical conductivity of the sample after adsorbing methanol vapor. As a result, after adsorbing methanol vapor, the electric current through the sample increases and the resistance decreases.

The table 3 shows the gas sensitivity and response time of all samples. The sample with 3% ZnO exhibits the highest gas sensitivity of 17% with 21 min response time. These results show that a small amount of ZnO significantly improves the sensing performance of thin films. However, further increase in ZnO concentration leads to a decrease in sensitivity due to particle aggregation and disruption of the conductive carbon network. Alouani et al. (2023) examined the gas sensing performance of G80/20 and G95/5 nanomaterials, with a weight ratio of 80:20 and 95:5, which were synthesized by the insertion of zinc oxide nanoparticles in the pristine graphene nanoplatelets, with bare graphene and pure ZnO as reference samples. Those samples have been tested for NO_2 gas in the range of 50 to 500 ppb at room temperature. Both G80/20 and bare graphene samples did not show a significant response. However, the G95/5 sample showed a relatively high performance of 2.6% ppm^{-1} in this concentration range, demonstrating that a lower amount of ZnO improves sensing, while excessive ZnO reduces performance [14].

Formation of percolation network in composites of metal oxide and carbon is attributed to the variation of gas sensitivity. Carbon materials facilitate a percolation network that supports electron transport. Although carbon has a higher conductivity, metal oxide is a semiconductor. At lower carbon concentrations, there isn't enough carbon to create a conductive pathway. At higher carbon concentrations, ZnO is insufficiently distributed, and the advantages of the composite synergy are minimized. With an optimal ZnO concentration of 3%, carbon particles support an interconnected network, while ZnO continues to provide positive contributions. Similar behavior was observed for composites of carbon and some other metal oxides. The highest gas sensitivity of CuO/carbon composites with carbon particles synthesized from acacia auriculiformis branches was obtained for 90% of CuO as measured in methanol vapor [15]. The highest gas sensitivity of composites of carbon derived from rubber tree and SnO_2 in ethanol vapor was observed at 92% SnO_2 [16]. The optimum ratio of carbon and metal oxide depends on many factors such as particle size and porosity.

IV. CONCLUSION

Graphitic carbon was successfully employed to detect 200 ppm methanol vapor. Higher surface area, defects and edges in graphene layers, □-electron system of graphitic carbon, residual oxygen functional groups, and porous structure of graphitic carbon are responsible for the gas sensing mechanism. Methanol behaves as a reducing gas, when it reacts with adsorbed oxygen species in graphitic carbon. Addition of ZnO created a percolation network in the composite. As a result, the gas sensitivity was optimized at 3% concentration of ZnO. Because the optical band gap gradually decreases with ZnO concentration, the optical band gap is not responsible for the optimum gas sensitivity at 3% concentration of ZnO. However, the crystallize size is minimum at 3% ZnO according to XRD analysis. Therefore, the minimum crystallite size can be attributed to highest gas sensitivity of 3% concentration of ZnO. Although addition of 2% of ZnO to carbon did not make any significant impact on gas sensitivity, the addition of 3% ZnO doubled the gas sensitivity. The gas sensitivity gradually decreased toward the higher ZnO concentrations.

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