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Proceeding Paper

Synthesis, Characterization, and Hydrogen Gas Sensing of ZnO/g-C_3N_4 Nanocomposite [†]

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Abstract: In this paper, the preparation of the ZnO/g-C_3N_4 nanocomposite is discussed. The synthesis of nanocomposite is performed by the direct pyrolysis of the precursor (zinc acetate hexahydrate). The material synthesis is validated by different characterization tools, such as X-ray Diffraction (XRD), Scanning electron microscopy (SEM), Transmission electron microscopy (TEM). The SEM and TEM analysis revealed the formation of nanorods on g-C_3N_4 support. The gas sensing property of the ZnO/g-C_3N_4 was studied for various concentrations of hydrogen gas. Response and recovery times were recorded by the sensor.

Keywords: hydrogen gas sensor; graphitic carbon nitride; ZnO/g-C_3N_4

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1. Introduction:

Hydrogen is increasingly important as a clean energy source due to its relatively easy availability and eco-friendliness. Moreover, hydrogen is efficient and renewable, and its common by-product is water [1]. For these features, hydrogen is the most promising green energy source for automotive and other industries and technologies, such as fuel cells, defence, petroleum, etc. Hydrogen gas is very explosive and fatal above its lower explosion limit (1–4%). The g-C_3N_4 is a 2D conjugated polymer semiconductor [2]. It is an n-type semiconductor, which has nitrogen in abundance. It is a thermally stable, non-poisonous, metal-free, and low-cost material [3]. The g-C_3N_4 has suitable band structures that are highly thermal; have chemical stabilities; have excellent electronic properties, and are abundant in nature [2,3]. The g-C_3N_4 has been applied in photosynthesis, energy conversion and storage, carbon dioxide storage and reduction, solar cells, sensing, and imaging [4]. The g-C_3N_4 comprises excellent properties as a gas sensing material. It possesses an indirect bandgap of 2.7–2.8 eV, consisting of carbon (C) and nitrogen (N) atoms that are organized in the graphite-like layered structure; each layer has tri-s-triazine, which is connected to the amino group [5].

2. Preparation of ZnO/g-C_3N_4nanorods

To synthesize the ZnO/g-C_3N_4nanocomposite melamine was taken as a precursor material for g-C_3N_4 and zinc acetate dehydrates for ZnO. All the materials taken for the synthesis were used as-received (i.e., without any further purification). For the synthesis, a very simple and cost-effective pyrolysis process was adopted. The ZnO and g-C_3N_4 precursor materials were transferred to the alumina crucible and closely packed using aluminum foil. Thereafter, obtained powder was heated up at 550 °C for 5–6 h at a 3

°C/min ramp rate. As a result of the calculations, brown to dark brown powder was obtained. The ZnO nanorods on g-C_3N_4 sheets were formed after the completion of the process, which was established by various morphological and microstructural characterizations.

3. Material Characterizations

3.1. X-ray Diffraction

The XRD spectrum for ZnO/g-C_3N_4 is shown in Figure 1. g-C_3N_4 gives two peaks at 12.9 and 27.5, which corresponds to 100 and 002 lattices [6,7]. The peak at 27.5 corresponds to interplanar stacking peaks of aromatic rings, and that at 12.9 corresponds to interlayer structure [6,7]. The interplanar spacing of the two peaks was obtained as 0.685 nm and 0.323 nm, respectively. The ZnO peaks are recorded at 31.8, 34.5, 36.3, 47.6, 56.7, 62.9, and 69.2 which correspond to 100,002,101,102,110,103,112 lattices respectively (JCPDS 36-1451). No other peaks were recorded, which confirmed the very high purity of the material [119].

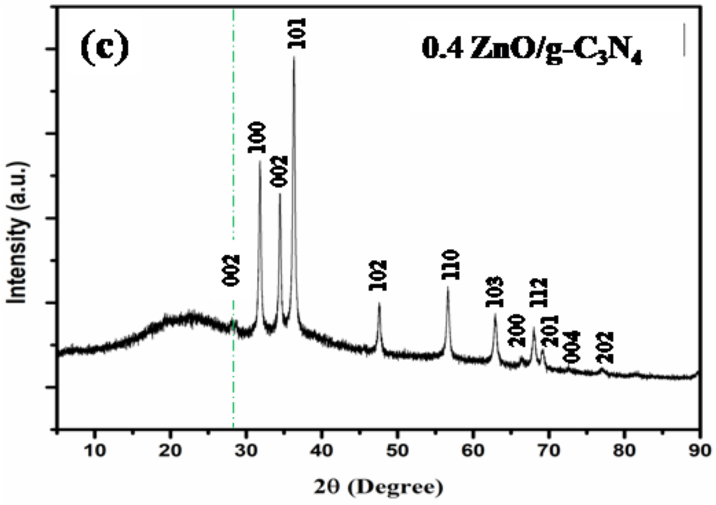

Figure 1. XRD spectra of (**a**) 0.1 ZnO/g-C_3N_4 (**b**) 0.2 ZnO/g-C_3N_4 (**c**) 0.3 ZnO/g-C_3N_4.

3.2. FESEM and TEM

SEM and TEM analyses were performed to investigate the surface morphology and microstructure of the prepared sample. SEM image reveals the irregular sheets of g-C_3N_4 with variation in size, as shown in Figure 2a. The ZnO/ g-C_3N_4 show the excellent formation of ZnO nanorods on the g-C_3N_4 sheets. The size of the rods is about 400–500 nm. TEM images of the GCN and ZnO/ g-C_3N_4 are shown in Figure 2b. The g-C_3N_4 has 2D sheets with irregularity in shape and size, as also depicted in SEM. The ZnO/g-C_3N_4TEM analysis shows the formation of nanorods. ZnO nanorods are anchored to the g-C_3N_4 sheet. This confirms the presence of ZnO in the composites.

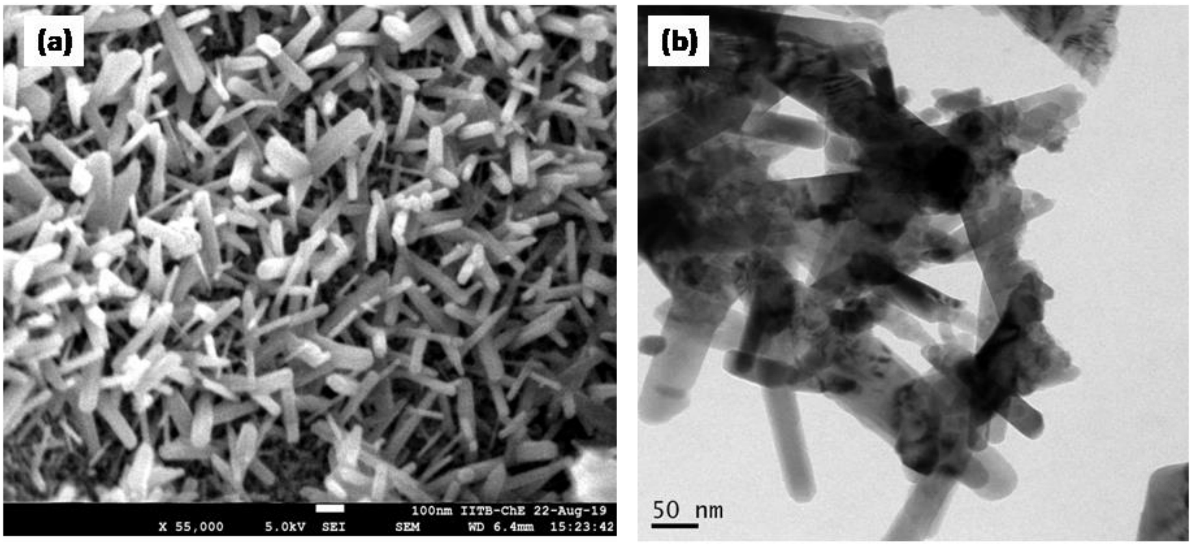

Figure 2. (**a**) FESEM of ZnO/g-C_3N_4 (**b**) TEM image of ZnO/ g-C_3N_4.

3.3. Energy Dispersive X-ray (EDX) Spectroscopy

EDX analysis for the synthesized ZnO/g-C_3N_4 composite was done to confirm the presence of elements. As shown in Figure 3, EDXwas performed in the selected area of the ZnO/g-C_3N_4 composite. The peaks for C, N, O, and Zn can easily be seen in the EDX spectrum. Apart from these elements, some other element peaks were also observed (which are attributed thin coating required for improving the conductivity and imaging).The elemental area mapping for the selected areaand the presence of all the expected elements iscleary evident. Therefore, it is confirmed that the ZnO/g-C_3N_4 nanocomposite has all the expected elements present. As there are no other peaks (except for those corresponding to coating), the synthesized ZnO/g-C_3N_4composite was concluded to be of high purity.

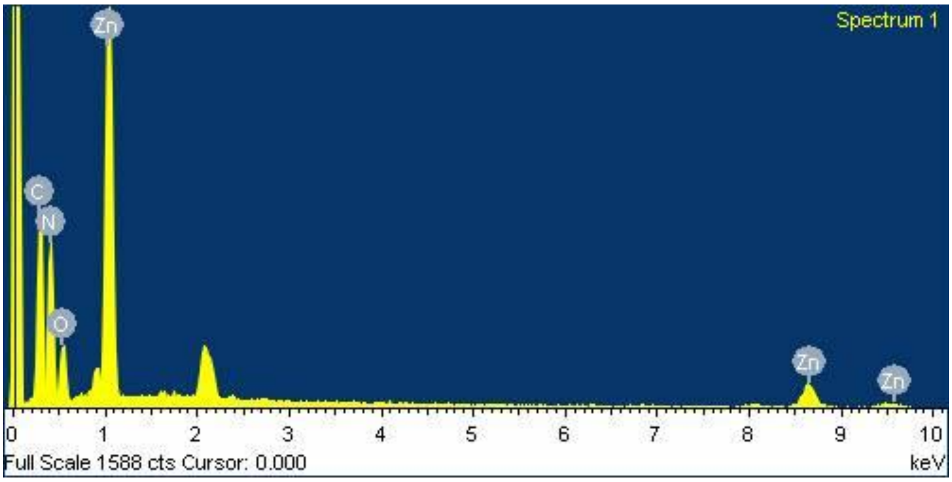

Figure 3. EDX analysis of ZnO/GCN. Elemental area mapping of Zn,O,N,C.

4. Gas Sensing Measurement

Gas sensing measurement has been performed on a fabricated sensor, as shown in Figure 4. Carbon interdigitated electrodes were fabricated on top of pre-cleaned glass substrates. Sensing material was deposited by using drop-casting. The figure below shows the change in resistance with respect to various concentrations of gas. The response and recovery time of the sensor were measured for 4% and 10% hydrogen gas. The response time for the 4% hydrogen concentration was observed to be 65 s, and it was 90 s for 10% hydrogen concentration, respectively.

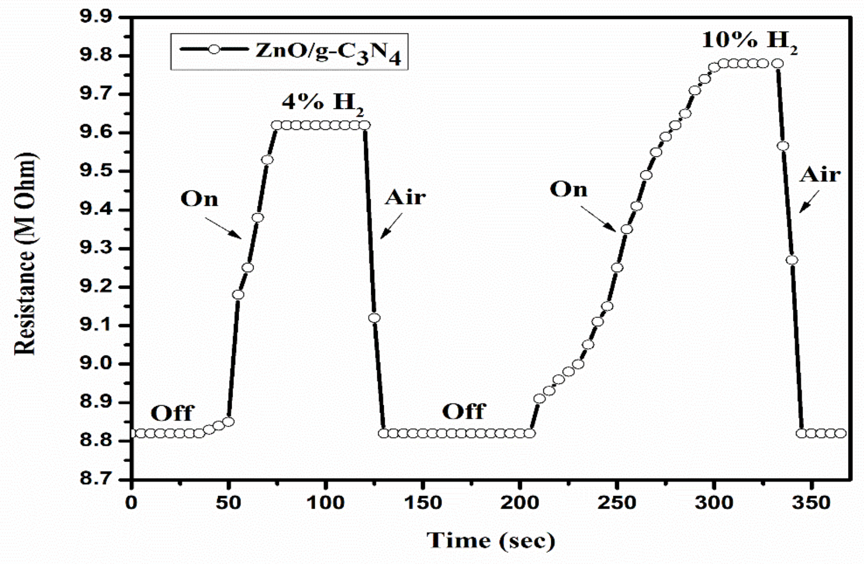

Figure 4. Real-time electrical resistance response of ZnO/g-C_3N_4at 4% and 10% H2 concentration at room temperature.

5. Conclusions

The ZnO/g-C_3N_4 nanocomposite was synthesized, followed by various characterizations to explore its properties. XRD showed the presence of all phases and purity of the material. The nanorods were more than 500 nm in length. ZnO nanorods were successfully impregnated on top of the graphitic carbon nitride matrix. The hydrogen gas sensing was studied, and promising data was recorded.

Institutional Review Board Statement:

Informed Consent Statement:

Data Availability Statement:

Conflicts of Interest: The authors declare no conflict of interest.

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