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Fabrication of Carbon Dots-Based Hydrogen Sulfide Gas Sensor

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Abstract
H_2S gas is a colorless and highly toxic gas found in natural sources like swamps and sewage, and produced during petroleum refining. As environmental safety concerns rise, the demand for efficient H_2S gas sensors increases. We have reported cost-effective nanocomposites synthesized by integrating green carbon dots (CDs) with zinc oxide (ZnO) for gas sensing application. Using a simple hydrothermal method, we achieved a chemiresistive H_2S gas sensor that operates at room temperature, with a 20% response at 5 ppm and quick response and recovery times of 14 and 19 seconds, respectively. Characterization techniques like FT-IR and XRD confirmed the successful synthesis of CDs-ZnO nanocomposites.

Keywords: H_2S Gas; Chemiresistive Gas Sensor; Room Temperature; CDs-ZnO Nanocomposites.

Introduction
As global awareness grows, there is an increasing concern regarding pollution, workplace safety, healthcare access, and the management of air quality. A range of toxic gases, such as hydrocarbons (HCs), nitrogen oxides (NOx), carbon monoxide (CO), carbon dioxide (CO_2), sulfur dioxide (SO_2), ammonia (NH_3), hydrogen sulfide (H_2S), and volatile organic compounds (VOCs), are released into the environment from various sources [1]. These harmful substances can lead to serious health problems, both directly and indirectly, and may contribute to dangerous situations that cause risks to individuals and communities [2]. Among all these gases, hydrogen sulfide (H_2S) is a colorless and highly toxic gas with the distinct smell of rotten eggs. Table 1 shows the harmful effects of inhaling H_2S gas at different concentrations [3]. As a result, this triggering gas motivates the researcher to develop a highly selective gas sensor with excellent sensing response, low energy consumption, and operational capabilities at room temperature [4].

Table 1: Harmful effects of H_2S when inhaled by workers at different concentrations.

S. No	Inhaling concentration	Duration	Harmful effects
1.	50-100 ppm	1 hr	Loss of appetite, irritation in the eyes, breathing problem
2.	100-150 ppm	15-30 min	drowsiness
3.	500-700 ppm	1 hr	Death within a minute
4.	1000-2000 ppm	-	Instant death

Transition metal oxide semiconductor nanomaterials, such as MgO, In_2O_3, WO_3, ZnO, SnO_2 etc are commonly used to fabricate H_2S gas sensors due to their oxygen vacancies, small grain size, and high surface volume ratio. ZnO stands out among these materials because of its wide band gap (3.3 eV), high conductivity, biocompatibility, good stability, and strong sensing response to both reducing and oxidizing gases. However, its high operating temperature limits practicality in real-world applications, leading researchers to explore carbon-based nanomaterials, which offer unique structural and electrical properties. From last 10 years, Carbon dots (CDs) have emerged as a promising 0-D nanomaterial with a size of 0-10 nm, high surface area, easy functionalization, and excellent electrical and thermal conductivities. Since these materials are functional at room temperature, so, the thought of hybridizing carbon nanomaterials with semiconducting oxides came to mind. In this work, we have reported CDs-ZnO nanocomposites-based gas sensor for H_2S gas sensing applications. We synthesized green CDs-ZnO nanocomposites using candle carbon soot (CCS) as a precursor by adopting in-situ hydrothermal method. This CDs-ZnO nanocomposite act as an active layer for the detection of 5 ppm concentrations of H_2S gas with low recovery time, good repeatability, complete resistance recovery at room temperature and excellent response time.

2. Experimental details

In the present work, in-situ carbon dots (CDs) incorporated with ZnO nanocomposites were synthesized by simple one step hydrothermal approach using candle carbon soot (CCS) as a precursor. The glass slide was successively pre-treated using different reagents, namely acetone, ethanol (purity Z99.9%, Hayman Ltd., England) and DI water. Afterwards, the candle was lit and the pre-treated glass slide was placed just above the candle flame and soot was collected. Meanwhile, ZnO nanoparticles were synthesized using Zinc acetate dihydrate Zn $(CH_3COO)_2.2H_2O$, (Thermofisher Scientific India Pvt Ltd, 97+%) and a 1M NaOH solution prepared by using NaCl pellets (Merck Life Science Pvt Ltd). For this 0.4 g of Zn $(CH_3COO)_2.2H_2O$ is added to 40ml DI water and further treated with 1M NaOH solution by adjusting the pH nearly to 12. Further, the collected CCS was then added subsequently to this mixture and subjected to magnetic stirring for half an hour to obtain a homogeneous solution as shown in Fig.1. This homogeneous solution is then treated hydrothermally for 10 hrs at 160° C in a Teflon-lined autoclave. However, after the completion of the reaction time, a clear solution with dispersed CDs-ZnO composites was obtained. The solution was subsequently centrifuged, filtered and washed with DI water until the pH value reached a neutral value.

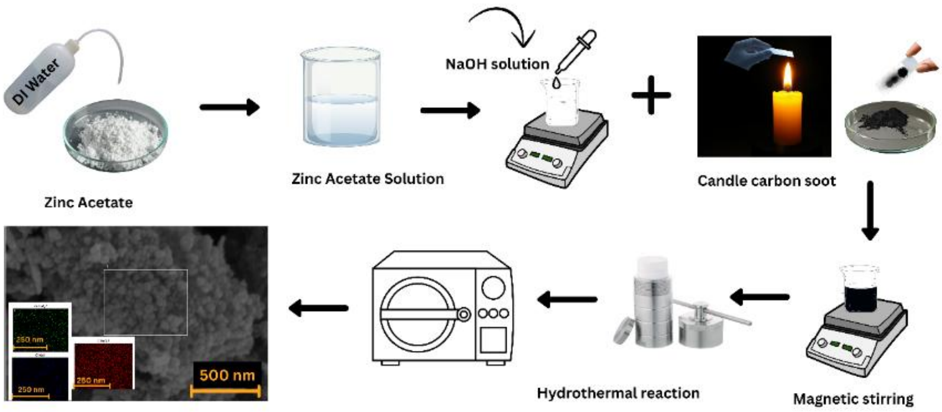

Fig. 1: Schematic diagram for the synthesis of CDs-ZnO nanocomposites.

3. Results and discussion

FTIR spectroscopy is utilized to investigate the surface functionalization of synthesized CDs and CDs-ZnO nanocomposites, as illustrated in Fig 2. The FTIR spectrum of the CDs exhibits a peak at 2327 cm^{-1}, indicative of O=C=O stretching. Upon incorporating ZnO, this peak intensifies and shifts to 2346 cm^{-1}, suggesting a blue shift due to stiffer bonding. A broad band near 2675 cm^{-1}, associated with carboxylic acid stretching from the hydrothermal synthesis, is present in both CDs and CDs-ZnO composites. At 2900 cm^{-1}, a –CH stretching peak appears in the CDs-ZnO nanocomposite, absent in the CDs. The spectrum also reveals –OH stretching in the range of 3400-3700 cm^{-1}, which increases in the CDs-ZnO nanocomposites, likely due to ZnO reduction producing oxygen. Overall, the CDs-ZnO composites show more intense peaks, indicating a higher density of functional groups and enhanced polar interactions, vital for effective sensing.

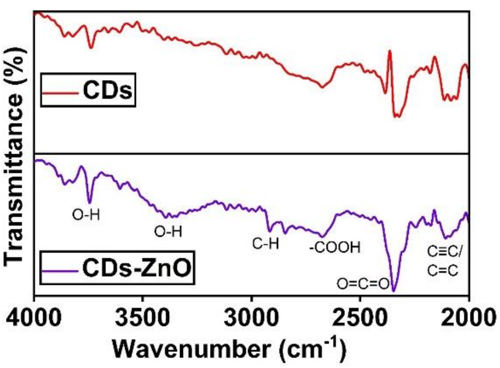

Fig.2: FT-IR of CDs and CDs-ZnO nanocomposite.

Moreover, the XRD pattern of CDs showing broad band at 2θ=21.6° corresponding to the (002) plane, with an interlayer spacing calculated as 0.41 nm approximately. Furthermore, this wide band may be attributed to a limited number of closely arranged layers. [5]. The appearance of broad band indicates the graphitic structure of CDs and similar findings were reported by Pan *et al* [6]. Moreover, this broad band is also observed in XRD pattern of CDs-ZnO nanocomposite along with some other prominent peaks are also recorded at 2θ angles of 31.9°, 34.6°, 36.4°, 47.7°, 56.7°, 63.06°, 66.5°, 68.12°, 69.3°, and 77.3°, which on comparing with (JCPDS No:36-1451) corresponds to the crystal planes (100), (002), (101), (102), (110), (103), (200), (112), (201), and (202) respectively and confirms that presence of the ZnO nanoparticles. It was observed that incorporating ZnO into CDs leads to a decrease in interplanar spacing from 0.41 nm to 0.25 nm. This change is observed may be due to the structural modifications, such as the attachment of functional groups, which might have created internal stresses within the material and these stresses may push the atomic planes close together and thereby decrease the interplanar spacing [7].

4. Fabrication and Sensing Measurement of CDs-ZnO Gas sensor

Measurements for H_2S gas sensing were performed in dynamic environment and conducted by applying a sensing film onto SiO_2 platform and followed by connecting copper wire using silver paste as shown in Fig 3. The silver paste was dried to maintain good adhesion between electrodes and tips of wire. SiO_2 substrate with thickness 200 nm was procured commercially by Graphene Supermarket, USA. For H_2S gas, 99.99% pure gas cylinder with 5 ppm concentration was purchased from Chemtron Science Laboratories Pvt.ltd.

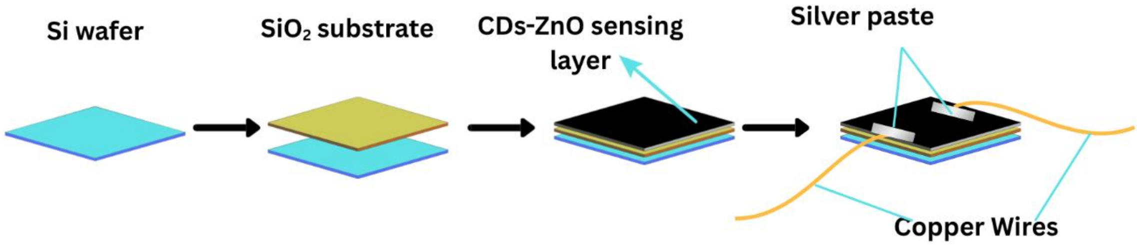

Fig 3: Schematic showing the fabrication of CDs-ZnO sensing device.

To investigate the response of the CDs-ZnO sensing device to H_2S gas, sensing device was positioned upside down in a sealed test chamber. The schematic of sensing set up is shown in Fig. 4 (a). The measurement process began by introducing air and recording the initial resistance. A 5-ppm concentration of H_2S gas was then introduced through a mass flow controller, and the change in resistance was recorded. Afterward, the sensor was exposed to air to reset to its baseline resistance. Response curves were obtained by applying a constant 0.1 V bias, with resistance changes recorded using a PC-based data acquisition system connected to a Keithley sourcemeter. Measurements were conducted at room temperature (25°C) and 40% relative humidity. We observed that the resistance of both the CDs and CDs-ZnO nanocomposite sensors increased with H_2S exposure and returned to baseline when the gas flow stopped. This resistance change is attributed to the adsorption and desorption of gas molecules. Additionally, the carbon in the CDs layer acts as a p-type material, further increasing the resistance of the sensing layer [8].

The sensing application of reported sensor is studied by assessing the sensing response of H_2S gas sensor and calculated according to the formula:

$$S\,(\%) = 100\left[\frac{R_{(Gas)} - R_{(Air)}}{R_{(Air)}}\right]\%$$

where R_{Gas} is the resistance of sensor when exposed to H_2S gas and R_{Air} is the resistance of sensor after exposure. Fig. 4(b) presents the sensitivity curve for both the CDs-ZnO nanocomposite and the CDs alone (shown in the inset). Observably, the sensor's response markedly improves upon exposure to H_2S gas, and it gracefully reverts to its baseline state following the air purge, demonstrating its effective sensing capabilities.

The gas sensing mechanism employed by the fabricated sensor, as shown in Fig. 5(c), is fundamentally driven by reactions involving chemisorbed surface oxygen species as discussed in Eq.No. 2 & 3. ZnO, known as an n-type semiconductor, acts effectively as a sensitive material due to its ability to form an electron depletion layer. [9]. When exposed to air, free electrons, which are the majority charge carriers in the conduction band of ZnO, interact with certain oxygen species that get chemisorbed onto the surface. Upon interaction with the target gas H_2S, the adsorbed oxygen species create active sites for the gas reaction. This process releases electrons, as shown in Eq.No. 4, [10] that subsequently recombine with holes present in the CDs layer of the CDs-ZnO composite which act as p-type because of the presence of carbon as discussed earlier. This recombination occurs as ZnO nanoparticles partially cover the CDs surface as evident from FE-SEM.

$$O_2\,(gas) \rightarrow O_2\,(ads) \qquad \text{Eq. No (2)}$$

$$O_2\,(ads) + e^- \rightarrow O_2^-\,(ads) \qquad \text{Eq. No. (3)}$$

$$2H_2S\,(gas) + 3O_2^-\,(ads) \rightarrow 2H_2O\,(gas) + 2SO_2\,(gas) + 3e^- \qquad \text{Eq. No. (4)}$$

This recombination process results in widening of the depletion layer, consequently increasing the overall resistance of the sensor. Thus, the enhanced sensing response observed can be directly linked to the morphological interaction between the CDs and the ZnO in the nanocomposite.

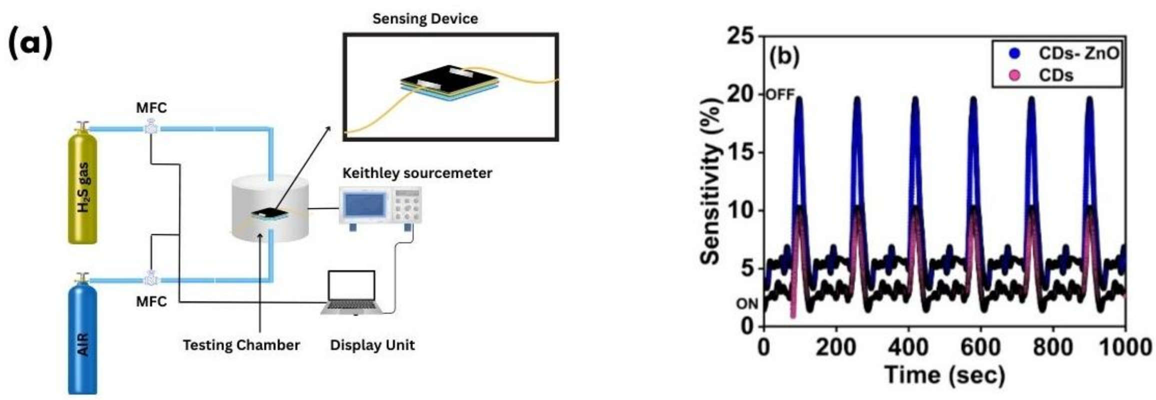

Fig. 4: (a) Schematic view of home-made sensing set up, (b) Sensing response of CDs-ZnO and CDs.

5. Conclusion

The investigation centers on the synthesis of a green nanocomposite material, CDs-ZnO, for use as a gas sensor. FT-IR analysis of the CDs-ZnO sensing layer shows a range of functional groups that enhance its capabilities, marking a significant advancement for sensing applications. The layer has been tested for detecting H_2S gas and demonstrates a commendable sensing response at room temperature, with quick response and recovery times even at concentrations as low as 5 ppm of H_2S.

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