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Room temperature ammonia sensor based on copper phthalocyanine

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Abstract
Ammonia, a colorless and highly irritating gas with a suffocating odor, which is widely used in industries and household cleaning materials. Overexposure to Ammonia may cause invisibility, lung damage, coma and sometimes may lead to death. Thus, sensing of ammonia becomes important. Conducting polymers and metallophthalocyanines have been found to be good gas sensing materials. The present study reports detection of ammonia using Cu-Phthalocyanine as a sensor. Cu-Phthalocyanine is synthesized by chemical method. The prepared sample is characterized using XRD, SEM, UV spectroscopy and FTIR and later developed in form of pellets. Using a two-probe sensing assembly, resistance of pellet is measured in presence and absence of ammonia at room temperature. The resulting change in electrical conductivity of Cu-Phthalocyanine on exposure to ammonia is used to evaluate the sensitivity. It is surmised that Cu-Phthalocyanine can serve as a good applicant for sensing ammonia.

Keywords: ammonia, Cu-Phthalocyanine, sensors, conducting polymers.

Introduction

Among many gases present in the environment, ammonia is one of the gases that is mainly used in industries such as agricultural industry, textile industry, rubber industry etc. It is also used in various metallurgical processes and also acts as a catalyst in production of some of the synthetic resins. It is also used in many household cleaning products. Over exposure of Ammonia can cause eyes, nose, throat irritation, lung damage, severe chest pain and may sometimes lead to death. The Occupational Safety and Health Administration (OSHA) states that the concentration of Ammonia over a 15-minute short period should be less than 35 ppm, hence sensing of ammonia is essential. Metallophthalocyanines (MPcs) viz. Copper Phthalocyanine (CuPc), Nickel Phthalocyanine (NiPc), Zinc Phthalocyanine (ZnPc) which are the derivates of phthalocyanine (Pcs) possesses π electron containing aromatic macrocycle and structural symmetry similar to Porphyrin system. Among all metallophthalocyanines, Copper Phthalocyanine possesses high thermal and chemical stability and are also rich in substitution chemistry. Copper Phthalocyanine responds to the exposed gas by changing electrical conductivity due to exchange of electrons. Copper Phthalocyanine has high electrical stability and Ammonia can be sensed over a wide range of concentrations. Copper Phthalocyanine is more easily synthesized and processed, and has been reported to exhibit greater sensitivity and selectivity towards some analyte gases at room temperature.
In this paper, we report synthesis, characterization and ammonia sensing study of Copper Phthalocyanine.

2. Experimental

Copper Phthalocyanine is synthesized by reacting phthalic anhydride, urea and copper nitrate. Phthalic anhydride, urea and copper nitrate were taken in the proportion of 4:4:1. These precursors were mixed properly on crushing. Then the mixture was stirred constantly and kept for heating. After heating, the slurry of the mixture was obtained and the cyclotetramerization reaction took place after heating it again. The reaction was observed to be very active in nature. The reaction took place at about 200-250 °C. After completion of reaction there was colour change of the product obtained. The surplus of the material was converted to fine powder. The obtained powder was washed with methyl alcohol and distilled water for more than 20 times so that the precursors which were inactive were removed. Then the powder was air-dried under IR lamp and was further crushed into fine particles. Then the sample was shaped in form of pellets. Copper phthalocyanine powder of 300 mg was converted into pellets. Pellets were formed by applying a pressure of 5 tonnes for 2 min with the help of a hydraulic press. The diameter of the pellet was 12 mm.

3. Characterization
Synthesized Metallophthalocyanines were characterized using various characterization techniques such as X-ray diffraction (XRD), UV-visible absorption Spectra, Fourier Transform Infrared Spectra (FTIR) and Scanning Electron Microscopy (SEM).

3.1 X-ray Diffraction
The characterization of the Copper phthalocyanine powder was carried out using a Philips X-ray diffractometer with Cu K_α radiation (=1.546 A°) for crystal structure as well as particle size determination. The result of XRD pattern of Copper Phthalocyanine is discussed below. The average grain size of the sample is 56.4 nm which is calculated by Scherer formula. The (100), (102), (112), (311) are the planes observed in the figure given below. The grain size of copper phthalocyanine is in agreement with the standard JCPDS number (11-0893). The copper phthalocyanine is found to be in β stable form.

3.2 Ultraviolet-Visible Absorption Spectra
For Phthalocyanine characterization method, UV Visible Absorption Spectroscopy is one of the most versatile methods. The spectrum of Metallophthalocyanines basically originates from molecular orbitals. At several different wavelengths the electronic transitions are given by the simplest unit that is 18 π electron system. The most prominent bands occurring are from 600 nm to 750 nm. The absorption peaks are obtained using absorption spectrometer JASCO V-607. The absorption peaks observed by Ultra Violet (UV) region from 200 nm to 350 nm reveals three peaks which are known as soret bands. The results obtained from absorption spectra of Copper Phthalocyanine shows the λ_{max} value of Copper Phthalocyanine is 545 nm.

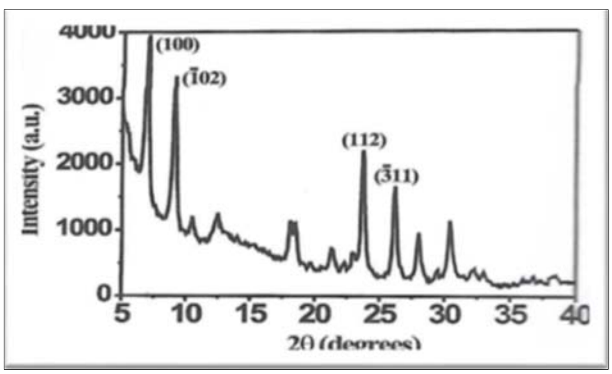


Fig 1: XRD of Copper Phthalocyanine

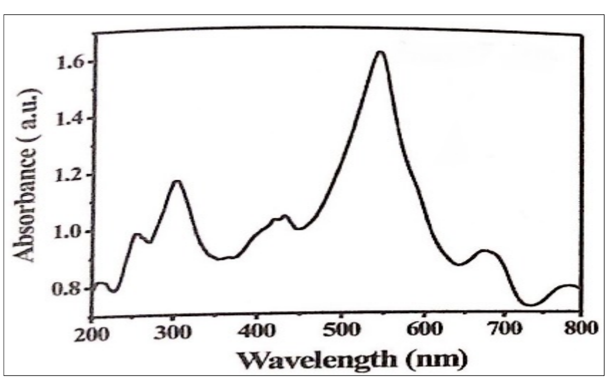


Fig 2: UV-Visible Spectra of Copper Phthalocyanine

3.3 Fourier Transform Infrared Spectra
With the help of JASCO FT/IR-6100 type A spectrophotometer having range from 400 cm^{-1} to 2000 cm^{-1}, IR spectra of the synthesized Copper Phthalocyanine were performed at room temperature. The copper phthalocyanine material is mixed with IR-Potassium Bromide (KBr) powder. The peaks observed in IR spectrum at wave numbers 3446.7, 2369.3, 1559.8, 1472.4, 1296.7, 1121.8, 799.7 cm^{-1} confirm the formation of copper phthalocyanine.

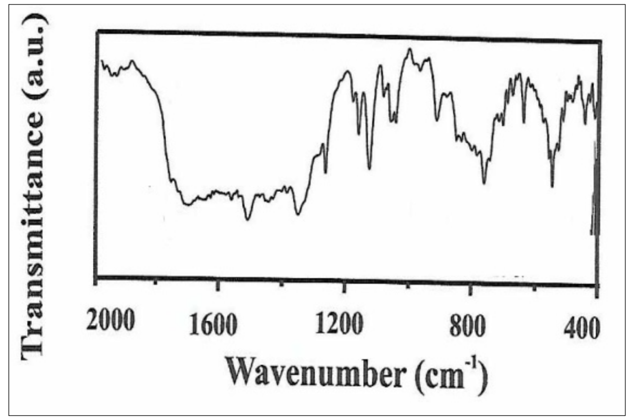


Fig 3: FTIR of Copper Phthalocyanine

3.4 Scanning Electron Microscopy
SEM image of synthesized powder of Copper Phthalocyanine is shown in the figure. SEM image shows the crystalline nature of Copper Phthalocyanine. SEM image of Copper Phthalocyanine shows uniform size distribution in tubular form having average size 2800 nm. Formation of crystal structure of Copper Phthalocyanine nano particle is seen in the image of SEM.

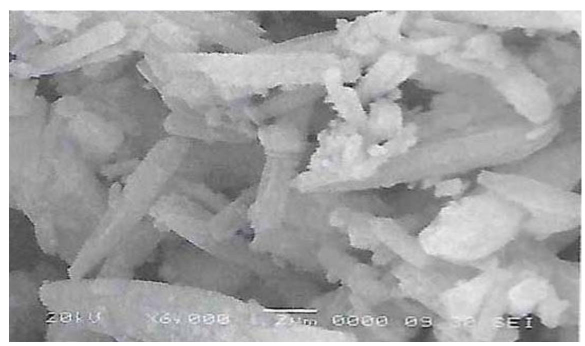
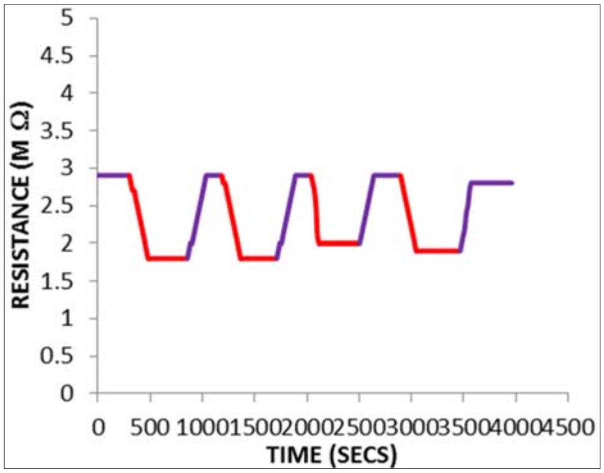


Fig 4: SEM of Copper Phthalocyanine

4. Results and Discussion
The electrical resistance of copper phthalocyanine was determined on exposure of ammonia. The copper phthalocyanine pellet of diameter 12 mm was placed in a gas chamber. The gas sensitivity characteristics were investigated by recording resistance when pellet was exposed to 100 ppm concentrated ammonia at room temperature. The significant change in the electrical resistance of the sample was recorded when it was exposed to 100 ppm of ammonia. Figure below shows variation in resistance with exposure and removal of ammonia.



Sensitivity was calculated as a ratio of change in resistance of the pellet after exposing to ammonia to resistance of pellet before exposure to ammonia.

Cycle wise comparison of Copper Phthalocyanine

Cycle No.	Sensitivity (%)	Response Time (sec)	Recovery time (sec)
1.	89.47	540	300
2.	84.21	525	270
3.	57.89	450	375
4.	94.73	555	495

Chemoresistive response of metallophthalocyanine is due to the changes of electrical conductivity of samples on the adsorption of electrons donating or electrons accepting gas species.

The significant change in electrical resistance of Copper Phthalocyanine sample was observed when it was exposed to ammonia. It showed that resistance of material decreases when it is exposed to ammonia since it is a reducing gas where Copper reacts with Nitrogen in ammonia. The effect of donating electrons to sensing materials results into reduction of number of holes which decreases the resistance of sensing material. The change in resistance of sensing material is due to change in charge carrier concentration which is due to adsorption of gas molecules on the surface of sensing material. Nitrogen is electron rich element responsible for showing decrease in resistance. When the pellet was exposed to air again, reversible nature was recorded.

5. Conclusion

We can conclude that Copper Phthalocyanine is a good sensing material for the detection of ammonia at room temperature. Sample of Copper Phthalocyanine showed good reproducibility with relatively faster response for ammonia.

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