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Effect of Poly Ethylene Glycol on Moisture Sensing of Copper Ferrite Nanocomposite

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Abstract Present paper reports, synthesis of $CuFe_2O_4$ nanocomposite via chemical precipitation route and effect of poly ethylene glycol on their humidity sensing properties. The variations of resistance with and without PEG at different value of %RH of the sensing elements were investigated. The maximum average value of sensitivity was found 13.7 MΩ/%RH respectively over the entire range of relative humidity. Results are found to be reproducible after three months with ±5 % hysteresis. Sensing material synthesized through PEG has been characterized by X-ray diffraction (XRD) and Scanning electron microscopy (SEM). XRD pattern revealed cubic crystal system. The average crystallite size of material was found to be 85.7 nm. SEM images show porous nature of sensing material with a number of active sites throughout the surface. The average size of pores of material was found to be 2µm.

Keywords: *humidity, sensors, sensitivity, morphology, composite materials*

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1. Introduction

In recent years on account of the attractive scientific and industrial applications of Ferrite nanoparticles, novel methods for their synthesis and new approaches for their characterizations have been reported [1,2,3]. It has been reported that different techniques of preparation lead to different phases and different degrees of size control [4,5,6,7]. Humidity affects mankind directly or indirectly. The irreversible effect due to humidity eventually causes permanent damage to the exposed surfaces. Therefore, there is an urgent need to measure and control precisely the humidity in various environments [8]. Ferrites show very good surface reactivity and they have temperature dependent surface morphology [9]. They also show remarkable catalytic properties in oxidation reactions due to the high oxygen ion mobility at the film surface and thus are highly interesting for the development of sensors [6]. Copper ferrite material exposes porous nature and various constructive applications in different fields [10,11].

2. Synthesis of Material

The stoichiometric amount of $[CuCl_2.2H_2O]$ and $Fe(NO_3)_3$ were separately dissolved in isopropyl alcohol and solution ammonium hydroxide (NaOH) was added drop by drop to obtain material in hydroxide form. Vigorous stirring was done 20-24 h to ensure complete and intimate reaction between various components.

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Later the materials were dried for 8-10 h at 100°C in an oven and calcined at 400°C for 2 h, resulting in complete crystallization into powder. The powders of synthesized materials S_1 and S_2 were compacted in to pellets of about 3 mm thickness and 9 mm diameter, at a pressure of 618 MPa using hydraulic press (KBR Press, Germany).

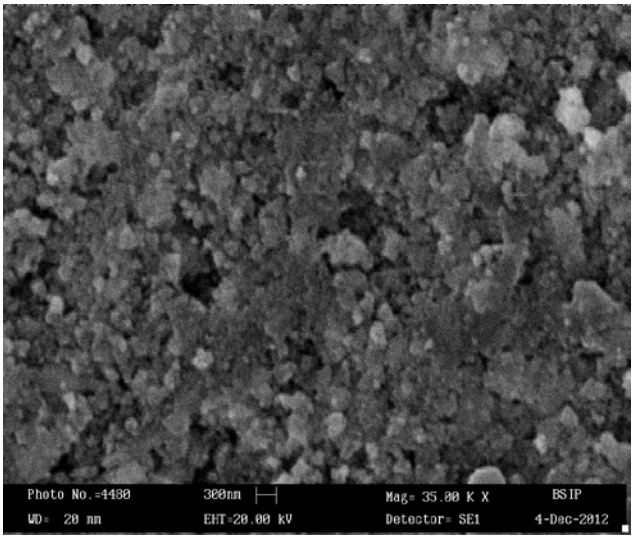

Figure 1(a). SEM image of pellet surface of material S_2 at nano scale

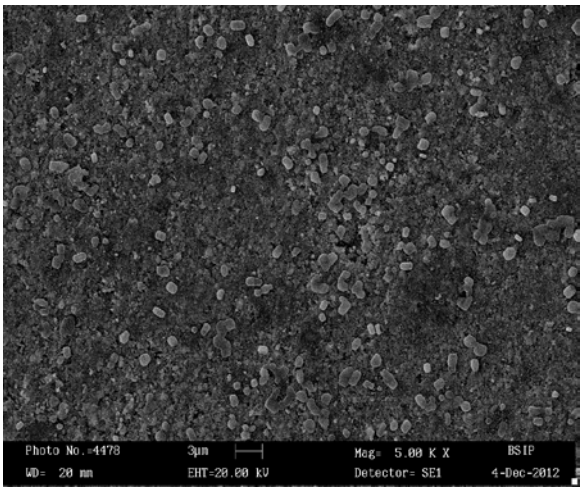

Figure 1(B). SEM image of pellet surface of material S_2 at micro scale

3. Characterizations of Material

a. Scanning Electron Microscopy (SEM)

Scanning electron micrograph as shown in Figure 1(a) and 1 (b) reveal that surfaces of the pellet prepared from sample S_2 is macro porous and macro porous and the clustering of the particles occurs over the different crystallites of materials which enables larger surface area for the adsorption of water molecules.

Therefore the pellet can absorb more atmospheric oxygen due to more exposed surface area of the pellet to react yielding high sensitivity towards moisture. The average size of pores was calculated by using Scanning Electron Micrographs. The average size of pores of nanocomposite $CuFe_2O_4$ was found to be 2µm.

b. X-Ray Diffraction

The crystal structure and phase of the powdered material S_2 was analyzed using X-ray Diffractometer (X-Pert, PRO PANalytical XRD system, Nether land) with Cu K_α radiations as source having wavelength 1.5418 Å. XRD pattern of the synthesized powder shown in Figure 2 reveals that copper ferrite is cubic crystal structure. It illustrate that the sensing material consists of $CuFe_2O_4$ along with peaks CuO, Fe_2O_3. The high intensity peak, centered at 2θ = 35.57º is assigned to cubic crystal system having 'd' spacing 2.523 Å and FWHM 0.1171.

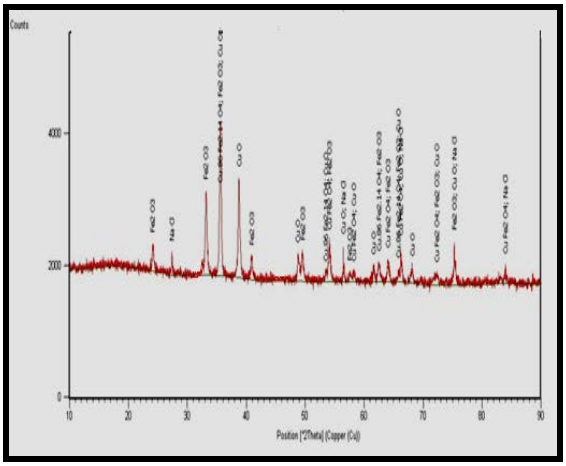

Figure 2. XRD pattern of synthesized powder S_2

The average crystallite size (D) of the sensing material can be calculated by the Debye-Scherrer's formula, which is given by

$$D = Kl / b\cos\theta$$

where K= 0.94 is Scherrer's coefficient, which depends on the shape of the crystallite and the type of defects present, λ is the wavelength of X-ray radiation, β is the full width at half maximum (FWHM) of the diffraction peak and θ is the angle of diffraction. The average crystallite size of copper ferrite calculated from Debye Scherer's formula was found to be 85.7 nm.

Experimental set up as shown in Fig. 3 was used for this investigation. A saturated solution of potassium hydroxide was used as a dehumidifier and a saturated solution of potassium sulphate was used as humidifier. The humidifier/dehumidifier was kept in a dish over a stand. Variations in %RH were measured with the help of hygrometer (Huger, Germany) and variations in resistance were measured Kithely Electrometer (Model: 6514). The temperature of the chamber remained the same throughout the observations. The chamber was then dehumidified up to 10%RH.

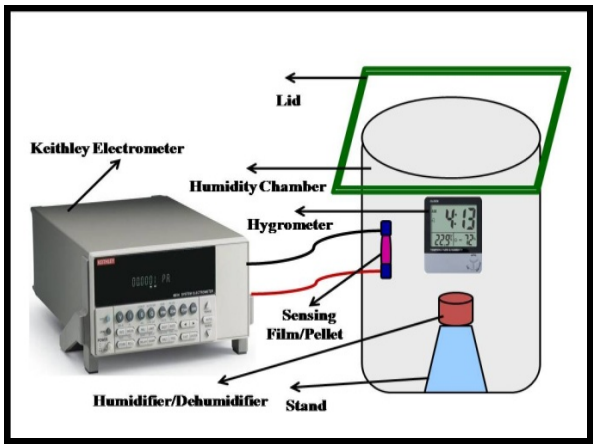

Figure 3. Experimental-set-up

The prepared pellet of sensing material was put within a conductivity-measuring holder having Cu electrode-pellet-Cu electrode arrangement. The Cu electrode was square-shaped of a side length 1.2 cm having a gap 0.4 cm between them. It was observed that as %RH inside the chamber increases from 10-90%RH, resistance of the sensing material decreases over the entire range of RH. Sensitivity of humidity sensor has been defined as the change in resistance (ΔR) of the sensing element per unit change in %RH [12].

4. Result and Discussions

Sensitivity of sensing pellet was calculated. Figure 4 clearly reflects that as sensing material prepared sample (S_1) without and with PEG (S_2) show that as RH increases, resistance decreases sharply up to 50%RH and shows highest sensitivity in this range followed by a less rapid decrease up to 90%RH as relative humidity increases.

The average sensitivity for samples S_1 and S_2 were found to be 8.11 MΩ/%RH and 13.7 MΩ/%RH over the entire range of relative humidity. From the Figure 5 and Figure 6, it was observed that the sensing pellet prepared

from samples S_2 (with PEG) shows less hysteresis between the curves for increasing and decreasing relative humidity over the entire range with resistance. The average values of hysteresis were obtained as ±7% and ±2 % from the original curves for samples S_1 and S_2 respectively. The effect of aging after three months from the fabrication of pellet from material S_2 was shown in Figure 7. The results were found to be reproducible with ±5% hysteresis.

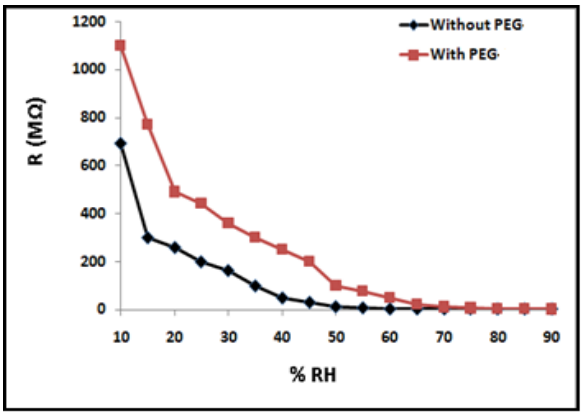

Figure 4. Variations of resistance for sensing element prepared from S_1 and S_2 with relative humidity

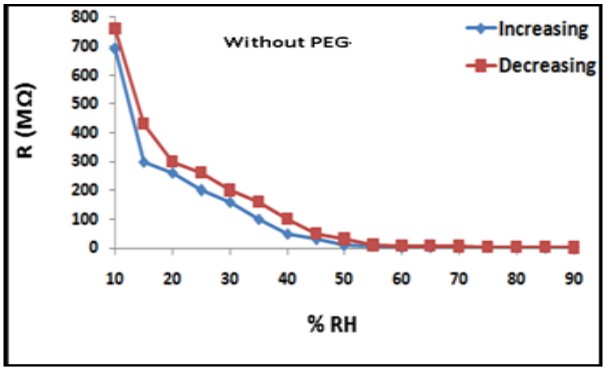

Figure 5. Hysteresis curve for pellet prepared from sample S_1

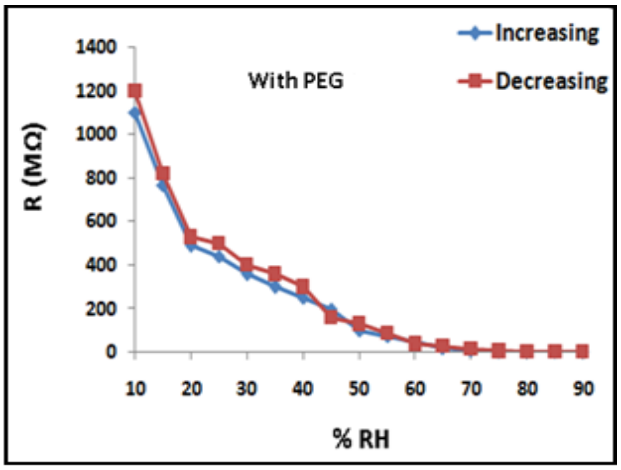

Figure 6. Hysteresis curve for pellet prepared from sample S_2

5. Humidity Sensing Mechanism

The composite material is porous in nature and has surface oxygen atoms which essentially arise due to the sample preparation technique. When the material adsorbs the humidity, its resistance decreases due to the increase of charge carriers, protons, in the ferrite and water system [12]. The adsorption of water on the surface of the material leads to the dissociation of hydrogen ions. These hydrogen ions bonded with the surface lattice oxygen atom, forms the hydroxyl groups [13] as shown in the equation:

$$H^+ + O_o \leftrightarrow [oh]^-$$

where O_o corresponds to oxygen at lattice sites. The hydroxyl groups thus produced are bonded with the lattice of the atoms and liberate the free electrons [14]. This electron is responsible for electrical conduction.

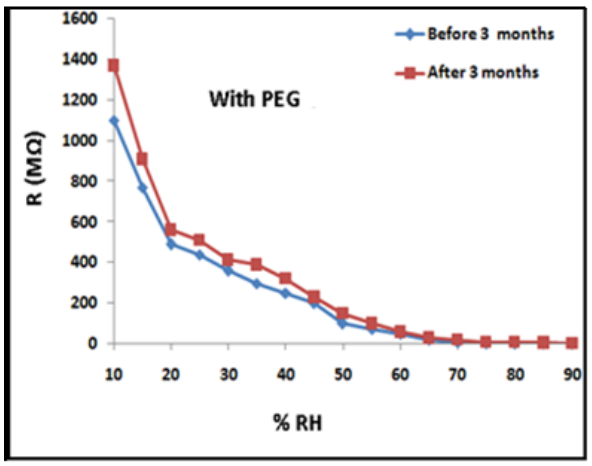

Figure 7. Reproducibility curve for sensing pellet fabricated from material S_2

6. Conclusion

Nanocomposite $CuFe_2O_4$ prepared at room temperature gave good humidity sensing properties for water vapors. The maximum average sensitivity 13.7 MΩ/%RH was achieved for sample synthesized through with poly ethylene glycol over the entire range of relative humidity. The poly ethylene glycol achieves a great role for preventing the grain grow and provide the bulky surface area for adsorption of water vapor through the materials which exhibit maximum sensitivity at room temperature. The results are found to be reproducible with less hysteresis. Thus humidity sensor made of $CuFe_2O_4$ nanocomposite based on electrical resistance is cost effective, easy to fabricate and user friendly and can be used for both indoor and outdoor applications for entire range of the %RH.

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