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Comparative humidity sensing based on Fe_2O_3 synthesized via different methods

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Abstract
Ferric oxide nanomaterial was synthesized by two hydrothermal precipitation methods as B_1 and B_2. Pellets as sensing elements were subjected to specially designed humidity chamber and variations in resistance with relative humidity (%RH) were measured. Pellet prepared from B_2 reveal maximum average sensitivity 6.61 MΩ/%RH. Structural analysis confirmed the formation of Fe_2O_3 with α-phase and rhombohedral structure. Average crystallite size of materials for B_1 and B_2 were found 40 and 18 nm respectively. SEM images show more porosity (largest surface area) of material for B_2. TEM image of material for B_2 shows uniform distribution of particles having average particle sizes are around 2 nm. Optical and thermal properties were investigated by using UV-visible absorption spectroscopy and Differential scanning calorimetric techniques.

Keywords: Humidity, SEM, Sensitivity, Nanometer, Pores.

Introduction

Ferric oxide has a wide range of applications as magnetic material [1], but in recent years it is very sensitive to humidity and can be used as humidity sensor element [2].

Synthesis of Materials

For Method B_1, hydrated ferric oxide was formed as equ.(1). The solution with the precipitate was thoroughly stirred at higher speed for 2h in ambient environment. After filtration the precipitate was dried at 400°C for 2 h to be convert ferric oxide in powdered form

$$Fe_2(SO_4)_3 + 6NH_4OH \longrightarrow 2Fe(OH)_3 + 3(NH_4)_2SO_4 \quad (1)$$

For Method B_2, Sodium hydroxide was added drop wise into 0.1 M ferric chloride solution with vigorous stirring as equ. (2). The flask was magnetically stirred for 2 h. The mixture was cooled naturally to room temperature. The product thus obtained was washed successively by deionized water and then dried at 600°C in electrical furnace for 2h.

$$FeCl_3 + 3NaOH \longrightarrow Fe(OH)_3 + 3NaCl \quad (2)$$

The powders synthesized from methods B_1 and B_2 were compacted in to pellets of about 3 mm thickness and 9 mm diameter, at a pressure of 618 MPa using hydraulic press.

Characterizations

Fig.1a and 1b shows the XRD patterns of materials prepared from methods B_1 and B_2. These patterns show pure α-Fe_2O_3 phase. The average crystallite sizes of samples synthesized from methods B_1 and B_2 were found 40 and 18 nm respectively. SEM of pellet prepared from method B_1 (Fig. 2a) shows that crystallites of Fe_2O_3 combining with each other form clusters leaving more spaces as pores. The sample prepared from B_2 (Fig. 2b) shows more porosity, giving largest effective surface area. The morphology of the sample synthesized from B_2 has also been observed by TEM. Fig. 3 shows the TEM micrograph of the ferric oxide prepared from method B_2. This shows the crystalline nature and uniform distribution of particles with the average particle sizes are around 2 nm. Fig. 4 depicts the UV-visible absorption spectra of ferric oxide (method B_2) in the photon energy range 1.24 to 6.19 eV (i.e. wavelength range 200-1000 nm). DSC curve of as powder synthesized from method B_2 is shown in Fig. 5. The curve shows three exothermic peaks of about 40°C, 92°C and 204°C and is due to the evaporation of chemical impurities and water.

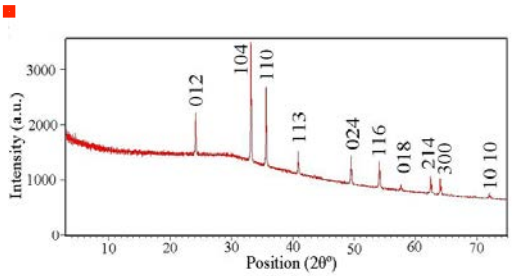

Fig. 1a XRD patterns of materials prepared from methods B_1

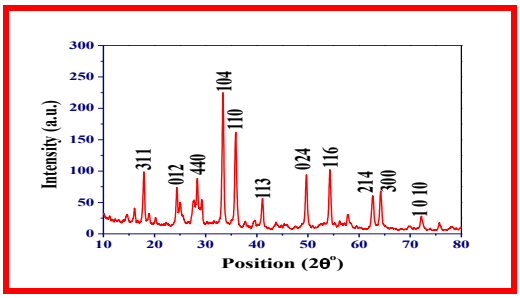

Fig. 1b XRD patterns of materials prepared from methods B_2

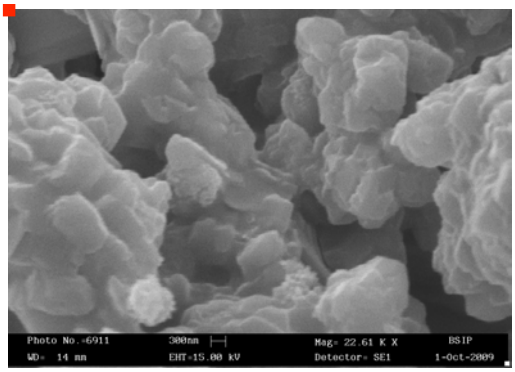

Fig. 2a SEM of pellet prepared from method B_1

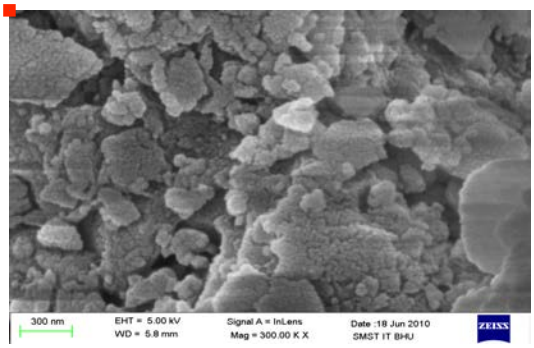

Fig. 2b SEM of pellet prepared from method B2

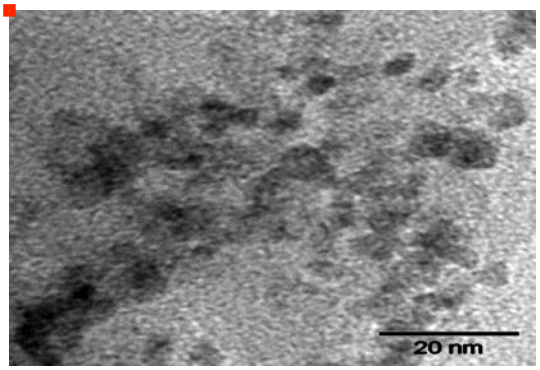

Fig.3 TEM micrograph of the ferric oxide prepared from method B_2

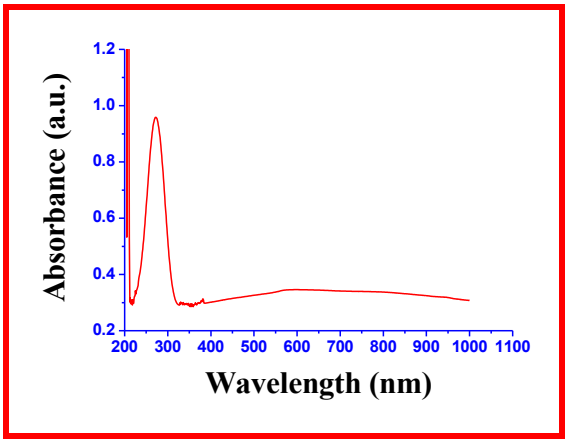

Fig. 4 UV-visible absorption spectra of ferric oxide prepared from method B_2

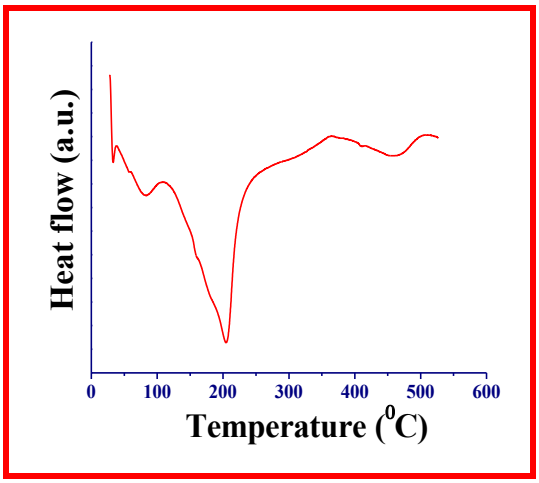

Fig.5 DSC curve of as powder synthesized from method B_2

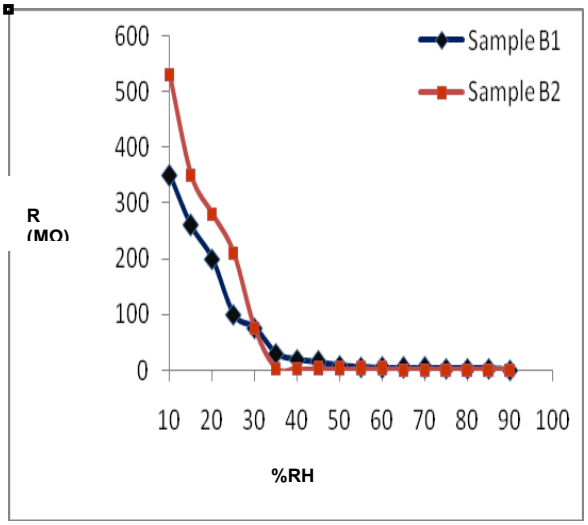

Fig. 6 Variation of relative humidity with %RH for samples prepared from methods B_1 and B_2

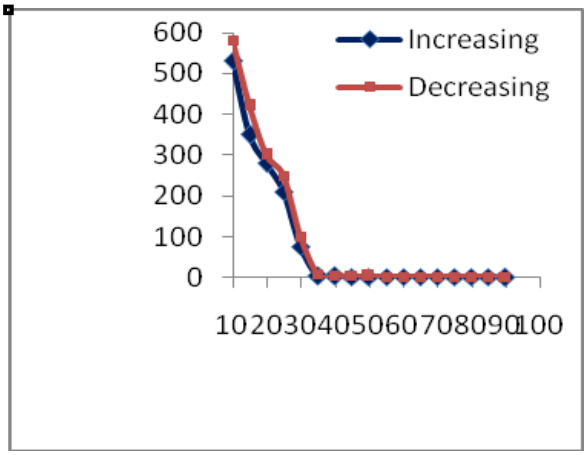

Fig. 7 Hysteresis for sensing element prepared from method B_2

Measurements of humidity sensing properties

Experimental set up reported earlier [3] was used for this investigation. The prepared pellet of sensing material was put within humidity chamber and it was observed that as %RH inside the chamber increases from 10-90%RH, the resistance of the sensing materials decreases over the entire range of RH. Fig. 6 clearly reflects that as sensing material prepared film method B_2 shows rapid decrease of resistance with increase of %RH from 10 to 90%. Fig. 7 shows the material prepared from method B_2 shows less hysteresis between the curves for increasing and decreasing relative humidity. Results are found to be reproducible with less hysteresis.

Results and Conclusions

In the present investigation we have successfully synthesized the ferric oxides with different morphologies. The average sensitivities of sensors for methods B_1 and B_2 were found 4.37 MΩ/%RH and 6.61 MΩ/% RH respectively over the entire range of relative humidity. The improved sensing performance of ferric oxide prepared from method B_2 may be attributed to their porous spherical structure and minimum crystallite size as 18 nm. Ferric oxide prepared from B_2 provides many sites to absorb the water vapor molecule and shows good sensitive property. Thus the experimental results demonstrate that nano-sized ferric oxide (B_2) without incorporation of dopants appears to be a promising material for the Humidity sensing.

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