

COPRECIPITATION SYNTHESIS OF ZINC FERRIT ($\text{Fe}_2\text{O}_3/\text{ZnO}$) NANOPARTICLES PREPARED BY CTAB SURFACTANT

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ABSTRACT

Zinc ferrite ($\text{Fe}_2\text{O}_3/\text{ZnO}$) nanocomposites were successfully synthesized by simple co-precipitation method via iron (III) nitrate 9-hydrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) and zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) as precursor in the presence of cetyltrimethylammonium bromide (CTAB) surfactant. The samples were characterized by high resolution transmission electron microscopy (HRTEM), field effect scanning electron microscopy (FESEM), X-ray diffraction (XRD), X-ray fluorescence (XRF) and Fourier transform infrared spectroscopy (FTIR) in different temperature. The XRD results showed that Fe-doped ZnO was single-phased with a hexagonal wurtzite structure ZnO and spinel phase of rhombohedral $\text{-Fe}_2\text{O}_3$. The particle size of as-prepared sample was in the range size of 30-40 nm and annealed one was around 37 nm in diameter at 500 °C for 3 hours. The TEM studies showed the rod-like shaped nanosized particles. The sharp peaks in FTIR spectrum determined the element of Fe-Zn nanoparticles. The result of XRF analysis showed the peaks of iron and ferrite elements with less Cl, F and Ni impurities.

Keywords: $\text{Fe}_2\text{O}_3/\text{ZnO}$; CTAB; chemical synthesis; coprecipitation.

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1. INTRODUCTION

Magnetic nanoparticles embedded in different non-magnetic matrices have been intensively investigated recently [1–8]. The synthesis of composite particles consisting of magnetic cores and luminescent cells such as $\text{-Fe}_2\text{O}_3/\text{ZnO}$ has gained acceptance in few years due to its magnetic, photoluminescence and catalytic properties [9] and also, as active element in gas sensors [10]. This type of composite particles has biological and biomedical potential applications such as detection of cancer cells, bacteria and viruses, and magnetic separation [11]. Recently Ruipeng Fu *et al.* [12] prepared $\text{-Fe}_2\text{O}_3/\text{ZnO}$ composite particles via a simple solution method, and investigated its morphology; indicating that the $\text{-Fe}_2\text{O}_3/\text{ZnO}$ composite particles are of typical sphere-like morphology with diameter in the range of 300-400 nm. Also, the $\text{-Fe}_2\text{O}_3/\text{ZnO}$ composites exhibit magnetic response to an external magnetic field and efficient characteristic emissions of ZnO under UV excitation. In this work, we report a simple synthesis of Fe/Zn samples by co-precipitation route using ferric nitrate $[\text{Fe}(\text{NO}_3)_3]$, zinc nitrate as source material and (CTAB) as surfactant and catalyst. Structural and surface morphological properties are discussed by XRD, HRTEM, FESEM, XRF and FTIR analyses.

2. EXPERIMENTAL DETAILS

$\text{-Fe}_2\text{O}_3/\text{ZnO}$ nanoparticles were synthesized by a simple co-precipitation synthesis according to the following manner. Firstly 2g, $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was dissolved in 60mL and then 1 g, CTAB surfactant was added to the solution with stirring at room temperature. After 5 min, 2 g, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ was added to the solution and then 20 mL ethanol was slowly added to the solution. After 30 min, the temperature was increased from 30°C to 90°C . The color of solution changed from milky color to red color by adding iron precursor. The pH was adjusted 2 during the synthesis. The product were evaporated for 2 hours, cooled to room temperature and finally calcined at 500°C for 4 hours. All analyses were done for samples without any washing and purification.

The specification of the size, structure and optical properties of the as-synthesis and annealed nanoparticles were carried out. X-ray diffractometer (XRD) was used to identify the crystalline phase and to estimate the crystalline size. The XRD pattern were recorded with 2θ in the range of $4-85^\circ$ with type X-Pert Pro MPD, Cu-K : $\lambda = 1.54 \text{ \AA}$. The morphology was

characterized by field emission scanning electron microscopy (SEM) with type KYKY-EM3200, 25 kV and transmission electron microscopy (TEM) with type Zeiss EM-900, 80 kV. Fourier transform infrared spectroscopy (FTIR) with WQF 510. All the measurements were carried out at room temperature.

3. RESULTS AND DISCUSSION

X-ray diffractometer (XRD) with CuK radiation, operated at 40 kV, 250 mA was used to identify crystalline phases and to estimate the crystalline sizes. Figure 1 shows the X-ray diffraction patterns of the powder before and after heat treatment. Figure 1(a) shows the XRD pattern of Iron ceria sample before annealing. Figures 1(b) shows the XRD patterns of iron cerium after annealing at 500 °C. The exhibited peaks correspond the γ -Fe₂O₃ and ZnO nanoparticles clearly show all the diffraction peaks of rhombohedral γ -Fe₂O₃ and hexagonal wurtzite ZnO respectively. The mean size of the ordered FeCe nanoparticles has been estimated from full width at half maximum (FWHM) and Debye-Sherrer formula according to equation the following:

$$D = \frac{0.89\lambda}{B \cos \theta} \quad (1)$$

where, 0.89 is the shape factor, λ is the x-ray wavelength, B is the line broadening at half the maximum intensity (FWHM) in radians, and θ is the Bragg angle. The mean size of as-prepared samples was around 40 nm from this Debye-Sherrer equation.

SEM analysis was used for the morphological study of nanoparticles of samples. These analyses show that high uniformity emerged in the samples surface by increasing annealing temperature. With increasing temperature size of the particles decreased from 45 nm to 35 nm. Figure 2(a) shows the SEM image of the as-prepared Fe/Zn nanoparticles prepared by this method. Figure 2(b) shows the SEM image of the annealed FeCe nanoparticles at 500°C for 3 hours. It can be seen that the particles were changed from sphere-liked shaped to rod-shaped with less aggregation. The diameter of as-prepared nanoparticles was in the range size of 45 nm and for annealed one was around 37 nm in diameter at 500°C.

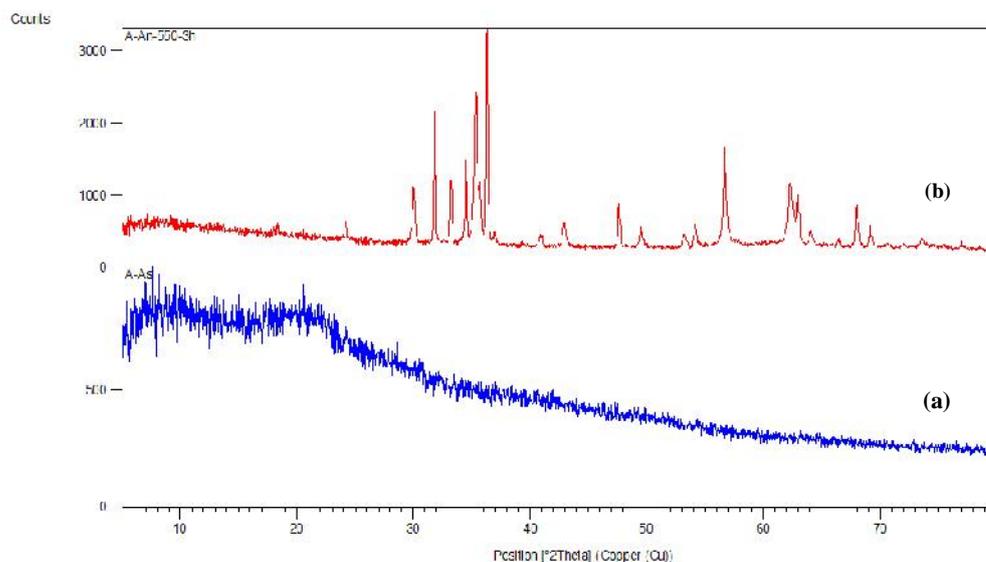


Fig.1. XRD patterns of (a) as-prepared and (b) annealed Fe-Zn samples

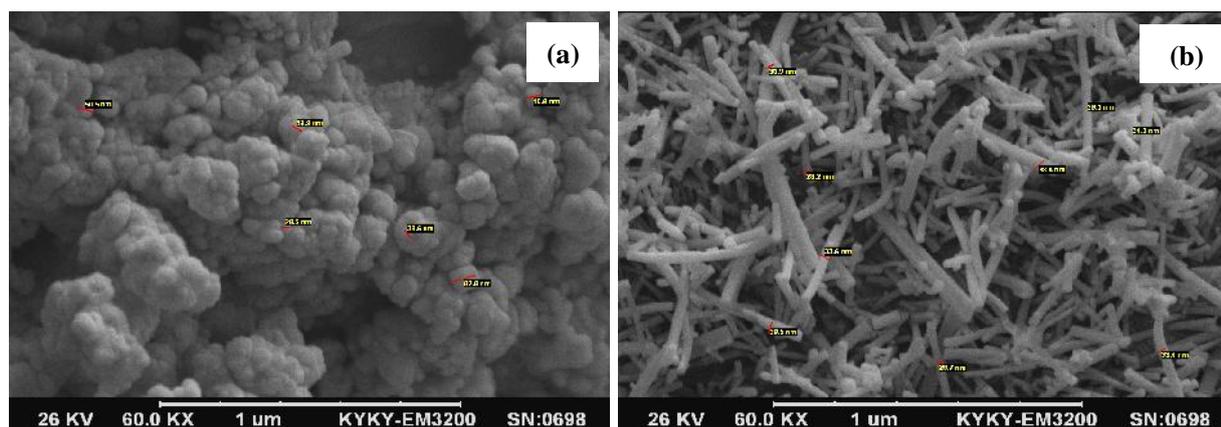


Fig.2. SEM images of the (a) as-prepared and (b) annealed Fe/Zn nanoparticles at 500⁰C

The transmission electron microscopic (TEM) analysis was carried out to confirm the actual size of the particles, their growth pattern and the distribution of the crystallites. Figure 3 shows the as-synthesized TEM image of squared-like Fe/Zn nanoparticles with average diameter of 35 nm prepared by coprecipitation synthesis route.

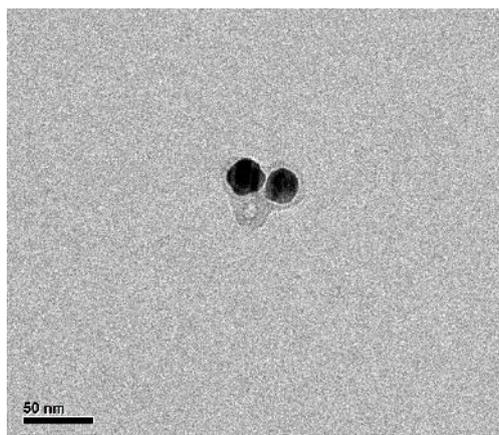


Fig.3. TEM image of the as-prepared FeZn nanoparticles

In figure 4, the infrared spectrum (FTIR) of the synthesized Fe/Zn nanoparticles was in the range of 400-4000 cm^{-1} wavenumber which identify the chemical bonds as well as functional groups in the compound. The large broad band at 3402 cm^{-1} is ascribed to the O-H stretching vibration in OH^- groups. The absorption peaks around 1646 cm^{-1} , 1465 cm^{-1} are due to the asymmetric and symmetric bending vibration of C=O. The strong band below 700 cm^{-1} is assigned Fe-Zn stretching mode. The bands corresponding to Fe-Zn stretching mode are seen at 680 cm^{-1} and 450 cm^{-1} .

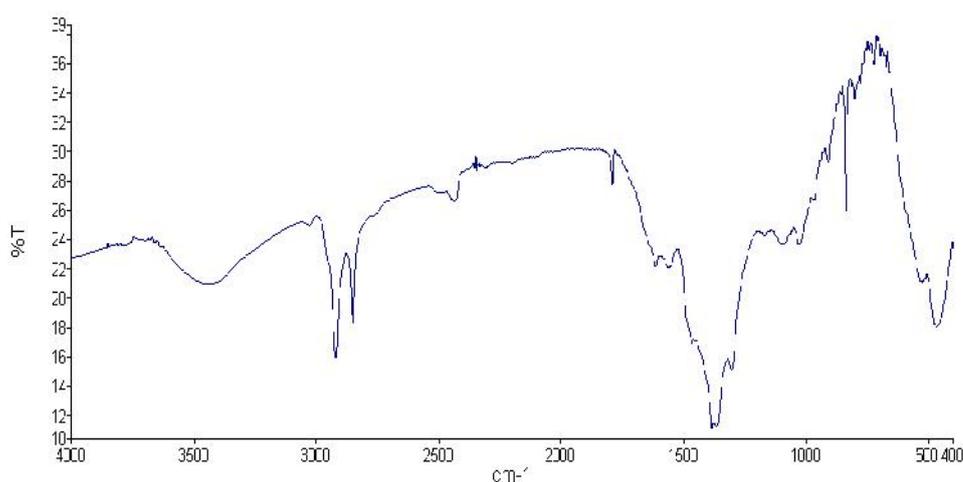


Fig.4. FTIR spectrum of as-prepared Fe/Zn sample

X-ray fluorescence analysis (XRF) of zinc ferrite nanoparticles prepared by wet synthesis is

shown in Figure 5 which confirms the existence of Zn and Fe. EDS was used to analyze the chemical composition of a material. XRF shows peaks of iron and ferrite elements with less Cl, F and Ni impurities.

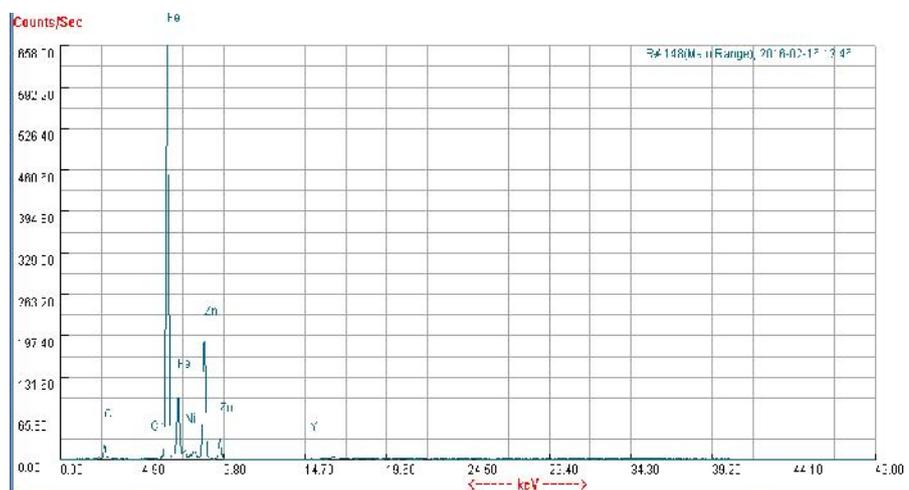


Fig.5. XRF analysis of Fe-Zn sample

4. CONCLUSION

Fe-doped ZnO nanoparticles have been successfully synthesized using iron nitrate and zinc nitrate precursors in the presence of CTAB surfactant and ethanol solvent. XRD spectrum showed rhombohedral $\text{-Fe}_2\text{O}_3$ and hexagonal wurtzite ZnO of the samples. SEM images indicated that the nanoparticles changed from sphere-like shaped to rod-like shaped with increasing temperature. TEM image exhibits that the as-synthesized Fe/Zn nanoparticles with an average diameter about 35 nm with good uniformity. FTIR data demonstrated the presence of Fe-Zn stretching mode. XRF analysis showed peaks of iron and ferrite elements with less Cl, F and Ni impurities.

5. ACKNOWLEDGMENTS

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