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Synthesis and Characterization of $ZnFe_2O_4$ Nanoparticles at Annealing Temperature

Physics

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ABSTRACT

Nanoparticles of $ZnFe_2O_4$ have synthesized at room temperature through Chemical Co-Precipitation method. These particles are synthesized without annealing.EDX and FTIR analyses were used for composition and structure investigation. FTIR frequency data for the respective sites are also analyzed. The higher frequency band and lower frequency band are assigned to the tetrahedral octahedral complex. X-ray diffraction pattern confirms the formation of cubic structure. Their corresponding reflection planes are also determined. The lattice constant calculated from XRD peaks is 8.39 A. Crystallite size calculated from XRD peak broadening confirms an average particles size of 12nm. Particles size measured using SEM show good agreement with the calculated value.

INTRODUCTION

Ferrites are one of the most important classes of magnetic ceramic materials and are used extensively in electronic devices for the communications industry [1]. Recently, nanometer sized magnetic ferrites have attracted considerable attention as improvements of their physical properties are achieved over that of the conventional feedstock [2-5]. Zinc ferrite ($ZnFe_2O_4$) is of particular interest among ferrite spinels because of the because of its use in providing a comprehensive scope for mixed ferrites meeting different specifications of magnetic materials[6-9], thereby, requiring a fine control over the size, crystalinity, stoichiometry and phase purity of the nanopowders.

EXPERIMENTAL TECHQUE

The Magnetite Nanoparticles of Zink ferrite ($ZnFe_2O_4$) synthesized using chemical co-precipitation method. A. R. Grade Zinc chloride ($ZnCl_2$), ferric chloride ($FeCl_3$, $6H_2O$), Oleic acid and sodium hydro-oxide (NaOH) were the basic ingredients used for the synthesis of this ferrite. Initially, the required amount of $ZnCl_2 6H_2O$ and $FeCl_3 6H_2O$ were taken in molar ratio of 1: 2 and dissolved in 40 ml of distil water. The required amount of NaOH is added in to the solution in order to adjust the pH value 7. The pH value of the solution plays an important role in controlling the precipitation and size of the precipitate particles. Solutions consisting of ZnFe2O4 were slowly heated up to 80-90 °C. Thereafter, a specified amount of oleic acid (5ml) was added to the solution. This act as the surfactant and coating material. The solution was stirred continuously and allowed to cool down up to room temperature. Few drops of Nitric acid (HNO_3) were added to precipitate the ferrite particles. The precipitate was taken out and washed, number of time with hot distilled water so as to remove traces of sodium chloride. Finally water was removed by washing it with acetone. This acetone wet-slurry was dispersed in 20ml of kerosene oil and heated at 70°C for 5 minutes. The resulting fluid was than centrifuged at 1200 rpm for about 10 minutes. After centrifuged the precipitate settled down at bottom of the pipette. The portion of the fluid was then taken out from pipette. Acquired substance was then dried at room temperature for 48 hours. The co-precipitated ferrite agglomerates were than ground for few minutes using a pestle to have very fine particles. These particles were subsequently heat treated in a box furnace at 500, 700 and 900°C, respectively. The final products obtained were dark brown in colour.

RESULT AND DISCUSSION

Figure 1 (a, b, c) show the XRD pattern of $ZnFe_2O_4$ samples annealed at different temperatures viz. 500, 700 and 900 °C. The reflection peaks (220), (311), (400), (422), (333) and (440) appear in all the samples. The appearance of these reflection peak proves that the present system have a cubic spinal structure [10]. The formation of a spinal $ZnFe_2O_4$ structure along with the other impurity phase was observed in the sample annealed at 500 °C. The sample annealed at 700 and 900 °C clearly shows the peaks related to $ZnFe_2O_4$ crystal structure only. The peaks related to other impurity phase get disappeared. It reveals that the increase in annealing temperature yields the sharpness of the peaks, i.e. of the grain size as well as crystallinity increases with the increase in annealing temperature [11]. The sizes of particle in the sample were calculated by measuring the FWHM for the most intense peak (311) by using Scherrer equation.

$$D = 0.9\lambda / \beta \cos\theta$$

The results are as shown in table. It is clear from these results that particle size increases with increasing annealing temperature. Similar trend in the variation of crystallite size with annealing temperature was observed by *Marykutty Thomas et.al* [12].

The lattice parameters "a" of the $ZnFe_2O_4$ annealed at different annealing temperatures have been calculated using of d values spacing. d-value spacing were calculated by using Bragg law. These values are shown in **table 1.** It can be seen that the lattice parameter decreases with the increase in particles size and annealing temperature.

Table 1 The change in particle size, Lattice parameter and X-ray density of Zn-ferrite Nanoparticles prepared at annealed temp. 500, 700, and d 900 °C respectively.

Annealing Temp.	Particle Size (nm)	Lattice Parameter (nm)	d-Values (Calculated)	X-ray density (gm/ cm3)
500 °C	10	0.8499	2.58304	5.2149
700 °C	24	0.8441	2.54519	5.3233
900 °C	45	0.8399	2.53240	5.4044

The X-ray densities were calculated using the equation

$$d_x = 8M/Na^3$$

Where digit 8 corresponds to the number of molecules in a unit cell of spin lattice, M is the molecular weight of the sample, N is the Avogadro's number and 'a' is the lattice constant.

The X-ray density (d_x) depends on the lattice constant and molecular weight of the sample. The values so obtained are given in table. Its (d_x) variation as a function of particle size and annealing temperature are shown in figure. 2 (a, b). It can seen from the figure that the X-ray densities increases with the increase annealing temperature, as it is inversely proportional to the lattice constant, which decreases with increasing annealing temperature. Increase in X-ray density is because of the decrease in lattice constant as also reported *D. M. Hemeda et al* [13].

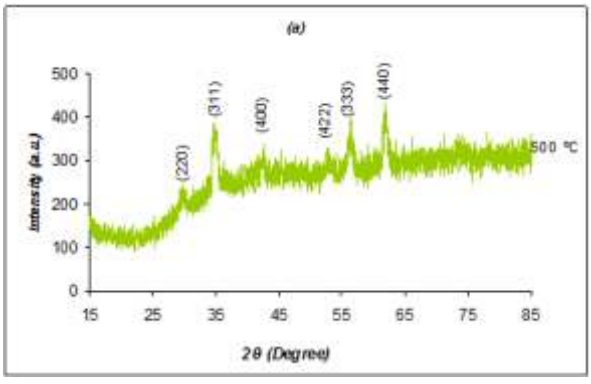

Figure 1(a) XRD pattern of $ZnFe_2O_4$ samples annealed at temperature 500 °C

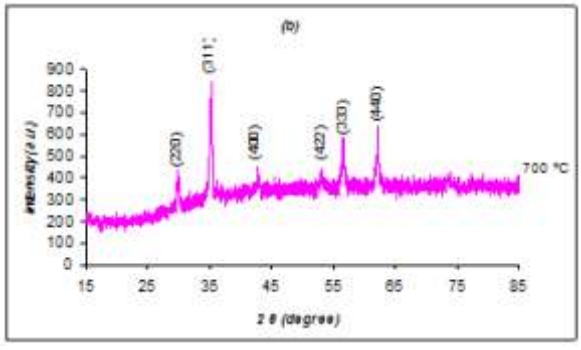

Figure 1(b) XRD pattern for $ZnFe_2O_4$ samples annealed at temperature 700 °C

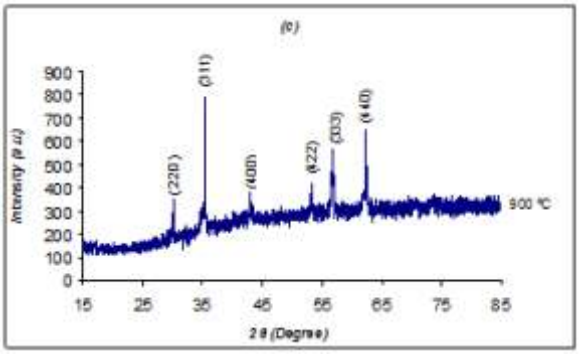

Figure 1 (c) XRD patterns for $ZnFe_2O_4$ samples annealed at temperature 900 °C

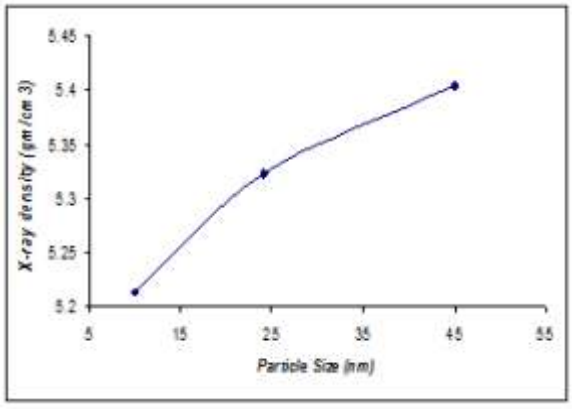

Figure 2 (a) Variation in X-ray Density with Particle size

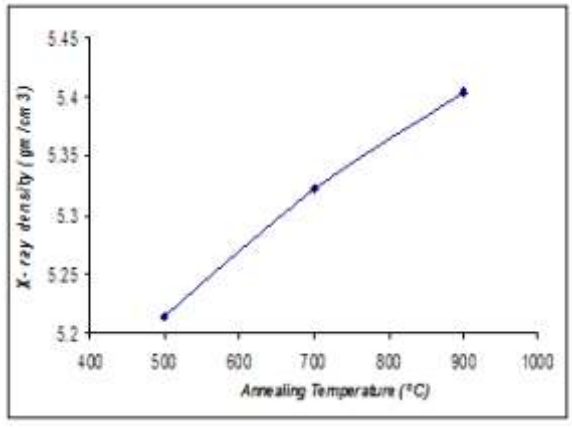

Figure 2 (b) Variation in X-ray Density with annealing temperatures

The SEM is used to characterize the morphology and size distribution of nanoparticles. The micrographs for Zn-ferrite system taken at annealing temperature are shown in figure 3 (a b and c). These micrographs clearly show the presence of two distinct phases; one is more grayish and aggregated. In all the samples the morphology of maximum grain structure, consists of almost spherical shape in agreement with the cubic structure as indicated by X-ray diffraction studies. The size of the nanoparticles calculated using SEM micrographs were found to be in the range of 12 to 56 nm. At annealing temperature 900 °C, these morphological features completely appears and a homogeneous system with agglomerates of submicronic particles is observed. The similar observation was reported by Dey et al [14]. Some particles are agglomerated because of magnetic dipole interactions between particles.

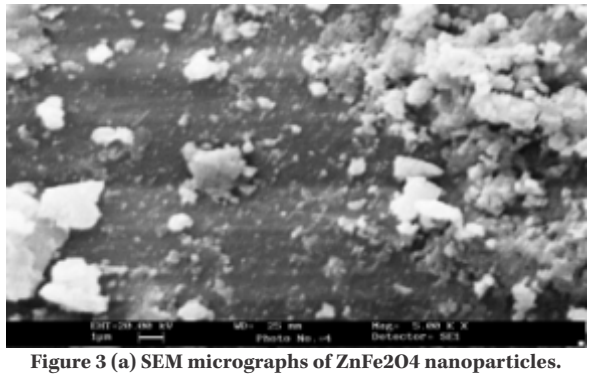

Figure 3 (a) SEM micrographs of ZnFe2O4 nanoparticles. Annealing temperature - 500 °C

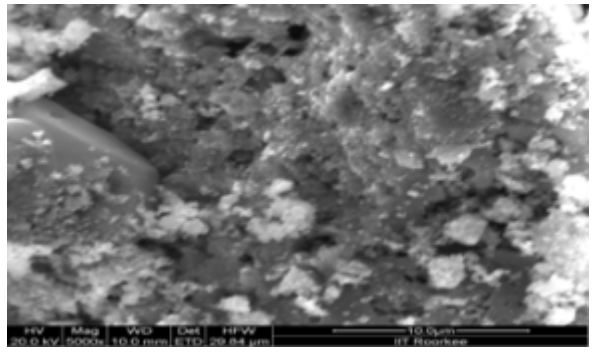

Figure 3 (b) SEM micrographs of $ZnFe_2O_4$ nanoparticles. Annealing temperature - 700 °C

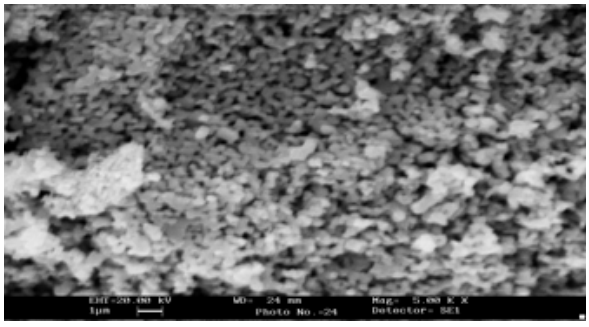

Figure 3 (c) SEM micrographs of $ZnFe_2O_4$ nanoparticles. Annealing temperature - 900 °C

The FTIR spectra in the wave length range from 400 to 600 cm^{-1} of $ZnFe_2O_4$ annealed at different temperature 500, 700 and 900 °C, respectively are shown in figure 4. No change FTIR spectra with annealing temperature. The position of vibration bands reveals distortion of the bonds due to the behavior of neighboring ions. The high frequency bands are very sensitive to the changes in interaction between oxygen and cation, in tetrahedral and octahedral position [15, 16]. The peaks observed between 400 to 552 cm^{-1} are principle peaks of ferrites. These peaks are observed to be in two pair. One pair of bands exist around 547- 552 cm^{-1} and next pair around 400-476 cm^{-1}. These pair of bands corresponds to the tetrahedral and octahedral site.

Chandrasekaran et al [17] has also attributed the occurrence of the first pair of band to intrinsic vibration in tetrahedral coordination compounds and the second to octahedral coordination. *Yongbo et al* [18] have reported that a strong band at 572 cm^{-1} is assigned to the Fe-O stretching vibration mode in the spectrum of $ZnFe_2O_4$.

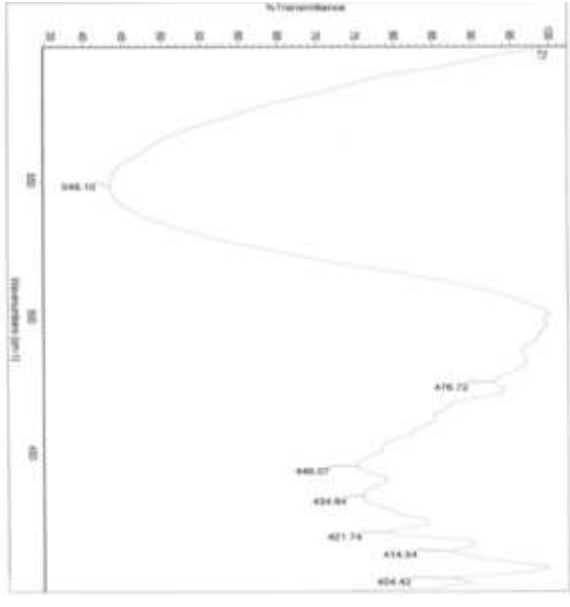

Figure 4.The FTIR spectra of $ZnFe_2O_4$

CONCLUSIONS

XRD pattern of the sample prepared by chemical co-precipitation method shows nanocrystalline nature of the sample. It has been observed that the material prepared is having the lattice constant of 0.8441nm. The average particle size resulting synthesized were observed to be 10-45 nm, which is well supported by the SEM micrographs. The FTIR spectrum has two absorption bands at approximately 400–600 cm^{-1}, which correspond to the octahedral and tetrahedral sites of positive ions of $NiFe_2O_4$, respectively.

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