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Original Research

Ferromagnetic Behavior in Zinc Ferrite Nanoparticles Synthesized using Coprecipitation Technique

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Abstract

Zinc ferrite have been produced and used by humans since long time, however understanding of $ZnFe_2O_4$ as a nano structured materials is very useful in order to be used for technological applications. $ZnFe_2O_4$ structural, magnetic and electrical properties are different when synthesized using different techniques. Therefore, it would be interesting to investigate the structural and magnetic properties of $ZnFe_2O_4$ when in nanosize. In the present work nanocrystalline $ZnFe_2O_4$ was synthesized using coprecipitation technique. The structural and magnetic properties of $ZnFe_2O_4$ nanopowders were investigated using X-ray diffraction (XRD), scanning electron microscope (SEM), dynamic light scattering (DLS), infrared spectroscopy (FTIR) and vibrating sample magnetometer (VSM). The XRD of $ZnFe_2O_4$ nanoparticles showed the single phase spinel structure. The average particle size of $ZnFe_2O_4$ calculated from XRD was observed to be 45 nm. DLS measurements showed the average particle size to be 42 nm. Further, the phase formation of $ZnFe_2O_4$ was confirmed from the IR measurements. The IR spectra showed the bands corresponding to $ZnFe_2O_4$. We observed the room temperature ferromagnetic behavior in synthesized $ZnFe_2O_4$ nanoparticles which may be due to the random distribution of Zn^{2+} and Fe^{3+} at the tetrahedral (A) and octahedral [B] sites. In our future work, we want to investigate the defect induced magnetic properties of $ZnFe_2O_4$ nanoparticles which is likely to contribute for ferromagnetic behavior in this material.

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INTRODUCTION

Nanoferrites are being extensively investigated due to their potential applications in magnetic refrigeration, drug delivery, high-density information storage, magnetic fluids etc. (Habibi *et al.,* 2013, Choi *et al.,* 2008). Spinel ferrites have been intensively investigated due to their versatile physical and chemical properties and due to their technological applications in magnetic sensors, biosensors, photocatalysts, nanoelectronics, biomedical (Ling *et al.,* 2013, Durka Prasad *et al.,* 2014). Zinc ferrite is one of the most important technological material having applications in radio engineering, radio technology, semiconductors etc. Bulk zinc ferrite have the normal spinel structure with Zn^{2+} ions occupying tetrahedral (A) site and Fe^{3+} ions being occupied in octahedral (B) site and is antiferromagnetic material having Neel temperature around 10 K (Pettit *et al.,* 1971). The magnetic properties of zinc ferrites significantly depend when a small amount of Fe^{3+} ions occupy A site (Choi *et al.,* 2008).

The magnetic properties of zinc ferrite nanoparticles was observed to have higher magnetization values compared to the bulk materials and also the synthesis technique might also play important role in achieving the ferromagnetic nature of zinc ferrite (Chen *et al.,* 1980, Singh *et al.,* 2010 and Thirupathi *et al.,* 2012). Several reports on $ZnFe_2O_4$ have showed that, $ZnFe_2O_4$ is paramagnetic at room temperature with Néel temperature $T_N \approx 10$ K. But, recently when $ZnFe_2O_4$ synthesized by different techniques and due to creation of oxygen vacancies ferromagnetic behavior was observed. Based on the above interesting properties of $ZnFe_2O_4$ nanoparticles, in this work we aimed to achieve room temperature ferromagnetic behavior of $ZnFe_2O_4$ nanoparticles synthesized using coprecipitation technique.

MATERIALS AND METHODS

$ZnFe_2O_4$ nanoparticles were synthesized using co-precipitation method (Raghavender *et al.,* 2011). The AR grade sodium hydroxide (NaOH), zinc (II) nitrate hydrate ($Zn(NO_3)_2.6H_2O$), ferric (III) nitrate nonahydrate ($Fe(NO_3)_3.9H_2O$) (98%) were used as starting materials. The metal nitrates were dissolved together in a minimum amount of deionized water to get a clear solution. In the metal nitrates precursor solution NaOH solution was added drop by drop under vigorous stirring. The precipitation occurred immediately to change the reaction solution to dark brown. The entire reaction was carried out at 75 °C for 2 h. The pH of the solution was varied by NaOH. The precipitate was filtered using a filter paper and

then dried at 220 °C for 3 h in oven. The structural characterization of zinc ferrite powders was carried out using Philips (France) X-ray diffraction (XRD) system with Ni filter using Cu –Kα radiation (wave length λ = 1.54 A^o). The $ZnFe_2O_4$ structure was confirmed by ABB Bomem MB 102 (Canada) infrared (FTIR) spectrometer. The samples were mixed with KBr and made in the form of pellets for IR transmission measurements. The particle size and morphology was verified using FEI Quanta (USA) FEG 200 High Resolution Scanning Electron Microscope (HR-SEM) and DLS measurements using LS spectrometer (Switzerland). The $ZnFe_2O_4$ powder was made in the form of pellet by applying 5 Tons of pressure and then SEM measurements was carried. For DLS measurements $ZnFe_2O_4$ powder was dissolved in ethanol and a high intensity laser beam was passed on to the dissolved sample to capture the particle size image. Room temperature magnetic properties were investigated using Lakeshore (USA) VSM 7410.

RESULTS AND DISCUSSION

Figure 1 shows the X-ray diffraction patterns of $ZnFe_2O_4$ nanoparticles. The XRD pattern shows the formation of single phase spinel structure. The lattice constant *a* from the XRD analysis was observed to be 8.409 Å, which is in agreement with the $ZnFe_2O_4$ nanoparticles synthesized by different techniques (Anjaneyulu *et al.,* 2014). The average particle size D was calculated using most intense peak (3 1 1) employing the Scherrer formula (Raghavender 2013).

$$D = \frac{0.9\lambda}{\beta Cos\theta}, \quad (1)$$

where β is the angular line width at half maximum intensity and y is the Bragg angle for the actual peak.

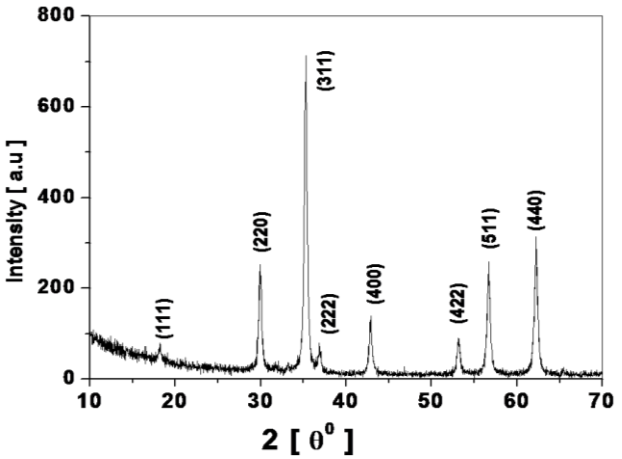
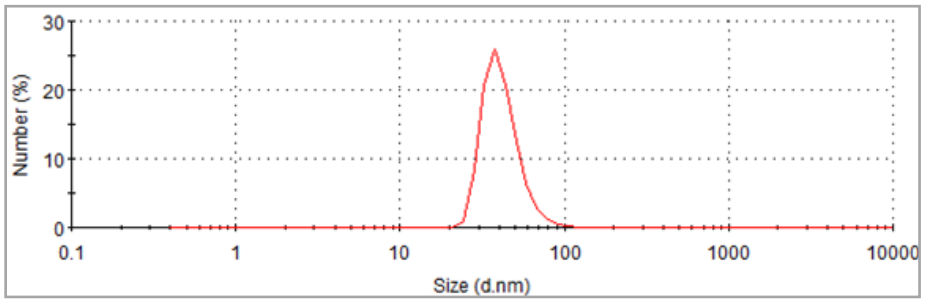


Figure 1: X-ray diffraction patterns of $ZnFe_2O_4$ nanoparticles

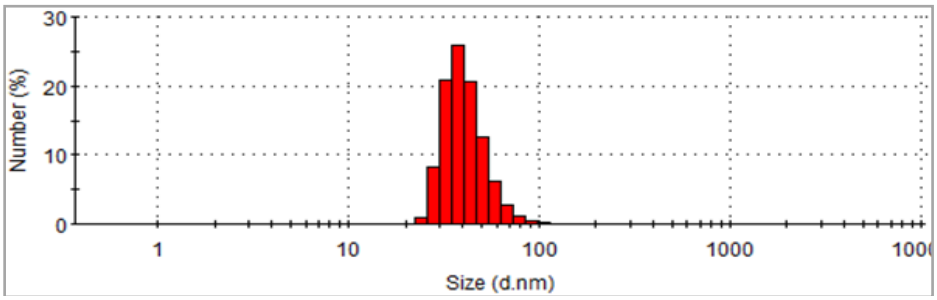
The average particle size calculated from XRD spectra was observed to be 45 nm (Table 1). The particle size was also measured from DLS system. The DLS measurement shows the particle size to be 42 (Figure 2). Therefore the average particle sizes calculated from XRD and DLS measurements are very well in agreement. Figure 3 shows the SEM image of synthesized $ZnFe_2O_4$ nanoparticles. Due to agglomeration in the synthesized nanoparticles we could not clearly observe the particle size distribution. Generally, when the particle size is in nano regime, due to nature and forces between them, agglomeration occurs which seems to be unavoidable when synthesized by few techniques. However, the XRD and DLS measurements support the existence of the nanoparticles in the synthesized $ZnFe_2O_4$ powder.

Table 1: Particle size *D* from XRD, DLS, lattice constant *a*, IR bands v_1, v_2, v_3, Coercivity H_c, remanence magnetization M_r, maximum magnetization M for $ZnFe_2O_4$ nanoparticles

D_{XRD} (nm)	D_{DLS} (nm)	a (Å)	v_1	v_2	v_3	H_c (Oe)	M_r (emu/g)	M (emu/g)
45	42	8.409	544	388	329	57	1.2	10.6



Size distribution by Number



Statistics graph

Figure 2: DLS image by distribution and statistics graph of $ZnFe_2O_4$ nanoparticles.

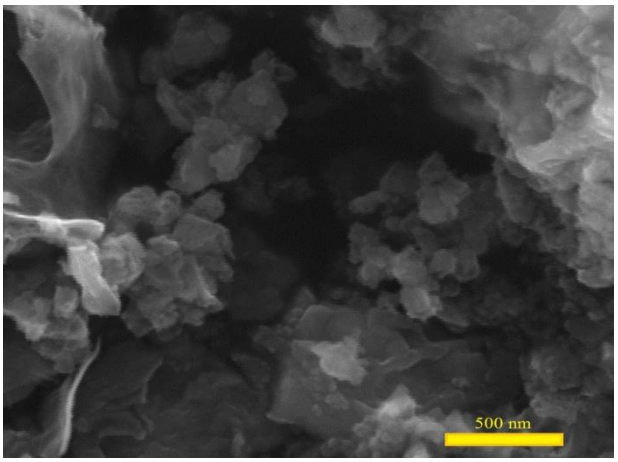


Figure 3: SEM image of $ZnFe_2O_4$ nanoparticles

The IR spectra of $ZnFe_2O_4$ nanoparticles are shown in Figure 4. IR spectra shows the bands corresponding to $ZnFe_2O_4$. The IR spectra shows the three absorption bands. The first band, v_1, is located in the 800 – 500 cm^{-1} range, the second band, v_2, in the 500 – 350 cm^{-1} range, while the third band, v_3, occurs between 350 and 280 cm^{-1} range. According to Waldron (Waldron 1955), v_1 band corresponds to the stretching vibrations of Zn^{2+}- O band in tetrahedral sites, while v_2 is assigned to Fe^{3+}- O stretching of octahedral sites. On the other hand, v_3 is result of oscillations of metal atoms in the isotropic force fields of their tetrahedral or octahedral environment. The IR results observed for $ZnFe_2O_4$ are in support with the reported data in the literature (Raghavender *et al.,* 2011; Raghavender 2013; Anjaneyulu *et al.,* 2014).

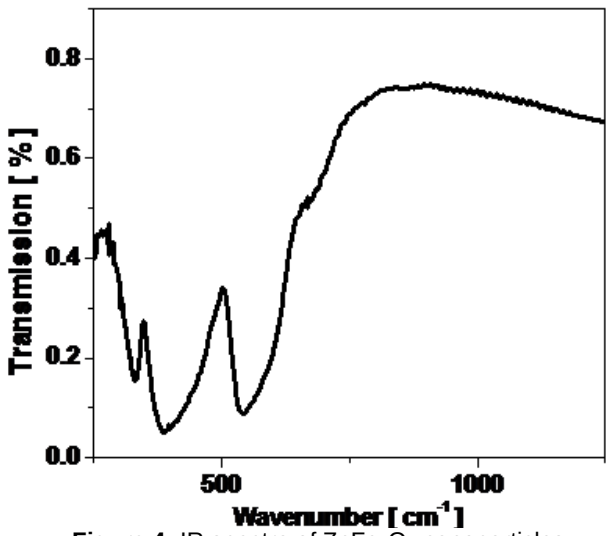


Figure 4: IR spectra of $ZnFe_2O_4$ nanoparticles

Figure 5 shows the room temperature hysteresis curve of $ZnFe_2O_4$ nanoparticles. The derived parameters are listed in Table 1. The hysteresis curve shows ferromagnetic nature of $ZnFe_2O_4$ nanoparticles. The magnetization curve does not seem to be saturated even with the maximum applied field of 10 kOe. From the hysteresis curve, the coercivity H_c was observed to be 57 Oe, remanence magnetization M_r was observed to be 1.2 emu/g and the maximum magnetization M to be 10.6 emu/g. $ZnFe_2O_4$ nanoparticles / bulk materials measured at low temperatures or synthesized at low temperatures shows ferromagnetic behavior. Generally, $ZnFe_2O_4$ shows the paramagnetic behavior at room temperature as reported in literature. The probable reason for the observed ferromagnetic behavior in our case may be due to several facts such as structural rearrangement, which may induce changes due to the superexchange interaction in the tetrahedral (A) and octahedral [B] sites (Deraz *et al.,* 2012). In the case of $ZnFe_2O_4$ thin films, the random distribution of Zn^{2+} and Fe^{3+} at (A) and [B] sites or the defects causes the ferromagnetic behavior (Raghavender, 2011). In $ZnFe_2O_4$ nanoparticles or bulk materials, the superexchange interaction between (A) and [B] sites does not seem to favor at room temperature, and when measured below room temperature $ZnFe_2O_4$ shows ferromagnetic behavior (Ayyapan *et al.,* 2010; Goya *et al.,* 1999). The ferromagnetic behavior in our case may also be due to nonequilibrium distribution of Fe^{3+} ions in (A) and [B] sites (Bohra *et al.,* 2006). In $ZnFe_2O_4$ the inversion parameter leads to the ferromagnetic behavior (Hofmann *et al.,* 2004). In fact, the magnetic properties of $ZnFe_2O_4$ are influenced mainly due to the synthesis routes, annealing temperature, cation distribution and the grain size (Deraz et al., 2012). The synthesis route makes a lot of difference in the properties of materials while no difference in some other properties (Upadhyay *et al.,* 2007). The ferromagnetic behavior may also arise due to the spin or structural disorder when $ZnFe_2O_4$ is synthesized by different routes. Therefore, the observed magnetic properties in the present work may be due to the above mentioned facts. In our future work, we will try to investigate in detail the magnetic properties of $ZnFe_2O_4$ nanoparticles prepared under several experimental conditions to clearly understand the origin of ferromagnetic behavior.

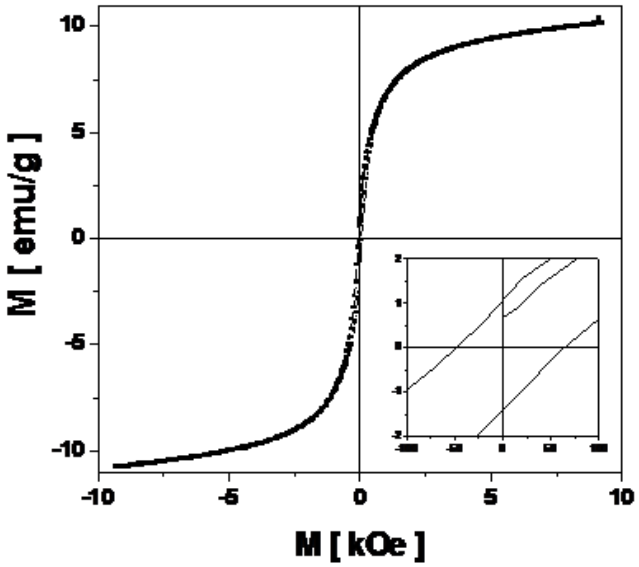


Figure 5: Room temperature M-H curve for $ZnFe_2O_4$ nanoparticles. The inset of the figure shows the expanded lower field curve.

CONCLUSION

$ZnFe_2O_4$ nanoparticles were synthesized using coprecipitation technique. The average particle size of synthesized $ZnFe_2O_4$ were observed to be 45 nm. DLS measurements showed the average particle size around 42 nm. The particle size calculated form XRD and DLS measurements are very well in agreement. The IR spectra showed the bands corresponding to $ZnFe_2O_4$.

Room temperature ferromagnetic behavior was observed for synthesized $ZnFe_2O_4$ nanoparticles. The ferromagnetic behavior in the synthesized $ZnFe_2O_4$ nanoparticles may be due to the random distribution of Zn^{2+} and Fe^{3+} at the tetrahedral (A) and octahedral [B] sites, inversion parameter, synthesis techniques etc.

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