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Morphological and Magnetic Studies of Zinc Spinel Ferrite Nanoparticles

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Abstract—*Zinc spinel ferrite ($ZnFe_2O_4$) nanoparticles having super paramagnetic nature were synthesized at lower temperature by low combustion synthesis method. The resulting 'as synthesized' powder was heat treated (HT) at 500°C for 4 h in nitrogen atmosphere. As-synthesized particles had sizes ~ 15 nm with spherical shape. Further, these spherically shaped nanoparticles tended to change their morphology to hexagonal plate shape when HT at 500°C. The hysteresis loops showed an increase in saturation magnetization from 1.131 to 33.20 emu/g with increasing HT temperatures. According to literature, the hexagonal shaped morphology and high saturation magnetization make these ferrites a potential material for RADAR absorption applications.*

Keywords*: RADAR, Saturation Magnetization, Hexagonal.*

1. INTRODUCTION

The zinc spinel ferrite has been the material of interest ever since the discovery of ferrites due to its intriguing electrical and magnetic properties such as low coercivity, high permeability, good chemical stability, high electrical resistivity and low eddy current losses. The zinc spinel ferrite has applications in data storage system, medical diagnostic instruments, microwave absorbers, and magnetic bulk cores [1-5]. The advantage of spinel ferrites over other magnetic materials is their high electrical resistance due to which they have low eddy current losses at high frequencies. This makes the soft ferrites an ideal choice for many electronic applications operating at high frequencies [3, 4]. Zinc ferrite has the normal spinel structure having generic formula MFe_2O_4, here M is a divalent cation such as Zn, Ni etc. and Fe is the trivalent cation. The magnetic properties of the spinel ferrites emerge due to the arrangement of the divalent and the trivalent cations in the unit cell and the interaction between them. The unit cell of the spinel ferrite consist of 32 oxygen atoms having cubic close packing , 8 tetrahedral sites and 16 octahedral occupied sites. These tetrahedral and octahedral sites are termed as A site and B site respectively. The M^{2+} ions occupy A sites where as Fe^{3+} ions occupies the B sites in the zinc spinel structure [5-9]. The spinel ferrites having particle size in nanometer range show impressive properties which are different from the bulk material. This is due the change in their structure and arrangement between the ions [10-11]. In the present study zinc ferrite nanoparticles are synthesized at lower temperature by low combustion synthesis method. Further, morphological and magnetic studies of the zinc ferrite nanoparticles has been carried out.

2. EXPERIMENTAL PROCEDURE

The zinc spinel ferrite nanoparticles were synthesized by the low combustion synthesis method. The analytical grade ferric nitrate ($Fe(NO_3)_3 \cdot 9H_2O$), zinc nitrate ($Zn(NO_3)_2 \cdot 6H_2O$), and thiourea (($CS(NH_2)_2$) were used as a raw materials. The stoichiometric amount of the nitrates and the thiourea were dissolved in the deionised water to form the aqueous solution. The nitrates solution and the thiourea solution were mixed in 1:2 molar ratios under constant stirring. The liquid ammonia was used to maintain the pH of the formed solution at 9.5. The mixed solution was then heated at 80°C for 4-5 hours until the self ignition occurs. The obtained powder was then crushed thoroughly in an agate pestle mortar in order to produce the fine nanoparticles of zinc spinel ferrite. The 'as synthesized' zinc spinel ferrite nanoparticles were then heat treated at 500°C in the electric furnace for 4 hours in nitrogen atmosphere.

3. CHARACTERIZATION

The phase identification of 'as synthesized' and heat treated $ZnFe_2O_4$ nanoparticles was done by X-Ray Diffraction (XRD) using Bruker AXSD8 differactometer with Cu-Kα radiation. Morphological study was carried out by transmission electron microscope, TEM (Philips, EM 400; TECHNAI 20G2-STWIN). Magnetic measurements were taken out at room temperature in the applied field range of -10000 to +10000 gauss using vibrating sample magnetometer, VSM (155, PAR).

4. RESULTS AND DISCUSSION

4.1. XRD Study

Fig. 1 shows the XRD diffraction patterns of the 'as synthesized' and heat treated $ZnFe_2O_4$ nanoparticles. The diffraction peaks show the reflection planes (220), (311), (222), (400), (422), (511) and (440) which are consistent with the JCPDS Card No. 22-1012. From the XRD graph it is observed that the diffraction peaks of the heat treated sample is sharper than the as synthesized sample. The particle size of the zinc ferrite nanoparticles has been calculated from the XRD plane (311) by the Scherrer's formula: $d = 0.9\ \lambda/\beta\ \cos\theta$, where d is the average particle size, β is the full width half maximum (FWHM), λ is the X-ray wavelength of the target material used and θ is the Bragg angle.

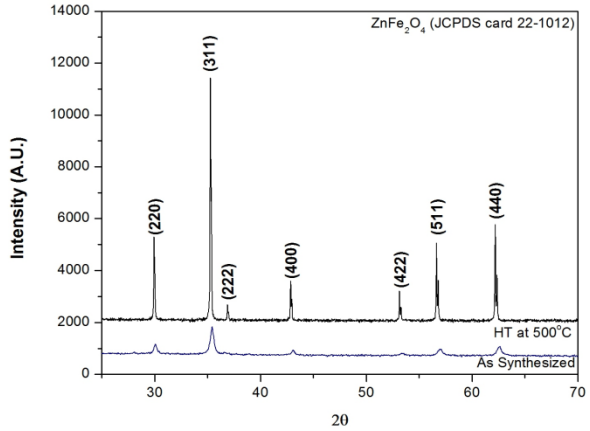
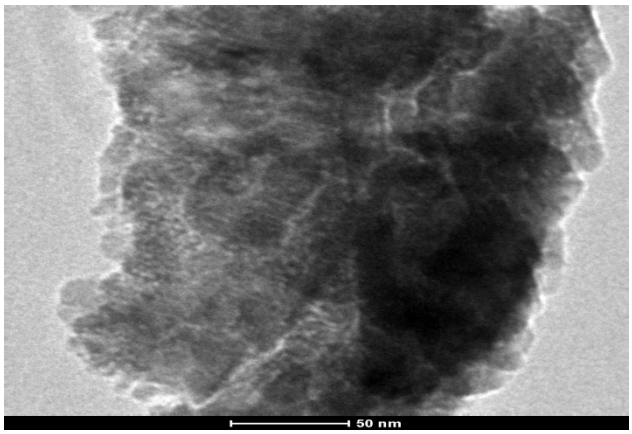


Fig. 1: Indexed XRD Pattern of 'As Synthesized' and Heat Treated Zinc Ferrite Nanoparticles.

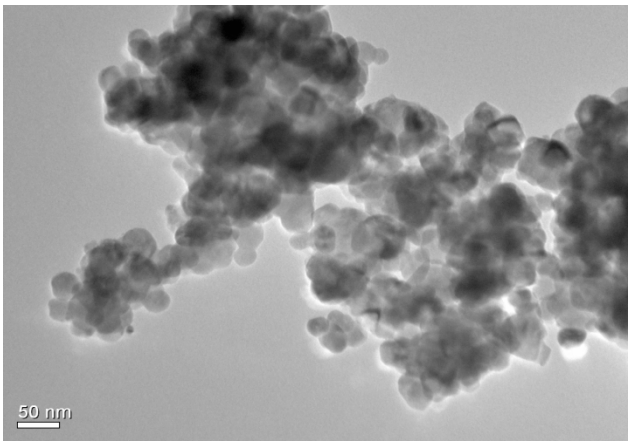
The crystallite size of zinc ferrite nanoparticles was observed to increase from 15 nm to 50 nm with increase in HT temperature.

4.2. Morphological Study

The Fig. 2 shows the TEM micrograph of the 'as synthesized' and heat treated Zinc ferrite nanoparticles at temperature 500°C.



(a)



(b)

Fig. 2: TEM Micrographs of $ZnFe_2O_4$ Nanoparticles (a) 'As Synthesized' (b) Heat Treated at 500°C in Nitrogen Atmosphere

The 'as synthesized' nanoparticles with the spherical shaped morphology are observed to have particle size in the range 15-20nm (Fig. 2a). However heat treated zinc ferrite nanoparticles possesses hexagonal plate shape with particle size ~ 50nm (Fig. 2b). This process of crystal growth and morphological evolution can be explained in terms of Ostwald ripening. As the HT temperature is increased, these nanoparticles slowly disappear except for the few that grow larger, at the expense of smaller ones. Thus particles of small size act as nutrients for the bigger ones. [11-14].

4.3. Magnetic Study

Fig. 3 show the hysteresis curve of the 'as synthesized' and HT zinc ferrite nanoparticles.

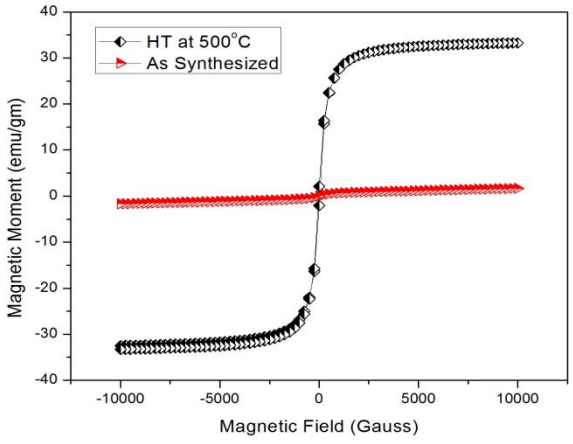


Fig. 3: The Effect of HT Temperature on Hysteresis Loop of $ZnFe_2O_4$ Nanoparticles in the 'As Synthesized' and Heat Treated Conditions.

The magnetic measurements of as synthesized $ZnFe_2O_4$ nanoparticles have almost negligible coercivity and remanance values (Fig. 3) showing the superparamagnetic behavior of the

material. But when the 'as-synthesized' zinc ferrite nanoparticles are heat treated at 500^{o}C, the particles appear to transform from superparamagnetic to ferromagnetic nature. Saturation magnetization is found to be dependent on HT temperature. It increases from 1.13 to 33.20 emu/g with increase in HT temperature.

5. CONCLUSIONS

The zinc ferrite nanoparticles (15-20nm) are successfully synthesized at lower temperature by low combustion synthesis method. The 'as synthesized' zinc ferrite nanoparticles are observed to have spherical shaped morphology which further changed to the hexagonal plate shape when heat treated at 500^{o}C in nitrogen atmosphere. The 'as synthesized' zinc ferrite nanoparticles are observed to be superparamagnetic which further transformed to ferromagnetic with increase in the HT temperature. The saturation magnetization of zinc spinel ferrite nanoparticles was observed to increased from 1.13 to 33.20 emu/g with increase in HT temperature

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