A simple and effective method of the synthesis of nanosized $Ni_{0.5}Zn_{0.5}Fe_2O_4$ particles

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Abstract:

Nanosized spinel Nickel zinc ferrite $Ni_{0.5}Zn_{0.5}Fe_2O_4$ has been synthesized by precipitation method. X-ray diffraction (XRD), transmission electron microscopy (TEM) and Vibrating sample magnetometer (VSM) are used to characterize the structural, morphological and magnetic properties. XRD studies show that Nickel zinc ferrite is having cubic spinel structure. The absence of hysteresis, negligible remanence and coercivity at 300K indicate the superparamagnetic character and single domain in the nanocrystalline $Ni_{0.5}Zn_{0.5}Fe_2O_4$ and hence can be used in magnetic devices. The particle size of the synthesized $Ni_{0.5}Zn_{0.5}Fe_2O_4$ was determined by TEM. TEM images show very fine nanoparticles of synthesized ferrite. Size of particles of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ varied from 20nm to 40nm with average particle size of 30.nm which is in good agreement of the theoretically predicted size of nanomaterials. Ms value was observed to be 30 emu/g at 300K This method is convenient, easy and effective in comparison to the known methods of the synthesis of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanomaterials like ultrasonic radiation, sol-gel approach, Fe implantation, thermal decomposition of metal-surfactant complexes, colloid mill, mechanical milling.

Key words: Nanaomaterial, $Ni_{0.5}Zn_{0.5}Fe_2O_4$, TEM, Nickel zinc ferrite, XRD analysis

1. Introduction:

Spinel ferrites are magnetic materials and have wide applications in magnetic devices and switching devices [1-4]. Nickel-zinc ferrite is a soft magnetic material having low magnetic coercivity, high resistivity and little eddy current loss in high frequency operations (10–500 MHz) values [5-6]. The high electrical resistivity and excellent magnetic properties make this ferrite an automatic choice as a core material for power transformers in electronic and telecommunication applications in megahertz frequency regions [7-8]. The properties of ferrite materials are strongly influenced by the materials composition and microstructure, which are sensitive to the preparation methodology used in their synthesis [9]. In addition, the sintering conditions employed and the impurity levels present in or added to these materials also change their properties [10]. The selection of an appropriate process is, therefore, the key to obtain good quality ferrites. These ferrites are usually prepared by the conventional ceramic method, in which the stoichiometric composition and final microstructure are extremely difficult to control. This method requires prolonged heating at high temperatures during preparation, which may cause some of the constituents to evaporate, thereby modifying the desired stoichiometry. Moreover, in Ni–Zn ferrites, the volatilization of zinc at high temperatures results in the formation of Fe^{2+} ions, which increase electron hopping and reduce resistivity [11]. The wet chemical methods of powder preparation appear to offer a better alternative, since they overcome the drawbacks of the conventional ceramic method. The wet chemical synthesis of highly reactive powders has proved to be one of the most effective routes to decrease the sintering temperature of ferrites. A variety of chemical synthesization methods such as co-precipitation, hydrothermal synthesis, the citrateprecursor method, the glass-ceramic route and the sol–gel process have been developed [12–18]. Very few reports have been published regarding the synthesis of Nickelzincferrite particles. In the present manuscript, synthesis of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanoparticles has been reported by simple aqueous precipitation method using chlorides of Nickel, zinc and iron and taking liquor ammonia as precipitating agent. This method involves a simple, cheap and one step process for synthesis of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanaoparticles as compared to other methods of synthesis The obtained particles of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ have size from 20-40 nm. The synthesized nanoparticles were characterized by XRD, Magnetic susceptibility and TEM.

2. Methods and materials

2.1 Chemicals:

All chemicals used in the experiment are analytic reagent grade. $FeCl_3$, $ZnCl_2$, $NiCl_2.6H_2O$ and liquor ammonia were purchased from Merck, India. Deionized water was used throughout the experiment.

2.2 Synthesis of $Ni_{0.5}Zn_{0.5}Fe_2O_4$:

Powder with a nominal composition of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ was prepared taking chlorides of (Ni, Zn and Fe) and aqueous ammonia (precipitating agent). $NiCl_2.6H_2O$, $FeCl_3$ and $ZnCl_2$ were taken in stoichiometry ratio and were dissolved in 500 mL of water. Aqueous ammonia (2M) was added drop wise with constant stirring until the pH of the solution reached 10. The precipitates thus obtained were filtered by Buckner funnel and were washed several times with distilled water. The precipitates were dried in oven at 70°C for 24 hrs and were calcined at 400°C in a muffle furnace for 5 hrs. Obtained material was ground and sieved through 100 mesh size sieve.

2.3 Characterization techniques:

The microstructure of the particles was characterized by X-ray diffraction (XRD), Philips PW 11/90 diffractometer using nickel filtered CuKα (l = 1.5405 Å) radiations. The average diameter (D) of the ferrite nanocrystals has been calculated from the broadening of the XRD peak intensity after $K\alpha_2$ corrections using the Debye-scherrer equation. Transmission electron microscopy (TEM) measurements of the sample were taken on Hitachi H7500 with a 70 kV accelerating voltage. The dispersions of nanoparticles in water were placed on carbon-coated 400 mesh copper grids, allowed to dry at room temperature before taking measurement. The obtained micrographs were then examined for particle size and shape. The magnetic properties of the solid was measured at 300K using a Vibrating sample Magnetometer Model 155.

3. Results and discussions:

3.1. X-ray studies:

X-ray diffraction of synthesized $Ni_{0.5}Zn_{0.5}Fe_2O_4$ is shown in Figure (1). X-ray diffraction pattern of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ pure indicated that Nickel zinc ferrite in the form of $Ni_{0.5}Zn_{0.5}Fe_2O_4$. In X-ray diffraction, some prominent peaks were considered and corresponding d-values were compared with the standard i.e. JCPDS file No. 08-0234 (Table-1).

X-ray diffraction shows that metal oxide is pure $Ni_{0.5}Zn_{0.5}Fe_2O_4$ having cubic spinel structure. The peaks indexed to (220), (311), (400), (422), (511) and (440) planes of a cubic unit cell, corresponds to single phase spinal crystal structure. Sharpness of the peaks shows good crystal growth of the ferrite particles. Average particle size (t) of the particles have been calculated using from high intensity peak (311) using the Debye-Scherrer equation

$$t = K\lambda / B \cos\theta$$

Where t is the average crystallite size of the phase under investigation, K is the Scherrer constant (0.89), λ is the wave length of X – ray beam used, B is the full-with half maximum(FWHM) of diffraction (in radians) and θ is the Bragg's angle.

The average crystallite size calculated is 30.nm which is in close agreement with the TEM results. Lattice constant of ferrite nanocrystals are computed using the d value (interplaner spacing) and their respective lattice (h k l) parameters. Lattice constant for ferrite nanocrystals has been found to be $8.37205A^0$. The actual (X-ray) density of $NiFe_2O_4$ nanoparticles is calculated using the formula Px = 8*M/Na*3 (26) and is given in Table 2. Where *M* is the molecular weight (kg) of the sample, *N* the Avogadro's number (per mol) and *a* the lattice constant (Å). Value of Px was calculated as 5.177 (g/cc).

3.2 TEM studies

TEM studies were carried to find out exact particle size of synthesized $Ni_{0.5}Zn_{0.5}Fe_2O_4$. Figure 2 shows the TEM image of the synthesized $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanoparticles. The single crystal nature of the $Ni_{0.5}Zn_{0.5}Fe_2O_4$ is revealed by TEM analysis. A very fine spherical NiFe2O4 nanoparticle in the range of 20-40 nm with average size of 30.are obtained. The average crystallite size $D_{XRD,}$ D_{TEM}, and the lattice constant (a) of the sample obtained have been summarized in Table 2

3.3 Magnetic measurements:

The magnetic measurement of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ was carried out at room temperature and it has been observed that $Ni_{0.5}Zn_{0.5}Fe_2O_4$ shows super paramagnetic behavior at room temperature (300 K) with saturation magnetization (Ms) value 28 (Fig.3). Previously reported values of *M*s for Nickel zinc ferrite nanoparticles prepared by various methods have been reported in Table2. The value of *M*s ranging from 12-88 $emug^{-1}$ shows that *M*s strongly depends on the synthesis method used [29-43]. This Ms value at room temperature is good and comparable with methods of synthesis as thermal decomposition method (Ms value 42 at 300K and Ms value 65.4 $emug^{-1}$ at 10K), ball milling (Ms value 20.7 in at 4.2K) and other co-precipitation routes which shows a maximum Ms 46.9 at 4.2 K [Table2].

4. Conclusion:

$Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanoparticles with cubic spinel structure are synthesized successfully by aqueous precipitation method. From TEM study it is found that particles are having size of 20-40 nm with average size . Magnetic measurements show that $Ni_{0.5}Zn_{0.5}Fe_2O_4$ is super paramagnetic in nature having saturation magnetization (Ms) value 29 emu/g. This method is advantageous over existing methods of synthesis of nanoparticles because other methods require specialized instrumentation, highly skilled labour, expensive materials and methods. Therefore, the proposed precipitation method is very promising, easy and cheap and may have extensive applications.

Table-1 X-RAY DIFFRACTION DATA FOR $Ni_{0.5}Zn_{0.5}Fe_2O_4$

S. No.	d=λ / 2sinθ (Observed)	d=λ / 2sinθ (Reported)	$I/I_0X100\%$ (Observed)	$I/I_0X100\%$ (Reported)
1.	2.96218	2.966	30	40
2.	2.52427	2.533	100	100
3.	2.41912	-	33	30
4.	2.09596	2.110	43	50
5.	1.70595	-	16	20
6.	1.61475	1.485	43	50

Reported value of saturation magnetization in literature

Nickel zinc ferrite	Ms (emu/g)	Temp	Size	Synthesis method	Ref Number
$Ni_{0.5}Zn_{0.5}Fe_2O_4$	28	500	20-40	Precipitation	This work
$Ni_{0.6}Zn_{0.4}Fe_2O_4$	30.83	180	-	Citrate gel method	22
$Ni_{0.5}Zn_{0.5}Fe_2O_4$	27	400	10	Reverse miscille	23
$Ni_{0.5}Zn_{0.5}Fe_2O_4$	54.70	1100	29	Combustion method	24
$Ni_{0.8}Zn_{0.2}Fe_2O_4$	44	RT	15	Precipitation	25
$Ni_{0.8}Zn_{0.2}Fe_2O_4$	67	1200	16-44	Precipitation	25
$Ni_{0.20}Zn_{0.44}Fe_{2.36}O_4$	25.9	RT	07	Reverse micelle process	26
$Ni_{0.20}Zn_{0.44}Fe_{2.36}O_4$	42	-	30	Thermal Plasma	27
$Ni_{0.6}Zn_{0.4}Fe_2O_4$	40	80	16	Precipitation	28

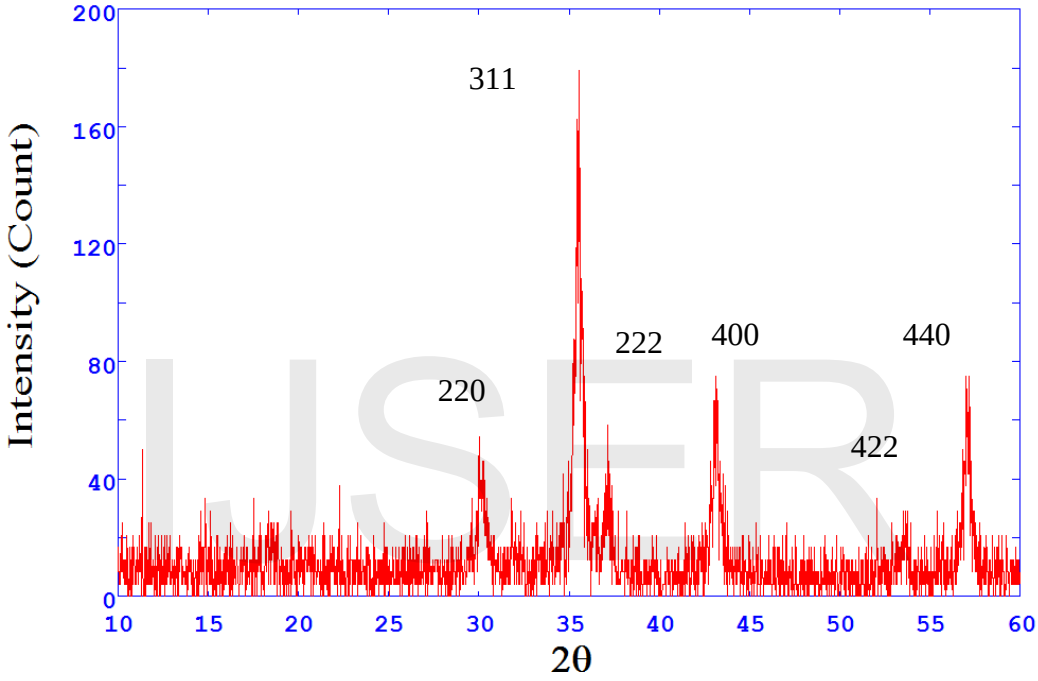


Figure 1 XRD spectra of Nickel Zinc Ferrite

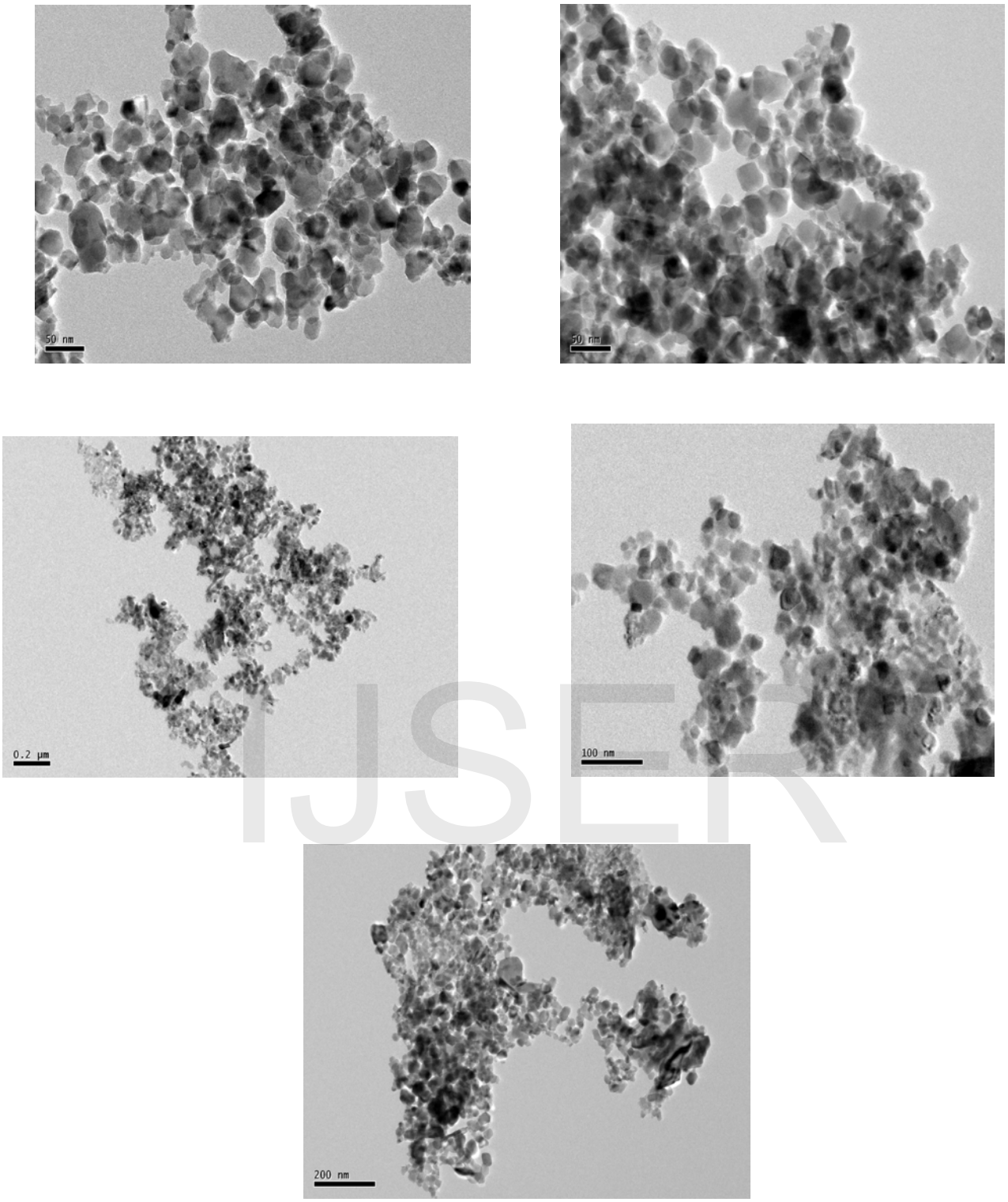
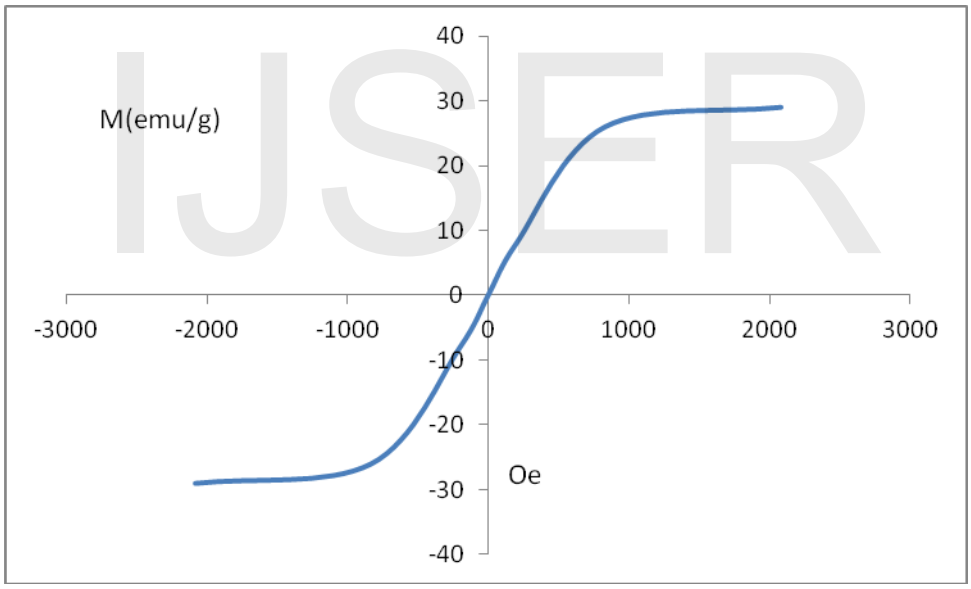


Figure 2: TEM images of Zinc ferrite particles



Magnetic measurements of synthesized nickel zinc ferrite

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