

# Synthesis and Characterization of Ni-Zn Ferrite Nanoparticles ( $\text{Ni}_{0.25}\text{Zn}_{0.75}\text{Fe}_2\text{O}_4$ ) by Thermal Treatment Method

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## ABSTRACT

Cubic structured nickel-zinc ferrite nanoparticles ( $\text{Ni}_{0.25}\text{Zn}_{0.75}\text{Fe}_2\text{O}_4$ ) have been synthesized by thermal treatment method. In this procedure, an aqueous solution containing metal nitrates as precursors, polyvinyl pyrrolidone as a capping agent, and deionized water as a solvent were thoroughly stirred, dried at 353 K for 24 h, and crushed into powder before calcination to remove organic matters and crystallize the particles. The structure and particle size were characterized by X-ray powder diffraction and transmission electron microscopy. The average particle size increased from 7 to 25 nm with increase of calcination temperature from 723 to 873 K respectively. The magnetic properties were determined by vibrating sample magnetometer and electron paramagnetic resonance electron paramagnetic resonance at room temperature. By increasing the calcinations temperatures from 723 to 873 K it showed an increase of the magnetization saturation from 11 to 26 emu/g and the g-factor from 2.0670 to 2.1220. The Fourier transform infrared spectroscopy was used to confirm the presence of metal oxide bands at all temperatures and the removal of organic matters at 873 K.

**Keywords:** Thermal Treatment; Nickel Zinc Ferrite; Nanoparticles; Magnetic Property

## 1. Introduction

The last two decades have seen a remarkable progress in the synthesis of spinel ferrites nanocrystals, aiming at a better material with excellent chemical stability, low magnetic coercivity, moderate saturation magnetization, high permeability, high electrical resistivity and low eddy current. In particular, the nickel-zinc (Ni-Zn) ferrite nanocrystals have been extensively studied for their super-paramagnetic properties, which are suitable for high-frequency applications such as rod antennas and cores of inductors and transformers [1,2]. The magnetic properties of ferrites of spinel structural formula  $\text{AB}_2\text{O}_4$  are mainly controlled by the divalent cations, which occupy the tetrahedral A sites and the trivalent cation, which has high degree affinity for octahedral B sites [3,4]. Zn ferrite bulk material has a normal spinel structure, where all divalent cations are located on the tetrahedral sites and

trivalent cations all located on the octahedral sites. Ni ferrites bulk materials on the other hand have an inverse spinel structure, where half of trivalent cations occupy the tetrahedral sites while the other half remain on octahedral, while divalent cations all migrate to octahedral positions. It has been shown that for the Ni-Zn ferrite nanoparticles, octahedral sites prefer for Ni and tetrahedral prefers for Zn [5]. Substituting Zn for Ni cations leads to the formula  $(\text{Zn}_x^{2+} \text{Fe}_{1-x}^{3+})(\text{Ni}_{1-x} \text{Fe}_{1+x}^{3+})\text{O}_4$  ( $0 \leq x \leq 1$ ), in which the first and second brackets indicate occupancy of the A and B sub-lattices respectively. Beside the distribution of divalent and trivalent cations in the spinel structure, the properties of Ni-Zn ferrite nanoparticles are highly sensitive to the quantum confinement effect of particle size, which in turn depends on the method of preparation of the nanoparticles [6].

A variety of methods have been proposed for the synthesis of Ni-Zn ferrite nanoparticles with controllable size, shape, and chemical stability such as sol-gel methods [7],

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thermal combustion method [8], citrate precursor route [9], co-precipitation method [6,10], thermal plasma synthesis [11], reverse micelle [12,13], hydrothermal [14], micro-emulsion [15] and sonochemical reaction [16]. Some disadvantages of these methods include complicated procedure, high reaction temperature, long reaction time, and use of reduction agents, which are potential upsetting the environment. In the present study, Ni-Zn ferrite nanoparticles ( $\text{Ni}_{0.25}\text{Zn}_{0.75}\text{Fe}_2\text{O}_4$ ) were synthesized from an aqueous solution containing only metal nitrates as precursors, polyvinyl pyrrolidone (PVP) as a capping agent and deionized water as solvent by a simple thermal treatment at moderate calcinations temperatures. No other chemicals were added, thus this method offers the advantages of simplicity, low reaction temperatures, a low cost, and an environmentally friendly operation since it produces no by-product effluents [17-20].

## 2. Experimental Procedure

### 2.1. Preparation

Iron (III) nitrate,  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ , nickel(II) nitrate,  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , and zinc nitrate,  $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  were purchased from Acros Organics and PVP (MW = 10,000) was supplied by Sigma Aldrich. All the chemical reagents were of research grade and used without further purification. 3 g of PVP was dissolved in 100 ml of deionized water at 343 K before mixing 0.2 mmol of iron (III) nitrate, 0.025 mmol of nickel nitrate and 0.075 mmol zinc nitrate and the solution stirred for 2 hours. No precipitation occurred in the solution. The brown solution was poured into a glass Petri dish and heated at 353 K in an oven for 24 hours to release most of the water. The brown solid material was crushed into powder and the samples were heated for 3 hours in alumina boat at different calcination temperatures of 723, 773, 823 and 873 K to decompose the organic matters and crystallize the nanoparticles.

### 2.2. Characterization

The textural and morphological characteristics of the prepared  $\text{Ni}_{0.25}\text{Zn}_{0.75}\text{Fe}_2\text{O}_4$  nanoparticles were studied with several techniques to verify the particle size, shape and size distribution as well as to explore the parameters of interest. The structure was characterized by the XRD technique using a Shimadzu diffract meter model XRD 6000 employing  $\text{Cu K}_\alpha$  (0.1542 nm) radiation to generate diffraction patterns from powder crystalline samples at room temperature in  $2\theta$  range of  $10^\circ - 70^\circ$ . The infrared spectra in the range 280 - 4000  $\text{cm}^{-1}$  were recorded using FTIR spectrometer (Perkin Elmer model 1650). The FTIR spectra are used to confirm the presence of metal oxide bands and the removal of organic matters at 873 K. The structural and particle size of the calcined  $\text{Ni}_{0.25}$

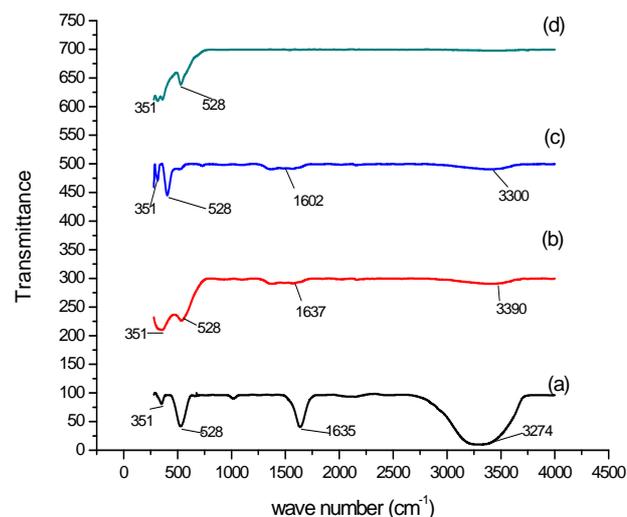
$\text{Zn}_{0.75}\text{Fe}_2\text{O}_4$  nanoparticles were determined at room temperature by using transmission electron micrograph (TEM) (JEOL 2010 UHR version microscopy) at an accelerating electron voltage of 200 kV. The magnetization was measured using a vibration sample magnetometer (VSM) (Lake Shore 4700) at room temperature with a maximum magnetic field of 15 kOe. The electron paramagnetic resonance (EPR) spectra were recorded on a JEOL JES-FA200 EPR spectrometer (JEOL, Tokyo, Japan) at room temperature.

## 3. Results and Discussion

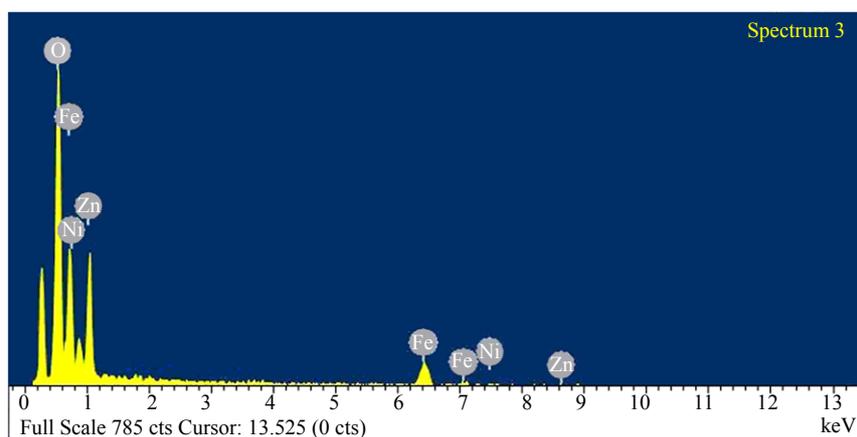
### 3.1. Structural Studies

The FTIR spectra recorded for  $\text{Ni}_{0.25}\text{Zn}_{0.75}\text{Fe}_2\text{O}_4$  nanoparticles in the range between 280  $\text{cm}^{-1}$  and 4000  $\text{cm}^{-1}$  are shown in **Figure 1**. The spectra give information about the chemical and molecular structure changes in the synthesized ferrites after calcination treatments. For all calcined samples at calcination temperatures from 673 to 873 K, shown in **Figures 1(a)-(d)**, two assigned absorption bands appear at 528  $\text{cm}^{-1}$ , which is attributed to stretching vibration of octahedral group Fe-O stretching band and that at 351  $\text{cm}^{-1}$  are attributed to the tetrahedral group Zn-O and Ni-O stretching bands. All absorption peaks above 1000  $\text{cm}^{-1}$  were attributed to covalent bonds of PVP. The bending mode around 1602 - 1637 and 33,274 - 3390  $\text{cm}^{-1}$  were associated with C = O stretching and O-H stretching vibration respectively. The absence of these peaks at 873 K confirmed the organic sources were removed from the calcined samples and pure  $\text{Ni}_{0.25}\text{Zn}_{0.75}\text{Fe}_2\text{O}_4$  nanoparticles were obtained.

**Figure 2** shows EDX pattern of the sample calcined at 873 K, which confirms the Ni, Zn, Fe and O peaks appearing along with the C substrate peak. No contaminat-



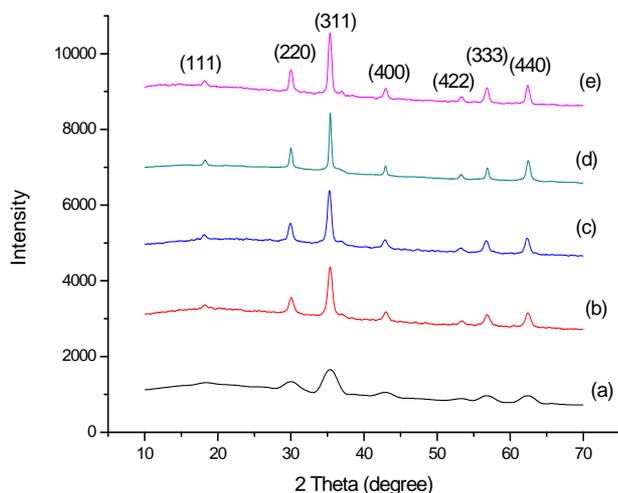
**Figure 1.** FTIR spectra of  $\text{Ni}_{0.25}\text{Zn}_{0.75}\text{Fe}_2\text{O}_4$  nanoparticles calcined at (a) 723, (b) 773, (c) 823 and (d) 873 K.



**Figure 2.** The EDX spectra of  $\text{Ni}_{0.25}\text{Zn}_{0.75}\text{Fe}_2\text{O}_4$  nanoparticles grown on carbon at calcination temperature of 873 K.

ing elements from organic reagents, such as hydrogen or nitrogen were detected.

**Figure 3** shows the XRD diffraction patterns of the  $\text{Ni}_{0.25}\text{Zn}_{0.75}\text{Fe}_2\text{O}_4$  nanoparticles at 353 K and at calcination temperatures from 723 to 873 K. The diffraction peaks show the reflection planes (111), (220), (311), (400), (422), (333) and (440) which are consistent with the standard powder diffraction reported from XRD library code (00-052-0279) and no other metal oxides could be identified. The diffraction peaks become sharper when the calcinations temperature increased. It can be said that all samples formed the spinel phase with a face centered cubic structure (f.c.c). The particle size of the ferrite nanoparticles has been estimated from the XRD plane (311) by the Scherrer's formula:  $d = 0.9 \lambda / \beta \cos \theta$ , where  $d$  is the average particle size in nm,  $\beta$  is the FWHM of the intensity measured in radians,  $\lambda$  is the X-ray wavelength and  $\theta$  is the Bragg angle. The average particle size increased from 9 nm to 23 nm at calcination



**Figure 3.** XRD pattern of  $\text{Ni}_{0.25}\text{Zn}_{0.75}\text{Fe}_2\text{O}_4$  nanoparticles at (a) 353 K, and calcined at (b) 723, (c) 773, (d) 823 and (e) 873 K.

temperature from 723 K to 873 K, as listed in **Table 1**.

The TEM images in **Figure 4** show the size, shape and size distribution of the  $\text{Ni}_{0.25}\text{Zn}_{0.75}\text{Fe}_2\text{O}_4$  nanoparticles at different calcination temperatures. The particle size increased from 7 nm at 723 K to 25 nm at 873 K (**Table 1**). The reason for the increase in the particle size is that the surface of the nanoparticles melted and fused with the neighboring particles at higher calcination temperatures [17-20]. Thus it is possible to control particle size by the calcination temperature. Smaller particle size has been reported for  $\text{Ni}_{0.25}\text{Zn}_{0.75}\text{Fe}_2\text{O}_4$  nanoparticles prepared by wet co-precipitation routes with the average particle size of about 4 nm [21] and 7 nm for  $\text{Ni}_{0.3}\text{Zn}_{0.7}\text{Fe}_2\text{O}_4$  prepared by co-precipitating aqueous solutions in alkaline medium [6]. By ball-milling on  $\text{Ni}_{0.2}\text{Zn}_{0.8}\text{Fe}_2\text{O}_4$  nanoparticles fabricated by mechanochemical method, particle size of about 11 nm was obtained but following annealing at 773 K the particle size increased to about 26 nm [22].

### 3.2. Magnetic Studies

**Figure 5** shows the curves of magnetization measured at room temperature. **Table 1** depicts the values of saturation magnetization ( $M_s$ ) and coercivity ( $H_c$ ) of different samples. Increasing the calcination temperatures from 723 K to 873 K, the  $M_s$  value increased from 11 emu/g to 26 emu/g and the coercivity value also increased from 7 to 29 G, when the particle size increased from 7 to 25 nm, as listed in **Table 1**. The magnetization curves demonstrate a typical superparamagnetic behavior. The  $H_c$  is in direct proportional to the volume of single domain grains. Therefore,  $H_c$  increased when the particles size increased. Higher  $M_s$  values of about 30 emu/g have been achieved for 15 nm  $\text{Ni}_{0.25}\text{Zn}_{0.75}\text{Fe}_2\text{O}_4$  nanoparticles prepared by co-precipitation [21], 22 emu/g for 6 - 7 nm  $\text{Ni}_{0.2}\text{Zn}_{0.8}\text{Fe}_2\text{O}_4$  synthesized by forced hydrolysis in diethyleneglycol [23] and 28 emu/g for 6 - 8 nm  $\text{Ni}_{0.35}\text{Zn}_{0.65}\text{Fe}_2\text{O}_4$  obtained by thermal decomposition [6].

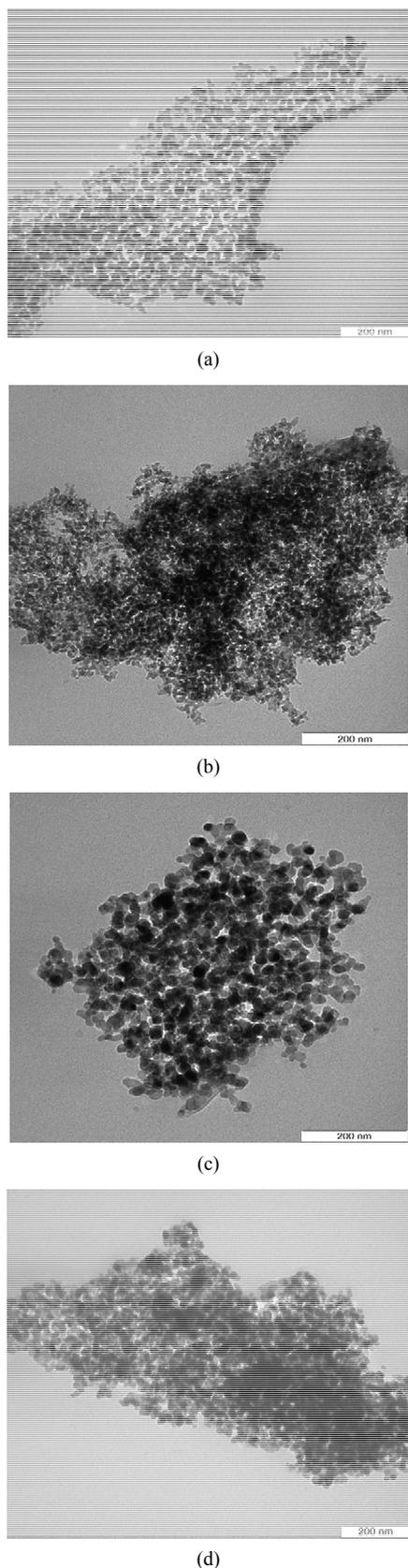


Figure 4. TEM images of  $\text{Ni}_{0.25}\text{Zn}_{0.75}\text{Fe}_2\text{O}_4$  nanoparticles calcined at (a) 723, (b) 773, (c) 823 and (d) 873 K.

Figure 6 shows the EPR spectra of the samples calcined at (a) 723, (b) 773, (c) 823 and (d) 873 K. Peak-to-peak line width ( $\Delta H_{pp}$ ), resonant magnetic field ( $H$ ), and  $g$ -factor are three parameters that characterize the magnetic properties. The  $g$ -factor can be calculated according to the equation:  $g = h\nu/\beta H$  where  $h$  is Planck's constant,  $\nu$  is the microwave frequency,  $\beta$  is the Bohr magneton ( $9.274 \times 10^{-21} \text{ erg}\cdot\text{G}^{-1}$ ), and  $H$  is resonant magnetic field. The values of  $g$ -factor increased from 2.0670 to 2.1220 correspond to the decrease of the resonance magnetic field from 3170 to 3090 G with the increase of calcination temperature from 723 K to 873 K.

#### 4. Conclusion

This paper presented a thermal treatment method for the

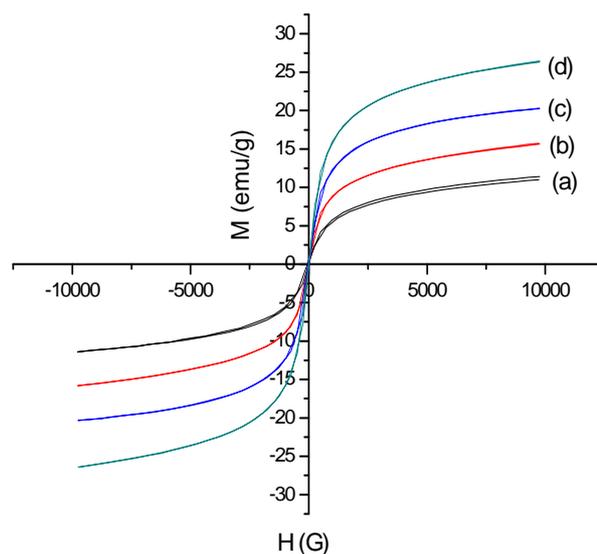


Figure 5. VSM curves of  $\text{Ni}_{0.25}\text{Zn}_{0.75}\text{Fe}_2\text{O}_4$  nanoparticles calcined at (a) 723, (b) 773, (c) 823 and (d) 873 K.

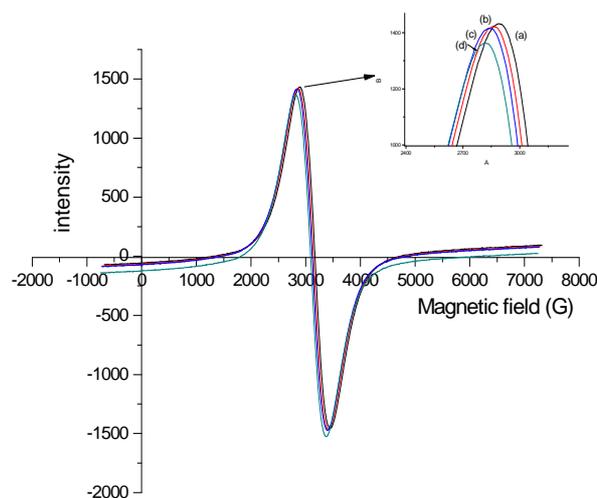


Figure 6. EPR spectra of  $\text{Ni}_{0.25}\text{Zn}_{0.75}\text{Fe}_2\text{O}_4$  nanoparticles calcined at (a) 723, (b) 773, (c) 823 and (d) 873 K.

**Table 1. The average particle sizes (nm) of Ni<sub>0.25</sub>Zn<sub>0.75</sub>Fe<sub>2</sub>O<sub>4</sub> nanoparticles determined from XRD and TEM and magnetic properties observed from VSM and EPR at room temperature.**

Ni <sub>0.25</sub> Zn <sub>0.75</sub> Fe <sub>2</sub> O <sub>4</sub> nanoparticles	Calcinations temperature (K)	Average particle size XRD (nm)	Average particle size TEM (nm)	Saturation magnetization, M <sub>s</sub> (emu/g)	Coercivity field H <sub>c</sub> (G)	Peak to peak line width ΔH <sub>pp</sub> (G)	g-factor	Resonance field, H <sub>r</sub> (G)
Ni-Zn ferrite 1	723	9	7	11.414	7	758	2.0670	3170
Ni-Zn ferrite 2	773	11	9	15.785	10	755	2.0885	3140
Ni-Zn ferrite 3	823	14	13	20.320	26	753	2.1050	3118
Ni-Zn ferrite 4	873	23	20	26.447	29	750	2.1220	3090

synthesis of Ni<sub>0.25</sub>Zn<sub>0.75</sub>Fe<sub>2</sub>O<sub>4</sub> nanocrystals with grain size ranging from 7 to 25 nm at the calcination temperatures from 723 to 873 K as measured by XRD and TEM. The PVP stabilized the particles and prevented them from agglomerating. The FTIR measurement confirmed the removal of all organic matters and leaving pure metal oxides at 873 K. The VSM results showed that the saturation magnetization increased from 11 emu/g to 26 emu/g and the coercivity value increased from 7 to 29 G at calcination temperatures from 723 K to 873 K due to the increasing of the volume of single domain grains. The values of g-factor increases with increase of calcination temperature and particle size were increased. This simple method, which is cost-effective and environmentally friendly, produces no toxic byproduct effluents and can be used to fabricate pure, crystalline spinel ferrite nanocrystals.

## 5. Acknowledgements

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