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Research Paper

Simple microwave combustion method and magnetic characterizations of spinel Ni-Zn ferrite nanoparticles

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Abstract

Spinel Ni-$Zn_{1-x}Fe_2O_4$ (x = 0.0 and 0.5) nanoparticles were synthesized by simple microwave combustion method. The structural, morphological and magnetic properties of the Ni-Zn ferrites were determined by X-ray diffraction (XRD), high resolution scanning electron microscopy (HR-SEM) and vibrating sample magnetometer. The XRD confirms that all the compositions crystallize with cubic spinel $ZnFe_2O_4$. HR-SEM images revealed that the as-prepared samples are crystalline with particle size distribution in 22-27 nm range. The saturation magnetization (M_s) increased with increase in Ni content.

Keywords: Microwave combustion; Spinel ferrites; Electron microscopy; Magnetic properties.

1. Introduction

Spinel Ni-$ZnFe_2O_4$ ferrite has attracted a vast of interest, because of its high resistivity, high permeability, and low dielectric loss in high frequency device applications [1]. Many methods have been used to prepare the spinel ferrite nanoparticles, such as solvothermal, co-precipitation, hydrothermal, citrate precursor, sol–gel. Nevertheless, the above methods have some disadvantages such as, high-energy consuming, requirement of complicated equipment,

requirement of a strong base, like NaOH, higher processing temperature and also require rather long reaction time to complete the crystallization of $ZnFe_2O_4$ [2-10].

Nanocrystalline spinel ferrites possess unique structural and opto-magnetic properties than that of their same bulk counterparts [1]. Spinel ferrites have applications in the area of magnetic resonance imaging and multilayer chip indicator, etc. Spinel ferrites with a general formula MFe_2O_4 (M= Co^{2+}, Ni^{2+}, Zn^{2+}, etc.) have been investigated for their usual optical and magnetic properties. Zinc ferrite ($ZnFe_2O_4$) is a normal spinel structure with Zn^{2+} ions located at the tetrahedral sites and Fe^{3+} ions at the octahedral sites [2]. $ZnFe_2O_4$ is a commercially important material and has been widely used in many areas, such as photo-catalysts [3], gas sensors [4], catalysts [5, 6] absorbent materials [7], and information storage [8].

Microwave combustion method has recently gained importance than the above said methods. The microwaves interact with the reactants at the molecular level, which leads to a uniform heating. During the microwave combustion, the microwave energy is transferred and converted into heat, because of the motion of the molecules. This results in the formation of $ZnFe_2O_4$ nanoparticles within few minutes of time and leads to a higher efficiency [11-19].

In this present study, we have synthesized $Ni_xZn_{1-x}Fe_2O_4$ (x = 0.0 and 0.5) nanoparticles by microwave combustion method using glycine as the fuel. The structural phase of the prepared samples was characterized by powder X-ray diffraction (XRD) analysis. The particle size and morphologies were determined by high resolution scanning electron microscopy (HR-SEM) and the chemical composition was determined by the energy dispersive X-ray (EDX) analysis. The magnetic behavior of the samples was studied by the vibrating sample magnetometer (VSM).

2. Experimental

2.1. Materials and methods

All the chemicals used in this study were of analytical grade obtained from Merck, India and were used as received without further purification. Zinc nitrate ($Zn(NO_3)_2{\cdot}6H_2O$, 98%), ferric nitrate ($Fe(NO_3)_3{\cdot}9H_2O$, 98%) and nickel nitrate ($Ni(NO_3)_2.6H_2O$) were used as precursors and glycine ($C_2H_5NO_2$) as a fuel for this method. The compositions were prepared with the addition of nickel by molar ratios ($Ni_xZn_{1-x}Fe_2O_4$with x = 0.0 and 0.5) to $ZnFe_2O_4$. For the preparation of pure and Ni-doped zinc ferrite using the microwave combustion technique, the

precursor mixture in glycine was placed into a domestic microwave oven and exposed to the microwave energy in a 2.45 GHz multimode cavity at 750 W for 10 min. After the completion of the reaction, the solid powder was obtained and then it was washed with ethanol and dried at 70ºC for 1 h. The obtained powders were labeled as $ZnFe_2O_4$ and $Ni_{0.5}Zn_{0.5}Fe_2O_4$.

2.2. Characterization techniques

The structural characterization of spinel $Ni_xZn_{1-x}Fe_2O_4$ (x = 0.0 and 0.5) nanoparticles were performed using a Rigaku Ultima X-ray diffractometer equipped with Cu-Kα radiation (λ = 1.5418 Å). Morphological studies and energy dispersive X-ray analysis of Ni-doped $ZnFe_2O_4$ nanoparticles have been performed with a Jeol JSM6360 high resolution scanning electron microscope (HR-SEM). Magnetic measurements were carried out at room temperature using a PMC MicroMag 3900 model vibrating sample magnetometer (VSM) equipped with 1 T magnet.

3. Results and discussion

3.1. XRD analysis

The structural analysis of $Ni_xZn_{1-x}Fe_2O_4$ (x = 0.0 and 0.5) samples was done by powder X-ray diffraction (XRD) technique using Cu Kα radiation. Fig. 1a,b shows the XRD patterns of $Ni_xZn_{1-x}Fe_2O_4$ (x = 0.0 and 0.5) samples. It can be observed that all the peaks of pure as well as Ni-doped $ZnFe_2O_4$ powders can be easily indexed with cubic $ZnFe_2O_4$ spinel structure (JCPDS No. 22-1012). The diffraction peaks at 2θ values 29.99º, 35.24º, 36.82º, 42.83º, 53.14º, 56.65º, 62.19º and 73.44º can be ascribed to (220), (311), (222), (422), (400), (511), (440) and (533) planes of the spinel crystal structure, respectively. There is no additional peak for all compositions, which indicates that all the samples crystallize in single-phase cubic structure with Fd3m space group [20].

In addition, the crystallite size is estimated from the most intense (311) reflection peak using the Debye Scherrer formula [21],

$$L = \frac{0.89\lambda}{\beta \cos\theta}$$

where L is the crystallite size, λ , the X-ray wavelength, θ, the Bragg diffraction angle and β, the full width at half maximum (FWHM). The crystallite size of $ZnFe_2O_4$ and $Ni_{0.5}Zn_{0.5}Fe_2O_4$

samples are 22.22, and 21.12 nm, respectively. It shows clearly that by increasing the amount of Ni^{2+} ions, the crystallite size decreased. It is observed that lattice parameter decreased from 8.444 Å to 8.436 Å with increase in nickel concentration, which attributes to the replacement of larger Zn^{2+} (0.83 Å) ions by smaller Ni^{2+} (0.74 Å) ions [22-27].

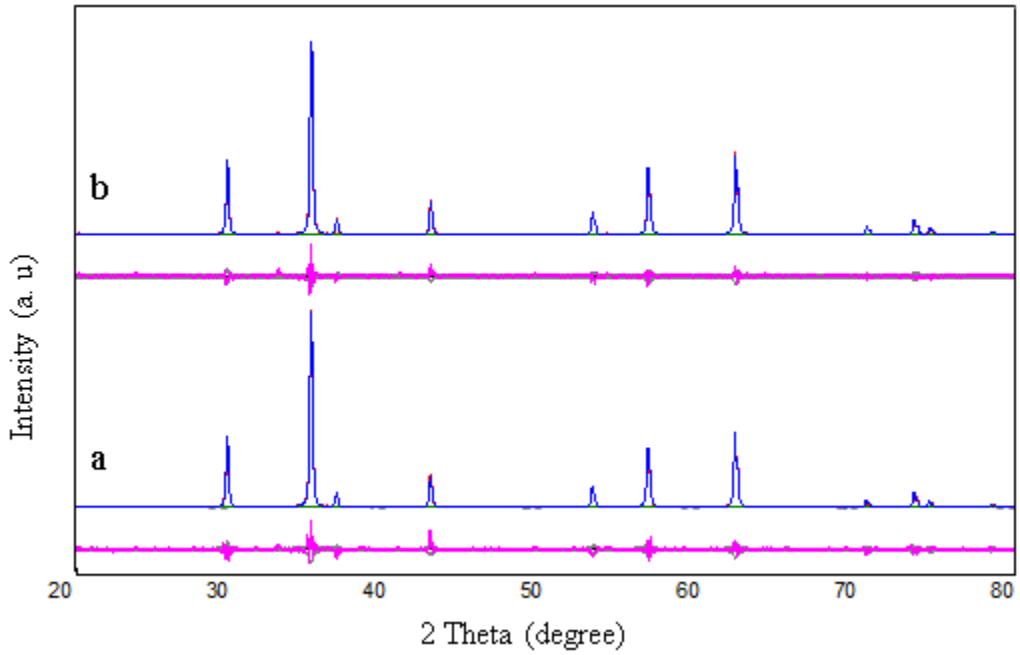

Fig. 1. Powder XRD patterns of Ni-$ZnFe_2O_4$ spinel ferrites.

3.2. Scanning electron microscopy (SEM) studies

High resolution scanning electron microscope (HR-SEM) studies was used to investigate the microstructures with the change in Ni composition in $ZnFe_2O_4$ nanoparticles. HR-SEM micrographs of the as-synthesized samples exhibited uniform, almost spherical shaped and loosely agglomerated particles as shown in Fig. 2. The average particle size of the ferrite nanoparticles prepared via this route is found to be in the range of 23-25 nm. It is observed that the particle size increases as the concentration of Ni ion increases.

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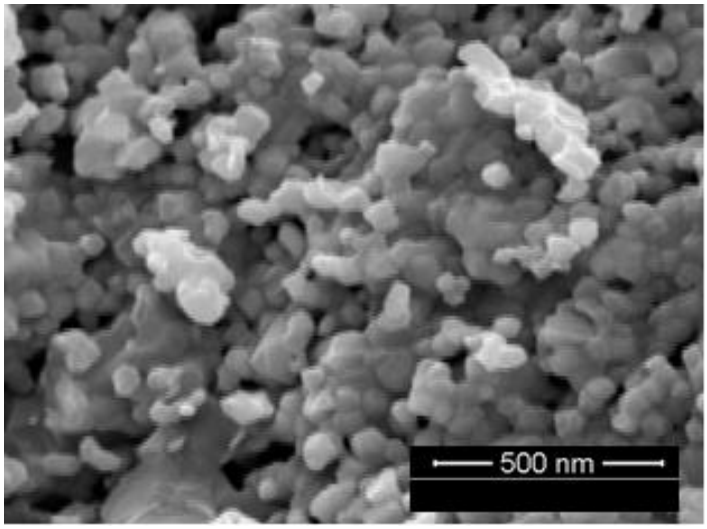

Fig. 2. SEM images of Ni-$ZnFe_2O_4$ spinel ferrites.

3.3. Energy dispersive X-ray analysis (EDX)

Energy dispersive X-ray analysis (EDX) of the respective samples is shown in Fig. 3. The peaks corresponding to the elements Fe, Zn and O were observed in pure $ZnFe_2O_4$ (Fig. 3) and the peaks of the elements Fe, Zn, Ni and O were observed in Ni-doped $ZnFe_2O_4$ samples. A small peak is appeared at 2.1 KeV for all the samples, which indicates the presence of gold (Au) peak that has been used as a coating, while preparing the sample for HR-SEM analysis for the better visibility of the surface morphology.

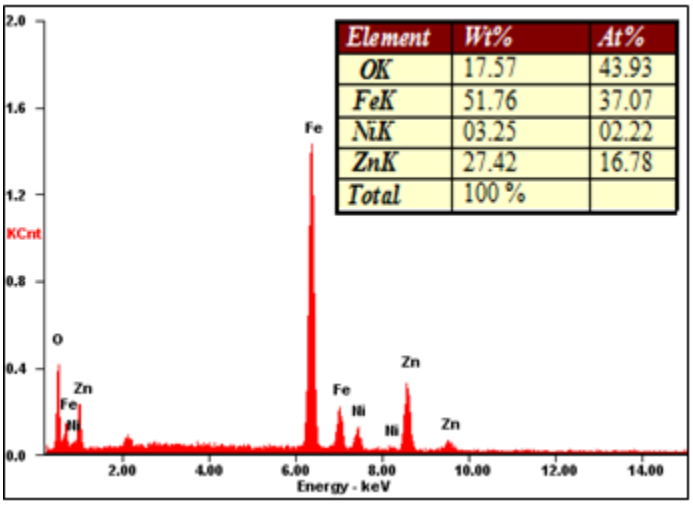

Fig. 3. EDX spectra of Ni-$ZnFe_2O_4$ spinel ferrites.

3.4. HR-TEM analysis

The structure and size of $Ni_xZn_{1-x}Fe_2O_4$ NPs was confirmed by TEM images (Figure 4). The particles size decreasing with the increase of Ni-content, which is similar to the crystallite sizes confirmed by XRD analysis. Also, the nanoparticles appear as agglomerated spherical particles. The spherical shapes of particles may be the result of the synthesis method and surface properties, while the agglomeration may be due to the interfacial surface tension phenomenon.

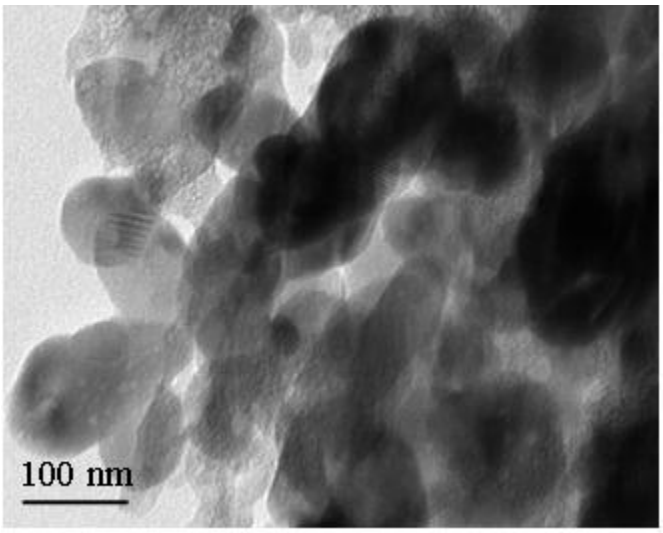

Fig. 4. TEM images of Ni-$ZnFe_2O_4$ spinel ferrites.

3.5. Magnetic measurements

The magnetic property of the as-prepared $ZnFe_2O_4$ and $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanoparticles was investigated with a vibrating sample magnetometer (VSM) at room temperature with the applied field ranging from -10000 to +10000 Oe. Fig. 5 shows the magnetic measurements of $ZnFe_2O_4$ and $Ni_{0.5}Zn_{0.5}Fe_2O_4$ samples. From the results, it is clearly understood that the magnetic properties of the samples are affected by the composition and cation distribution [28-35]. When non-magnetic Zn^{2+} in $ZnFe_2O_4$ is substituted by magnetic Ni^{2+} ions, there is a drastic change in magnetic properties such as M_s, M_r and H_c. The magnetization curve demonstrates a typical superparamagnetic behavior of the as-prepared pure $ZnFe_2O_4$ nanoparticles with lower remanence and coercivity. This is confirmed by the non saturation observed in MH loop and the

absence of the hysteresis, M_r and H_c. The superparamagnetic nature can be attributed to their fine crystalline size, which makes it easier for them to be thermally activated to overcome the magnetic anisotropy [36]. For $Ni_{0.5}Zn_{0.5}Fe_2O_4$ sample, a hysteresis was observed, thus indicating the ferromagnetism. This is due to the increase in the magnetic nature of the Ni^{2+} concentration. The value of M_s for pure $ZnFe_2O_4$ is 1.638 emu/g which increased with the increase in Ni^{2+} concentration. The observed differences in the VSM results (M_s, M_r and H_c) between pure and Ni-doped $ZnFe_2O_4$ can be attributed to the finite size effects. Moreover, the very low values of H_c and M_r, indicate that they are also soft magnets.

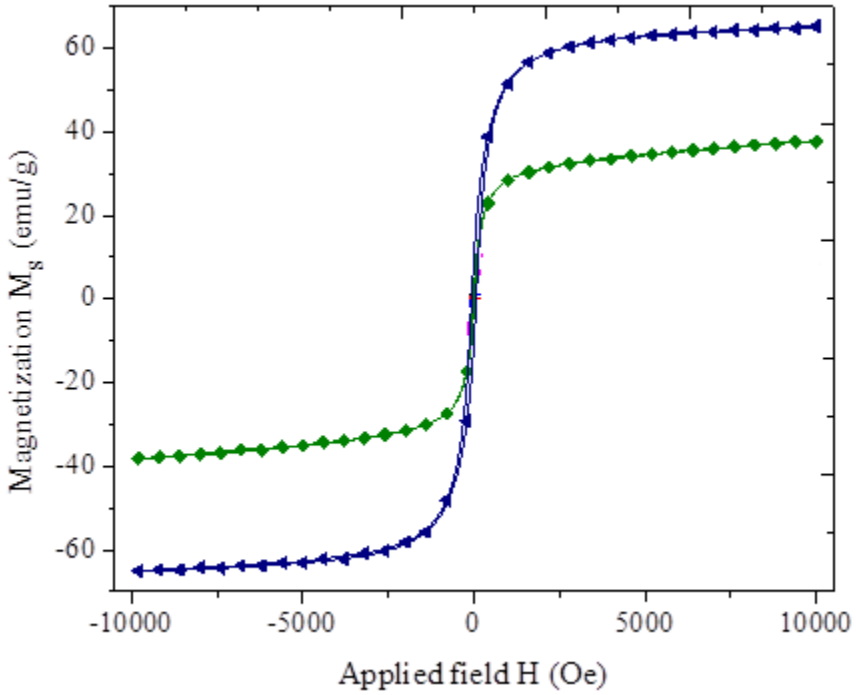

Fig. 5. Magnetic characteristics of Ni-$ZnFe_2O_4$ spinel ferrites.

4. Conclusions

Nanocrystalline $Ni_xZn_{1-x}Fe_2O_4$ (x = 0.0 and 0.5) nanoparticles were successfully prepared by microwave combustion method using glycine as the fuel. Microwave combustion method is suitable for preparing the spinel structure with good crystallinity and reproducibility. The crystallite size was found to vary within the range of 25.21 to 26.13 nm. The results revealed that

the decreases in Zn concentration lead to the decrease in particle size, which ultimately affects the magnetic properties of the sample. Room temperature VSM measurements revealed that the saturation magnetization of the as-synthesized $Ni_xZn_{1-x}Fe_2O_4$ nanoparticles lies in the range of 1.638 to 64.73 emu/g depending upon the Ni content. The synthetic methodology of magnetic materials is technically simple and effective, because the starting materials are very cheap and this method does not require any complex equipment and complicated operation, thus offers more advantages than the other methods reported so far.

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