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RESEARCH ARTICLE

MAGNETIC ZINC SUBSTITUTED NICKEL FERRITE NANOPARTICLES SYNTHESIZED BY SOL-GEL

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ABSTRACT

The sol-gel method was used to produce nanoparticles of nickel ferrite. The brown powders were sintering at the temperature of 800˚C for 24 hrs. The ferrite nanoparticles were determined by X-ray diffraction (XRD). The structural characteristics of calcined sample of Ni_{1-x} Zn_x Fe_2 O_4 (x = 0.0, 0.1, 0.2, 0.3, 0.4, 0.5).The FT-IR analyzed the functional group. The prepared samples have cubic structure and crystalline size decreases from 25-55nm calculated using Debye's Sherrer formula. The preparation method investigated brought about formation of Nickel ferrite single phase. TG/DTA measurement showed the weight loss between 0-200˚C, 200-400˚C and 400-600˚C which corresponding the endothermic and exothermic processes. An exothermic peak observed at around 400˚C is due to the thermal decomposition of the ingredients to form Ni Fe_2O_4. The Vibrating Sample Magnetometer was used to obtain the Hysteresis parameters. The saturation magnetization value is 0.2 emu/g to 0.7emu/g for the sample sintered at 800˚C.

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INTRODUCTION

The size of nanoparticles spans the range between 1 to 100 nm. (Satoshi Horikoshi and Nick Serpone, 2013). The small particles contain 10 or 100 of atoms, with dimensions at the scale of nanometers-hence nanoparticles (Cristina Buzea *et al*., 2007). The application of interest, nanoparticles may be known by a number of alternative and trade-specific names, including particulate matter, aerosols, colloids, nano composites, nano powders, and nano ceramics etc. Nickel ferrite is known to exist in tetrahedral and octahedral spinel cubic structures. They are recently attracted considerable research interest on their structural, magnetic properties (Hou *et al*., 2010). Zn^{2+}substituted Nickel ferrite has been synthesized by sol-gel method (Bobade and Brabers, 1967). Nickel ferrite have many advantages such as high electromagnetic performance, moderate saturation magnetization, hysteresis and good chemical stability (Awati *et al*., 2013). Zn^{2+} ion concentration in Ni spinel ferrite by sol gel method has been investigated single phase cubic structure and surface morphology (Karthick and Venkatesh, 2015).

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MATERIALS AND METHODS

Preparation

The samples were synthesized using nickel nitrate, zinc nitrate, and iron nitrate. All the chemicals were supplied by Modern scientific chemicals Ltd. Ethylene glycol, oxalic acid as a gelling agent because it plays an important role in homogeneous mixing. Nickel nitrate dissolve in ethanol, Zinc nitrate dissolve in ethanol, and ferric nitrate dissolve in ethanol then Zinc nitrate slowly added with Nickel nitrate, next Nickel nitrate added with ferric nitrate, and then oxalic acid dissolve in 2ml of ethanol, finally drop wise ethylene glycol added while stirring at 90˚C, after the solutions was evaporated with continuously stirrered to obtain uniform gel. After 1-2 hours the gel is formed. The brownish powder obtained is subjected to calcinations at 800˚ C for 3hrs.

RESULTS AND DISCUSSION

TG-DTA analysis

The thermal decomposition of Ni Fe_2O_4 nanoparticles is shown in Figure (2). The TGA curve of Nickel ferrite shows three main steps of mass loss. The first stage of degradation can be observed between 0 and 200°C, with respect to the output of

waste water which is adsorbed on the material during the synthesis step. The corresponding DTA curve indicates that these processes are exothermic at temperature 200 and 400˚C. The second stage of degradation occurs between 200-400°C, Loss in weight results due to evaporation of moisture or solvent. The third step weight loss occurs in the temperature range of 400-600ºC with the removal of organic impurities still remaining in the material after 800°C. A continuous line in the TGA curve is related to the end of the degradation of ions and oxalic acid.

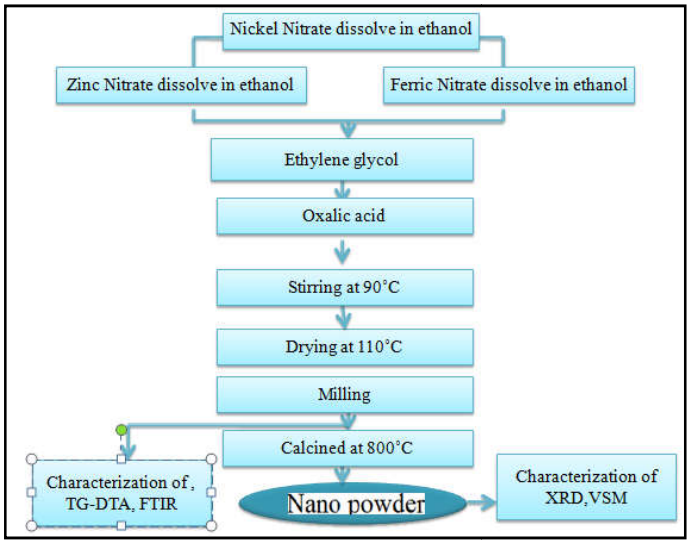

Fig.1. Synthesis of nickel ferrite nano powder

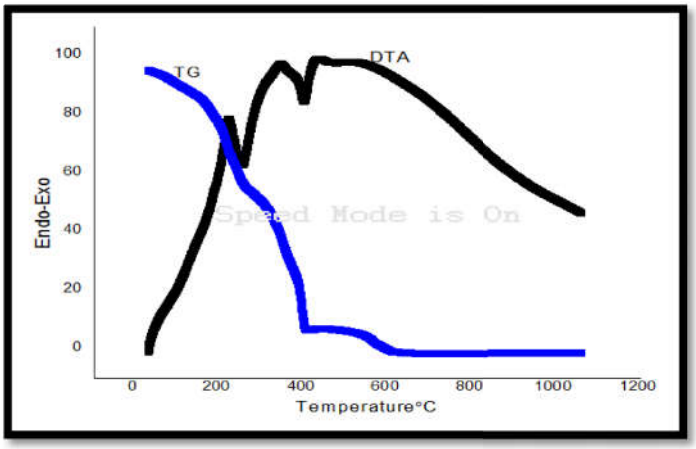

Fig.2. DTA and TG plots of the dried gels for $Ni_{1-x}Zn_xFe_2O_4$

FT-IR analysis

Figure (3) shows the FTIR spectrum of the Ni Fe_2O_4 nanoparticles synthesized by sol gel method, which was acquired in the range of 400-4000 cm^{-1}. Nickel ferrite IR curve fig (3) shows the absorptions of ferrite with a strong absorption around 1357cm^{-1} to the intrinsic vibrations of the nitro compounds groups with respect to the N-O symmetric stretch bond. Then 661 Cm^{-1} is the C-Br stretching represented by alkyl halides groups. There are two weak and broad absorption peaks at around 1500 and 1600 cm^{-1} corresponding to the presence of small amounts of residual carbon in the samples. The peaks are at 2398 Cm^{-1} and 3367 Cm^{-1} corresponding to the stretching vibration of C≡N,N-H respectively as shown in the Fig(3).

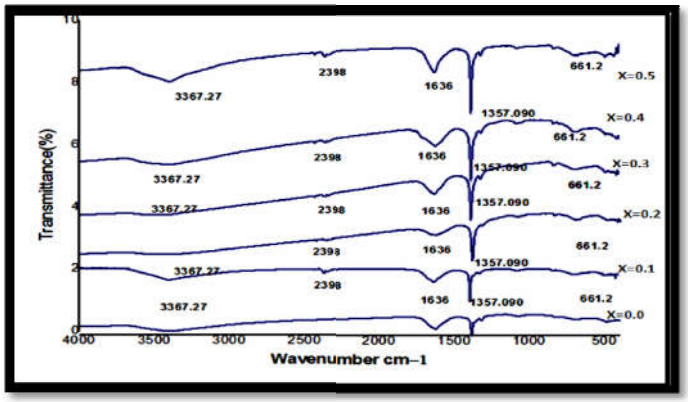

Fig.3. FTIR spectra of $Ni_{1-x}Zn_x$ Fe_2O_4 with (x=0.0, 0.1, 0.2, 0.3, 0.4, and 0.5)

Structural analysis

Figure (4) shows the X-ray diffraction (XRD) patterns of typical samples of Ni_{1-x} Zn_x Fe_2O_4 (Where x=0.0, 0.1, 0.2, 0.3, 0.4, 0.5). In the XRD pattern of nickel ferrite nanoparticles diffraction peaks at 35˚, 42˚, 55˚, 57˚and 63˚can be assigned to cubic spinel structure. The broadening of the Bragg's peaks indicates the formation of nickel. The major planes correspond to (531), (220), (311), (400), (422), (511) were found to be matched which confirmed the presence of cubic nanoparticles. The X-ray diffraction patterns show well developed diffraction lines assigned to cubic single phase, with all major peaks matching with the standard pattern of Ni-Zn ferrite, JCPDS 08-0234. The average crystallite sizes decreases from 25-55nm using Scherrer's formula.

$$D=K\lambda / (\beta \cos\theta)$$

In which, where k is a constant taken as0.9, θ is the diffraction angle; λ is the wavelength of the X-ray radiation. β is the full width half maximum (FWHM) of each phase and D=average particle size of crystallite.

$$a = d_{hkl}/(h^2+k^2+l^2)^{1/2}$$

Where (hkl) are the miller indices and d_{hkl} is interplanar spacing. The X-ray density ($\rho_{X\text{-}ray}$) was calculated from the relation,

$$\rho_{X\text{-}ray} = 8M/Na^3$$

Where, M is the molecular weight, N the Avogadro number and "a" is the lattice constant. The calculated values of crystallite size, lattice constant, d spacing, and X-ray densities are tabulated in Table (1).

SEM Morphology

Figure (3) shows morphological pattern of the prepared Nickel ferrite powder taken by scanning electron microscope. The

SEM figure clearly indicates that microstructure of the grains of the sample is ultra-small. Most of the grains containing a large number of atoms are very small in dimensions, having an average grain size of 10 µm. The size distribution is extraordinarily narrow. Also, there exist some grains of larger size but having normal growth.

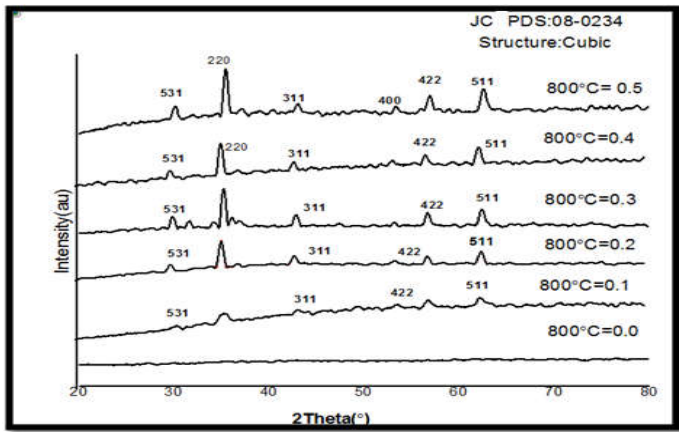

Fig.4. XRD patterns of $Ni_{1-x}Zn_x$ Fe_2O_4 with (x=0.0, 0.1, 0.2, 0.3, 0.4 and 0.5)

Table 1. Variation of crystallite size, lattice constant, d-spacing, and X-ray density with nickel ferrite concentration

Ni Fe_2O_4 Concentration	Crystallite size (nm)	Lattice constant (A)	d spacing (A)	X-ray density (g/cm3)
X=0.0	55.38	8.3429	2.1430	3.7325
X=0.1	43.50	8.3558	2.0640	3.7263
X=0.2	37.72	8.3731	2.1151	3.7186
X=0.3	31.20	8.3864	2.2619	3.7128
X=0.4	29.97	8.4213	2.1374	3.6973
X=0.5	25.24	8.4456	2.1468	3.6867

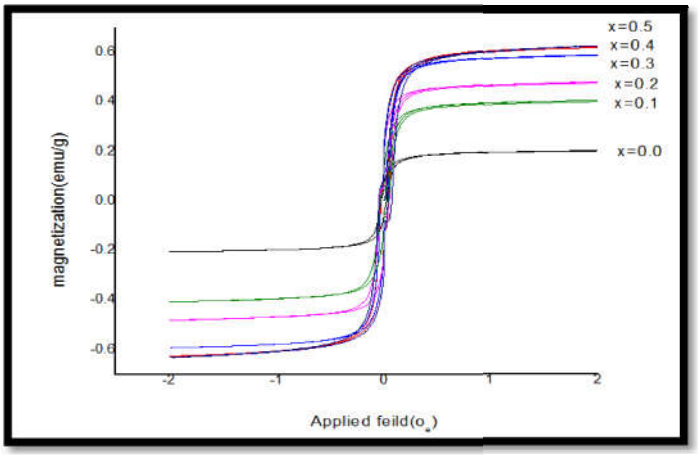

Fig.5. Hysteresis loops of $Ni_{1-x}Zn_xFe_2O_4$ sintered at 800˚C

TG-DTA

Fig (4) shows the thermal analysis of Nickel ferrite. The TG-DTA curves indicate that the degradation of dried gel show multi -step weight loss.

The TG data plots the weight loss of the Nickel Ferrite nanoparticles which shows that a small amount of weight loss has occurred around 400˚C to 600˚C, thereby indicating the evaporation of moisture. The DTA curve obtained shows multistate decomposition without forming any intermediates.

Magnetic measurements

The magnetic properties were studied from the VSM graph shown in the Figure (5). Temperature values of saturation magnetization (Ms). All the samples show narrow hysteresis loop which indicates the soft ferrite nature. Hysteresis loops of synthesized sample are shown in fig (5) saturation magnetization value is 0.2 emu/g to 0.7emu/g for the sample sintered at 800˚C.The increase of saturation magnetization with increasing Zn concentration .The particles are soft magnetic material at 800˚C.

Conclusion

Zn substituted $NiFe_2O_4$ nanoparticles can be prepared by the sol-gel method. From FTIR patterns analyzed the functional group. The formation of cubic phase Nickel ferrite was confirmed by the XRD technique. The synthesis of nanoparticles with crystalline size and lattice constant decreases from 25-55nm for 800˚C. From VSM studies, the effect of Zinc substitution on saturation magnetization was analyzed. It showed that there is an increase in saturation magnetization for all the concentration of Nickel ions and were explained using Neel's two sub lattice model. The 800˚C B-H hysteresis curve show that particles are soft magnetic at 800˚C.

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