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Synthesis and Structural Analysis of Ni-Zn Ferrite Nanoparitcles

Raje Shaikh B.B.[1], Chaitali Vilasrao More [2], Bhagwan G Toksha[3], Syed Qadeeruddin Chishty [4]

[1] *Assistant Professor, Department of Physics, Government College of Arts and Science, Maharashtra, India*
[2] *Research scholar, Department of Physics, Dr. Babasaheb Ambedkar Marathwada University, Aurangabad, Maharashtra, India.*
[3] *Assistant Professor, Basic Sciences and Humanities department, Maharashtra Institute of Technology, Maharashtra, India*
[4] *Associate professor, Department of Physic, Dr. Rafiq Zakaria College for women Aurangabad, Maharashtra, India*

ABSTRACT

Ferrite composition having general formula Ni1−xZnxFe2O4 with 0.7 as Ni0.7Zn0.3Fe2O4 was prepared by sol gel auto combustion method. The method used for synthesis involves use of nitrates of respective elements and by keeping metal nitrate to citrate ratio 1:1. The article is mainly focused on synthesis and structural properties were studied by means of X-ray diffraction, Thermogravimetry (TGA) and Scanning electron microscope to confirm chemical composition, to determine the particle size and to study surface morphology. Particle Size Analyzer was employed to determine the particle size of the sample. The X-ray diffraction analysis confirmed the single-phase formation of the samples. The lattice parameter obtained from XRD data was found in agreement with literature.
Keyword Ferrites, Sol-gel auto combustion, nanoparticles, synthesis, Structural properties

1. INTRODUCTION

In spinel structure ferrites both divalent and trivalent cations are distributed among tetrahedral (A) and octahedral (B) sites. The site preference shown by divalent ions defines whether the spinel is normal, inverse or mixed. In most cases, magnetic divalent cations (such as Ni2+) prefer the octahedral sites and produce an inverse spinel structure. Diamagnetic divalent cations (such as Zn2+) have preference for the tetrahedral positions and the resulting structure is a normal spinel [1]. Of all ferrites, Ni-Zn is the most versatile because of their many technological applications. These ferrites are known to have high resistivity, low eddy current losses, and relatively low initial permeability. Ni-Zn ferrite has unique high dielectric properties which makes them useful in designing electronic devices [2-3]. Owing to their low power loss (LPL) property, Ni-Zn ferrites can be used in switch mode power supplies (SMPS) for the working frequencies climbing above 1MHz. Hot pressed and single crystal Ni-Zn ferrites are found quite suitable for magnetic recording head due to high thermal stability, wear and corrosion resistance with low magnetostriction and high frequency response. For high frequency applications knowledge of dielectric properties of ferrites is of prime importance. The parameters that need to be tuned properly are dielectric constant (ε′), dielectric loss (ε″) and loss tangent (tanδ). High frequency applications of Ni-Zn ferrites include large number of microwave components such as circulators, isolators, gyrators, phase shifters, YIG tuned filters, and switches and substrates for microwave integrated circuits [4]. It is found that there are systematic studies have been reported on the properties of Ni-Zn ferrite [5-8]. Soft ferrites are usually prepared by ceramic method, which suffers from many drawbacks [9-10]. Recently some chemical methods have been used to synthesize soft ferrites for the reason that they produce nano-size particles [11-14]. The systems made up of nano-particles are intensively studied both theoretically and practically, due to their electrical and magnetic properties that are sensibly different from those of their bulk counterpart [15-16].

2. SYNTHESIS

The nano-crystalline sample of the formula *Ni1−xZnxFe2O4 with 0.7 as Ni0.7Zn0.3Fe2O4 was* prepared by citrate nitrate sol-gel auto-ignition synthesis route. The A.R. grade citric acid (C6H8O7·H2O), Nickel nitrate (Ni(NO3)2·6H2O), Zinc nitrate(Zn(NO3)3·9H2O), ferric nitrate (Fe(NO3)3·9H2O) and ammonia (>99% sd-fine) were used as starting materials. In the present system, product was synthesized by keeping metal nitrates to citrate ratio 1:1 and adding ammonia maintaining pH at 6. The as-prepared powder was heat treated separately at 500 °C for 4 h to get the final product.

3. STRUCTURAL ANALYSIS

The X-ray diffraction investigation of the sample was carried out and is as shown in Figure1. Part of the powder was X-ray examined by a Bruker X-ray diffractometer (model D8 advance) using Cu-Kα radiation (λ = 1.5405 Å). The graph shown in Figure 1, portrays X-ray diffraction spectra has highest intensity of 5647 counts at 36.49º. The patterns are in good agreement with the standard diffraction data of Ni-Zn ferrite nanoparticles (JCPDS file No. cardno.08-0234). The peaks observed at (220), (311), (222), (400), (422), (333) and (440) diffraction planes reveal the cubic phase of Ni-Zn ferrite nanoparticles. The average crystallite size for NiZn nanoparticles is calculated from intensity peaks by the Debye-Scherrer equation. In addition, the Williamson and Hall (W-H) plots [17] are also used to calculate the crystallite size. Similar results are reported in literature for closely related composition [18]. The crystallite size of 40 nm was estimated using Scherer equation. Dynamic light scattering (DLS) and particle size measurements were performed using a Zeta-potential and Particle Size Analyzer (Zetasizer ZS; Malvern Panalytical). For DLS and zeta potential tests, suspensions of 5 mg nanoparticles in 50 mL deionized water were subjected to ultrasound (15 min) before the analyses. The mean hydrodynamic size obtained by DLS was 285 nm for Ni-Zn ferrite NPs. The measurements of the zeta potential were used to assess the effects of nanoparticles in the colloidal phase and their aggregates. Higher zeta potentials indicate stable nanoparticles systems [19]. Thus, it can be concluded that well dispersed Ni-Zn ferrite nanoparticles solution in water was synthesized in the present work.

The morphology of the sample was investigated by SEM as shown in Figure 2. SEM micrograph showed freely distributed sharp edged grains. Shapes of the grains are spherical or elliptical. The sample image revealed smaller size distribution. The as prepared sample was investigated by thermogravimeter (TGA) in air (Model: TGA-50, Shimadzu) with rate of heating maintained at 10^0C per minute. In TGA pattern, the system undergoes fast decrease in weight between 329^0C to 401^0C of 44.33% (1.01mg) is ascribed to burning of organic material in the sample and accompanied by the gradual crystallization process. The weight loss at 401^0C is assigned to crystal perfection and grain growth. Beyond 450^0C no weight loss is detectable.

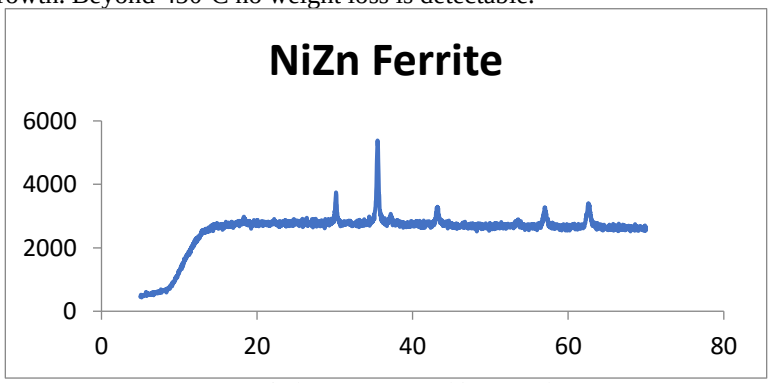

Fig -1 XRD pattern of Ni1−xZnxFe2O4 with 0.7 as Ni0.7Zn0.3Fe2O4

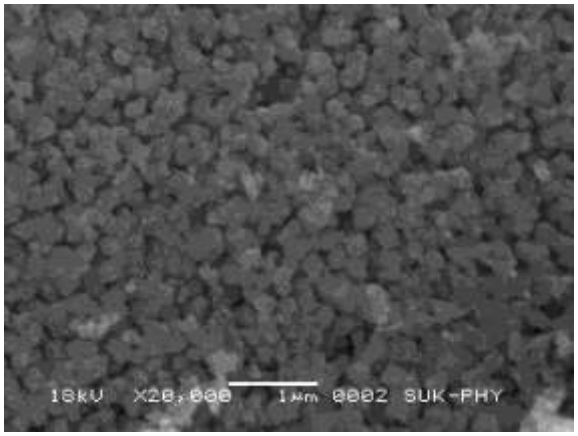

Fig -2 SEM images of Ni1−xZnxFe2O4 with 0.7 as Ni0.7Zn0.3Fe2O4

The recorded diffraction pattern identified that the sample has single phase cubic spinel structure. The diffraction pattern is without appearance of any extra peaks representing no existence of secondary phases. All peaks

in the XRD pattern matches well with the characteristic reflections. The Inter-planer spacing (d) values were calculated for the recorded peaks using Bragg's law and the lattice constant 'a' was calculated for each plane. The value of lattice constant was obtained from XRD data with an accuracy of ±0.002 ˚A and given in Table I. A comparison of the values of lattice constant obtained by the standard ceramic method (Table I) with those obtained in the present investigation shows considerable change. Theoretical lattice constant (ath) was calculated using the equation discussed elsewhere [20]. The calculated agree with that of 'a' however the values of 'ath' are little smaller than the values of 'a'.

Table 1. Lattice constant (a), Theoretical lattice constant (ath), X-ray density (dx), Measured density (dm), Porosity (P), particle size (t) and Specific surface area (S) of *Ni1−xZnxFe2O4 with 0.7 as Ni0.7Zn0.3Fe2O4*

Lattice constant Å	Theoretical lattice constant Å	X-ray density (gm/cm3)	Measured density (gm/cm3)	Porosity %	Particle size nm	Specific surface area (m2/gm)
8.405	8.401	5.353	4.371	18.11	40	27

The bond length RA (the shortest distance between A-site cations and oxygen ion) and RB (the shortest distance between B-site cations and oxygen ion) were calculated using the relations discussed elsewhere [21]. The values calculated are in good agreement with literature values. According to Levine [22], there exists an inverse relationship between the covalent character of the spinel and bond lengths. The distance between magnetic ions, the hopping lengths in tetrahedral sites (LA) and in octahedral sites (LB) was also calculated using the relation reported in Ref. [23]. This may be explained on the basis of difference in ionic radii of the constituent ions. Hopping length (LA), (LB), and bond lengths (RA), (RB) of present sample are calculated and found out to be 3.630, 2.978, 1.901, 2.051 respectively, lengths are all in Å.

4. CONCLUSIONS

Sample of Ni-Zn spinel ferrite with x=0.7 was successfully prepared by sol gel auto combustion method with relatively low cost. The fine particle nature of the synthesized powder results from the by sol gel auto combustion method. The XRD peaks are broader as compared to ceramic sample, which indicates nanoparticle nature. A change in the structural parameter is observed compared with the ceramic samples and in agreement with literature value. The lattice constant was found in agreement with literature.

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BIOGRAPHIES (Not Essential)

	Raje Shaikh B.B.:M.Sc. Physics from Pune University in 1992. Working as Assistant Professor and Head Dept. of Physics Govt. College of Arts and Science, Aurangabad from last 25 years. Published 4 research papers at National and international level. Also working as reviever for International journal of nano dimension, Iran. My research work is related with thin film and currently on ferrite material.
	Chaitali Vilasrao More: I have completed my masters in Physics in 2015 from Dr. Babasaheb Ambedkar Marathwada University, Aurangabad. I am pursing Ph.D. in Nuclear Physics. I have 6 International peer reviewed journal papers and 8 International and National conference papers to my credit. I am being trained in research activities and have the ability of understanding challenging research problems. I also have sound knowledge of computer applications in research.
	M. Sc. in Physics in 2004 and Ph. D. in Physics from Dr. Babasaheb Ambedkar Marathwada University in 2010. I am currently working as assistant professor Maharashtra Institute of Technology. My research interests include Nano materials, Use of ICT in education and Materials Science. I have authored two books on Physics, published 23 international journal papers, published 4 International conference papers, and presented 8 national conference papers.
	Dr Syed Qadeeruddin Chishty :MSc physics 1990 and PhD in physics 2003 from Dr BAM university Aurangabad M.S. India working as Associate professor and head dept of physics Dr. Rafiq Zakaria College for women Aurangabad from last 27 years. Also working as research guide in physics.published 22 research papers at national and international level .Also working as reviewer for Elsevier journal Fractal and chaos .My research work is related with material science and fractals and chaos .Also published Nine Books at international level