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Synthesis and Characterization of Ni $Sm_xFe_{2-x}O_4$ Nano-Ferrites by auto combustion method

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ABSTRACT
Ni-Sm Nano ferrites with chemical formula $NiSm_xFe_{2-x}o_4$ (x=0.00, 0.010, 0.030, 0.050, 0.070, 0.090 and 0.10) were synthesized by the citrate-gel auto combustion method. Synthesized powders are sintered at 500^0C for four hours in air and characterized by XRD, SEM, EDS and FTIR. X-ray diffraction (XRD) analysis showed cubic spinel structure of the ferrites and the values of lattice parameter (a) and X-ray density (d_x) increases with the increase of Sm content. Scanning electron microscope (SEM) studies revealed morphology of the Nano crystalline samples. An elemental composition of the sample was studied by Energy Dispersive spectroscopy (EDS). The FTIR spectra shows the two significant absorption bands in the wave numbers range of 370-600cm^{-1}arising due to the inter-atomic vibrations in the tetrahedral and octahedral coordination compounds The observed results can be explained on the basis of composition

Keywords: Ni-Sm Nano ferrites, citrate-gel auto combustion Technique, XRD, SEM, FTIR.

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I. INTRODUCTION

Spinal ferrite of nano particle chemical formula ($NiSm_xFe_{2-x}O_4$) enjoys in a significant role in microwave applications such as microwave devices, microwave control components, biosensors, isolators. (1) Among different ferrites, nickel samarium ferrite plays a special attention because of its vast applications in high technological importance micro wave device such as electrical devices. [2, 3]. The structural properties of ferrites strongly depending on their stoichiometric ratio of chemical composition.

Nano ferrites with samarium substitution of nano ferrites synthesized by using and available method citrate gel auto combustion. Substitution of large rare earth metal of samarium change the structure and morphology of images will result in strain which induce structural distribution and there by modify the properties of samples in Nano-region
Various methods are used for synthesizing nano sized spinel ferrites such as sol-emulsion-gel technique, electro deposition, plasma enhanced vapour decomposition, hydrothermal, and reversemicelle [4, 5].

II. EXPERIMENTAL DETAILS

SamariumsubstitutedNickel nano ferrites having the chemical formula $NiSm_xFe_{2-x}O_4$ were synthesized by low temperature citrate gel auto combustion method using the following below raw materials

2.1 Raw Materials

- Nickel nitrate 99%pure(AR Grade)[$Ni(NO_3)_2$]
- Samarium nitrate 99.9% sigma Aldrich [$Sm(NO_3)_3$] $6H_2O$
- Ferric nitrate –99.9%pure(AR Grade) ($Fe(NO_3)_3$ $9H_2O$
- Citric acid –99%pure(AR Grad) ($C_6H_8O_7.H_2O$)
- Ammonia Solution -99%pure(AR Grade)(NH_3)

2.2 The synthesis nano ferrites

The detailed procedure for the preparation a set of ferrites $NiSm_xFe2_{-x}O_4$ (where x =0.00, 0.010, 0.030, 0.050, 0.070, 0.090 and 0.10) with step of increment prepared by citrate gel auto combustion method. The required amount of metal. Nitrates and citric acid are taken so as to have a molar ratio of 1:3 and dissolved in 200 ml of distilled water. Metal nitrates are weighed according to stoichiometric ratios. a required amount of ammonia is added drop by drop into the solution in order to adjust the pH value at 7since base solution are employed in order to speed up the reaction. these solution is mixed with continuous stirring and heated up to 100 ^{o}c for 10-12hours then viscous gel is formed this gel dried at 200 ^{o}c which self-ignites to form a sol this is then form a brown coloured product. This is a desired sample. prepared samples were sintered at 500 ^{o}c 4 h these powders characterization of XRD SEM FTIR AND EDS further studies.

2.3 Structural characterization by XRD

The structural characterization was done by X-Ray Diffract meter were recorded using Regakumaniplex powder X-ray diffractometer (Cu-

Kα**λ**=1.5406A°)with diffracted mono chromatic beam with radiation of wave length (1.5405A^0) the diffraction pattern of Ni-Sm between brag Angles 10^o to 80^o in the steps of 0.04^o/sec. nano ferrites particles Were shown in fig.

The crystalline size was calculated for the sample using the high intensity 311 peak and using DebayScherrer formula **[7]** while taking into account the intensity broadening [8].

The Crystalline size of the sample $D = \frac{0.91\lambda}{\beta \cos\theta}$ [6].

Where λ the wavelength of X-ray is used [7]. βis the width of diffraction peak i, e. full width half Maximum (fwhm) ,θ is the peak position .

Lattice parameter (a) of the sample was calculated by the formula

$\boldsymbol{a = d * \sqrt{h^2 + k^2 + l^2}}$[8].

Where a is Lattice constant, (hkl) are the Miller indices, d is the inter planner space

The X-ray density of the prepared sample was calculated using the relation

$d_X = \frac{nM}{a^3 N}$g/cm^3 [9]

Where n is the number of molecules in a unit cell of spinel lattice(n=8), M= molecular weight of the sample , a is the lattice parameter and N is the Avogadro number.

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The experimental density of the prepared samples was calculated by Archimedes's principle with xylene media using following relation.

$$d_E = \frac{\text{Weight of the sample in air}}{\text{Weight of the sample in air} - \text{weight of the sample in xylene}} \times \text{Density of xylene}$$

The Percentage of Porosity P of the ferrite sample was then determined by employing the relation

$$P = \frac{d_X - d_E}{d_X} \times 100$$

Where d_x is the X-ray density &$d_E$is the experimental density,

III. Result and Discussion

3.1. XRD Analysis:

A phase analysis using X-ray diffraction technique was performed to confirm the formation of single-phase cubic spinel structure as shown in Fig. (1) .With no extra lines corresponding to any other crystallographic phase. The results obtained from XRD pattern for all the samples of $NiSm_xFe_{2-x}O4$ with the (hkl) values corresponding to the diffraction peaks of different planes (220), (311), (400), (422), (511), and (440) are spinel cubic phaseThe calculated values of crystalline size for the different compositions are given in the table (1)It can be seen from the table that the values of the crystal size varies from 28.53nm to 39.45nm of the various Sm concentrations

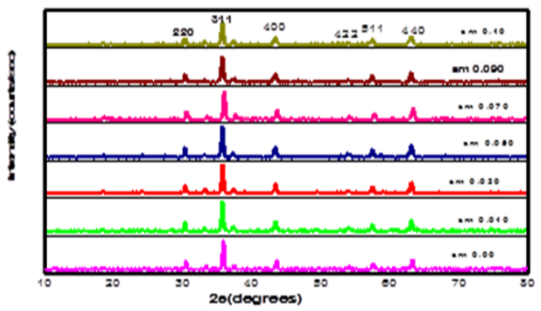


Fig.1 XRD pattern of Ni-Sm Nano ferrites

The lattice parameter values of all the composition of Samarium doped Iron ferrites have been calculated from the d- spacing and are given in the above table.

A plot is drawn between the lattice parameter vs Samarium composition is shown in below Fig.(2)

Table 1: crystalline size (D), lattice parameter (a), X-ray density (d_x), expert density (d_E), porosity (p)

Ferrite Composition	D [nm]	a (A°)	d_x (gm/c)	d_E (gm/c)	p (%)	ϑ_1(cm^{-1})	ϑ_2(cm^{-1})
NiFe2O4	39.45	8.307	5.432	5.273	2.92	574	385
$NiSm_{0.010}Fe_{1.99}O_4$	33.37	8.338	5.393	5.347	0.85	580	397
$NiSm_{0.030}Fe_{1.97}O_4$	30.72	8.061	6.162	5.338	13.37	580	389
$NiSm_{0.050}Fe_{1.95}O_4$	30.74	8.329	5.679	5.360	5.61	576	395
$NiSm_{0.070}Fe_{1.93}O_4$	31.65	8.281	5.638	5.381	4.55	574	397
$NiSm_{0.090}Fe_{1.91}O^4$	28.53	8.458	5.332	5.320	0.22	580	389
$NiSm_{0.10}Fe_{1.9}O_4$	29.53	8.334	5.596	5.389	3.69	580	387

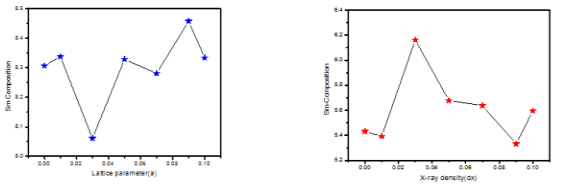


Fig.2 variation of lattice constant with Sm+3 content and **Fig. (3)** shows the X-ray density (dx) vsSm Concentration.

Fig. (3) variation of lattice constant with Sm^{+3} content in $NiSm_xFe_{2-x}O_4$is (X=0.00 to 0.10 with a step of 0.01)is observed that the Lattice parameter increases with increase in Sm content. This is attributed to replacement of smaller ionic radii Ni (0.78A°) by larger ionic radii Sm^{+3} (A°) ions. This linear variation indicates that the Ni-Sm ferrite system obeys Vegard's law [8].

The Fig. (2, 3) shows the X-ray density (dx) vs concentration, The X-ray density (dx) is depend on the lattice parameter and molecular weight of the sample. From the table one can observe that molecular weight of the sample is increases with Samarium concentration and lattice parameter is also increases with the increase of Sm compositions. This may due to the grater atomic weight of Sm 150.36gm/mol and lesser atomic weight of Fe-55.845gm/mol(8) shown in figure[4, 5]. Samarium was very small .the calculated values of the lattice constant tabulated in table:1 show that there has been no structural distortion for the various ratios. The fact that samarium goes as substitution into the cubic spinel lattice evident from the absence of any extra peaks in the XRD spectrum.

3.2. Morphology by SEM

Studied microscope (prepared samples by Citrate- Gel Auto combustion method was using scanning electron SEM) where the secondary electron images were taken at different magnifications to study the synthesized samples were shown in Fig.(4).

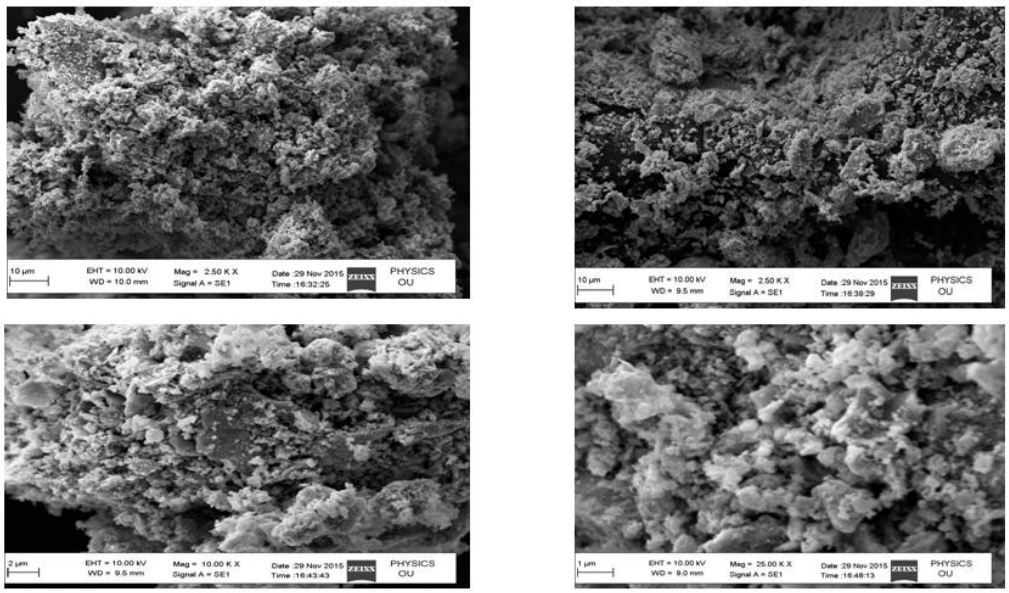


Fig. 4. SEM micrographs of Ni-Sm ferrites samples at x=0.030 0.050 0.070 0.090

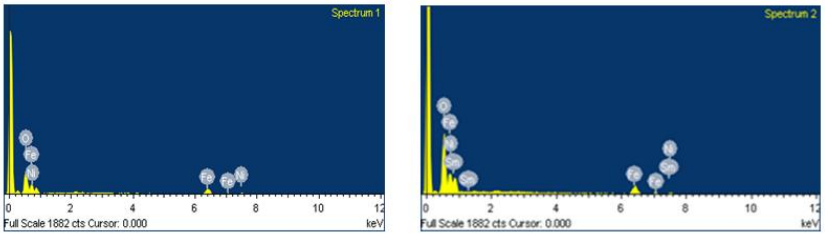
The sem images of Ni-Sm ferrite are shown in the Fig the images show that the particles have an almost homogeneous distribution, Shows the presence of a monophasic microstructure homogeneous microstructure with an average agglomeration [10] and some of the samples are agglomerated form .it evidenced by SEM images that the agglomeration of particles lies in Nano region. The particles were observed as uniform grain (in different SEM images) sizes [9].

3.3. Elemental Analysis by EDS

The EDS spectra give information about the chemical composition of the elements present from the surface to the interior of the solids, and they are used to confirm the homogeneity of the investigated samples.The spectra indicated the presence of O, Fe,

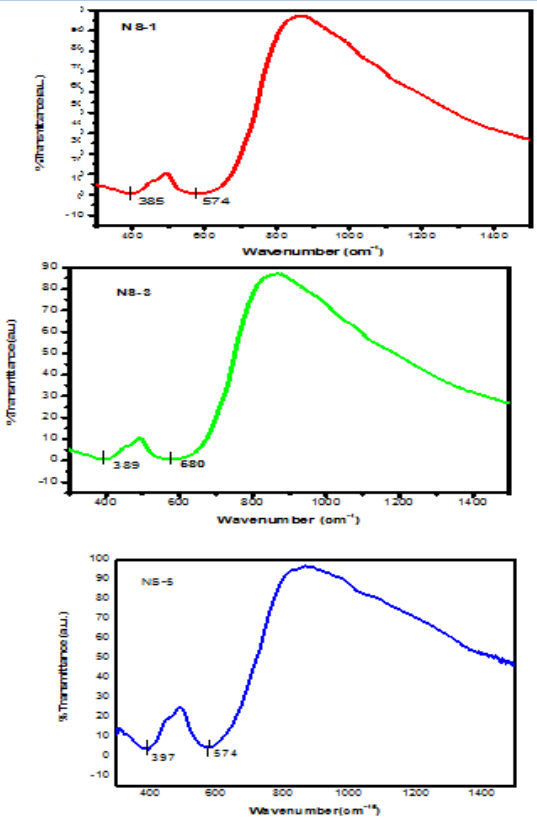
Ni, and Sm as the major elements in the synthesized material with no impurities are observed along with elemental mapping. The EDX spectra of the samples are shown in Fig. (5).

Fig. 5. EDS micro graphs of Ni-Sm nano ferrites



IV. FTIR SPECTRAL ANALYSIS

FTIRspectra of $NiSm_xFe_{2-x}O_4$ nano ferrites of prepared samples two absorption bands are obtained in the range of 387-397cm^{-1} and 574-580cm^{-1} corresponds to the stretching vibrations of the metal at the site and octahedral site it explain that the normal mode of vibration of tetrahedral cluster is higher than that of octahedral cluster. [10, 11] It is attributed to the shorter bond length of tetrahedral cluster and longer bond length of octahedral cluster. Shown in below Fig (6).



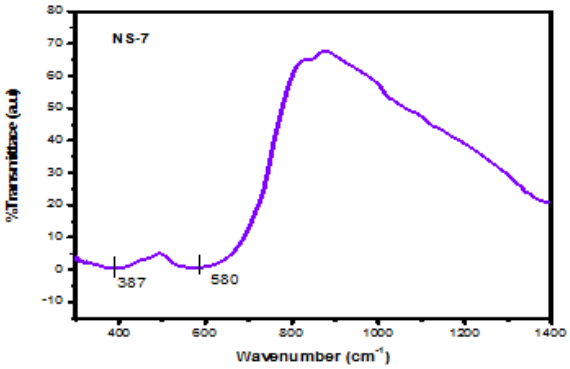


Fig. 6 shows FTIR spectra of the ferrite samples

V. CONCLUSION

- Citrate Gel Auto Combustion technique is a convenient way for obtaining a homogeneous nano sized mixed ferrites, it is very simple and economical method where no specific heating or cooling rate is required .
- X-ray diffraction pattern confirms the formation of single phase cubic spinel structure. from standard data JCPDS.
- The crystalline size of the various Ni-Sm ferrites was in the range of 28 to 39 nm.
  The Lattice parameter has increased with the increase of Sm concentration in Ni-Sm ferrites which indicate that the mixed Ni-Sm ferrite system obey the Vegard's law
- SEM micrographs of various compositions indicate the morphology of particles is similar. They reveal largely agglomerated.
- EDS data give the elemental % and atomic % in the mixed Ni-Sm ferrites and it shows the presence of(Ni, Sm, Fe and O) without impurities and which indicates the completeness of solid state reaction
- The FTIR spectra of the ferrite samples in the range of wave numbers from 387 to 580 cm^{-1}.

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