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Structural and Dielectric Studies of Samarium Substituted Cobalt Nano Ferrites

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Abstract
The Samarium Substituted Cobalt Nano Ferrites With Chemical Formula $Cosm_xfe_{2-X}O_4$ (Where X=0.0, 0.010, 0.030, 0.050, 0.070, 0.090 And 0.10) Were Synthesized By Citrate-Gel Auto Combustion Method. Synthesized Powders Were Sintered At 500^0C For Four Hours In Air And Characterized By XRD, SEM, EDS, FTIR. X-Ray Diffraction (XRD) Analysis Showed Cubic Spinel Structure Of The Ferrites And The Values Of Lattice Constant (A) And X-Ray Density (D_x) Increased With The Increase Of Sm Content. The Surface Morphology Of The Samples Was Observed By Scanning Electron Microscopy (SEM). An Elemental Composition Of The Sample Has Studied By Energy Dispersive Spectroscopy (EDS). The Observed Results Can Be Explained On The Basis Of Composition. The Dielectric Parameters Like Real Part Of Dielectric Constant(E´), Imaginary Part Dielectric Constant (E″) Of The Samples Were Studied As A Function Of Frequency In The Range Of 50Hz To5mhz At Room Temperature Using Agilent E4980 Were Studied. The Observed Result Can Be Explained On The Basis Of Conduction Mechanism.

Keywords: *Co-Sm Nano Ferrites; Citrate-Gel Auto Combustion Technique; XRD; SEM; EDS; FTIR.*

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I. INTRODUCTION

Among All The Spinel Type Structures. Cobalt Samarium Nano Ferrites Have Interested The Researches Much Due To Its Commercial Importance In Industrial Electronic Applications. This Importance For This Materials Because The Increasing Use Of Electronic And Wireless Systems, Those Are Personal Communication Devices, Satellite Communication Systems, Radios, Sensors, Tvs, Etc. In The Problem Of Leakage In The Electromagnetic Radiation From Such Devices Is The All-Time Need. Also, The Problem Of Leakage In The Electromagnetic Interference Becomes More Serious, Which May Cause The Misoperation Of Precise Electronic Equipment And The Leak In Secret Information Communication [1]. Co-Smnanoferrites Deal With Special Features Such As Low Dielectric Loss, Good Strength And Toughness, Etc.[2].

II. EXPERIMENTAL DETAILS

The Cobalt Samarium Nano Ferrites Having The Chemical Formula $Cosm_xfe_{2-X}O_4$ Were Synthesized By Citrate Gel Auto Combustion Method Using The Raw Materials Are .Samarium Nitrate ($Smno_3$), Cobalt Nitrate ($Cono_3$), Ferric Nitrate ($Fe(NO_3)_3$ $9H_2O$), Citric Acid ($C_6H_8O_7.H_2O$) And Ammonia Solution (NH_3). Thesynthesis Of Co-Smnano Ferrites: The Chemicals Are Weighed According To The Stoichiometric Proportion; The Calculated Quantities Of Metal Nitrates Were Dissolved In Minimum Amount Of Distilled Water To Get Clear Homogeneous Solution. An Aqueous Solution Of Citric Acid Is Used As A Fuel Because; Among All Other Fuels Citric Acid Has Better Complexing Ability. The Metal Nitrate To Citric Acid Ratio Was Maintained As 1:3 For All The Samples, And Nitrate-Citrate Solution Is Obtained To That Ammonia (NH_3) Solutions Are Added Drop By Drop To Maintain PH-7.The Solution Is Mixed And Heated By Continuous Stirring Up To100^oc For 10-12 Hours, Then Viscous Gel Is Formed, Again Water In The Mixture Is Evaporated Forming A Dry Gel Generates Internal Combustion And Forms A Brown Colored Product. Which Is A Desired Sample, Collected Ferrite Powder Is Subjected To Calcinations At 500^0 C For 4hrs These Powder Are For Characterized By XRD, SEM, FTIR And EDS.

The Structural Characterization Was Done By X-Ray Diffract Meter Bruker D8 Advanced System With Diffracted Monochromatic Beam With Cu Kα Radiation Of Wavelength ($1.5405A^0$). The Diffraction Pattern Of Co-Sm Between The Bragg Angles 10º To 80º In The Steps Of 0.04º/Sec.

The Crystalline Size Was Calculated For The Sample Using The High Intensity 311 Peak And Using Debayscherrer Formula [3] While Taking Into Account The Intensity Broadening

The Crystalline Size Of The Sample $D = \frac{0.91\lambda}{\beta \cos\theta}$ (1)

Where λ The Wavelength Of X-Ray Is Used. β Is The Width Of Diffraction Peak I, E. Full Width Half Maximum (FWHM), θ Is The Peak Position.

Lattice Parameter (A) [4].Of The Sample Was Calculated By The Formula

$$a = d * \sqrt{h^2 + k^2 + l^2} \quad (2)$$

Where A Is Lattice Constant, (Hkl) Are The Miller Indices, D Is The Inter Planner Space

The X-Ray Density [5] Of The Prepared Sample Was Calculated Using The Relation

$$d_X = \frac{nM}{a^3 N} \text{G/Cm}^3 \quad (3)$$

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.

The Distance Between The Magnetic Ions (Hoping Length) On A-Site (Tetrahedral) And B Site (Octahedral) Is Calculated According To The Following Relations

$D_a = 0.25a\sqrt{3}$ And $D_b = 0.25a\sqrt{2}$ [6] (4)

The Drift Mobility (M_d) Of The Charge Carrier In The Synthesized Samples Is Calculated Using The Fallowing Relation

III. RESULT AND DISCUSSION

3.1. XRD Analysis

A Phase Analysis Using X-Ray Diffraction Technique Was Performed To Conform The Formation Of Single-Phase Cubic Spinel Structure As Shown In Figure (1) With No Extra Lines Corresponding To Any Other Crystallographic Phase. The Results Obtained From XRD Pattern For All The Samples Of $Cosm_xfe_{2-X}O_4$ With The (Hkl) Values Corresponding To The Diffraction Peaks Of Different Planes (220), (311), (400), (422), (511), And (440) Are Spinel Cubic Phase

The 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 11nm To 17nm Of The Various Sm Concentrations

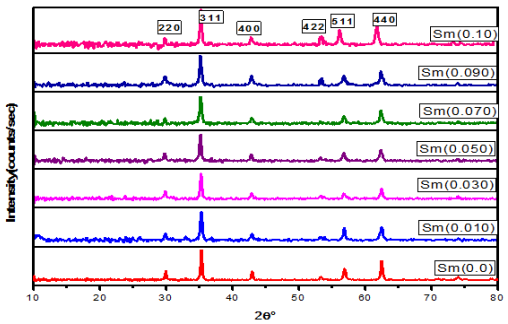

Fig.1. XRD Pattern Of $Cosm_xfe_{2-X}O_4$ Nano Crystalline Ferrite

Table1. Crystalline Size (D), Lattice Parameter (A), X-Ray Density (D_x), Expert Density (D_e), Happing Length A, B- Site.

Ferrite composition	D[nm]	a [A⁰]	d_X [gm/cc]	d_E [gm/cc]	A-site(d_A)	B-site(d_B)
$CoFe_2O_4$	23.78	8.419	5.222	5.151	3.6454	2.9761
$CoSm_{0.010}Fe_{1.99}O_4$	21.95	8.428	5.227	5.165	3.6493	2.9793
$CoSm_{0.030}Fe_{1.97}O_4$	14.63	8.431	5.263	5.198	3.6506	2.9803
$CoSm_{0.050}Fe_{1.95}O_4$	26.75	8.441	5.286	5.191	3.6549	2.9838
$CoSm_{0.070}Fe_{1.93}O_4$	21.94	8.449	5.313	5.255	3.6584	2.9867
$CoSm_{0.090}Fe_{1.91}O_4$	21.95	8.453	5.347	5.271	3.6601	2.9881
$CoSm_{0.10}Fe_{1.9}O_4$	17.55	8.458	5.358	5.298	3.6623	2.9899

The Lattice Parameter Values Of All The Composition Of Samarium Doped Cobalt Nano Ferrites Have Been Calculated From The D- Spacing And Are Given In The Table (1). A Plot Is Drown Between The Lattice Parameter Vs Samarium Composition Is Shown In Fig. (2).

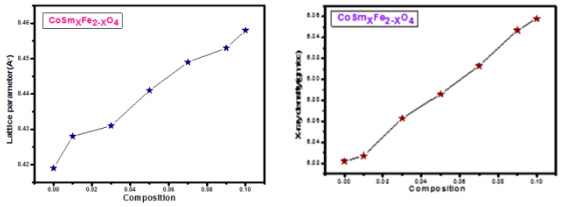

Fig. 2. Lattice Parameter Vs Sm Composition
Fig. 3. X-Ray Density Vs Sm Composition

The Variation Of Lattice Constant With Sm^{3+} Content In $Cosm_xfe_{2-X}o_4$ (Where X=0.0, 0.010, 0.030, 0.050, 0.070 0.090 And 0.10) Is Observed That The Lattice Parameter Increases With Increase In Smcontent. This Is Attributed To Replacement Of Smaller Ionic Radii Fe (0.69A°) By Larger Ionic Radii Sm^{3+} (1.098A°) Ions. This Linear Variation Indicates That The Co-Sm Ferrite System Obeys Vegard's Law [7].

From Fig (3) Shows The X-Ray Density (Dx) Vs Sm Concentration, The X-Ray Density (Dx) Is Depend On The Lattice Parameter And Molecular Weight Of The Sample. From The Table (1) We 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/Mo)[8] The Sample.

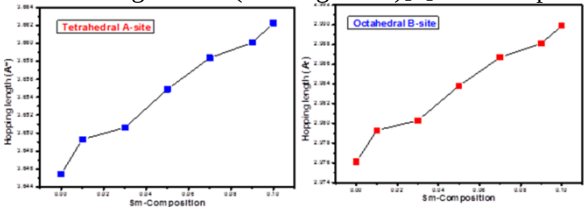

Fig. 4. Tetrahedral A-Site And Octahedral B-Site Of $Cosm_xfe_{2-X}O_4$

3.2. Morphology By SEM

Morphology Of Co-Sm Prepared Samples Was Done By Using Scanning Electron Microscope

(SEM) As The Instrument Ziess Special Edition-18 Where The Secondary Electron Images Were Taken At Different Magnifications To Study The Synthesized Samples Were Shown In Fig (5)

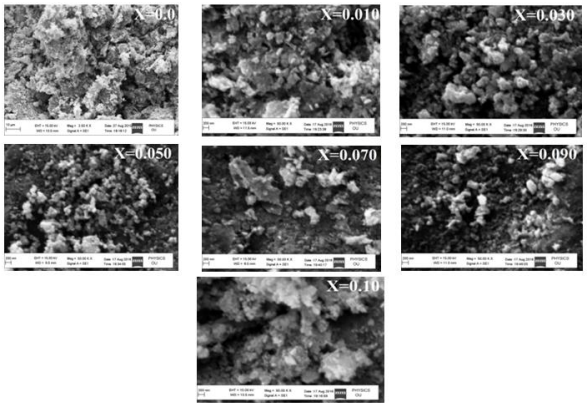

Fig. 5. SEM Micrographs Of $Cosm_xfe_{2-X}O_4$ferrites Samples At (X=0.0, 0.010, 0.030, 0.050, 0.070, 0.090 And 0.10)

The Sem Images Show That The Particles Have An Almost Homogeneous Distribution, And Some Of The Samples Are Agglomerated Form. It Is Evidenced By SEM All The Samples Exhibit A Very Low Porosity And Large Grain Size (52nm-150nm) That The Agglomeration Of Particles Lies In Nano Region. The Particles Were Observed As Uniform Grain (In Different SEM Images) Sizes, The Increasing Sm Content Particle Size Will Be Decreases From 0.010 To 0.050, And Again Increase Sm Content Particle Size Will Increases Upto 0.10.

3.3. Elemental Analysis By EDS

By Using The EDS Spectra We Observe The Chemical Composition Of Prepared Samples Of The Elements Present From The Surface To The Internal Of The Solids, And They Are Used To Confirm The Homogeneity Of The Investigated Samples. The Spectra Indicated The Presence Of Elements (O, Fe, Co, And Sm) Without Impurities And Which Indicates The Completeness Of Solid State Reaction. The Edsmicro Graphs Of The Samples Are Shown In Fig (6)

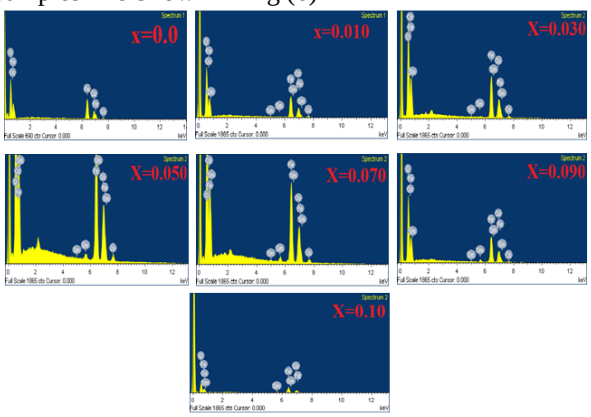

Fig .6. EDS Micro Graphs Of $Cosm_xfe_{2-X}O_4$nano Ferrites

3. 4. Dielectric Properties

The Study Of The Dielectric Properties Give Valuable Information About The Behaviour Of Electric Charge Carriers Which Leads To Good Result And Explanation Of Conduction Mechanism In Ferrites. The Dielectric Behaviour Is One Of The Most Important Study Of Ferrites Which Is Depend On The Preparation Condition, For Example Sintering Temperature And Time, Type And Quantity Of Additives. The Real Part Dielectric Constants (E´), Imaginary Part Of Dielectric Constant (E″) Results Are Due To The Heterogeneous Structure Of The Ferrite Material. The Frequency Dependence Of Dielectric Constant For The Sample Is Shown In Fig. 8 Then It Can Be Seen That All The Samples Showed Similar Trend In Dielectric Behaviour. I.E. Dielectric Constant Decreased Initially At Room Temperature With Increase In Frequency From (50Hz To 5mhz) And Reached At Constant Value At Higher Frequency. Then After A Certain Increase In Frequency All The Samples Exhibit A Frequency- Independent Behaviour. This Can Be Explained On The Basis Of Maxwell-Wagner Interfacial Type Polarization, Which Is In Agreement With Koop's Theory [9.10].

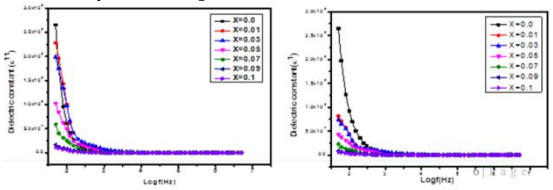

Fig. 7. Variation Of Real Part Of Dielectric Constant (E´) Imaginary Part Of Dielectric Constant (E″) With Frequency

IV. 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 Cubic Spinel Structure In Single Phase With Standard Data From JCPDS. The Crystalline Size Of The Various Co-Sm Ferrites Was In The Range Of 14 To26 Nm. The Lattice Parameter Has Increased With The Increase Of Sm Concentration In Co-Sm, Ferrites Which Indicate That The Mixed Co-Sm Ferrite System Obay 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 Co-Sm Ferrites And It Shows The Presence Of (Co, Sm, Fe And O) Without Impurities And Which Indicates The Completeness Of Solid State Reaction. Dielectric

Constant Decreased Initially At Room Temperature With Increase In Frequency From (50Hz To 5mhz) And Reached At Constant Value At Higher Frequency. Then After A Certain Increase In Frequency All The Samples Exhibit A Frequency-Independent Behavior.

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