Synthesis and Characterization of nanocrystalline Ni-Co-Zn ferrite by Sol-gel Auto-Combustion method.

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Abstract:

The structural properties of nanocrystalline $NiFe_2O_4$ (NF), $CoFe_2O_4$ (CF) and $ZnFe_2O_4$ (ZF) ferrites were synthesized by Sol-gel auto combustion method. The powders were sintering at Normal temperature, 400°c and 700°c for 2hrs to densify properly. The samples were characterized by X-RD, SEM and FT-IR. The X-RD used to analyze phase structure and lattice parameters. The FT-IR spectra confirmed that synthesis material is ferrite. Morphology of ferrite powders were investigated by using SEM. Porosity of synthesis ferrite is measured.

Key Words: Nano crystalline, Structural Properties, Sol-gel Auto Combustion method, X-RD, FT-IR Spectra, SEM.

1. Introduction

Ferrite of the type $NiFe_2O_4$ (NF), Co Fe_2O_4 (CF) and $ZnFe_2O_4$ (ZF) with the spinal structure are magnetic ceramics of great importance in the production of electronic and magnetic components. The electric and magnetic properties of ferrite are depends on various parameters such as processing conditions, sintering temperature and time as well as on their chemical composition [1]. Several attempts have been made by researchers to deposited ferrite films by a variety of techniques including alternative sputtering technology [2], Pulse-Laser deposition [3], and spin-spray plating [4]. However most of them cannot be economically applied on a large scale because they required high vacuum system, complicated experimental steps and high reaction temperatures. In this study, chemical synthesis route called sol-gel auto combustion method has been applied synthesize NF, CF and ZF [1-4]. This method is useful to achieve the fabrication of magnetic nano ferrites at low annealing temperature. In recent years very few studied based on different composition of Ni Co and Zn have been carried out. The size and morphology of nano particles and their properties may be controlled by modifying the composition of the nano composites and by thermal treatment conditions [5]. Due to the small size of the nano crystals, an important part of the atoms are located at the surface this is the reason why the sol-gel synthesis method gone on intensive development [6]. Up to this stage the research work on Ni CO Zn Ferrite is very limited. The sol-gel method was used for synthesis of this nano ferrite material. This method involves hydrolysis [7].

In this paper we present a study on the synthesis of $Ni_{0.6}CO_{0.2}Zn_{0.2}Fe_2O_4$, $Ni_{0.4}CO_{0.2}Zn_{0.4}Fe_2O_4$ and $Ni_{0.2}CO_{0.2}Zn_{0.6}Fe_2O_4$ nano composites of different composition. The composition, crystal structure, morphology, and size distribution of Ni, Co, Zn-Ferrite nano crystals can be controlled by adjusting the synthesis route and molar ratio of materials in the initial mixtures. The synthesized nano crystals have been characterized by X-RD, SEM, and FT-IR, presented below are the details of investigation.

2. Experimental:

The $Ni_{0.6}CO_{0.2}Zn_{0.2}Fe_2O_4$, $Ni_{0.4}CO_{0.2}Zn_{0.4}Fe_2O_4$ and $Ni_{0.2}CO_{0.2}Zn_{0.6}Fe_2O_4$ ferrite powders were prepared by co-precipitation and hydro thermal technique using iron nitrate, zinc nitrate, cobalt nitrate, and nickel nitrate as reaction agent. To obtain $Ni_{0.6}CO_{0.2}Zn_{0.2}Fe_2O_4$, $Ni_{0.4}CO_{0.2}Zn_{0.4}Fe_2O_4$ and $Ni_{0.2}CO_{0.2}Zn_{0.6}Fe_2O_4$ ferrite powders we have mixed AR grade iron nitrate, zinc nitrate, cobalt nitrate, and nickel nitrate with double distilled water. Citric acid was used as a chelating agent because it plays an important role in homogeneous mixture formation of metal cations. Reaction procedure was carried out in air atmosphere at room temperature. The composition was well shake and pH of solution is maintained as 7 by adding ammonia. The prepared solution was stirred on magnetic stirrer at low temperature 80^0C to form a gel. The prepared ferrite samples were annealed for 400^0C and 700^0C. The general chemical reaction involves in synthetic process can be written as

$$(x\text{-}0.8)Ni(NO_3)_2.6H_2O+(0.2)Co(NO_3)_2.6H_2O+ (x)Zn(NO_3)_2.6H_2O+2Fe(NO_3)_3.9H_2O+3C_6H_8O_7 \rightarrow Ni_{x-0.8}CO_{0.2}Zn_x\ Fe_2O_4+4N_2\uparrow+18CO_2\uparrow+12H_2O.$$

The synthesized nano crystalline samples were characterized by X-Ray Diffraction techniques at room temperature by using Philips powder X-Ray Diffractometer (model PW3710) with CuKα radiations having wavelength 1.5406 $A^{0.}$ The morphological behavior of the investigated samples was determined by using Scanning Electron Microscopy (SEM) techniques (model HITACHI Japan). Fourier Transform Infrared (FT-IR) spectra were recorded in the range of 4000-400 cm^{-1} at room temperature by using Brukar Spectrometry.

3.Result and discussion:

3.1 X-Ray Diffraction.

To identify the possible formation of phase in Ni Co Zn Ferrite an X-RD analysis was done. The most intense peaks in all the specimens were found to match well with spherical spinel ferrite (JCPD). Lattice parameters and crystalline sizes of sintered ferrites specimens, evaluated by X-RD analysis are shown in table (1) along with their composition, density, crystalline size, Porosity. There was a minor increase in lattice parameter which may be due to increasing concentration of Ni and Zn. But lattice parameter increases with in increasing annealing temperature. Decreasing densification may be due to the evolution of excess Ni, and Zn in the composition for Fe at Room Temp., 400^0C and 700^0C respectively. Decreasing in density may be due to vary with concentration of Ni and Zn. Increasing porosity is depends on increasing temperature. The X-RD patterns are shown as in fig 1(a), (b), and (c).

Table No. 1

Constant	Temperature ^{0}C	Lattice Constant (a) A^0	Inter planer Dist (d)nm	Density Dx. gm/cc^3	Porosity (%P)
I] Ni Co(0.2) Zn	Room	6.76	38.00	$10.15x10^6$	20.4
	400	6.09	49.30	$13.85x10^6$	22.35
	700	6.75	27.72	$10.15x10^6$	24.49
II] Ni Co(0.2) Zn	Room	5.45	31.7	$19.46x10^6$	03.60
	400	7.35	44.88	$7.93x10^6$	05.26
	700	8.62	57.61	$4.91x10^6$	09.00
III] Ni Co(0.2) Zn	Room	6.09	23.49	$13.86x10^6$	27.45
	400	6.75	28.63	$10.15x10^6$	29.14
	700	6.76	30.62	$10.153x10^6$	31.99

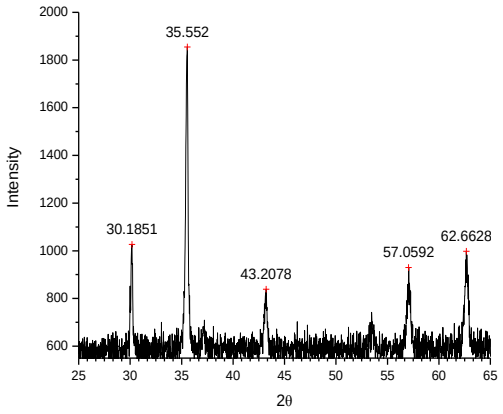

Fig-1(a) X-RD Pattern of Sintered $Ni_{0.6}CO_{0.2}Zn_{0.2}Fe_2O_4$ Ferrites. Of peaks (210,211,311,411,421.)

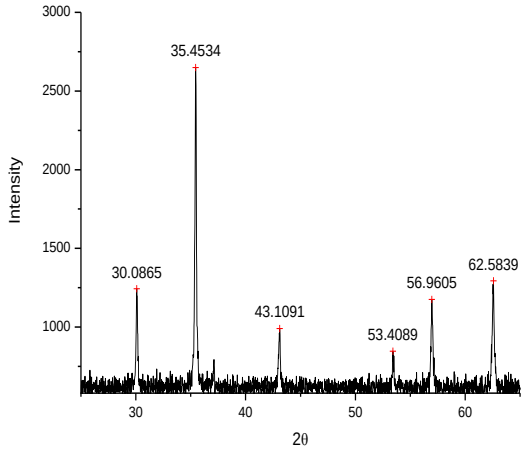

Fig-1(b) X-RD Pattern of Sintered $Ni_{0.4}CO_{0.2}Zn_{0.4}Fe_2O_4$ Ferrites of peaks (300,222,410,510,520,530)

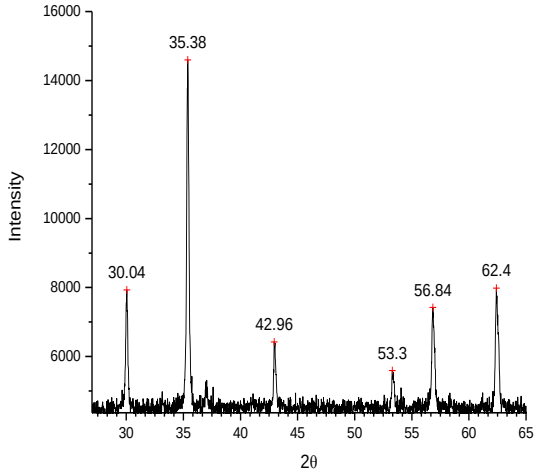

Fig-1(c) X-RD Pattern of Sintered $Ni_{0.2}CO_{0.2}Zn_{0.6}Fe_2O_4$ Ferrites of peaks (200,211,220,222,321,410)

3.2 FT-IR Spectra:

In the present study $Ni_{0.6}CO_{0.2}Zn_{0.2}Fe_2O_4$, $Ni_{0.4}CO_{0.2}Zn_{0.4}Fe_2O_4$ and $Ni_{0.2}CO_{0.2}Zn_{0.6}Fe_2O_4$ ferrite has been synthesized at room temperature, 400^0C, and 700^0C.The synthesis process is carried out using sol-gel auto combustion method. The citric acid was used a reducing agent in reaction. The of $Ni_{0.6}CO_{0.2}Zn_{0.2}Fe_2O_4$ IR curve fig2(a) of sintered powder shows strong absorption band 1598.699Cm^{-1} to 1345.10Cm^{-1} indicates N-H Bending structure, the strong absorption band at 2342.11Cm^{-1} indicating C triple bond N- Stretched. The band at 724.13 Cm^{-1} indicating C-H out of plane bending carbohydrates which is very weak and shifted to low frequency.

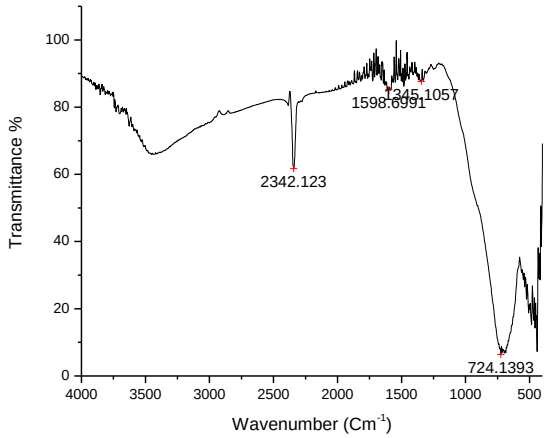

Fig-2(a) FT-IR Pattern of Sintered $Ni_{0.6}CO_{0.2}Zn_{0.2}Fe_2O_4$ Ferrites.

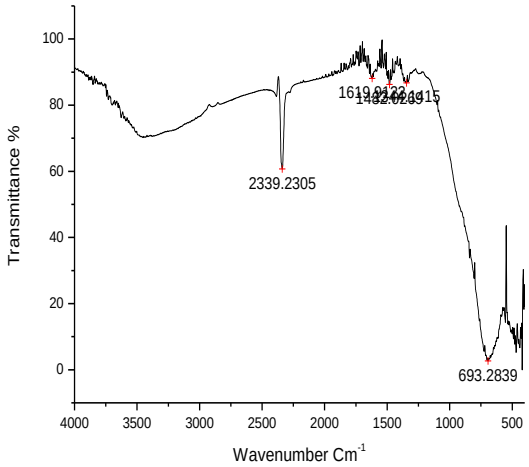

Fig-2(b) FT-IR Pattern of Sintered $Ni_{0.4}CO_{0.2}Zn_{0.4}Fe_2O_4$ Ferrites.

The of $Ni_{0.4}CO_{0.2}Zn_{0.4}Fe_2O_4$ IR curve fig2(b) of sintered powder shows strong absorption band 1619.91Cm^{-1} to 1344.14Cm^{-1} indicates N-H Bending

structure, the strong absorption band at 2339.23Cm^{-1} indicating C triple bond N- Stretched. The band at 693.28 Cm^{-1} indicating C-H out of plane bending carbohydrates which is very weak and shifted to low frequency.

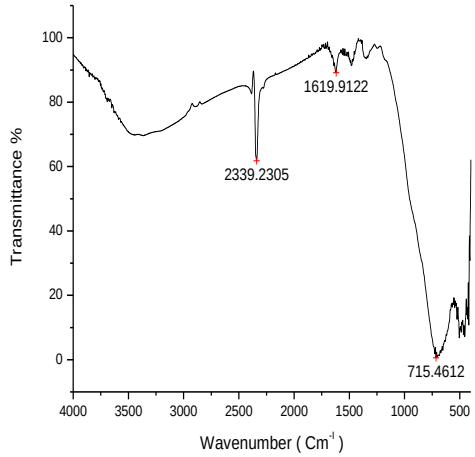

Fig-2(c) FT-IR Pattern of Sintered $Ni_{0.2}CO_{0.2}Zn_{0.6}Fe_2O_4$ Ferrites

The of $Ni_{0.2}CO_{0.2}Zn_{0.6}Fe_2O_4$ IR curve fig2(c) of sintered powder shows strong absorption band 2339.23Cm^{-1} to 1619.91Cm^{-1} indicates N-H Bending structure, the strong absorption band at 2341.15Cm^{-1} indicating C triple bond N- Stretched. The band at 715.46 Cm^{-1} indicating C-H out of plane bending carbohydrates which is very weak and shifted to low frequency.

3.3 SEM Morphology:

Performing SEM we analyzed the structure of $Ni_{0.6}CO_{0.2}Zn_{0.2}Fe_2O_4$, $Ni_{0.4}CO_{0.2}Zn_{0.4}Fe_2O_4$ and $Ni_{0.2}CO_{0.2}Zn_{0.6}Fe_2O_4$ shows typical morphology in fig3(a,b,c). For samples synthesized by sol-gel method the surface has compact structure with smallest particle size typically less than 100 nm. The micrograph of samples sintered at 400^{0}C and 700^{0}C indicating that microstructure is completely form these temperature. The grain size increases with increase in temperature.

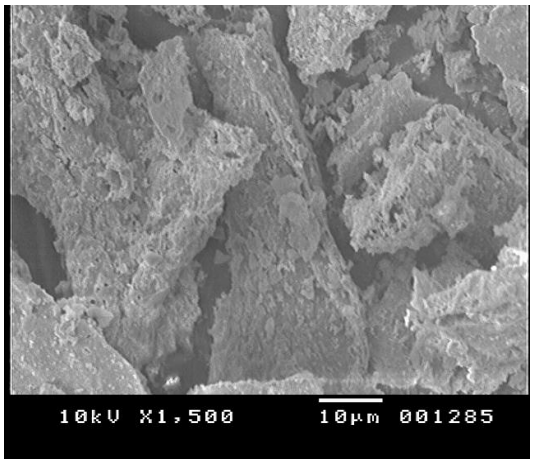

Fig-3(a)SEM Morphology of Sintered $Ni_{0.6}CO_{0.2}Zn_{0.2}Fe_2O_4$ Ferrites.

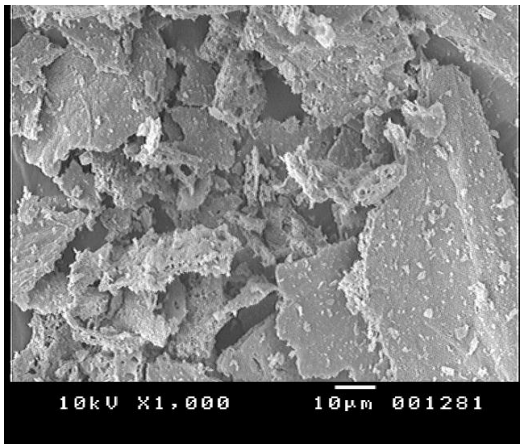

Fig-3(b) SEM Morphology of Sintered $Ni_{0.4}CO_{0.2}Zn_{0.4}Fe_2O_4$ Ferrites.

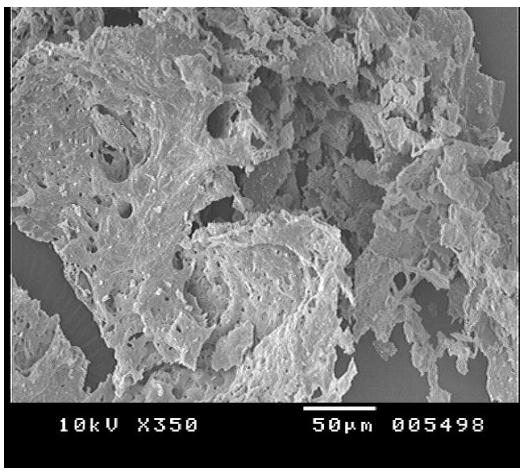

Fig-3(c) SEM Morphology of Sintered $Ni_{0.2}CO_{0.2}Zn_{0.6}Fe_2O_4$ Ferrites

4. Conclusion:

The NiCoZn Spinel ferrite nano particles were successfully synthesized by sol-gel auto combustion method. By varying the concentration of $NiFe_2O_4$ (NF), and $ZnFe_2O_4$ (ZF) at constant Co Fe_2O_4 (CF), there is increase in Lattice Constant, Inter planer distance and Porosity and decrease in Density, at increasing temperature. The FT-IR investigation shows strong absorption of Ni and Zn ions. The nano crystalline natures confirm from SEM and X-RD. SEM shows spherical structure. X-RD pattern confirm the formation of spherical spinel phase. The lattice parameters, Porosity and Density of ferrite materials are changes for different concentration and different temperature.

5. References:

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