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Sol-Gel Synthesis and Characterization of Zinc Substituted Cobalt Ferrite Magnetic Nanoparticles

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***Abstract:* - Synthesis and characterization of soft $Co_{1-x}Zn_xFe_2O_4$ magnetic nanoparticles have been synthesized using sol-gel method. The prepared nanoparticles were characterized by using Fourier transform infrared spectroscopy(FTIR),X-ray diffraction(XRD), Thermo gravimetric-Differential Thermal Analysis(TG-DTA),Scanning electron microscopy (SEM),Energy Dispersive x-ray spectrum(EDX) and vibrating sample magnetometer(VSM).Using XRD,it is confirmed that the samples were cubic structure in nature and the mean crystalline size were decreases 16nm to 11nm respectively.The morphology and the quantitative analysis of the prepared analysis of the prepared particles were studied by using SEM and EDX spectrum. The FTIR was used to study the presence of functional groups.Finally,the magnetic properties of the powders have been studied at room temperature from the hysteresis loop measurements using a vibrating sample magnetometer(VSM).From this analysis, the values of the saturation magnetization increase and the coercive field of $Co_{1-x}Zn_xFe_2O_4$ nanoparticles were found to decrease with increasing degree of Zn substitution.**

Keywords: Nanoparticles, Magnetic properties, vibrating sample magnetometer,XRD,FT-IR,Solgel.

1. INTRODUCTION

Ferrites nanoparticles are of great interest because of their scientific aspect and various applications[1].A number of chemical routes have been used for the synthesis of ferrite nanoparticles.These methods includes sol-gel, micro emulsion, chemical co-precipitation etc.,Among these methods sol-gel method is widely used for the synthesis of nanoparticles of ferrite[2].The combination of magnetic and electrical properties makes ferrite useful in many technological application[3].Ferrites are ferromagnetic oxides consisting of ferric oxide and metal oxides[4].Metal-oxide nanoparticles are of interest because of their unique optical, electronic and magnetic properties[5].Cobalt ferrite($CoFe_2O_4$)nanopowders have high permeability, good saturation magnetization, and no preferred direction of magnetization.[6].Cobalt ferrite is the most important and abundant magnetic materials that have large magnetic anisotropy, moderate saturation magnetization, remarkable chemical stability and mechanical hardness, which make it good candidate for the recording media[7].Cobalt ferrite is a well known hard magnetic material with a high coercivity and a moderate magnetization[8].Zn substituted cobalt ferrite nanoparticles were prepared by sol-gel method[9].The nanoferrites are interesting materials owing to their wide range of applications in modern science and technology[10].Magnetic and structural properties of ($CoFe_2O_4$) magnetic materials prepared by the sol-gel method will be presented in this report[11].

2. EXPERIMENTAL DETAIL:

Materials

All the reagents used for the synthesis of cobalt ferrite nanoparticles were analytical grade and used as received, without further purification. The chemicals used are Zinc nitrate [$Zn(NO_3)_2.6H_2O$],Cobalt nitrate [$Co(NO_3)_2.6H_2O$],Ferric nitrate [$Fe(NO_3)_3.9H_2O$],ethylene glycol [$C_2H_6O_2$] and oxalic acid [$C_2H_2O_4$].

Synthesis of $Co_{1-x}Zn_xFe_2O_4$ magnetic nanoparticles:

The process for synthesizing at room temperature was carried out as follows: In a typical synthesis, Cobalt nitrate [$Co(NO_3)_2.6H_2O$],Zinc nitrate [$Zn(NO_3)_2.6H_2O$],Ferric nitrate [$Fe(NO_3)_3.9H_2O$],and oxalic acid [$C_2H_2O_4$].mixing in ethanol under constant magnetic stirring for 20 minutes. Then the gelling agent ethylene glycol is added to the solution. The final solution is magnetically stirred for 2 hours and then surplus water is removed by using a vacuum rotary evaporator at 80̊ C until the gel is obtained. Then the gel is dried in hot air oven at 110̊ C.Then, the gel was dried and grinded into powders. After that, the powder was annealed at 800°C for 4 hrs in furnace under air atmosphere. Finally, magnetic nanoparticles in different size were synthesized, as shown in the below flow chart.

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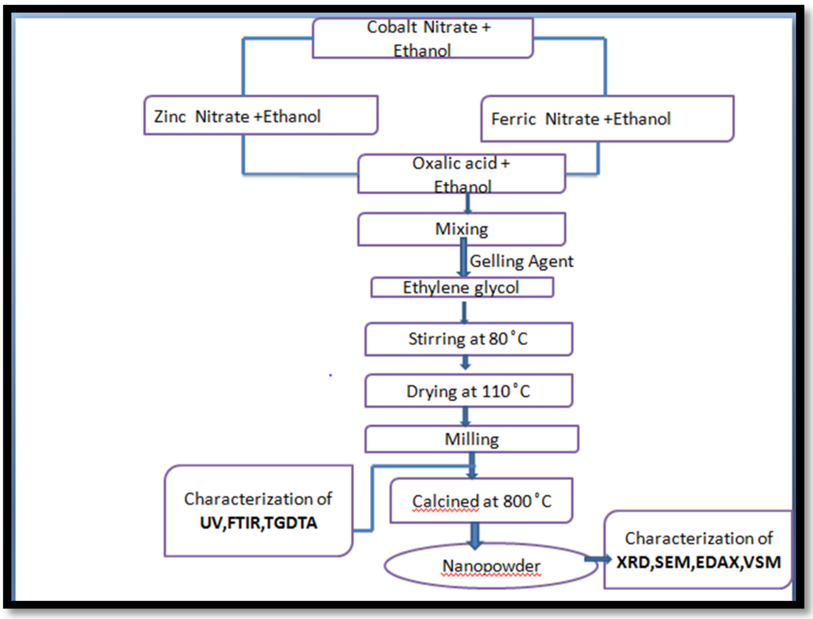

Figure (1) Flowchart for the Sol-Gel method to prepare $Co_{1-x}Zn_xFe_2O_4$ Magnetic Nanoparticles

3. RESULT AND DISCUSSION:

A.FT-IR:

Figure (2) using KBR pellets the Fourier Transform Infrared Spectra (FTIR) of the pure $Co_{1-x}Zn_xFe_2O_4$ and doped powder was recorded range 400^{-1}cm to $4000cm^{-1}$.$Co_{1-x}Zn_xFe_2O_4$ IR curve figure shows strong absorption band $1619.912Cm^{-1}$ to $2339.23Cm^{-1}$ indicates N-H Bending structure, the strong absorption band at $2341.15Cm^{-1}$ indicating C triple bond N- Stretched. The band at 1715.46 Cm^{-1} indicating C-H out of plane bending carbohydrates which is very weak and shifted to low frequency.

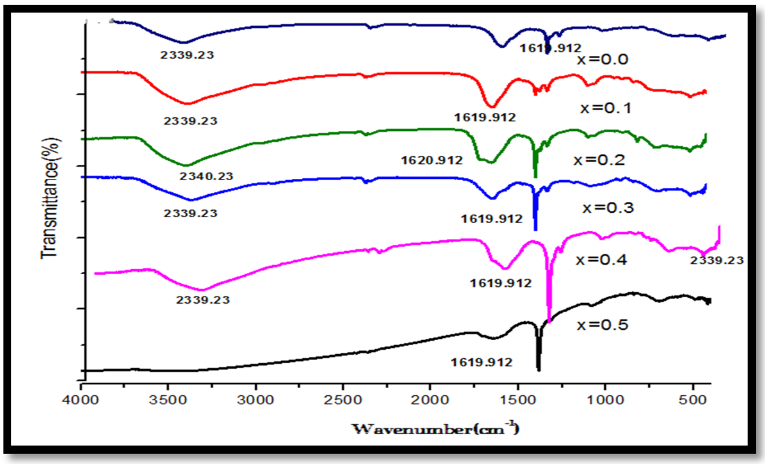

Fig (2) FTIR spectra of $Co_{1-x}Zn_xFe_2O_4$ Precursors with (x= 0, 0.1,0.2,0.3,0.4,0.5).

B. XRD:

Figure (3) shows the XRD Patterns of Zinc substituted cobalt ferrite nano powders calcined at 800˚C temperature. The synthesized material structure corresponds with the cubic structure of $Co_{1-x}Zn_xFe_2O_4$ [JCPDS card No: 89-4307].The crystalline peaks(111),(200),(220)and(002) indexed as cubic $Co_{1-x}Zn_xFe_2O_4$.The crystallite size of the nanoparticles is calculated by Debye-Scherer formula:

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

Where K is a constant taken as 0.9.λ is the wavelength of the X-ray radiation.β is the Full Width Half Maximum (FWHM) of each phase and Θ is the diffraction range. The mean crystallite sizes of the $Co_{1-x}Zn_xFe_2O_4$ nanoparticles synthesized at 800°C are 16nm and 11 nm respectively.

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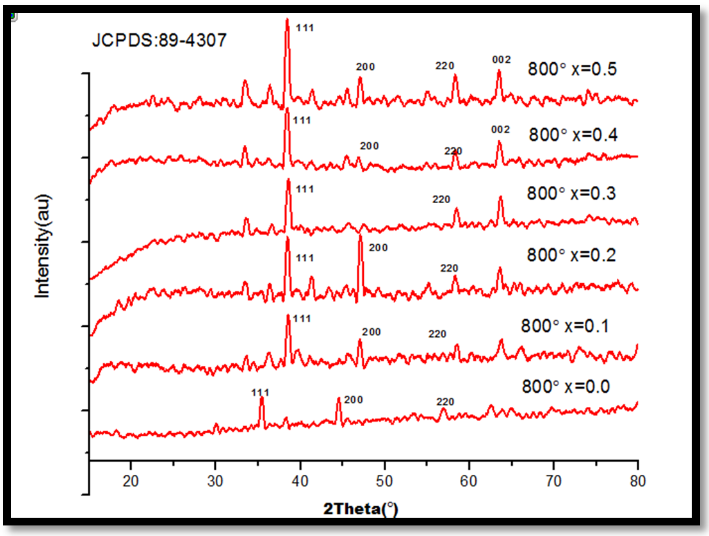


Fig(3).X-ray diffraction pattern of $Co_{1-x}Zn_xFe_2O_4$ at 800°C annealing temperature with (x=0,0.1,0.2,0.3,0.4,0.5).

C.TGA-DTA:

The TGA-DTA has been taken of the sample to determine the temperature range for growth of these systems Thermo Gravimetric Analysis Differential Thermal Analysis and in the temperature range 0˚C-1200˚C was performed**.** Figure shows the TGA-DTA graph of $Co_{1-x}Zn_xFe_2O_4$.It may be noted that the transformation from precursor powder to final phase is accompanied 400˚C. The conversation process starts at around 100̊ C and finally get converged into the well-grown ferrite particles at a temperature 400̊ C.The thermo gravimetric analysis goes with the DTA curve as well where we see a significant endothermic peak. This gives an indication that the ferrite formation get completed at a temperature around 400̊ C.It shows the phase formation at 400̊ C.

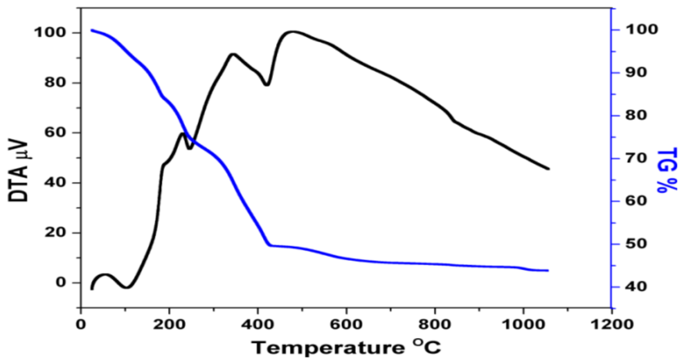
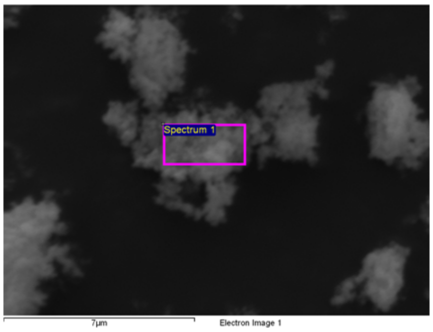
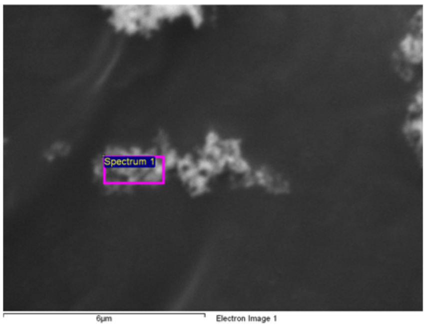


Fig (4) TGA-DTA spectra of $Co_{1-x}Zn_xFe_2O_4$ Precursors with (x= 0)

D.SEM:

Figure (5) shows the surface morphology of cobalt ferrite powder prepared by changing the molar ratio of cobalt ferrite.The typical spherical morphology is found for the cobalt ferrites calcined at 800°C for 4 hrs.Morphology of the prepared samples was studied using Scanning electron microscope(SEM).Where the secondary electron images were taken at different magnification to study the morphology.Figure(5) represents the scanning electron micrographs for typical(x=0,0.1,0.2,0.3,0.4,0.5)sample. Scanning electron micrographs indicates the formation of nano-sized grains of the $Co_{1-x}Zn_xFe_2O_4$ ferrite powder.





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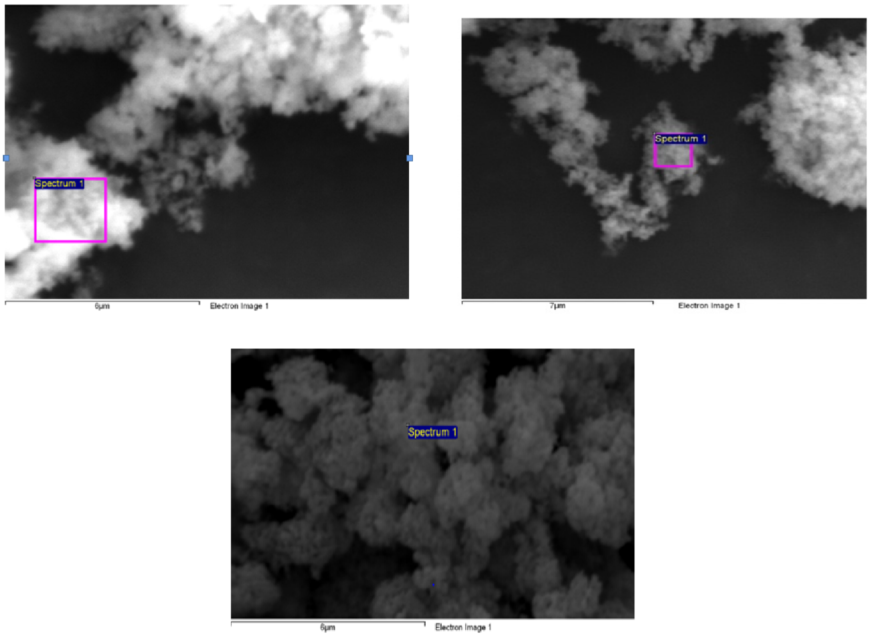
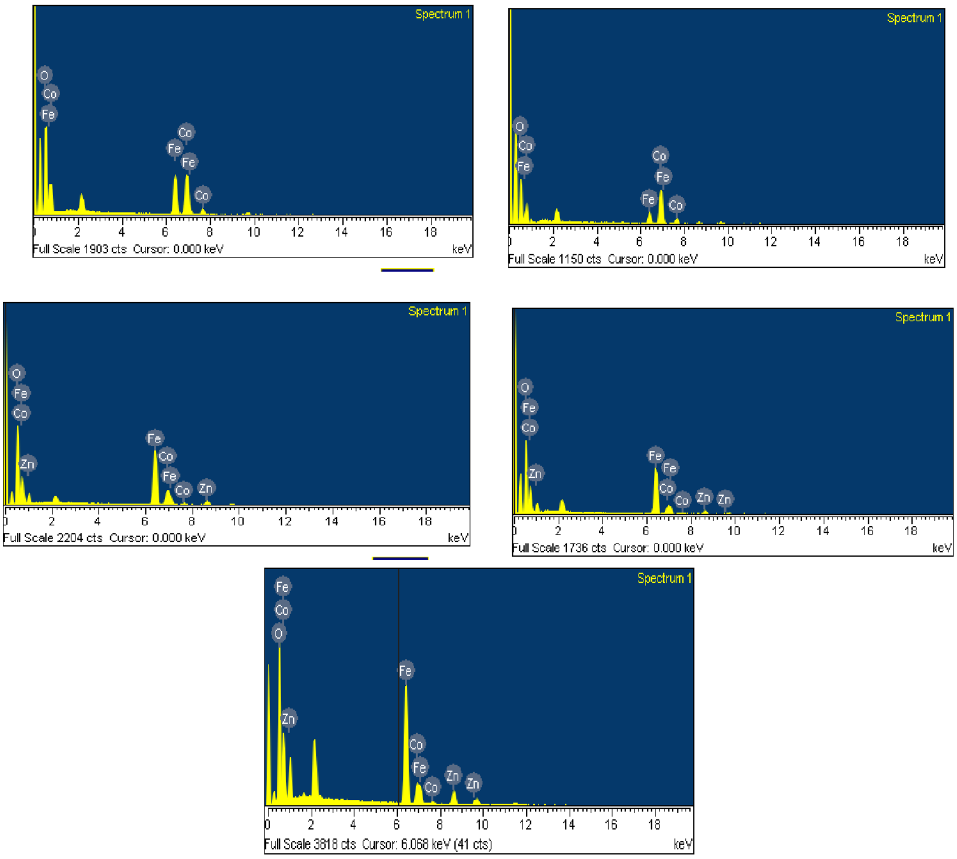


Fig(5) SEM images of the $Co_{1-x}Zn_xFe_2O_4$ at 800°C annealing temperature. with (x=0,0.1,0.2,0.3,0.4,0.5).

E.EDAX:

Figure(6) shows the Energy dispersive X-ray spectroscopic (EDS)analysis shows that there are elements Co,Fe,Zn,and O in the sample Figure(6),and the atom ratio of Co;Fe;Zn;O is Which is close to that of $Co_{1-x}Zn_xFe_2O_4$ formula.All the above analyses confirm that the synthesized sample is $Co_{1-x}Zn_xFe_2O_4$ without any impurities.



Fig(6) EDAX spectra of the $Co_{1-x}Zn_xFe_2O_4$ at 800°C annealing temperature with (X=0,0.1,0.2,0.3,0.4,0.5)

F.Magnetic study:

Magnetic characterization of the samples was performed by VSM at room temperature with a maximum applied field of ±10kOe. Figure(7) shows the room temperature hysteresis loops of samples(0,0.1,0.2,0.3,0.4,0.5) It can be observed that both formulations reveal typical ferromagnetic behavior. The ferromagnetic behavior of the prepared nanocrystals is clearly shown by coercivity (Hc), saturation magnetization (Ms) and remanence magnetization (Mr). The saturation magnetization is the maximum induced magnetic moment that can be obtained in a magnetic field, beyond this field no further increase in magnetization occurs. High saturation magnetization magnetic materials are required for further high-frequency inductors [31].

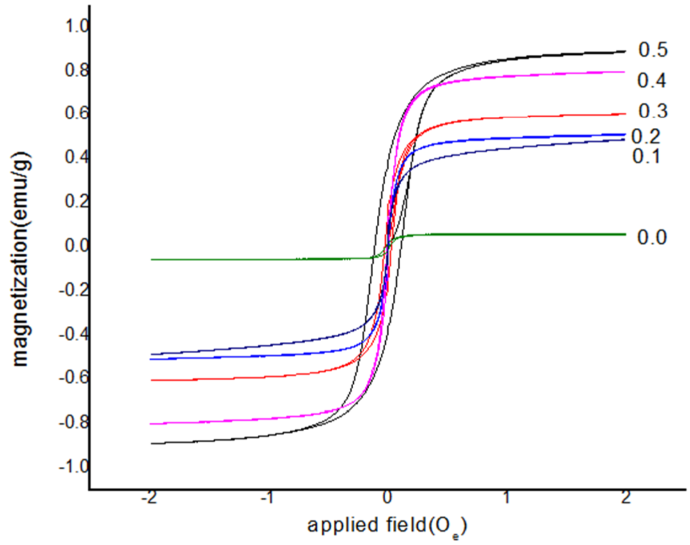


Figure (7) Room temperature hysteresis loops of $Co_{1-x}Zn_xFe_2O_4$ at 800°C annealing temperature

4. CONCLUSIONS

The magnetic zinc substituted cobalt ferrite nano powder was successfully synthesized by sol-gel Method. The functional group analyzed using FTIR. The zinc substituted cobalt ferrite nanoparticle phase formation was confirmed by x-ray diffraction pattern. The particle size from 16nm to 11nm.The morphology of the prepared samples was studied using scanning electron microscope(SEM).The EDS shows the presence of Co,Fe,Zn,and O.The magnetic properties are measured by vibrating sample magnetometer(VSM).

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