

Effect of Fe³⁺ Ions on Structural and Magnetic Properties of Co-Precipitated Strontium Cobalt Chromite

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Abstract – Pure and Fe doped nano Strontium-Cobalt Chromites were synthesized by Co-precipitation method with pH as 10. Effect of doping Fe (x= 0.0, 0.5, 1.5, 2.0) on structural and magnetic properties are reported in the present work. All samples are sintered at 1173 and characterized. X-ray Diffraction (XRD) used to reveal phase and structural parameters. Morphology has been obtained from Scanning Electron Microscope. Compositions of the synthesized samples are scaled with Energy Dispersive X-ray Spectroscopy (EDX). FTIR is equipped to record vibration of nanoferrite sites and two prominent absorption bands ν_1 and ν_2 corresponding to the stretching vibration of tetrahedral and octahedral sites around 600 cm⁻¹ and 400 cm⁻¹. The magnetic parameter such as Saturation magnetization (M_s), Remanent magnetization (M_r), Coercive field (H_c) and Squareness ratio are determined by Vibrational Sample Magnetometer (VSM). The average crystallite size calculated from XRD pattern and it ranging between 41.7 and 57.8 nanometers. Maximum Anisotropy constant obtained at x= 1.5.

Index Terms – Sr_{0.5}Co_{0.5}Fe_xCr_{2-x}O₄, Co-Precipitation method, Magnetic Properties.

1. INTRODUCTION

Spinel ferrites are huge fundamental and technological important due to their structural, electronic, magnetic and catalytic properties [1-3]. Among the pigment classes, one of the most important is the spinel group AB₂O₄ due to its capacity of accommodating different cations combination, leading to a variety of colors and tonalities. It has been used as magnetic fluids, microwave absorber and medical diagnostics [4], magneto optical devices [5]. The physio-chemical properties of the ferrites are strongly dependent on the sites and the nature of the catalyst [6,7] which are closely related to the method of preparation. Normal spinel can be described as A²⁺[B(Fe²⁺)]O₄ divalent atom occupy at tetrahedral sites and trivalent occupy at octahedral site and inverse ferrite as A(Fe³⁺)[B(M²⁺, Fe³⁺)]O₄ trivalent atom occupy tetrahedral site and both divalent and trivalent occupy octahedral site. [8].

The structural and magnetic properties of this type magnetic material depend on the purity, size and morphology of the precursor powder [9]. Thus, obtaining fine, high chemical homogeneity and monodispersed particles is the most

important process in material manufacturing. Recently fine particles of spinel ferrites synthesized by wet-chemical methods were shown to have markedly different properties from those prepared by the ceramic method [10]. Several chemical processing techniques are available for the synthesis of ferrites [11-15]. The selection of an appropriate synthetic procedure often depends on the desired properties and the final applications. Among these methods, Chemical co-precipitation was selected as a best method to synthesize nanoparticles. It is least expensive simplest approach for making nanoparticles, it produces large quantities (order of grams) in relatively short interval of time [16].

In recent years, several researchers studied the effect of non-magnetic Sr²⁺ substitution on structural, optical and electromagnetic properties of different ferrites Sr_{0.2}Mn_{0.8}Fe₂O₄ [17], Co_{1-x}Sr_xFe₂O₄ [18-20], Zn_{1-x}Sr_xFe₂O₄ [21]. To the best of our knowledge, no research works have been available on the literature related to Sr_{0.5}Co_{0.5}Fe_xCr_{2-x}O₄. In present work, we have studied the structural and magnetic properties of Sr_{0.5}Co_{0.5}Fe_xCr_{2-x}O₄ (x= 0, 0.5, 1.5, 2.0) prepared by Co-precipitation method.

2. MATERIALS AND METHODS

2.1 Material for production

Magnetically potential Fe doped Strontium Cobalt nano chromite at various concentration of Fe ion has been prepared from the starting materials CoCl₂.4H₂O, SrCl₂.6H₂O, CrCl₃.6H₂O, FeCl₃.6H₂O and NaOH. Analytical grade of these precursors are purchased from SIGMA ALDRICH, Germany with 98% purity.

2.2 Synthesis Methodology

A profitable synthesizing methodology involves correct choice of precursor, its composition and reaction environment. Particularly for wet chemical methods like sol-gel, hydrothermal, co-precipitation and colloid emulsion technique, pH controller plays an important role. For the present work eco-friendly NaOH is used to maintain pH. The physio-chemical properties of nanoparticles are greatly influenced by particle

size, morphology, purity and chemical composition. Chemical method has been conformed to efficiently control the morphology and chemical composition of prepared nano powder. Among wet chemical techniques sol-gel, hydro thermal and colloid emulsions are time consuming and involve highly unstable alkoxides and difficult to maintain reaction conditions. Co-precipitation is one of the more successful techniques for synthesizing ultrafine nanoparticles having narrow particle size distribution [14]. These advantages on Co-precipitation method motivated authors to synthesize $\text{Sr}_{0.5}\text{Co}_{0.5}\text{Fe}_x\text{Cr}_{2-x}\text{O}_4$ ($x = 0, 0.5, 1.5, 2.0$) nano ferrites by Co-precipitation method. The precursors for Fe ion are taken as 2 M and 1M for other Metals chlorides. They are mixed in stoichiometric ratio and added one by one on the basis of their electronegativity value. Mixture of Aqueous solution is stirred rigorously at 338K for 30 minutes, meanwhile NaOH is added to the brain solution by drop by drop using a burette till solution reaches pH value 10. The required composition of nano ferrites are formed from conversion of metal salt into hydroxide and then transformed into ferrites. The precipitates obtained were thoroughly washed more than three times with double distilled water and acetone. The final product was dried and sintered at 1173 K for the formation of spinel ferrite crystal structure.

2.3 Physical measurements

Crystal structure of all the samples were examined by powder X-Ray diffraction XRD patterns at room temperature PANalytical-X'Pert PRO powder diffractometer using $\text{Cu-K}\alpha_1$ radiation. Scanning Electron Microscopy (SEM) study was performed by VEGA 3 TESCAN Scanning Electron Microscope, operated at 120 KV. Elemental analysis has been done with BRUKER EDS. Fourier Transform Infrared (FT-IR) spectra were recorded on SHIMADZ FT-IR spectrophotometer using KBr pellet method in the range $4000\text{-}400\text{ cm}^{-1}$. The magnetic properties were measured at room temperature by LAKESHORE vibrating sample magnetometer (VSM).

3. RESULT AND DISCUSSION

3.1 X-Ray Diffraction analysis

Fig.1 shows x-ray diffraction pattern of Fe doped Cobalt strontium nanochromite for the concentration $x = 0, 0.5, 1.5$ and 2.0 . Major peaks (220), (311), (400), (511) and (440) are present in XRD pattern of all the samples which reveals these are having the disordered spinel ferrite formation of space group $\text{Fd}\bar{3}m$. In addition to major peaks, minor reflections planes (111) and (422) are present. These matches with the JCPDS file CoCr_2O_4 No 780711 [22] CoFe_2O_4 ICSD No 22-1086 [23], SrFe_2O_4 ICDD# 00-001-1027 [24]. (111) is present in $x=0$ and (422) present in $x=0$ and $x=2.0$. This disappearance of reflection peaks is due to conversion of Fe^{2+} into Fe^{3+} induced by more number of Co^{2+} and Sr^{2+} ions [25,26]. When increasing Fe content impurity peaks gets

decrease and in $X=1.5, X=2.0$ there is no impurity peak is present.

Generally, intensity of XRD peaks corresponds to the crystalline nature of the samples. For $x=2.0$, the intensity of XRD peaks is higher than all other samples. Thus, higher value of Fe results in more crystalline in nature. Intensity of XRD pattern related to particle size and crystallinity of the samples. The intensities of (220) and (440) planes are more sensitive to cations in tetrahedral and octahedral sites respectively [25,26]. From TABLE 1 it is clear that intensity of (220) and (440) varies non-linearly with increase in Fe^{2+} ion, after adding Fe into the SrCoCr (220) and (440) value gets increase this indicates Fe will occupy and replace Chromium ions on both tetrahedral and octahedral sites. At $x=2.0$ the values get decreased this is due to absence of Chromium in this ratio. This happens because of the presence of Fe^{2+} ion in octahedral site formed due to reduction of Fe^{3+} ion to Fe^{2+} ion for higher Fe concentration and leads to migration of Fe^{3+} ion from octahedral to tetrahedral site.

Average crystallite size 'D' and lattice constant has been estimated from X-ray reflections indexed (111), (220), (311), (222), (400), (422), (511), (440) and (620), using Scherer's equation $D = 0.9 \lambda / \beta \cos \theta$, where D is the average crystallite size, β is the full width half maxima, λ is the X-Ray wavelength and θ is the Bragg's angle. Lattice constant has been calculated from equation $a = d (h^2 + k^2 + l^2)^{1/2}$ Where 'a' is lattice constant, d be the inter planar distance, hkl is miller indices. Lattice strain were determined using the Williamson-Hall formula $\epsilon = \beta / 4 \tan \theta$, where ϵ is the lattice strain of the structure. X-ray Density can be calculated by $\rho_x = ZM/\text{Na}^3$, Where Z is number of molecules per unit cell, here it is 8. M is Molecular weight of the sample, N is Avogadro's Number, 'a' lattice constant. Dislocation density has been found by using the relation $\delta = 15 \epsilon / a D$, here δ be the dislocation density [27,28]. All these structural parameters are calculated and tabulated in TABLE 2.

The average crystallite size 'D' estimated for $\text{Sr}_{0.5}\text{Co}_{0.5}\text{Fe}_x\text{Cr}_{2-x}\text{O}_4$ nanoferrites for different 'x' values lie in between 41 nm and 58 nm. The samples having lattice constant between 8.2676 Å and 8.3596 Å. Even though addition of equal ionic radius Fe^{2+} ion ($r_{\text{Fe}} = 0.64 \text{ \AA}$) and ($r_{\text{Cr}} = 0.64 \text{ \AA}$) in Strontium cobalt chromites variation in the lattice constant value is occur. In crystallite size, initially the value gets increase further it decrease. When the particle size reduced the lattice gets more strain and dislocated ions number also gets increased. When the particle size reduced to nano dimension there is change in cation distribution Co^{2+} occupies both tetrahedral and octahedral sites. Molecular weight of the $\text{Co}_{0.5}\text{Sr}_{0.5}\text{Fe}_x\text{Cr}_{2-x}\text{O}_4$ composition increases with replacement of higher atomic mass Fe^{3+} (55.85 a.m.u) by lower atomic mass Cr^{3+} (51.996 a.m.u) in the composition. X-Ray density values varies with respect to lattice constant and atomic mass of the ions, here $x=0.5$ and 1.5

the values gets decrease due to variation in lattice constant and number of Fe content in these ratios.

3.2 Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopic (EDS) analysis

The morphological characteristics of the obtained $\text{Sr}_{0.5}\text{Co}_{0.5}\text{Fe}_x\text{Cr}_{2-x}\text{O}_4$ nanoferrites sintered at 1173 K have been investigated with the help of VEGA 3 TESCAN for all concentration of the samples. Fig.2 shows morphology of $\text{Sr}_{0.5}\text{Co}_{0.5}\text{Fe}_x\text{Cr}_{2-x}\text{O}_4$ nano ferrite samples for the increase in concentration of Fe ($x = 0, 0.5, 1.5, 2.0$). The micrographs show the agglomerated grain structure with increasing iron the agglomeration gets reduced. Agglomeration is due to increase in magnetic interaction among the particles influenced by Fe^{3+} [29]. Thus, results of SEM well in agreement with X-Ray diffraction pattern. Especially for $x = 1.5$ and 2.0 agglomeration are less, this matches with disappearance of superstructure reflection planes due to amorphization. The surface of the ferrite samples has a number of fine pores or voids that are attributed to the large amount of Oxygen and chlorine gas liberated during the sintering process. Presence of vacancies results in contraction of Lattice even higher ionic radius dopant is added to the sample [30].

EDS spectrum for the $\text{Sr}_{0.5}\text{Co}_{0.5}\text{Fe}_x\text{Cr}_{2-x}\text{O}_4$ ($x = 0, 0.5, 1.5, 2.0$). nanoferrites are recorded with BRUKER EDS and illustrated in Fig.3. The result shows each peak corresponds to the element added in the prepared nanoferrite which confirmed the presence of elements in respective concentration. Chromium, Iron and Oxygen are the major constituents in the composition, Strontium and Cobalt are the next major constituent. It is interesting to note that the preparation condition completely favors the formation of nano spinel ferrite and allow us to study the effect of increasing the Fe content on the properties of the Sr-Co-Chromites. The values of Chromium vary with the increase in Iron concentration.

3.3. Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FTIR spectrums of the investigated sample are shown in Fig.4. Infrared spectroscopy study supported the formation of Sr-Co-Fe-nanochromite with enlightening two strong absorption bands around 400 cm^{-1} and 600 cm^{-1} that are common features of all spinel ferrites. The spinel structure is attributed to the stretching vibrations of the unit cell of the spinel in the tetrahedral (A) site and the metal-oxygen vibration in the octahedral (B) site. These absorption bands are highly sensitive to changes in interaction between oxygen and cations, as well as to the size of the obtained nano-particles [31]. The vibration frequency depends on the cation mass, cation-oxygen distance and bending force [32]. From Fig 4, Intrinsic stretching vibration frequency of metal-oxygen at tetrahedral site observed in a range $624\text{ cm}^{-1} - 594\text{ cm}^{-1}$ and octahedral site observed in the range of $495\text{ cm}^{-1} - 407\text{ cm}^{-1}$. No subsidiary

bands present in mixed form of Chromium and Iron. The replacement of Cr^{3+} ions by bigger atomic mass (Fe^{3+}) results in increase in metal oxygen bond length and consequently decrease the wave number of octahedral and tetrahedral sites [33, 34]. Intensity of the peaks corresponds to tetrahedral site increase up to $x = 0.15$ after that decrease. And in octahedral site alternate decrease and increase in peak value is occur. It is well known that the intensity ratio is function of change of dipole moment with the internuclear distance. This value represents the contribution of ionic bond Fe-O in the lattice. So the observed decrease and increase in the absorption band intensity with increase in Fe content, is due to perturbation occurring in Fe-O bonds. The electronic distribution of Fe-O bonds greatly affected by the dopant Fe^{2+} which is having comparatively higher atomic weight than the Chromium ions. (Cr^{3+} -51.996 a.m.u, Fe^{2+} 55.845 a.m.u).

3.4 Vibrational Sample Magnetometer (VSM) analysis:

MH-loops reveals the magnetic properties of $\text{Sr}_{0.5}\text{Co}_{0.5}\text{Fe}_x\text{Cr}_{2-x}\text{O}_4$ nanochromites ($x = 0, 0.5, 1.5, 2.0$). Hysteresis loops for the samples are recorded with LAKESHORE Vibrational sample magnetometer at 300 K with applied field as 30 KOe are shown in Fig.5. The values of saturation magnetization (M_s), Remanence Magnetization (M_r), Coercivity (H_c) are measured from the hysteresis loop. The value of anisotropy constant (K) was calculated from Stoner-Wohlfarth relation as follows $H_c = K/M_s$ [35]. Calculation of magnetic moment in bohr magneton was carried out using the following relation, $n_B = (\text{Molecular Weight} \times M_s)/5585$ [36]. Magnetic parameters saturation magnetization (M_s), Remanence Magnetization (M_r), Coercivity (H_c), Squareness ratio, Magnetic anisotropy constant (K) and magneton number are tabulated in Table 4. Generally magnetic properties in the prepared sample arise from coupling between spin and orbital angular momentum (L-S coupling) and electron spin (S-S coupling) [37]. In the case of spinel nano magnetic ferrite material magnetic parameters are influenced by cation distribution, collinearity and non-collinearity (canting) of spins on their surface, crystallite size and dopant. In the present study increasing Fe content transformation of paramagnetic to ferromagnetic to diamagnetic behaviour is occur. When increasing Fe content the saturation magnetization gets increase. The drastic increase occurs at $x=1.5$, M_s is 155.229 emu/g . at $x=2.0$ M_s value reduced nearly equal to negative zero value this indicates diamagnetic nature. Respective to M_s value the Remanance Magnetization value is obtained. The transformation of paramagnetic nature to ferromagnetic nature and then to anti-ferromagnetic is supported by the Coercivity value. At $x = 0$, H_c value is zero indicates paramagnetic nature. $x=0.5, 1.5$ increase in H_c indicates Ferromagnetic nature. $x=2.0$ value indicates diamagnetic nature. The increase in Coercivity value from 855 to 2099 Oe is due to the decrease in crystallite size of the samples, which changes from 57.8 to 52.8 nm . Moreover, the deterioration of magnetic properties at SrCoFeO content

$x=2.0$ can also be due to the presence of large amount of nonmagnetic Sr^{2+} ions at (B) sites. It has highest K value at $x=1.5$. The increase in saturation magnetization and remanence is clear indication that the synthesized material can be used in high-density recording media and high K value will release more thermal energy in alternating magnetic field, and should be act as best candidate for magnetic fluid hyperthermia and in targeted drug delivery system[38].

4. CONCLUSION

Iron doped Strontium Cobalt nanochromite at various concentration has been successfully synthesized by uncomplicated and cheap co-precipitation method. With average crystallite size between 41.7 nm and 57.8 nm. All physical properties are studied for samples sintered at 1173K. Addition of Fe^{3+} ion influenced the structural properties such as Average crystallite size, lattice constant, lattice strain, dislocation density and X-Ray density of the synthesized samples. Absence of super structure reflection peaks from XRD analysis suggest that nano crystal lattice having disordered spinel structure in the higher concentration of the dopant. SEM shows morphology manipulated by Fe^{3+} ion in the sample and FTIR observation have fine match with results of XRD. From VSM, at $x=1.5$ it has maximum magneto crystalline anisotropy value.

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TABLE 1: Comparison of X-Ray Intensity

Fe content 'x'	Composition	I ₂₂₀	I ₄₄₀
0	Sr_{0.5}Co_{0.5}Cr₂O₄	38.99	50.45
0.5	Sr_{0.5}Co_{0.5}Cr_{1.5}Fe_{0.5}O₄	28.87	43.50
1.5	Sr_{0.5}Co_{0.5}Cr_{0.5}Fe_{1.5}O₄	42.89	62.67
2	Sr_{0.5}Co_{0.5}Fe₂O₄	32.07	36.70

TABLE 2: Structural parameters of $\text{Sr}_{0.5}\text{Co}_{0.5}\text{Fe}_x\text{Cr}_{2-x}\text{O}_4$ ($x=0.0, 0.5, 1.5, 2.0$) sintered at 1173K

Fe content 'x'	Composition	Crystallite Size D (nm)	Lattice Constant a (Å)	Molecular Weight g/mole	X-ray Density g/cm ³	Lattice Strain 10 ⁻³	Dislocation Density 10 ¹⁵
0	Sr_{0.5}Co_{0.5}Cr₂O₄	41.7	8.2676	241.215	5.6757	4.296	3.9068
0.5	Sr_{0.5}Co_{0.5}Cr_{1.5}Fe_{0.5}O₄	57.8	8.3431	243.14	5.5619	1.802	0.5914
1.5	Sr_{0.5}Co_{0.5}Cr_{0.5}Fe_{1.5}O₄	53.4	8.3596	246.99	5.6166	1.993	0.7437
2	Sr_{0.5}Co_{0.5}Fe₂O₄	52.8	8.3495	248.915	5.6809	1.997	0.8134

TABLE 3: Vibrational frequency of tetrahedral and octahedral sites

Fe content 'x'	Composition	ν_1 tetra cm ⁻¹	ν_1' tetra cm ⁻¹	ν_2 octa cm ⁻¹	ν_2' octa cm ⁻¹
0	Sr_{0.5}Co_{0.5}Cr₂O₄	624.51	523.80	-	407.00
0.5	Sr_{0.5}Co_{0.5}Cr_{1.5}Fe_{0.5}O₄	604.14	-	495.73	-
1.5	Sr_{0.5}Co_{0.5}Cr_{0.5}Fe_{1.5}O₄	594.25	-	431.997	-
2	Sr_{0.5}Co_{0.5}Fe₂O₄	594.09	-	438.65	412.76

TABLE 4: Magnetic parameters of $\text{Sr}_{0.5}\text{Co}_{0.5}\text{Fe}_x\text{Cr}_{2-x}\text{O}_4$ ($x=0.0, 0.5, 1.5, 2.0$) sintered at 1173K

Fe content 'x'	Composition	M_s (emu/g)	M_r (emu/g)	H_c (Oe)	M_r/M_s	K (erg)	n_B
0	Sr_{0.5}Co_{0.5}Cr₂O₄	0.6037	0.0000	0.0000	0.0000	0.00	0.0261
0.5	Sr_{0.5}Co_{0.5}Cr_{1.5}Fe_{0.5}O₄	2.3539	0.6508	855.1000	0.2765	2012.82	0.1025
1.5	Sr_{0.5}Co_{0.5}Cr_{0.5}Fe_{1.5}O₄	155.2290	62.1292	1197.2000	0.4002	185840.20	6.8648
2	Sr_{0.5}Co_{0.5}Fe₂O₄	0.889	2.7100	2099.1000	3.0484	1866.10	0.0396

M_s -Saturation Magnetization, M_r - Remanent Magnetization, H_c -Coercivity, K-MagnetoCrystalline Anisotropy, n_B -Magnetron number

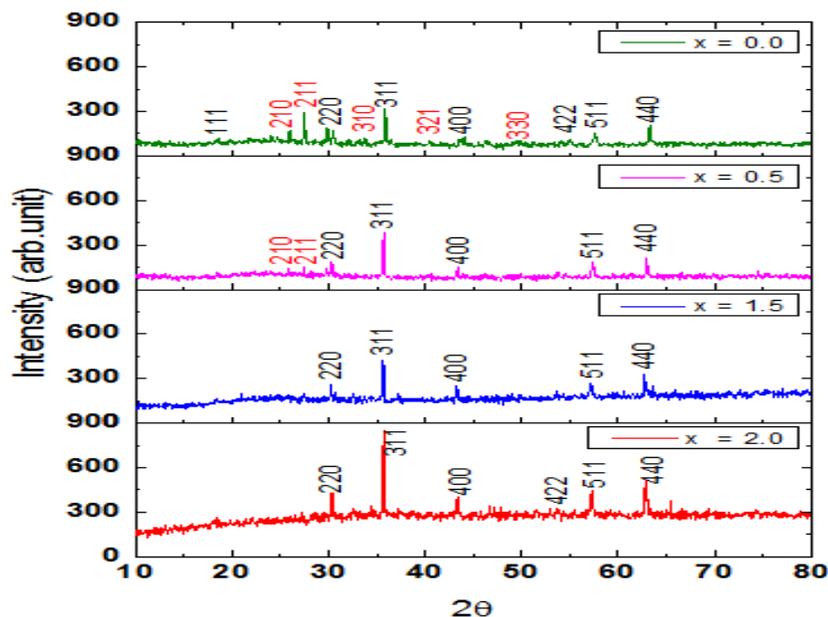


Figure 1: X-Ray Diffraction pattern of $\text{Sr}_{0.5}\text{Co}_{0.5}\text{Fe}_x\text{Cr}_{2-x}\text{O}_4$ ($x=0.0, 0.5, 1.5, 2.0$) sintered at 1173K

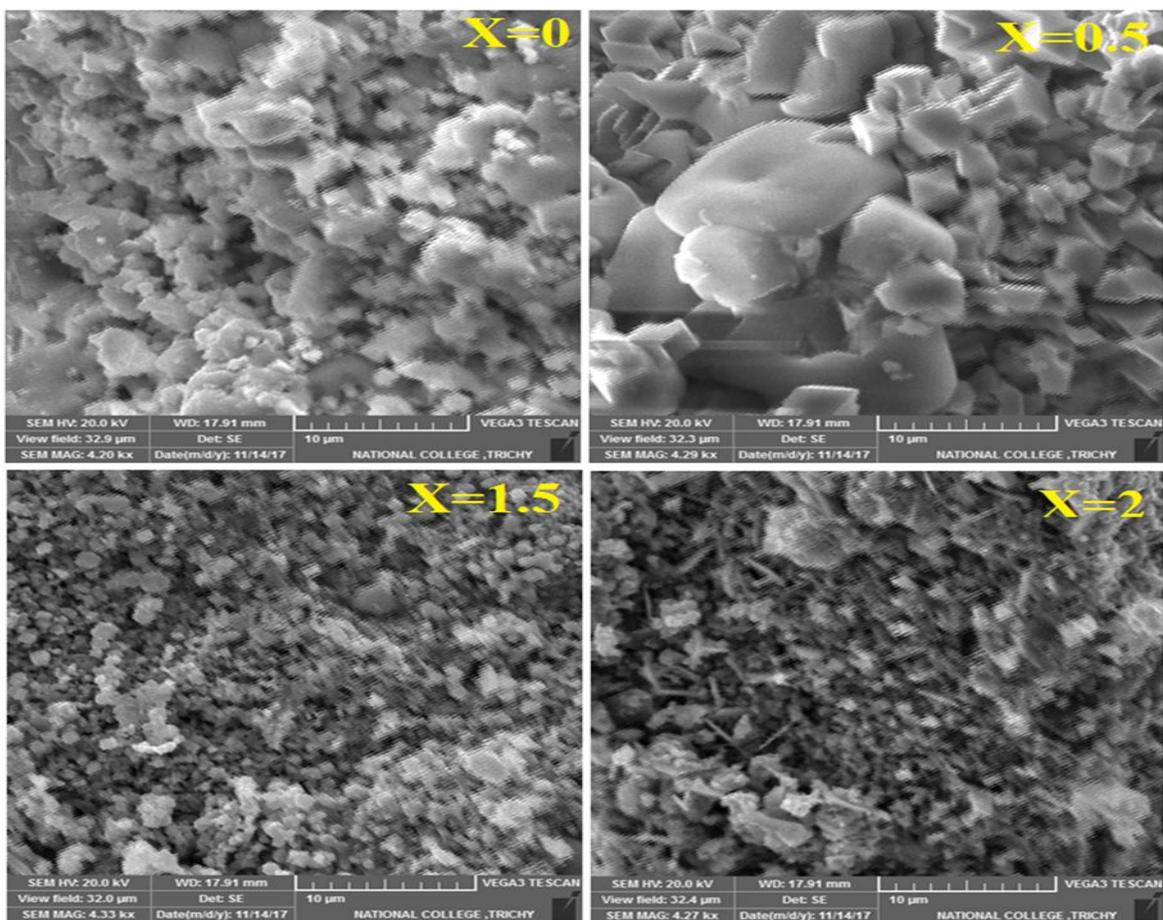


Figure 2. SEM micrograph of $\text{Sr}_{0.5}\text{Co}_{0.5}\text{Fe}_x\text{Cr}_{2-x}\text{O}_4$ ($x=0.0, 0.5, 1.5, 2.0$) sintered at 1173K

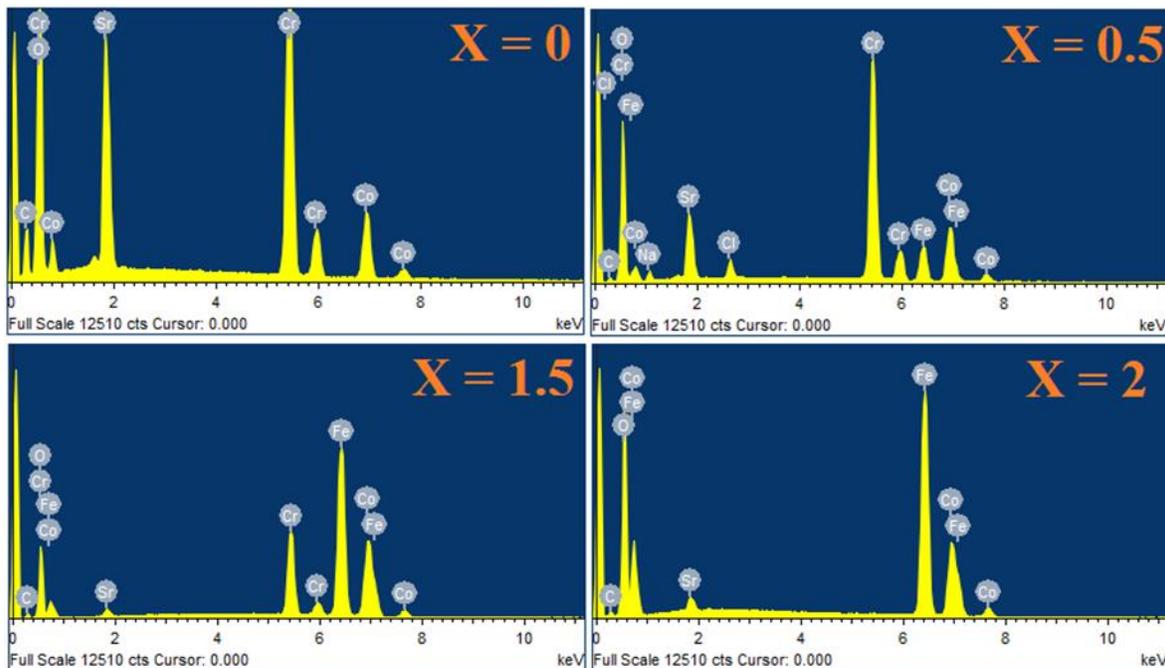


Figure 3: EDS of $\text{Sr}_{0.5}\text{Co}_{0.5}\text{Fe}_x\text{Cr}_{2-x}\text{O}_4$ ($x=0.0, 0.5, 1.5, 2.0$) sintered at 1173K

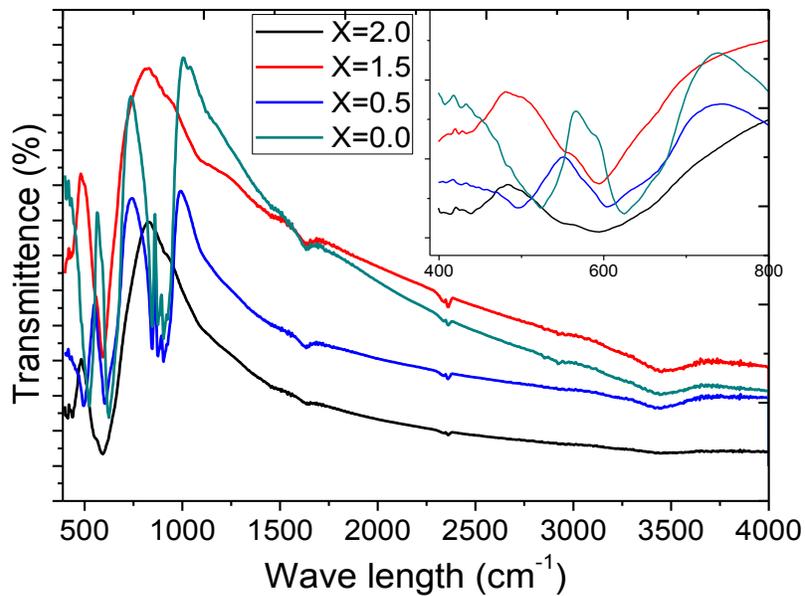


Figure 4: FTIR spectra of $\text{Sr}_{0.5}\text{Co}_{0.5}\text{Fe}_x\text{Cr}_{2-x}\text{O}_4$ ($x=0.0, 0.5, 1.5, 2.0$) sintered at 1173K

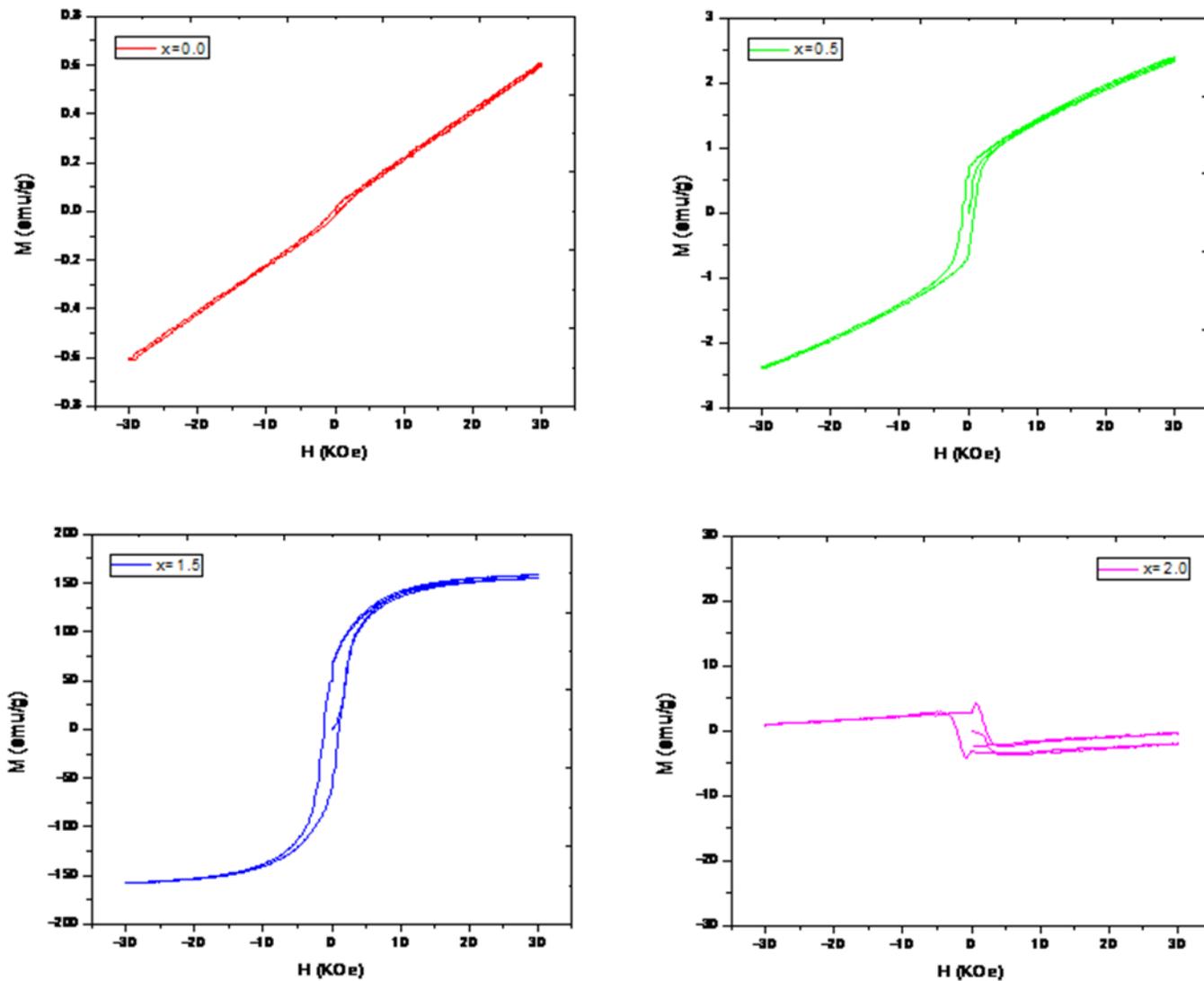


Figure 5: Magnetic hysteresis curves of $\text{Sr}_{0.5}\text{Co}_{0.5}\text{Fe}_x\text{Cr}_{2-x}\text{O}_4$ ($x=0.0, 0.5, 1.5, 2.0$) sintered at 1173K