

## Structural and Magnetic Properties of NiCd Ferrites

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**Abstract:** The ferrite compositions of  $Ni_{1-x}Cd_xFe_2O_4$  with  $x=0, 0.3, 0.5, 0.7, 1$  were synthesized at low temperature through the chemical co-precipitation method. The synthesized powders were characterized using X-ray Diffraction (XRD) for crystallite size and lattice parameter calculation. It reveals the presence of cubic spinel structure of ferrites with crystallite size of 15-23nm. The lattice parameter is found to increase with increase in cadmium concentration obeying Vegard's law. Scanning Electron micrograph (SEM) shows uniform distribution of ferrite particles with some agglomeration and Energy Dispersive X-ray analysis (EDAX) was used to determine the compositional mass ratio. Magnetic measurements of the samples were carried out by means of vibrating sample magnetometer and these studies reveal that addition of  $Cd^{2+}$  concentration greatly affects the magnetic properties.

**Keywords:** Ferrites, X-ray diffraction, Lattice parameter, Magnetic properties.

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### I. Introduction

Spinel ferrites have received appreciable interest owing to their promising magnetic properties and are extensively used in Ferro-fluids, microwave devices, ferroseals and memory cores of computers [1-3]. Spinel type oxides ( $MFe_2O_4$  where M is divalent metal), which include the magnetic ferrites are often denoted by the formula  $AB_2O_4$  where A and B refer to tetrahedral and octahedral sites, respectively. Magnetic properties of ferrites can be suitably tailored by varying composition of cations. The Doping of ferrite with small amount of non-magnetic ions such as  $Zn^{2+}$  or  $Cd^{2+}$  results in augmentation of saturation magnetization [4]. A variety of methods such as co-precipitation, sol-gel, gas condensation, aerosol reactions have been used in the preparation of nanoscale materials [5]. Each technique have unique advantages, chemical precipitation affords greater control over the size and shape of the particles and results in highly uniform morphologies. Among all the ferrites, nickel substituted ferrites are considered to be the most versatile and found wide spread application in the electronics and microwave devices, due to their high electrical resistivity, low-eddy current and dielectric loss [6]. Ni-Cd ferrite, is a soft magnetic material, with a spinel crystal structure with widespread application in recording heads, antenna rods, loading coils, microwave devices, core material for power transformers due to their high resistivity and low eddy current losses. The aim of this paper is to report and discuss the results of structural and magnetic properties of  $Ni_{1-x}Cd_xFe_2O_4$  particles with varying compositions ( $x=0, 0.3, 0.5, 0.7, 1$ ) synthesized at low temperature by chemical co-precipitation method.

### II. Experimental

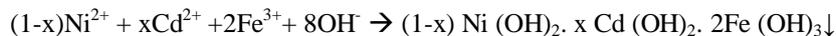
The magnetization of substituted ferrite nanoparticles synthesized by co-precipitation depends mostly on parameters such as reaction temperature pH of the suspension, initial molar concentrations etc. [7]. The aqueous solutions of  $FeCl_3 \cdot 6H_2O$ ,  $NiCl_2 \cdot 6H_2O$  and  $Cd(CH_3COO)_2$  of analytical grade were prepared and kept at 60 °C. To this 8M sodium hydroxide was added with constant stirring. The pH of the solution was maintained at 11.5 for 40 minutes. The resulting precipitate was washed with warm water followed by acetone and filtration was repeated several times until the discarded water was free of chlorides. The washed powder was dried and kept in an oven for 3 hours at 100 °C. The samples were then characterized through various characterization techniques.

The average crystallite size of the ferrite nanoparticles were analyzed using powder X-ray diffractometer (XRD) using  $CuK\alpha$  ( $\lambda=1.5418\text{\AA}$ ) radiation. The scanning was done in the  $2\theta$  range from 20° to 80°. The morphology, structure and elemental composition of the sample were characterized by Scanning electron microscopy (SEM) and Energy dispersive X-ray spectra (EDS). Room temperature magnetic measurements were carried out using vibrating sample magnetometer (VSM) and various parameters were evaluated.

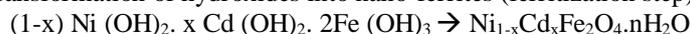
### III. Results And Discussion

#### 3.1. Synthesis of $Ni_{1-x}Cd_xFe_2O_4$ Nanoparticles

Ultra-fine particles of  $Ni_{1-x}Cd_xFe_2O_4$  with  $x$  varying from  $x=0, 0.3, 0.5, 0.7, 1$  were prepared by co-precipitation method, which is two step process. At first, conversion of metal salts into hydroxides, this takes place immediately (Co-precipitation step).



Secondly, the transformation of hydroxides into nano-ferrites (ferritization step)



#### 3.2. Particle Characterization

The precipitated fine particles were characterized by XRD as shown in “Fig.1”. The structure and their crystallite size were evaluated. The crystallite size of the nano-crystalline samples was measured using Debye-Scherer formula [8],

$$D_{XRD} = 0.98\lambda/\beta \text{ Cos } \theta$$

Where  $\lambda$  is the wavelength of X-ray used in  $\text{\AA}$ ,  $\beta$  is the full width at half-maximum (FWHM) in radians in the  $2\theta$  scale,  $\theta$  is the Bragg angle,  $D_{XRD}$  is the crystallite size in nm. The value of  $D$  decreases from 23 to 15nm on increasing value of  $x$ . This can be attributed to the liberation of latent heat of surface which rises the local temperature, consequently slowing down the growth process and lowering ferrite concentration in the vicinity [9].

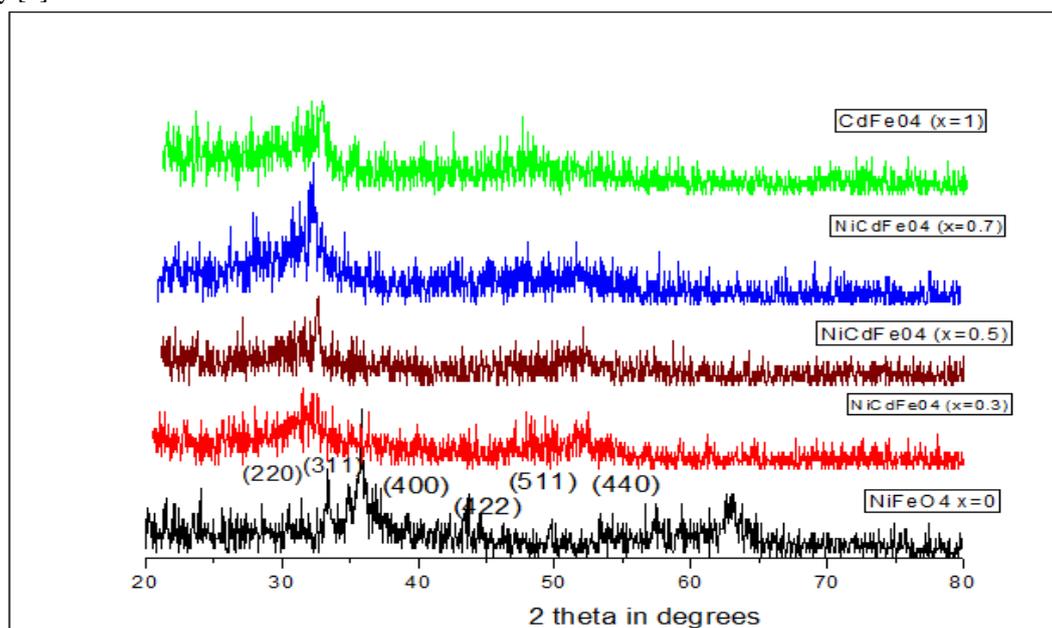


Fig.1. XRD pattern of the  $Ni_{1-x}Cd_xFe_2O_4$  powders

The lattice constant ( $a$ ) was computed using the ‘ $d$ ’ value and with their respective (hkl) parameters. Analysis of the diffraction pattern confirms the formation of cubic spinel structure for all the samples. The peaks indexed to (220), (311), (400), (422), (511) and (440) planes of a cubic unit cell, correspond to cubic spinel structure. The lattice constant calculated using the equation given below and tabulated in “Table 1”  $a=d(h^2+k^2+l^2)^{1/2}$

Table 1: XRD parameters of  $Ni_{1-x}Cd_xFe_2O_4$  powders

x	Chemical Formulae	d-spacing	Lattice constant ‘a’ in angstrom	Average Particle size ‘D’ in nm
0	$NiFe_2O_4$	2.50742	8.3162	23.1055
0.3	$Ni_{0.7}Cd_{0.3}Fe_2O_4$	2.50882	8.32083	22.9120
0.5	$Ni_{0.5}Cd_{0.5}Fe_2O_4$	2.52423	8.37194	17.8776
0.7	$Ni_{0.3}Cd_{0.7}Fe_2O_4$	2.53515	8.40814	15.3860
1.0	$CdFe_2O_4$	2.5094	8.3227	21.3340

The lattice constant was found to increase from 8.31 to 8.41Å with increase in cadmium concentration, which is in good agreement with reference to Vegard's law [10] as shown in “Fig.2”. The addition of cadmium displaces the Fe ions from tetrahedral to octahedral site and larger ionic radius of Cd (0.97Å) elongates the lattice. It is also observed that the sample with higher cadmium content has a larger lattice parameter. Similar variation of lattice constant is observed in the literature, when the ion of larger ionic radii is substituted in nickel ferrite [11-12].

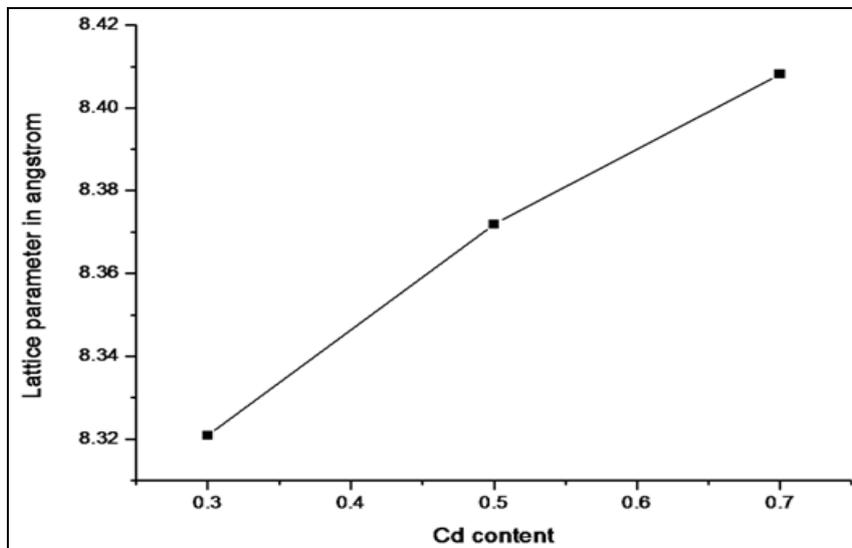


Fig.2. Variation of lattice parameter with cadmium content.

The cation distribution in spinel ferrite can be obtained from an analysis of the X-ray diffraction pattern. In the present work, the Bertaut method [13] is used to determine the cation distribution and their composition  $Ni_{1-x}Cd_xFe_2O_4$  is given by



Where the brackets ( ) and [ ] indicates the tetrahedral (A-site) and the Octahedral (B-site) respectively.

The X-ray density was calculated using the molecular weight and the lattice constant. The XRD density was calculated by formula [14] and tabulated in “Table-2”.

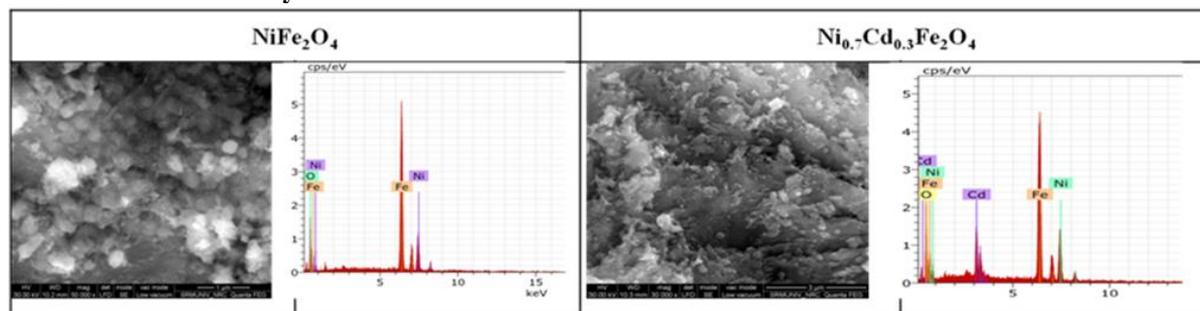
$$XRD \text{ density} = 8M/Na^3$$

Where M is molecular weight of the sample and N is the Avogadro number and ‘a’ is the lattice parameter. X-ray density also exhibited increasing trend on increasing the cadmium concentration.

Table 2: Parameters deduced from XRD studies

x	Chemical Formulae	Molecular mass (g)	X-ray density ( $\times 10^3$ kg/m <sup>3</sup> )	A-site	B-site
0	NiFe <sub>2</sub> O <sub>4</sub>	235	5.427	(Fe <sup>3+</sup> )	[Ni <sup>2+</sup> <sub>1</sub> Fe <sup>3+</sup> <sub>1</sub> ] O <sub>4</sub> <sup>2-</sup>
0.3	Ni <sub>0.7</sub> Cd <sub>0.3</sub> Fe <sub>2</sub> O <sub>4</sub>	251.02	5.787	(Cd <sup>2+</sup> <sub>0.3</sub> Fe <sup>3+</sup> <sub>0.7</sub> )	[Ni <sup>2+</sup> <sub>0.7</sub> Fe <sup>3+</sup> <sub>1.3</sub> ] O <sub>4</sub> <sup>2-</sup>
0.5	Ni <sub>0.5</sub> Cd <sub>0.5</sub> Fe <sub>2</sub> O <sub>4</sub>	261.7	5.9238	(Cd <sup>2+</sup> <sub>0.5</sub> Fe <sup>3+</sup> <sub>0.5</sub> )	[Ni <sup>2+</sup> <sub>0.5</sub> Fe <sup>3+</sup> <sub>1.5</sub> ] O <sub>4</sub> <sup>2-</sup>
0.7	Ni <sub>0.3</sub> Cd <sub>0.7</sub> Fe <sub>2</sub> O <sub>4</sub>	272.38	6.0863	(Cd <sup>2+</sup> <sub>0.7</sub> Fe <sup>3+</sup> <sub>0.3</sub> )	[Ni <sup>2+</sup> <sub>0.3</sub> Fe <sup>3+</sup> <sub>1.7</sub> ] O <sub>4</sub> <sup>2-</sup>
1.0	CdFe <sub>2</sub> O <sub>4</sub>	288.4	6.644	(Cd <sup>2+</sup> )	[Fe <sup>3+</sup> <sub>2</sub> ] O <sub>4</sub> <sup>2-</sup>

### 3.3. SEM and EDX analysis



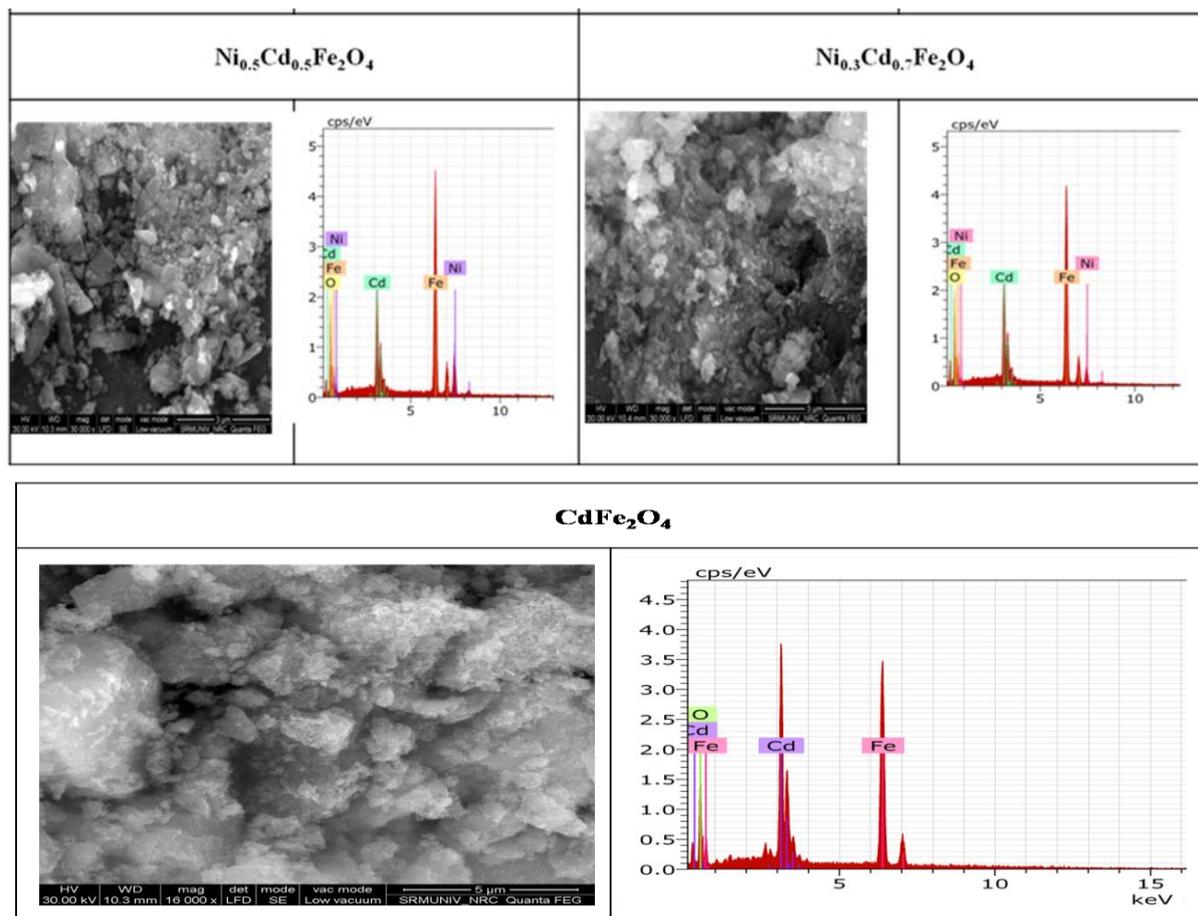


Fig.3 SEM and EDX images of  $Ni_{1-x}Cd_xFe_2O_4$  NPs

“Fig.3” shows scanning electron micrograph of the ferrite samples with various ratios. The uniform nature of the ferrite particle is shown with some agglomeration, since it is prepared by low temperature coprecipitation technique. The EDX (Energy dispersive X-ray spectroscopy analysis) was done in order to determine the chemical composition on the surface of the sample to support our observations on the structure of the ferrite. The results of EDX analysis evaluates the atomic weight percentage of various cations in the investigated samples are found to be approximately correct, which corresponds to a composition ratio and these ratios are expected by the preparation method.

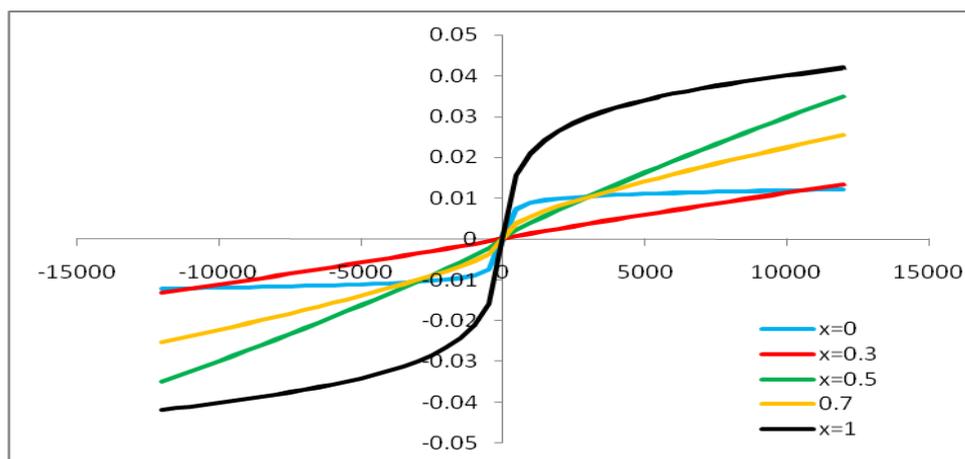
### 3.4. Magnetic Studies

Magnetic properties of  $Ni_{1-x}Cd_xFe_2O_4$  were investigated by VSM at room temperature and the various parameters like Saturation Magnetization ( $M_s$ ), Coercivity ( $H_c$ ) were tabulated in Table-3 and Magnetic hysteresis loop is shown in “Fig.4”.

Table 3: Magnetic Parameters of  $Ni_{1-x}Cd_xFe_2O_4$  NPs

x	Chemical Formulae	Coercivity $H_c$ (Gauss)	Magnetization $M_s$ (emu/gm)
0	$NiFe_2O_4$	53.189	12.125
0.3	$Ni_{0.7}Cd_{0.3}Fe_2O_4$	12.952	13.229
0.5	$Ni_{0.5}Cd_{0.5}Fe_2O_4$	4.0102	34.905
0.7	$Ni_{0.3}Cd_{0.7}Fe_2O_4$	3.0637	25.381
1.0	$CdFe_2O_4$	0.34250	41.97

The value of Coercivity decreases with increasing cadmium concentration. The saturation magnetization increases with increase in cadmium content upto  $x=0.5$  and shows decreasing trend later on. This is due to the fact that cadmium ions have the preference for tetrahedral A sites, nickel ions for octahedral B sites and iron ions are distributed among octahedral and tetrahedral sites.



**Fig.4** Magnetization plot of Ni<sub>1-x</sub>Cd<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> NPs

The substitutions of non-magnetic divalent cadmium ions on the A-sites transfer the trivalent iron ions on B sites affecting the magnetic moments of individual sub lattice and A-B interactions. As the cadmium ion increases at A site, the magnetization decreases, this results in an increase of net magnetization, which is in agreement with Neel's two sub lattice model. Other researchers have also found such type of behaviour in the other ferrite systems [15-16]. With further increase in cadmium concentration the A moments becomes too weak to affect the B moments and net saturation magnetization decreases, indicating the predominance of Yafet and Kittel [17] model.

#### IV. Conclusion

Ni<sub>1-x</sub>Cd<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> ferrites have been successfully synthesized by co-precipitation method at low temperature and successful formation of cubic spinel structure confirmed by X-ray diffraction analysis. The lattice parameter and x-ray density were found to increase with increase in cadmium concentration obeying Vegard's law. SEM micrographs and EDX analysis confirms the chemical compositions, which support our observations on the structure of the ferrite. The saturation magnetization is found to increase with increase in cadmium concentration and then decreases.

#### References

- [1]. J.D. Adams, L.E. David, G.F. Dionne, E.F. Schloemann, S. Stitzer, Ferrite devices and materials, IEEE Trans. Microw. Theory Technol., 5, .2002, 721-737.
- [2]. M.P. Horwath, Microwave applications of soft ferrites, J. Magn. Magn. Mater., 215, 2000, 171-183.
- [3]. S.W. Charles, R. Chandershekar, K.O. Geady, M. Walker, Ionic magnetic fluids based on cobalt ferrite nanoparticles, J.Appl. Phys., 64, 1988, 5840-5844.
- [4]. B.Viswanathan, V.R.K. Murthy, Ferrite Materials, Narosa Publications, 1990, P.11.
- [5]. I.H. Gul, W. Ahmed, A. Maqsood, Electrical and magnetic characterization of nano-crystalline Ni-Zn ferrite synthesis of co-precipitation route, J. Magn. Magn. Mater., 320, (3-4), 2008, 270-275.
- [6]. D. Stoppels, Developments in soft magnetic power ferrite, Journal of Magnetism and Magnetic Materials, 160(1), 1996, 323-8.
- [7]. B. Jayadevan, C.N. Chinnasamy, K. Shinoda, K. Tohji, J. Appl. Phys. 93 ,2003, 8450.
- [8]. E. Warren, X-ray diffraction, Addison Wiley, Reading, 1969:
- [9]. R.F. Strickland-Constable, Kinetics and Mechanism of Crystallization, Academic Press, New York, 1968.
- [10]. L.H. Gul, J. Magn. Magn. Mater., 320, 2008, 270—275.
- [11]. Sagar.E. Shirsath, S.M. Patange, R.H. Kadam, M.L. Mane, K.M. Jadhav, J. Mol. Struct. 1024, 2012, 77.
- [12]. H. Kavas, A. Baykal, M.S. Toprak, Y. Koseoglu, M. Sertkol, B.Aktas, J. Alloy. Comp., 479 2009, 49.
- [13]. L. Weil, E.F. Bertaut, L. Bachirol, Journal de physique et Le Radium, 11, 1950, 208.
- [14]. ASTM card No. 17-484.
- [15]. R.G. Kharabe, S.A. Jadhav, A.M. Shaikh, D.R. Patil, B.K. Chogule, Material Chemistry and Physics, 72, 2001, 77-80.
- [16]. S.A. Masti, A.K. Sharma, P.N. Vasambekar, A.S. Vaingankar, Journal of Magnetism and Magnetic Materials, 305, 2006, 436-439.
- [17]. Y.Yafet, C. Kittel, Physical Review, 87, 1952, 290-294.