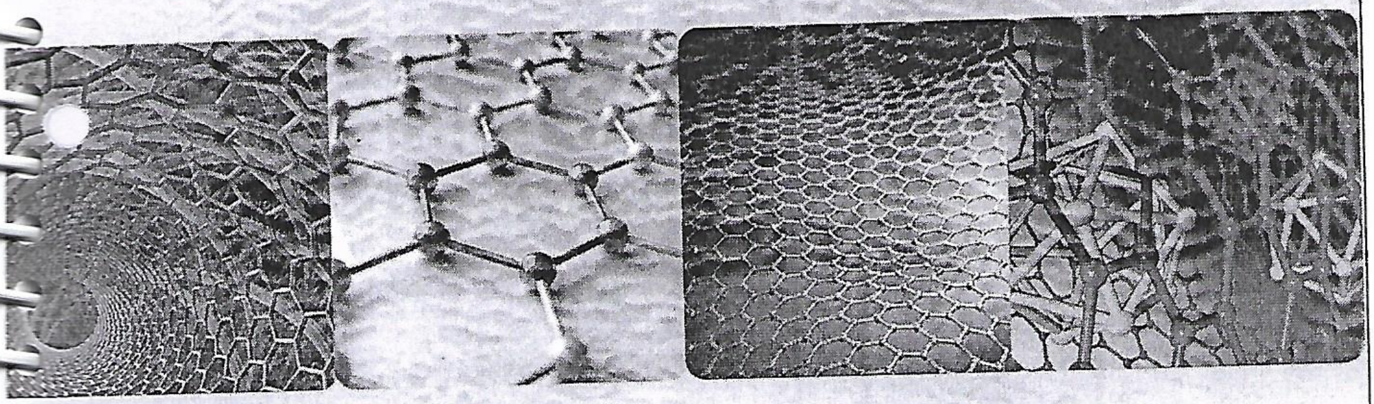
ISBN: 978-93-91768-77-5

Advances in Functional and Sustainable Materials



Editors

Prin. (Dr.) M. M. Rajmane

Prof. (Dr.) J. B. Thorat

Dr. S. H. Pisal

Dr. I. A. Dhole

Published By: Bhumi Publishing

First Edition: 2022

MICROWAVE ASSISTED SYNTHESIS AND CHARACTERIZATION OF MG-CO-ND-CD NANOPARTICLES

S. S. Barate[1*], L. S. More[1] , A. D. Pawar[1], B. B. Patil[1], S.R. Bhongale[2], J. S. Ghodake[3], J. B. Thorat[4], U. K. Mohite[5], T. J. Shinde[1]

[1]P. G. Department of Physics, Smt. KRP Kanya Mahavidyalaya, Islampur, (MS), 415409

[2] Department of Physics, Yashwantrao Chavan Institute of Science (Autonomous), Satara, (MS), India - 415001

[3]Departments of Physics, PDVP Mahavidyalaya, Tasgaon (MS), India- 416312

[4]Department of Physics, Sadguru Gadage Maharaj College, Karad (MS), India- 415110

[5]Department of Physics, M.B.S.K.K.M. Kadegoan (MS), India- 415304

*Corresponding author E-Mail: shrutibarate2016@gmail.com

Abstract:

Nd^{3+} doped Mg-Co-Cd ferrite nanoparticles with composition $Mg_{0.4}Co_{0.2}Cd_{0.4}Nd_{0.02}Fe_{1.98}O_4$ were synthesized successfully by sol-gel method. The sintering of synthesized powder was carried out microwavely at 60W for 20 min and characterized by XRD, FTIR and FESEM techniques. These techniques are utilized to evaluate crystal structure, phase purity and surface morphology of ferrite material. X-ray analysis study shows the formation of single-phase cubic spinel structure. The FTIR results confirm the presence of two prominent vibrational bands in the wave number range 399.73-569 cm^{-1}. The irregularly shaped grains with slight agglomeration observed in the FESEM photograph. EDAX confirmed the formation of desired ferrite in required stoichiometric ratio without any impurity phase.

Keywords: Mg-Co-Nd-Cd nano-ferrite; Sol-gel method; XRD; FTIR; FESEM; EDAX.

1. Introduction:

Nanoparticles of spinel ferrites proved themselves as a potential candidate in various applications that include permanent magnets, chemical sensors, microwave absorbers, magnetic storage information devices microwave devices and also in biomedical applications [1-3]. Unique properties such as high initial magnetic permeability, electrical resistivity and saturation magnetization and low power losses make nanoferrites one of the most diverse magnetic materials [4]. Nanoferrites have very versatile structural, magnetic, electrical, and dielectric properties that are tailored by method of preparation and adding divalent or trivalent ions of suitable valences with appropriate thermal treatments [5].

Different synthesis techniques, such as ceramic method [6], sol–gel technique [7], microwave [8], hydrothermal method [9], oxalate co-precipitation technique [10], microemulsion

[11], and thermolysis [11] have been used to produce nanoferrites. Among these, owing to the good stoichiometric control and production of nano size particles at quite low temperature, the sol–gel technique is an attractive synthesis technique [12]. Several researchers studied the structural, electrical and magnetic properties of ferrites, because with substitution of a small proportion of rare-earth elements in ferrites will drastically modify its properties [13]. Effects of substitution of different types of rare earth ions on the ferrite structures and electromagnetic properties were studied by Rezlescu et al [12]. Nd^{3+} substituted nano-sized magnesium ferrite have been reported by seldom et.al [14].Cobalt ferrite prepared by microwave-assisted synthesis was studied by Komarneni et al. [15].Effect of sintering temperature on the magnetic and morphological characteristics of magnesium ferrite have been investigated by Reddy et al. [16]. Dippong et al. prepared cobalt ferrites using sol-gel method and they observed the structural and magnetic properties of prepared samples [17].

In the present paper we report the structural properties of Nd^{3+} doped Mg-Co-Cd ferrite nanoparticles prepared by sol-gel method using microwave sintering technique.

2. Experimental:

Neodymium substituted Mg-Co-Cd ferrite with chemical composition $Mg_{0.4}Co_{0.2}Cd_{0.4}Nd_{0.02}Fe_{1.98}O_4$ was prepared by well-known sol–gel method. Analytical grade magnesium nitrate, cobalt nitrate, cadmium nitrate, neodymium nitrate, ferric nitrate and citric acid ($C_6H_8O_7$) were used as starting materials. These materials were weighted in its stoichiometric amounts and dissolved in distilled water with stirring. The ratio of entire nitrates to citric acid was taken as to be 1:1. For homogeneity, the solution was constantly heated at 80^0C for 2 hours. After cooling, ammonia was added in the solution in order to maintained pH at 7. The solution was then heated until it converted into the form of gel. The process of heating was continued till gel is ignited and converted into powder. The pre-sintered powder was milled in agate mortar for an hour using acetone as base. The resulting powder was sintered at 60W for 20 minutes using microwave oven (ONIDA 23L 800 watts). The structural properties of prepared ferrites were investigated by using X- ray diffraction, FTIR, FESEM and E-DAX techniques. The Rigaku miniflex-600 make X-ray diffractometer having Cu-Kα radiation (λ = 0.15406 nm) was used to determine the crystalline phase of ferrite nanoparticles. The FTIR spectrum ferrite was recorded in range 350 cm^{-1} to 850 cm^{-1} by using Bruker ALPHA 100508. The morphological study of the ferrite was carried out on Zeiss Ultra-55 of FE-SEM.

The elemental composition of prepared ferrite was studied by using EDAX model ASTM E1508-98 Oxford.

3. Result and Discussion:-

3.1. X-ray Diffraction Analysis (XRD):-

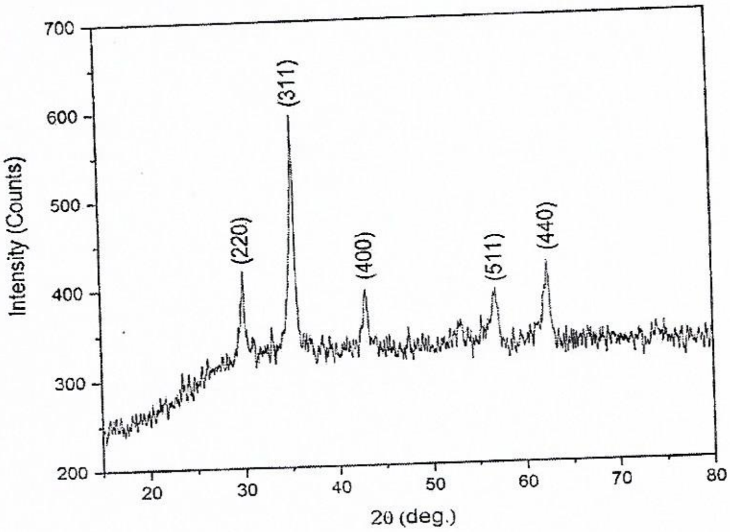


Fig. 1 XRD pattern of $Mg_{0.4}Co_{0.2}Cd_{0.4}Nd_{0.02}Fe_{1.98}O_4$

X-ray diffraction patterns of microwavely sintered $Mg_{0.4}Co_{0.2}Cd_{0.4}Nd_{0.02}Fe_{1.98}O_4$ ferrite samples are shown in Fig.1. The presence of nominated planes such as (220), (311), (400), (431/511), (440) in the diffraction patterns confirms the formation of single phase cubic spinel structure without any ambiguity phase. Lattice constant 'a' of the ferrite was calculated for most intensive peak (311) plane by using Bragg's equation and was found to be 8.4149 A° and tabulated in the Table 1. The crystallite size (D_A) was determined by using Scherrer formula:

$$D = \frac{0.94\lambda}{\beta\cos\theta}$$

Where, $\lambda = 1.5425$ A° is the wavelength of X-ray radiation, θ is the diffraction angle and β is the full-width half maxima.

Mg-Cd-Nd ferrites have been prepared by oxalate co-precipitation method by Bhongale et.al [18]. They reported that, well defined intense peak is observed at 70w microwave sintering, but we observed higher intensity peak at 60w microwave sintering.

Table 1: XRD parameters of $Mg_{0.4}Co_{0.2}Cd_{0.4}Nd_{0.02}Fe_{1.98}O_4$

Crystallite size D (nm)	Lattice consta nt a (Å)	Ionic radii Å		Bond length Å		X-Ray density (ρ) 10^{-6} gm/cm³	Wave number cm⁻¹	
		r_A	r_B	L_A	L_B		ϑ_1	ϑ_2
17.10	8.4149	0.287	0.907	7.288	5.95	5.4373	569	399.73

3.2. Fourier Transform Infrared Spectroscopy (FTIR):

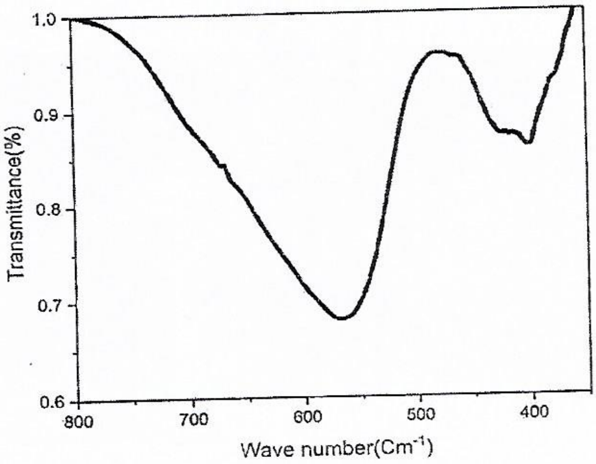


Fig. 2 FTIR spectra of $Mg_{0.4}Co_{0.2}Cd_{0.4}Nd_{0.02}Fe_{1.98}O_4$

FTIR spectra of $Mg_{0.4}Co_{0.2}Cd_{0.4}Nd_{0.02}Fe_{1.98}O_4$ system is shown in Fig. 2.To conclude ferrite phase formation and detection of chemical bond present in spinel structure the measurement were carried out from FTIR spectrometer. Mainly two absorption bands ϑ_1 and ϑ_2 corresponding to tetrahedral (A) and octahedral (B) sites appeared in the most of the ferrites and which is characteristic of spinel structure ferrite. In prepared ferrite, the absorption band ϑ_1 corresponding to tetrahedral (A) sites observed at 569 cm^{-1}, and this can be related to metal cation–oxygen bond stretching vibrations. The absorption band ϑ_2 appears at 399.73 cm^{-1} corresponding to octahedral (B) sites and it relates metal cation oxygen bond stretching vibrations in octahedral site. It confirms that typical bands of spinel structure are formed under microwave sintering.

3.3. Field Emission Scanning Electron Microscopy (FESEM):

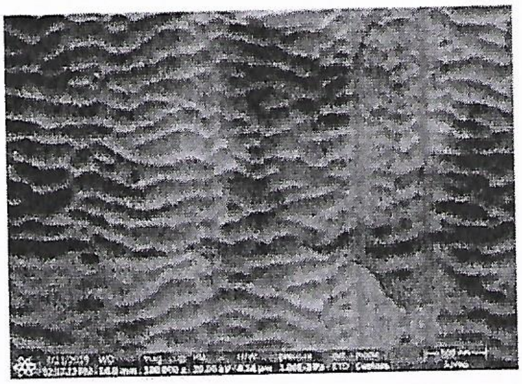
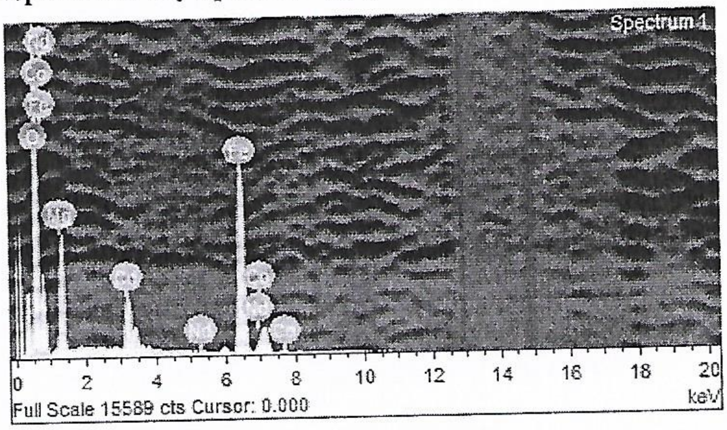


Fig. 3 FESEM microphotographs of $Mg_{0.4}Co_{0.2}Cd_{0.4}Nd_{0.02}Fe_{1.98}O_4$

The FESEM microphotograph of $Mg_{0.4}Co_{0.2}Cd_{0.4}Nd_{0.02}Fe_{1.98}O_4$ sample is shown in Fig. 3. The morphology of ferrite shows clustered like grains. It can be normally acquired as a result of partial agglomeration of nanoparticles. Due to weak Van der Waals bonds or magnetic forces, irregular agglomeration of particles take place.

3.4.Energy Dispersive X-ray Spectroscopy (EDAX):



The energy dispersive X-ray analysis (EDAX) spectra of $Mg_{0.4}Co_{0.2}Cd_{0.4}$ $Nd_{0.02}$ $Fe_{1.98}O_4$ system is shown in Fig. 6. The spectra confirm the presence of stoichiometric elements Mg, Cd, Co, Nd, Fe and O with required proportion without impurity phase.

Conclusion:

Nd^{3+} doped Mg-Co-Cd ferrite nanoparticles with composition $Mg_{0.4}Co_{0.2}Cd_{0.4}$ $Nd_{0.02}$ $Fe_{1.98}O_4$ were synthesized successfully by sol-gel method at lower sintering temperature and smaller duration. X- ray diffraction analysis confirms the formation of cubic spinel structure. Lattice constant 'a' of the ferrite was found to be 8.4149 A°. The appearance of two main absorption bands in the FTIR spectra shows the formation of ferrite with cubic spinel structure. Morphological study shows irregular agglomeration that may be due weak Van der Waals bonds or magnetic forces. EDAX spectra indicate the formation of required stoichiometric ferrites.

References:

[1] R. M. Anderson, C. R. Vestal, A. C. S. Samia and Z. J. Zhang, Appl. Phys. Lett., 2004, 84, 3115–3117.

[2] N. Rezlescu, E. Rezlescu, D. Dorofei and P. D. Popa, J. Phys.: Conf. Ser., 2005, 15, 296–299.

[3] E. Rezlescu, L. Sachelarie, P. D. Popa and N. Rezlescu, IEEE

[4] G. Ott, J. Wrba, R. Lucke, J. Magn. Magn. Mater. 535 (2003) 254–255.

[5] P. Kumar, J. Chand, S. Verma, M. Singh, : Int J. Theoretical and App Sci. 3 (2011) (2) 10-12

[6] Patil, M.R., Rendale, M.K., Mathad, S.N., and Pujar, R.B., Int. J. Self-Propag. High-Temp. Synth. , 2015, vol. 24, no. 4, pp. 241–245.

[7] Shi Y, Ding J and Yin H 2000 J. Alloys Compounds 308 290–5

[8] Liu Q, Sun J, Long H, Sun X, Zhong X and Xu Z 2008 Mater. Chem. Phys. 108 269–73

[9] Maaz K, Mumtz A, Hasanain S K and Ceylan

[10] Kosak, A., Makovec, D., Znidarsic, A., and Drofenik, M., *J. Eur. Ceram. Soc.*, 2004, vol. 24, no. 6,pp. 959–962.

[11] Bao, N.Z., Shen, L.M., Wang, Y.H., Padhan, P., and Gupta, A., *J. Am. Chem. Soc.*, 2007, vol. 129,no. 41, pp 12374–12375.

[12] N. Rezlescu, E. Rezlescu, Solid State Commun. 88 (2) (1993) 139.

[13] Sun G-L, Li J-B, Sun J J and Yang X Z 2004 J. Magn. Magn. Mater. 281 173.

[14] Rezlescu N, Rezlescu E, Pasnicu C and Craus M L 1994 J. Phys.: Condens. Matter 6 5707.

[15] Thomas Dippong, Lucian Diamandescu, Ion Bibicu, Cristian Leostean,Gheorghe Borodi, Lucian Barbu Tudoran, J. Magn. Magn. Mater. 394 (2015) 111–116.

[16] Bhongale SR, Ingawale HR, Shinde TJ, Vasambekar PN, Journal of Rare Earths (2018),