

A Brief Review on Structural, Morphological, Magnetic and Dielectric Behavior of Divalent Cation-substituted Nanocrystalline Cobalt Ferrite

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Abstract: Spinel ferrites nanoparticles have received much more focus due to their promising applications in various technologies, such as magnetic refrigerators, microwave devices, colour imaging, high-density recording media and magnetic fluids. Nanocrystalline cobalt ferrite has been widely studied for its distinctive properties, like cubic magneto crystalline anisotropy, high coercivity, reasonable saturation magnetization, great chemical constancy, wear resistance and electrical insulation. Thus, cobalt ferrite has its own applications in the arena of high-frequency devices, magneto-optical devices, memory cores, recording media and spintronics and also in the biomedical sector, such as MRI, drug delivery schemes and magnetic hyperthermia. Reduced dimensionality of ferrite nanoparticles is having differences in properties when compared to its bulk counterparts. Size modulation of the physico-chemical properties of cobalt ferrite gives a distinctive response in the nanosized scale, permitting for material engineering in order to meet different necessities while addressing its different applications. The present study gives a brief review of significant effects on structural, dielectric and magnetic properties of cobalt spinel ferrite with different cation substitutions, like Mg^{2+} , Ni^{2+} , Zn^{2+} , Cu^{2+} , In^{2+} ...etc. This study regarding the size and its dispersity, core shell design, shape, crystallinity and surface decoration with hybrid derivatives opens up a wide variety of prospects for elementary studies as well as for growth and revolution in the field of ferrites.

Keywords: Spinel ferrite, Structural properties, Microstructural properties, Magnetic properties, Dielectric properties.

Introduction

Spinel ferrite has the general formula $(A)[B_2]O_4$, with the larger oxygen anions forming a close-packed FCC structure with the smaller metal cations occupying interstitial sites: the tetrahedral (A) sites and the octahedral [B] sites. In cubic spinel MFe_2O_4 structure, the cations will locate at tetrahedral (A) and octahedral (B) interstitial sites. Generally, in the normal spinel ($MnFe_2O_4$, $ZnFe_2O_4$), divalent cations (Zn^{2+} , Mn^{2+}) occupy tetrahedral A sites, while octahedral B sites are occupied by trivalent cations Fe^{2+} . However, in inverse spinel

($CoFe_2O_4$, $NiFe_2O_4$, $MgFe_2O_4$), divalent cations Co^{2+} , Ni^{2+} , Mg^{2+} occupy octahedral sites and trivalent cations Fe^{3+} are distributed equally at A and B sites. So, the formula of MFe_2O_4 ferrites can be stated as $(M_{1-x}^{2+}Fe_x^{3+}) [M_x^{2+}Fe_{2-x}^{3+}]O_4$, where round and square brackets denote tetrahedral and octahedral sites, respectively. The interaction between these two sub-lattices A and B, in a spinel structure (AB_2O_4), comprise inter-sublattice super exchange (A-B) interactions and intra-sublattice exchange

interactions; i.e., (A-A) and (B-B) interactions. The super-exchange interactions (A-B) are more dominant than intra-sub lattice exchange interactions [1]. The type of spinel structure (inverse, normal and mixed) decides the lattice occupancy of the divalent metal ions and hence, these metal ion occupancy on lattice sites influences the properties of ferrite nanoparticles. The structural, electrical and magnetic properties of two sub-lattice ferrites can be evaluated by using tools such as elemental category, composition with concentration and preparation methods as well as outer perturbations such as temperatures and pressures. Correspondingly, octahedral and tetrahedral sub-lattices antiferromagnetic coupling is a key source for the magnetic moment of the spinel ferrite. In normal spinel ferrite structure of CoFe_2O_4 , the tetrahedral A-site is occupied by divalent (Co) atoms, whereas the octahedral B-site is filled by trivalent (Fe) atoms. While, in an inverse spinel structure of CoFe_2O_4 , A-site is populated by Fe^{3+} ions, while B-site is equally populated by Co^{2+} and Fe^{3+} ions. Therefore, the structural, transport and magnetic properties of CoFe_2O_4 are highly based on the synthesis method and crystallite size [2]. Cobalt ferrite exhibits ferromagnetic behavior at the transition temperature around 790 K and crystallizes in tetrahedral A-site coordination ($\text{Co}_{1-\delta}\text{Fe}_\delta\text{O}_4$) and octahedral B-site coordination ($\text{Co}_\delta\text{Fe}_{1-\delta}\text{O}_4$), where δ is the degree of inversion whose value depends on the thermal treatment during the synthesis process. $\delta=1$ corresponds to the formation of inverse spinel structure and $\delta=0$ corresponds to the formation of normal spinel structure. The cation redistribution leads to the microstructure variation, which further affects the magnetic properties of the ferrite nanoparticles. Hence, the internal structure with cation distribution can be well controlled by using appropriate synthetic methods. Thus, by tuning the microstructure and cation distribution within the ferrites, the magnetic properties will be strongly influenced.

Literature Review and Discussion

Microstructure and magnetic properties of MFe_2O_4 (M = Co, Ni, Mn) ferrite nanocrystals were studied by Wei Wang *et al.* [3] who found that all the obtained samples exhibit higher saturation magnetization (Ms). SEM and TEM images indicate that CoFe_2O_4 and MnFe_2O_4 samples contain nanosphere, whereas NiFe_2O_4

samples consist of big cauliflower-like nanoparticles and small nanoplatelets.

Structural and magnetic properties of cobalt ferrite nanoparticles were studied by C. R. Stein *et al.* [4] confirming the inverse cubic spinel structure with Fd-3m space group with diameters in nano-size range (below 10 nm). In the field of magnetic recordings, a large value of magnetic moment is demanded for video and audio modes. Tailhades *et al.* [5] tried to substitute Cu into the CoFe_2O_4 to increase magnetization, but found cation migration during annealing. Reports [6] are also existing on Al- as well as Ti-substituted CoFe_2O_4 prepared by sol-gel method with obtained saturation magnetization of 72.1 and 62.6 emu/gm, respectively, which shows low values of magnetization as compared to Ni-substituted CoFe_2O_4 and mismatch of the synthesized particle size.

The effect of nickel on cobalt ferrite was studied by Ninad B. Velhal *et al.* [7] confirming the formation of cubic-phase spinel structure with 30-44 nm crystallite size variation with nickel content. Porous agglomerated morphology of powder samples was shown in the SEM and dispersion behavior, which shows the high resistance of cobalt nickel ferrite. The saturation magnetization with maximum value of 92.87 emu/gm was observed at 30K temperature by substituting cobalt ferrite with 0.4 nickel concentration. The dielectric constant and loss tangent decrease with frequency, whereas conductivity increases with increase in frequency. As we increase Ni content, the magnetic moment of the sample decreases, due to which Ms, Mr, Hc and Mr/Ms decrease at constant temperature. As we decrease the temperature of the sample, all these magnetic values decrease.

Zinc-doped cobalt ferrite nanoparticles were synthesized by Khalid M. Batoor *et al.* [8] using sol-gel method and approved the formation of single-phase cubic structure with an average particle size between 55.38 and 32.87 nm, which was also authorized by transmission electron microscopy. As we increase the doping concentration of Zn, the lattice parameter was found to increase. The sample display of Maxwell-Wagner type of interfacial polarization with normal dielectric behavior decreases with increasing the applied field frequency. With the doping of non-magnetic Zn, the ac conductivity, dielectric constant and loss tangent were found

to decrease. Also, the same type of change was observed for other magnetic parameters; i.e., saturation magnetization, remanence and coercivity.

In doped cobalt ferrite nanoparticles studied by Razia Nongjai *et al.* [9] for magnetic and electrical properties, the major dependence of magnetic and electric properties on grain size was found. The variation of dielectric properties, $\tan \delta$ and as conductivity with frequency exposes, the dispersion is owed to Maxwell-Wagner type of interfacial polarization in general and the hopping of charge between Fe^{2+} - Fe^{3+} and between Co^{2+} - Co^{3+} ions at B-sites.

S. Muthurani *et al.* [10] synthesized nanostructured $Cu_xCo_{1-x}Fe_2O_4$ by auto-combustion method to sense the magnetic and humidity properties and observed a significant increase in saturation magnetization (M_s) and remanence magnetization (M_r) due to the substitution of Cu^{2+} ions in cobalt ferrite, while coercivity (H_c) decreases. The temperature variation of electrical conductivity shows a thermal hysteresis and definite break in conductivity, which corresponds to ferromagnetic-paramagnetic transition and exhibits highest sensitivity for humidity.

Comparative Observations

The different cation substitutions and their significant effects on the characteristics (structural, dielectric, magnetic) of ferrites have been intensively studied. In particular, the effect of partial interstitial substitution of divalent cations like Cu^{2+} , Ni^{2+} , Zn^{2+} , Mg^{2+} ... etc. was studied.

Cu Substitutions

A. Samavari *et al.* [11] results showed that increasing Cu concentration causes a reduction in the nanoparticle size. Saturation magnetization (M_s), remanence magnetization (M_r) and coercivity (H_c) of the cobalt ferrite diminished by the substitutions of Cu^{2+} ions. B. Chandra Sekhar *et al.* [12] confirmed the cubic spinel structure and improved strain derivatives standards as compared to pure cobalt ferrite, thus making them suitable for the application of stress sensing. N. Sanpo *et al.* [13] also reported the same results. M. Margabandhu *et al.* [14] synthesized Cu^{2+} -substituted cobalt ferrite magnetic nanoparticles by chemical co-precipitation method. The VSM results showed

that the magnetic parameters coercivity (H_c) and retentivity (M_r) decrease with increase in Cu^{2+} substitution. Saturation magnetization (M_s) shows increment and decrement with Cu^{2+} substitution. Rakesh K. Singh *et al.* [15] synthesized copper-substituted cobalt ferrite nanoparticles using citrate precursor method and observed a cubic spinel structure with sharp changes in particle size, lattice constant, magnetization and retentivity with respect to Cu content.

Ni Substitutions

Sonal Singhal *et al.* [16] prepared nano-size nickel-substituted cobalt ferrite using aerosol route with a particle size of 10 nm which rises up to 80 nm on annealing at 1200°C. With the nickel concentration, the unit cell parameter decreases linearly due to smaller ionic radius of nickel. Won-Ok Choi *et al.* [17] used sol-gel method and found that when the nickel-substitution was increased, the lattice constant and size of particles of the ferrite powders decreased. The nickel-substituted cobalt ferrites showed lower coercivity and saturation magnetization than the pure cobalt ferrite powders, due to cation distribution, magnetic moment and magneto crystalline anisotropy constant of the substituted ions. The variation of lattice parameter from 8.350 to 8.300 Å was observed by Mozaffari *et al.* [18] with the increase of Ni content. Saturation magnetization (M_s) and coercive forces drops with increase in nickel content, but the Curie temperature increases, which was explained by the changes in magneto crystalline anisotropy. Abdul Gaffor *et al.* [19] manufactured nickel-cobalt nanoparticles by citrate gel auto-combustion method and confirmed cubic spinel structure of ferrites. For magnetic data storage purpose, the crystallite size in the range of 20 nm to 31 nm is desirable. N. B. Velhal *et al.* [20] reported the cubic spinel phase using the auto-combustion technique at low temperature. Due to the lower magnetic moment of nickel, the magnetic properties; i. e., M_s , M_r , H_c and M_r/M_s decrease with the increase of Ni content and show a temperature-dependent behavior. Nickel-substituted cobalt ferrite was prepared by Uday Bhasker Sontu *et al.* [21] using self-combustion method at low temperatures and observed the tuning of magnetic and electrical properties of the ferrites from hard magnetic and lower-resistivity cobalt ferrite to soft magnetic and

high-resistivity nickel ferrite. Ajaypal Singh *et al.* [22] synthesized nanoscale ferrite particles by using solution combustion method and exposed single-phase spinel structure to X-ray diffraction studies. Magnetic studies showed the variation of coercivity and saturation magnetization with nickel substitution and observed higher values of coercivity and saturation magnetization than in pure nickel ferrites. The single cubic spinel phase for all the samples was also observed by P. P. Hankare *et al.* [23]. It is also noticed that as we increase the nickel content in the cobalt ferrites, the lattice parameter decreases and the crystallite size of the ferri-spinel increases. The size of the synthesized nanoparticles lies between 20 and 25 nm.

Zn Substitutions

Faheim, A. S. *et al.* [24] synthesized zinc-substituted nano-crystalline cobalt ferrite powders, $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ by the co-precipitation method. X-ray analysis tells that the samples were in cubic spinel. Bond length between the magnetic ions, lattice constant, X-ray density and ionic radii were found to increase as we increase the zinc concentration. The crystallite size is within the range of 6-24 nm, approved by XRD & HR-TEM. The saturation magnetization and coercivity decrease with the increase in zinc content. The chemical composition of $\text{Co}_{0.3}\text{Zn}_{0.7}\text{Fe}_2\text{O}_4$ sample exhibits super-paramagnetic behavior. El-Saady *et al.* [25] showed the same results and exhibited super-paramagnetic behavior. The single-phase spinel cubical structure was confirmed by S. Nasrin *et al.* [26] who observed that the average grain size increases with the sintering temperature and decreases with the increase of zinc content. Thus, the sintering temperature and zinc content both affect the lattice constant. The saturation magnetization, remanent magnetization, coercivity and magnetic moment have been found to show a decreasing behavior with the increase of Zn content. Experimental data shows that Curie temperature increased with the increase of sintering temperature and decreased with the increase of Zn content. S. Jadhav *et al.* [27] also confirmed the decrease in Curie temperature with the increase in Zn concentration. Polina Yaseneva *et al.* [28] also reported the effect of Zn substitution on the Curie temperature and found that Zn substitution decreases the Curie temperature from around 440 °C for the pure sample to 180 °C for the

doped sample with $x = 0.5$. Coppola, P. *et al.* [29] found structural and morphological changes in the sample with respect to zinc substitution. The shapes of the nanoparticles vary from spheres, cubes to octahedrons as per TEM analysis. In the nanoparticle composition, the proportions of Zn and Co strongly influence the properties of hysteresis loop. G. Vaidyanatham *et al.* [30] studied $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ nanoparticles and showed that the samples were cubic spinel with average crystallite size varying from 6.92 to 12.02 nm with the increase of zinc concentration; therefore, the lattice constant increases with zinc. Magnetic factors such as M_s , M_r and H_c were found to reduce with the increase of zinc content. M. T. Jamila *et al.* [31] studied the effect of Zn content on the structural parameters and confirmed single-phase face center cubic structure. They revealed that the crystallite size was found in the range 30-70 nm while lattice parameter and X-ray density decrease with the increase of Zn concentration. Linear growth of unit cell parameter with the increase in zinc concentration was investigated by Swati Tapdiya *et al.* [32] due to the larger ionic radii of Zn^{2+} ion and they also found an increase in saturation magnetization owing to the variation of exchange interaction among the tetrahedral and octahedral sites. Sonal Singhal *et al.* [33] synthesized zinc-substituted cobalt ferrite *via* sol-gel method, characterized using IR spectroscopy, TEM, X-ray diffractometry (XRD) and magnetic measurements. It is observed that the lattice parameter and X-ray density increase with increasing Zn concentration. The saturation magnetization first increases and then decreases. C. Nlebedim *et al.* [34] studied the temperature dependence of structural and magnetic properties of zinc-substituted cobalt ferrite from 50 K to 300 K and found no observable changes in the crystal structure. At all temperatures, magnetization increased with x , indicating A-site Zn substitution. An inverse relation was seen between magnetic susceptibility and coercive field, while a direct relationship was seen between coercive field and magnetocrystalline anisotropy coefficient.

Mg Substitutions

Vithal Vinayak *et al.* [35] & Shaik Jesus Mercy *et al.* [36] successfully synthesized the nanocrystalline $\text{Co}_{1-x}\text{Mg}_x\text{Fe}_2\text{O}_4$ by sol gel auto-combustion method and showed the formation of

single-phase cubic spinel structure by X-ray technique. The lattice constant was found to decrease with increasing Mg^{2+} concentration. The particle size of the sample was obtained in the range of 11-24 nm by Debye-Scherrer formula. The tetrahedral and octahedral bond lengths and edges decrease as Cu^{2+} content increases. In overall, the substitution of magnesium in cobalt ferrite influences the structural properties. V. V. Dhole *et al.* [37] synthesized the nanocrystalline $Co_{1-x}Mg_xFe_2O_4$ by sol-gel auto-combustion technique. X-ray diffraction results showed the formation of single-phase cubic spinel structure. The lattice constant is found to decrease with increasing Mg^{2+} concentration. The particle size of the sample calculated using Debye-Scherrer formula was obtained in the range of 12-32 nm. The average grain size determined from scanning electron microscopy technique is of the order of 45-66 nm. The signs of modification in structural and magnetic properties due to substitution of Mg for Ni in $Co_{0.5}Ni_{0.5}Fe_2O_4$ sample were also observed by S.V. Bhandare *et al.* [38].

Conclusion

In the present paper, different cation substitutions like Ni^{2+} , Zn^{2+} , Cu^{2+} , Mg^{2+} ... etc. in cobalt ferrite and their significant effects on

structural, dielectric and magnetic properties of the ferrites have been intensively studied. Hysteresis study of the curve shows enrichment in coercivity and remanence, which is attributed to the shift from multidomain to single-domain environment and reduction in saturation magnetization with the substitution of divalent metal ions into cobalt ferrite materials. The variation of dielectric properties, $\tan \delta$ and ac conductivity with frequency reveals the dispersion relation due to Maxwell-Wagner (two-layer models) type of interfacial polarization that decreases with increasing the frequency of the applied field. The lattice parameter is found to increase with increasing divalent metal ion doping concentration into the cobalt ferrite crystals. Porous and agglomerated morphology of the bulk sample was displayed in the scanning electron microscopy (SEM). The dispersion behavior of the doped sample shows high resistance of cobalt-nickel ferrite with ferromagnetic nature. X-ray diffraction pattern of the doped sample reveals single-phase cubic spinel structure. It is also seen that increment or decrement in these properties depends on the dopant concentration, the type of dopant, sintering temperature and also on the method of preparation of the sample.

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