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A REVIEW: EFFECT OF SUBSTITUTION ON MAGNETIC PROPERTIES OF CALCIUM HEXAGONAL FERRITES

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Abstract

Calcium hexaferrites are a subject of great interest due to their promising applications and narrow range of particle distribution. It offers good magnetic, electrical and magneto-optical properties with thermal and chemical stability along with low cost. The present review investigates the effect of divalent and trivalent ion substitution on Magnetic properties of Calcium Hexagonal Ferrites. The magnetic characteristics namely saturation magnetization (Ms), remanence (Mr) and coercivity (Hc) change with substitution of suitable divalent or trivalent ions occupying sublattice spin-up and spin-down sites into the crystal of Calcium hexagonal ferrites. In the present work we have studied calcium hexaferrites containing different types of substitutions. From the technological point of view substitution of different types of ions was found to be an effective way of improving and enhancing the quality of material and their magnetic characteristics which can lead to various potential applications.

Introduction:-

Hexagonal Ferrites commonly known as hexaferrites are hard magnetic materials commonly referred to as Magnetoplumbites. These magnetic materials are reported to exhibit promising properties for nanomaterials synthesis leading to microwave absorption devices, antenna materials, recording media, permanent magnets and permanent magnets. [i,ii]Hexaferrites are hexagonal or rhombohedral ferromagnetic oxides with formula $MFe_{12}O_{19}$, where M is anelement like Barium, calcium, Lead or Strontium. In these types of ferrites, oxygen ions have closed packedhexagonal crystal structure. They are used at very high frequency. The structure and lattice of hexagonal ferrites isvery similar to the spinel structure with closely packed oxygen ions, but there are also metal ions at some layers withthe same ionic radii as that of oxygen ions. Hexagonal ferrites have larger ions than that of garnet ferrite and areformed by the replacement of oxygen ions. Most of these larger ions are barium, strontium or lead.[iii]Hexagonal ferrites are known to possess good chemical stability, enhanced magnetic loss property, large to moderate magnetization and high Curie temperature. [iv]The space group of hexagonal ferrites is $P6_3/mmc$ including layers of iron atoms, oxygen or (barium/ calcium/strontium)- oxygen in the structure occupying one tetrahedral, one

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bipyramidal and three types of octahedral sites. [v]In the hexagonal structure the tetrahedral sites is $4f_1$, bipyramidal site is 2b and octahedral sites are $4f_2$, 12k and 2a.[vi,vii] Hexaferrites are classified into X-type ($Ca_2Me_2Fe_{28}O_{46}$), Z-type($Ca_3Me_2Fe_{24}O_{41}$), W-type ($CaMe_2Fe_{16}O_{27}$),Y-type ($Ca_2Me_2Fe_{12}O_{22}$) and Mtype($CaFe_{12}O_{19}$). This classification is based on the crystal structure and the chemical formulae. Magnetic and structural properties of hexagonal ferrites vary with change in synthesis methods, temperature, dopants and chemical composition.[viii,ix,x,xi,xii,xiii,xiv,xv]The focus is now on synthesizing hexaferrites at the nanoscale having unique properties due to modified electronic structure closed to isolated atom.[xvi]Hexaferrites make interesting study because of their unique magnetic and electrical behavior.[xvii]Hexagonal ferrites find potential applications in data storage devices, high density magnetic recording devices, Microwave absorption for radars, permanent magnets and electronic circuits.[xviii]Barium hexaferrite gives very high uniaxial magnetocrystallilne anisotropy, good magnetic properties and high stability. [xix,xx] Calcium has the same electronic configuration and lies in the same periodic table group as Srontium and Barium. But Calcium substituted hexaferrites are not investigated much although they have enhanced properties and require reduced sintering temperautes. Apart from having similar electronic configuration as barium, lead and strontium, calcium is the most abundant element on earth and comparatively less cheaper.[xxi]Hence, research and study on Calcium substituted Hexagonal ferrites seems interesting and much needed.[xxii]Calcium W- type hexaferrite with formula $CaM_2Fe_{16}O_{27}$;where M can be a divalent ion like Mg, Mn, Fe, Sr, Co, Ni, Cu or Zn; when subjected to change in applied magnetic field or temperature has spin reorientation transitions between various anisotropy configurations[xxiii]. The change in chemical composition can alter the transition temperatures in W-type hexagonal ferrite. Few of the spin reorientation transitions between various anisotropy configurations are of first order mainly which leads to the use of W-type calcium hexagonal ferrites in Magnetic Refrigeration at room temperature. [xxiv]The crystal structure M –type Hexagonal ferrite ($CaFe_{12}O_{19}$) are similar. Both have alternating stacks of R blocks (as shown in Fig.1) and Spinel blocks in the direction of c-axis.

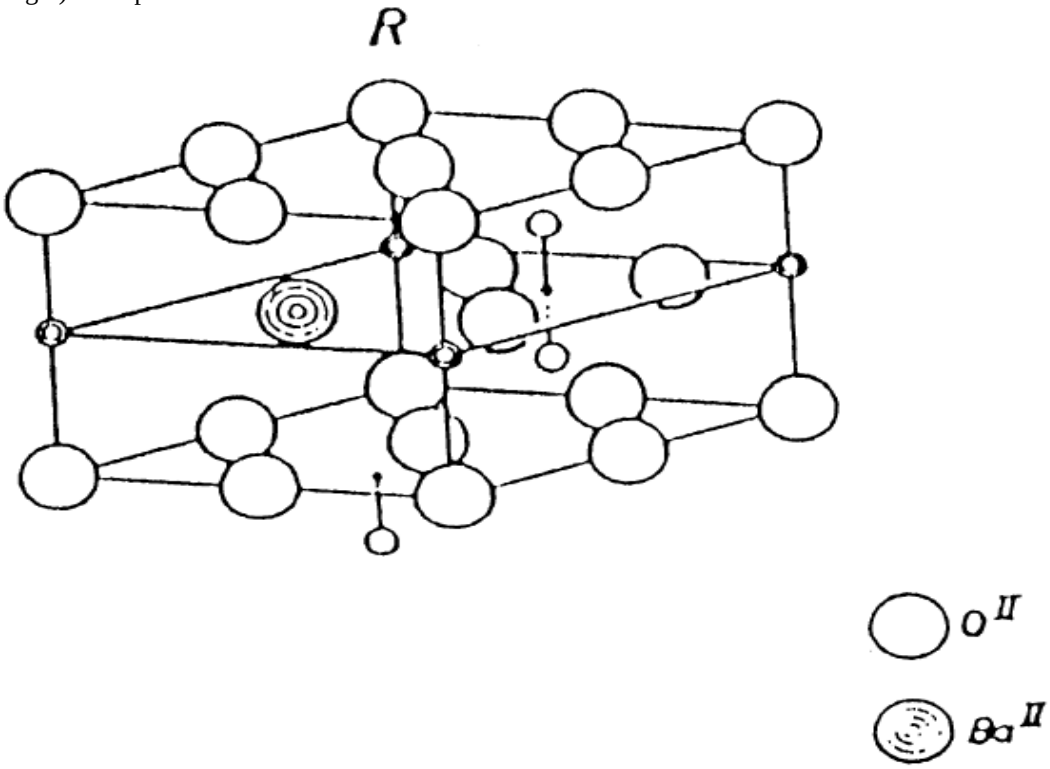

Fig. 1:- Diagram of R-block.[xxv]

In Calcium M-type hexagonal ferrite, a total of 24 Fe^{3+} ions occupy the sites 2a, $4f_1$(tetrahedral site), $4f_2$, 2b and 12K(trigonal bipyramidal site). It is observed that the Fe^{3+} ions located at 12k, 2aand 2b sites have up spins and those at 4f1 and 4f2 sites have got the down spins. When any other trivalent metal ion or a combination of divalent and tetravalent metal ions replaces the Fe3+ ion partially, the properties of the Ca-M ferrite change which changes their magnetic behavior. [xxvi]There are 64 ions in the structural unit cell on total 11 sites of symmetry. The magnetic of these ferrites is due to several magnetic interactions in the lattice because of various site distributions. Calcium hexaferrites belong to magneto plumbite family having ferrimagnetic nature with a preferred direction of

magnetisation. There are two lattice parameters 'a' and 'c' which denote the width and height of the hexagonal structure respectively. The magneto crystalline anisotropy lies along the 'c' axis and is often parallel to the 'c'axis when the field is applied. [xxvii]Calcium hexagonal ferrites are of great interest of their good electrical, magnetic, structural, magneto-optical, magnetic and thermal properties. They are chemical stable materials with narrow size distribution of their particles. By substituting the non- magnetic trivalent of divalent ions into the lattice, the magnetic moments (of Fe^{3+} atomsRSR*S* configuration) can be disturbed leading to change in magnetic properties.[xxviii] Single domain Calcium Hexaferrites possess large coercivity(H_c) values inherently, coercivity values can be further enhanced by substituting Fe^{3+} ions with other suitable paramagnetic or diamagnetic cations like cobalt.[xxix] The best synthesis technique for synthesizing very fine, homogeneous powder with single domain involves various parameters. It includes low calcination temperature, short reaction time and energy efficiency.[xxx] Calcium Hexaferrites have potential industrial applications owing to their unique properties. Hence an effort to study the Structural and Magnetic properties of Calcium Hexaferrites is made in this review.

Magnetic Properties of Calcium Hexaferrites:-

It is seen that the magnetic hysteresisloops of calcium substituted hexagonal ferrite are dependent on the crystalline anisotropy, synthesis methods, size of the grains, composition and sintering temperature.[xxxi] The magnetic properties of hexagonal ferrites vary with the substitutions of divalent or trivalent ions into the lattice as well as on the site occupancy of Fe^{3+} ions in the sublattice.[xxxii]Table 1 denotes the magnetic and structural properties of the magnetic sublattices in the crystal structure of hexagonal ferrite. [xxxiii]

Sublattice	Sites	Block	Number of ions	Spin
2a	Octahedral	S	1	UP
2b	Pseudo-tetrahedral	R	1	UP
4f1	Tetrahedral	S	2	DOWN
4f2	Octahedral	R	2	DOWN
12k	Octahedral	S	6	UP

Table 1:- Magnetic Sublattices and Spins. [26]

The coercivity, saturation magnetization and remnant magnetization decide the application of the ferrites. Ferrites having high value of coercivity find applications in hard magnets and energy storage devices. Low coercivity materials are used in soft magnets and recording media of information. [xxxiv]Hexagonal ferrites have uniaxial magnetocrystalline anistrophy which is the source of coercivity. Magnetic properties are of great importance in order to tailor the properties of the ferrites for potential applications.[xxxv]The observations suggest that Calcium Ferrite exhibit high values of coercivity in single domain phase. This is because of the substitution of Co^{3+} ions without changing the magnetism much significantly.[29]Magnetocrystalline anistrophy, sintered grain size and magnetic moment per unit cell influence the magnetic properties of ferrite magnets.[xxxvi] Hexagonal ferrites have any easy c-axis of magnetization which is perpendicular to the bottom plane. The molding processes accompanied by application of external magnetic fields can facilitate manufacturing of ferrite magnets by aligning the direction of easy magnetization. As per the processes of molding, when the powdered ferrite is shaped by applied magnetic field then the magnet formed is called as dry press molding and when the ferrite slurry is applied with magnetic field then it is wet press molding.[xxxvii]The wet press molding method is carried out by mixing the ferrite powder with water or other solvent then drying the slurry. The slurry makes the ferrite powder move easily aligning it in the direction of applied external magnetic field molding or pressing it further. This leads to enhancement in the magnetic properties of the ferrite.[36] In W-type Hexaferrites, the variable site distributions accompanied by various sites of oxidations leads to magnetic variations. [xxxviii]The Gorter collinear spin model suggests that the magnetic structure in ferrites having three parallel sublattices (2a, 2b and 12k) and two anti-parallel sublattices (4f1 and 4f2) undergo super-exchange interactions through O^{-2}ions. Thus, the magnetic characteristics of ferrites can be improvised by substituting divalent or trivalent ions partially.[xxxix] Table 2 denotes the magnetic parameters like saturation magnetization, residual magnetization and coercivity of various substituted calcium hexaferrites.

Sample Number	Compounds	M_s (emu/gm)	M_r (emu/gm)	H_c (Oe)	M_r/M_s	Reference
1	$\mathbf{CaFe_{12}O_{19}}$	0.4975	0.1495	875	0.3005	(a)
2	$\mathbf{Ca(Zr\text{-}Co)_2Fe_8O_{19}}$	0.3055	0.0305	250	0.0998	(a)
3	$\mathbf{Ca(Zr\text{-}Co)_4Fe_4O_{19}}$	0.1985	0.0265	125	0.1335	(a)
4	$\mathbf{CaSr_2Fe_{16}O_{27}}$	28.41	15.77	5217.3	0.5550	(b)

5	**$CaMg_2Fe_{16}O_{27}$**	6.857	0.048	20.76	0.0070	(c)
6	**$Ca(Co\text{-}Sn)Fe_{10}O_{19}$**	328.79	63.21	479	0.1922	(d)
7	**$Ca(CoZr)Fe_{10}O_{19}$**	23.969	8.336	717	0.3477	(e)
8	**$Ca(ZnZr)Fe_{10}O_{19}$**	26.6	1.45	26	0.004	(f)
9	**$CaCr_{10}Fe_2O_{19}$**	3.9884	0.60436	49.203	0.1515	(g)
10	**$CaAlFe_{11}O_{19}$**	67.765	28.009	3386.1	0.4133	(h)

Table 2:- Magnetic Parameters for Calcium Hexaferrites.

(a) A. D. Deshpande, K. G. Rewatkar, V. M. Nanoti, Materials Today: Proceedings 4 (2017) 12174–12179.

(b) C. L. Khobaragade, S. B. Deshpande, S. V. Soni and K. G. Rewatkar, I J R B A T, Issue (3), Vol. II, May 2015: 51-56.

(c) P S Sawadh and DKKulkarni, Bull. Mater. Sci., Vol. 24, No. 1, February 2001, pp. 47–50. © Indian Academy of Sciences.

(d) Sharad N. Sable, Kishor G. Rewatkar, Vivek M. Nanoti, Materials Science and Engineering B 168 (2010) 156–160.

(e)J.N. Christy, K.G. Rewatkar, P.S. Sawadh, Materials Today: Proceedings 4 (2017) 11857–11865.

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(g) B.C. Manjunatha, K.M. Rajashekara, Materials Today: Proceedings, https://doi.org/10.1016/j.matpr.2020.05.361.

(h) A.P. Bhat, S.J. Dhoble, D. K. Sahu, K. G. Rewatkar, Materials Today: Proceedings 5 (2018) 22181–22187.

With reference to table 2, the values of M_s and M_r is seen to decrease while Coercivity $H_c$increases with substitution of Zirconium and Cobalt in calcium M-type hexaferrite $CaFe_{12}O_{19}$(refer sample numbers 1, 2 and 3 in table 2). Furthermore, with increase in the concentration of zinc and cobalt, the values of M_s and M_r decrease and H_c increases even more. This decrease in the values of M_s and M_r is due to zinc and cobalt occupying the spin down ($4f_1$) and also in spin up (2a, 2b and 12k) sublattices of Fe^{3+} ions. Increase in the concentrations of zinc and cobalt lowers the magnetocrystalline anisotropy which results in decreased coercivity. As per M.V. Cabanas et. al, the Fe^{3+} ions at 12k and $4f_2$ sites contribute majorly to the magnetocrystalline anisotropy and replacement of Fe^{3+} ions with Zr or Co ions at those sites leads to decrease in anisotropy and coercivity.[xl, xli]Zirconium ions prefer octahedral spin up sites and cobalt ions have affinity for tetrahedral spin down sites. Fe^{3+} ions when replaced by these ions, Zr^{4+} ions go to 2a, 2b or 12k sites whereas Co^{2+} ions go to $4f_1$ sites. These results in decrease in the net magnetization as spin up orientations contribute to net magnetization whereas spin down orientations don't. [xlii] Hence, substitution of Zirconium ions majorly affects the values of M_s, M_r and H_c.[xliii]With substitution of strontium in calcium W- type hexaferrite in $CaSr_2Fe_{16}O_{27}$, the coercivity , saturation magnetization and residual magnetization gets enhanced due to decreasedas seen in table 2. Magnesium substitution in Calcium W-type ferrite $CaMg_2Fe_{16}O_{27}$results in enhancement in magnetization and reduced coercivity. Diamagnetic Mg^{2+} ions prefer tetrahedral spin down sublattice sites which lowers the values of H_C. Further, this is because the magnetic moment of the minority sub lattices is reduced which increases the total magnetic moment.[25] M –type calcium hexaferrite synthesized compound $Ca(Co\text{-}Sn)Fe_{10}O_{19}$has enhanced saturation magnetization values with Co–Sn ions substitution. This can be credited to Co-Sn ions replacing Fe3+ ions in the spin down state. Furthermore, the synthesis of hexaferrite in the nanoscale thereby reducing the particle size improves the magnetic properties. [35] Figure 2 shows the B-H curve of $Ca(Co\text{-}Sn)Fe_{10}O_{19}$sample indicating magnetic properties very useful in military and aerospace applications.

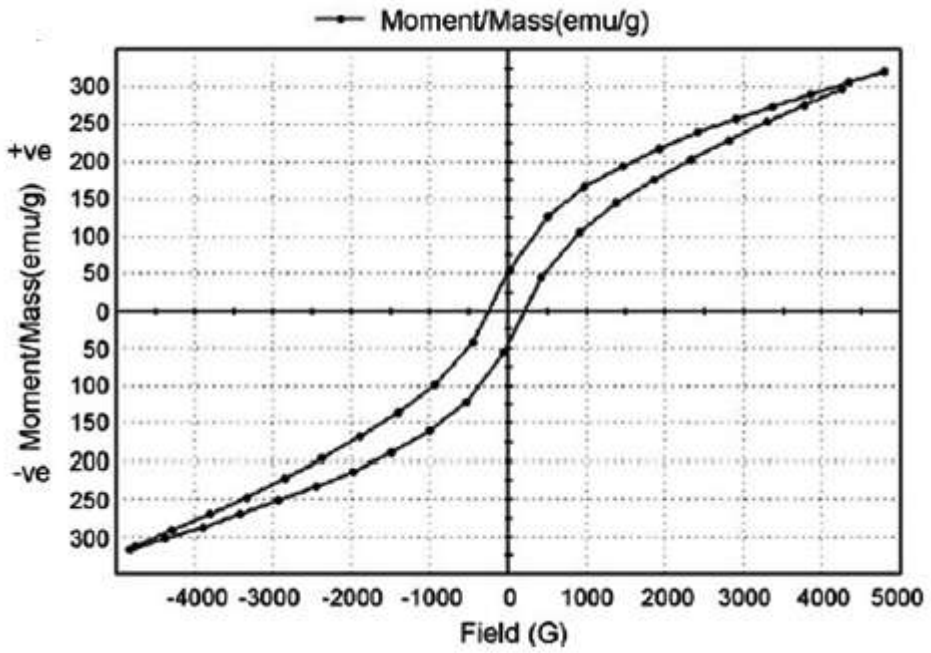

Fig. 2:- Hysteresis Loop of M- type calcium hexaferrite $Ca(Co\text{-}Sn)Fe_{10}O_{19}$.[35]

Co-Zr substituted M-type calcium hexaferrites with formula $Ca(Co\text{-}Zr)_xFe_{12-2x}O_{19}$ (x = 0.2, 0.4, 0.6, 0.8, 1.0) shows increase in values of saturation magnetization $M_s$from 5.827 emu/g for x = 0.2 to 23.969 emu/g for x = 1 as shown in Figure 3. Zr^{4+} ions being nonmagnetic in nature prefer distribution in both tetrahedral and octahedral sites and are responsible for stability of the lattice further.[xliv] Co^{2+} ions have preference towards $4f_1$ and $4f_2$ spin down sites. Both Zr^{4+} and Co^{2+} ions occupy the Fe3+ sites which leads to increase in M_s.[xlv,xlvi]

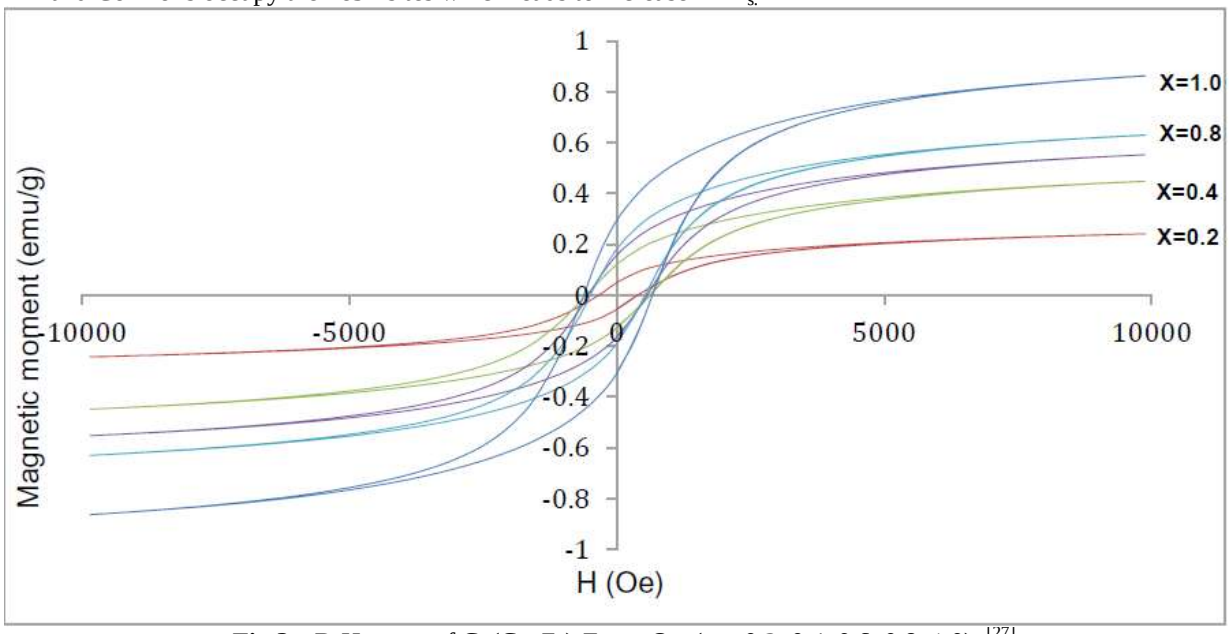

Fig.3:- B-H curve of $Ca(Co\text{-}Zr)_xFe_{12-2x}O_{19}$ (x = 0.2, 0.4, 0.6, 0.8, 1.0). [27]

Figure 3 reveals that the saturation magnetization M_s and residual magnetization Mr values increase with the concentration. The hysteresis loop suggests that the nanoparticles have a soft magnetic nature useful in magnetic recording media and high frequency applications. If in $Ca(CoZr)Fe_{10}O_{19}$ M-type Calcium hexaferrite, Zn replaces Co which leads to a slight increase in M_s and reduction in M_r and H_c as seen in table 2. Zinc is nonmagnetic ion like

zirconium and shows preference for tetrahedral spin down site $4f_1$. Residual Magnetization decreases to a value closer to zero and saturation magnetization increases by slight value. Magnetocrystalline isotropy gets affected negatively due to the presence of nonmagnetic Zr^{4+} and Zn^{2+} ions at 4f1 sites hereby reducing the coercivity values. [18] The sample synthesized with Zinc and Zirconium exhibits transition from hard ferrite to soft ferrite nearing superparamagnetic nature.

Conclusion:-

Calcium Hexaferrites have appealing magnetic properties at low cost. Calcium Hexaferrites are not investigated intensely as compared to Barium Hexaferrites. It has been observed that the magnetic properties like remnant magnetization, coercivity and saturation magnetization can be tuned by adjusting the amount of the non magnetic substitution for iron. The samples reviewed in the present study should be further investigated with different concentrations of doping to seek their potential applications in recording devices , permanent magnets and microwave absorption properties by obtaining ferrites with enhanced saturation magnetiozation and low values of coercivity**.**

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