

Structural, Electrical and Magnetic Properties of La/Al Substituted Nano Calcium Hexaferrites prepared by Sol–Gel Auto-Combustion Method

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Abstract: The Samples of Calcium Nano Hexaferrites $\text{CaLa}_1\text{Fe}_{11}\text{O}_{19}$, $\text{CaLa}_1\text{Al}_1\text{Fe}_{10}\text{O}_{19}$ and $\text{CaAl}_1\text{Fe}_{11}\text{O}_{19}$ were prepared by Sol-gel auto combustion technique using urea as a fuel. The powders were characterized with X-ray diffraction analysis, revealed the formation of mono phase M type hexagonal ferrite with space group $P6_3/mmc$ (no194). The SEM and TEM were studied, which confirmed that the sample exhibit relatively well defined hexagonal grains with average particle size in the range 43 nm to 31nm. The values of the lattice parameters supports this confirmation. DC conductivity of bulk samples have been investigated as a function of reciprocal temperature. The measurements were carried out at constant frequency (100Hz). The Activation energies were calculated at both ferrimagnetic and paramagnetic regions. The enhanced resistivity of aluminum doped calcium hexaferrite is a prospective application in microwave devices. The magnetic measurements were carried by using Vibration Sample Magnetometer (VSM). Results of VSM studies shown that, the Al doped sample shows rise in saturation magnetization, significant increase in the coercive field and magnetic remenance. Increase in coercivity and retentivity illustrate that they are hard ferrite materials. Thus the magnetic characters of the Al doped samples are superior than that of La and La-Al doped calcium hexaferrites. It is known that the nano-hexaferrites with such special magnetic properties are highly useful in Data storage applications.

Keywords: M type Nano-hexaferrite, Sol-gel Auto-combustion, XRD, VSM, D C Conductivity, Activation energy etc

1. Introduction

Nano hexagonal ferrites continued to grab the attraction of many researchers due to their unique structural, electrical and magnetic properties and proved to be a promising candidate for various technological applications [1] such as permanent magnets, microwave absorber, plastoferrite, and magnetic recording media. The unique property of nanostructure materials are due to changed electronic structure closed to that of isolated atom or molecule [2]. Hexagonal ferrites form a large family of ferrimagnetic materials. The Calcium ferrite having general formula $\text{CaFe}_{12}\text{O}_{19}$ is one of the most important hard magnetic materials, widely used for above applications [3].

The nanosized ferrite particles can be prepared by a large number of methods like co-precipitation of hydroxides, hydrothermal synthesis, sol gel synthesis, [4,5] etc. The properties of the ferrites are strongly influenced by their composition and synthesis method. In the present paper we have reported the results of compounds synthesized by sol gel auto combustion method as this route provides a good control over the particle size and produce samples in relatively short time [6].

There are many reports on replacement of Fe^{3+} ion in Ba/Sr hexagonal ferrites by different substitution like trivalent cations viz. Zn^{3+} , Gd^{3+} , Co^{3+} or by the combination of divalent - tetravalent cations like Zn^{2+} - Ti^{4+} , Co^{2+} - Ti^{4+} , Co^{2+} - Nd^{4+} , Zn^{2+} - Mg^{4+} , Zn^{2+} - Sn^{4+} , Zn^{2+} - Ti^{4+} , Co^{2+} - Zr^{4+} , Ni^{2+} - Zr^{4+} [7-14]. It has been observed that the Calcium Hexaferrites remain less attempted, hence in this paper effect of doping of La^{3+} , Al^{3+} and La^{3+} - Al^{3+} substituted ions

on structural, electric and magnetic properties of such prepared samples were discussed in detail.

2. Experimental Methodology

Analytical grade reactive nitrates such as $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, $\text{La}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and urea were used stoichiometrically to prepare the ferrite compositions ($\text{CaLa}_1\text{Fe}_{11}\text{O}_{19}$, $\text{CaLa}_1\text{Al}_1\text{Fe}_{10}\text{O}_{19}$, $\text{CaAl}_1\text{Fe}_{11}\text{O}_{19}$). The specified amount of metal nitrate dissolved into distilled water and heated at the temperature of 80°C for about 3 hr with urea used as the fuel, which gives requisite energy to initiate exothermic reaction [15]. The gel formed were fired in digitally controlled microwave oven, yielding powder (ash). The resulting powder were followed by grinding for about 5 hours in pestal mortar to get ultra-fine homogeneous crystallize nanoparticles powder of samples and then heated in electric furnace up to 800°C for about 8 h by increasing the temperature slowly ($100^\circ\text{C}/\text{hr}$) and then cooled at the same rate. Finally the samples powder of all the specimens were subjected to the structural and magnetic characterization and the crack free pellets of corresponding samples were used for electrical measurements.

The phase structure of hexaferrite powders were investigated by (Philips diffractometer PW 3710) X ray diffraction Cu-K α radiation ($\lambda = 1.54060\text{\AA}$). The Phase identification was executed using a X powder software and X-ray diffractograms were also plotted with X-Powder software. The structure morphology was identified using SEM instrument Cameca SU – SEM Probe and the TEM studies of prepared samples was done by using TEM Model

Philips CM – 200. The DC electrical properties such as conductivity, resistivity, activation energy and transition temperature of synthesized ferrite samples were studied in the temperature range 300K to 700K by using four probe method (Wyne Kerr 6500B model).

The magnetic measurements on all the samples have been carried out by using high field hysteresis loop technique at room temperature. The magnetic hysteresis loops (magnetization (M) vs. applied field (H) curves) for the samples has been measured using a Vibrating Sample Magnetometer (VSM) instrument, (14 T model) with a maximum field of 20000 Oe. Characterization were focused on Saturation magnetization M_s , Coercivity (H_c), Remanence magnetization (M_r),

3. Results & Discussion

X-ray diffraction studies of investigated samples (fig. 1) confirm the formation of M-type calcium hexaferrites with the space group $P6_3/mmc$ (No 194) the JCPDS Files had been referred to for all the peak position identification. In XRD pattern absence of secondary peaks confirms the formation of purely crystallized single phase [16]. Moreover, the intensity of the peaks becomes stronger and narrower, indicating a better structural quality of materials [17]. The average crystallite size of the powders was measured by X-ray line-broadening technique employing the Debye-Scherrer formula [18] reveal size in nanometer scale.

The compositional dependence of the lattice parameters, bulk density (d), X-ray density (d_x), porosity (P) and

average particle size of all the samples are listed in Table 1. The recorded lattice parameters values lie within the range ($a = 5.8-5.9 \text{ \AA}$ and $c = 22-23 \text{ \AA}$) of pure magnetoplumbite phase of hexaferrites. The M-type hexagonal structure can be assumed if lattice parameter ratio (c/a) lies between 3.80 - 3.98. In all synthesized compounds this ratio remains fairly constant at 3.81, as the ratio of lattice parameters lies below 3.98 [19] which confirms that prepared mixed Ca-M ferrites reveals the hexagonal structure.

Composition analysis of samples shows that the grains are uniform and roundish hexagonal in shape having a size in the range 31 to 43nm respectively, which was also confirmed by SEM and TEM results, (fig. 2) and (fig.3). In TEM results, electron diffraction pattern shows ring pattern superimposed with bright spots. It reveals the polycrystallinity of individual crystallites and also confirming the formation of calcium hexaferrite phase [20]. It is observed that the lattice parameters are smaller for the content Al^{3+} as compared to the contents La^{3+} and $La^{3+}-Al^{3+}$ respectively. This is due to small ionic radius of Al^{3+} (0.53 \AA) compared to that of La^{3+} (1.03 \AA). Similar behavior was reported by Prakash et al for Me substituted calcium ferrite. (where Me = Al, Cr and Co) [21] and Co-Al substituted calcium ferrite by Rewatkar et al. [22]. In the investigated compounds due to comparatively small lattice constant and particle size, interaction and solubility between Fe^{3+} ion and Al^{3+} ion is higher than other trivalent ion. The variation in the densities shows that the actual (experimental) density d values were found to be in general less than those of X-ray density d_x (theoretical density) which are expected due to presence of unavoidable pores created during firing [23].

Table 1: Structural data

Compound	Lattice parameters		Axial ratio (c/a)	Cell volume (\AA^3)	Bulk density, (d) g/cm ³	X ray density (d_x) g/cm ³	Porosity %	Particle size D nm
	a (\AA)	c (\AA)						
CaLa ₂ Fe ₁₁ O ₁₉	5.811	22.15	3.81	647.15	2.5851	5.4645	52.70	43
CaLa ₁ Al ₁ Fe ₁₀ O ₁₉	5.800	22.88	3.81	643.82	2.6044	5.5083	52.72	38
CaAl ₁ Fe ₁₁ O ₁₉	5.805	22.11	3.80	645.38	2.6101	5.0505	48.34	31

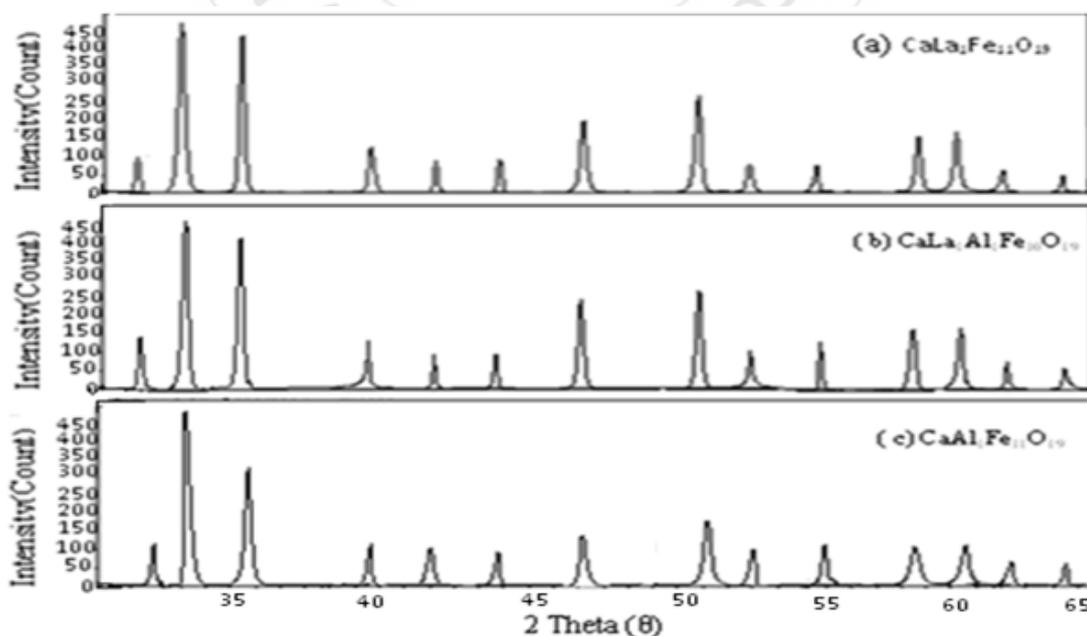


Fig. (1): XRD Pattern of synthesized Ca Substituted Hexaferrite

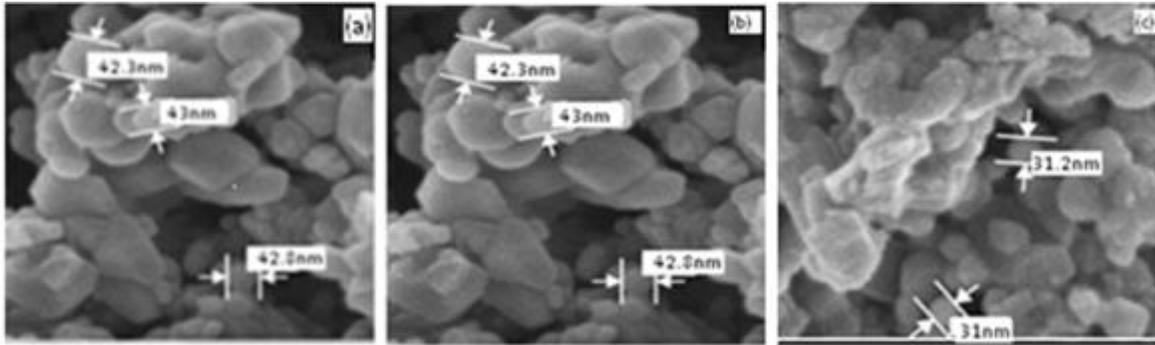


Fig (2): SEM Micrographs of Synthesized compounds

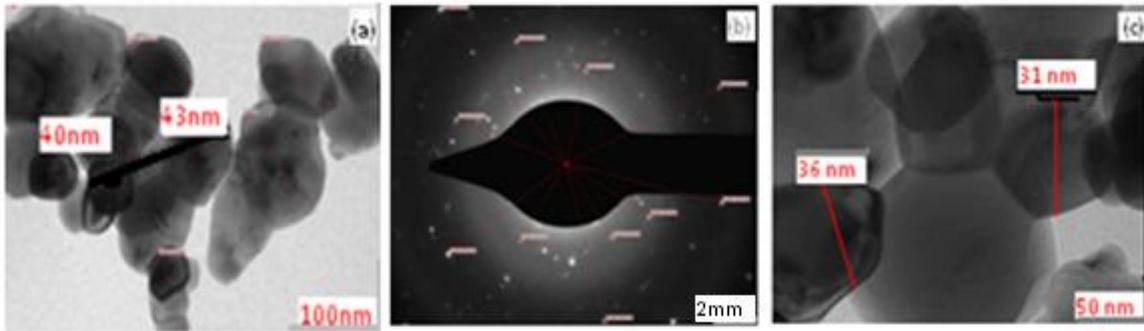


Fig (3): TEM Micrographs of Synthesized compounds

The variation of DC electrical conductivity synthesized samples in terms of log σ with inverse absolute temperature

for all samples are represented in figure (4), and values of its various electrical parameters of are listed in table 2.

Table 2: Electrical parameters

Compound	Activation energy E_g in eV		Resistivity at Room Temperature (Ω -cm)	Conductivity at Room temperature (Ω -cm) ⁻¹	Transition Temperature k
	Paramagnetic	Ferrimagnetic			
CaLa ₁ Fe ₁₁ O ₁₉	0.59	0.28	1.76x10 ⁷	5.69x10 ⁻⁸	565
CaLa ₁ Al ₁ Fe ₁₀ O ₁₉	0.35	0.21	2.65x10 ⁷	3.77x10 ⁻⁸	540
CaAl ₁ Fe ₁₁ O ₁₉	0.91	0.25	6.80x10 ⁷	1.47x10 ⁻⁸	525

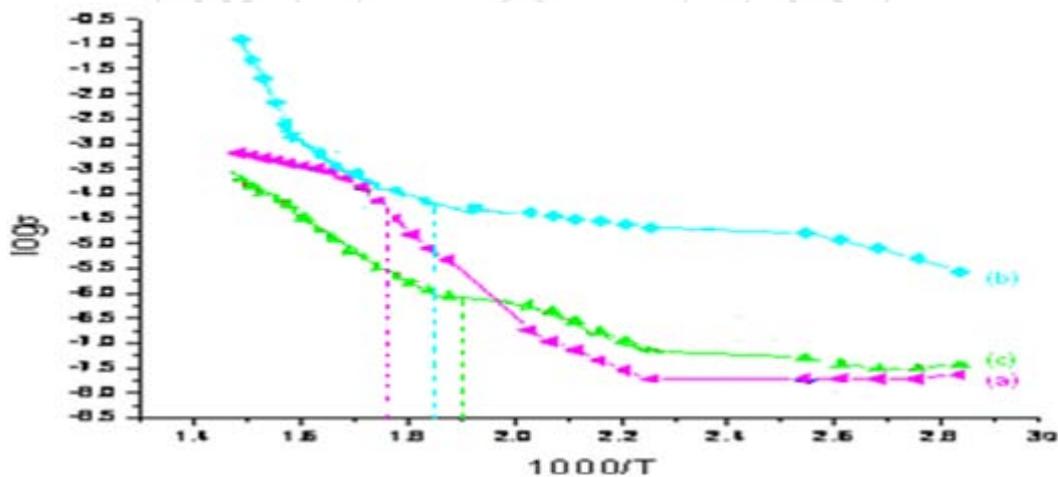


Figure 4: Temperature dependence of conductivity of samples (a) CaLa₁Fe₁₁O₁₉ (b) CaLa₁Al₁Fe₁₀O₁₉ (c) CaAl₁Fe₁₁O₁₉ at constant frequency

It is observed that the values of activation energy for all samples are above 0.2 eV clearly indicate the polaron hopping in the system [24]. The resistivity increases rapidly with doping of Al⁺³ ions for Fe⁺³ ions. The increase of the electrical resistivity for the investigated system gives a promising property for communication systems as the eddy current loss decreases [25]. Variation in the resistivity of

these ferrites can be explained on the basis of Verwey model, where in the conduction is due to electron hopping exchange, the conduction in ferrite at room temperature is due to the impurities where as at high temperature it is due to polaron hopping. According to Verwey, Hopping mechanism in electronic conduction in ferrites is mainly due to electron hopping exchange between ion (Fe⁺³ + e⁻ → Fe⁺²) of same

element, present in more than one valence state, distributed randomly over different lattice sites [26,27]. From the plots it is seen that, the conductivity increases with increasing temperature for all samples this is just like normal semiconductor behavior. Similar results was reported for Cr³⁺ substituted ferrite by S.A. Masti [28]. The values of log σ decays almost linearly with increasing reciprocal temperature up to certain value called transition temperature (T_t), where change in slope (kink) occurs in plots. The temperature where kink is observed for the various composition of ferrites is in neighborhood of magnetic transition temperature (Curie temperature T_c). This reveals that the kink observed in the samples undertaken in this research module can be attributed to the magnetic order to disorder phase. The lower activation energy in the ferrimagnetic region is attributed to the magnetic disordering [29], Similar results were reported for Al substituted Ca₂Y hexaferrite powder by P. R. Moharkar et al. [30] and Co-Ferrite by R. A. Bugad et. al. [31]. The cause of increase in activation energy in disordered state while increase in temperature may be due to creation of more cations and development of oxygen vacancies. Finally it can be concluded that concentration of oxygen vacancies is an important factor for DC electrical resistivity [32].

In the present work, Typical hysteresis loops of the samples are shown in Fig.5 data obtained were tabulated in Table 3. shows magnetic moment found in the range between 34 and 100 emu/g, retentivity between 7 and 29 emu/g, and the coercivity between 190 and 3400G for all the samples confirming the good quality of samples. It is observed that sample CaAl₁Fe₁₁O₁₉ shows rise in value for magnetic saturation (M_s), and significant increase in values for retentivity (M_r), and coercivity (H_c). The observed values for magnetic moment, retentivity, and coercivity showed high solubility and interaction between Al³⁺ as compared to other dopents. The utility of a magnetic material for a specific application depends on its relevant properties like coercivity. Hard magnet demands high coercivity which is essential property for energy storage applications while soft magnet demands low coercivity which is requirement of information storage recording media [33]. In Al³⁺ doped CaAl₁Fe₁₁O₁₉ sample high coercivity (3386 G) is observed,

which might be due to uniaxial magneto crystalline anisotropy along c-axis [34] and there is remarkable fall in coercivity with the substitution of La³⁺ ions (192.85 G). The replacement of Fe³⁺ ions by substituted ions at 12k and 2b sites underlies the fast reduction in coercivity. These two sites contribute to a large anisotropy field, Mendoza-Surez [35]. The lowest value of coercivity for the La³⁺ ion doped samples CaLa₁Fe₁₁O₁₉ may be due to the replacement of iron ions from 12k site to results in a reduction in the magneto-crystalline anisotropy [36]. This consequently decreases the coercivity for the samples of CaLa₁Fe₁₁O₁₉ hexaferrite. Similar type of variations have reported by Fang et al. (2005) [37], The lowest value of coercivity for CaLa₁Fe₁₁O₁₉ may be due to the fact that a part of the particles in the sample do not have a perfect hexagonal shape, so that structural defects exist, which may also cause the reduction in the sample coercivity. Kubo et al. [38] The rapid fall in coercivity indicates transformation of hard ferrite into soft ferrite. Thus CaAl₁Fe₁₁O₁₉ assumes more hard ferrite in nature than that of CaLa₁Fe₁₁O₁₉ and CaLa₁Al₁₁Fe₁₀O₁₉ Ferrite. The squareness ratio (SQR), for the samples is denoted by the ratio of M_r to M_s. SQR values of all the samples are depicted in the observation Table 3. The values of squareness ratio are essentially a measure of squareness of the hysteresis loop. The (SQR) should be less than 0.5 for the single domain magnetic structure of the sample. In case, all the sample have SQR less than 0.5 which eventually confirms the single domain structure for the samples [39]. It is known that in general, the nano-hexaferrites with large SQR values (in the region of 0.5) are highly useful in recording media. It is known that the nano-hexaferrites with such special magnetic properties are highly useful in data storage applications [40,41]. In present investigation the sample CaAl₁Fe₁₁O₁₉ has high value of SQR. Hence it can be the promising material for recording media application.

Table 3: Magnetic parameters

Compound	M _s	M _r	H _c	SQR ratio (M _r / M _s)
CaLa ₁ Fe ₁₁ O ₁₉	99.52	14.71	192.85	0.1477
CaLa ₁ Al ₁ Fe ₁₀ O ₁₉	33.75	07.64	2440.1	0.2027
CaAl ₁ Fe ₁₁ O ₁₉	66.76	28.10	3386.0	0.4133

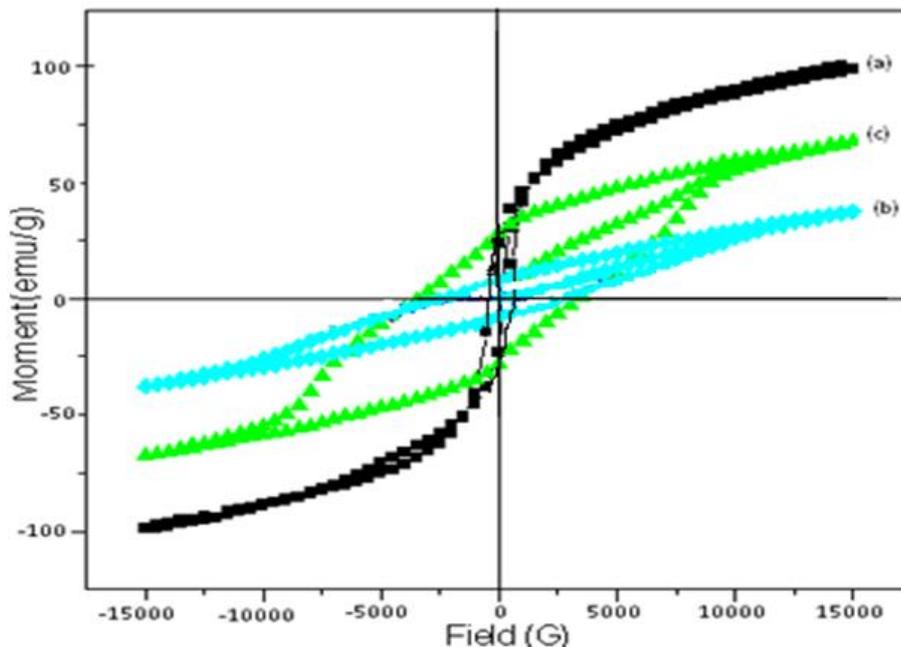


Figure 5: Hysteresis loops of (a) $\text{CaLa}_1\text{Fe}_{11}\text{O}_{19}$ (b) $\text{CaLa}_1\text{Al}_1\text{Fe}_{10}\text{O}_{19}$ (c) $\text{CaAl}_1\text{Fe}_{11}\text{O}_{19}$ at room temperature

4. Conclusions

Single-phase M-type La, La-Al and Al substituted calcium hexaferrite samples were successfully synthesized by the microwave induced sol-gel auto-combustion technique. The X-ray diffraction studies confirm the formation of single phase M type hexaferrites. The values of lattice parameters „a“ and „c“ of the sample supports this confirmation. Structural studies have confirmed the space group of samples to be $P6_3/mmc$.(No. 194). The SEM and TEM study confirmed that samples exhibit relatively well defined, hexagonal like grains with average size in the range 43 nm to 31 nm.

The activation energy for all the samples is found to be different for ferrimagnetic and paramagnetic regions. The activation energy in ferrimagnetic region is observed to be less than that in paramagnetic region. It is observed that the values of activation energy for all samples are above 0.2 eV clearly indicate the polaron hopping in the system. The d c electrical resistivity of the samples increases rapidly with doping of Al^{+3} ions for Fe^{+3} ions. This is attributed to the fact that the substituted ions, localized Fe^{2+} ions and this localization induces Verwey-de Boer hopping mechanism between Fe^{2+} and Fe^{3+} ions. The increase in DC electrical resistivity suggests that the synthesized material ($\text{CaAl}_1\text{Fe}_{11}\text{O}_{19}$) can be used for applications in microwave devices as eddy current loss decreases.

The reduction of particle size of hexaferrites samples to nano range greatly improves the magnetic properties such as saturation magnetization, remanent magnetization and coercivity. The particle size of Al doped sample is small as compared to La and La-Al doped samples. It was found that doping of Al leads to a rise in saturation magnetization and to a significant increase in the coercive field which might be due to uniaxial magneto crystalline anisotropy along c-axis. The rapid rise in coercivity for sample $\text{CaAl}_1\text{Fe}_{11}\text{O}_{19}$ indicates that it is hard ferrite compare to that of $\text{CaLa}_1\text{Fe}_{11}\text{O}_{19}$ and $\text{CaLa}_1\text{Al}_1\text{Fe}_{10}\text{O}_{19}$ Ferrite. All the sample

have squareness ratio less than 0.5 which eventually confirms the single domain structure for all the samples. The magnetic characters of the Al doped samples are observed to be of immense vital improvisation. Thus, purely Al doped calcium hexaferrites have superior magnetic properties than that of La and La-Al substituted. In present investigation the sample $\text{CaAl}_1\text{Fe}_{11}\text{O}_{19}$ has high value of SQR (in the region 0.5), hence it can be the promising material for recording media application.

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