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Structural, Electrical and Magnetic properties of Aluminum Substituted Nanocalcium Hexaferrites

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Abstract: M-type calcium Hexaferrites with substitution of trivalent Aluminum ions were prepared by Solution combustion technique using metal nitrates as oxidants and ODH as reducing agents. The powders were characterized with XRD, SEM, and EDS. XRD analysis indicated the formation of single-phase substituted M-type calcium hexaferrites with space group of $P6_3/mmc(194)$. The average crystallite size is calculated and found to be in Nano size. DC conductivity and AC conductivity of bulk samples have been investigated as a function of frequency and temperature. The measurements were carried out in the frequency range of 100 to 10×10^5 Hz, at room temperature. 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. Magnetic properties are studied with the help of vibrating sample magnetometer (VSM). Results of VSM studies show the decrease in saturation magnetization with the increase of Al concentration. Increase in coercivity and Retentivity illustrated that these are hard ferrite materials.

Keywords: NanoHexaferrite, Activation Energy, Coercivity and Retentivity.

1. Introduction and Experimental

Hexagonal ferrites form a large family of ferrimagnetic materials. Among these, M-type hexaferrites are the most popular due to their large applications as permanent magnets with unique magnetic performance, low cost and chemical stability [1-2]. Substituted hexaferrites belong to M-Type with general formula $MFe_{12}O_{19}$ where M is usually barium, strontium, Calcium or Lead, are of the significant attractions for researchers.

The basic structure of magnetoplumbites consists of 38 oxygen and 24 ferric ions. All ferric ions occupy five different locations in the unit cell such as 2a, 4f₂ and 12k, which are octahedral sites, 4f₁ tetrahedral and 2b-bi-pyramidal sites [3-6]. The magnetic properties of magnetoplumbites are determined by the substituted trivalent ions for ferric ions, which occupy different sites in the structure [7]. When doped with other trivalent metal ions, the magnetic properties of the calcium ferrite would get altered [8-9].

There are many methods for synthesis of Hexagonal ferrite nanoparticles [10]. In the present work, Calcium hexaferrites doped with trivalent Al ions with general formula $\text{CaAl}_x\text{Fe}_{12-x}\text{O}_{19}$ ($x=3$ and 4) have been synthesized successfully by solution combustion technique. Samples were prepared with AR grade calcium nitrate, iron nitrate and aluminum nitrate. ODH ($\text{C}_2\text{H}_6\text{N}_4\text{O}_2$) was used as fuel. Samples were calcinated at 900°C for 3 hours and allowed to cool gradually [10]. The structural analysis was performed by XRD. The morphology and compositional analysis were studied using SEM and Energy Dispersive Spectroscopy (EDS) attached with SEM. The magnetic measurements were carried out using (VSM) at room Temperature. For conductivity measurements, the powder was made into pellet and sintered at 800°C . Resistivity of the samples at room temperature and activation energies in both ferri and paramagnetic regions were calculated and reported (Table 1).

Table: Calculated Electrical and magnetic data of samples

Compound	H_c (Koe)	M_s (emu/g)	M_r (emu/g)	Resistivity At RT ρ ($\text{M}\Omega/\text{cm}$)	Activation Energy ΔE (ev)	
					Para	Ferri
$\text{CaAl}_3\text{Fe}_9\text{O}_{19}$ (CFA3)	2.017	3.874	0.869	17.017	0.09	0.98
$\text{CaAl}_4\text{Fe}_8\text{O}_{19}$ (CFA4)	5.092	3.791	1.278	28.189	0.18	1.2

2. Results and Discussions

X-ray diffraction studies confirm the formation of hexaferrites with the space group $\text{P6}_3/\text{mmc}$. The average crystallite size of the powders was measured by X-ray line-broadening technique employing the Debye-Scherer formula. The results show drastic improvement in the grain size at the Nano level, as compared to similar work reported elsewhere [8]. The morphology and composition analysis of samples shows that the grains are uniform and cylindrical in shape having a size of 30.8 and 23.4 nm respectively for Al_3 and Al_4 substitutions. EDS analysis shows that the samples contain only Ca, Al, Fe, and O.

To study the magnetic properties of synthesized compounds, the magnetic moment was recorded as a function of magnetizing field in the range of -20 kG to $+20$ kG at room temperature. Calculated Saturation magnetization (M_s), Coercivity (H_c) and Retentivity (M_r) values are described in Table 1. Results of VSM studies show the decrease in saturation magnetization (M_s) with the increase of Al concentration. Increase in magnetic properties such as coercivity (H_c) and Retentivity (M_r) confirmed the nature of hard ferrites. The dc electrical resistivity, activation energy of the samples showed a gradual increase with increase in the content of Al at room temperature.

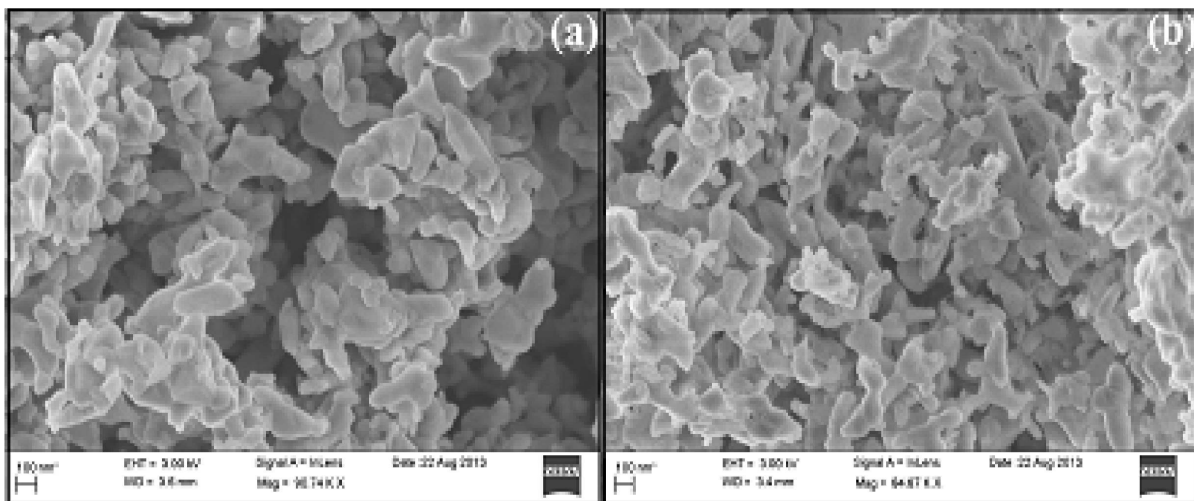


Figure1: SEM images of (a) $\text{CaAl}_3\text{Fe}_9\text{O}_{19}$, (b) $\text{CaAl}_4\text{Fe}_8\text{O}_{19}$

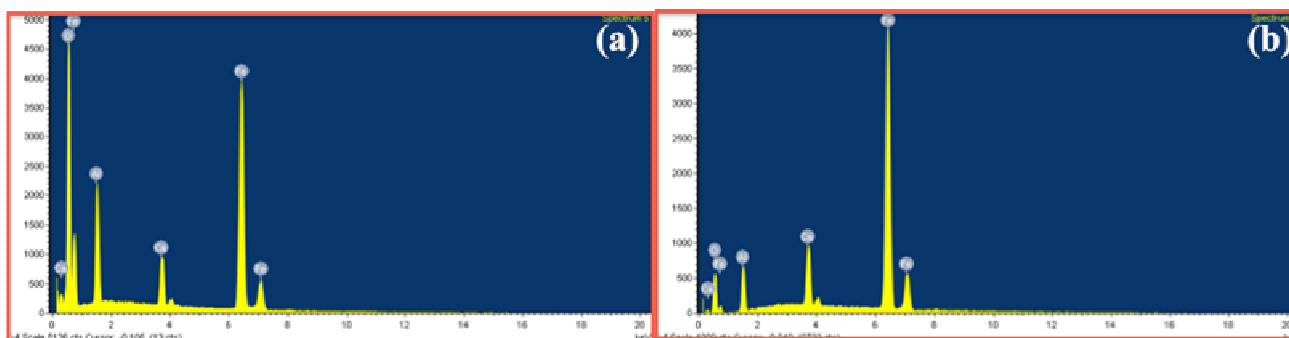


Figure 2: EDS, Spectrum of (a) $\text{CaAl}_3\text{Fe}_9\text{O}_{19}$, (b) $\text{CaAl}_4\text{Fe}_8\text{O}_{19}$

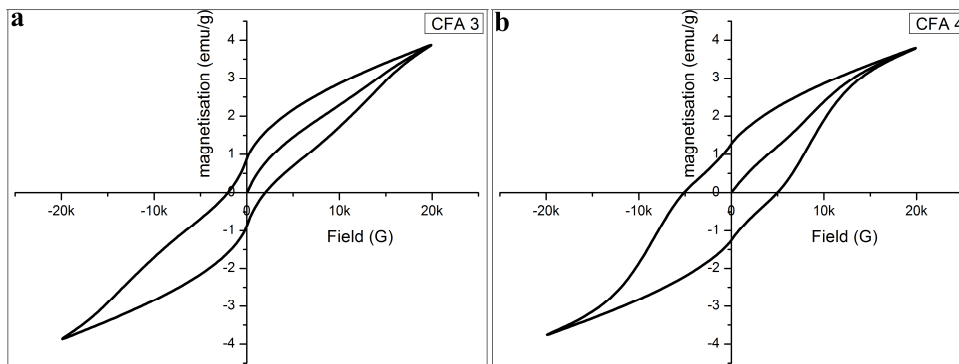


Figure 3: Hysteresis loop of (a) $\text{CaAl}_3\text{Fe}_9\text{O}_{19}$ (b) $\text{CaAl}_4\text{Fe}_8\text{O}_{19}$

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