



X-ray Diffraction Studies of Synthesized M-type Compound

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ABSTRACT

M-type calcium Hexaferrites with Substituted of Trivalent Aluminum ions (Al^{+3}) substituted $CaAl_6Fe_6O_{19}$ were synthesized by Standard ceramic method. The Characterization by using various instrument Techniques. The structural studied of the sample were studied by using X-ray Diffraction (XRD), and Scanning Electron Microscope (SEM). XRD shows hexagonal magnetoplumbite M-type structuring and having unit cell dimension 'a=b' & 'c' varies between 5.7639\AA & 22.31\AA . The SEM Morphology of the particle was studied & particle size was confirmed using SEM.

Keyword: Hexagonal ferrites (hexaferrite), X-ray Diffraction (XRD), Scanning Electron Microscope (SEM).

I. INTRODUCTION

Hexagonal ferrites (Also known as hexaferrites), were discovered in the 1938 at V. Adelskold [1]. Since then, the degree of interest in these ferrites has been increasing exponentially due to their cost effectiveness and suitability for a wide range of industrial and technological application [2-9]. Hexaferrite are industrially important material that have numerous technological application such as permanent magnets, microwave device, magnetic recording media and magneto-optical device. Magnetoplumbite (M) type is a hard ferromagnetic material possessing hexagonal structure which was first observed by the general Chemical formula by which M-type hexaferrite are represented is $MFe_{12}O_{19}$ [10]. 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 $2a$, $4f_2$ and $12k$, which are octahedral sites, $4f_1$ -tetrahedral and $2b$ -bipyramidal sites [11-

14]. The magnetic properties of magnetoplumbites are determined by the substituted trivalent ions for ferric ions, which occupy different sites in the structure [15]. When doped with other trivalent metal ions, the magnetic properties of the calcium ferrite would get altered [16-17]. There are many methods for synthesis of Hexagonal ferrite nanoparticles [18]. In the present work, Calcium hexaferrites doped with trivalent Al ions with general formula $CaAl_xFe_{12-x}O_{19}$ ($x=6$) have been synthesized successfully by Standard ceramic method.

II. EXPERIMENTAL DETAILS

Synthesis:

The preparation of compounds with chemical formula $CaAl_xFe_{12-x}O_{19}$ (with $x=6$) by standard ceramic method. The molecular concentration x substituted cations in the chemical formula. The oxides CaO , Al_2O_3 , Fe_2O_3 of Merck grade (with 99.90% purity) were used as starting material for the synthesis of present series of compounds. The stoichmetric proportion of weight oxides were mixed thoroughly by grinding for 6 hours in agate mortar

with help of acetone to get ultra-fine homogeneous powder of sample. The resulting powder pre-sintered at 200°C for half hours to moisture free, homogeneous, calcinations. The calcination powder were pressed into the pellet machine to form pellet at 75 tons kg/cm² and then sintered at 1130°C in air atmosphere for about 72 hours and slowly cooled to room temperature at the rate of 200°C /half hours using a microprocessor controlled furnace. The synthesized pellet break with hydraulic pressure of pellet machine at 120 kg/cm² . Then grinding in agate mortar to get ultra-fine powder of sample. The synthesized powder of sample again heated at 300°C for 30 minute to remove impurity.

Characterization:

- X-ray diffraction pattern of CaAl₆Fe₆O₁₉ hexagonal ferrite under investigation were obtained using X-ray Diffractometer.
- Determination of grain size and aspect factor from SEM data.

III. RESULT AND DISCUSSION:

Structural analysis: The XRD pattern of CaAl₆Fe₆O₁₉ powder (fig1) investigated ferrite sample synthesized by ceramic method correspond to M-type calcium hexaferrite structure fig (1). The hexagonal M-structure with space group (SG:P63/mmc) (No. 194), which confirms that phase belongs to magnetoplumbite, indicating that the crystal structure were single phase hexagonal magnetoplumbite after substitution with La³⁺ ions respectively. The lattice constant a and c of hexagonal calcium ferrite were calculated using equation (1)

$$\frac{1}{d^2} = \frac{4(h^2+k^2+hk)}{3a^2} + \frac{l^2}{c^2} \text{-----(1)}$$

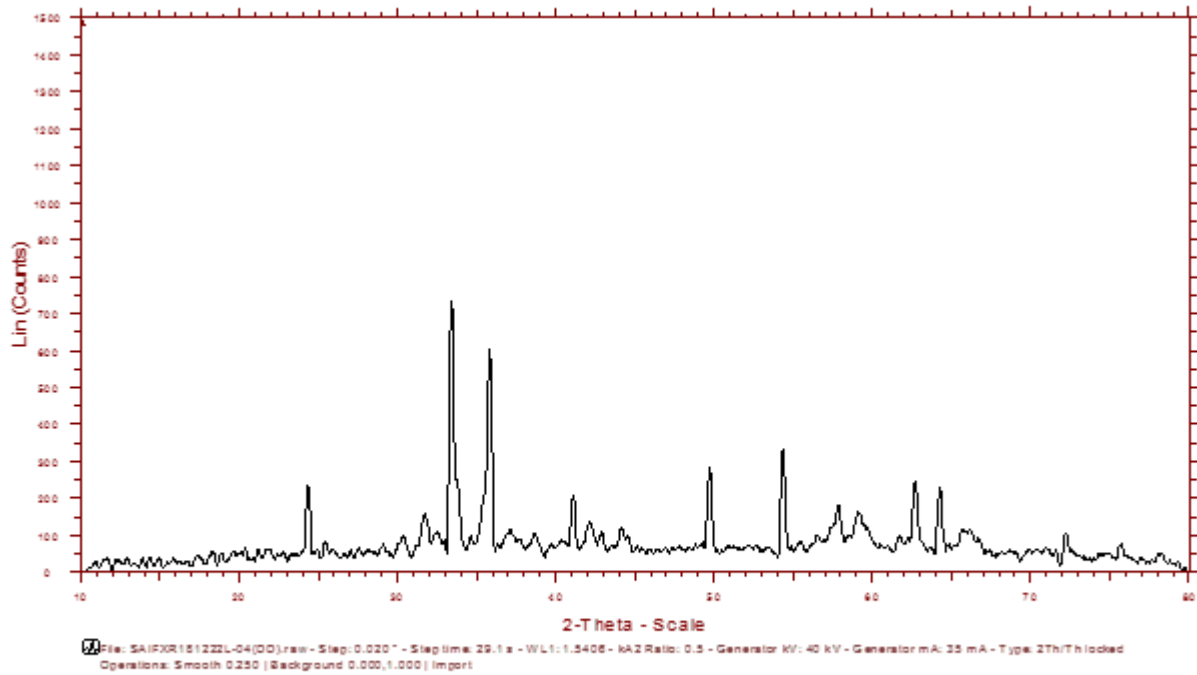
Where h, k, l are miller indices, d is interplaner distance. The lattice parameter a and c found to be 5.7639 and 22.31 respectively. The crystallite size measurements were also carried out using the XRD data and using scherrer equation

$$D = \frac{k\lambda}{\beta \cos\theta} \text{----- (2)}$$

Where β is width of the observed diffraction peak at its half maximum intensity, K is the space factor which take value of about 0.94 & λ is the wavelength (Cu). And the average particle size was found to be about 46.49nm.

Table 1

Sample	a (Å)	c (Å)	Particle size (D)nm	Volume (Å ³)	Maximum intensity	c/a ratio
CaAl ₆ Fe ₆ O ₁₉	5.7639	22.31	46.49	641.89	735	3.8706



Fig(1) X-Ray Diffraction of $\text{CaAl}_6\text{Fe}_6\text{O}_{19}$

Table 2. Observation table of $\text{CaAl}_6\text{Fe}_6\text{O}_{19}$

Sr. No.	2θ	d value (\AA)	Observe intensity count (I)	Intensity in (I) %	h k l
1	24.297	3.66035	232	31.5	(1 1 2)
2	30.308	2.94663	96	13.1	(1 2 2)
3	31.687	2.82154	156	21.2	(0 1 3)
4	33.371	2.68290	735	100	(1 1 3)
5	35.792	2.50675	600	81.7	(0 2 3)
6	41.073	2.19580	206	28	(0 1 4)
7	42.223	2.13861	132	17.9	(1 1 4)
8	44.181	2.04830	116	15.8	(1 3 3)
9	49.717	1.8329	283	38.5	(2 2 4)
10	57.812	1.59358	171	23.3	(0 4 4)
11	62.719	1.48018	244	33.2	(0 1 6)
12	64.298	1.4476	228	31.4	(1 1 6)

SEM Studies:

The scanning electron microscopy (SEM) Fig(2) shows the morphological information of the hexagonal ferrite containing irregular grain, but nearly vertically arranged shaped flakes. The particle

seemed to be hexagonal plate like mould which spread out homogeneously were examined by ceramic method.

Figure 2 shows the representative SEM micrographs of fractured surface of Al-substituted sintered CaM. The micrograph shows that Al substitution reduces the particle size. Al substituted calcium hexaferrite. Aspect factor calculated from following relation.

$$\text{Aspect Factor} = \frac{\text{length}}{\text{diameter}}$$

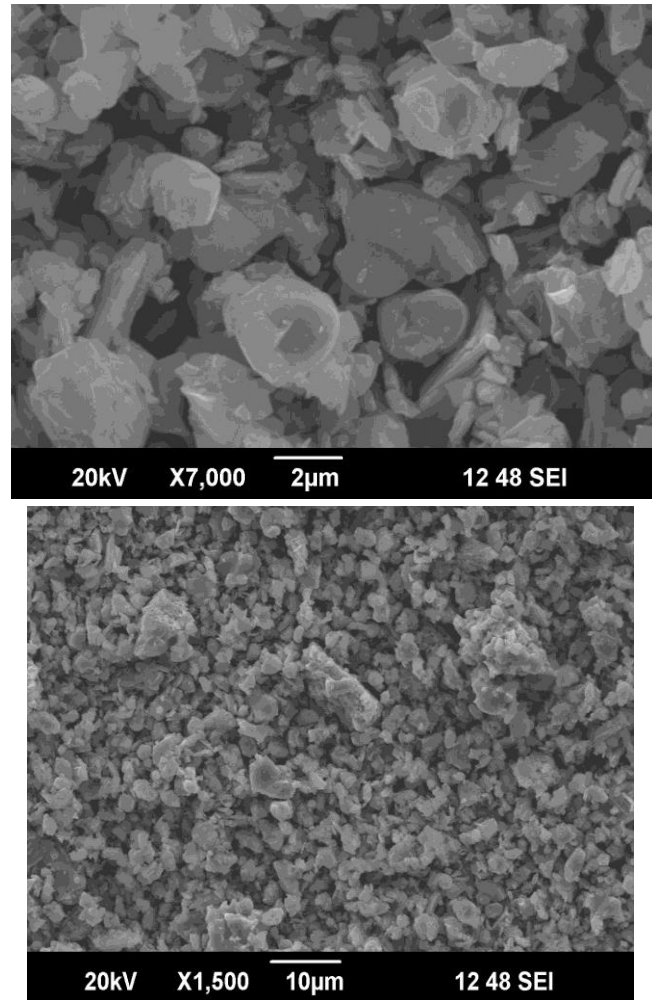
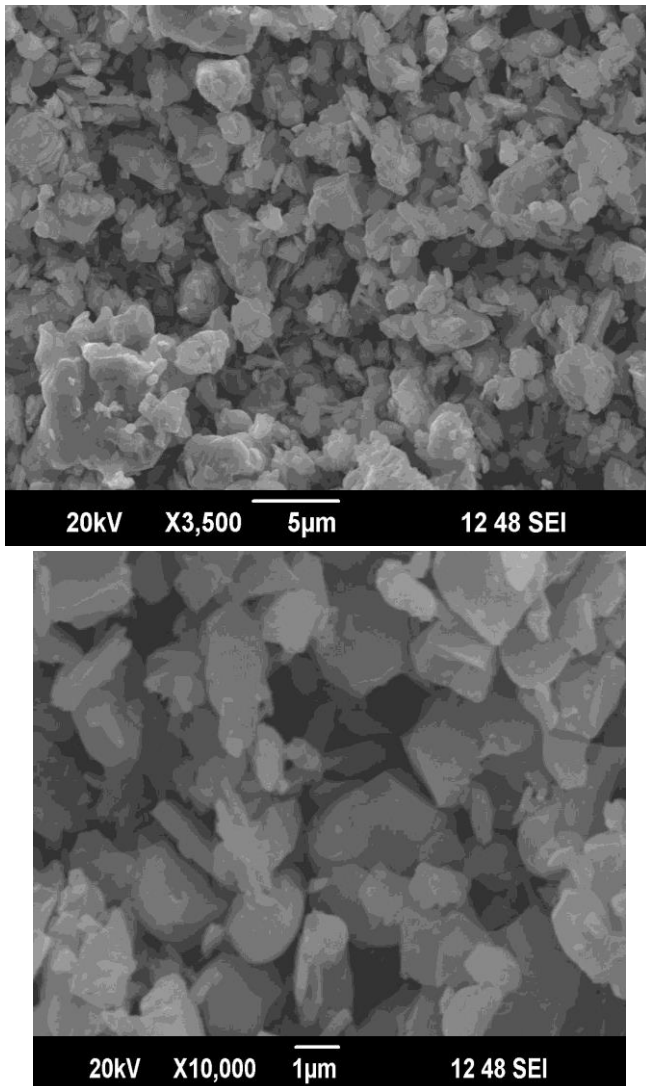


Figure 2. SEM image of $\text{CaAl}_6\text{Fe}_6\text{O}_{19}$

Table 3. Observation table of $\text{CaAl}_6\text{Fe}_6\text{O}_{19}$

Sample	Grain size (nm)	Aspect factor
$\text{CaAl}_6\text{Fe}_6\text{O}_{19}$	376.7	2.05

IV. CONCLUSION

The ferrites $\text{CaAl}_6\text{Fe}_6\text{O}_{19}$ with Al Substituted were synthesized by Standard ceramic method. The X-ray diffraction studied confirm the formation of monophasic M-type hexaferrite and the a and c value of the sample supports this confirmation. Structural studies have confirmed the space group of the sample to be P6/mnc. The nanorange of particle size of hexaferrites helps to improve many magnetic properties mentioned earlier. The substitution of Al^{3+}

ion for Fe^{3+} ion greatly improves the magnetic parameters.

V. ACKNOWLEDGEMENT

The authors thanks to sophisticated test and Instrumentation Centre, STIC, Cochin University Keral, India for giving us best result for X-ray Diffraction (XRD) and Scanning Electron Microscope (SEM) in needed hour. The authors are also thankful to the management, Principal Prof. Dr. K. P. Kariya of VMV College, Nagpur for providing the basic facilities of synthesis of the sample.

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