# STRUCTURAL PROPERTIES OF AL AND CR DOPED M-TYPE NANO HEXAFERRITES

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## ABSTRACT

A series of single phase and nanostructured Aluminum ($Al^{3+}$) ion and chromium ($Cr^{3+}$) iondoped with M-Type hexa-ferrites $CaAl_6Fe_6O_{19}$ and $CaCr_6Fe_6O_{19}$ were synthesized by solution combustion technique. The powdered samples were sintered at $900^0C$ for about 3hrs and characterized by X-ray diffraction (XRD), Scanning electron microscopy (SEM) and Fourier Transform Infrared spectroscopy(FTIR) analysis was carried out to observe the transformation of different phases during heat treatment. X-ray diffraction (XRD) conformed that the single phase and nanostructured Aluminum and Chromium doped M-Type hexa-ferrites with space group 167: R-3c, hexagonal of P63/mmc. Morphology of the particle was studied and particle size was confirmed using SEM. The crystal size calculated by the Scherer equation was found to be in the range of 20.37nm and 34.81nm.The occurrence of absorption bands in FTIR spectra are due to stretching vibrations to the metal oxygen in the lattice confirmed the formation of hexagonal structure. This is small enough to obtain a suitable signal to noise ratio in the high density recording media and permanent magnets.

**Keywords:** Hexa-ferrites, solution combustion, X-Ray Diffraction, SEM, FTIR.

## I. INTRODUCTION

Hexagonal ferrites are large family of ferromagnetic materials. Ferrites are classified into four types; they are spinel, garnets, hexagonal and ortho-ferrites. Hexagonal ferrites have attracted much attention of several workers [1-3]due to their large applications as permanent magnets.

The doped calcium hexa-ferrites belong to M type are of the significant attractions for researchers because of their applications in the field of material science as permanent magnets, microwave device etc[4-5]. The basic structure is hexagonal with all 38 oxygen ions occupying the interstitial sites forming a closepacked assembly 24 ferric ions occupy ,five different location in the unit cell such as $2a,2b,4f_1,4f_2$ and 12k where $2a,4f_2$ and 12k are octahedral $4f_1$ is tetrahedral and 2b is bi-pyramidal sites[6]. The magnetic nature of magnetoplumbites is determined by the doped with trivalent ions for ferric ions which occupy different sites in the structure [7-9].The $Fe^{3+}$ ions when replaced partially by other trivalent metal ions , the magnetic properties of the calcium ferrites undergo changes[10,11]. To prepare hexagonal ferrites various synthesis method like chemical co-precipitation method [12], hydrothermalmethod [13], sol gel method (14). In the present work calcium hexa-ferrites doped with $Al^{3+}$and $Cr^{3+}$ ions synthesized by solution combustion method [16] are studied and reported.

## II. EXPERIMENTAL DETAILS

### A. Sample Preparation:

Samples of M-type Aluminum and chromium substituted calcium hexa-ferrites with general formula $CaAl_6Fe_6O_{19}$ and $CaCr_6Fe_6O_{19}$ have been synthesized successfully by solution combustion. The samples were prepared with AR grade calcium nitrate$Ca(No_3)_24H_2O$, Iron nitrate $Fe(No_3)_39H_2O$, Aluminum nitrate $Al(No_3)_39H_2O$ and Urea ($NH_2$-Co-$NH_2$) was used as fuel.

Solution combustion method the stoichiometric amounts of metal nitrates which act as oxidizing agent and fuel acts as reducing agentexothermic reaction . The maximum heat is released when oxidizer to fuel to ratio (O/F) become equal to unity [15]. Stoichiometric amount of metal nitrates along with urea were dissolved completely in the double distilled water to form an aqueoussolution. After continuous stirring for about 3hrswith the help of magnetic stirrer, the solution was kept in the pre heated mufflefurnaceat $300^0$c. Thence the solution

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boils and undergoes dehydration followed by decomposition within a few minutes. After reaching spontaneous combustion, at around 400 $^0$C,the solution ignites like smoldering with the evolution of a large volume of gases ($N_2$ and $Co_2$).It releases large amount of heat vaporizing and all the solution turns into a solid. Prepared powders are of loose foamy pores and slightly fire-able.After cooling, the samples crushed into powder using agate motorand were sintered at 900$^0$C for about 3hrs and allowed to cool gradually on sand bath.

### B. Structural Analysis:

The characterization of doped nanohexa-ferrites by X-ray diffraction (XRD), Scanning electron microscopy (SEM) and Fourier Transform Infrared spectroscopy (FTIR) analysis was carried out to observe the transformation of different phases during heat treatment as follows.

## III. RESULTS AND DISCUSSION

### A. X-ray diffraction analysis:

The structural characterization of the sample was performed PAN analytical X pert pro- diffractometer with Cu-kα radiation in the 2 theta range of 20$^0$ to 80$^0$in angular steps of 0.02$^o$. The average crystal size D was determined from line broadening ions using Debye Scherer formula D = kλ/ β cos θ  where β is the angular line width at half maximum intensity and θ is the Bragg's angle for that peak . k is Schererconstant(k=0.89 for hexagonal ferrites) and λ is the wavelength of x-rays( λ=1.5405 A$^0$ ). The value of lattice constant a, c and the unit cell volume ($V_{cell}$)were calculated by taking (hkl) parameter and distance between the planes (d), x-ray density ($\rho_{x-ray}$) were calculated by using following equations [18].

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

$$V_{cell} = 0.866\ a^2c$$

$$\rho_{x-ray} = \frac{2M}{V\ N_A}$$

Where, M is the molar mass,V-cell volume,'$N_A$' is Avogadro's number.

X- ray diffraction spectrum for all the sample is shown in the Fig. 1(a&b) X-ray diffraction studies confirmed the formation of hexa-ferrites of space group p63/mmc. There is a small shift peak position with the increase of substituting. This is due to the ionic radius of $Al^{3+}$(0.184 A$^0$) and $Cr^{3+}$(0.2A$^0$)compared to the ionic radius of $Fe^{3+}$(0.65A$^0$).Structural parameters of  these variation in the particle size with substitution .The Volume of the cell also slightly decreases with the increase in substitution.
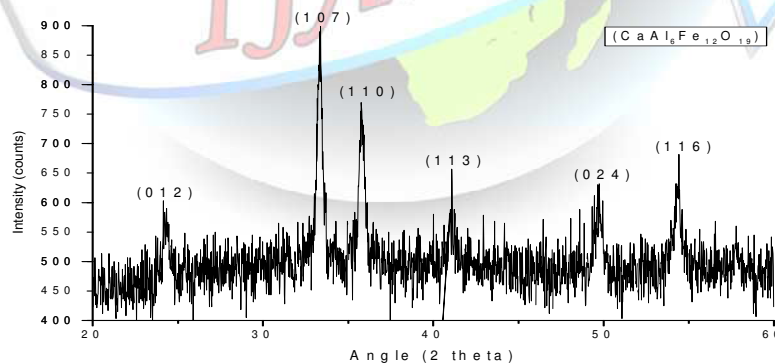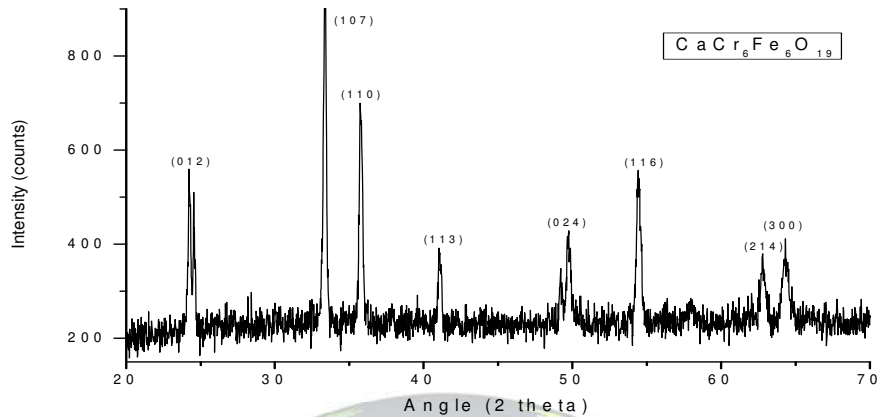


**Fig.1(a) XRD spectrum of CaAl$_6$Fe$_6$O$_{19}$**

**Fig1 (b). XRD spectrum of CaCr$_6$Fe$_6$O$_{19}$**

**Table 1. Grain size and lattice parameters**

| Sample | D(nm) | a A$^0$ | c A$^0$ | v (A$^0$)$^3$ | $\rho_{x\text{-ray}}$(gm/cm$^3$) |
|---|---|---|---|---|---|
| CaAl$_6$Fe$_6$O$_{19}$ | 20.37 | 5.017 | 13.666 | 297.945 | 1.3107 |
| CaCr$_6$Fe$_6$O$_{19}$ | 34.81 | 5.017 | 13.645 | 297.510 | 1.4310 |

**B.   FTIR spectroscopy:**

Fig. 2(a) and 2(b) shows FTIR profile of two sintered samples with nominal compositions CaAl$_6$Fe$_{12}$O$_{19}$ and CaCr$_6$Fe$_{12}$O$_{19}$. The FTIR graphs of as prepared and sintered sample in the range of wave number 400-4000cm$^{-1}$.The absorption bands of CaAl$_6$Fe$_{12}$O$_{19}$at 580.63, 1385.52, 1638.74, and 3421.94cm$^{-1}$and CaCr$_6$Fe$_{12}$O$_{19}$at 895.96, 1137.14, 1154.75 and 1261.74cm$^{-1}$respectively were observed. The occurrence of absorption bands in FTIR spectra are due to stretching vibrations to the metal oxygen in the lattice confirmed the formation of hexagonal structure. [7] proposed a principle in which another NN yield input control law was created for an under incited quad rotor UAV which uses the regular limitations of the under incited framework to create virtual control contributions to ensure the UAV tracks a craved direction. Utilizing the versatile back venturing method, every one of the six DOF are effectively followed utilizing just four control inputs while within the sight of un demonstrated flow and limited unsettling influences. Elements and speed vectors were thought to be inaccessible, along these lines a NN eyewitness was intended to recoup the limitless states. At that point, a novel NN virtual control structure which permitted the craved translational speeds to be controlled utilizing the pitch and the move of the UAV. At long last, a NN was used in the figuring of the real control inputs for the UAV dynamic framework. Utilizing Lyapunov systems, it was demonstrated that the estimation blunders of each NN, the spectator, Virtual controller, and the position, introduction, and speed following mistakes were all SGUUB while unwinding the partition Principle.
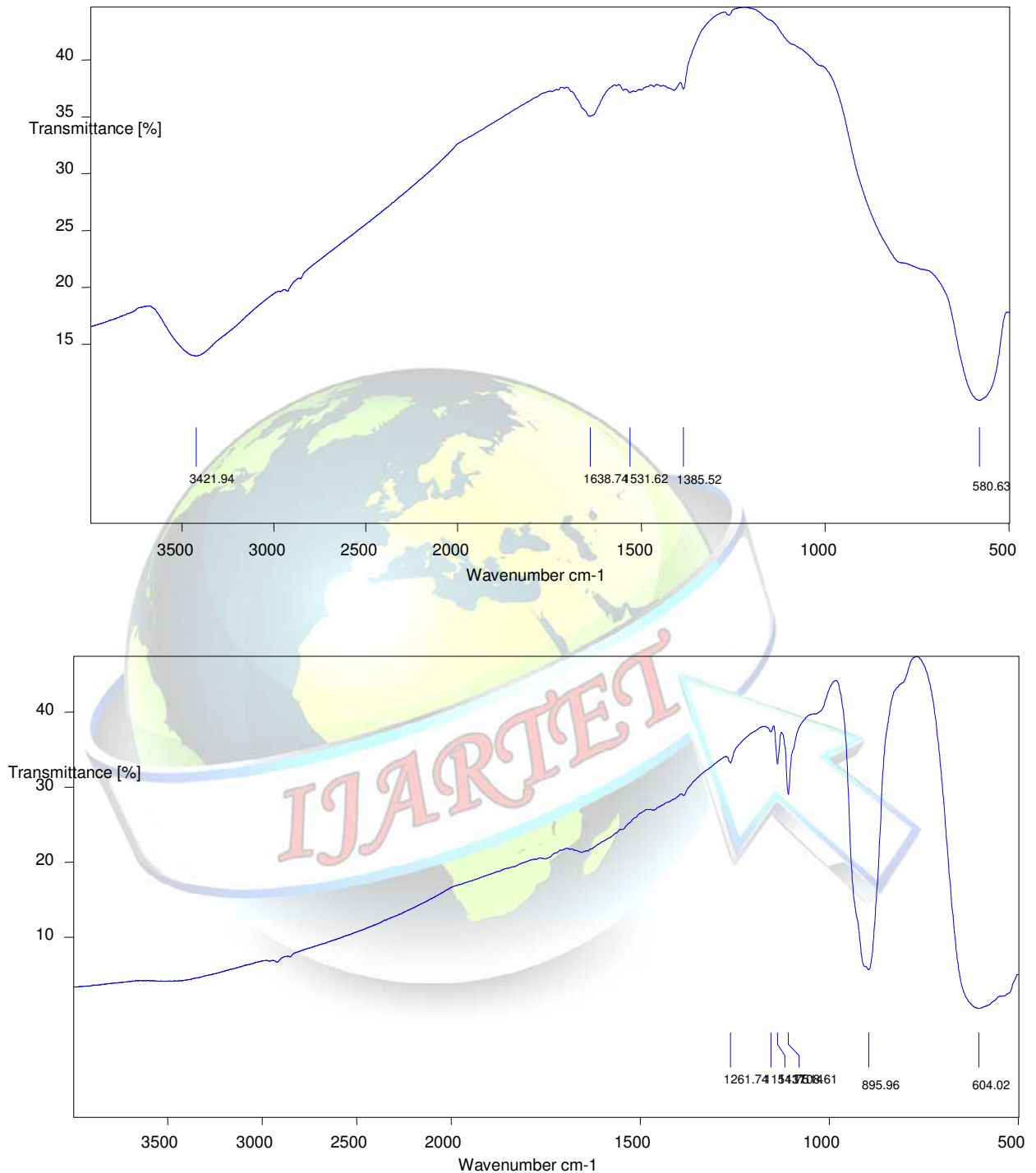
Fig. 2(a) and 2(b) shows FTIR spectrum of $CaAl_6Fe_{12}O_{19}$ and $CaCr_6Fe_{12}O_{19}$

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### C. Micro Structural analysis:

The Scanning Electron Microscopy results of the samples are shown in fig 3(a, b, c, d). It is the evidence that particle are nano size and almost spherical in shape [17].
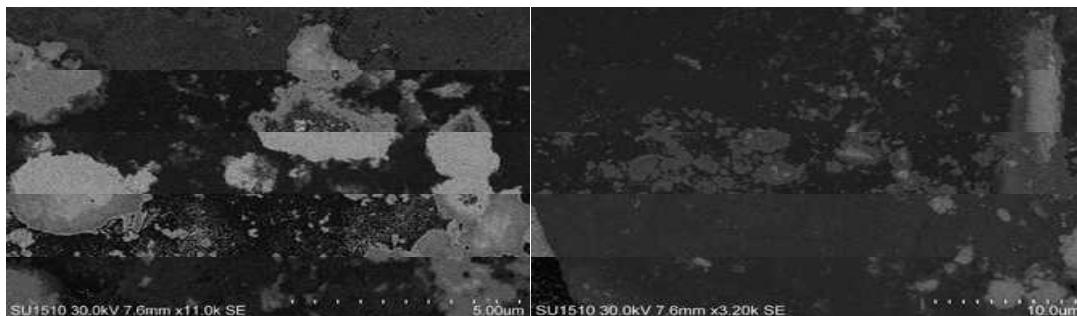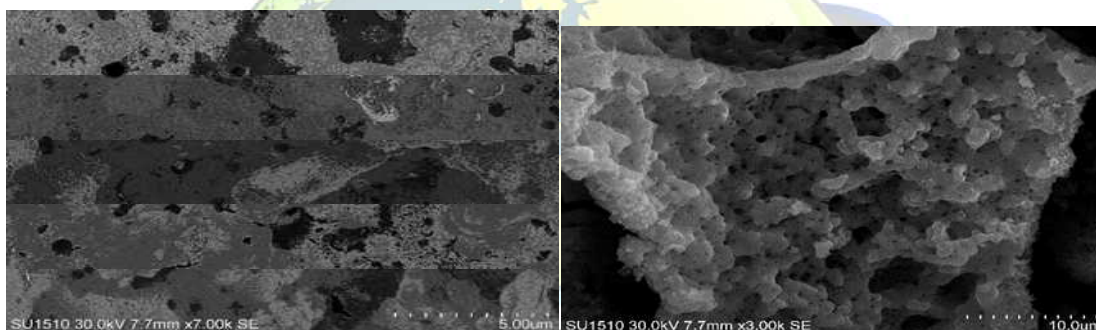


| Fig.3 (a) | fig.3(b) |



| Fig.3(c) | fig.3 (d) |

**Figure 3: SEM images of (a), (b)$CaAl_6Fe_6O_{19}$ and (c),(d)$CaCr_6Fe_6O_{19}$**

## IV. CONCLUSION

A series of Aluminum (Al) and chromium (Cr) substituted M-Type hexa-ferrites $CaAl_6Fe_6O_{19}$ and $CaCr_6Fe_6O_{19}$ have been synthesized by solution combustion technique. The X-ray diffraction pattern reveals that the formation of hexagonal structure with space group of P63/mmc, without any trace of secondary phases. A decrease in the lattice parameter 'c' with increasing $Al^{3+}$ and $Cr^{3+}$ substitution is due to difference in atomic radii of $Al^{3+}$ ion($0.184 A^0$) and $Cr^{3+}$ ion ($0.2 A^0$). The most prominent peak observed at ($33.31^0$) and ($33.36^0$),the average crystal size was calculated with the Debye Scherer formula. The particle size was<50nm which can be used for high–density recording media, since a reasonable signal to noise ratio can be obtained. The morphology of the particle agglomeration is increased with substitutions of aluminum and chromium contents. SEM confirmed that the particles are nano particles. The occurrence of absorption bands in FTIR spectra are due to stretching vibrations to the metal oxygen in the lattice confirmed the formation of hexagonal structure.

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