

## Synthesis and Magnetic study of Zirconium–Cobalt Substituted Calcium Hexaferrites

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### ABSTRACT

The Zirconium – Cobalt substituted ferrite ceramics with chemical composition  $\text{Ca}(\text{Zr-Co})_4\text{Fe}_8\text{O}_{19}$  and  $\text{Ca}(\text{Zr-Co})_8\text{Fe}_4\text{O}_{19}$  having magnetoplumbite structures were synthesized using perfect stoichiometric mixtures of nitrates by the novel method of synthesis ‘Microwave Induced Sol Gel Combustion Route’. XRD results show a single-phase formation of calcium hexaferrite. The lattice parameters ‘a’, ‘c’ and (h k l) values confirms the formation of hexagonal unit cell. Response to the external applied magnetic field is studied using vibrating sample magnetometer (VSM), which shows the moderate values of coercivity, saturation and remnant magnetization. The saturation magnetization ( $M_s$ ), Remanence ( $M_r$ ) and Coercivity ( $H_c$ ) found to decrease with increase in the value of substitution which is attributed to occupation of sub lattice spin-up and spin-down sites by diamagnetic ions and magneto crystalline anisotropy. The variation in magnetic parameters give rise to the possibility of controlling magnetic properties by varying the degree of substitution.

**Keywords** - About five key words in alphabetical order, separated by comma

### I. INTRODUCTION

Low cost, easy manufacturing, and interesting electric and magnetic properties lead the hexagonal ferrite to be one of the most important materials that has attracted a considerable attention in the field of technological applications [1]. Possibility of tailoring the electric and magnetic properties depending upon the application is the most important characteristic of the hexaferrites. Which can be achieved by the partial substitution of divalent-tetravalent metal ion or other compatible combinations for Iron (Fe) in the parent hexaferrite matrix [2,3]. The properties are strongly depend upon the particle size which is again mainly decided by the method of synthesis, sintering temperature, etc [4] The magnetic particles produced via conventional solid state reaction are often larger than particles produced using the sol–gel method. Larger particles of magnetic oxides usually exhibit multi-domain magnetism, whereas nano-sized particles have the characteristic of exhibiting single domain properties[5]. To achieve higher homogeneity and to reduce the particle size of the final product, Microwave induced sol–gel combustion route is employed in this work. In this module, samples with chemical formulae  $\text{Ca}(\text{Zr-Co})_4\text{Fe}_8\text{O}_{19}$  and  $\text{Ca}(\text{Zr-Co})_8\text{Fe}_4\text{O}_{19}$  are synthesized by Microwave Induced sol-gel combustion route are described and their structural, magnetic properties are discussed.

### II. PREPARATION

The synthesis of  $\text{Ca}(\text{Zr-Co})_4\text{Fe}_8\text{O}_{19}$  and  $\text{Ca}(\text{Zr-Co})_8\text{Fe}_4\text{O}_{19}$  is done using the sol–gel route. The synthesis basically consists of the formation of the gel in distilled water by taking proper stoichiometric ratios of reactive nitrates, combustion of the gel in domestic microwave oven and finally the heat treatment at  $800^\circ\text{C}$  for 5 hr. Urea is used as a fuel [6]. It provides requisite energy to carry out exothermic reaction. The samples were analysed using X-ray powder diffractometer Philips X’pert Diffractometer and Cu  $K\alpha$  radiation with wavelength  $\lambda=1.542 \text{ \AA}$ . The measurement of transition temperature  $T_c$  is done by Guoy’s balance setup in the temperature range 300-850K. The magnetic measurements were carried out by using a vibrating sample magnetometer in the applied field up to 12KG.

### III. RESULT AND DISCUSSION

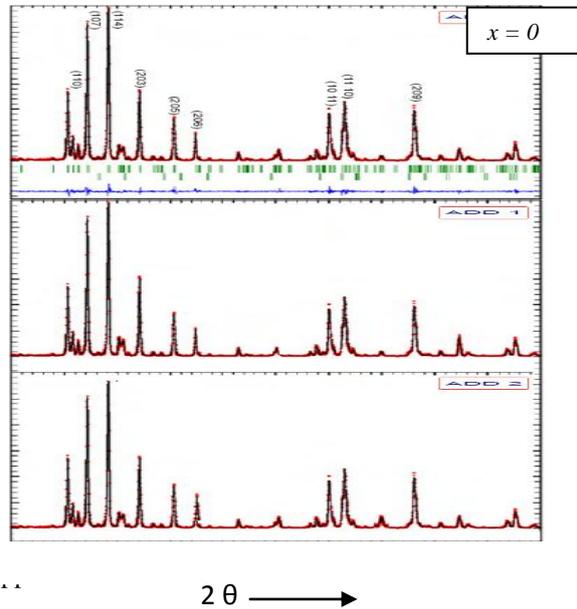
X-ray powder diffraction data is shown in Table 1(a) and Table 1(b). The lattice parameters and consolidated data of magnetic properties are enumerated in Table 3.

#### Structural Characterization

The diffraction patterns of samples are taken with Philips X’pert Diffractometer, using Cu  $K\alpha$  radiation with wavelength  $\lambda=1.542 \text{ \AA}$ . Figure 1

shows the intensity graphs for samples  $\text{Ca}(\text{Zr}-\text{Co})_4\text{Fe}_8\text{O}_{19}$  and  $\text{Ca}(\text{Zr}-\text{Co})_8\text{Fe}_4\text{O}_{19}$  respectively.

**Figure 1 . X-ray powder diffraction patterns of samples**



By comparing the diffraction patterns with JCPDS standards, using  $2\theta$  values, observed d-values and intensity variations, d-values are recalculated and (hkl) planes are finalized. Values of lattice parameters confirms the formation of single phase hexagonal ferrites.

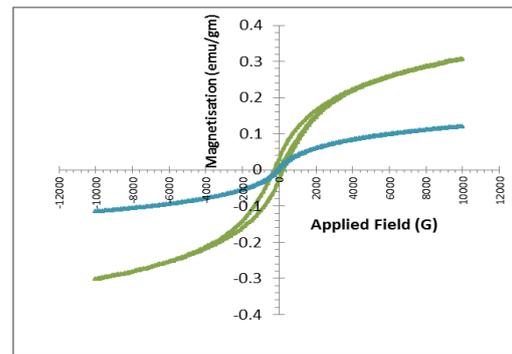
$d_{\text{obs}}$ (Å)	$d_{\text{cal}}$ (Å)	$\frac{I}{I_0}$ (%)	$h k l$
4.8730	4.9263	9.4	(1 1 0)
2.9752	2.9820	97.7	(1 0 7)
2.5443	2.5448	100	(1 1 4)

### 3.2 Magnetic Characterization

Figure 2 shows hysteresis curves for the sample depicting magnetic behavior of the sample in the presence of external magnetic field. Values of saturation magnetization  $M_s$ , remnant magnetization  $M_r$  and coercivity  $H_c$  found to decreases with the substitution of cations as mentioned in Table 2. Decrease in the values of  $M_s$ ,  $M_r$  with increase in values of substitution may be due to the substitution of diamagnetic ions not only into the spin-down sublattices  $4f_1$  of  $\text{Fe}^{3+}$  ions but also into other octahedral spin-up sublattices  $12k$ ,  $2a$ ,  $2b$  of  $\text{Fe}^{3+}$  ions. Decrease in the value of coercivity  $H_c$  with increase in the value of substitution is due to lowering of magneto crystalline anisotropy. Replacement of the

$\text{Fe}^{3+}$  ions from these two sites by substituted cations will cause a lowering of magneto crystalline anisotropy and magnetic coercivity  $H_c$ . Replacement of  $\text{Fe}^{3+}$  ion by non magnetic  $\text{Zr}^{4+}$  in spin up position decreases the net magnetization.  $\text{Co}^{2+}$  ions occupy tetrahedral  $4f_1$  site of  $\text{Fe}^{3+}$  ion with spin down orientation which do not contribute to net magnetization. The effect of tetravalent substitution is more dominant reducing the values of  $M_s$ ,  $M_r$  and  $H_c$  as compared to divalent substitution. Also the area under hysteresis curves decreases with increase in substitution level which indicates that the ferrite become soft with increasing degree of substitution. Comparatively low value of coercivity made the materials to be more useful for the information storage recording media. [8]

**Table 1(a) : X-Ray Diffraction Result for Sample  $x = 2 :: \text{Ca}(\text{Zr}-\text{Co})_4\text{Fe}_8\text{O}_{19}$**



**Figure 2. Hysteresis curves for the samples  $\text{Ca}(\text{Zr}-\text{Co})_4\text{Fe}_8\text{O}_{19}$  and  $\text{Ca}(\text{Zr}-\text{Co})_8\text{Fe}_4\text{O}_{19}$**

**Table 1 : Lattice parameters (  $a$  and  $c$  ), Values of saturation magnetization  $M_s$  , remnant magnetization  $M_r$  and coercivity  $H_c$**

Samples	$\text{Ca}(\text{Zr}-\text{Co})_4\text{Fe}_8\text{O}_{19}$		$\text{Ca}(\text{Zr}-\text{Co})_8\text{Fe}_4\text{O}_{19}$		
	$a$ (Å)	$c$ (Å)	$M_s$ (emu/gm)	$M_r$ (emu/gm)	$H_c$ (G)
$\text{Ca}(\text{Zr}-\text{Co})_4\text{Fe}_8\text{O}_{19}$	5.876	22.61	0.3055	0.0305	250
$\text{Ca}(\text{Zr}-\text{Co})_8\text{Fe}_4\text{O}_{19}$	5.888	23.63	0.1985	0.0265	125

**Table 1(b) : X-Ray Diffraction Result  $x = 4 :: \text{Ca}(\text{Zr}-\text{Co})_8\text{Fe}_4\text{O}_{19}$**

#### **IV. CONCLUSION**

Magnetic and structural properties of zirconium-cobalt substituted calcium hexaferrites powders synthesized by the 'Microwave Induced Sol Gel Combustion Route' are studied from which following conclusion were drawn

1. The improvised method 'Microwave Induced Sol Gel Combustion Route' found to be successful for the synthesis of zirconium-cobalt substituted calcium hexaferrites
2. X-ray spectra shows a hexagonal peaks. The values of lattice parameters  $a$  and  $c$  confirms the formation of hexagonal unit cell.
3.  $\text{Ca}(\text{Zr-Co})_4\text{Fe}_8\text{O}_{19}$  and  $\text{Ca}(\text{Zr-Co})_8\text{Fe}_4\text{O}_{19}$  shows soft magnetic properties which are more useful in the field of information storage recording media.

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