

# Synthesis and Characterization of Cobalt Substituted $\text{NaZnFe}_2\text{O}_4$ by Sol-Gel Method

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## Abstract

The samples of  $\text{NaCo}_{0.11}\text{Zn}_{0.39}\text{Fe}_2\text{O}_4$  and  $\text{NaCo}_{0.24}\text{Zn}_{0.26}\text{Fe}_2\text{O}_4$  were synthesized by sol-gel method. The structural characterizations of these samples were performed by X-ray diffraction (XRD), Scanning electron microscope (SEM), Fourier transform infrared spectroscopy (FTIR). The XRD show that the single phase spinal ferrite was formed when sintering the sample at 1000°C. The crystal sizes were about 24nm and 16nm respectively. The SEM images revealed that the grain was agglomerated and their size measured by line intercept method and it was found to be in the range of 300nm to 400 nm. The FTIR results show that the functional group of the samples are carbon- carbon double bond (C=C), while it lies on the alkaline and alkenes group. Here the coercivity is depending upon the grain size, when the grain size is decreased then the coercivity is increased.

**Keywords:**  $\text{NaZnFe}_2\text{O}_4$ ; Ferrite; Phase analysis; Microstructure

## Introduction

The ferrite is basically a type of ceramic compound which can be represented as iron oxide ( $\text{Fe}_2\text{O}_3$ ) combined chemically with one or more than metallic elements<sup>[1]</sup>. The ferrites can be categorized into three different groups. The First group is Hexagonal Ferrites. The hexagonal ferrites is expressed is due to their high unidirectional magneto crystalline anisotropy which can be used in hard magnet application<sup>[2]</sup>. The other group of ferrite is Grants which have a unique magnetic ceramics. All the species of Grants is expressed which is similar in physical properties in crystal forms, but different in chemical form<sup>[3]</sup>.

The third group of ferrite is known to be the Spinal Ferrite. The spinal ferrities is also discussed in above but in the spinal ferrite the two most important ceramic magnets are the Nickel Zinc Ferrite (NZF) and the other is Magnesium Zinc Ferrite (MZF). They have been most popular ceramic materials due to their high electrical resistivity and high magnetic permeability<sup>[4,5]</sup>. The first ferrite compound was proposed by Yogoro Kato and Takeshi of the Tokyo institute of technology in 1930<sup>[6]</sup>. The ferrite consists of two families which are based on their magnetic coercivity, their resistance to being remove the magnetic properties (Demagnetized)<sup>[7]</sup>. In the two families one is hard ferrite and the other is soft ferrite. The Hard Ferrite is that ferrite which have high coercivity, so they are difficult to remove the magnetic properties, thus this used as a magnet<sup>[8]</sup>. For device in which hard ferrite is used in refrigerator magnets, loudspeaker and small electric motors. While the soft ferrite are those ferrite which have low coercivity. So they are used in such electronics industry to make the ferrite cores for such as inductor, transformer and various microwaves<sup>[9]</sup>. The ferrite compounds are economically very cheap and easy available in market. The ferrites have also

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a good corrosion resistance. The ferrite are basically very stable and very difficult to demagnetized, and are made of both with high and low coercivity forces<sup>[10]</sup>. Ferrites are produced by heating the material for some time of period, after this the mixture is like a gel and this gel is to heat in the specific temperature. After this it becomes to dry and the mixture of small powdered are pressed into a cavity/mould. During this process Calcinations of Carbonates are occurs<sup>[11]</sup>. Those ferrite in which the current cannot easily flow have a very low circular current or eddy current losses process. This type of ferrite is commonly used in lamp in computer cable which is called the ferrite choke. This ferrite choke (bead) are used to prevent the electrical devices from the high frequency electrical sound which are entering or existing. From very beginning the hard ferrite cores are used to store the computer memories data in the form of magnetic field, which were collected these data in specific arrangement in core memory. The ferrites powders are generally used in the coatings of magnetic recording tapes. The particles of ferrite are used for absorbing materials for Radar. And also used for the coating of the aircraft stealth. Most of the ferrite magnets are used in the loudspeakers. These ferrite magnets have largely displaced in the aluminium, nickel and cobalt magnet.

### Experimental Procedure

The samples were prepared by sol-gel method, because this method is very simple, economically very cheap and more helpful method which can produce the high value coatings. The sol-gel method can make the thin bond-layer which creates the better overlapping among the metallic reactant and the peak coat. Due to the sol-gel method which can create the thick layered on the metals surfaces to avoid from the corrosion. In the 1st step we set the weight balance to zero point and measure the weight of the beaker. Now put the zinc nitrate in one beaker and the iron nitrate in another beaker also put the sodium and cobalt in a separate beakers. Now putting the 80 ml De-ionized water + citric acid in each of the beaker in which the chemicals are present. Now shaking all these beakers in which the chemicals are solvable. When the chemicals are completely solve then put all these in a one beaker and stirring it for 30 minutes with the help of magnetic stirrer to mix. After the stirring check the PH levels, when the PH level is less than 7 then put some amount of ammonia to neutralize the sample. Now put this beaker in a magnetic stirrer and giving the temperature up to 70- 80°C for 6<sup>th</sup> to 7<sup>th</sup> hours.

### Results and Discussion

The formation of single phase of  $\text{NaCo}_{0.11}\text{Zn}_{0.39}\text{Fe}_2\text{O}_4$  and  $\text{NaCo}_{0.24}\text{Zn}_{0.26}\text{Fe}_2\text{O}_4$  and sintered at temperature 1000°C was confirmed by the X-ray diffraction. XRD profile of two samples is shown in Figure 1. The  $2\theta$  ranges are from 30° to 70°. The well defined diffraction peaks at corresponding angles were compared by Joint Committee of Powder Diffraction Standard (JCPDS) which confirmed the formation of spinal cubic structure. Peaks are shown in the figure, depicted that samples are in single phase. From XRD peaks at the corresponding angles the lattice parameters, crystallite size was calculated. The interplanar distance was calculated by using the Bragg's law;

$$"n\lambda = 2d\sin \theta" \text{ (where } n=1)$$

So we have,  $\lambda = 2d\sin \theta$

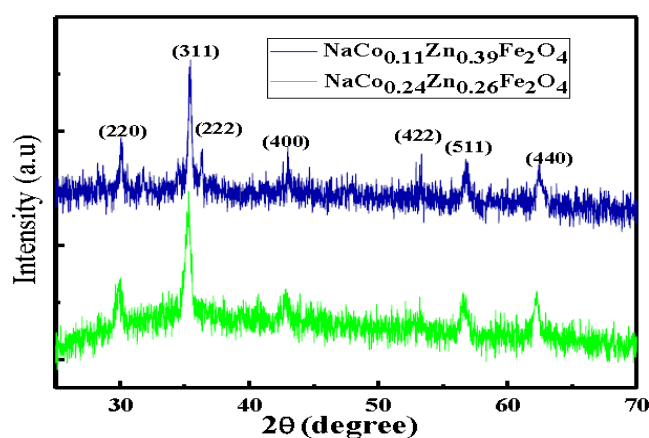
And  $d = \lambda / 2\sin \theta$  (where "d" is the inter planning spacing)

Since the structure is cubic so the formula for lattice parameter is given that;

$$a = d\sqrt{h^2 + k^2 + l^2}$$

Where "a" is lattice constant and (h,k,l) are miller indices, and obtained from diffracted peaks. The average crystallite size (D) was calculated from the Debye Scherer Formula:

$$D = \frac{K\lambda}{\beta \cos \theta}$$

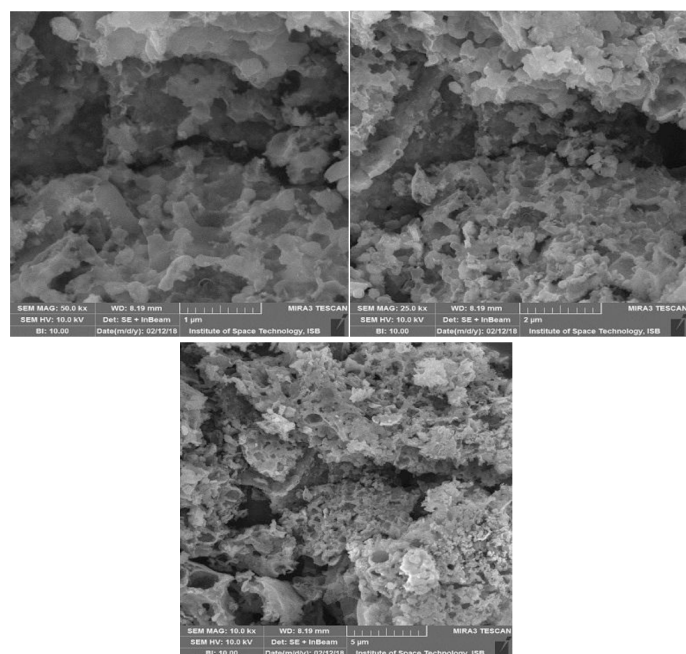


**Figure 1:** XRD patterns of the  $\text{NaZnFe}_2\text{O}_4$  sintered at 1000°C in air

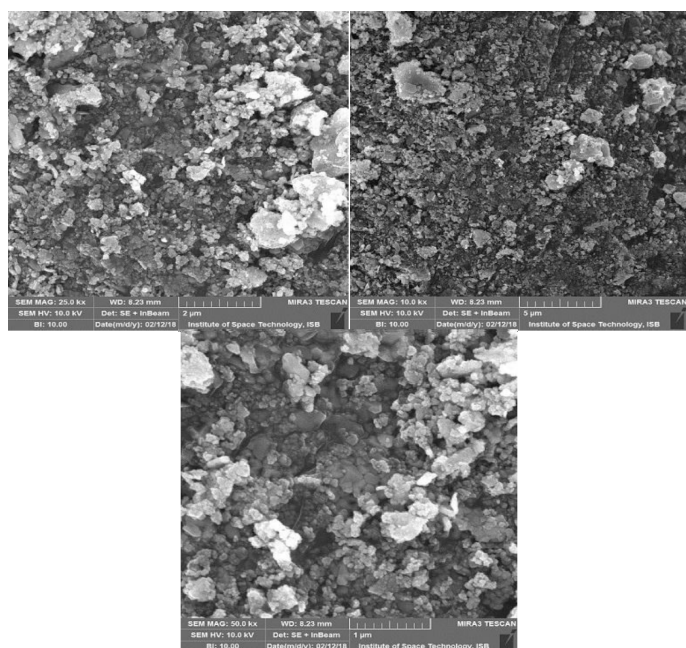
In the below table showed that the lattice parameter and crystallite size slightly changes because the ionic radii of Co and Zn are approximately near, while the crystallite size is slightly decreased at the same sintering temperature with increasing concentration of Co. The stresses in the sample released due to small size of ionic radii of Co. The diffraction peak are slightly shift in the direction of higher theta point as shown in the Figure 1, which reveal that lattice parameter decreased and the crystallinity increased and samples are free the strain which it may be removed the crystal defect.

The Scanning Electron Microscope (SEM) was use the samples morphology and topography. The scanning electron micrograph with magnification 1 $\mu\text{m}$ , 2 $\mu\text{m}$  and 5 $\mu\text{m}$  of both samples as shown in Figure 2 and 3. The particles agglomerated in samples at sintering temperature 1000°C as shown in Figure 2. With increasing the concentration of Co the particles separated nevertheless with the substitution of Co the particles nearly spherical-shaped with the apparent boundary and there are small amount of pore are also present in the sample ( $\text{NaCo}_{0.24}\text{Zn}_{0.26}\text{Fe}_2\text{O}_4$ ) as shown in Figure 3. The particles sizes were measured by using line intercept method as shown from table 1 is between 300nm to 400nm. The particles in sample  $\text{NaCo}_{0.11}\text{Zn}_{0.39}\text{Fe}_2\text{O}_4$ , shows that concentration of cobalt and zinc having effect on the grain morphology. The combined particle size occurs between 300 nm to 400 nm. From the SEM graphs the approximate particle size can be estimated by using the line intercept method;

Particle size= length of line/numbers of grains



**Figure 2:** SEM micrograph of the  $\text{NaCo}_{0.11}\text{Zn}_{0.39}\text{Fe}_2\text{O}_4$  sintered at  $1000^\circ\text{C}$  in air.



**Figure 3:** SEM micrograph of the  $\text{NaCo}_{0.24}\text{Zn}_{0.26}\text{Fe}_2\text{O}_4$  sintered at  $1000^\circ\text{C}$  in air.

**Table1:** Lattice parameter, Crystal size, Density and Average Grain Size of  $\text{NaCoZnFe}_2\text{O}_4$

| Samples   | Lattice parameter (Å) | Crystal Size (nm) | Dx [ $\text{g}/\text{cm}^3$ ] | Average Grain Size (nm) |
|---|-----------------------|-------------------|-------------------------------|-------------------------|
| $\text{NaCo}_{0.11}\text{Zn}_{0.39}\text{Fe}_2\text{O}_4$ | 8.31                  | $24 \pm 1$        | 5.2                           | 394                     |
| $\text{NaCo}_{0.24}\text{Zn}_{0.26}\text{Fe}_2\text{O}_4$ | 8.42                  | $16 \pm 2$        | 4.99                          | 356                     |

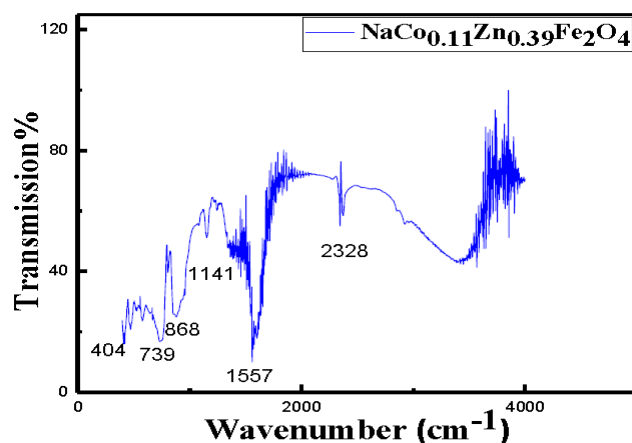
The material density can be calculated from the given below

equation:

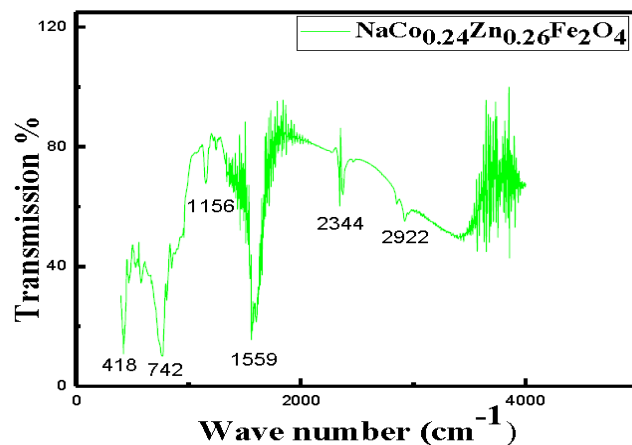
$$D = \frac{8M}{N_A V}$$

Where “8” is the number of molecules per unit cell, “M” is molar mass; “ $N_A$ ” is the Avogadro number and “V” is the volume in cubic cell which is equal to ( $V=a^3$ ).

The Infrared (IR) spectra of the samples  $\text{NaCoZnFe}_2\text{O}_4$  (where  $x=0.26$  and  $x=0.39$ ) are shown in the Figure 4 and 5. The infrared (IR) spectra of a sample will be confirmed that the transmission wavelength ranges are from  $600 \text{ cm}^{-1}$  to  $400 \text{ cm}^{-1}$  respectively. The composition of infrared spectra is under study which tells about the structure of a single part of cubic spinel. According to IR absorption band theory this the first band location which lies at the  $550 \text{ cm}^{-1}$  to  $600 \text{ cm}^{-1}$  is recognized the tetrahedral site, while the second absorption band which showed that  $335 \text{ cm}^{-1}$  to  $430 \text{ cm}^{-1}$  which locate in octahedral group. The functional group of the samples are carbon- carbon double bond ( $\text{C}=\text{C}$ ), while it lying on the alkaline and alkenes group. The location of absorption band will be compositional dependence, so there dependent can be recognized by the change in the cation oxygen bonds.



**Figure 4:** FTIR patterns of the  $\text{NaCo}_{0.11}\text{Zn}_{0.39}\text{Fe}_2\text{O}_4$  sintered at  $1000^\circ\text{C}$  in air



**Figure 5:** FTIR patterns of the  $\text{NaCo}_{0.24}\text{Zn}_{0.26}\text{Fe}_2\text{O}_4$  sintered at  $1000^\circ\text{C}$  in air

## Conclusion

In the present work, the effect of cobalt substitution in sodium zinc ferrite with chemical composition  $\text{NaCo}_{0.5-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  (where  $x=0.26$  and  $0.39$ ) on its structure. Physical properties were studied the sample  $\text{NaCo}_{0.11}\text{Zn}_{0.39}\text{Fe}_2\text{O}_4$  and  $\text{NaCo}_{0.24}\text{Zn}_{0.26}\text{Fe}_2\text{O}_4$  were successfully synthesized by sol-gel method. The samples were characterized by X-ray diffraction (XRD), Scanning Electron Microscope (SEM) And Fourier Transform Infrared Spectroscopy (FTIR). The XRD profile confirmed the sample sintered at  $1000^\circ\text{C}$  were angle phase cubic spinel structure. The crystalline size of prepared nano-particles was calculated by Scherer equation and to be between 15.5 nm to 24 nm. The SEM results show decreased that the particles were agglomerated and there were no sharp boundaries between particles on sample  $\text{NaCo}_{0.11}\text{Zn}_{0.39}\text{Fe}_2\text{O}_4$  but with increase of concentration of Co a particle appeared have definite boundaries and particle size slightly decreased. The infrared spectroscopy tells about the structure of a single part of simple cubic spinel. In this infrared spectroscopy the carbon- carbon double bonding are occur which are called Alkenes bonding.

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