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# Study of Structural and Magnetic Behaviour of $Ni_{0.75}Zn_{0.25}Fe_2O_4$ and $Ni_{0.25}Zn_{0.75}Fe_2O_4$ Ferrite Nanoparticles Annealed at 550°C, 650°C and 700°C, Synthesized by Citrate Precursor Method

• **Tarbia Jamil** • **Rashmi Kumari** • **Priya Kumari**
• **Rakesh Kumar Singh**

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Corresponding Author : Rakesh Kumar Singh

**Abstract :** *$Ni_{0.75}Zn_{0.25}Fe_2O_4$ and $Ni_{0.25}Zn_{0.75}Fe_2O_4$ ferrite nanoparticles were synthesized using citrate precursor method. The citrate precursor was annealed at temperature 550°C, 650°C and 700°C. The annealed powders were characterized using X-ray diffractometer. (XRD) and vibrating sample magnetometer (VSM). Observed XRD data display cubic spinel structure. The average particle size was observed 34nm, 35nm and 35 nm for $Ni_{0.75}Zn_{0.25}Fe_2O_4$ ferrite and 34nm, 36nm and 36nm for $Ni_{0.25}Zn_{0.75}Fe_2O_4$ ferrite at the given annealing temperatures 550°C, 650°C and 700°C,. The Magnetization, Retentivity and Coercivity for $Ni_{0.75}Zn_{0.25}Fe_2O_4$ was found 61.38 emu/g, 13.11 emu/g and 113G at annealing temperature 550°C, 39.09 emu/g, 8.10 emu/g and 80G at annealing temperature 650°C and 43.21emu/g, 6.77 emu/g and 90G at annealing temperature 700°C respectively. Similarly the Magnetization, Retentivity and Coercivity for $Ni_{0.25}Zn_{0.75}Fe_2O_4$ ferrite were found to be 15.05 emu/g, 1.35 emu/g and 55G at annealing temperature 550°C, 29.16emu/g, 1.27 emu/g and 43G at annealing temperature 650°C and 32.08emu/g, 2.12 emu/g and 55G at annealing temperature 700°C, respectively*

***Key words**: Ni-Zn Ferrite Nanoparticles, Citrate precursor method, Magnetic properties.*

**Tarbia Jamil**
B.Sc. II year, Physics (Hons.), Session: 2011-2014,
Patna Women's College, Patna University, Patna,
Bihar, India

**Rashmi Kumari**
B.Sc. II year, Physics (Hons.), Session: 2011-2014,
Patna Women's College, Patna University, Patna,
Bihar, India

**Priya Kumari**
B.Sc. II year, Physics (Hons.), Session: 2011-2014,
Patna Women's College, Patna University, Patna,
Bihar, India

**Rakesh Kumar Singh**
Assistant Professor, Department of Physics,
Patna Women's College, Patna University, Bailey Road,
Patna – 800 001, Bihar, India
E-mail : rakeshsinghpu@gmail.com

## Introduction:

Ferrites are technologically important engineering material and an object of study for quite long time (Ishino and Narumiya, 1987; Sugimoto,1999). Nickel-Zinc ferrite has been extensively used as high permeability material and low eddy current loss. Research in this field has received a major boost in the recent years when new methods for synthesis and characterization of small size nanoparticles were developed. Several research groups are exploring the possibility of preparing ferrites with novel properties by forcing the system to acquire metastable and nonequilibrium configurations (Singh et al.2011, Albuquerque *et al.,* 2000). Apart from the application aspects, investigations have been directed towards understanding the basic physics of nanoscale interactions. Synthesizing these materials in nanophase leads to different drastic properties (Upadhyay *et al.,* 2001, Singh et al. 2010). The magnetic properties of these ferrites are mainly controlled by the cation distribution of Ni, Zn and Fe among the available tetrahedral A sites and octahedral B sites. Zinc is known to have a high degree of affinity for the tetrahedral sites in the spinel structure and Nickel has a similar affinity for octahedral B site. The cation preferences can be greatly altered by preparing the spinel ferrites in nanosize. Several results have indicated that for particle size less than or around 10nm, sizeable fraction of zinc present in spinel structure occupies octahedral B sites, against its normal preference. The magnetic properties are likewise altered once the cation distribution is changed. $Ni_{0.5}Zn_{0.5}Fe_2O_4$ offers the most complex cationic distribution in the series of Ni-Zn ferrites (Singh et al. 2010). Most of the studies on nanophase soft and hard ferrites therefore have been associated with the cationic distribution in the structure (Peng Zhijian et al., 2011,Singh et al 2012). However, very less emphasis has been paid on the growth process of these systems. In this work we will try to study the systematic growth of this system by allowing them to grow under the influence of thermal energy, annealed at temperature 550°C, 650°C and 700°C and study their magnetic and cationic distribution in the structure by X-ray diffractometer (XRD) and vibrating sample magnetometer (VSM).

**Materials and Methods :**

Several research groups have used citrate precursor method for the synthesis of nano ferrites (Singh e*t al.*, 2011, Costa *et al.,* 2003; Gajbhiye *et al.,* 1996). In this method citrates/acetates of the salts are homogenized in presence of citric acid, and proper refluxing, drying and further annealing leads to the final product. In the present study the citrate route was followed for synthesis of nickel-zinc ferrite.The starting materials used for preparation of nickel-zinc ferrite were: ferric nitrate, zinc nitrate, nickel nitrate and citric acid (AR Grade). The solutions were then mixed together. The solution was then heated to 80°C and maintained at that temperature for 2 hours in a round bottom flask using a heating mantle under constant refluxing and stirring conditions using a magnetic stirrer. The refluxed solution was then slowly cooled to form a viscous solution. Finally, the viscous solution was dried at 60°C for 24 hours to form a brown fluffy mass, which was crumbled to form the precursor powder.

**Results and Discussion:**

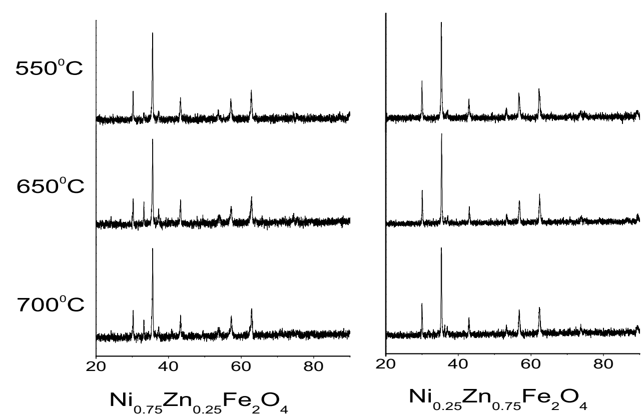


**Fig 1. XRD spectrum of nano ferrite samples**

Figure 1 shows the x-ray diffraction of the Precursor Powder samples annealed at different temperature. It is very clear from the pattern that all the samples have been formed in single phase and well crystalline in nature. In $Ni_{0.75}Zn_{0.25}Fe_2O_4$ nanoparticles, additional peak of small intensity was observed near prominent peak. The particles size of these samples has been estimated by Scherrer formula using the most intense peak (Callister, 2005; Culity and Graham, 2009). The average particle size of the sample annealed at 550°C turns out to be around 34 nm and 33 nm for samples $Ni_{0.75}Zn_{0.25}Fe_2O_4$ and $Ni_{0.25}Zn_{0.75}Fe_2O_4$ · On further increase in the annealing temperature the size increases to 35 nm and 36 nm (Table-1) for temperature 650°C and 700°C annealed sample. However as expected there is no change in the particle size of the sample annealed at 650°C and 700°C. This indicates that the sample grows up to a temperature somewhere below 650°C and become almost constant afterwards.

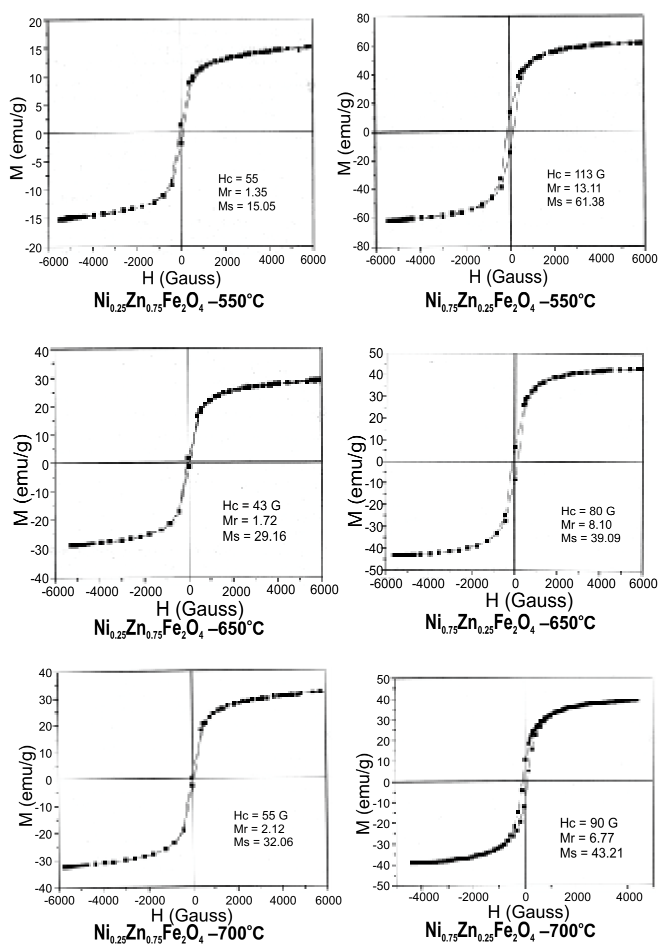


**Fig 2. Magnetization Curves of Nano ferrite samples annealed at temperature 550°C, 650°C and 700°C**

| Composition | $Ni_{0.75}Zn_{0.25}Fe_2O_4$ | | | | $Ni_{0.25}Zn_{0.75}Fe_2O_4$ | | | |
|---|---|---|---|---|---|---|---|---|
| Annealing Temp | Magnetization (emu/g) | Retentivity (emu/g) | Coercivity (G) | Particle size (nm) | Magnetization (emu/g) | Retentivity (emu/g) | Coercivity (G) | Particle size (nm) |
| 550 °C | 61.38 | 13.11 | 113 | 34 | 15.05 | 1.35 | 55 | 33 |
| 650 °C | 39.09 | 8.10 | 80 | 35 | 29.16 | 1.72 | 43 | 36 |
| 700 °C | 43.21 | 6.77 | 90 | 35 | 32.06 | 2.12 | 55 | 36 |

**Table 1. Observed data of nano ferrite samples from XRD And VSM**

In order to look in to the magnetic behaviour of these samples we performed magnetization studies on them using VSM. Similar behaviour was also observed for $Ni_{0.75}Zn_{0.25}Fe_2O_4$ and $Ni_{0.25}Zn_{0.75}Fe_2O_4$ nano ferrite samples. Figure 2 shows the M vs H loop of these samples. The inset of each graph shows the behaviour of the sample at low applied field. Here also we see striking difference between the samples annealed at 550°C, 650°C and 700°C. For sample annealed at 550°C shows a saturation magnetization value of 61. 38 emu/g, where as the sample annealed at 700°C shows that of 43.21 emu/g. This clearly indicates that the magnetic configurations of these samples are different. It is known for Ni-Zn ferrite to have a very strong preference for a particular site when they are in bulk form. Mainly it is octahedral preference for Ni and tetrahedral preference for Zn. Having a relatively high value of saturation magnetization for the sample annealed at 550°C clearly indicates that cations are surely deviated from their normal preferences. When annealed at higher temperature, the cations under the influence of the heat energy tries to regain their normal preferences, which leads to the higher value of the saturation magnetization in the samples annealed at 700°C and 650°C (Table). Such behaviour in structural and magnetic properties of nanoferrite sample, synthesized by same precursor method was also observed (Kumari Arpana et al 2011, Singh Shanta et al, 2011).

**Conclusion:**

$Ni_{0.75}Zn_{0.25}Fe_2O_4$ Samples have almost same particle size( 34nm, 35nm and 35nm) at annealing temperature 550$^0$C, 650$^0$C and 700$^0$C. While magnetization was found to change.The highest magnetization 61.38 emu/g was observed at annealing temperature 550$^0$C of $Ni_{0.75}Zn_{0.25}$ $Fe_2O_4$. $Ni_{0.25}Zn_{0.75}$ $Fe_2O_4$ Samples have also have same particle size (33nm, 36nm and 36nm) at annealing temperature 550$^0$C, 650$^0$C and 700$^0$C. While the magnetization was found to increased with increase in annealing temperature. The highest magnetization was observed 32.06 emu/g for sample annealed at 700$^0$C of $Ni_{0.25}Zn_{0.75}Fe_2O_4$ Nanoparticles. In this work, at anneeling temperature 550$^0$C, 650$^0$C and 700$^0$C, the particle size is almost same. But due to different stoichiometric ratio of Ni and Zn, cation distribution and annealing temperature the magnetization, Retertentivity and Coercivity were found different. This megnetic properties depends on annealing temperature and cations distribution, while the nanomaterial was synthesized using Citrate precursor method. Synthesized nanoparticles grows up to a temperature somewhere below 650$^0$C becomes constant afterwards.

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