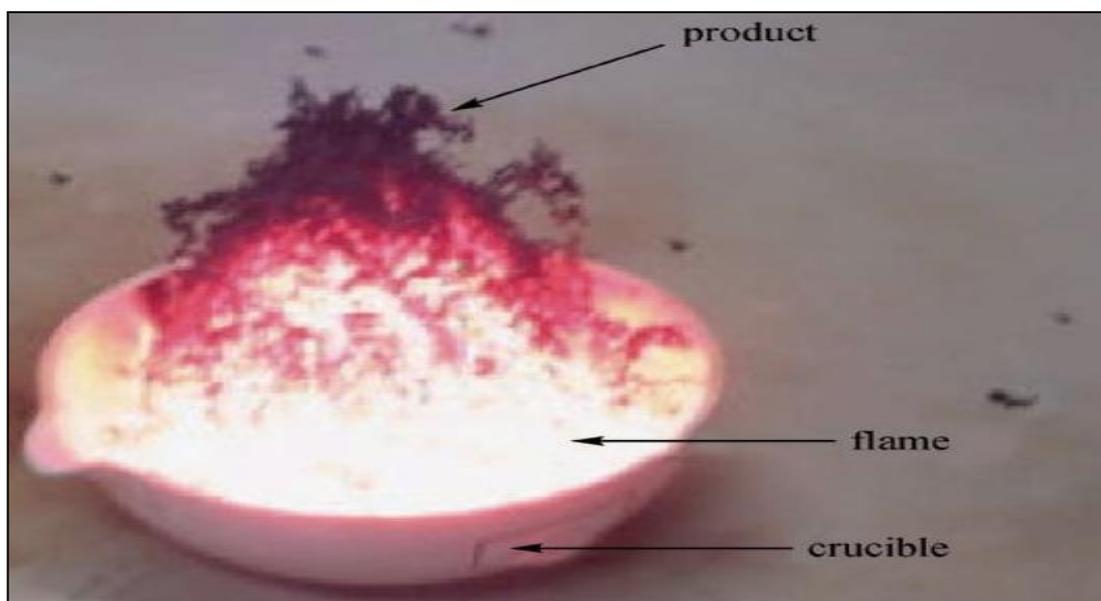




**SYNTHESIS AND CHARACTERIZATION OF NICKEL-ZINC SPINEL
FERRITE BY SOL-GEL AUTOCOMBUSTION TECHNIQUE**



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ABSTRACT

The spinel ferrite can be synthesized in nano crystalline form using various methods-such as chemical co-precipitation,hydrothermal sol-gel etc. we have synthesized nanocrystalline Nickel-Zinc ferrite by sol-gel auto combustion method. The prepared sample was sintered at 700⁰c for 6 hr. Structural analysis of prepared sample was examined by using x-ray diffraction. The surface morphology was investigated by SEM.

KEYWORD- Ni-Zn spinel ferrite, sol-gel autocombustion technique,XRD, SEM.

1.INTRODUCTION-

Ferrites are ferrimagnetic materials with good magnetic,dielectric properties and large number of technological applications in satellite, communication, memory devices, computers, components,filter components,antenna rods, transformer cores etc. because of their good electronic and magnetic properties[1].The high electrical resistivity, low eddy current and dielectric losses,

moderate saturation magnetization, easy and low cost of preparation, high curie temperature and high permeability are the remarkable characterization of ferrite material which makes them useful in variety of applications. The properties of ferrites depends upon magnetic interaction, cation distribution in the two sub lattice, method of preparation, preparative parameters, type and amount of dopant etc.[2-4].The dielectric properties of ferrites are depends upon different factors including the method of preparation ,chemical composition and grain structure. Ni-Zn ferrite possess mixed spinel has high electric resistivity, low eddy current and dielectric loss, moderate saturation magnetization, high curie temperature and wide applications in many fields[5-6].

Taking into consideration the importance of Ni-Zn ferrite the aim of present work was to synthesize metal ferric nanoparticle of mixed nickel-zinc spinel($\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Fe}_2\text{O}_4$) ferrite using wet chemical sol-gel auto combustion method and find out various structural, morphological and dielectric properties.

2. EXPERIMENTAL

In the synthesis of Nickel -Zinc spinel ferrite AR grade with high purity (99.99 %) nitrates of metal ions(Nickel, Zinc and ferric) were used and L-Ascorbic acid was used as fuel.All as nitrates and L-Ascorbic acid as per calculation was taken and dissolved in minimum amount of double distilled water in separate beaker. The metal nitrate to fuel ratio was taken 1:3. All thesesolution are mixed homogenously in single beaker by continues stirring. Ammonia was added to adjust thep^Hofvalue 8 of initial solution. This solution was evaporated at 75⁰c to get a dense sticky gel, then temperature was increased to 110⁰c for the dehydration process. The temperature was then increased rapidly and when it reached approximately 120⁰c large of gases((CO₂, H₂O, N₂) were liberated and dark brown power was produced through combustion process. The prepared power of Ni-Zn spinel ferrite is sintered at 700⁰c for 6 h and various properties of Ni-Zn ferrite were studied.

3. RESULTS AND DISCUSSIONS

A) Structuralproperties:- X-ray diffraction(XRD) is an important tool to check the phase purity and crystal structure of prepared sample.In present study x-ray diffraction technique was used to characterize $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Fe}_2\text{O}_4$ spinel ferrite sintered at 700⁰ c for its phase purity. X-ray diffraction pattern recorded at room temperature for sample is shown in fig1

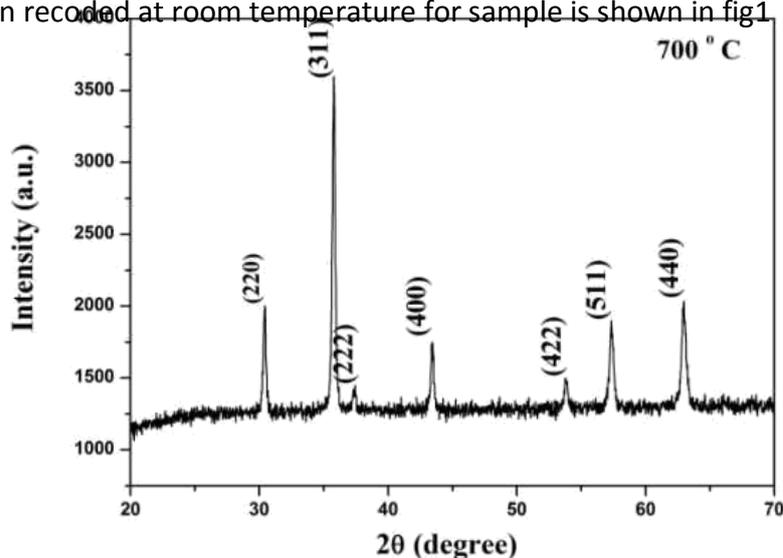


Fig 1 XRD pattern of $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Fe}_2\text{O}_4$ nanoparticle at temperature 700⁰c.

A close observation of XRD pattern suggest that planes with miller indices (220),(311),(222), (400),(422),(511), and (440) belongs to cubic spinel structure. These planes have sufficient intensity and are broader. The values of Bragg's angle 2θ , inter planer spacing d and intensity of various planes for sample are given in Table 1

Table 1: Miller indices (h k l), Bragg's angle (θ) inter planar spacing (d), intensity (I) and relative intensity ratio (I/I_0) of $Ni_{0.65}Zn_{0.35}Fe_2O_4$ nanoparticle for $700^\circ C$

(h k l)	2θ (degree)	θ (degree)	$\sin(\theta)$	$\sin(\theta)/\lambda$	d (Å)	I (a.u)	I/I_0
(220)	30.422	15.211	0.262	0.17017	2.935	1901.3	54
(311)	35.789	17.895	0.307	0.19929	2.506	3508.0	100
(222)	37.423	18.712	0.321	0.20807	2.401	1404.9	40
(400)	43.437	21.719	0.370	0.24001	2.081	1686.0	48
(422)	53.834	26.917	0.453	0.29361	1.701	1469.1	42
(511)	57.354	28.677	0.480	0.31124	1.605	1814.0	52
(440)	62.962	31.481	0.522	0.33870	1.475	1965.5	56

The most intense peak (311) observed in XRD pattern of sample is considered for determining the crystallite size. The crystallite size is obtained by using relation [7] The obtained values of crystallite size are given in table 2..From table 2 it is clear that crystallite size was 18 nm indicating nan crystalline nature of sample

Table 2: Lattice parameter(a), X-ray density(d_x), bulk density(d_B), Porosity(P%), unit volume(v) and crystallite size(t) of $Ni_{0.65}Zn_{0.35}Fe_2O_4$ nanoparticle for $700^\circ C$

Sintering temp. maintain at	a (Å)	d_x (gm/cm ³)	d_B (gm/cm ³)	P %	V (Å ³)	t (nm)
$700^\circ C$	8.324	5.4527	2.6317	51.73	576.76	18

Lattice constant(a) of sample was calculated using standard relation for cubic spinel structure. From table 2 it is observed that lattice constant of Ni-Zn sample is in between lattice constant of pure Nickel ferrite and Zinc ferrite.

X-ray density (d_x) of sample was calculated using following relation [8] and obtained value is presented in table 2.

$$d_x = 8M/NV \text{ gm/cm}^3$$

M-molecular weight, N-Avogadro's number and $V=a^3$ unit cell volume.

Bulk density (d_B) of present sample was measured by using Archimedes principle. The value of bulk density is given in table 2, which is less as compared to x-ray density.

Percentageporosity (P%) is calculated by taking known values of bulk density and x-ray density. The percentageporosity was calculated [9] and its value is given in table 2.

Hopping length of tetrahedral and octahedral site (L_A) and(L_B) for the present sample was calculated using following relation[10].

$$L_A = a\sqrt{(3)}/4 \text{ \AA}$$

$$L_B = a\sqrt{(2)}/4 \text{ \AA}$$

The value of hopping length L_A and L_B are given in table 3.

Table 3: Hopping length (L_A , L_B), Tetrahedral bond (d_{AX}),Octahedral bond (d_{BX}),Tetra edge (d_{AXE}) and Octa edge (d_{BXE}) of $Ni_{0.65}Zn_{0.35}Fe_2O_4$ nanoparticles for (700 ° C).

Sintering temp. maintain at	L_A (Å)	L_B (Å)	d_{AX} (Å)	d_{BX} (Å)	d_{AXE} (Å)	d_{BXE} (Å)	
						Shared	Unshared
700 ° C	3.6044	2.9430	1.8887	2.0323	3.0842	2.8017	2.9446

From table it is seen that hopping length L_A is less than L_B .

Table 4: Ionic radii (r_A , r_B) $Ni_{0.65}Zn_{0.35}Fe_2O_4$ nanoparticles for (700 ° C)

Sintering temp. maintain at	r_A (Å)	r_B (Å)
700 ° C	0.5687	0.7111

The tetrahedral radius(r_A) and octahedral radius(r_B) for present sample were calculated using the following relations[11].and their values are given in table 4

$$r_A = \left(u - \frac{1}{4}\right) a\sqrt{3} - r(O^{2-})\text{Å}$$

$$r_B = \left(\frac{5}{8} - u\right) a - r(O^{2-})\text{Å}$$

Where u is oxygen positional parameter($u = 0.381$) $r(O)$ is radius of oxygen ion(1.32 \AA)

Tetrahedral and Octahedral edge:- The structural parameters like tetrahedral edge and octahedral edge, shared and unshared were calculated using following relations[12].

$$d_{AX} = a(3(u-1/4))^{1/2} \text{ \AA}$$

$$d_{BX} = a[3u^2 - (11/4)u + (43/64)]^{1/2} \text{ \AA}$$

$$d_{AXE} = a[2(2u-1/2)]^{1/2} \quad \text{Å}$$

$$d_{BXE} = a[2(1-2u)]^{1/2} \quad \text{Å}$$

$$d_{BXEU} = a\sqrt{(4u^2 - 3u + 11/16)} \text{Å}$$

Using the values of the lattice constant (a), the various other structural parameters like tetrahedral bond length (d_{AX}), octahedral bond length (d_{BX}), tetra edge (d_{AXE}) and octa edge (d_{BXE})

B. Morphological Characterization:-

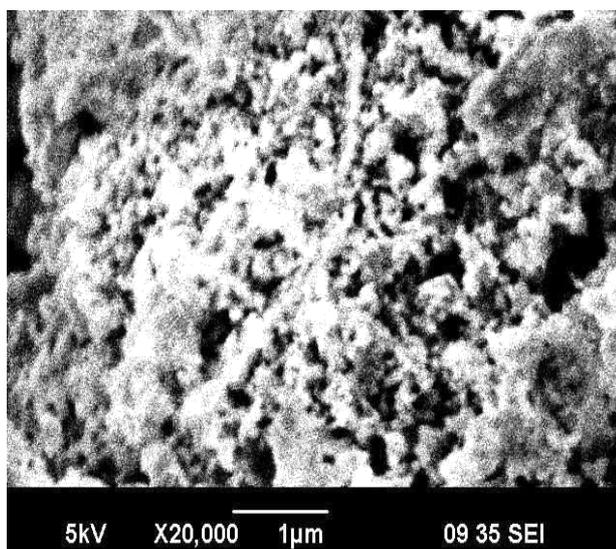


Figure 2 SEM image of $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Fe}_2\text{O}_4$ nanoparticle at temperature 700°C .

Surface morphology and average grain size of sample were determined by using scanning electron microscopy technique. The average grain size and specific surface area was calculated by relation [13] and presented in table 5

$$G_{\text{avg}} = \frac{1.5L}{MN}$$

L-total test line length

M-magnification

N-total number of interception.

Table 5 Grain size(G) and specific surface area(S) from SEM image for $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Fe}_2\text{O}_4$ nanoparticle at 700°C

Sintering temp. maintain at	Grain Size (nm) SEM	Specific Surface Area (m^2/g)
700°C	79	13.62

4. CONCLUSIONS

The nano crystalline $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Fe}_2\text{O}_4$ ferrite for sintering temperature 700°C was successfully synthesized by sol-gel auto combustion method. The XRD results formation of single phase cubic spinel structure. The crystallite size confirms the nano crystalline nature of sample. The crystallite size of sample calculated by using Debye Scherrer's formula was obtained is 18 nm. The average grain size determined from SEM technique is 79 nm.

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