FTIR STUDIES OF TEMPERATURE DEPENDENT NICKEL ZINC FERRITES SYNTHESIZED BY SOLUTION AUTO COMBUSTION METHOD

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ABSTRACT
Nickel zinc ferrite samples were synthesized from metal nitrates precursors via solution auto combustion method, using different synthesis conditions. Samples were prepared at different temperatures 400°C, 600°C, 800°C and 1000°C with heating rate 10°C/min. for 4 hours. The optical measurements are carried out by a FTIR. The nickel zinc ferrite exhibits a single-spinel structure. From FTIR spectrum two fundamental bands around 600–400 cm\(^{-1}\) were observed, that are the common features of all ferrite materials. Improved optical properties was found for sample prepared at 1000°C for 4 hours (with 10°C/min) show better Sample prepared at 10°C/min (800°C for 4 hours) show improved results as compared to sample at 25°C/min (800°C for 4 hours).

Keywords: Nickel zinc ferrite, auto combustion method, calcination, FTIR studies.

I. INTRODUCTION
Nickel-Zinc ferrites are considered as one of the most versatile soft ferrites materials because of its high resistance and low eddy current losses [1]. Ni-Zn ferrite has been commercially used in radio frequency circuits, high quality filters, rod antennas and transformer cores [2, 3]. Ferrites crystallize into spinel type which has tetrahedral A site and octahedral B site. The properties of the ferrites can be tuned by varying the cation and their distribution among tetrahedral A site and octahedral B site. Further bulk properties of ferrite change as its dimensions are changed to nanoscale [1–4]. Superparamagnetism, spin canting, and metastable cation distribution are some of the phenomena which have been observed in nanoparticles of various ferrites. The structural, magnetic & dielectric parameters depend on the distribution of these ions on tetrahedral and octahedral sites. This distribution is different when the ferrite is synthesized at low temperature [4]. The method of powder synthesis plays an important role in the properties of the ferrite nanoparticles. There are many methods to synthesize Ni-Zn ferrite, such as co-precipitation, thermal plasma [6], hydrolysis [7] etc. The influences of calcinations temperature on the structural and magnetic properties of Ni-Zn ferrites have been reported by various researchers [8]. It is also believed that several researchers synthesized Ni-Zn ferrite by conventional methods like double sintering method, solid state reaction method, and chemical co precipitation method, hydro thermal process etc. [9-12]. Priyadharsini et al. [13] have reported the FTIR investigation of nanostructured Ni-Zn ferrite. In the present investigation we report the FTIR studies of the Ni-Zn ferrite samples prepared by solution auto combustion method. The influences of heating rate and calcinations temperature on optical properties (FTIR) were also investigated.
II. EXPERIMENTAL DETAILS

Materials and Method Used
Nickel Nitrate Hexahydrate, Zinc Nitrate Hexahydrate, Iron Nitrate Nonahydrate, Citric Acid (Himeida, AR), Ammonia Solution (Sigma Aldrich, 33%). Method used for sample preparation was sol-gel auto combustion method.

Samples Preparation
The powders of Ni-Zn ferrites (Ni$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$) were prepared by solution auto combustion method. Zinc nitrate hexahydrate, Zn(NO$_3$)$_2$6H$_2$O, nickel nitrate hexahydrate Ni(NO$_3$)$_2$6H$_2$O and iron nitrate nonahydrate Fe(NO$_3$)$_3$9H$_2$O were used Zinc, nickel and iron precursors respectively. The starting materials used in this preparation are zinc nitrate hexahydrate, nickel nitrate hexahydrate and iron nitrate nonahydrate and citric acid were dissolved in deionized water. Citric acid acts as fuel for combustion process. The pH of the solution was adjusted to 8 using ammonia solution [9]. The solution was uniformly heated at 100°C with constant stirring to transform it into a gel and. The black brown viscous gel was combusted with the evolution of large amount of gases and it resulted in the formation of loose powder. Ground the as prepared powder (S0) for one hour and heat the powder at temperature 800°C (S5) with 25°C/min. for 4 hours. Another series are 400°C(S1), 600°C(S2), 800°C(S3) and 1000°C(S4) with heating rate of 10°C/min. for 4 hours. Fig. 1 demonstrates the solution auto combustion process.

Characterization Techniques
The FTIR (Perkin Elmer-Spectrum RX-IFTIR) spectra recorded to measure the optical properties of the samples

III. RESULTS AND DISCUSSIONS

FTIR
The FTIR spectra are shown in Figure 2 and 3 which helps in confirming the formation of the spinel structure in the nickel-zinc samples. The IR spectral studies on ferrite compounds were recorded between 400 and 3000 cm$^{-1}$. Several absorption bands were recorded in a decreasing number as the calcined temperature increased from 400 to
1000°C. With increase in temperature from 400°C to 1000°C the lower frequency bands slight shift towards high frequency and intensity of bands increases. Sample S4 shows the high intense band at $586.42 \text{cm}^{-1}$.

![FTIR spectra of samples S3 and S5](image1)

**Fig.2** The FTIR spectra of samples S3 and S5

![FTIR spectra of samples S0, S1, S2, S3, S4](image2)

**Fig.3** The FTIR spectra of samples S0, S1, S2, S3 and S4.

The spectra show the position of the ions in the crystal structure and their vibration modes, which represents the various ordering positions on the structural properties of the synthesized compounds. In the present study, the absorption bands for the synthesized ferrites are in the expected range. The spectra show two main absorption bands below 1000 cm$^{-1}$ which is a common feature of ferrites. As synthesized NZF shows absorption bands at 420.29(1) and 558.57 cm$^{-1}$(2). The high frequency band lies in the range 550–600 cm$^{-1}$ while the low frequency band lies in the range 474–420 cm$^{-1}$. These bands are assigned to the vibrations of the metal ion-oxygen complexes in tetrahedral and octahedral sites, respectively [14, 15]. Waldron [15] attributed the high frequency band to the intrinsic vibrations of the tetrahedral groups and the low frequency band to the octahedral groups. Puri and Varshney [16] have reported that Zn$^{2+}$ showed a strong preference for tetrahedral A site and Ni$^{2+}$ ion showed preference for B site. At the temperature of 1000°C, there were two considerable bands appeared at 574.61 (1) and 2340.90 cm$^{-1}$ (3) which were assigned to the metal ion-oxygen complexes in the tetrahedral sites and traces of adsorbed or atmospheric CO$_2$, respectively. The light band around 1630(3) could be attributed to the adsorbed water or humidity.
The variation in the band positions is due to the difference in the Fe–O distances for the octahedral and tetrahedral complexes [19, 20]. The band values of the different samples are given in Table 1. Sample S5 (25°/min.) show less intense band around 570 cm⁻¹ as compared to sample S3(10°/min.).

<table>
<thead>
<tr>
<th>Position/Sample</th>
<th>S0</th>
<th>S1</th>
<th>S2</th>
<th>S3</th>
<th>S4</th>
<th>S5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Octahedral Sites</td>
<td>420.29</td>
<td>436.75</td>
<td>419.93</td>
<td>424.99</td>
<td>426.87</td>
<td>418.95</td>
</tr>
<tr>
<td>H₂O</td>
<td>1632.62</td>
<td>1632.53</td>
<td>1627.93</td>
<td>1627.94</td>
<td>1628.92</td>
<td>1627.94</td>
</tr>
<tr>
<td>Tetrahedral Sites(M-O)</td>
<td>558.56</td>
<td>554.48</td>
<td>571.68</td>
<td>575.59</td>
<td>586.42</td>
<td>574.61</td>
</tr>
<tr>
<td>Traces of CO₂</td>
<td>2353.75</td>
<td>2340.75</td>
<td>2342.91</td>
<td>2343.90</td>
<td>2337.75</td>
<td>2340.90</td>
</tr>
<tr>
<td>Humidity</td>
<td>1598.23</td>
<td>1588.93</td>
<td>1627.93</td>
<td>1627.94</td>
<td>1628.93</td>
<td>1627.94</td>
</tr>
</tbody>
</table>

IV. CONCLUSIONS

Ni-Zn ferrite samples were synthesized by sol-gel auto combustion technique. The FTIR spectral studies on ferrite compounds were studied between 400 and 3000 cm⁻¹. Several absorption bands were recorded in a decreasing number as the calcined temperature increased from 400 to 1000°C. The FTIR spectra demonstrate the position of the ions in the crystal structure and their vibration modes, which shows the various ordering positions on the structural properties of the synthesized compounds. In the present study, the absorption bands for the synthesized ferrites are in the expected range. As synthesized NZF shows absorption bands at 420.29 and 1632.62 cm⁻¹. There were two considerable bands appeared at 574.61 (1) and 2340.90 cm⁻¹ (3) which were assigned to the metal ion-oxygen complexes in the tetrahedral sites and traces of adsorbed or atmospheric CO₂, respectively at the temperature of 1000°C. Our results demonstrate that optical properties of sample improved by enhancement in temperature upto 1000°C. Effect of heating rate was also investigated. Sample prepared at heating rate 10°/min. show better results as compared to sample at 25°/min.

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REFERENCES