X-ray Diffraction, Infra-Red Spectroscopy and Magnetic Properties of Ni$_{0.7}$Zn$_{0.3}$Fe$_2$O$_4$+0.6wt%V$_2$O$_5$ Prepared via Ceramic Method

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Abstract: In view of the growing demand to the additives, we promote the compound formation of Ni$_{0.7}$Zn$_{0.3}$Fe$_2$O$_4$ loaded with 0.6wt%V$_2$O$_5$ by ceramic technique. The structural analysis and magnetic were determined by diverse characterization techniques. The phase formation and purity over the addition of V$_2$O$_5$ was identified by XRD pattern. In IR studied, the functional group scrutiny of Ni$_{0.7}$Zn$_{0.3}$Fe$_2$O$_4$+0.6wt%V$_2$O$_5$ was examined by locating the intrinsic vibrational bands and confirmed the phase formation. The lattice constant ($a$) and x-ray density ($dx$) were calculated from the post-examination of XRD data. We noted an inflation in the Saturation magnetization (Ms), Remanence magnetization (Mr) and magneton number ($n_0$) of Ni$_{0.7}$Zn$_{0.3}$Fe$_2$O$_4$+0.6wt%V$_2$O$_5$ as a principle cause of V$_2$O$_5$ loading. Admitting that, the rate of coercivity (Hc) was drastically declined suggesting the alternated magnetic parameters. This inspection was focused on the characteristic ramification of additive 0.6wt%V$_2$O$_5$ on physical properties of Ni$_{0.7}$Zn$_{0.3}$Fe$_2$O$_4$ ferrite.

INTRODUCTION

Nickel ferrite (NiFe$_2$O$_4$) is a inverse spinel, cubic structured soft ferrite along the general formula AB$_2$O$_4$; space group Fd$$_{3}$$m$O$_h$ [1, 2]. Precisely, the dynamic crystal nature of the ferrite materials has been studied by many researchers. In the nickel ferrite structure, eight tetrahedral voids (A)-sites are occupied by Fe$^{3+}$ ions, and sixteen octahedral voids (B)-sites are occupied by Fe$^{3+}$ and Ni$^{2+}$ ions equally [3]. Dimensionally, NiFe$_2$O$_4$ has a far-reaching spectrum of applications in various territories supported by its physico-chemical properties. Some of the paramount applications comprises environmental monitoring, bio-imaging, thermal analysis, chemical supercapacitors [4], EMV chips [5], Drug delivery systems [6], Ni-based batteries [7], MRI [8], sensors and actuators [9], etc. An inclusion of additives in the spinel ferrite performs an important role in modulating the microstructural properties of spinel ferrite [10]. Eventually, various oxides like niobium oxide (Nb$_2$O$_5$) [11], vanadium pentoxide (V$_2$O$_5$) [12], tungsten (IV) oxide (WO$_3$) [13], Lithium carbonate (Li$_2$CO$_3$) [14], Bismuth(III) oxide (Bi$_2$O$_3$) [15], Cobalt oxide (Co$_2$O$_3$) [16] etc. are used as additives. Vanadium pentoxide is an inorganic compound with the formula V$_2$O$_5$ is recognized as an ‘oxidizing agent’ by reason of its high degree of oxidation. Vanadium alloys are used as the surgical instruments and tools, steel additives, ferrovanadium, automobile crankshafts, axels, dental implants. Xueying Wang et.al has observed that the excess addition of non-magnetic V$_2$O$_5$ may depress the magnetic properties and disintegrates the grains. however, the appropriate proportion of V$_2$O$_5$ loading may complement the magnetic properties as well [17]. In this article, we anticipated to formulate the Ni$_{0.7}$Zn$_{0.3}$Fe$_2$O$_4$+0.6wt%V$_2$O$_5$ spinel ferrite practicing the solid state reaction method to study the structural and magnetic properties as a role of V$_2$O$_5$ mole percent.
EXPERIMENTAL

Essential Materials and Synthesis

The high purity (≥ 99.9%) Nickel oxide (NiO), Zinc oxide (ZnO), Ferric oxide (Fe₂O₃) and Vanadium oxide (V₂O₅) of were chosen as virgin ingredients for the compound construction of Ni₀.₇Zn₀.₃Fe₂O₄ + 0.6wt% V₂O₅ via solid state reaction. The respective oxides were weighed accurately and mixed homogeneously using agate pestle mortar for the better yield. Over the comprehensive grinding of initial ingredients, we have obtained Ni₀.₇Zn₀.₃Fe₂O₄+0.6wt%V₂O₅. On set, we heated the oxide ingredients at 200°C for the deportation of moisture from the compound. Next to this, the assimilated powder was heated at 900 °C for 24 h and repeatedly grounded it for 3 h and pressed into pellet shape (10×3 mm). Polyvinyl alcohol (PVA) was used as binder for the preparation of pellets. The sintering course at 1080°C was performed for the Ni₀.₇Zn₀.₃Fe₂O₄+0.6wt%V₂O₅ for 6 h in a muffle furnace.

CHARACTERIZATION

X-Ray Diffraction of Ni₀.₇Zn₀.₃Fe₂O₄+0.6wt%V₂O₅

XRD impressions of Ni₀.₇Zn₀.₃Fe₂O₄+0.6wt%V₂O₅ were taken by applying Cu-κα radiation (λ = 1.54056 Å) on (Bruker X-Advanced D8) ray diffractometer. The XRD parameters were characterized at room temperature supervised at 20 mA current 40 Kv voltage. The consolidated XRD pattern was inscribed in the 2θ range of 20° to 80° with a scanning rate of 0.01 deg/min and the major Bragg’s reflections of Ni₀.₇Zn₀.₃Fe₂O₄+0.6wt%V₂O₅ were indexed for the analysis of structural parameters.

Infrared spectroscopy of Ni₀.₇Zn₀.₃Fe₂O₄+0.6wt%V₂O₅

Infrared spectroscopy was recorded by NICOLET-NEXU-870 spectrometer using KBr pellet at room temperature in the wavenumber range of 0 cm⁻¹ - 4000 cm⁻¹. IR spectroscopy used to understand chemical bonding information and confirmed the formation of spinel structure.

Magnetic Property of Ni₀.₇Zn₀.₃Fe₂O₄+0.6wt%V₂O₅

The pulse field hysteresis loop tracer supplied by MAGNETA Pvt. Ltd. Mumbai was used to measure the magnetic properties of the samples at room temperature. The field applied during measurements was ±5000 Oe.

RESULTS AND DISCUSSIONS

X-ray diffraction analysis of Ni₀.₇Zn₀.₃Fe₂O₄+0.6wt%V₂O₅

The recognized peaks in XRD patterns are fine and intensified; without any extra peaks expressing the formation of complete ferrite phase. The existence of (220), (310), (311), (222), (400), (422), (511) and (440) planes in the XRD pattern affirms the architecture of single phase cubic spinel structure which can be seen in Figure 1. The loading of V₂O₅ additive in Ni-Zn ferrite boosts the mass transport mechanism forming a liquid phase during the sintering process, assisting the pore-free grains formation in due course of capillary force effect within the particles. The incorporation of V⁵⁺ ions into the spinel crystals of ferrite introduces some convoluted structures viz. constitution of Fe²⁺ ions and precipitation of Fe₂O₃ as a partial completion of ferrite phase.
The values of Miller Indices (hkl) and Intensity of various reflections of Ni0.7Zn0.3Fe2O4 + wt6% V2O5 are depicted in Table 1.

<table>
<thead>
<tr>
<th>(hkl)</th>
<th>2θ</th>
<th>θ</th>
<th>sinθ</th>
<th>sinθ/λ</th>
<th>d(Å)</th>
<th>I (a.u.)</th>
<th>I/I₀</th>
</tr>
</thead>
<tbody>
<tr>
<td>(220)</td>
<td>30.5</td>
<td>15.25</td>
<td>0.263</td>
<td>0.171</td>
<td>2.927</td>
<td>4691</td>
<td>55.653</td>
</tr>
<tr>
<td>(310)</td>
<td>33.5</td>
<td>16.75</td>
<td>0.288</td>
<td>0.187</td>
<td>2.672</td>
<td>3466</td>
<td>41.119</td>
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<tr>
<td>(311)</td>
<td>35.84</td>
<td>17.92</td>
<td>0.308</td>
<td>0.2</td>
<td>2.502</td>
<td>8429</td>
<td>100</td>
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<tr>
<td>(222)</td>
<td>37.46</td>
<td>18.73</td>
<td>0.321</td>
<td>0.208</td>
<td>2.398</td>
<td>3387</td>
<td>40.183</td>
</tr>
<tr>
<td>(400)</td>
<td>43.44</td>
<td>21.72</td>
<td>0.370</td>
<td>0.24</td>
<td>2.081</td>
<td>3758</td>
<td>44.584</td>
</tr>
<tr>
<td>(422)</td>
<td>53.82</td>
<td>26.91</td>
<td>0.453</td>
<td>0.294</td>
<td>1.702</td>
<td>3737</td>
<td>44.335</td>
</tr>
<tr>
<td>(511)</td>
<td>57.36</td>
<td>28.68</td>
<td>0.479</td>
<td>0.312</td>
<td>1.605</td>
<td>4991</td>
<td>59.212</td>
</tr>
<tr>
<td>(440)</td>
<td>62.98</td>
<td>31.49</td>
<td>0.522</td>
<td>0.339</td>
<td>1.474</td>
<td>5396</td>
<td>64.017</td>
</tr>
</tbody>
</table>

The values of Hopping length L_A, L_B, tetrahedral bond d_{AX}, octahedral bond d_{BX}, tetra edge d_{AXE}, octa edge d_{BXE} and octa edge unshared d_{BXEu} of Ni_{0.7}Zn_{0.3}Fe_{2}O_{4}+wt6% V_{2}O_{5} are given in Table 2.

<table>
<thead>
<tr>
<th>Ni_{0.7}Zn_{0.3}Fe_{2}O_{4} + 0.6wt% V_{2}O_{5}</th>
<th>L_A</th>
<th>L_B</th>
<th>d_{AX}</th>
<th>d_{BX}</th>
<th>d_{AXE}</th>
<th>d_{BXE}</th>
<th>d_{BXEu}</th>
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<tr>
<td></td>
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<td>(')</td>
<td>(')</td>
<td>(')</td>
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<td>(')</td>
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</tbody>
</table>
Structural parameters of Ni₀.₇Zn₀.₃Fe₂O₄+0.₆wt%V₂O₅

The average lattice constants a = 8.379 Å for Ni₀.₇Zn₀.₃Fe₂O₄+0.₆wt%V₂O₅ was calculated using the standard relation $a = \sqrt[3]{h^2 + k^2 + l^2}$; Where $d_{hkl}$ the interplaner spacing of two planes, and $(hkl)$ is the miller indices of each plane. Although, $V^{5+}$ ions have a smaller radius (0.59 Å) than that of the $Fe^{3+}$ ions (0.64 Å) and Ni²⁺ ions (0.69 Å), the incorporation of V₂O₅ into lattice results in the creation of some Fe²⁺ ions which have an ionic radius (0.74 Å) [18]. The crystallite size (t) was calculated by using the Debye-Scherrer method:

$$t = \frac{k\lambda}{\beta \cos \theta}$$

Where, $\lambda$ is the X-ray wavelength, $\beta$ is the full width at half maximum, $\theta$ is the Bragg’s angle, and $k = 0.89$. The X-ray density ($d_\chi$) of Ni₀.₇Zn₀.₃Fe₂O₄+0.₆wt%V₂O₅ was calculated by the relation; $d_\chi = 8M/Na^3$; Where, M is molecular weight and $N_A$ is the Avogadro’s number, $\alpha$ is lattice constant. The X-ray density for Ni₀.₇Zn₀.₃Fe₂O₄+0.₆wt%V₂O₅ was obtained as 5.415 (g/cm³). The values of Lattice parameter $\alpha$, X-ray density $d_\chi$, Bulk density $d_B$, Volume V, and Porosity P%, of Ni₀.₇Zn₀.₃Fe₂O₄+0.₆wt%V₂O₅ are listed in Table 3.

<table>
<thead>
<tr>
<th>Ni₀.₇Zn₀.₃Fe₂O₄+wt6%V₂O₅</th>
<th>$\alpha$(Å)</th>
<th>$d_\chi$(g/cm³)</th>
<th>$d_B$(g/cm³)</th>
<th>V(Å³)</th>
<th>P%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni₀.₇Zn₀.₃Fe₂O₄+wt6%V₂O₅</td>
<td>8.379</td>
<td>5.415</td>
<td>4.376</td>
<td>588.3</td>
<td>19.187</td>
</tr>
</tbody>
</table>

Infrared spectroscopy of Ni₀.₇Zn₀.₃Fe₂O₄+0.₆wt%V₂O₅

IR spectroscopy stands an essential tool to endorse the functional group fix up in the materials and the bands linked within Ni₀.₇Zn₀.₃Fe₂O₄+0.₆wt%V₂O₅ structure. All the intensified peaks in the IR spectra hinted the intrinsic vibrations confirming the formation of the ferrite phase. We support the results out come in the IR spectra of the Ni₀.₇Zn₀.₃Fe₂O₄+0.₆wt%V₂O₅ considering as continuously bonded crystals, based on the studies accomplished by Waldron [19].
**Figure 2.** Infrared spectra of Ni$_{0.7}$Zn$_{0.3}$Fe$_2$O$_4$+wt6%V$_2$O$_5$

The IR absorption bands seen in Figure 2 were accredited to the vibrations of oxide-metal ions (M-O) in the crystal lattice.

**Magnetic Property of Ni$_{0.7}$Zn$_{0.3}$Fe$_2$O$_4$+wt6%V$_2$O$_5$**

The magnetic specifications of Ni$_{0.7}$Zn$_{0.3}$Fe$_2$O$_4$+0.6wt%V$_2$O$_5$ synthesized via ceramic method were resolute using the pulse field hysteresis loop tracer technique on the device MAGNETA, at room temperature engaging the magnetic field of 5000 Oe (Figure 3). The saturation magnetization (Ms) and remanence magnetization (Mr) over the loading of 0.6wt%V$_2$O$_5$ was recorded to be increased in comparison to Ni$_{0.7}$Zn$_{0.3}$Fe$_2$O$_4$ [23] employing the similar synthesis method. The coercivity (Hc) appealed to be reduced drastically from 18.71 Oe to 3.04 Oe as a ramification of wt4%V$_2$O$_5$. Also, increased slight enhancement in Magneton number ($n_B$) was observed as a result of wt4%V$_2$O$_5$ loading.

![M-H curve](image)

**Figure 3.** M-H curve of magnetization for Ni$_{0.7}$Zn$_{0.3}$Fe$_2$O$_4$+wt6%V$_2$O$_5$

**Table 4.** The Saturation magnetization (Ms), Remanence magnetization (Mr), and Coercivity (Hc), Magnetic remanence to saturation ratio (Mr/Ms), and Magneton number for Ni$_{0.7}$Zn$_{0.3}$Fe$_2$O$_4$+wt6%V$_2$O$_5$

<table>
<thead>
<tr>
<th>Ni$<em>{0.7}$Zn$</em>{0.3}$Fe$_2$O$_4$+wt4%V$_2$O$_5$</th>
<th>Ms (emu/gm)</th>
<th>Mr (emu/gm)</th>
<th>Hc (Oe)</th>
<th>$n_B$ (µb)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni$<em>{0.7}$Zn$</em>{0.3}$Fe$_2$O$_4$+wt6%V$_2$O$_5$</td>
<td>72.23</td>
<td>2.19</td>
<td>3.04</td>
<td>2.352</td>
</tr>
<tr>
<td>Ni$<em>{0.7}$Zn$</em>{0.3}$Fe$_2$O$_4$ [23]</td>
<td>74.60</td>
<td>2.26</td>
<td>18.71</td>
<td>2.429</td>
</tr>
</tbody>
</table>

**CONCLUSIONS**

In this article, we have summarized the efficacious preparation of Ni$_{0.7}$Zn$_{0.3}$Fe$_2$O$_4$+wt6%V$_2$O$_5$ by using conventional ceramic method, belongs to the inverse; single-phase; cubic spinel ferrite structure. The gained data from XRD was calculated using the standard formula. The lattice constant of Ni$_{0.7}$Zn$_{0.3}$Fe$_2$O$_4$+wt6%V$_2$O$_5$ was reported to be 8.379 Å which is in a good agreement with the proclaimed literature and found to be consistent over the wt4%V$_2$O$_5$. X-ray density ($d_f$) was obtained as 5.415 g/cm$^3$. The values of $L_A$, $L_B$, $d_{AX}$, $d_{BX}$, $d_{AXE}$, and $d_{BXE}$ derived from the XRD data were found to be consecutive towards the reported range in literature. The IR spectroscopy has affirmed the ferrite phase formation in Ni$_{0.7}$Zn$_{0.3}$Fe$_2$O$_4$+wt6%V$_2$O$_5$. On loading the V$_2$O$_5$, we report an escalation in the saturation...
magnetization, remanence magnetization and magneton number (n_B) of Ni_{0.7}Zn_{0.3}Fe_{2}O_{4}+0.6wt\%V_{2}O_{5}. Whereas, the value of coercivity was drastically decreased implying an alteration in magnetic property.

ACKNOWLEDGMENT

One of the authors Dr. M.S. Patil is thankful to the Savitribai Phule Pune University, for providing the X-ray diffraction facility for the present investigation.

REFERENCE

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