CHAPTER-5

RESULTS DISCUSSION FOR Mg$_x$Fe$_{(1-x)}$O

5.1 XRD Calculations:

To know about the crystal data of prepared samples, XRD study of prepared samples was made. The prepared samples were subjected to copper Ka X-ray of wave length 1.1504 nm. The XRD pattern of Mg$_x$Fe$_{(1-x)}$O consists of well defined and well resolved peaks. The appearance of these peaks in XRD pattern is a configuration of the the poly crystalline and monophonic nature of prepared samples of Mg$_x$Fe$_{(1-x)}$O. The diffraction peaks corresponding to the planes (111), (220), (311), (400), (422), (440), (533), and (440) is an evidence for the spinnel structure of ferrites. Using XRD data full width half maximum, lattice parameter, crystalline size and interplanar distance “d” are determined.
Fig 5.1.1. XRD pattern of 9 samples
Table of Crystalographic Information of Total Samples

Table 5.2.1 gives the information of all crystallographic parameters of all samples prepared by chemical combustion method.

<table>
<thead>
<tr>
<th>Sample</th>
<th>System</th>
<th>structure</th>
<th>Interplanar distance $d$ (A°)</th>
<th>Lattice parameter $a$ (A°)</th>
<th>Crystalline size $t$ (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mg$<em>{0.1}$Fe$</em>{0.9}$</td>
<td>cubic</td>
<td>FCC</td>
<td>29.23</td>
<td>9.33</td>
<td>12.23</td>
</tr>
<tr>
<td>Mg$<em>{0.2}$Fe$</em>{0.8}$</td>
<td>cubic</td>
<td>FCC</td>
<td>29.88</td>
<td>9.14</td>
<td>13.53</td>
</tr>
<tr>
<td>Mg$<em>{0.3}$Fe$</em>{0.7}$</td>
<td>cubic</td>
<td>FCC</td>
<td>31.12</td>
<td>9.89</td>
<td>11.78</td>
</tr>
<tr>
<td>Mg$<em>{0.4}$Fe$</em>{0.6}$</td>
<td>cubic</td>
<td>FCC</td>
<td>29.01</td>
<td>9.34</td>
<td>14.23</td>
</tr>
<tr>
<td>Mg$<em>{0.5}$Fe$</em>{0.5}$</td>
<td>cubic</td>
<td>FCC</td>
<td>30.21</td>
<td>9.11</td>
<td>12.23</td>
</tr>
<tr>
<td>Mg$<em>{0.6}$Fe$</em>{0.4}$</td>
<td>cubic</td>
<td>FCC</td>
<td>32.45</td>
<td>10.02</td>
<td>13.22</td>
</tr>
<tr>
<td>Mg$<em>{0.7}$Fe$</em>{0.3}$</td>
<td>cubic</td>
<td>FCC</td>
<td>30.91</td>
<td>9.08</td>
<td>14.11</td>
</tr>
<tr>
<td>Mg$<em>{0.8}$Fe$</em>{0.2}$</td>
<td>cubic</td>
<td>FCC</td>
<td>3423</td>
<td>10.75</td>
<td>14.29</td>
</tr>
<tr>
<td>Mg$<em>{0.9}$Fe$</em>{0.1}$</td>
<td>cubic</td>
<td>FCC</td>
<td>33.34</td>
<td>10.22</td>
<td>16.23</td>
</tr>
</tbody>
</table>

Table 5.2.1 Table of Crystallographic Information of Total Samples
5.2. SEM image of Mg$_{0.2}$Fe$_{0.8}$O of After Calcination at 300$^\circ$C for 3 hours:

Scanning electron microscope (SEM) shows the morphological state of the sample, the electrons interact with atoms that make up the sample producing composition and other properties.

![SEM image of Mg$_{0.2}$Fe$_{0.8}$O](image)

Fig 5.2 SEM image of Mg$_{0.2}$Fe$_{0.8}$O

SEM image of Mg$_{0.2}$Fe$_{0.8}$O nano particles after calcination at 2.5 magnification. The image shows amorphous nature and cottony lumps of masses. An individual particles of mass was measured which is also in agreement with XRD approximately around 300 nm. The effect of calcination is clearly evident with the mass of lump growing around 20 µm. An individual particle found to have approximately 300-350 nm.
5.3. Energy Dispersive X-ray analysis (EDAX)

Energy dispersive x-ray analysis (EDAX) is an analytical technique. By using this technique we can identify the elements present in the samples and also their weight percentage in the sample. That means we can assess the purity of the synthesized sample. By the chemical combustion of the synthesized sample we can estimate the elements present in the

<table>
<thead>
<tr>
<th>Element</th>
<th>Element %</th>
<th>Atomic %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon</td>
<td>1</td>
<td>2.28</td>
</tr>
<tr>
<td>Oxygen</td>
<td>34.24</td>
<td>58.57</td>
</tr>
<tr>
<td>Magnesium</td>
<td>11.55</td>
<td>13.03</td>
</tr>
<tr>
<td>Iron</td>
<td>53.21</td>
<td>26.12</td>
</tr>
<tr>
<td>Total</td>
<td>100</td>
<td>100</td>
</tr>
</tbody>
</table>

Table 5.3 EDAX analysis of \( \text{Mg}_{0.2}\text{Fe}_{(0.8)}\text{O} \)
Table 5.3 shows the EDAX analysis of Mg$_{0.2}$Fe$_{0.8}$O with inert gas atmosphere after calcination. The synthesized sample of Mg$_{0.2}$Fe$_{0.8}$O contains 1% carbon, 34% of oxygen, 11.55% of magnesium, 53.2% of iron. From this analysis it is clear that Mg$_{0.2}$Fe$_{0.8}$O has 97% purity.

**Procedure for C$_p$ Calculation:**

The specific heat of unknowns can be determined by exploiting the sensitivity of DTA/DSC baseline total heat capacity. The procedure for determining the C$_p$ value of the given sample is described as follows. First an empty sample container versus an empty reference container is run. In the next step, a known mass of standard material is taken in a sample container. Here the standard material is α-alumina and it’s C$_p$ value is 1.089 J/mol-K. Now the container containing standard material is exposed to identical heating rate. In the final step, a known quantity of material whose C$_p$ value is to be determined is taken in the sample container. Now the container is exposed to identical rate of heating. All these traces are plotted on the same graph as shown in fig. 5.4.1.
If the total heat capacities of the empty sample and reference containers were perfectly matched, then the empty sample container versus empty reference container trace should appear as a flat baseline of zero value during the entire scan. This is rarely the case (due to asymmetries in instrument construction) and is actually not necessary for this calculation. The temperature deviation from this base line (empty panels line) for other traces is a result of the extra thermal mass on the sample side, causing it to lag the reference in temperature during the heating ramp. Defining $C_p$ as the specific heat ($J/g/K$), the sample specific heat is determined by the ratio;

$$\frac{\Delta T_{\text{unknown}}}{C_p \text{unknown} M_{\text{unknown}}} = \frac{\Delta T_{\text{standard}}}{C_p \text{standard} M_{\text{standard}}}$$

Where $M$ is mass as determined from an analytical balance, and $\Delta T$s are measured at the same temperature. For reproducibility, the standard and reference granules should be of the same size and the sample container should be of the same size and the sample container should be placed in same position in the DTA cell.

At 300°C

$\Delta T_{\text{unknown}} = 11.76382 - 9.99126 = 1.77256 \text{uV}$

$\Delta T_{\text{standard}} = 21.7464 - 9.99126 = 11.75514 \text{uV}$

$M_{\text{unknown}} = 7 \text{mg}$
\[M_{\text{standard}} = 21.34\text{mg}\]

\[C_{p, \text{standard}} = 1.083 \text{J/g/k}\]

\[C_p \text{ of } \text{Mg}_{0.4}\text{Fe}_{0.6} = ?\]

Relation \(\frac{(\Delta T_{\text{unknown}})}{(M_{\text{unknown}} \cdot C_{p, \text{unknown}})} = \frac{(\Delta T_{\text{standard}})}{(M_{\text{standard}} \cdot C_{p, \text{standard}})}\)

\[C_p \text{ of } \text{Mg}_{0.4}\text{Fe}_{0.6} = 0.4978 \text{J/g/k}\]

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**Fig 5.4.1** Procedure for Calculation of Specific Heat Capacity (C).

**Specific Heat of 9 Samples;**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Specific heat in J/g/K</th>
</tr>
</thead>
</table>

4.4. FTIR Analysis:

**FTIR Analysis of Mg$_{0.2}$Fe$_{0.8}$O:**

The Fourier transform infrared spectroscopy is a technique, which can be used for identification of organic materials in the synthesized sample. FTIR is a non-destructive analytical method. FTIR is mainly a study of absorption spectra of the sample. By studying this absorption spectra, we can obtain information regarding the molecular structure of the material and chemical bonds in the material.

The FTIR spectrum of the sample Mg$_{0.2}$Fe$_{0.8}$O, This spectra displays broad absorption around the intensive band at 3433.3 cm$^{-1}$ was assigned as a OH stretching, H-O-H bonding at 1631.14 cm$^{-1}$ and the typical metal oxygen absorption band for spinal structure of ferrite at 597.10 cm$^{-1}$. This bond intrinsic stretching vibrations of the metal (mg-fe) at tetrahedral site.
FTIR Analysis of Mg$_{0.4}$ Fe$_{(0.6)}$O

The FTIR spectrum of the sample Mg$_{0.4}$Fe$_{(0.6)}$O, This spectra displays broad absorption around the intensive band 3428.68 cm$^{-1}$ cm$^{-1}$ was assigned as a OH stretching, H-O-H bonding at 1632.22 cm$^{-1}$ and the typical metal –oxygen absorption band for spinal structure of ferrite at 600 cm$^{-1}$. This bond intrinsic stretching vibrations of the metal(Mg-Fe) at tetrahedral sites.
FTIR Analysis of Mg\textsubscript{0.6}Fe\textsubscript{(0.4)}O

The FTIR spectrum of the sample Mg\textsubscript{0.6}Fe\textsubscript{(0.4)}O, This spectra displays broad absorption around the intensive band 3400 cm\textsuperscript{-1} cm\textsuperscript{-1} was assigned as a OH stretching, H-O-H bonding at 1634.55 cm\textsuperscript{-1} and the typical metal –oxygen absorption band for spinal structure of ferrite at 598.84 cm\textsuperscript{-1}. This bond intrinsic stretching vibrations of the metal (Mg-Fe) at tetrahedral sites.
FTIR Analysis of Mg$_{0.9}$Fe$_{(0.1)}$O

The FTIR spectrum of the sample Mg$_{0.9}$Fe$_{(0.1)}$O, this spectra displays broad absorption around the insensitive band 3434.3 cm$^{-1}$ was assigned as a OH stretching, H-O-H bonding at 1635.31 cm$^{-1}$ and the typical metal–oxygen absorption band for spinal structure of ferrite at 590 cm$^{-1}$. This bond intrinsic stretching vibrations of the metal(Mg-Fe) at tetrahedral sites.
5.5 Vibration sample magnetometer:

VSM of Mg$_{0.4}$Fe$_{(0.6)}$O

Nano ferrites are characterized by using vibrating sample magnetometer and there by magnetic properties of nano ferrites can be understood. Fig 5.6.1 shows the Hysteresis loop of Mg$_{0.4}$Fe$_{(0.6)}$O at room temperature. Hysteresis loop explains super paramagnetic nature. From VSM data the saturation magnetization, coercivity and retentivity of Mg$_{0.4}$Fe$_{(0.6)}$O nano particles are obtained and they are 3.272 emu/gm, 63.431 Oe/gm, 1.48 emu/gm respectively.

![Hysteresis loop graph](image)

5.6.1. VSM of Mg$_{0.4}$Fe$_{(0.6)}$O
4.5.2 VSM of Mg$_{0.6}$Fe$_{(0.4)}$O

The Hysteresis loop traced at room temperature for Mg$_{0.6}$Fe$_{(0.4)}$O is. The hysteresis loop explains the super paramagnetic nature. The values of the saturation magnetization (MS), coercivity (HC) and retentivity (MR) are obtained. The experimental magnetic moment (mB) is determined from the saturation magnetization (MS) data. The values of magnetic parameters such as $M_S$, $H_C$ and $M_r$ of nanoparticles of Mg$_{0.6}$Fe$_{(0.4)}$O obtained from the VSM data are 4.321 emu/g, 97.506 oe and 3.748 emu/gm, respectively.

Fig. 5.6.2 VSM of Mg$_{0.6}$Fe$_{(0.4)}$O
Magnetic NPs and MPs have been used in different types of biosensors based on different physical principles. Some achieve high sensitivity and, with rapid advances in instrumentation, maybe useful as point-of-care sensors. The continued rapid development of sensors using magnetic materials seems assured.

![Figure 5.6.4](image)

**Figure 5.6.4 Effect of the No. of Ferro magnets**

Figure 5.6.4 indicates that the intensity of magnetic field and the magnetic response of Fe₃O₄ magnetic nanoparticles weaken as the distance increases, moreover, the magnetic response depends on the magnetic intensity. The magnetic response increases nearly 64 times when the distance is changed from 3 cm to 1 cm. Thus, in the clinical application application the distance between ferromagnet and drug should be highlighted especially.
Fig. 5.6.5 illustrates the magnetic response of Fe₃O₄ magnetic nanoparticles increases rapidly as the increase of the number of the ferromagnets initially, but not obviously when the number of the ferromagnets exceeds four. Therefore, from the economic point of view, a 32.6 mT magnetic field which is produced by four ferromagnets was sufficient to excite the dipole moments of the 0.05 g Fe₃O₄ powder 2 cm away from the ferromagnets. Ferromagnet and drug should be highlighted especially.