Synthesis And Some Physical Properties Of Magnetite ($\text{Fe}_3\text{O}_4$) NPs

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Abstract—In this study , ($\text{Fe}_3\text{O}_4$) thin film which prepared by chemical method and deposited by drop casting technique on glass. The structural ,optical and chemical analysis have been investigated . X-ray diffraction (XRD) measurements relieve that the ($\text{Fe}_3\text{O}_4$) thin film was single crystalline, cubic structure and there is no trace of the other material. UV-Vis measurements reveal that the energy gap of ($\text{Fe}_3\text{O}_4$) thin film was found 2.2 eV. The Fourier Transform Infrared Spectroscopy (FTIR) spectrum of ($\text{Fe}_3\text{O}_4$) thin film shows the O–H in plane and out of plane bonds appears at (1583.45–1481.23 and 935.41–838.98)cm$^{-1}$ and absorption band Fe–O bonds in the crystalline lattice of $\text{Fe}_3\text{O}_4$.

Keywords—Thin film; XRD; $\text{Fe}_3\text{O}_4$; energy gap; drop casting.

1. Introduction

Many authors prepared Magnetite and study its different properties, but still Magnetite inspires researcher's new researachable ideas, this due to its characteristics, which has a great significance in various fields, especially, when this material in a nanosize. For example, Magnetite ($\text{Fe}_3\text{O}_4$) nanoparticles have attracted much interest not only in the field of magnetic recording media such as audio, videotape, high-density digital recording disks, magnetic fluids and data storage, but also in the areas of medical care such as drug delivery systems (DDS), medical applications, including radio-frequency hyperthe-mia, photomagnetics, magnetic resonance imaging (MRI), medical diagnostics, cancer therapy, microwave devices, magneto-optics devices, sensors, high frequency applications, catalysis and magnetic sensing [1-11].

2. Experimental

In a typical procedure, 1.5g of $\text{Fe(NO}_3\text{)}_2$ , 179.8548 g/mol Molar mass (BDH Chemicals Ltd Pool England) was dissolved in 50ml of PVP (Sigma Aldrich USA) 1WT. % and Re-distilled water was used throughout the experiment. The solut-ion was added into a round-bottom flask with stirring. The color of the mixture was dark yellow . About 15ml of NaOH (1M) was rapidly added to the mixture, and a nanopowder suspension was formed as shown in figure 1 . The suspension was kept at 75 °C for 1 h. After cooling to room temperature, the particles were separated by centrifugation and were washed with distilled water to remove any contaminations.

Figure 2 shows that $\text{Fe}_3\text{O}_4$ colloidal nanoparticles which are prepared by chemical method and deposited by drop casting technique on glass substrate. It has been taken from the solution by pipette and then drop on glass surface only 5 drops, the particles were then dried by using heater at 80 °C , then the film is ready.

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**Fig.1:** $\text{Fe}_3\text{O}_4$ freshly colloidal nanoparticles(left) which are prepared by chemical method and the solution(right).

**Fig.2:** Schematic diagram drop casting method experimental set up.

X-ray diffractometer (XRD-6000, Shimadzu) was used to investigate the structure and crystalinity of
nano-particles. The absorption of the colloidal nanoparticles solution was measured by using UV–Vis double beam spectro-photometer (CECIL, C. 7200, France).

3. Results and discussion
The precipitated fine particles were characterized by XRD for structural determination and estimation of crystallite size. XRD can be used to characterize the crystallinity of nano-particles, as well as the average nanoparticles diameter. The lattice constant (a) was found to be 8.310 Å, which was compared with the lattice parameter for the magnetite of 8.39. Finally, the analysis of the diffraction pattern showed the formation in the sample of a cubic phase structure, due to the strongest reflection that proceeds from the(220) plane (Fig.3). The peak indexed as plane (220) corresponded to a cubic unit cell, characteristic of a cubic structure. Therefore, it was confirmed that the crystalline structure of obtained mag-netite nanoparticles, agreed with the structure of an inverse phase type oxide. Crystallite size measurements were determined from the full-width at half maximum (FWHM) of the strongest reflection of the (220) peak, using the Scherrer approximation, which assumes the small crystallite size to be the cause of line broadening (Equation 1):

\[ G_s = \frac{A \lambda}{\beta \cos \theta} \]  

(1)

Here, \( G_s \) is the crystallite mean size, \( A \) is a shape function for which a value of 0.9 is used, \( \lambda \) is the wavelength of the radiation, \( \beta \) the full width at half maximum (FWHM) in radians in the 2\( \theta \) scale, and \( \theta \) the Bragg angle. The crystal size calculated were (56.61 and 111.22)nm. Due to the broad diffraction pattern lines, it can be said that particles have average size about of nanometers scale ; therefore, also, it can be seen that, the site and intensity of the diffraction peak are consistent with the standard pattern for JCPDS Card No. (79 - 0417) Magnetite - synthetic. The sample show broad peak, indicating the ultra-fine nature and small crystallite size of the particles.

The microstrain value ‘\( \varepsilon \)’ and the dislocation density ‘\( \sigma \)’ was evaluated by using the following relations [13, 14]:

\[ \varepsilon = \frac{\beta \cos \theta}{4} \]  

(2)

\[ \sigma = \frac{1}{G_s^2} \]  

(3)

The strain and dislocation density of Fe\(_3\)O\(_4\) nanoparticles films chemical reaction were around 5.85 and 3.39 \( 10^{14} \) lines/m\(^2 \) respectively.

The transmittance characteristics can be a valuable tool for analyzing nanostructure.

**Table 1: powder X-ray diffraction data of (Fe\(_3\)O\(_4\)) thin film**

<table>
<thead>
<tr>
<th>2 Theta (deg)</th>
<th>( \beta ) (deg)</th>
<th>( G_s ) (nm)</th>
<th>( \sigma ) lines/m(^2 ) ( 10^{14} )</th>
<th>( \varepsilon ) ( 10^{-4} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>29.44</td>
<td>0.138</td>
<td>56.610</td>
<td>3.12</td>
<td>5.85</td>
</tr>
<tr>
<td>29.60</td>
<td>0.080</td>
<td>111.26</td>
<td>0.80</td>
<td>3.39</td>
</tr>
</tbody>
</table>

Figure 4 shows the transmittance spectrum of (Fe\(_3\)O\(_4\)) thin film. The data are corrected for glass transmission in UV region. The transmittance is sharply increasing above ~ 600 nm due to a wide distribution of particlesize.
Fig.4: Transmittance spectrum of (Fe$_3$O$_4$) thin film which prepared by chemical method and deposited by drop casting technique on glass.

Therefore using the fundamental relation of photon transmission and absorbance, the absorbance (A) is defined as the logarithm (base 10) of the reciprocal of the transmittance:

$$A = \log_{10}\frac{1}{T} \quad (4)$$

If T is the transmittance and A is the absorbance of the (Fe$_3$O$_4$) thin film which prepared by chemical method and deposited by drop casting technique on glass. The reflection of the film has been found by using relationship:

$$R + T + A = 1 \quad (5)$$

The reflection of the (Fe$_3$O$_4$) thin film increases with increasing the wavelength above 600 nm after that there is about 0.2 as shown in figure 5 due to stability in transmittance.

![Transmittance spectrum](image1)

![Reflection spectrum](image2)

Fig.5: Reflection spectrum of (Fe$_3$O$_4$) thin film which prepared by chemical method and deposited by drop casting technique on glass.

From the reflection R of the thin film, the refraction index can be calculated from the following relationship:

$$n = \frac{1 + \sqrt{R}}{1 - \sqrt{R}} \quad (6)$$

Figure 6 shows the refraction index of the (Fe$_3$O$_4$) thin film which prepared by chemical method and deposited by drop casting technique on glass which is similar to the behavior of the refraction spectra and the maximum value is 1.5.

![Refractive index spectrum](image3)

![Reflectance index spectrum](image4)

Fig.6: Reflectance index spectrum of (Fe$_3$O$_4$) thin film which prepared by chemical method and deposited by drop casting technique on glass.

The optical absorption coefficient $\alpha$ was evaluated by the Tauc relation $\alpha h v = A (h v - E_g)^n$ where $A = 2.303 \frac{A}{t}$ where $t$ is the film thickness, $h v$ is the photon energy, $E_g = \frac{1240}{\lambda(nm)}$ and $n = 0.5$ for allowed direct transition.

Plotting the graph between $(\alpha h v)^2$ versus photon energy $(h v)$ gives the value of the band gap. The extrapolation of the straight line to $(\alpha h v)^2 = 0$, gives the value of the band gap, shown in figure 7. The optical band gap is 2.2 eV, in other word, the excitons wavelength ~ 516 nm. This result is very important to relieve that the (Fe$_3$O$_4$) thin film can be use in solar cell device.

![Optical conductance](image5)

Fig.7: $(\alpha h v)^2$ versus photon energy plot of (Fe$_3$O$_4$) thin film which prepared by chemical method and deposited by drop casting technique on glass.

The optical conductance is obtained using the relation,

$$\sigma = a n c \varepsilon_o \varepsilon = \frac{a n c}{4 \pi} \quad (7)$$

Where $\sigma$ is the optical conductance, $c$ is the velocity of the radiation in the space, $n$ is the refractive index and $\alpha$ is the absorption coefficient.

Figure 8 shows the relation between the optical conductance and photon energy for (Fe$_3$O$_4$) thin film. Figure 8 shows the optical conductance increases at 2.2 eV to 2.6 eV then it is constant over that.
In particular, this occurs with certain types of chemical substances adsorbed on the surface of nanoparticles [15,16].

4. Conclusions

The synthesized (Fe$_3$O$_4$) thin film by chemical method had minimum nanosized is around 56.61 nm and the optical properties revealed that the energy gap of (Fe$_3$O$_4$) thin film indicated to the effect of quantum size. X-ray diffraction (XRD) exhibits spectrum that the (Fe$_3$O$_4$) are monocristalline by splitting the orientation (220) plane in tow peaks. FTIR measurements shows that the absorption bond Fe–O bonds in the crystalline lattice of Fe$_3$O$_4$.

References