

Synthesis and Specific Absorption Rate of Fe₃O₄ Nanoparticles

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Abstract: The sample of Fe₃O₄ magnetic nanoparticles (MNPs) was synthesized via co-precipitation method from ferrous and ferric solutions. X-ray diffraction (XRD) used to characterize the powder synthesized of Fe₃O₄. The average lattice parameter and the average size of Fe₃O₄ MNPs were $a = 8.3 \text{ \AA}$ and $t = 9.8 \text{ nm}$. Induction heater operated at low frequencies 100 kHz was used to study the heating rate ($\Delta T/\Delta t \text{ } ^\circ\text{C}/\text{sec}$) and the specific absorption rate (SARW. g^{-1}) of the Fe₃O₄ MNPs and were found to be $0.025 \text{ } ^\circ\text{C}/\text{sec}$ and 105 W. g^{-1} respectively.

Keywords: Magnetic nanoparticles, hyperthermia

1. Introduction

The capability of magnetic nanoparticles (MNPs) to act as effective heating agents for magnetic hyperthermia (MH) was demonstrated many years ago [1].

Fe₃O₄ MNPs have attracted much interest in the field of magnetic recording media such as audio and videotape, and high-density digital recording disks, data storage, magnetic fluids. Fe₃O₄ MNPs also used in the areas of medical care such as drug delivery systems (DDS), medical applications including radiofrequency hyperthermia (RFH), photo magnetic (PM), and magnetic resonance imaging (MRI), medical diagnostics, cancer therapy, microwave devices, magneto-optics devices, sensors, high and low frequency applications. Several studies have shown the Fe₃O₄ MNPs energy absorption in alternative current magnetic field (ACMF) [2].

2. Experimental details

2.1 Sample preparation

Fe₃O₄ prepared by co-precipitating aqueous solutions of (NH₄)₂ Fe(SO₄)₂ and FeCl₃ mixtures, respectively, in alkaline medium ($\text{Fe}^{+2}:\text{Fe}^{+3} = 1:2$). The mixture is kept at 80 °C. This mixture is added to the boiling solution of NaOH (0.5 mol. is dissolved in 600 ml of distilled water) within 10 second under constant stirring. Magnetite is formed by conversion of metal salts into hydroxides, which take place immediately, and transformation of hydroxides into ferrites. The solution is maintained at 100 °C for 1.5 h. The Fe₃O₄ particles are washed several times by distilled water, the Fe₃O₄ particles are just dried at 100 °C for 1h [2,3].

2.2 X-ray diffraction

The Shimadzu X-ray Diffractometer (XRD-6000) in Department of Physics, King Fahd University of Petroleum and Minerals, Kingdom of Saudi Arabia was used in this study. The XRD-6000, an X-ray diffractometer analyzed crystalline states under normal atmospheric conditions. This method is nondestructive. X-rays focused on a sample fixed on the axis of the spectrometer (goniometer) were diffracted by the sample. The changes in the diffracted X-ray intensities measured, recorded and plotted against the rotation angles of the sample. The result is referred to as the XRD pattern of the sample. Computer analysis of the peak positions and intensities associated with this pattern enables qualitative analysis, lattice parameter determination and/or stress determination of the sample. Qualitative analysis may be conducted on the basis of peak height or peak area. The peak angles and profiles may be used to determine particle diameters and degree of crystallization, and are useful in conducting precise X-ray structural analysis. The XRD-6000 will accept various types of X-ray tubes, our sample of Fe₃O₄ analyzed by the standard Cu X-ray tube.

2.3 Induction heater

An induction heater operated at low frequencies and low powers (100 kHz and 100 W), was used to study the heating rate ($\Delta T/\Delta t \text{ } ^\circ\text{C}/\text{sec}$) and the specific absorption rate (SAR W. g^{-1}) of the Fe₃O₄ MNPs was prepared previously.

3. Result and Discussion

3.1 Sample characterizations

The sample of Fe₃O₄ prepared, as described in the previous was characterized by X-ray diffraction (XRD) in the 2θ range from 10 to 80 degrees. The obtained result is shown in fig.1.

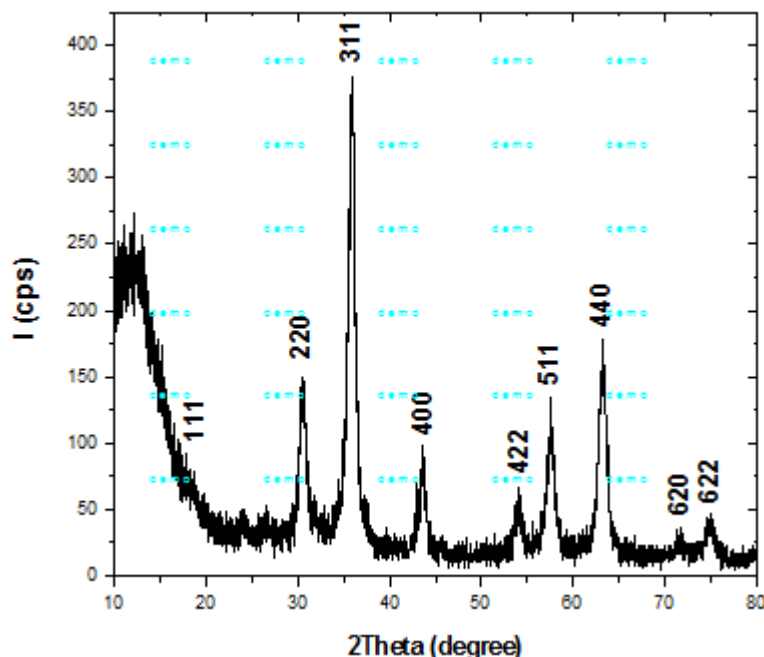


Figure 1: XRD for Fe₃O₄

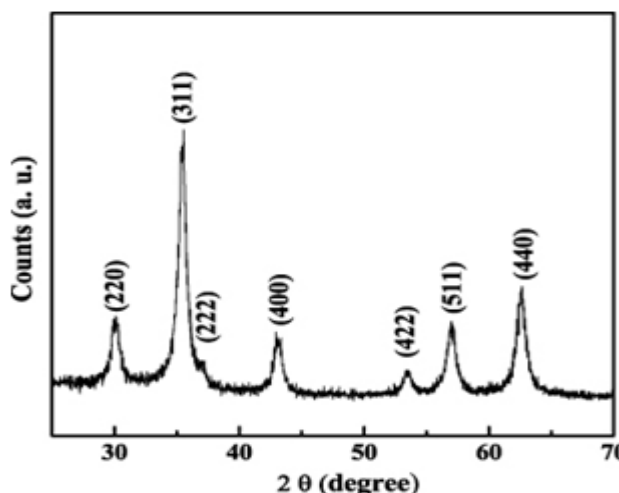


Figure 2: XRD for Fe₃O₄ [4]

Table 1: The parameters of the three strongest peaks

| No | 2 theta (deg.) | d (Å) | FWHM (deg.) | t nm | hkl | a(Å) |
|----|----------------|---------|-------------|------|-------|-------|
| 1 | 35.8583 | 2.50227 | 0.85940 | 9.80 | 2 1 1 | 8.299 |
| 2 | 63.1667 | 1.47077 | 0.92000 | 10.2 | 4 4 0 | 8.320 |
| 3 | 30.4993 | 2.92862 | 0.89470 | 9.30 | 2 2 0 | 8.283 |

To determine the prepared sample of Fe₃O₄ size "t", the interplanar distance of the crystal " d_{hkl} " and the lattice parameter "a" from the XRD data, the peaks positions and the full width at half maximum of the three strongest peaks, shown in table 1, are substituted in the Scherrer's formula (1) and Bragg's equation (2) are given by

$$t = \frac{K\lambda}{B \cos \theta} \quad (1)$$

$$d_{hkl} = \frac{\lambda}{2 \sin \theta} \quad , \quad d_{hkl} = \frac{a}{\sqrt{(h^2 + k^2 + l^2)}} \quad (2)$$

Where; t is the thickness of the crystallite, a is the lattice parameter, hkl are Millar's indices, d_{hkl} is the interplanar distance of the crystal, $K \sim 0.9$ is constant depend on the crystallite shape, $\lambda = 1.456 \text{ \AA}$ is x-ray wavelength of the Cu target, B is full width at half maximum (FWHM) and θ_B is Bragg's angle [2, 5, 6].

The XRD patterns indexing of the prepared Fe₃O₄ showed that this sample is cubic with average lattice parameter is 8.3 Å and the average size is 9.8 nm. The crystal structure, the size and the lattice parameter in good agreement with published data fig.2. Such a small size indicates that the Fe₃O₄ sample is the magnetic nanoparticles MNPs [4].

3.2 The SAR of Fe₃O₄ MNPs suspension

To determine the heating rate $\Delta T/\Delta t$ and specific absorption rate SAR value of the Fe₃O₄ MNPs, a suspension of 1ml de-ionizing (DI) water + 1mg Fe₃O₄ MNPs was prepared.

First, 1 ml of DI water was exposed to the magnetic induction and no change on the temperature is noted. Then, 1 mg of Fe₃O₄ MNPs was added to 1ml DI water and exposed to the same magnetic induction; the temperature is seen to increase with the time. After 35 minutes the temperature reached 46°C and remained constant up to 60 minutes exposure to the magnetic induction heating. The temperature no longer changed over time when it reached to a certain value because the heat generated by the absorbed electromagnetic energy of Fe₃O₄ and the heat released towards the environment were equal that is shown in fig.3 [7, 8].

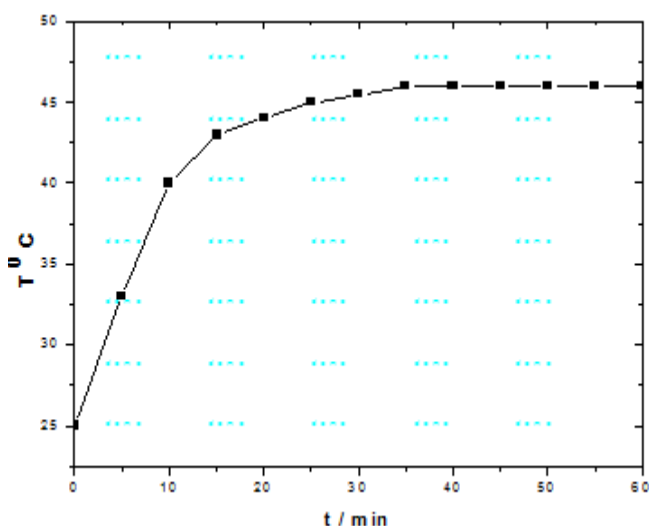


Figure 3: Fe₃O₄ heating rate

The SAR value can be calculated by the following equation

$$SAR = C \frac{\Delta T}{\Delta t} \frac{1}{m_{ferrite}} \quad (3)$$

Where; $C = 4.185 \text{ J g}^{-1} \text{ K}^{-1}$ is the sample specific heat capacity which is calculated as a mass weighed mean value of magnetite and water. $\Delta T/\Delta t = 0.025 \text{ }^\circ\text{C}/\text{sec}$ is the initial slope of the time dependent temperature curve, $m_{ferrite} = 1 \text{ mg}$ is the ferrite content per mg of the sample tube [8].

There are as good as the linear relations in the first rising of the temperature fig.3. We used the linear relation in 0 – 10 minutes intervals for calculating the SAR values of the samples Fe₃O₄ MNPs by equation (3). We find that the SAR value equal 105 W. g^{-1} .

4. Conclusions

The average size and the lattice parameter of the Fe₃O₄ magnetic nanoparticles sample MNPs was prepared by co

precipitation method were estimated to be 9.8 nm and 8.3 Å respectively.

The maximum temperature was 46°C and the needed time to reach this temperature was 35 min with the concentration of 1.0 mg Fe₃O₄/ 1ml of deionizing water. The temperature no longer changed over time when it reached to a certain value. The specific absorption rate SAR and the heating rate $\Delta T/\Delta t$ values of the Fe₃O₄ MNPs were founded to be 105 W. g^{-1} and $0.025^\circ\text{C}/\text{sec}$, these values indicating to use this sample in the magnetic hyperthermia treatment MHT.

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