

“Magnetic Properties and Induction Heating Ability Studies of Spinal Ferrite Nanoparticles for Hyperthermia Treatment of Tumors”

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IN this research synthesizes nanocrystalline CuFe_2O_4 spinel structure using co-precipitation method. X-ray diffraction (XRD), transmission electron microscopy (TEM), and vibrating sample magnetometer (VSM) are utilized in order to study crystalline size, lattice parameters, microstructure and optical and magnetic properties of the formed nanopowders. Hyperthermia is one of the most promising approaches in cancer therapy. The most commonly used heating method in the clinical setting is capacitive heating that uses a radiofrequency (RF) electric field. The induction heating behavior and heating properties of the CuFe_2O_4 nanoparticles in an alternating magnetic field at 150-300 kHz were estimated. The result making Cu ferrite appropriate for hyperthermia treatment of cancer. These results will help us to optimize the conditions for tumor treatment by magnetic nanoparticles.

Keywords: CuFe_2O_4 ; Spinel Ferrites; Optical properties; Magnetic Properties, Heat Induction.

Introduction

Spinel of the type $\text{M}^{2+}\text{M}_2^{3+}\text{O}_4$ attract a great deal of interest for their diverse practical applications [1]. In the case of $\text{M}^{3+} = \text{Fe}$, the resulting spinel ferrite having a general chemical composition MFe_2O_4 ($\text{M} = \text{Co, Ni, Mn, Fe, Cu, Zn, etc.}$), and they have a face-centered cubic (FCC) close packing structure [2]. Spinel ferrites are among the most widely used magnetic materials and exhibit interesting magnetic, magneto-resistive and magneto-optical properties. The development of uniform spinel ferrite nanocrystals has been intensively pursued in the applications of magnetic fluids [3], catalysis [4,5], biotechnology/biomedicine [6,7], magnetic resonance imaging [8,9], data storage [10,11] and microwave absorption [12]. There are several methods for synthesizing nanosized spinel ferrite particles such as co-precipitation [13], hydrothermal method [14,15], sol-gel [16,17], combustion method [17,18], precursor method [19], micro-emulsion method [20], ball milling method [21] and mechano-chemical method [22]. Many reports proved that the property of spinel ferrite can be improved by doping metal elements in MFe_2O_4 owing to their enhanced crystal anisotropy [23]. The substitution of various cations such as magnetic and nonmagnetic at different sub-lattices in ferrite materials renders different kinds of magnetic

and electrical properties [24-26]. Several cations such as Zn^{2+} , Cu^{2+} , Ti^{4+} , Co^{2+} and Y^{3+} have been attempted by many researchers in order to improve the electrical and magnetic properties of spinel ferrites [27-30]. The morphology and size of the synthesized ferrite can be tailored by different preparation methods.

Experimental

Materials and Preparation

The synthesis procedure is as follows: A mixture of iron (III) nitrate nonahydrate, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and copper chloride hexahydrate with the $\text{Fe}^{3+}:\text{Cu}^{2+}$ molar ratios 2:1 was dissolved in deionized water. The produced solution was treated with 5 M NaOH to form a precipitate at pH 12. The produced slurry was filtered, washed and dried at 100 °C. The formed precursor powders were annealing temperature at 1000 °C for 2 hrs in static air atmosphere.

Characterization

The crystalline phases present in the different annealed samples were identified by X-ray diffraction (XRD) on a Bruker axis D8 diffractometer using Cu-K_α ($\lambda = 1.5406$) radiation and secondary monochromator in the range 2θ from 10° to 80° operating at 40 kV and 30 mA at a rate

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DOI : 110.21608/ejbbe.2019.5193.1022

of $2^\circ/\text{min}$. The crystallite sizes of the produced mayenite for the most intense peak (3 1 1) plane determined from the X-ray diffraction data using the Debye-Scherrer formula [31]:

$$d_{RX} = k\lambda/\beta\cos\theta \quad (1)$$

where d_{RX} is the crystallite size, $k = 0.9$ is a correction factor to account for particle shapes, β is the full width at half maximum (FWHM) of the most intense diffraction peak (4 2 0) plane, λ is the wavelength of Cu target = 1.5406 Å, and θ is the Bragg angle. Fourier transform infrared spectroscopy (FTIR) by using a FTIR Thermo Electron Magna 760. The micrographs of produced samples were examined by direct observation via field emission scanning electron microscope (FE-SEM) model JEOL instrument (Japan) model JSM-7800F. The UV-Vis absorption and diffuse reflectance spectrum were recorded at room temperature using UV-VIS-NIR spectrophotometer (Jasco-V-570 spectrophotometer, Japan) fitted with integrating sphere reflectance unit (ISN) in the wavelength range 200 – 2000 nm. The magnetic properties of the powders were measured at room temperature using a vibrating sample magnetometer (VSM, 7410 Lakeshore, USA) in a maximum applied field of 20 KOe. From the obtained hysteresis loops, the saturation magnetization (M_s), remanence magnetization (M_r) and the coercive field (H_c) were determined.

Hyperthermia system

hyperthermia system consist of zero voltage switching [ZVS] induction high frequency heating board, an induction coil with [10 turns] of inner diameter [2 cm], and cut around hollow cylinder inner side, digital DC power supply and oscilloscope. ZVS converter DC current to AMF and insulating sample holder placed within a solenoid induction copper coil. The induction solenoid coils produce a uniform magnetic field inside volume within the coil. The coil formed from cylindrical sections of copper plate with other design elements that enhance performance over a comparable solenoid.

We adjust the voltage of DC power supply to get the desired frequency (195 kHz) for inducing hyperthermia. To avoid heat lost by convection we coated the tube by polyurethane foam (PUR) as a thermal insulator.

Magnetic heating induction study

Four different concentrations (20mg/ml, 40 mg/ml, 60 mg/ml and 80mg/ml) of the CuFe₂O₄ under investigation were suspended in 1 ml of *Egypt. J. Biophys. Biomed. Engng. Vol. , 19 (2018)*

double distilled water. The vials were ultra-sonicated in water bath sonicator for 20 min at 40 °C, and then the suspension was placed in the coil center without touching the walls for 10 min of the sample with desired magnetic field (H) 9.5 Oe. The temperature increasing recorded for the different concentrations and graphs it to show the behavior of different concentration in inducing hyperthermia.

Result and Dissection

X-ray diffraction analysis

The structure and phase purity of as prepared products were confirmed by analyzing the observed powder X-ray diffraction (XRD) patterns. The observed powder XRD pattern of the CuFe₂O₄ nanoparticles was shown in Fig. 1. All the observed reflections of the Cu ferrite (CuFe₂O₄) samples could be assigned to cubic spinel lattice indicating their single phase nature and the planes in the diffraction patterns confirm the formation of pure cubic spinel ferrite structure of CuFe₂O₄ phase (JCPDS # 08-0234) for all samples without any impurity or secondary phase [56]. The average crystallite sizes 't' the lattice constant 'a' and theoretical density 'd_x' of the samples are listed in Table 1. It is clear that Table 1 the crystallite size and lattice parameter, for all samples, seems to be independent of the type of the substituted Mn²⁺ ions. Crystallite size for each sample was calculated using the Scherrer formula which is considering the most intense peak (3 1 1).

The broadened diffraction peaks can be attributed to the nanocrystallite size of as prepared product. Unit cell parameters i.e. lattice parameter (a) was calculated by indexing the observed reflections of the XRD patterns by applying the following equation [32]:

$$a = d\sqrt{h^2 + k^2 + l^2} \quad (2)$$

The X-ray density or theoretical density was estimated by using the relation [33]:

$$d_x = \frac{\sum A}{N_x V} \quad (3)$$

where A is sum of the atomic weights of all the atoms in the unit cell, V is volume of the unit cell and N is the Avogadro's number. Since each primitive unit cell of the spinel structure contains eight molecules, the theoretical density, d_x was determined according to the following relation [34]:

$$dx = \frac{8M}{Nn^3} \tag{4}$$

Where M is molecular weight of the sample, N the Avogadro’s number, a lattice parameter, 8 represents the number of molecules per unit cell and a^3 is the volume of the cubic unit cell.

The mean ionic radii of the A-and B-sublattices (R_A and R_B) can be calculated for all samples using the cation distribution and the relations [35]:

$$R_A = (0.5 - x)r_{Zn^{2+}} + xr_{Co^{2+}} + 0.5r_{Fe^{3+}} \tag{5}$$

$$R_B = 0.5r_{Ni^{2+}} + zr_{Y^{3+}} + (0.5-z)r_{Fe^{3+}} \tag{6}$$

where r denotes to the ionic radius. The calculated values are given in **Table 2**. It is obvious that the trend of R_A and R_B decrease with x , which may be due to the substitution process. The oxygen positional parameter u can be determined using the relation [35]:

$$R_A = a\sqrt{3}(u - 0.25) - r_o \tag{7}$$

where r_o is the O^{2-} radius. The obtained value of u was ~ 0.386 for all samples. The tetrahedral bond length d_{AL} ($d_{A-O^{2-}}$) and the octahedral bond length d_{BL} ($d_{B-O^{2-}}$) can be calculated by the relations [35]:

$$d_{AL} = a\sqrt{3}(u - 0.25) \tag{8}$$

$$d_{BL} = a\left(3u^2 - \frac{11}{4}u + \frac{43}{64}\right)^{1/2} \tag{9}$$

The tetrahedral edge d_{AE} and the octahedral edge d_{BE} and unshared edge d_{BEU} can be determined using the relations [35]:

$$d_{AE} = a\sqrt{2}(2u - 0.5) \tag{10}$$

$$d_{BE} = a\sqrt{2(1 - 2u)} \tag{11}$$

$$d_{BL} = a\left(4u^2 - 3u + \frac{11}{16}\right)^{1/2} \tag{12}$$

The distance between the magnetic ions Co^{3+} and Fe^{3+} (hopping lengths) can be calculated by the relations $L_A = a_t(3/4)^{1/2}$ and $L_B = a_t(2/4)^{1/2}$ for A- and B- sublattices [35]. The determined values are listed in **Table 2**. Table 2 illustrates that the trends of R_A , R_B , d_{AL} , d_{BL} , d_{AE} , d_{BE} , d_{BEU} , L_A and L_B reflect. This is assigned to the substitution process and cation distribution. The values of u are higher than the standard values (0.375), which may point to a trigonal distortion of the B-site coordination. It may be due to the oxygen dissociation through the samples during the preparation.

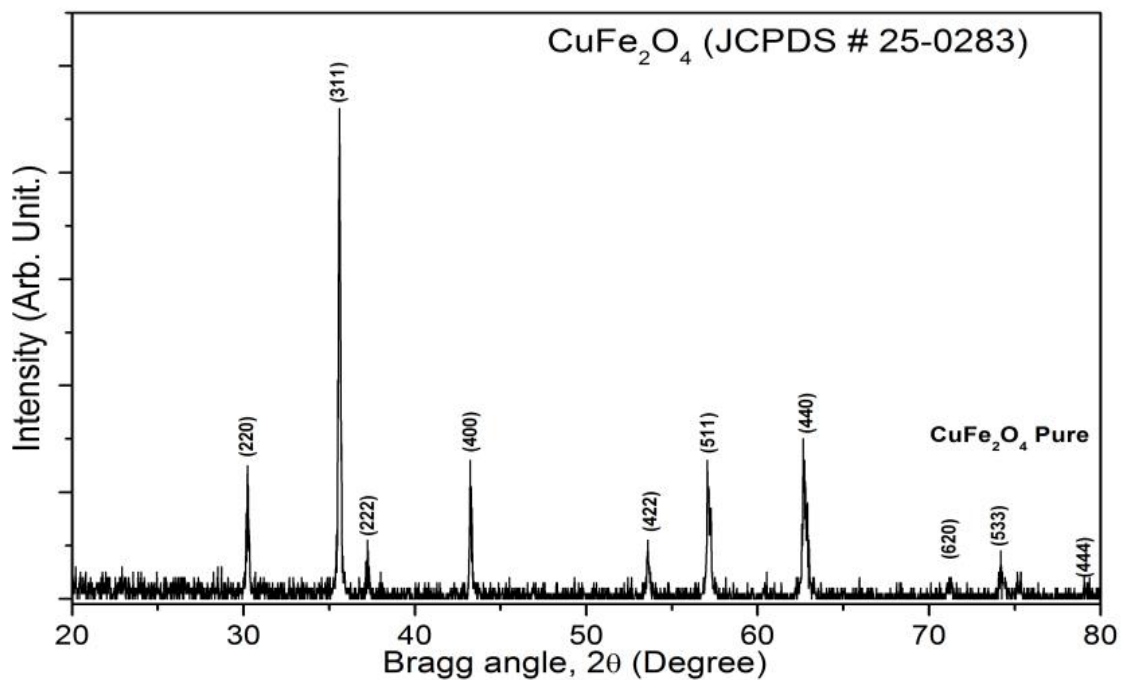


Fig. 1. XRD patterns of $CuFe_2O_4$ nanopowders.

TABLE 1. Crystallite size (t), Lattice constant (a), Lattice volume (V), theoretical density (d_x) and band gap energy (E_g) of CuFe_2O_4 nanoparticles.

Parameters	Crystallite size, t (nm)	Lattice constant, a (Å)	Lattice volume, V (Å ³)	X-ray density, d_x (g/cm ³)	Band gap energy, E_g (eV)
CuFe_2O_4	100.8	8.349	581.970	5.310	1.14

TABLE 2. The obtained lattice parameters, the ionic radii of the A- and B-sites (R_A, R_B), the A- and B-site bond lengths (d_{AL}, d_{BL}), the tetrahedral edge (d_{AE}) and the octahedral shared and unshared edges (d_{BE}, d_{BEU}) and are hopping lengths at A- and B-sites (L_A, L_B) of CuFe_2O_4 nanoparticles.

parameter	A-sites ionic radii, R_A (Å)	B-sites ionic radii, R_B (Å)	A-site bond length, d_{AL} (Å)	B-site bond length, d_{BL} (Å)	tetrahedral edge, d_{AE} (Å)	Octahedral shared edge, d_{BE} (Å)	octahedral unshared edge, d_{BEU} (Å)	A-site hopping length, L_A (Å)	B-site hopping length, L_B (Å)
CuFe_2O_4	0.575	1.170	1.975	2.005	3.225	2.698	1.051	7.254	5.923

Transmission electron microscopy analysis

The high-resolution transmission electron microscope (HRTEM) images in Fig. 2 show size, shape and distribution of zinc ferrite prepared by the co-precipitation method at 1000 °C at 2 h. The first

view showed that a group of very fine crystals self-organized and the large cubic structure morphology becomes the dominant and the particle size increased to 278 nm length and 503 nm width.

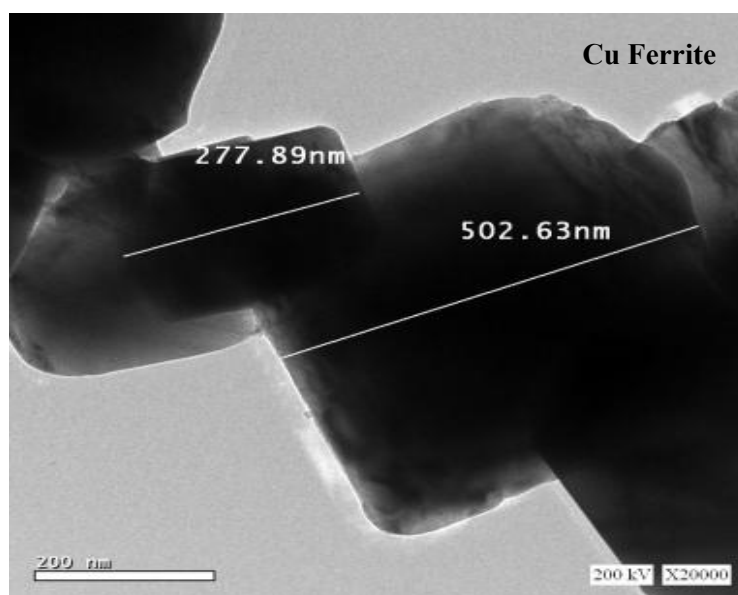


Fig. 2. TEM micrographs of CuFe_2O_4 nanopowders.

Optical properties

UV-Vis-NIR Spectra

The optical properties of the powders for annealed samples were examined using UV-Vis-NIR spectrophotometer using integrating sphere unit. Fig. 3 depicts the absorbance and reflectance spectrums of CuFe₂O₄ nanopowders for 1000 °C for 2h whereas The as deposited sample was exhibited the lowest optical reflectivity of around 45-10% in the infrared (IR) and visible regions. Further increasing in annealing temperature was found to enhance the average reflectivity of around 62-12% in the IR and visible regions, respectively. Additionally, the absorption peaks were corresponding to the characteristic peak of copper and iron ions.

Three bands were related to ${}^2B_{2g} \rightarrow {}^2B_{1g}$, ${}^2A_{1g} \rightarrow {}^2B_{1g}$ and ${}^2E_g \rightarrow {}^2B_{1g}$ energy transitions of octahedral symmetry copper ion for pure copper oxide [38]. The first two transitions were very close in energy and often appeared in the form of one broad band profile in near-infrared. The third band was observed in the visible region which exhibited a broad optical absorption band around 780 nm, assigned to the ${}^2E_g \rightarrow {}^2B_{1g}$ transition imputed to Jahn–Teller splitting of d levels of copper ions [38]. Otherwise, three transition bands ${}^6A_{1g}(S) \rightarrow {}^4T_{1g}(G) (\nu_1)$, ${}^6A_{1g}(S) \rightarrow {}^4T_{2g}(G) (\nu_2)$ were assigned ; ν_1 at 950 nm whereas ν_2 at 550 to 650 nm usually as a shoulder. Indeed, the bands corresponding to ${}^6A_{1g}(S) \rightarrow {}^4A_{1g}(G)$, ${}^4E_g(G) (\nu_3)$ was deduced around 450 nm[39].

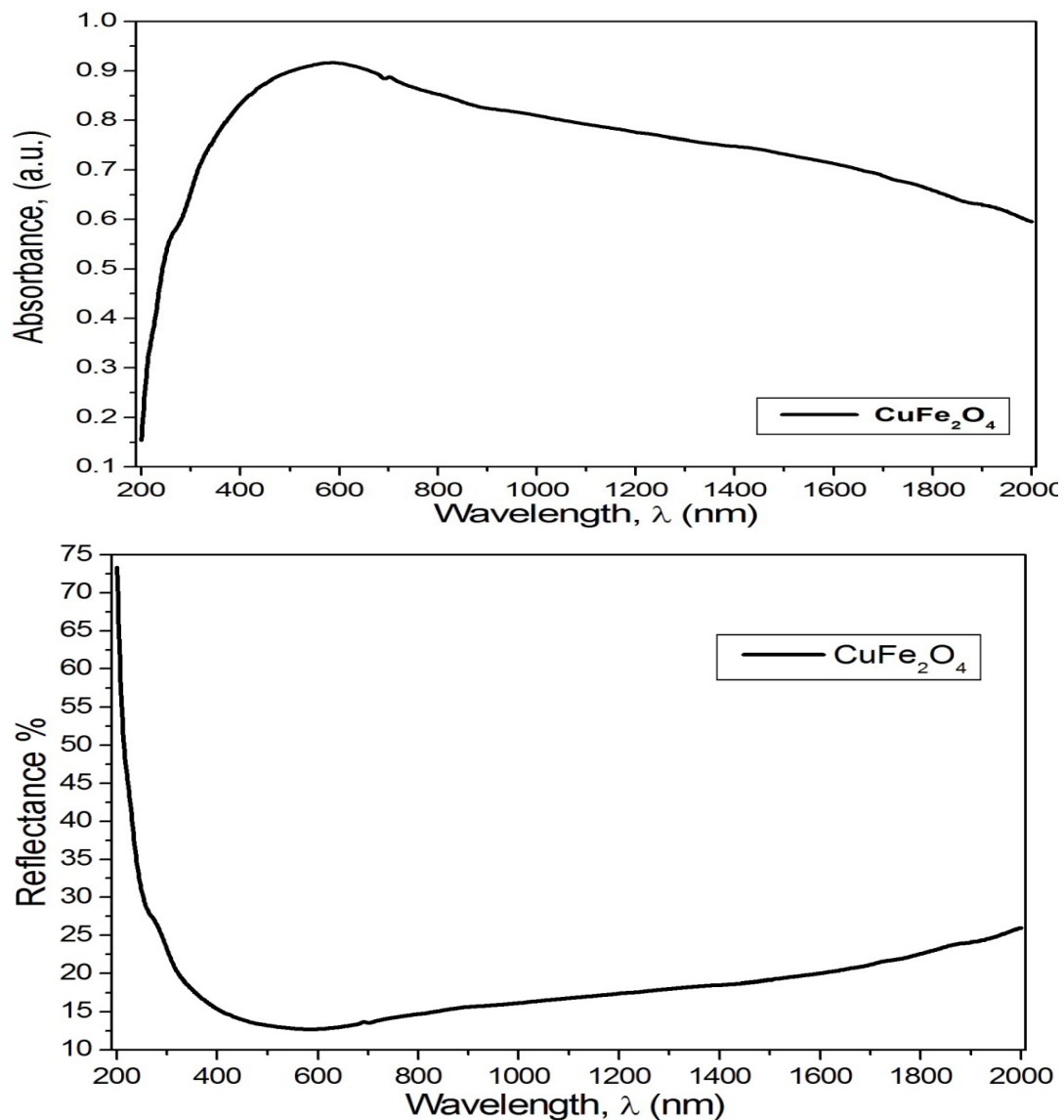


Fig. 3. Optical absorbance diffuse and reflectance spectra of CuFe₂O₄ nanoparticles .

In the limiting case of an infinitely powder samples, thickness and sample holder have no influence on the value of reflectance (R). In this case, the Kubelka-Munk equation at any wavelength becomes [40,41]:

$$F(R_\infty) = (100-R)/(2R) \quad (13)$$

$F(R_\infty)$ is the so-called remission or Kubelka-Munk function. In the parabolic band structure, the band gap (E_g), and absorption coefficient (α), of a direct band gap semiconductor are related through the well known equation [42]:

$$ahv = A(hv - E_g)^{1/n} \quad (14)$$

Where a is the linear absorption coefficient of the material, $h\nu$ is the photon energy and A is proportionality constant. When the material scatters in perfectly diffuse manner (or when it is illuminated at 60° incidence), the K-M absorption coefficient K becomes equal to $2a$ ($K=2a$). In this case, considering the K-M scattering coefficient S as constant with respect to wavelength, and using the remission function in Eq. (14) we obtain the expression [43,44]:

$$(hvF(R_\infty))^n = B(hv - E_g) \quad (15)$$

Therefore, obtaining $F(R_\infty)$ from Eq. (15) and plotting the $[F(R_\infty)hv]^2$ against $h\nu$, the band gap energy E_g of a powder sample can be extracted easily. The band gap energy of CuFe_2O_4 nanoparticles after Kubelka-Munk treatment

was shown in Fig. 4. The direct band gap energy of CuFe_2O_4 sample was 1.14 eV value was summarized and listed in Table 1.

Magnetic properties

The magnetization of the produced sample of CuFe_2O_4 nanoferrites nanoparticles was measured at room temperature under an applied field of 20 KOe and the hysteresis loops were determined. Plots of magnetization (M) as a function of the magnetic field (H) were plotted in Fig.5 and the corresponding data are collected in Table 3. The as-prepared products exhibited soft-magnetic properties. The values of saturation magnetization (M_s), coercivity (H_c) and remanence (M_r) are found to be in the range of (33.623) emu/g , (2.3627) emu/g , (71.489) Oe respectively. The squareness ratios for all the samples were also calculated from M_s and M_r data, and the calculated values are listed in Table 3.

Magnetic heating induction ability

The characteristic temperature-time evolution of the sample under fixed magnetic field strength 0.75 KA/m, at 195 KHz. After the magnetic field is switched on, the temperature initially rise the heating ability of the magnetic nanoparticles was found to be dependent on its concentration as in Table 4 in addition to its magnetic properties. The Nano-crystalline Cu ferrite show notice rise in temperature in short time (10 min) as shown in Fig.6 ready for killing tumor cells.

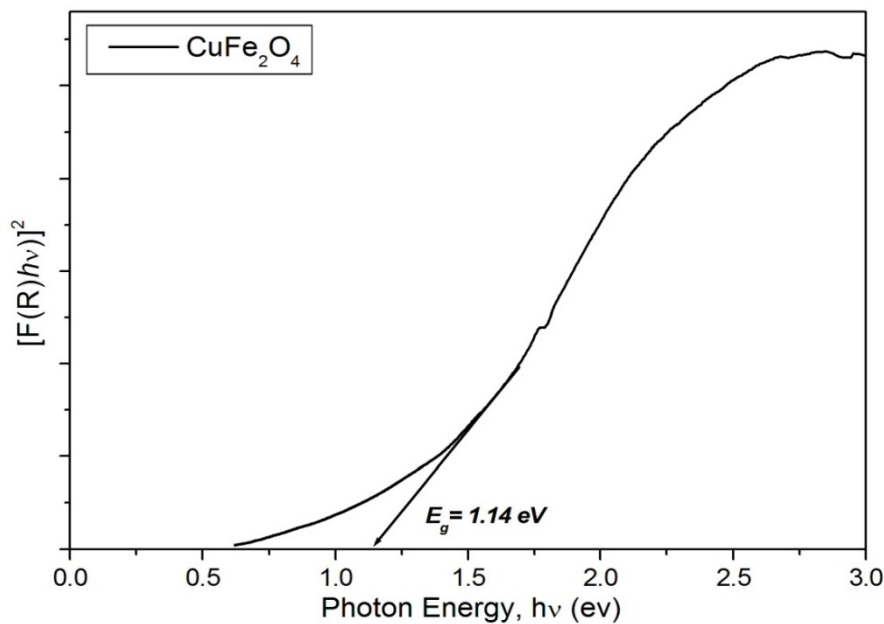


Fig. 4. Optical band gap energy of CuFe_2O_4 nanoparticles .

TABLE 3. Magnetic properties of CuFe₂O₄ nanoparticles.

Sample	<i>Ms</i> (emu/g)	<i>Mr</i> (emu/g)	<i>Hc</i> x10 ⁻³ (Oe)
CuFe ₂ O ₄	33.623	2.3627	71.489

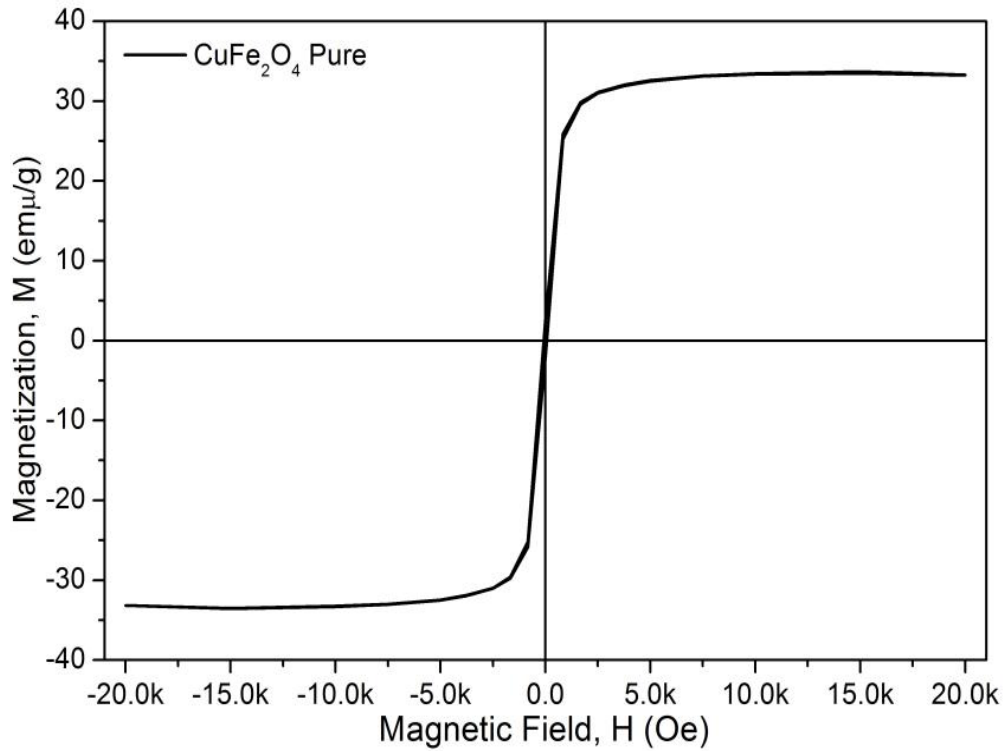


Fig. 5. M-H loops of CuFe₂O₄ nanopowders.

TABLE 4. Magnetic induction heating ability with different concentrations.

Time (min)	(20 mg/ml)	(40 mg/ml)	(60 mg/ml)	(80 mg/ml)
0	32	32	32	32
1	32.3	32.7	32.8	32.9
2	32.7	33.6	33.3	34
3	33.4	34.7	34	34.7
4	34	35	35.1	35.3
5	34.3	35.7	35.8	36.4
6	35.1	35.8	36.5	37.1
7	35.2	35.9	36.6	37.2
8	35.7	36.5	36.8	37.6
9	36.1	36.8	37.1	37.9
10	36.9	37.2	37.9	38.1

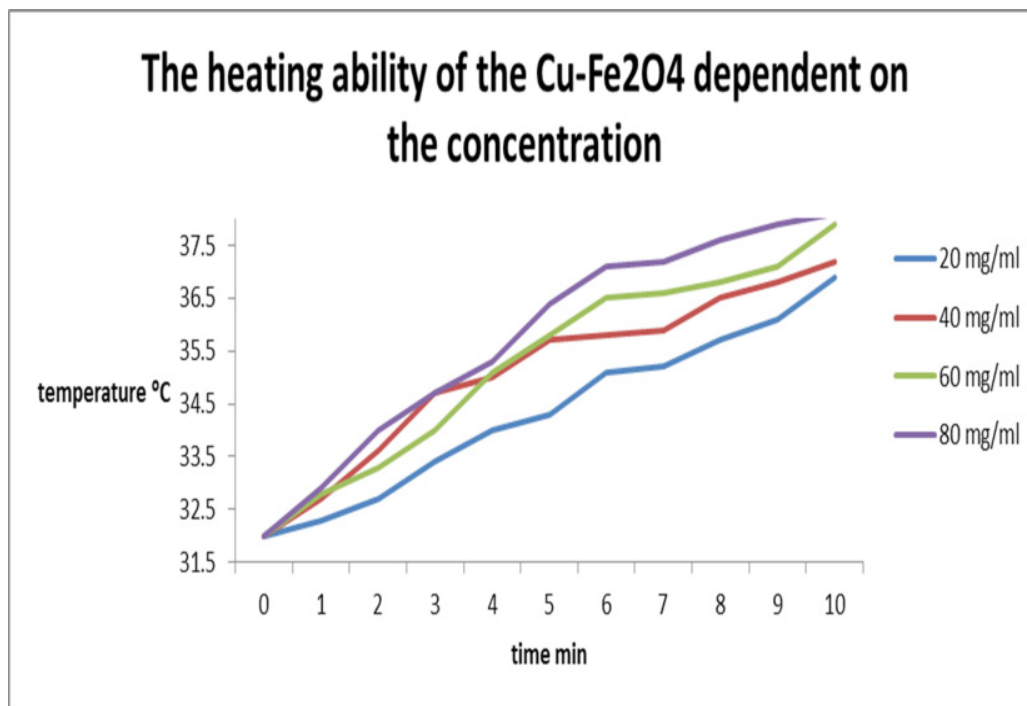


Fig. 6. Magnetic induction heating ability.

Conclusion

The degree to which magnetic hyperthermia can be applied to cancer therapy depends on the ability to deliver MNPs systematically to tumor cells in sufficient concentrations. The results indicate that a notice rise in temperature in short time induced by magnetic nanoparticles and the alternating magnetic field. Also, the heating ability of the magnetic nanoparticles was found to be strongly dependent on its concentration. The nanocrystalline Cu ferrite with the hyperthermia system is ready for killing tumor cells.

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(Received 19/9/2018 ;
accepted 18/1/2019)

دراسة الخواص المغناطيسية ونتاج الحرارة اللازمه لعلاج الأورام باستخدام درجات الحرارة المرتفعة

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في هذا البحث تم تحضير الجسيمات المغناطيسية النانومترية متناهية الصغر باستخدام طريقة الترسيب (TEM) والمجهر الإلكتروني النافذ (XRD) المصاحب. وتم توصيف العينات بأستخدام حيود الأشعة السينية من أجل دراسة الشكل البلوري، والبنية المجهرية والخصائص (VSM) وجهاز قياس الخواص المغناطيسية البصرية والمغناطيسية. رفع درجة الحرارة باستخدام الجسيمات المغناطيسية هو من أكثر الطرق الواعدة في علاج السرطان. إن طريقة رفع درجة الحرارة باستخدام الجسيمات المغناطيسية تتم بتطبيق مجال كهربائي متردد. وقد تم دراسة سلوك ارتفاع درجات الحرارة وخصائص التسخين للجسيمات النانومترية المغناطيسية بعد تطبيق مجال مغناطيسي متردد من ١٠٠-٣٠٠ كيلو هيرتز. والنتائج تشير إلى ان هذه الجسيمات النانومترية المغناطيسية مناسبة لانتاج الحرارة اللازمة لعلاج السرطان. كذلك ستساعدنا هذه النتائج في تحسين ظروف معالجة الأورام السرطانية