

DOI: <http://dx.doi.org/10.21123/bsj.2016.13.2.0331>

Synthesis and Characterization of Multishapes of Fe₃O₄ Nanoparticle by Solve-Hydrothermal Method Using Microwave Radiation

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Received 6/5/ 2015

Accepted 15/6/ 2015

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DOI:10.21123\bsj.2016.13.2.0000

Abstract:

Iron oxide(Fe₃O₄) nanoparticles of different sizes and shapes were synthesized by solve-hydrothermal reaction assisted by microwave irradiation using ferrous ammonium sulfate as a metal precursor, oleic acid as dispersing agent, ethanol as reducing agent and NaOH as precipitating agent at pH=12. The synthesized Fe₃O₄ nano particles were characterized by X-ray diffraction (XRD), FTIR and thermal analysis TG-DTG. Sizes and shapes of Fe₃O₄ nanoparticles were characterized by Scanning Electron Microscopy (SEM), and atomic force microscopy (AFM).

Key words: Magnetite, microwave, oleic acid, SEM, AFM

Introduction:

There has been an increasing interest recently towards the synthesis and fabrication of magnetic nanoparticles mainly magnetite (Fe₃O₄). Besides the high surface to volume ratio and high surface energy, these nanomaterials have been found to have distinguished thermal, magnetic and chemical properties that made them suitable for several applications. In industry magnetic nanomaterials have been used in magnetic fluids for heat transfer [1-3], catalysis [4-7], corrosion inhibitors [8], energy and magnetic storage devices [6, 9] electronic devices [5]. In environmental applications magnetite nanoparticles were used for removal of toxic heavy metal ions like Cu(II), Cr(VI), Cd(II), Pb(II), Hg(II), Tl(I),

As(III), Ag(I) [5, 10-15] and organic pollutants [16-19] from waste water. In biomedical applications magnetite nanoparticles were used in magnetic resonance imaging (MRI), contrast agents, drug delivery and therapy [4, 20-25]. The compatibility of these applications is mainly controlled by phase, size and morphology of nanomaterials. Several methods have been reported on the synthesis of different sizes and morphologies of iron oxide Fe₃O₄ and γ -Fe₂O₃ nanoparticles which mainly involve simple wet chemical [15, 19, 26-30], hydrothermal [9, 31-33], electrochemical [34] and microwave methods [35, 36]. Since magnetite nanoparticles are susceptible to oxidation by atmospheric oxygen, the

synthesis of this oxide is best performed in presence of protecting agents like PPy [31], polyethylene glycol [35] and reducing agents like trisodium citrate and ethylene glycol [31]. The microwave method is attracting more attention compared with conventional heating methods because of rapid and controlled production of high yield advanced materials as well as it is safe and needs less energy [35]. Da-Peng Yanga, et al [35] synthesized spherically shaped Fe_3O_4 nanoparticles with average size of ~100 nm by microwave using ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), ethylene glycol, anhydrous sodium acetate (CH_3COONa) and polyethylene glycol, while Z. Kozakova, et al. [36] obtained uniform spherical Fe_3O_4 nanoparticles with average dimensions of 200 nm in 30 min using microwave using ammonium acetate, ammonium carbonate ($\text{NH}_4)_2\text{CO}_3$ or aqueous NH_3 as precipitant. This work presents the synthesis of multi shapes of Fe_3O_4 nanoparticles like spherical, plate, and nano flowers by solve thermal method assisted by microwave radiation, by using $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ as iron precursor, oleic acid as reducing agent, ethanol and NaOH.

Materials and Methods:

All of the the following chemical reagents in this work are of analytical grade and were used as received from suppliers: $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ (Sigma Aldrich), sodium hydroxide NaOH, 96% (BDH), ethanol 99%, oleic acid ($\text{C}_{18}\text{H}_{34}\text{O}_2$) and acetone (Fluka).

Instruments:

FT-IR spectra were recorded on Shimadzu 8400 S FT-IR spectrophotometer using KBr pellet. Scanning electron microscope (SEM) analyses were acquired using type SEM TE SCAN VEGA 2 Cheech. A few drops of nanoparticle suspension were applied on a carbon coated copper grid followed by vacuum drying. Atomic

force microscope images AFM were acquired using AFM model AA 3000 SPM 220V Angstrom Advanced INC. USA. Samples were prepared by applying few drops of nanoparticles dispersed in acetone by sonication for 20 min, on a glass slide followed by vacuum drying at room temperature. Thermal gravimetric analyses (TGA) were performed using Perkin Elmer TGA 4000 thermo gravimetric analyzer in a synthetic N_2 atmosphere at heating range $30^\circ - 900^\circ\text{C}$ and heating rate of $20^\circ\text{C}/\text{min}$. X-ray diffraction analyses (XRD) were recorded using SHIMADZU 6000 X-Ray Diffract meter with a high-intensity Cu K α radiation ($\lambda=1.54180\text{\AA}$) and a graphite monochromatic source. Magnetic susceptibility of the prepared oxide was measured by using a magnetic balance Model MSB-MK I. Preparation of Fe_3O_4 nanoparticles by microwave irradiation was performed in a domestic microwave oven (mode LG, MS2042X frequency 2.450 MHz, maximum power 1000 W), fixed with a wireless camera to watch capsule reactor.

Preparation of Fe_3O_4 by solve-hydrothermal microwave method

Fan Zhang, et al [33] prepared Fe_3O_4 nanospheres by hydrothermal method by heating a mixture of $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ as iron precursor, oleic acid as reducing agent, ethanol and NaOH in an autoclave at 180°C for 10 h. In this work we used the same reactants to prepare Fe_3O_4 nano crystals but by using microwave technique. To a solution mixture of oleic acid ($\text{C}_{18}\text{H}_{34}\text{O}_2$) (6.2 ml) and ethanol (7.02 ml) in three-neck round-bottomed flask was added sodium hydroxide NaOH (0.562 g, 8.9 mmol) with vigorous stirring under argon atmosphere at room temperature A white viscous solution was formed. To this solution was added, the iron precursor $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$

(0.352g, 0.898 mmol) with vigorous stirring. The final pH of the reaction mixture was adjusted to 12. A dark green precipitate appeared immediately. After stirring for further 30 minutes under continuous argon flow, the mixture turned brown. The resultant suspension was then transferred into a homemade 30 mL Teflon autoclave which was sealed, and heated in the microwave domestic oven (second level, 6 second ON and 16 second OFF) at 190 ° C for 15 min. The system was then

allowed to cool naturally to room temperature. The color of the mixture was black. The steps of separation of the product are illustrated in Figures (1a-e). The product was collected and separated by permanent magnet which took more than 20 min, washed several times with distilled deionized water (DDW), followed by ethanol, then vacuum dried at 80°C for 10 h and stored under nitrogen atmosphere for characterization by XRD, FTIR, SEM, AFM, and TG analyses.

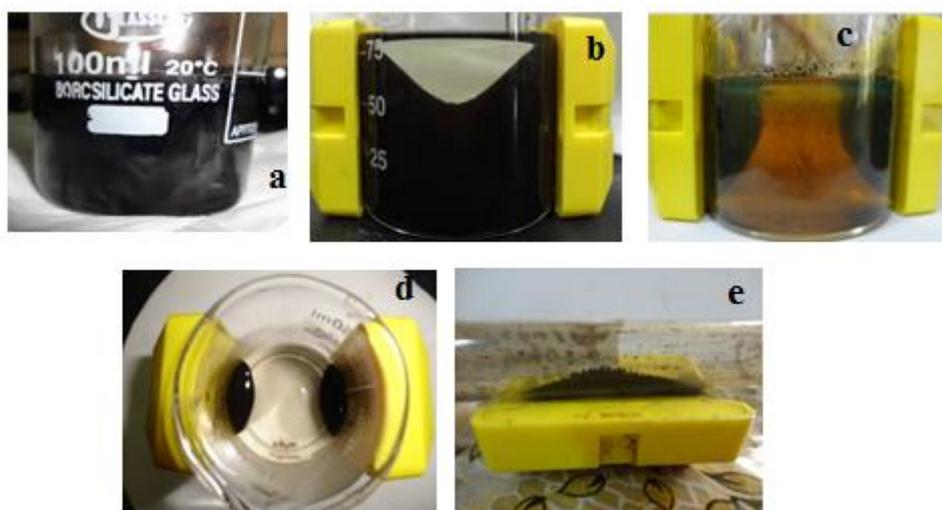


Fig. (1): Separation and of iron oxide Fe_3O_4 by permanent magnet

Results and Discussion:

X-Ray diffraction analysis

Figure (2) shows the XRD pattern of Fe_3O_4 nano particles obtained by using solve-hydrothermal by microwave radiation method, using Cu K α ($\lambda=1.54056 \text{ \AA}$) radiation with 2θ in the range (10-60°). Five main diffraction peaks were observed at $2\theta = 30^\circ, 35.6^\circ, 43.3^\circ, 54^\circ, 57.1^\circ$, correspond respectively to the planes (220, 311, 400, 422, and 511 respectively) of Fe_3O_4 nanostructures which came in agreement with the card (space group: JCPDS Nos. 26-1136) X-ray diffract meter with a high-intensity Cu K α radiation ($\lambda=1.54056 \text{ \AA}$) and a graphite monochromatic a. The results indicated a cubic structure of Fe_3O_4 with

lattice parameters $a=b=c=8.0903 \text{ \AA}$ and $\alpha = \beta = \gamma = 90^\circ$ and agreed with the structure of an inverse spinel type oxide [2, 35, 37, 38]. Crystallite size measurements were determined from the full-width at half maximum (FWHM) of the strongest peaks assigned to the reflection planes (220), (311), (400) and (511) (Table (1)) using the Debye-Scherrer approximation [39]

$$D = \frac{K \cdot \lambda}{\beta \cos \theta}$$

Where (D) is crystallite diameter, (K) = 0.9 is the constant, λ is the wavelength of the X-rays, (β) is the peak breadth of the XRD peak, and θ is the Bragg angle (in radians or degrees). The calculated crystal average size is (25.5) nm.

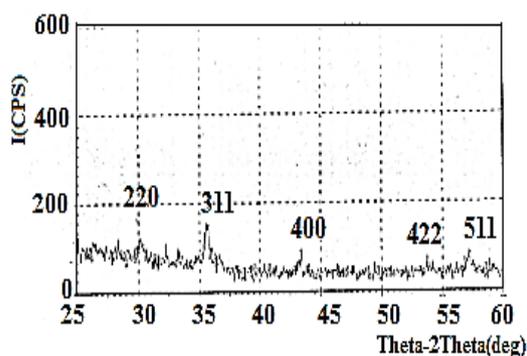


Fig. (2): XRD patterns of the Fe_3O_4 synthesized by solve-hydrothermal microwave method

Table (1): Average particle size of the synthesized Fe_3O_4 nanoparticles calculated from Scherer's equation

Diffraction peak at 2θ (deg.)	FWHM	Size (nm)	Average size (nm)
30°.2	0.416	25.5	25.55
35°.6	0.445	18.9	
43°.3	0.266	32.8	
57°.1	0.366	25.0	

FT-IR Spectrophotometry

Figure 3 shows the FT-IR spectrum of the prepared Fe_3O_4 coated with oleic acid (OA). The spectrum displayed an absorption band at 3006 cm^{-1} which was assigned to the stretch vibration of vinylic $=\text{C}-\text{H}$ group [40, 41]. The broad peak at 3122 cm^{-1} can be assigned to surface and bulk OH groups in magnetite [40]. Two absorption sharp peaks were observed at 2923.8 and 2852.5 cm^{-1} were attributed to the CH_2 and CH_3 stretching vibrations [41]. The intense peak at 1701 cm^{-1} was assigned to the $\text{C}=\text{O}$ stretching vibration of carboxylate anion [42]. The two new bands at 1639 and 1541 cm^{-1} were assigned to the asymmetric (COO^-) and symmetric (COO^-) stretching vibrations. This indicates that the carboxylate group is present as both mono and bidentate bonding group i.e. oleic acid has been both physically and coordinatively adsorbed on the surface

of magnetite nanoparticles. The bands observed at 1454.2 and 900 cm^{-1} were attributed to the O-H in-plane and out-of-plane vibrations respectively. The absorption peak observed at 584 cm^{-1} corresponds to the Fe-O stretching vibration related to the magnetite phase [40],

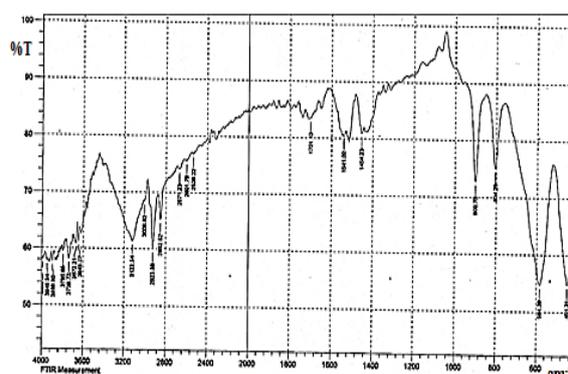


Fig.(3): FTIR Fe_3O_4 synthesized by solve- hydrothermal microwave Scanning electron microscopy (SEM)

The SEM micrographs of Fe_3O_4 reveal the presence of three types of morphology like spherical, sheets and flowers nanostructures as is shown in Figures 4a and b and the average particle sizes of spherical Fe_3O_4 nanoparticles are (50-200nm). The thickness of thorns in flower shaped nanostructures are about (8-10nm) while the diameter and length of nanosheets are $0.8-1\mu\text{m}$ and $1-2\mu\text{m}$ respectively. The same morphology of copper oxide nanoparticles was reported by Volanti et. al using the same method [1].

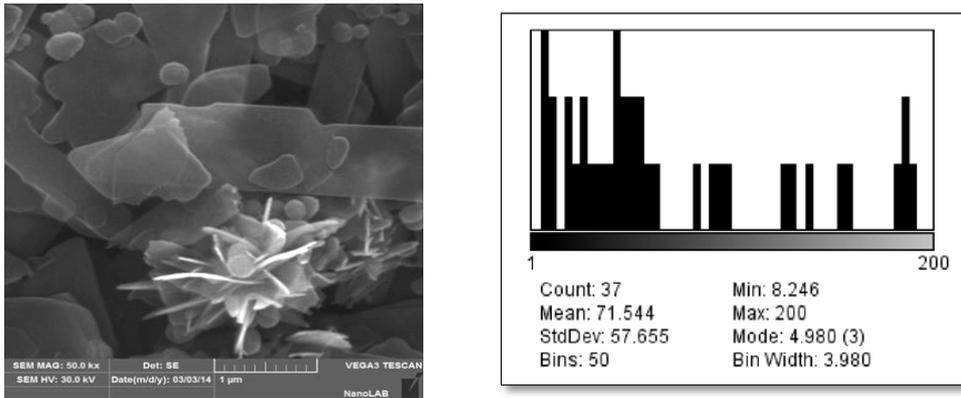


Fig.(4a): SEM micrograph and particle size distribution of solve-hydrothermally synthesized Fe₃O₄ nanoparticles under microwave radiation showing different sizes of nanospheres, nanosheets, and flower shaped nanostructures.

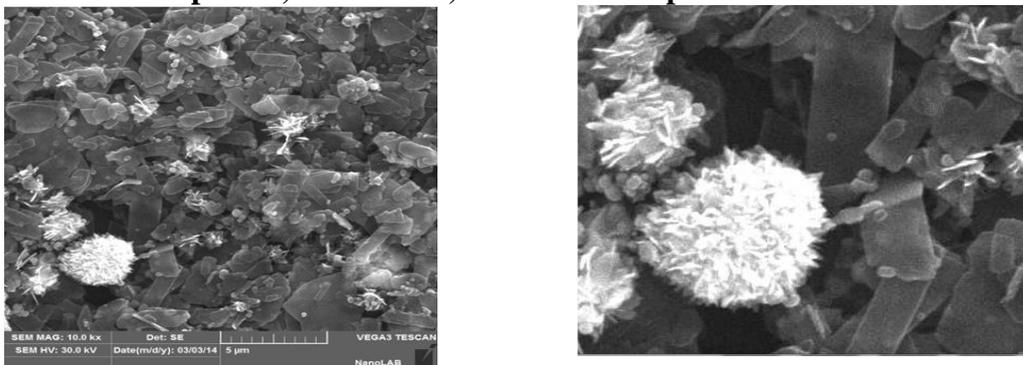


Fig. (4b): SEM micrograph of solve-hydrothermally synthesized Fe₃O₄ nanoparticles under microwave radiation showing several flower shaped nanostructures (left) and a zoom view that shows the presence of spherical and sheet like nanoparticles(right)

Atomic force microscopy (AFM)

Figure (5) shows the 2D, 3D views and particle size distribution of Fe₃O₄ by AFM analysis which reflects the variation and existence of large

aggregates of nanoparticle as plates and flower nanostructure as those shown in SEM micrograph with average size about 91.02 nm.

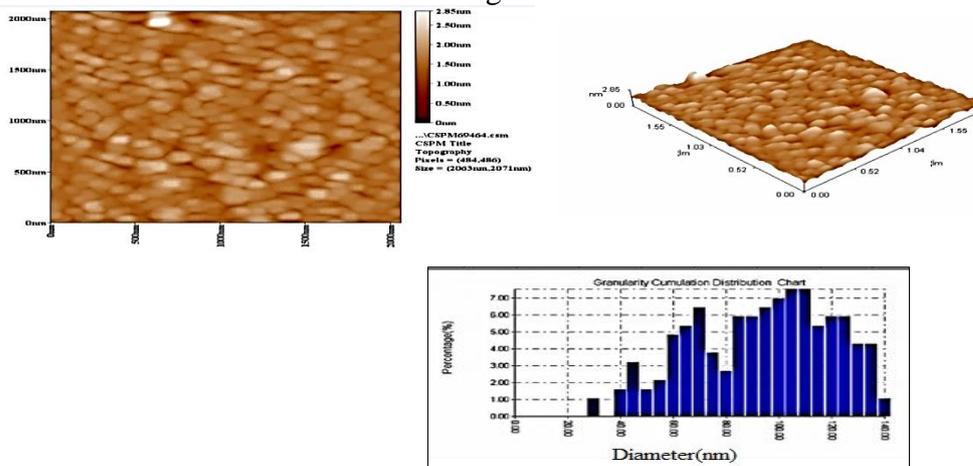


Fig.(5): AFM 2D, 3D views and particle size distribution of Fe₃O₄ nanoparticles synthesized by solve hydrothermal microwave method

Thermo gravimetric analysis (TGA)

The TGA/DTG curves of Fe_3O_4 MNPs coated with OA under N_2 atmosphere at heating range $30^\circ\text{-}900^\circ\text{C}$ and heating rate of $20^\circ\text{C}/\text{min}$ are shown in Figure (6). The TG curve displays three steps of thermal decompositions with weight losses of 0.1, 2.4 and 1.20 which at temperature ranges 30-150, 150-380 and $380\text{-}620^\circ\text{C}$ at peak temperatures 76.75 , 309.58 and 616.8°C

as demonstrated by the DTG curve. The first step corresponds to vaporization of adsorbed solvent molecules present in the sample. The second step is attributed to the removal of free oleic acid adsorbed on the surface of Fe_3O_4 MNPs [43]. The third step is attributed to oxidation and removal of the remaining coordinatively adsorbed oleic acid and phase transition from Fe_3O_4 to hematite [42]

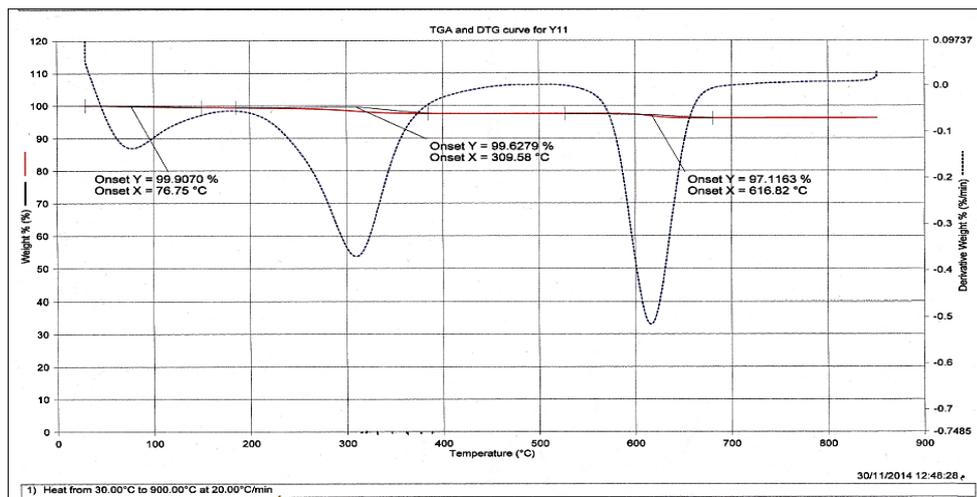


Fig. (6): TGA and DTG thermograph of Fe_3O_4 nanoparticles prepared by solve-hydrothermal microwave method

Conclusion:

Iron oxide (Fe_3O_4) nano particles of different sizes and shapes were synthesized by solve-hydrothermal reaction assisted by microwave irradiation using ferrous ammonium sulfate as a metal precursor, oleic acid as dispersing agent, ethanol as reducing agent and NaOH as precipitating agent at $\text{pH}=12$. XRD analysis supported the inverse spinel structure. The FTIR and thermogravimetric analyses supported the conjugation of Fe_3O_4 with oleic acid through the carboxyl group which behaved as mono and bidentate ligand

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تحضير وتشخيص دقائق Fe_3O_4 النانوية متعددة الاشكال بطريقة المذيب – الهيدروحرارية بواسطة اشعة الميكروويف

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الخلاصة:

حضرت دقائق Fe_3O_4 النانوية من مختلف الأحجام والأشكال بطريقة المذيب-هيدروحرارية بواسطة إشعاع الميكروويف باستخدام كبريتات الأمونيوم الحديد كمصدر للفلز، وحامض الأوليك كعامل مشتت، والإيثانول كعامل مختزل وهيدروكسيد الصوديوم كعامل ترسيب في دالة الحموضة = 12. شخصت دقائق Fe_3O_4 النانوية من حيود الأشعة السينية (XRD)، تحليل الأشعة تحت الحمراء والتحليل الحراري (TG-DTG). شخصت الحجم والأشكال للدقائق النانوية Fe_3O_4 بواسطة المجهر الإلكتروني الماسح (SEM)، ومجهر القوة الذرية (AFM).

الكلمات المفتاحية: مغناطيت، ميكروويف، حامض الأوليك، SEM، AFM.