

Synthesis of Fe₃O₄ nanoparticles from Ferric Nitrate by Sol-Gel Method

تخليق جزئيات النانوية
لأكسيد الحديد المغناطيسي "Fe₃O₄"
من نترات الحديد بواسطة طريقة محلول - غروي

Nabil Abdullah Noman Alkadasi

- *Hubei key lab of Materials Chemistry & Service Failure , School of Chemistry & Chemical Engineering ,Huazhong University of Science and Technology, Wuhan , Hubei , 430074 China .*
- *Doctor - Department of Chemistry ,Faculty of Education and Science, Rada'a, Al-Baida'a ,University , Yemen.*



جامعة الأندلس
للعلوم والتكنولوجيا

Alandalus University For Science & Technology

(AUST)

Synthesis of Fe₃O₄ nanoparticles from Ferric Nitrate by Sol-Gel Method

Abstract :

Fe₃O₄ nanoparticles were synthesized from Ferric Nitrate by Sol-Gel method with annealing under vacuum. The phase structures, morphologies, particle sizes, chemical composition, and magnetic properties of Fe₃O₄ nanoparticles were characterized by TEM ,U.V and XRD .

The results indicated that the size, the corresponding saturation magnetization value and coercivity value of Fe₃O₄ nanoparticles increase with the increase of temperature . Fe₃O₄ nanoparticles was studied under different atmospheres and temperatures.

Keywords: Ferric Nitrate , Fe₃O₄ nanoparticles , properties and characterization.

الملخص :

الحساسية بزيادة جزيئات النانوية لأكسيد الحديد المغناطيسي "Fe₃O₄" مع زيادة درجة الحرارة . جزيئات النانوية لأكسيد الحديد المغناطيسي "Fe₃O₄" درست تحت درجات حرارة مختلفة وتحت ضغط .
الأشياء الرئيسية: نترات الحديد ، جزيئات النانوية لأكسيد المغناطيسي "Fe₃O₄" والخواص و التحليل.

خلقت جزيئات النانوية لأكسيد الحديد المغناطيسي من نترات الحديد بواسطة محلول -غروي مع درجة الحرارة العالية وتحت ضغط. التراكيب الطوري، المرفولوجيات والاحجام الجزيئية تركيب الكيميائي ، الصفات المغناطيسية من جزيئات النانوية الأكسيد الحديد المغناطيسي "Fe₃O₄" دراسة التحليل بواسطة اجهزة TEM, XRD and U.V. والنتائج تشير إلى الحجم والقيم المغناطيسية لمواضع الارتباط والقيم

Introduction :

In the past decade, a variety of methods have been developed to form highly structure-controlled materials of functionalized metals, semiconductors and copolymer nanoparticles on the nano- or microscale. As a versatile kind of material, magnetite has attracted much attention in recent years.

Ferrite iron (Fe₃O₄) is a traditional magnetic material used in magnetic storage media, solar energy transformation, electronics, ferrofluids, biomedicine and catalysis [1-4]. During the last decade, significant research has been done in the field of nanosized magnetic particles, due to their potential for biomedical applications, such as improving the quality of Magnetic Resonance Imaging (MRI) , and drug delivery systems [5 – 6].

Fe₃O₄ nanoparticles have been the subject of intense interest because of their potential applications in several advance technological areas due to their promising physical and chemical properties. Generally, these properties depend on the size and structure of particles . Fe₃O₄ nanoparticles find wide applications in the field of biomedical, as anticancer agent and corrosion protective pigments in paints and coatings [7- 9]. The magnetic atoms or ions in such solid materials are arranged in a periodic lattice and their magnetic moments collectively interact through molecular exchange fields, which give rise to a long-range magnetic ordering.

Among all iron oxide nanoparticles, Fe₃O₄ represent the most interesting properties due to of its unique structure i.e. the presence of iron cations in two valence states, Fe²⁺ , Fe³⁺ on tetrahedral and octahedral sites with an inverse cubic spinel structure. The coercivity and remenance values for the super paramagnetic nano Fe₃O₄ nanoparticles have been found to be zero by the earlier reported methods [10-13]. Presently, cell labelling strategies find application of superparamagnetic ferrite either through conjugating the magnetic nanoparticles to the cellular surface of the stem cell or by internalization of the particles into the cell. Superparamagnetic ferrite can work in both of these ways, since the potential to manipulate their surface chemistry is plentiful and their sizes along

with other attributes promote their successful uptake into cells. The superparamagnetic nano ferrites also interface well with MRI technology.

The use of superparamagnetic particles play a crucial role in the diagnostic imaging modality technique finds application in the study of stem cell [14-15].

In this paper Fe₃O₄ nanoparticles were synthesized by heating to 40⁰C at 2 hours and at 80⁰C for 2 hours to obtained Sol Gel followed by drying for 6 hours at 120 ⁰C and then annealing at 200⁰C and 400⁰C in oven under vacuum to obtain Fe₃O₄ powder .

Experimental Materials :

Ferric nitrate (Fe(NO₃)₃.9 H₂O) and ethylene glycol (C₂H₆O₂) of analytical grade were obtained from Sinopharm Chemical Reagent Co ,Ltd ,China. The reagents were used without further purification.

Physical parameters of Ferric nitrate (Fe (NO₃)₃. 9 H₂O) and Glycol (C₂H₆O₂) are reported in table 1 and 2 respectively.

Table 1. General Characteristics of Ferric nitrate (Fe (NO₃)₃. 9 H₂O)

Molecular formula	Ferric nitrate (Fe (NO ₃) ₃ . 9 H ₂ O) ≥ 98.5 %
Appearance	White Powder
Molecular weight	404
Company	Sinopharm chemical reagent Co ,Ltd ,China

Table 2. General Characteristics of Ethylene Glycol (C₂H₆O₂)

Molecular formula	Ethylene Glycol (C ₂ H ₆ O ₂) ≥ 99 %
Appearance	liquid
Molecular weight	62.07
density	1.111- 1.115
Company	Sinopharm chemical reagent Co ,Ltd ,China

Preparation of Magnetite Nanoparticles :

The procedure of synthesizing magnetite nanoparticles [16-19] is described as follows:

20.2 gram ferric nitrate was first dissolved in 25 mL ethylene glycol with vigorous stirring speed 500r.p.m in 100 mL three necked flask glass with out cover for 2 hours at 40 °C, and then the solution was heated to 80 °C and kept at the temperature to obtain brown gel. The gel was obtained after 2 hours and then dried at 120 °C for about 4 hours in petri dish . After drying, the xerogel was annealed at temperature range 200 and 400 °C under vacuum. Finally, different size magnetite nanoparticles were obtained.

Transmission Electron Microscope (TEM) Test :

For TEM Test , a small amount of sample was dissolved in 3mL of deionized water in test tube and the solution was stirred by ultra-sonication . Then 10 µ L sample was transferred to clean Copper Grid and kept for drying for TEM test. The TEM micrographs of samples were observed by CM 12 Philips Transmission Electron Microscope .

Results and Discussion :

Plate 1,2,3 ,4,5,6 ,7 and 8 (TEM) at at 200 0C and plate 9,10,11,12,13,14,15,16,17,18 and 19 (TEM) at at 400 0C shows the top-view TEM images of the Fe₃O₄ Nanoparticle plate (TEM) 1 The size of the Fe₃O₄ nanoparticle is clear from the TEM. The surface of Fe₃O₄ nanoparticle shows several large meandering wrinkles. The size of Fe₃O₄ nanoparticle can be clear from TEM image . Fig (1 and 2) X-ray diffraction shown the graph all of Magnetite and Fe₃O₄ nanoparticle. Fig (3and 4) U.V shown the graph all of Magnetite and Fe₃O₄ nanoparticle respectively dispersed in chloroform.

References :

1. Mihajlović.G, Xiong. P, and von Molnár. S. Appl. Phys. Lett. **2005**, 87, 112502-1-3 .
2. Etier. M ,Shvartsman .V. V. , Gao .Y , Landers.J , Wende.H&Lupascu. D.C ,Ferroelectrics, Taylor & Francis,**2013**,[347]177-[355]185 .
3. Maiti.R , Chakraborty .M , J. Alloy and Compod. **2008** ,458 , 450–456.
4. Riyanto .A, Listiawati .D, Suharyadi .E, Abraha .d. K. ProsidingPertemuanIlmiah XXVI HFI Jateng& DIY, Purworejo ,**2012**, 14 April, 203-207.
5. Asuhan. S, Wan .H.L, Zhao. S, Deligeer .W, Wu. H.Y, Song.L, Tegus .O. Ceramics Int. **2012** ,38,6579–6584.
6. Zhoua .X, Shi.Y,Rena .L, Bao .S , Han.Y, Wu.S, Zhang.H,Zhong.L, Zhang. Q,J. Solid State Chem. **2012**,196,138–144.
7. Wang.B ,Wei .Q, Qu. S,Int. J. Electrochem. Sci., **2013**, 8, 3786 – 3793.
8. Sun.J, Zhou.S, Hou.P, Yang.Y, Weng.J,Li.X,Li.M,J. Biomed Mater Res 80A: **2007**,333–341.
9. Jiang.W, Yang.H.C, Yang. S.Y, Horng.H.E,Hung.J.C,Chene.Y.C , Hong.C.-Y.,J.Mag. Mag. Mater. **2004**,283, 210–214.
10. Hu.P, Zhang .S, Wang.H, Pan.D, Tiana.J, Tang.Z, Volinsky.A.A ,J.Alloy. Compd.**2011** ,509 , 2316–2319
11. J.O. Park, K.Y. Rhee, S.J. Park ,Appl. Surf. Sci,**2010** ,256 , 6945–6950.
12. Ur Rahman.O, Mohapatra.S. C, Ahmad. S,Mater. Chem. Phys, **2012**,132 , 196–202.
13. Mukherjee.J, Ramkumar.J ,Chandramouleeswaran. S.,Shukla.R,Tyagi. A. K.J. Radioanal.Nucl.Chem., **2013** ,22 January .
14. Miyauchi.M,Simmons.T.J, JianjunMiao,Gagner.J.E., Shriver.Z. H.,Aich.U, Dordick .J. S. and Linhardt.R. J, ACS Appl. Mater. Int. **2011**, 3, 1958–1964.

15. Wang .L.-L. , Jiang. J.-S.Nanoscale Res Lett , **2009**, 4:1439–1446 .
16. Nowosielski.R, Babilas.R ,J.Achievements in Mater. Manuf. Eng.**2011** , 48/2 , 153-160.
17. Astuti, Claudia.G., Noraida, and Ramadhani .M.,Makara J. Sci, August . **2013**, Vol. 17 ,No. 2 ,58-62 .
18. Kim.D, Lee.N, Park .M, Kim B.H., An .K and Hyeon T. J.Am.Chem .Soc,2009 ,131 ,454-455.
19. Xu.J ,Yang .H, Fu .W,Du.K, Sui.Y ,Chen.J,Zeng.Y, Li.M and Zou.G j.Mag.Mag. Mater , **2007** , 309 , 307-311.

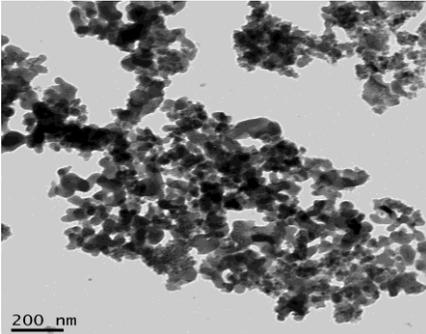


Plate1: Fe₃O₄ 200 °C

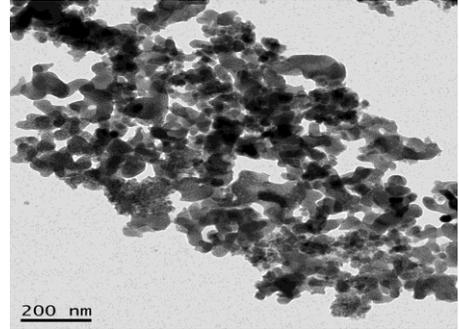


Plate2: Fe₃O₄ 200 °C

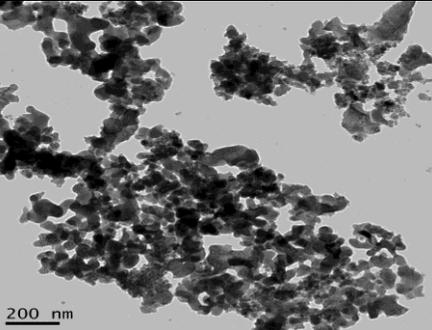


Plate3: Fe₃O₄ 200 °C

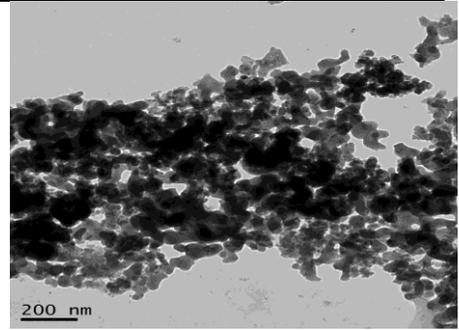


Plate4: Fe₃O₄ 200 °C

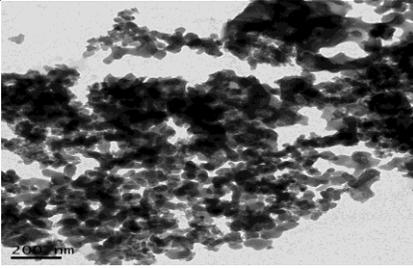


Plate 5: Fe₃O₄ 200 °C

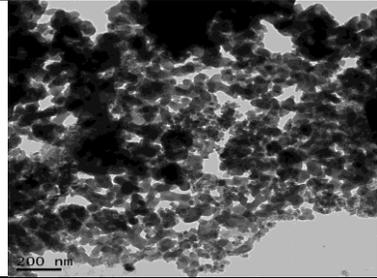


Plate 6: Fe₃O₄ 200 °C

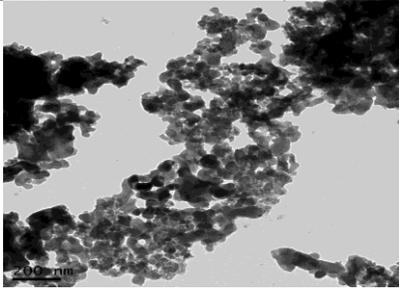


Plate 7: Fe₃O₄ 200 °C

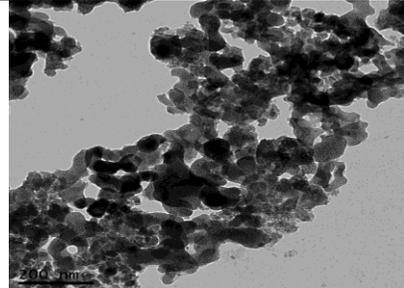


Plate 8: Fe₃O₄ 200 °C

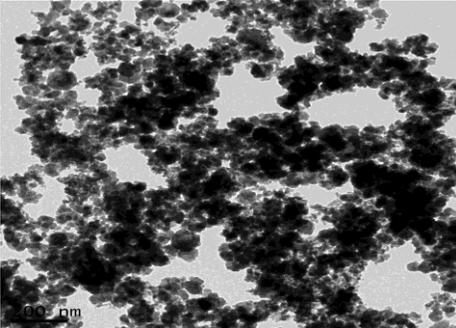


Plate 9: Fe₃O₄ 400 °C

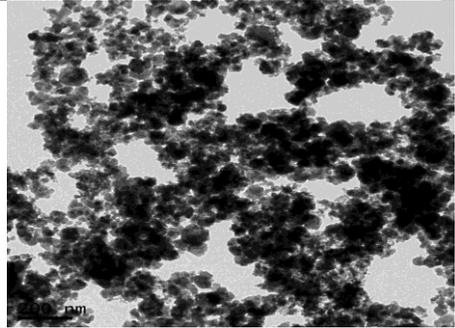


Plate 10: Fe₃O₄ 400 °C

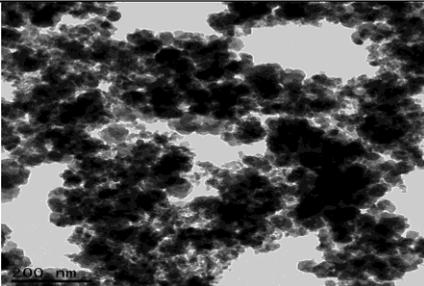


Plate 11: Fe₃O₄ 400 °C

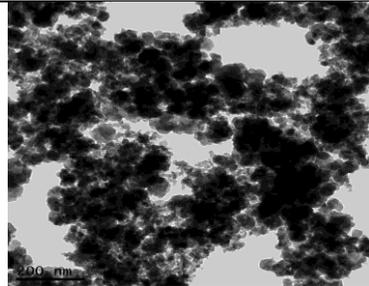


Plate 12: Fe₃O₄ 400 °C

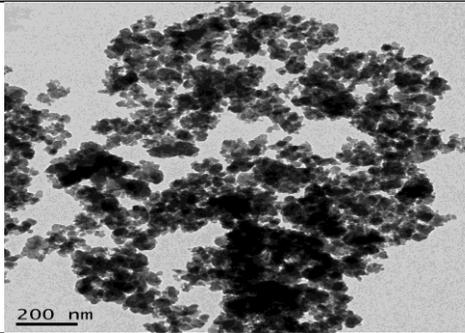


Plate 13: Fe₃O₄ 400 °C

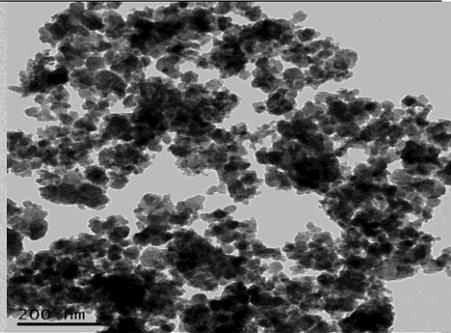


Plate 14: Fe₃O₄ 400 °C

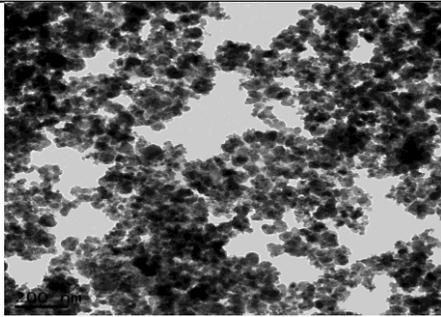


Plate 15: Fe₃O₄ 400 °C

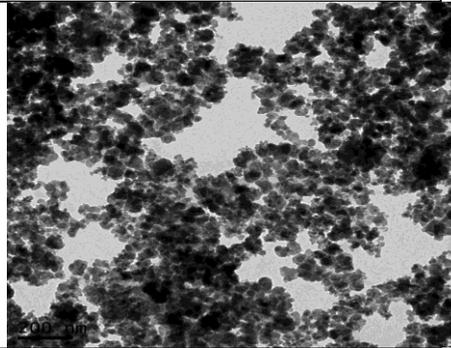


Plate 16: Fe₃O₄ 400 °C

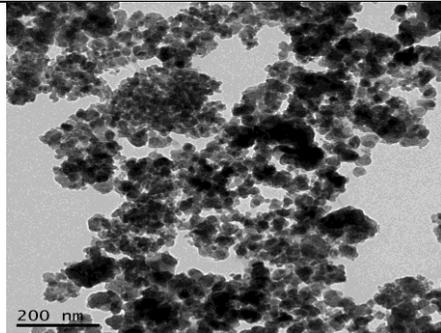


Plate 17: Fe₃O₄ 400 °C

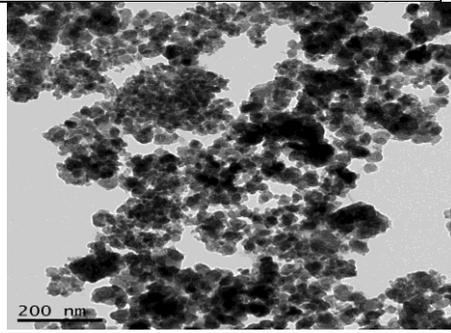


Plate 18: Fe₃O₄ 400 °C

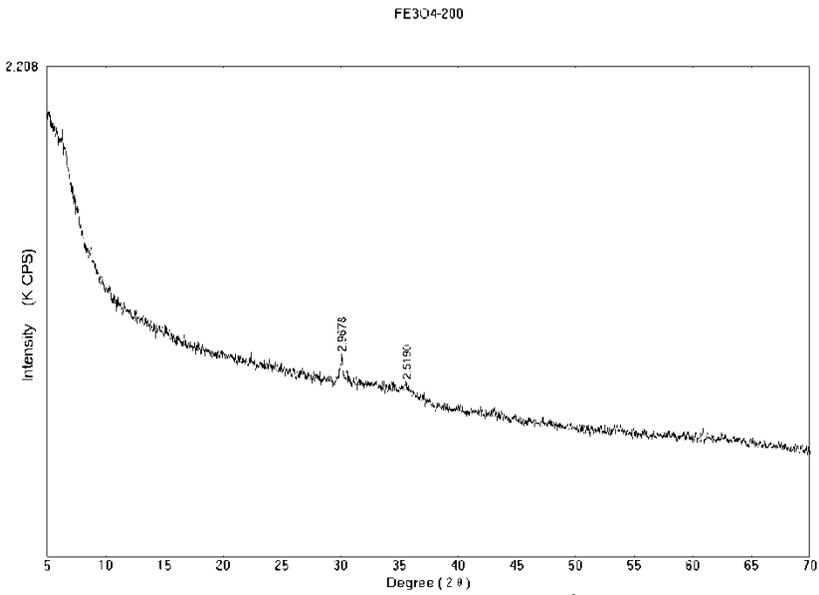


Fig .1: XRD for Fe₃O₄ Nanoparticle at 200 °C

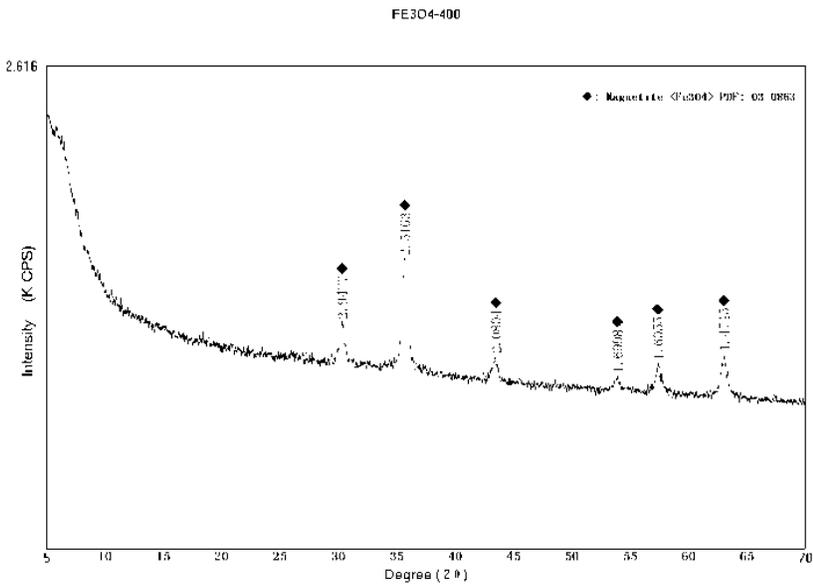


Fig .2: XRD for Fe₃O₄ Nanoparticle at 400 °C



Fig.3 : U.V of Fe₃O₄ 200 °C

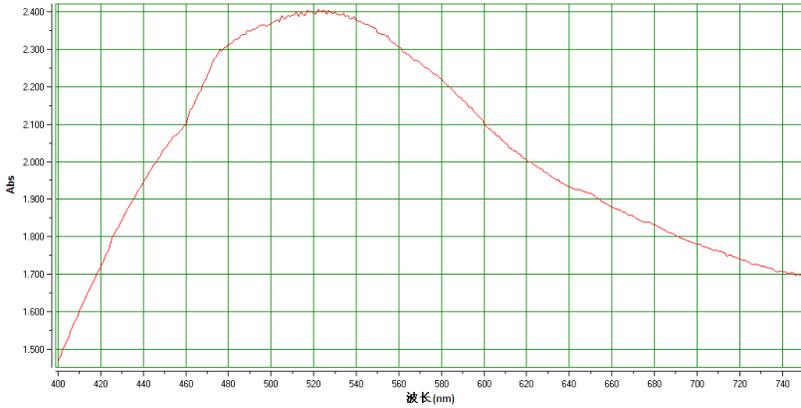


Fig. 4 : U.V of Fe₃O₄ 400 °C