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Synthesis of Magnetite Nanocubes (Fe_3O_4) from Iron (III) Acetylacetonate by Removal Gas and Higher Temperature Obtained

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ABSTRACT

Fe_3O_4 nanocubes were synthesized from Iron (III) acetylacetonate (99 % ,Across), 4- biphenylcarboxylic acid , oleic acid and benzyl ether 98 % at higher temperature with drying under vacuum. Fe_3O_4 nanocubes were obtained in powder form . The Fe_3O_4 nanocubes structures, nanoparticles size, chemical composition, and magnetic properties were characterized by TEM, U.V and XRD .

Key words: Iron (III) acetylacetonate , Fe_3O_4 nanocubes, properties and characterization.

INTRODUCTION

Recently, considerable research has been focused on iron oxides due to their potential uses in pigments, magnetic drug targeting, magnetic resonance imaging for clinical diagnosis, recording material and catalysts, etc[1-6].

The magnetic nanoparticles exhibit superparamagnetic behavior because of the infinitely small coercivity arising from the negligible energy barrier in the hysteresis of the magnetization loop of the particles as predicted .

There are many various ways to prepare Fe_3O_4 nanoparticles, which have been reported in other papers, such as arc discharge, mechanical grinding, laser ablation, microemulsions, and high temperature decomposition of organic precursors, etc[7- 10].

These methods are used to prepare magnetite nanoparticles with several controllable particle diameters. However, well-dispersed aqueous Fe_3O_4 nanoparticles have met with very limited success[11- 13].

In this paper, preparation of Fe_3O_4 nanocubes is reported by removal of the gas as well as higher temperature was used to obtain Fe_3O_4 nanocubes in powder form under oven vacuum at 80 ^0C temperature.

EXPERIMENTAL

Materials

Physical parameters of Iron (II) acetylacetonate (99 %, Across), 4- biphenylcarboxylic acid , oleic acid and Benzyl ether 98 % are reported in table 1 , 2 ,3 and 4 respectively.

Notes

Molecular sieves type 4 A 98.5% ,d = 0.69 – 0.75 heated them in oven at temperature at 400 ^0C for 2-3 hrs and then put them in 50 ml Benzyl ether 98 % in flash to remove water before starting the experimen.

Synthesis of Magnetite Nanocubes

Synthesis of ferrimagnetic nanocubes (Fe_3O_4) was carried out under nitrogen (N_2).Typical synthesis of mangntic nanocubes (0.71g,2 mmol) Iron (III) acetylacetonate (Fe (acac)$_3$ mixed with (0.41 g,2.1 mmol) 4-biphenylacarboxylic a cid added to mixture (1.129 g , 4 mmol) oleic acid and (10.40 g ,10 ml) benzyl ether . The mixture solution was degassed at room temperature for 1 hour .The solution was then heated to 290 ^0C at the rate of 20 ^0C /min with vigorous magnetic stirring at 290 rpm to get ferrimagnetic nanocubes. where the temperature was held for 30 min when temperature reached 290 ^0C . After cooling the solution to room temperature , a mixture of (40 ml) toluene and (10 ml) hexane was added to solution . The solution was then centrifuged at 5000 rpm for minutes to precipitate the magnetite nanocubes .The precipitate was washed using (10 ml) chloroform ($CHCl_3$) . Then after that used oven vacuum to obtain Fe_3O_4 nanocubes in powder form at 80 ^0C temperature[14- 18].

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.

UV Results

For UV results, a small amount of sample in test tube and then was dissolved in 3mL ethanol or chloroform ($CHCl_3$) into the sample and the solution was stirred by ultra-sonication to make sure the sample was uniform . Then solution was transferred to cavity of spectrophotometer to get the test. Spectra were recorded at 400 to 750 nm.

RESULTS AND DISCUSSION

Plate 1,2,3 ,4,5,6,7 and 8 (TEM) shows the top-view TEM images of the Fe3O4 nanocubes plate (TEM). The surface of Fe3O4 nanocubes shows several large meandering wrinkles. The size of Fe3O4 nanocubes about (between 39.62 –

Table 1: General Characteristics of Iron (III) acetylacetonate (99 %, Across)

Trade Name	**Iron (III) acetylacetonate , 99 %**
Appearance	Red powder
Molecular weight	353.17
content	25 G R
Company	ACROS ,Organics ,U.S.A

Table 2: General characteristics of 4- biphenylcarboxylic acid 97 %

Trade Name	**4- biphenylcarboxylic a cid, 97 %**
Appearance	White powder
Molecular weight	198.22
density	1.185
Company	Adamas-beta Reagent Co,Ltd, China

Table 3: General characteristics of oleic acid

Trade Name	**Oleic A cid ($C_{18}H_{34}O_2$) 99.9 %**
Appearance	Liquid
Molecular weight	282.46
Density (20 0C g/m)	0.870 - 0.90
pH (250 g /l ,25 0C	3.0 - 5.0
Company	Sinopharm Chemical Reagent Co,Ltd, China

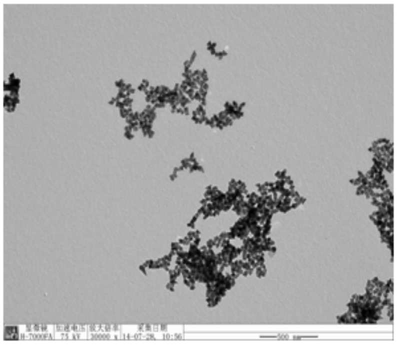

Plate 1: TEM of magnetite nanocubes

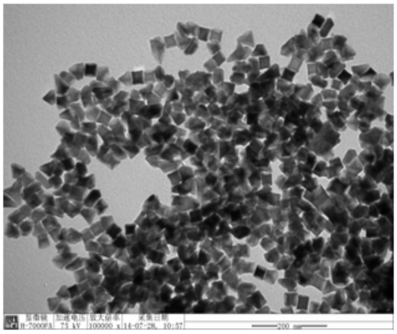

Plate 2: TEM of magnetite nanocubes

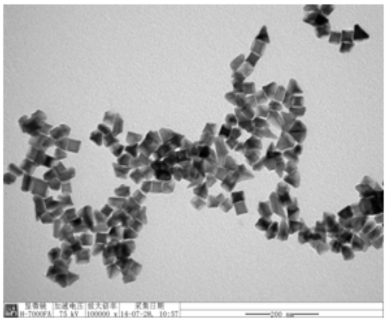

Plate 3: TEM of magnetite nanocubes

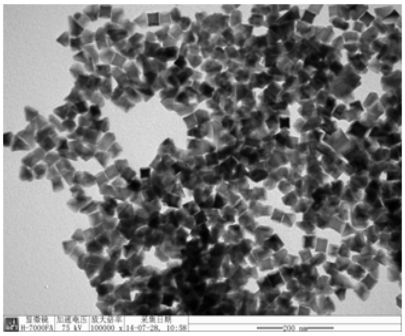

Plate 4: TEM of magnetite nanocubes

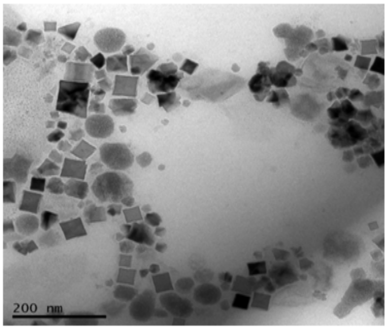

Plate 5: TEM of magnetite nanocubes

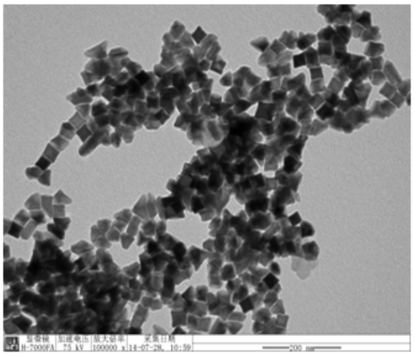

Plate 6: TEM of magnetite nanocubes

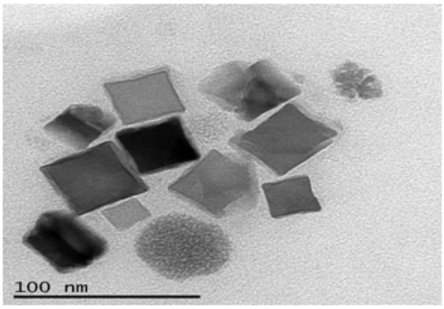

Plate 7: TEM of magnetite nanocubes

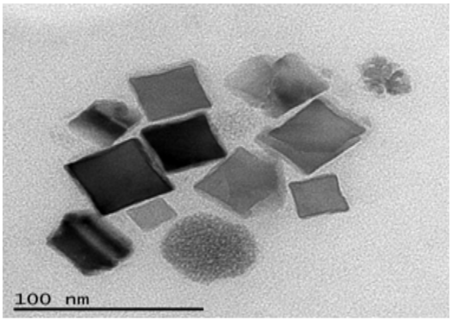

Plate 8: TEM of magnetite nanocubes

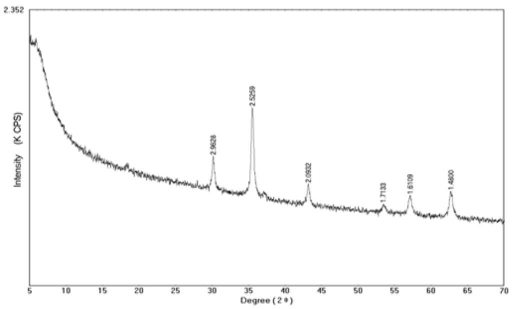

Fig. 1: XRD for magnetite nanocubes

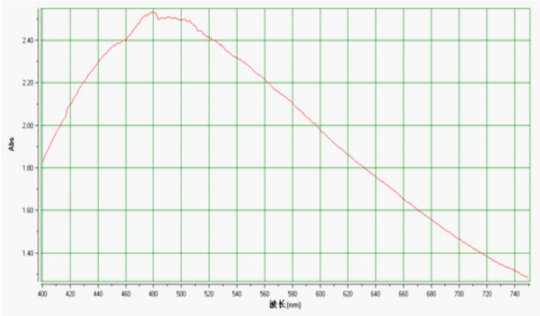

Fig. 2: U.V for magnetite nanocubes

Table 4: General characteristics of benzyl ether 98 %

Trade Name	Benzyl ether ($C_{14}H_{14}O_1$), 98 %
Appearance	Liquid
Molecular weight	198.26
Density (25 ^{0}C g/m)	1.043 g / ml at 25 ^{0}C
pH (250 g /l ,25 ^{0}C	3.0 – 5.0
Melting point	1.5 – 3.5 ^{0}C
Boling point	298 ^{0}C
Company	Al-drich Chemistry

48.35 nm) is clear from TEM image . Fig.1. X-ray diffraction showed the graph all of Magnetite Fe_3O_4 nanocubes. Fig .2. U.V shown the graph all of Fe_3O_4 nanocubes respectively dispersed in ethanol or chloroform .

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