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Review Article

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Synthesis and Characterization of Superparamagnetic (Fe_3O_4) Magnetite Nanoparticles Coated with Oleic Acid

Nabil Abdullah Noman Alkadasi

Department of Chemistry, Rada'a, Albaydha University, Yemen
Department of Civil and Mechanical Engineering, Thamar University, Yemen

Abstract Fe_3O_4 nanoparticles have been successfully synthesized by modification of the preparation method of Fe_3O_4 nano-particles, which were prepared from Iron (III) Chloride hexahydrate $FeCl_3.6H_2O$, Ferrous Chloride $FeCl_2.4H_2O$ and Ammonia Solution (NH_4OH), oleic acid. In this study, a new preparation of Fe_3O_4nanoparticles and Fe_3O_4nanoparticles coated with oleic acid were reported. The characterization of Fe_3O_4 nanoparticle and Fe_3O_4 nanoparticles coated with oleic acid is done by TEM , XRD and UV spectroscopy.

Keywords Fe3O_4, Nanoparticle and characterization.

Introduction

Magnetic iron oxide nanoparticles and their dispersion in various media have been scientific and technologic [1-3]. Owing to their unique properties in a magnetic field they are actively used in various industrial and medical application. The magnetic fluids based on magnetite and mineral oils or organic solvents are conventionally synthesized by alkaline hydrolysis of ferrous and ferric salts. The magnetite obtained is stabilized by surfactants.

Oleic acid is often used as a surfactant to form a waterproof shell around the magnetite particles since oleic acid has higher affinity to the surface of superfine magnetite compared to other surfactants .The treatment of magnetite by oleic acid is the most complex and important stage of the magnetic magnetite fluid preparation [4-8].

In conventional techniques, to prepare magnetic fluids, excess base (NH_4OH) is used to form magnetite precipitates and then oleic acid is added as surfactant by forming oleate directly after the complete crystallization of the magnetite precipitate. The main procedure begins with the co-precipitation of Fe (II) and Fe (III) ions with addition of excess concentrated NH_4OH .The sediment is then isolated by magnetic decantation and treated with oleic acid at heating. In finally an organic carrier liquid is added under intensive stirring. To obtain concentrated magnetic fluids, treated procedure such as phase separation and extraction of excess surfactant and solvent are often needed. In this paper, Fe_3O_4 nanoparticles are perpetrated in higher concentrated hydrophobic magnetite by adjusting the amount of ammonium hydroxide and oleic acid, and time of oleic acid addition. The key to the success of making such a hydrophobic magnetite is to add an appropriate amount of ammonium hydroxide and oleic acid. Finally solution has remained neutral and the magnetite precipitated. The oleic acid, as a reactant, was added immediately after the formation of magnetite crystal, simultaneously with the crystal growth. We posited that the oleic acid will efficiently coat the Fe_3O_4 crystal at the growth stage and will be create a highly concentrated hydrophobic magnetite precipitated [9-13].

We characterized the magnetite precipitated in terms of its morphology ,particle size , magnetite properties , structure , and hydrophobicity /hyrophilicity by transmission electron microscopy (TEM), ultraviolet (U.V) and powder X- ray diffraction) XRD .

Experimental Work

Materials

Iron (III) Chloride hexahydrate $FeCl_3.6H_2O$, 99.0 %, Ferrous Chloride $FeCl_2.4H_2O$, 98.0 %, Ammonia Solution (NH_4OH) and oleic acid were purchased from Sinopharm chemical reagent Co, Ltd, China. Physical parameters of Iron (III) Chloride hexahydrate $FeCl_3.6H_2O$, 99.0 %, Ferrous Chloride $FeCl_2.4H_2O$, 98.0 %, Ammonia Solution (NH_4OH), oleic acid and Hydrochloric acid (HCl) are reported in table 1, 2, 3, 4 and 5 respectively.

Table 1: General Characteristics of Iron (III) Chloride hexahydrate $FeCl_3.6H_2O$

Molecular formula	Iron (III) Chloride hexahydrate $FeCl_3.6H_2O$, 99.0 %
Appearance	Yellow- red crystal
Molecular weight	270.29
Company	Sinopharm chemical reagent Co, Ltd, China

Table 2: General Characteristics of Ferrous Chloride $FeCl_2.4H_2O$,98.0%

Molecular formula	Ferrous Chloride $FeCl_2.4H_2O$, 98.0 %
Appearance	Green Crystall
Molecular weight	198.82
Company	Sinopharm chemical reagent Co, Ltd, China

Table 3: General Characteristics of Ammonia (NH_4OH)

Molecular formula	Ammonia (NH_4OH)
Appearance	liquid
Molecular weight	17.03
Concentration	25-28 %
Company	Sinopharm chemical reagent Co, Ltd, China

Table 4: General characteristics of oleic acid

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

Table 5: General Characteristics of Hydrochloric acid (HCl)

Molecular formula	Hydrochloric Acid (HCl)
Appearance	liquid
Molecular weight	36 .5
Concentration	36-38 %
Company	Sinopharm chemical reagent Co ,Ltd ,China

Synthesis of oleic acid-coated magnetite

The magnetite was synthesized by modification method in the following procedure (14-19): 5.8 g $FeCl_3.6H_2O$ and 2.2 g $FeCl_2.4H_2O$ were dissolved in 200 ml deionized water under nitrogen gas with vigorous stirring at 90 °C for 30 minutes. 7.5 ml of 25 % NH_4OH was added to the solution. Then 4.9 mL oleic acid was added dropwise into the suspension. After 30 minutes, the upper solution became colorless and the tarlike black magnetite gel precipitated and was isolated by magnetic decantation. The magnetite was prepared by the same recipe except for omission of oleic acid. The black Magnetite gel precipitated was collected and washed with de-ionized water and pure ethanol three times or acetone to remove excess oleic acid. After drying, a black powder was obtained.

Figure 1: Solution of Fe_3O_4 Nanoparticles

Transmission Electron Microscope (TEM) Test

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

Results and Discussion

The Fe_3O_4 nanoparticle was synthesized by heating to 90 °C of Fe_3O_4 and Fe_3O_4 nanoparticle coated oleic acid in powder form. Plates 1 to 13 (TEM) show the top-view TEM images of the Fe_3O_4 Nanoparticle and Fe_3O_4 nanoparticle coated oleic acid. X-ray diffraction spectra shows of Magnetite nanoparticles and Fe_3O_4 nanoparticle coated oleic acid (figure 2 & 3). UV spectra shows of Magnetite nanoparticles and Fe_3O_4 nanoparticle coated oleic acid respectively dispersed in ethanol (figure 4 & 5).

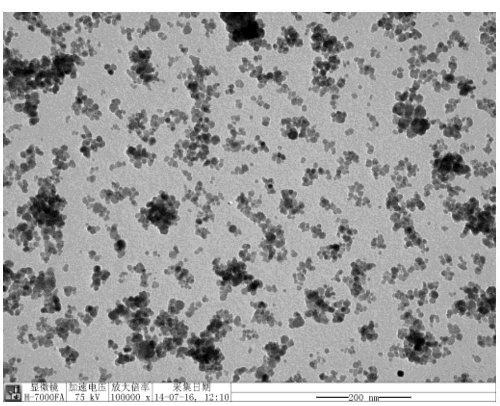

Plate 1: TEM of Fe_3O_4 nanoparticles

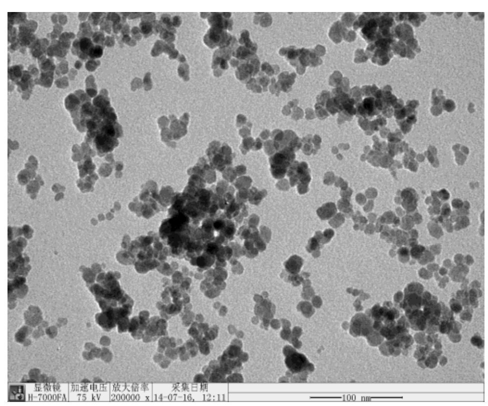

Plate 2: TEM of Fe_3O_4 nanoparticles

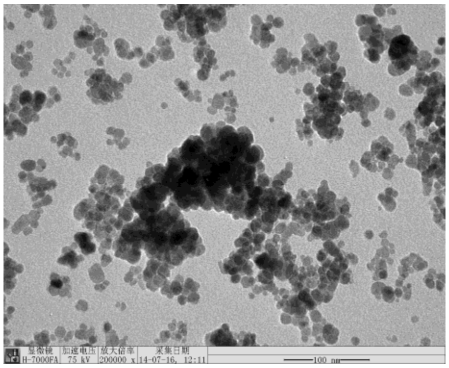

Plate 3: TEM of Fe_3O_4 nanoparticles

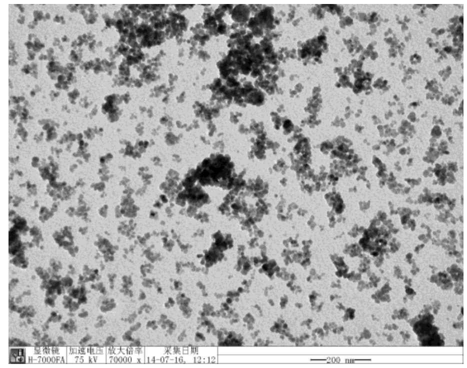

Plate 4: TEM of Fe_3O_4 nanoparticles

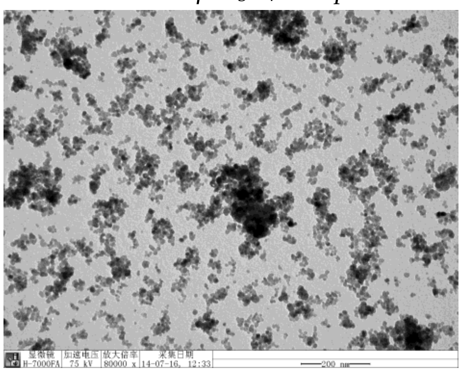

Plate 5: TEM of Fe_3O_4 nanoparticles

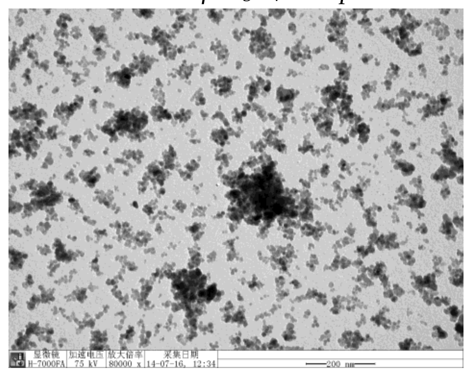

Plate 6: TEM of Fe_3O_4 nanoparticles

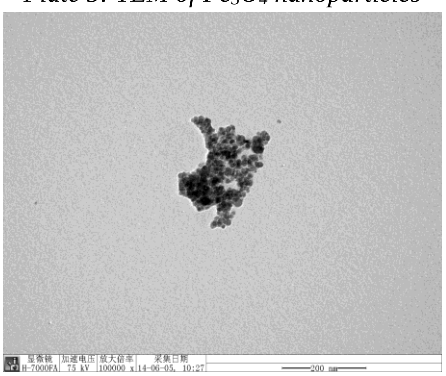

Plate 7: TEM of Fe_3O_4 nanoparticles

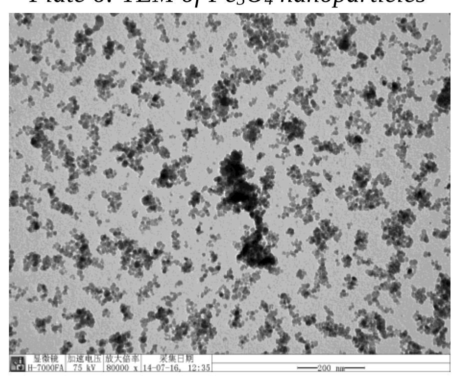

Plate 8: TEM of Fe_3O_4 nanoparticles

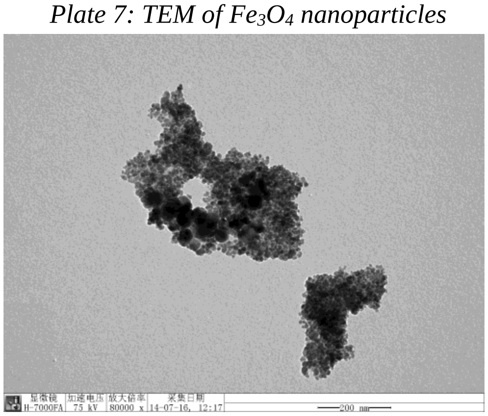

Plate 9: TEM of Oleic acid coating Fe_3O_4 nanoparticles

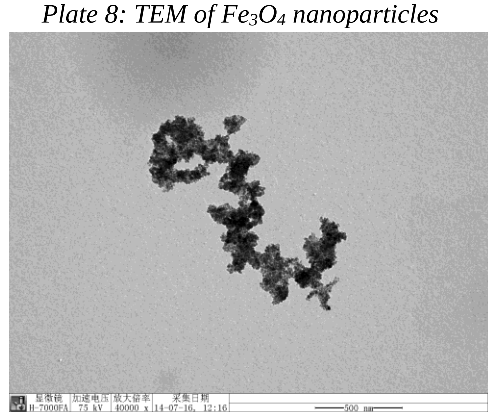

Plate 10: TEM of Oleic acid coating Fe_3O_4 nanoparticles

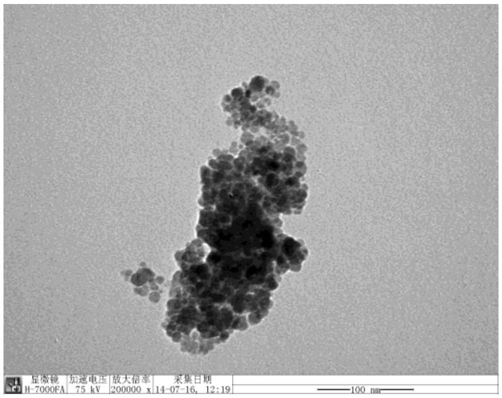

Plate 11: TEM of Oleic acid coating Fe_3O_4 nanoparticles

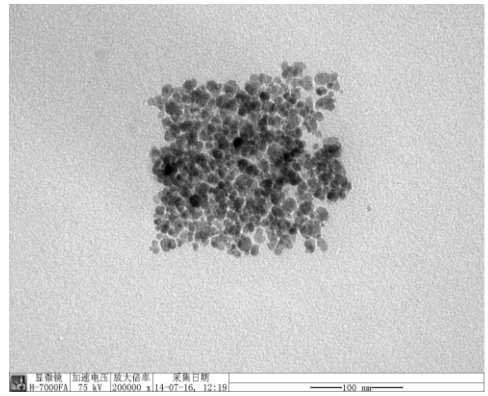

Plate 12: TEM of Oleic acid coating Fe_3O_4 nanoparticles

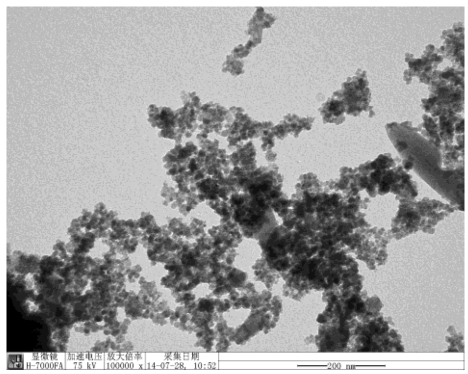

Plate 13 . TEM of Oleic acid coating Fe_3O_4 nanoparticles

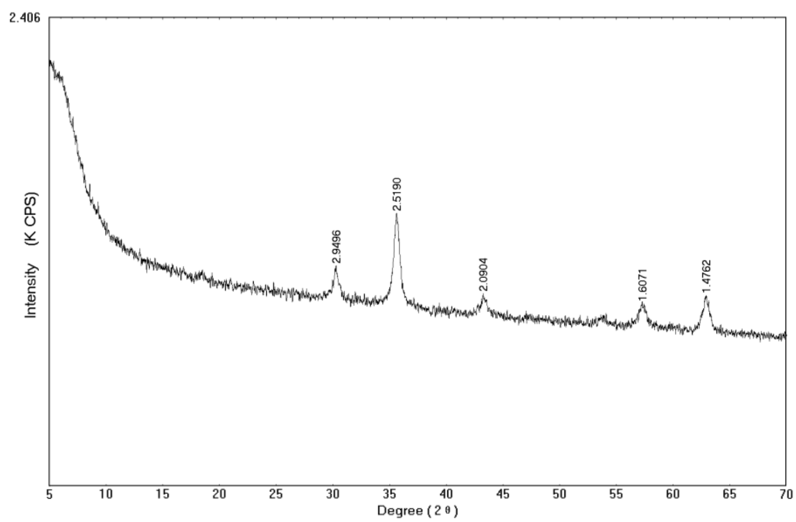

Figure 2: XRD Spectra for Fe_3O_4 Magnetite (Fe_3O_4) Nanoparticles

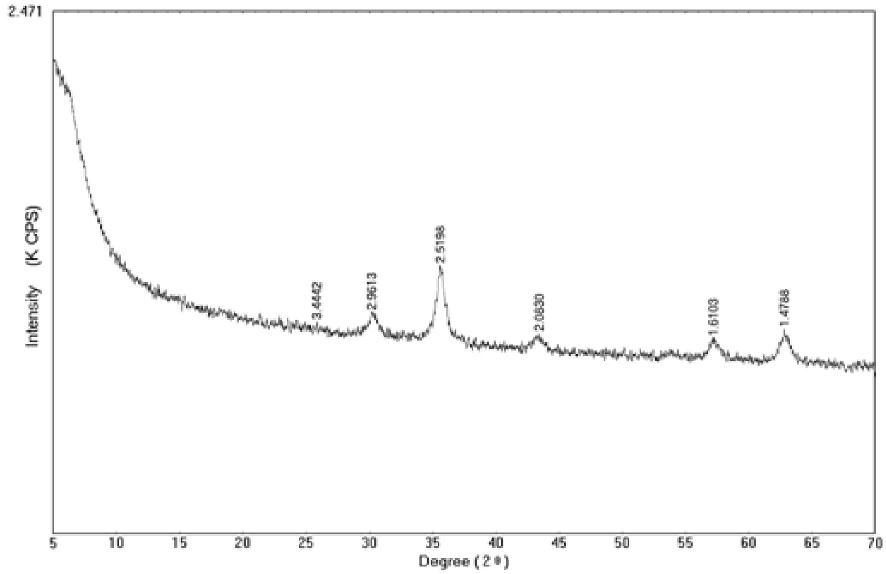

Figure 3: XRD Spectra for Oleic acid coating Magnetite (Fe_3O_4) nanoparticles

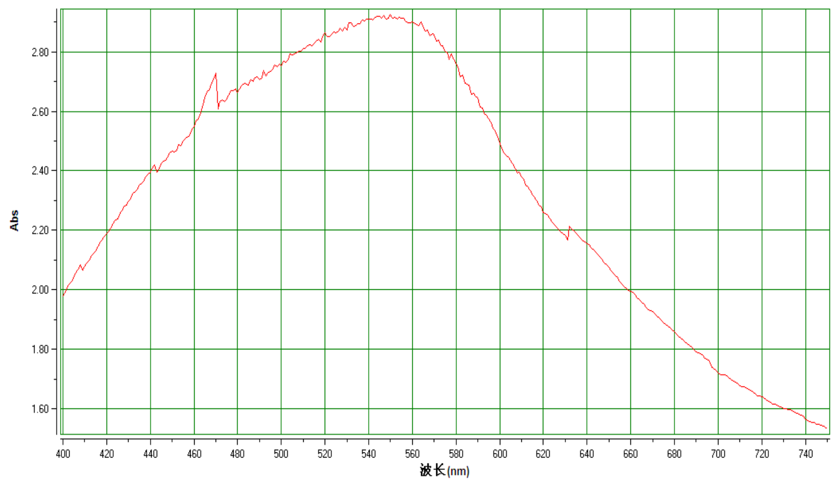

Figure 4: UV Spectra for Magnetite (Fe_3O_4) nanoparticles

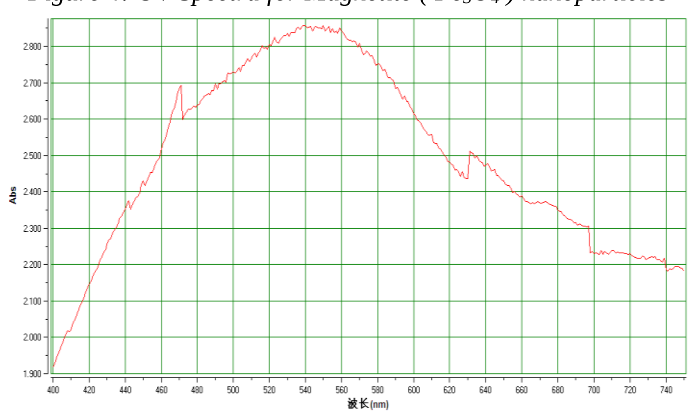

Figure 5: UV Spectra for Oleic acid coating Magnetite (Fe_3O_4) nanoparticles

Acknowledgements

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