



Synthesis of Fe_3O_4 Nanoparticles from Ironstone from The Republic of Yemen

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ABSTRACT

In this study, a new preparation of Fe_3O_4 nanoparticles is reported. Fe_3O_4 nanoparticles were successfully synthesized. This method consisted of two stages, beginning with the pulverization and separation of iron ore from ironstone by using the coprecipitation method of magnetite. The characterization of Fe_3O_4 nanoparticles was done by TEM, XRD and UV.

Key words: Magnetite, Fe_3O_4 nanoparticles and properties and characterization.

INTRODUCTION

Recently, a considerable amount of research focused on iron oxides, due to their potential uses in pigments, drug delivery and resonance imaging for clinical diagnosis, recording material and catalyst, etc¹⁻³. 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 predicted⁴. There are many 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. These methods are used to prepare magnetic nanoparticles with controlled

diameters. However, well-dispersed aqueous Fe_3O_4 nanoparticles have been met with very limited success. Several methods have been published for synthesizing Fe_3O_4 nanoparticles, and several research studies have reported the successful preparation of nano- or microscale Fe_3O_4 . Using different methods, such as the ultrasonic chemical coprecipitation method and the solvothermal method²⁻⁶ have been reported the synthesis of nanoparticles Fe_3O_4 in organic solvent, and Copper⁷ successfully fabricated magnetic Fe_3O_4 covered with a modifiable phospholipid coating. Of these methods, chemical coprecipitation was reported to be the most promising because of its simplicity and productivity⁸⁻¹⁰. The physics of nanoscale magnetic materials has been a vivid subject for researchers within the last few decades and the exploration of

iron sand from beaches or rivers to prepare magnetic materials on nanoscale has been reported in some studies¹¹. In this paper, magnetic materials from ironstone mining in Pasaman Barat West Sumatera were investigated, and it was found that ironstone in that area contained 12.462 ppm of iron (Fe), with a susceptibility magnetic value of 888.81×10^{-8} m³/kg by using an atomic absorption spectrophotometer and magnetic susceptibility meter. For these reasons, these materials have the potential to be developed and cultivated as raw materials for magnetite (Fe₃O₄). Although there have been many significant developments in the synthesis of magnetic nanoparticles, the stability of these particles without agglomeration or precipitation is an important issue. It began with the crushing of ironstone into powder form and then synthesizing Fe₃O₄ nanoparticles by using the coprecipitation method of magnetite ore .

EXPERIMENTAL

Materials

Hydrochloric Acid (HCl) and Ammonia Solution (NH₄OH) were purchased from Sinopharm chemical reagent Co ,Ltd ,China,and ironstone was obtained from Republic of Yemen.

Physical parameters of Hydrochloric Acid (HCl), Ammonia Solution (NH₄OH) and Fe₃O₄ powder are reported in table 1 , 2 and 3 respectively.

Experiment

Two steps of preparing samples have been reported here . In the first step ironstone was pulverized to obtain a powder . Then a permanent magnet was used to obtain the iron ore.In the second step the iron ore powders were prepared by the chemical coprecipitation method.

In typical coprecipitation synthesis procedure , 10 g Fe₃O₄ powder and 20 ml HCl were mixed and heated at 90 °C for one hour . The solutions were filtered and then 25 ml NH₄OH (90%) was added to the filtrate .The black precipitate was collected and washed with de-ionized water and pure ethanol three times.

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

The Fe₃O₄ nanoparticle was synthesized by heating to 90 °C of Fe₃O₄ powder . plate 1,2,3 ,4,5,6and 7 (TEM) 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

Table 1: 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

Table 2: General Characteristics of Ammonia Solution (NH₄OH)

Molecular formula	Ammonia (NH ₄ OH)
Appearance	liquid
Molecular weight	17.03
Concentration	25 – 28 %
Company	Sinopharm chemical reagent Co, Ltd, China

Table 3: General Characteristics of Fe₃O₄ powder

Molecular formula	Magnetite oxide powder (Fe ₂ O ₃)
Appearance	Brown powder
Fe ₃ O ₄ %	45.31-75
Chorite	37.73-13
Riebeckite	16.95-15
Country	Al-Baida'a ,Yemen.



Photo.1 : Fe_3O_4 Rock Fe_3O_4 in powder form



Photo 2 : Equipment Fe_3O_4 Nanoparticle (after dry)

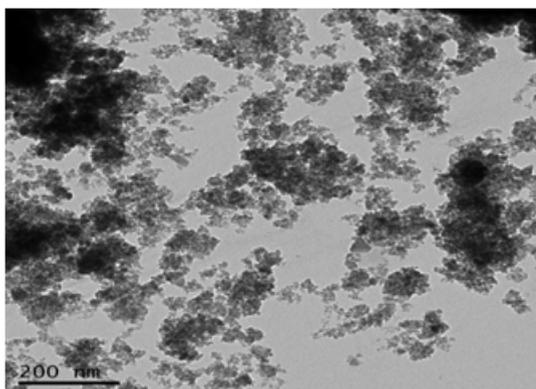


Plate 1: Fe_3O_4 nanoparticle

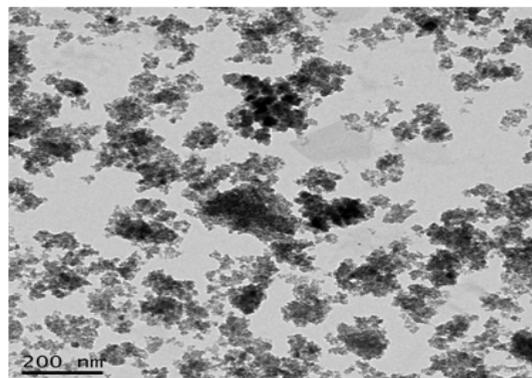


Plate 2: Fe_3O_4 nanoparticle

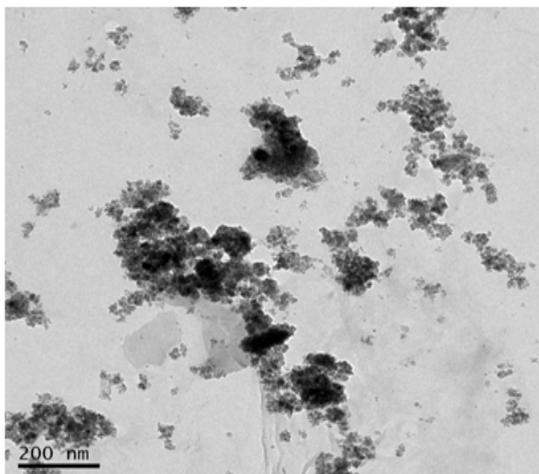


Plate 3: Fe₃O₄ nanoparticle

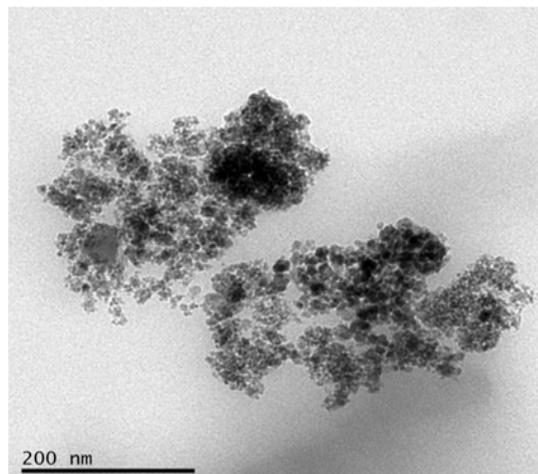


Plate 4: Fe₃O₄ nanoparticle

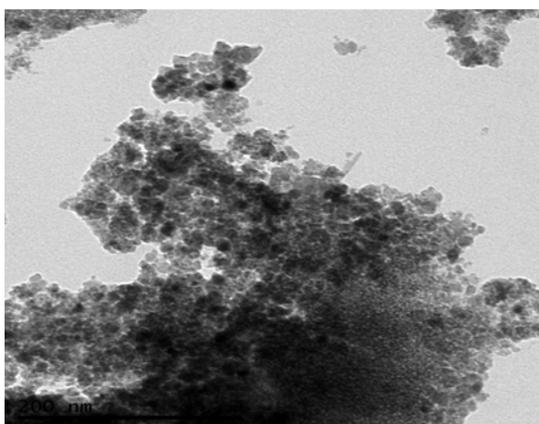


Plate 5: Fe₃O₄ nanoparticle

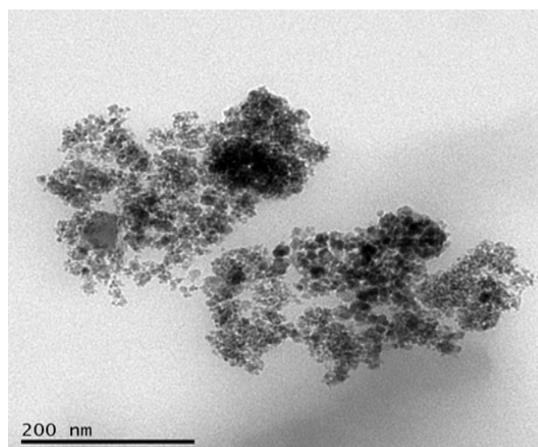


Plate 6: Fe₃O₄ nanoparticle

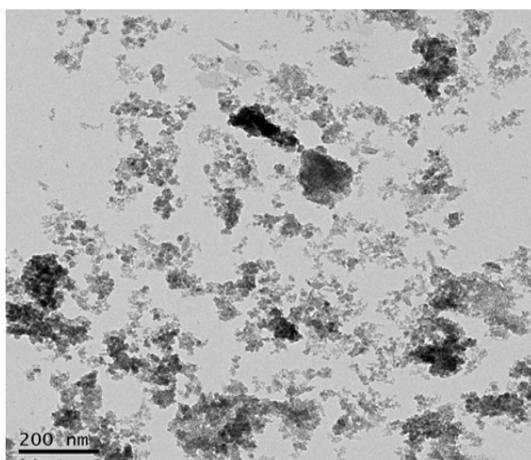


Plate 7: Fe₃O₄ nanoparticle

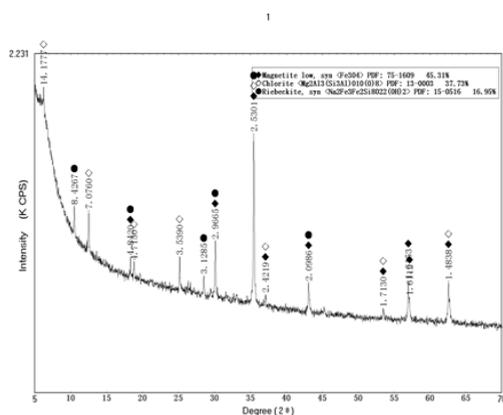


Fig. 1: XRD for Fe₃O₄ Iron Powder

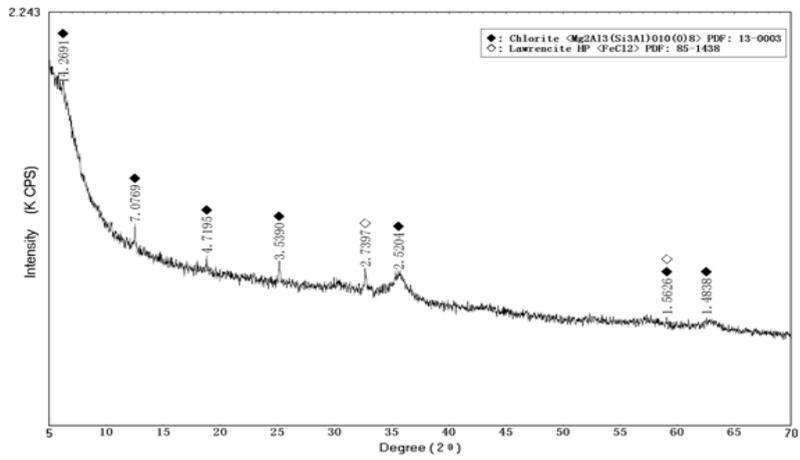


Fig. 2: Fe₃O₄ nanoparticle

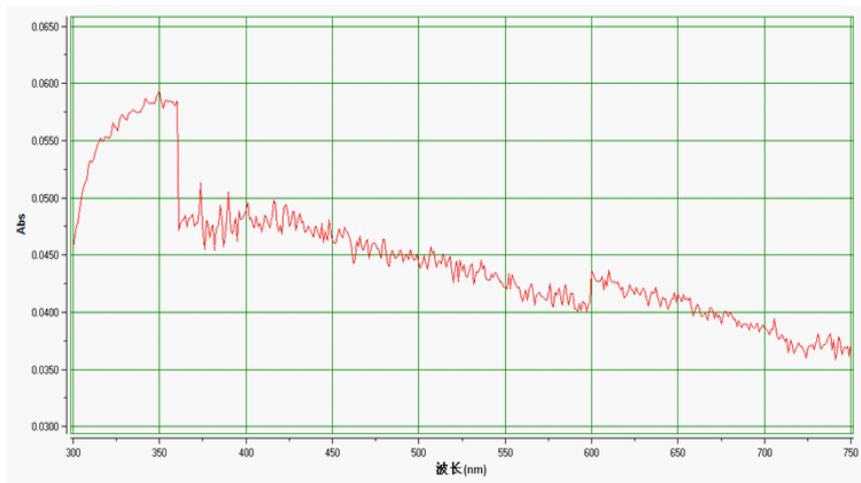


Fig. 3: Magnetite

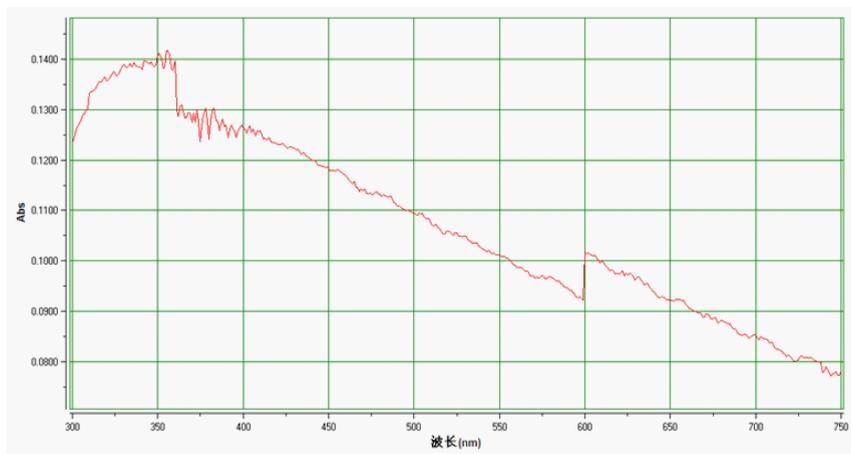


Fig. 4: Fe₃O₄ nanoparticle

nanoparticle can be clear from TEM image . Fig (1and 2) X-ray differaction shown the graph all of Magnitite and Fe₃O₄ nanoparticle. Fig (3and 4)

U.V shown the graph all of Magnitite and Fe₃O₄ nanoparticle respectively dispersed in chloroform.

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