Synthesis of Fe_2O_3 Nanoparticles From Limonite Stone the Republic of Yemen

حضرت الجسيمات النانوية لأكسيد الحديديك "Fe_2O_3" من صخر الليمونايت من الجمهورية اليمنية

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Abstract :

Fe_2O_3 nanoparticles have been successfully synthesized and the modification of the preparation method of Fe_2O_3 nano-particles, is reported here which consists of two stages, beginning with the destruction and separation of iron ore from ironstone. Then, the Fe_2O_3 nano-particles are synthesized using the coprecipitation method with iron oxide (Fe_2O_3).

It began with the destruction of ironstone and the separation of iron ore from purity stone. Fe_2O_3 nanoparticles were synthesized by

using the coprecipitation method. The characterization of Fe_2O_3 nanoparticle was done by SEM ,TEM ,XRD and U.V.

Keywords: limonite , Fe_2O_3 nanoparticles, properties and characterization.

الملخص :

حضرت الجسيمات النانوية لأكسيد الحديديك بنجاح، والتعديل في طريقة التحضير لجسيمات النانوية لأكسيد الحديديك "Fe_2O_3" سجلت هنا في هذه الورقة وتحتوي على مرحلتين في البداية الطحن والفصل للحديد الخام من صخر الليمونايت، وبعدها تحضير جسيمات النانوية لأكسيد الحديديك "Fe_2O_3" باستخدام طريقة ثنائية الترسيب لاكسيد الحديديك "Fe_2O_3" في البداية الطحن والفصل من الحديد الخام النقي وتحضير جسيمات "Fe_2O_3" بواسطة استخدام طريقة الترسيب الثنائي.

والتحاليل للجسيمات النانوية لأكسيد الحديديك "Fe_2O_3" عملت بواسطة أجهزة "TEM, XRD and U.V" .

كلمات المفتاحية:

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الليمونايت ، جسيمات النانوية لأكسيد الحديديك " Fe_2O_3 "، الخواص والتحاليل.

Introduction :

Limonite is an iron ore consisting of a mixture of hydrated iron(III) oxide-hydroxides in composition. The generic formula is as $FeO(OH)\cdot nH_2O$, although this is not entirely accurate as the ratio of oxide to hydroxide can vary quite widely. Limonite is one of the two principal iron ores, the other being hematite, and has been mined for the production of iron.

Limonite is bog iron ore in meadows and marshes. In its brown form it is sometimes called brown hematite or brown iron ore. In its bright yellow form it is sometimes called lemon rock or yellow iron ore. A single mineral, limonite is now recognized as a mixture of related hydrated iron oxide minerals, among them goethite, akaganeite,lepidocrocite, and jarosite. Individual minerals in limonite may form crystals, but limonite does not, although specimens may show a fibrous or microcrystalline structure,[7] and limonite often occurs in concretionary forms or in compact and earthy masses; sometimes mammillary, botryoidal, reniform or stalactite.

Limonite usually forms from the hydration of hematite and magnetite, from the oxidation and hydration of iron rich sulfide minerals, and chemical weathering of other iron rich minerals such as olivine, pyroxene, amphibole, and biotitic. It is often the major iron component in lateritic soils. It is often deposited in run-off streams from mining operations.

One of the first uses limonite was as a pigment. The yellow form produced yellow ochre for which Cyprus was famous,[8] while the darker forms produced more earthy tones. Roasting the limonite changed it partially to hematite, producing red ochers, burnt umbers and siennas[9] .

Bog iron ore and limonite mudstones are mined as a source of iron.

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Iron caps or gossans of siliceous iron oxide typically form as the result of intensive oxidation of sulfide ore deposits[10] .These gossans are used by prospectors as guides to buried ore. In addition the oxidation of those sulfide deposits which contain gold, often result in the concentration of gold in the iron oxide and quartz of the gossans.

In this paper Fe_2O_3 Nanoparticles were synthesized by heating at 90 ^{0}C for

30 minutes to obtain brown solution followed by drying for 24 hours at 60-80 ^{0}C in oven under vacuum to obtain Fe_2O_3 powder .

Experiment :

Materials:

Hydrochloric Acid (HCl) and Ammonia solution (NH_4OH) were purchased from Sinopharm chemical reagent Co ,Ltd ,China, Fe_2O_3 powder from a rocky mountain in the Republic of Yemen.

Physical parameters of Hydrochloric Acid (HCl) , Ammonia solution (NH_4OH) and Fe_2O_3 powder are reported in table 1 ,2 and 3 respectively.

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

Table 1. General Characteristics of Hydrochloric Acid (HCl)

Molecular formula	Ammonia (NH4OH)
Appearance	liquid
Molecular weight	17.03
Concentration	25 – 28 %
Company	Sinopharm chemical reagent Co ,Ltd ,China

Table 2. General Characteristics of Ammonia (NH4OH)

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Molecular formula	Limonite oxide powder (2 Fe2O3.H2O)
Appearance	powder
Fe2O3 %	40-78
CaCO3 %	60-21
Country	Yemen.

Table 3. General Characteristics of Limonite oxide powder

Limonite in powder form Photo. 1. Limonite Rock

Experiment :

The first step in the physical preparation method ,in which 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 precipitation method, the Fe_2O_3 properties were studied here in this paper [11-17].

In typical precipitation synthesis procedure, 5g Fe_2O_3 powder and 10 ml HCl were mixed at 90 ^{0}C for at least one hour . The solutions were filtered and then 12.5 ml NH_4OH solution

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(90%) was added to the filtrate .The brown precipitates were collected and washed with deionized water and pure ethanol three times .

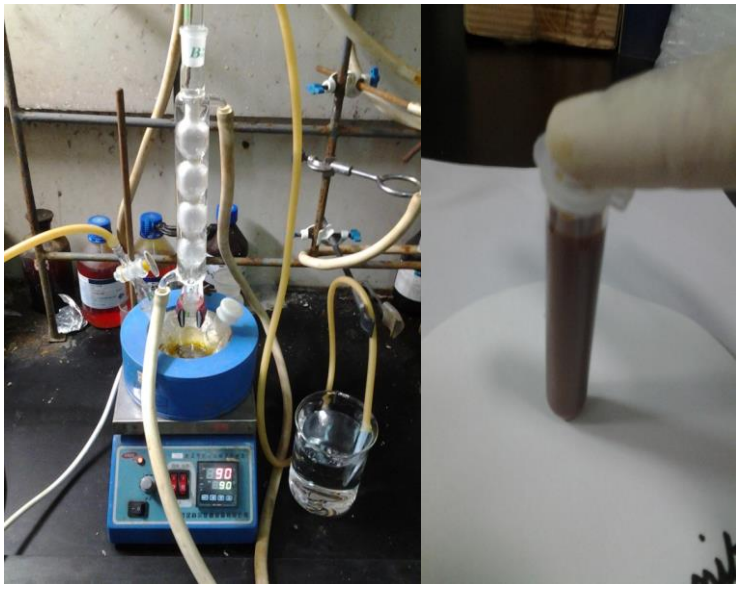

Photo. 2. Apparatus for preparation of Fe2O3 Nanoparticles from limonite stone

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SEM Test :

silicon wafer is cut into (3mm x 3mm) used Piranha solution a mixture consisting of sulfuric acid (H_2SO_4) and hydrogen peroxide (H_2O_2) to clean silicon wafer . It is typically mixed in concentration ratios of around 3:1 H_2SO_4:H_2O_2 heated one hour at 60 ^{0}C temperature . Then followed by triple rinsing in ethanol with ultrasonic cleaning for 30 min then with nitrogen (N_2) . Main applications of piranha solution are to remove organic contaminants from surface of the wafer during cleaning . Then Silicon wafer was used for SEM [18].

TEM Tests :

For TEM Test , a small amount of sample was dissolved in test tube and 3mL of chloroform or ethanol was added to it and the solution was stirred by ultra-sonication to make sure the sample was dissolved . Then 10 µ L solutions were transferred to clean Copper Grid and kept for drying for TEM test.

UV Results :

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

XRD Results :

For XRD results, a small amount of powder sample was put in the XRD machine connected with computer . The result was record between intensity (KCPS) and Degree (2ɵ) shown in the figures (1 & 2) .

Results and Discussion :

The Fe_2O_3 nanoparticles were synthesized by heating to 90 ^{0}C of Fe_2O_3 powder obtain Fe_2O_3 nanoparticles . Plate 1

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,2,3 ,4 and 5 (SEM) and plate 6,7,8,9,10,11,12,13 and 14 . The surface of Fe_2O_3 nanoparticle shows several. The thickness of Fe_2O_3 nanoparticle can be clear in high-magnification TEM image . Fig (1and 2) X-ray diffraction shows the graph all of limonite Stone and Fe_2O_3 nanoparticle. Fig (3and 4) U.V shows the graph all of limonite Stone and Fe_2O_3 nanoparticle respectively dispersed in chloroform ($CHCl_3$) or ethanol .

Conclusions :

In conclusion, I modified the synthesis method for Fe_2O_3 nanoparticles through using the coprecipitation method with iron oxide (Fe_2O_3).

Main application of Fe_2O_3 nanoparticles are in electrical material. The surface of Fe_2O_3 can be seen from the SEM image.

Acknowledgements :

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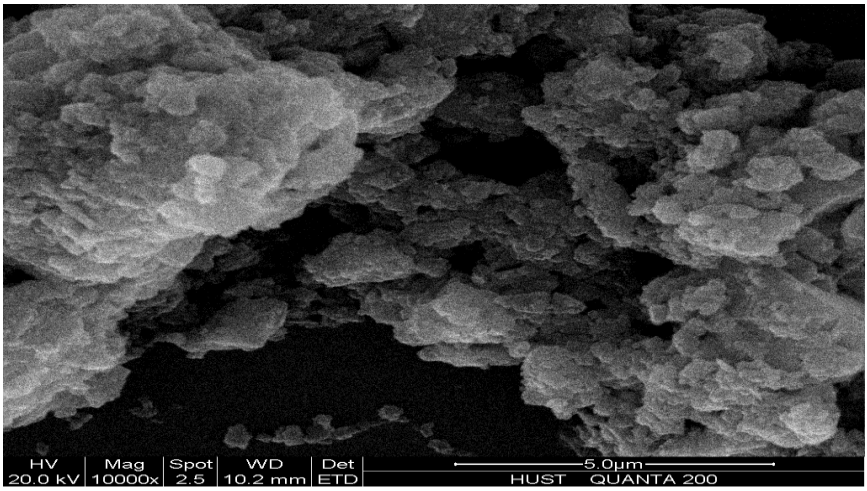

Plate (SEM) 1 : Limonite Rock

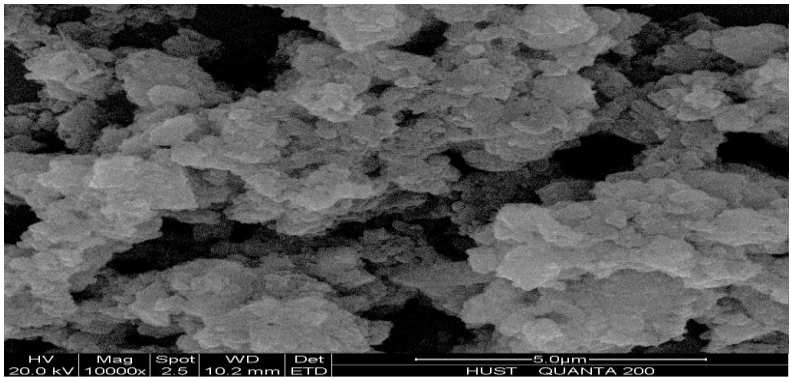

Plate (SEM) 2: Limonite Rock

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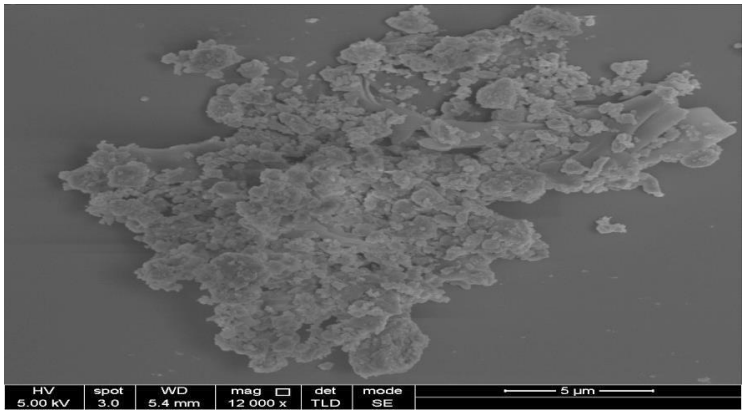

Plate (SEM) 3: Limonite Rock

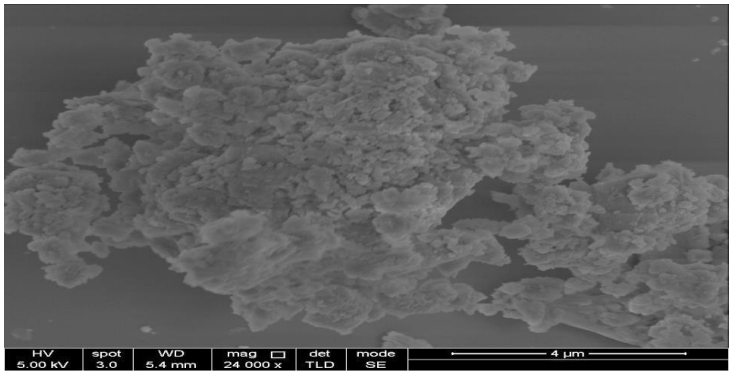

Plate (SEM) 4: Limonite Rock

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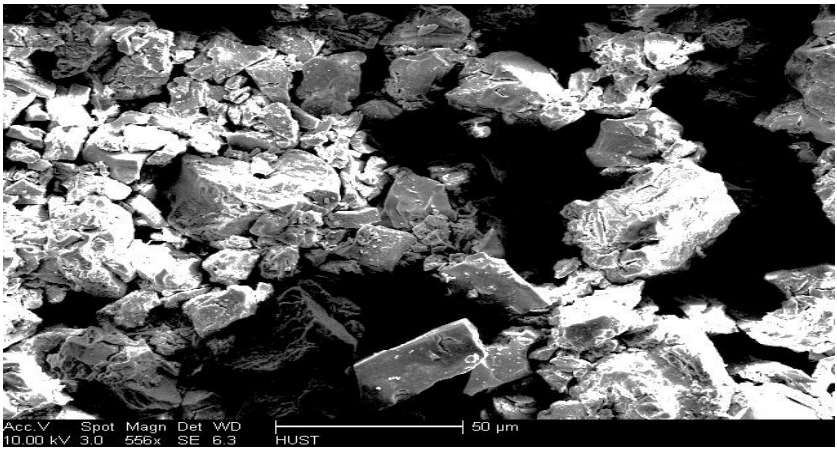

Plate (SEM) 5: Fe2O3 nanoparticles

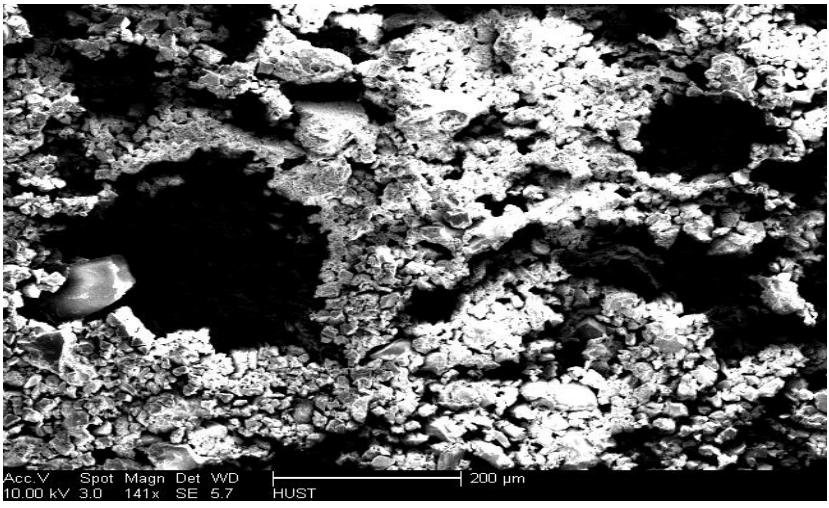

Plate (SEM) 6: Fe2O3 nanoparticles

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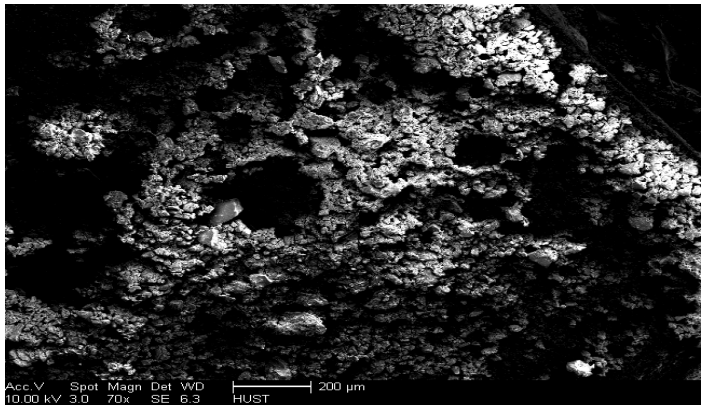

Plate (SEM) 7: Fe2O3 nanoparticles

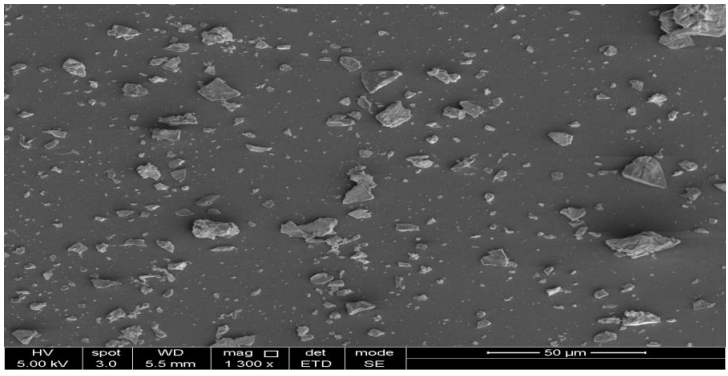

Plate (SEM) 8: Fe2O3 nanoparticles

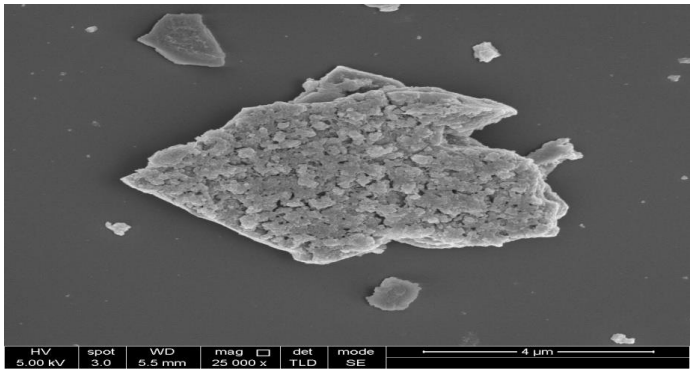

Plate (SEM) 9: Fe2O3 nanoparticles

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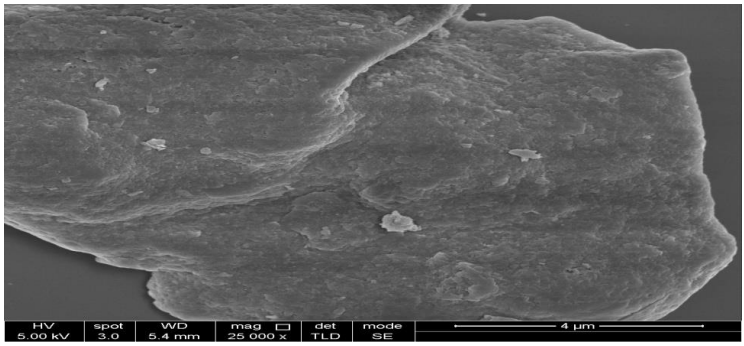

Plate (SEM) 10 : Fe2O3 nanoparticles

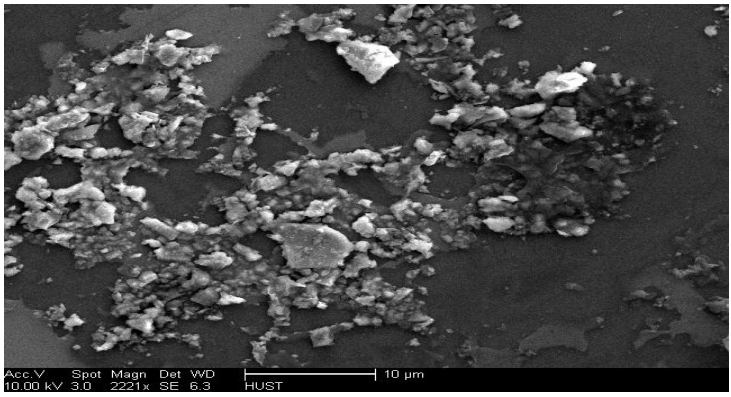

Plate (SEM) 11: Fe2O3 nanoparticles

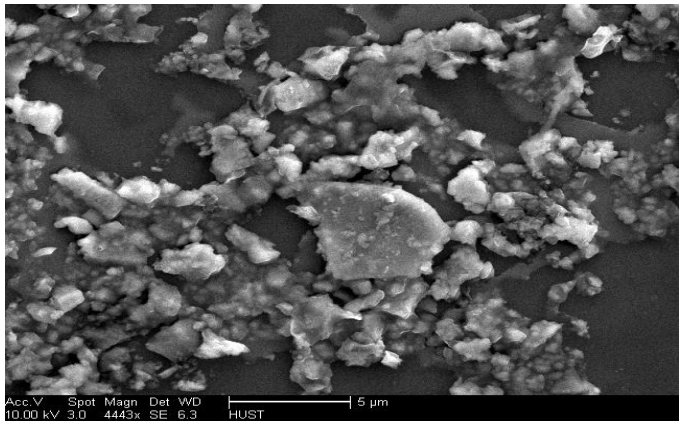

Plate (SEM) 12 : Fe2O3 nanoparticles

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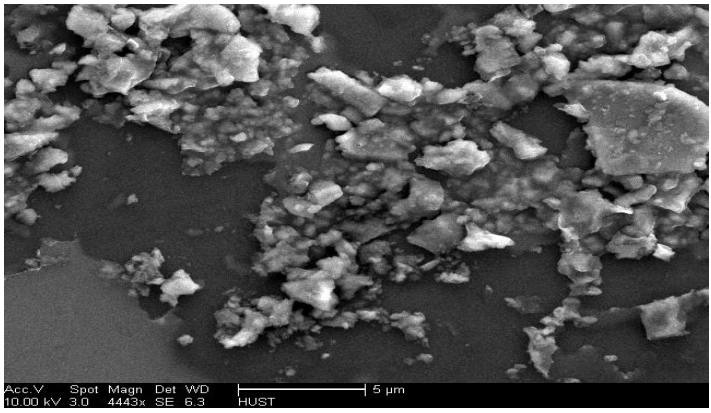

Plate (SEM) 13 : Fe2O3 nanoparticles

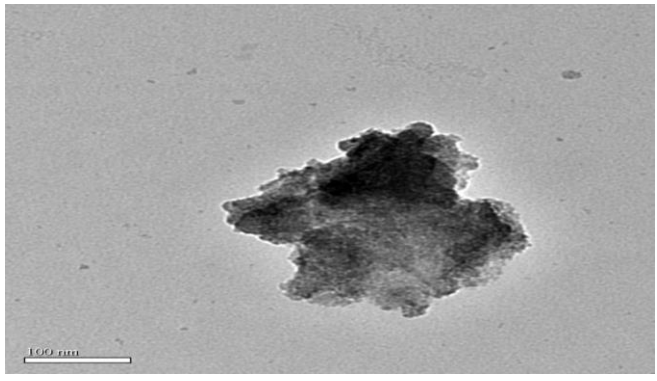

Plate (TEM) 14 : Fe2O3 nanoparticles

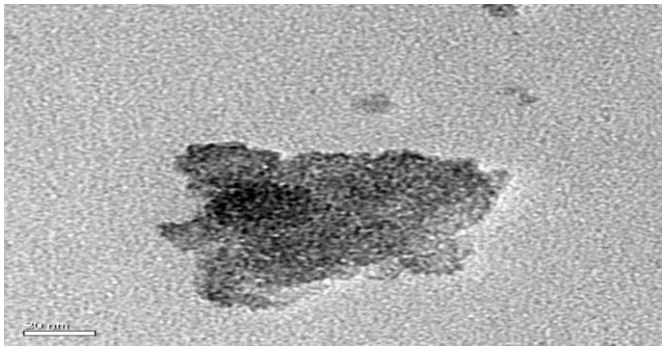

Plate (TEM) 15 . Fe2O3 nanoparticles

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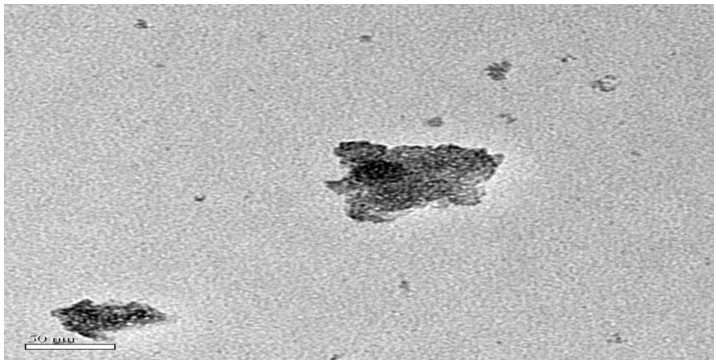

Plate (TEM) 16: Fe2O3 nanoparticles

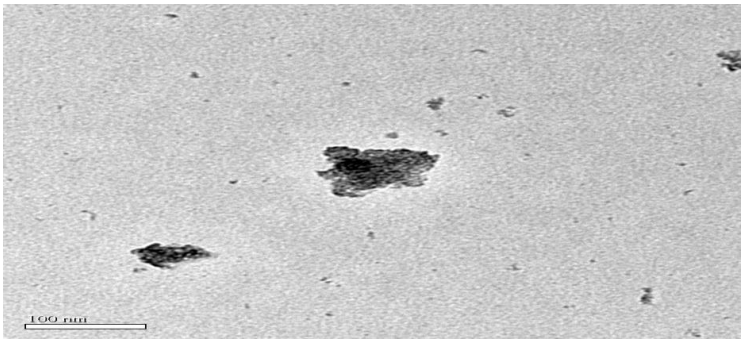

Plate (TEM) 17: Fe2O3 nanoparticles

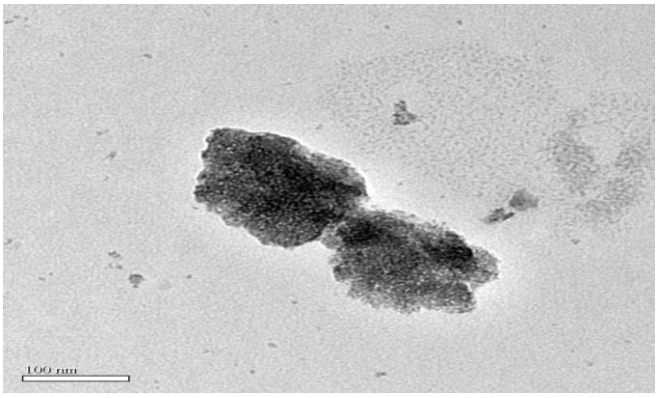

Plate (TEM) 18: Fe2O3 nanoparticles

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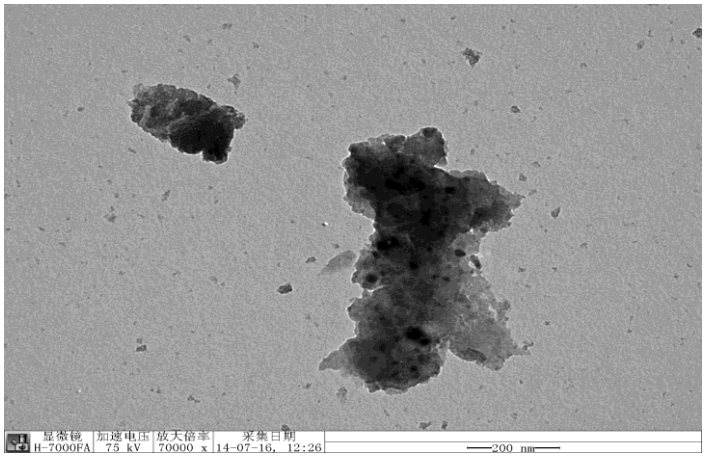

Plate (TEM) 19 : Fe2O3 nanoparticles

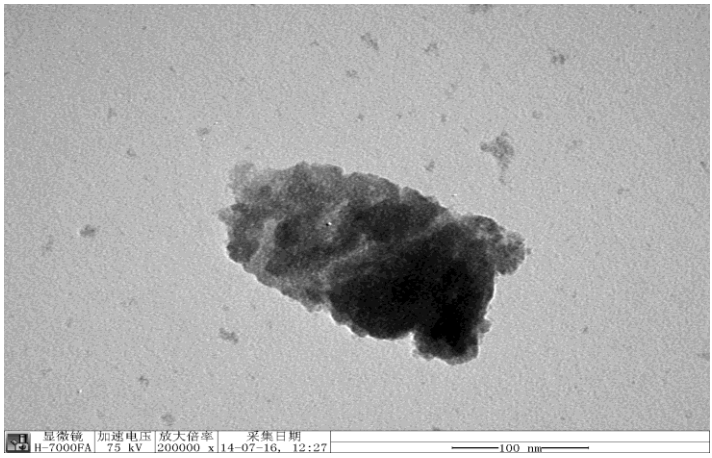

Plate (TEM) 20 : Fe2O3 nanoparticles

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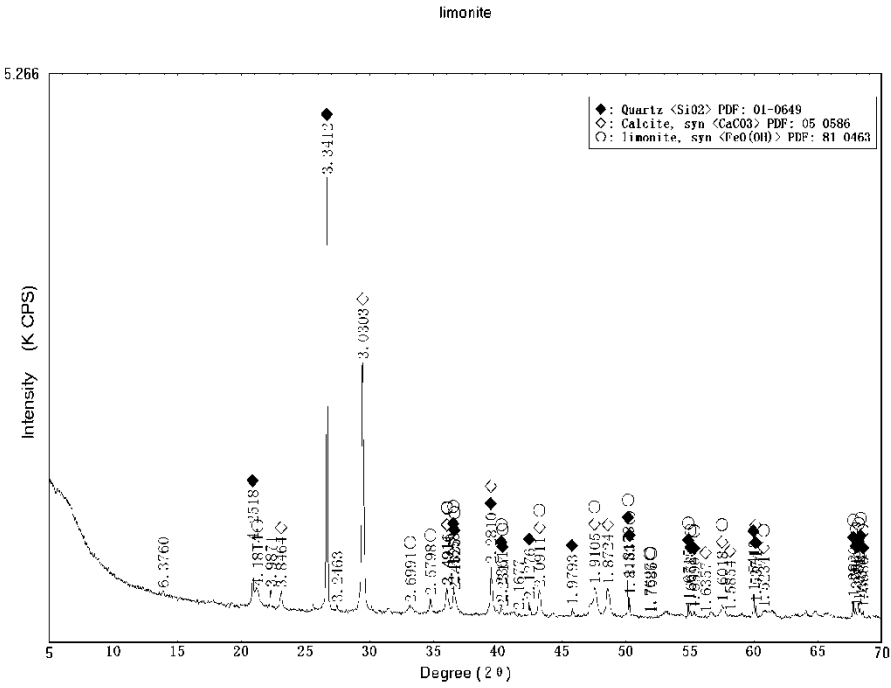

Fig. 1 : XRD for Limonite Iron Powder

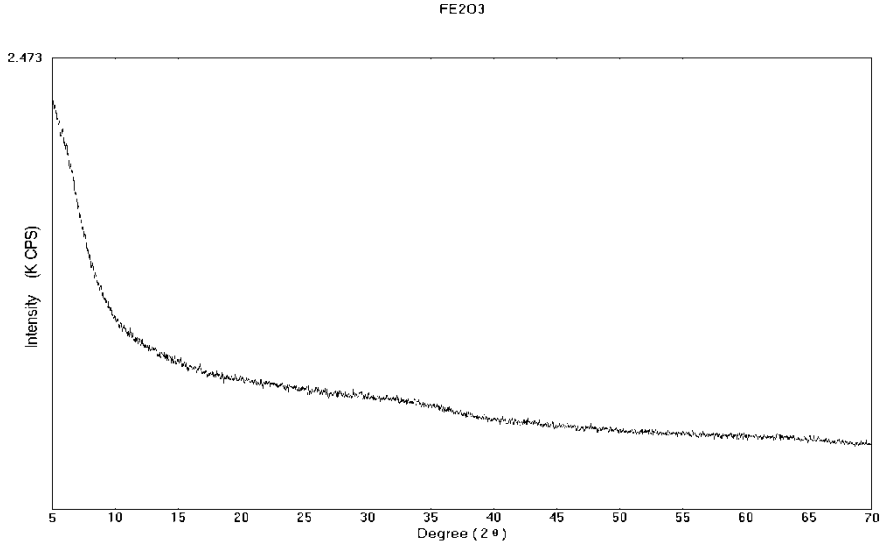

Fig. 2 : XRD for Fe2O3 nanoparticles

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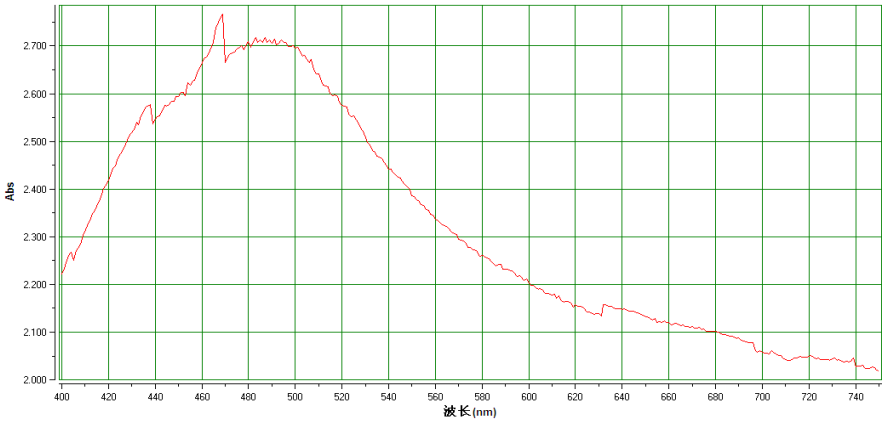

Fig. 3: U.V for Limonite stone

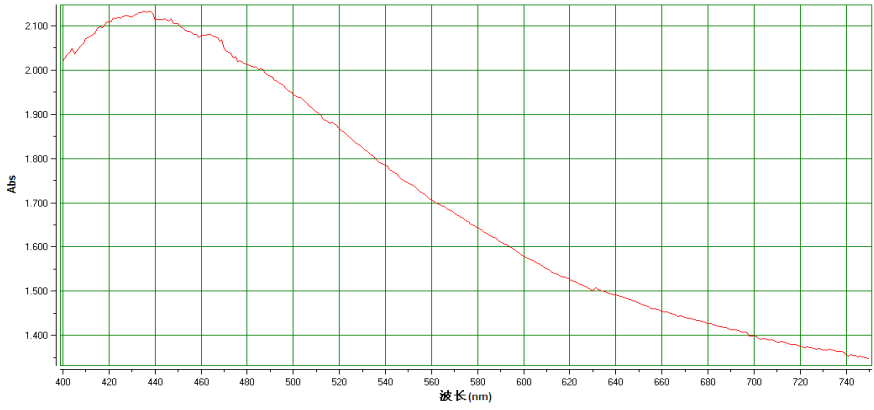

Fig .4: U.V for Fe2O3 nanoparticles