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Synthesis and Characterization of Iron Oxide Nanoparticles by Open Vessel Ageing Process

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Abstract

Nano-crystalline iron oxide nanoparticles (magnetite) was synthesized by open vessel ageing process. The iron chloride solution was prepared by mixing deionized water and iron chloride tetrahydrate. The product was characterized by X-Ray, Surface area and pore volume by Brunauer-Emmet-Teller, Atomic Force Microscope (AFM) and Fourier Transform Infrared Spectroscopy(FTIR) . The results showed that the XRD in compatibility of the prepared iron oxide (magnetite) with the general structure of standard iron oxide, and in Fourier Transform Infrared Spectroscopy, it is strong crests in 586 bands, because of the expansion vibration manner related to the metal oxygen absorption band (Fe–O bonds in the crystals of iron oxide). The results show that the prepared nano iron oxide is with average crystal size 75.92 nm, surface area was 85.97 m^2/g and the pore volume was found equal to 0.1566 cm^3/g.

Keywords: Nanomaterial; Magnetite; Iron oxide; (characterization; Iron (II) chloride) tetrahydrate.

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1- Introduction

Nano-technology has been considered as one of the most important recent advancements in science and technology. Nano-particles are one of the important building blocks in the fabrication and development of nano-materials [1]. As a consequence, the nanoparticle has drawn a huge interest from researchers globally due to specific characteristics such as shape, size, and distribution, which could be utilized in a distinct field of applications. Nanoparticles iron oxide has an essential role in many chemical, physical and materials science [2].

Among nanoparticles, iron magnetic nanoparticles have gained more interest due to their abundance, rapid reaction, superparamagnetic , high competence , non-toxic, enhanced stability and efficiency in chemical and physical adsorption of organic and inorganic pollutants including heavy metals from polluted waters [3]**.** These unique properties allow Fe_3O_4-NPs to be widely used in different areas of applications, such as catalysis [4], magnetic storage media [5], environmental treatment [6], magnetic resonance imaging (MRI)[7] , and targeted drug delivery [8]. Magnetic properties of nanoparticles magnetic can be fitted by their size distributions and particle sizes. The particle sizes and size distributions of nanoparticles magnetic are successively, affected by the synthesis path.

For these points, different synthesis methods have been advanced to make iron oxide nanoparticles in order to obtain desired properties [9],which have been reported in other papers, gas-phase deposition and mechanical techniques (Physical methods) [10] ,green synthesis (biological method) [11], co-precipitation method [12], microwave assisted synthesis [13], (chemical methods). The chemical method is the most common for preparing Fe_3O_4 nanoparticles. Chemical preparation methods, relatively less energy were consumed compared with that of physical methods. The size and morphology of the nanoparticles can be controlled by selectively choosing the reaction media, the physical parameters of the reaction, such as precursors, reactant concentration, base (NaOH and ammonium hydroxide), temperature, pH . Biological method represents an advantageous manufacturing technology with respect to high yield, good, as well as low costs and low energy input, but the fermentation process is rather time-consuming [14].

In other work presents the synthesis of multi shapes of Fe_3O_4 nanoparticles like spherical , plate, and nano flowers by chemical method by solve thermal method assisted by microwave radiation, by using FeSO4·(NH4)2SO4·6H2O as iron precursor, , ethanol and NaOH [15]. This work presents the synthesis and characterization of Fe_3O_4 nanoparticles from raw materials, cheap and available by iron (II) chloride tetrahydrate and sodium hydroxide by open vessel ageing process.

2- Experimental Work

2.1. Materials

Every component used for the preparation of Fe_3O_4 were analytical grade and used without further purification.

Iron (II) chloride tetrahydrate(98%), sodium hydroxide [NaOH](98%) were purchased from Sigma, Germany.

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2.2. The Procedure of Fe_3O_4 Synthesis

The preparation process of Fe_3O_4 including the following steps:

a. 1L of a 30 mM solution of $FeCl_2$ was prepared from deionized water and $FeCl_2.4H_2O$.
b. The solution was then titrated with sodium hydroxide solution at a rate about 1mL/min. The solution kept on constant mixing to attain a well-mixed blend.
c. Then the Fe(OH)x was put in the Teflon container and heated in a programmable electrical furnace with maximum temperature.
d. The particles were heated at constant temperature of 100ºC for 60 min and consequently cooled to room temperature.
e. The product obtained was filtered using Buckner funnel with the aid of a vacuum pump and washed twice with deionized water and then dried in an electrical oven for 24 hours at 100 °C.

$$FeCl_2+2NaOH \longrightarrow Fe(OH)_2+2NaCl$$

$$3Fe(OH)_2 \longrightarrow Fe_3O_4+2H_2O+H_2$$

Fig. 1 shows schematic diagram of preparation procedure of magnetite. On the other hand

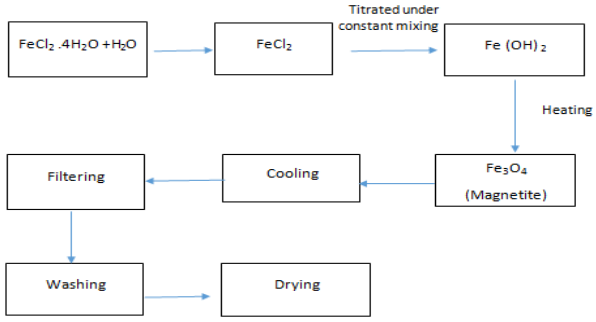

Fig. 1. The schematic diagram of preparation method

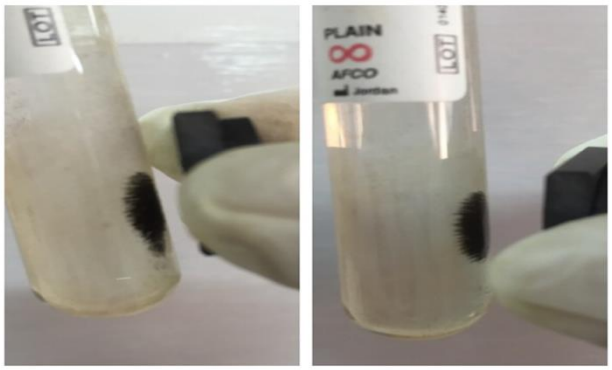

Fig. 2. Photographs of a solution in the absence and presence of a magnet

Fig. 2 the magnetic response of Fe_3O_4 MNPs was test by placing a magnet near the glass bottle. The particles were attracted toward the magnet; so, the Fe_3O_4 MNPs can be separated under an external magnetic field.

2.3. Characterization

Many characterization techniques were used to measure specification and properties of nanoFe_3o_4.

XRD analyses were carried out at room temperature using a Shimadzu 6000 (Japan) using CuKα radiation Nickel filter (λ= 1.5418A°).Data were collected within the 2Θ range of 2° and 50° with a 2θ step size of 0.02 and a step time of 0.24s per step (40kv and 30mA).

The surface area of prepared catalyst was measured by nitrogen adsorption at liquid nitrogen temperature at -196 °C using the BET method, Pore volume is a measure the void space in the catalyst.

The chemical composition of the prepared Fe_3O_4 was analyzed using XRF technique. Conducted the test for a device of the type SPECTRO XEROS, Germany by weight of sample is 3g in powder state, put in plastic cup 30mm diameter. Test conducted in inert atmosphere (Helium).

Atomic Force Microscope (AFM) is a powerful technique for surface investigation by providing material topology in high resolution. The test was performed by Device (type Angstrom, Scanning Probe Microscope, Advanced Inc, AA 3000, USA), performed for samples by ethanol dispersion to conducted the surface morphology and the particles size.

And FT-IR spectroscopy analysis of Fe_3O_4 was carried out to study the features their structural by the chemical bonds (functional group) between molecules. This test was determined using a Shimadzu FTIR 8400S (Japan) with wave number range (400-4000 cm^{-1}).

3- Results and Discussion

3.1. X-Ray Diffraction (XRD)

X-ray diffraction was implemented to check the required pattern of Fe_3O_4 and its crystalline. From Fig. 3 X-ray diffraction pattern of the prepared nanoFe_3O_4 is approximately comparable with the standard and Table1 comparison of lattice spacing and angle, between prepared nanoFe_3O_4 and standard.

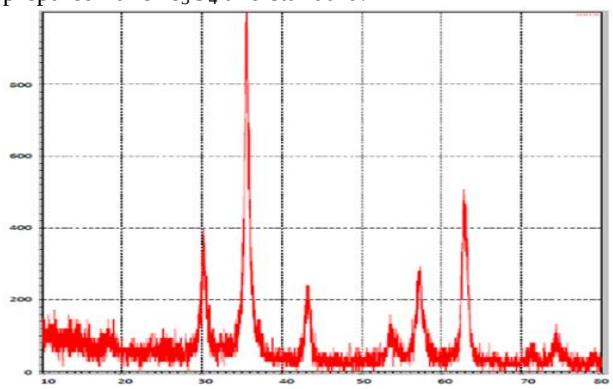

Fig. 3. XRD pattern of prepared nano Fe_3O_4

Table 1. Comparison of lattice spacing and angle, between prepared Fe_3O_4 and standard

Prepared catalyst		Standard of catalyst	
Angle(2Theta)deg	d, spacing(Å)	Angle(2Theta)deg	d, spacing(Å)
30.22	2.954	30.094	2.967
35.601	2.519	35.422	2.532
43.246	2.09	43.051	2.099
53.59	1.708	53.390	1.714
57.225	1.608	56.942	1.615
62.873	1.476	62.514	1.484
71.310	1.321	70.923	1.327

3.2. Surface Area and Pore Volume

The surface area of magnetite range from 4-100m^2/g .The obtained value of surface area of prepared nanomagnetite =85.97m^2/g, this value is in agreement with standard [16]. The pore volume for nanomagnetite (Fe_3O_4) was found equal to 0.1566cm^3/g.

3.3. Atomic Force Microscope (AFM)

The surface uniformity of the prepared nanoFe_3O_4 was studied using Atomic Force Microscope with 408 pixel density. Fig. 4 shows AFM on two-dimensional surface profile while

Fig. **5** shows AFM for two dimensional surface profiles. The two dimensional image showed hexagonal structure and three dimensional image of the Fe_3O_4 crystal obtained by AFM indicated hexagonal layers.

The particle size distribution for prepared nano Fe_3O_4 was obtained as shown in Fig. 6 From Fig. 6 show that the most volume percentage 12.98% of particle size distribution was at 90 nm and the lowest volume percentage 0.38 % was at 35nm and also show the prepared nano Fe_3O_4 consisted of particles with diameters ranged between 35 - 100 nm this means that the particles of prepared nanoFe_3O_4 are nanometer-sizes and the average particles diameter of nano$Fe_3O_4$was 75.92nm.

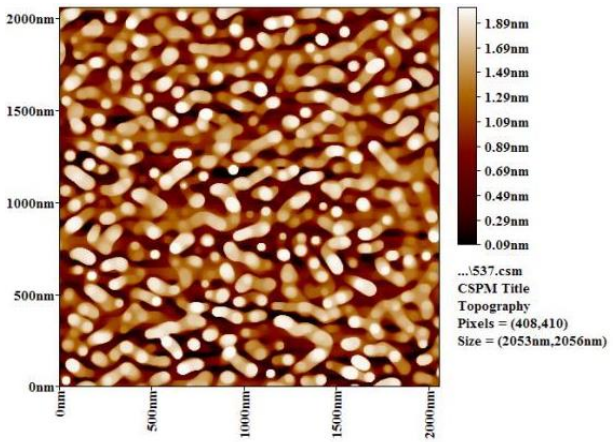

Fig. 4. AFM two-dimensional surface profiles for Fe_3O_4

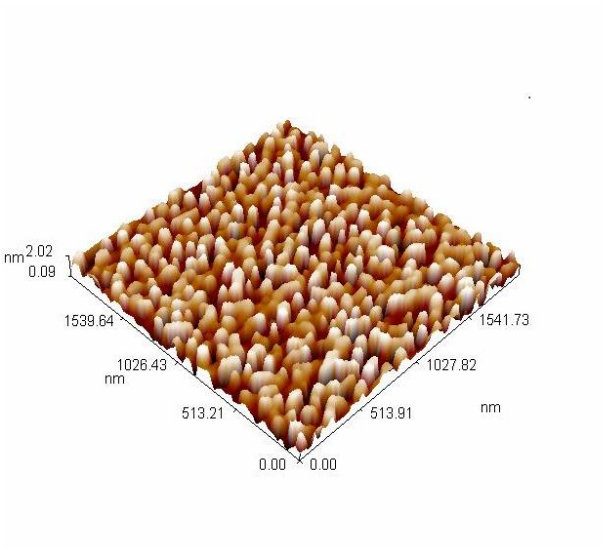

Fig. 5. AFM three-dimensional surface profiles for Fe_3O_4

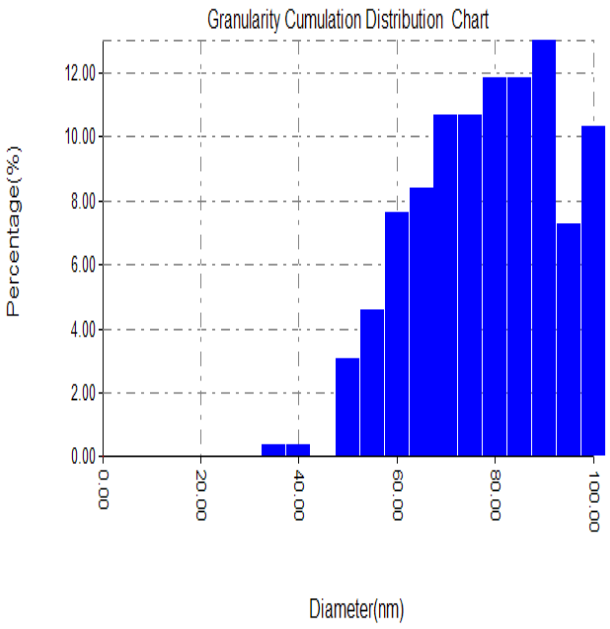

Fig. 6. Granularity cumulation distribution for prepared Fe_3O_4

3.4. X- Ray Florescence (XRF)

The chemical composition of the prepared Fe_3O_4 was analyzed using XRF technique. Table 2 represents the chemical composition of the prepared nano Fe_3O_4 expressing in weight percent. Fe and O elemental composition 69% and 28.24% respectively this value is not far from the value 71.58% for Fe and 28.4% for O**.**

Table 2. The chemical composition for the prepared Fe_3O_4

Oxides, wt. %	Fe_2O_3	P_2O_5	CaO	TiO_2	MgO
Fe_3O_4	77.8	0.5	0.25	0.45	0.37

3.5. Fourier Transform Infrared Spectroscopy (FTIR)

Fig. 7 illustrates the FTIR spectra of prepared nano Fe_3O_4. From this figure it can be observed that, the bands 586 is assigned to characteristic Fe–O vibrations of Fe_3O_4.The band O–H vibrations occur from 3160 to 3430 cm^{-1}. Slight differences occur in the peaks at 3414 cm^{-1} representing–OH functions .The band at 1616 cm^{-1} is due to bending modes of the water molecules adsorbed on magnetite surfaces [17].

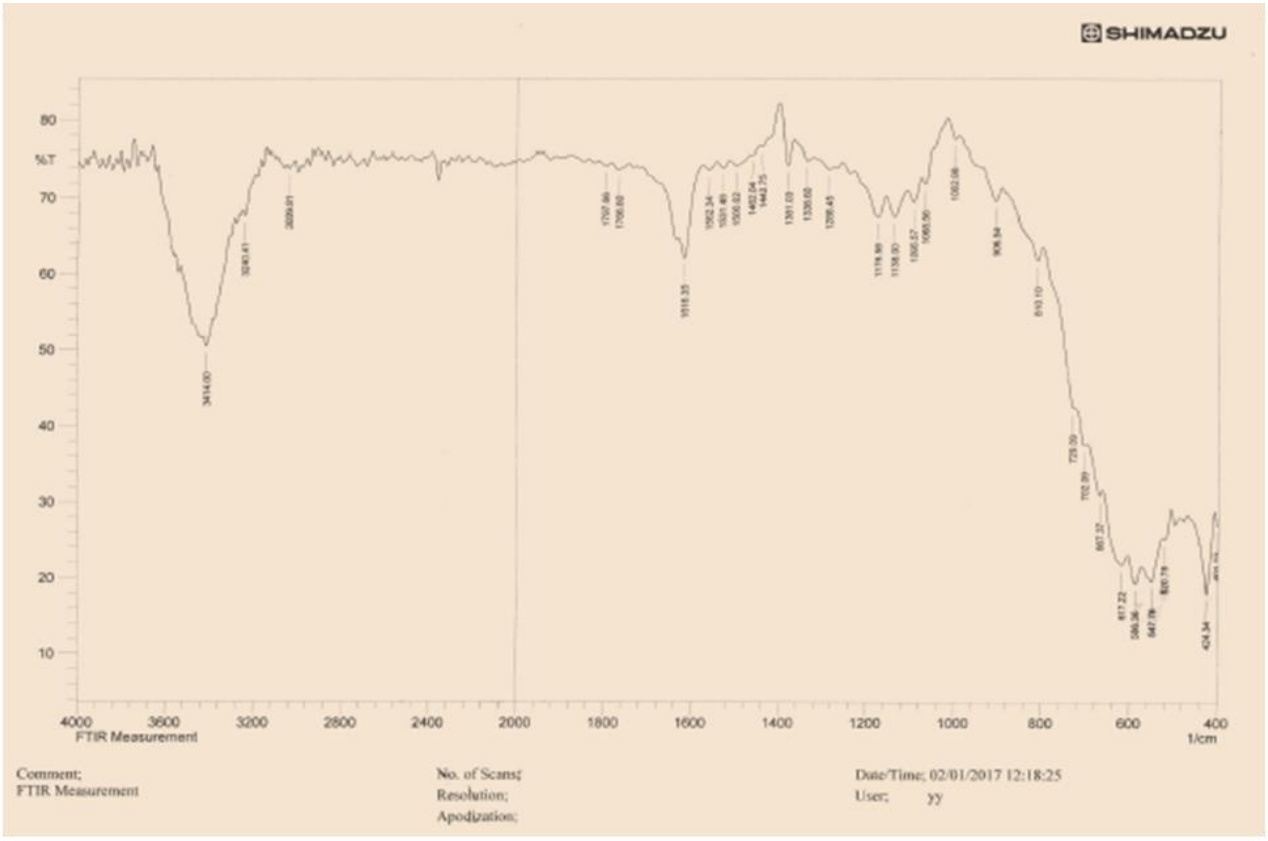

Fig. 7. FTIR of synthesized nano Fe_3O_4

4- Conclusion

According to the results obtained from this study, nanomagnetite can be synthesized successfully by using iron (II) chloride tetrahydrate and Sodium Hydroxide by open vessel ageing process. The X-Ray diffraction patterns of synthesized nanomagnetite show very good agreements with standard magnetite. The value of surface area of prepared nanomagnetite was 85.97m^2/g and the pore volume was find equal to 0.1566 cm3/g. Fe and O elemental composition 69% and 28.24% respectively

The average particles diameter of prepared magnetite was determine by AFM analysis and it was find equal to75.92nm.

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