The Synthesis of Maghemite and Hematite Nanospheres

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Abstract

Maghemite and hematite nanospheres were synthesized by using the Sol-gel technique. The structural properties of these nanosphere powders were characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM), field emission scanning electron microscopy (FESEM), and pore size distribution. Hematite phase shows crystalline structures. The mean particle size that resulted from BET and XRD analyses were 4.9 nm and 2 nm. It can be seen from transmission electron microscopy that the size of the particles are very small which is in good agreement with the FESEM and the X-ray diffraction. The BET and pore size method were employed for specific surface area determination.

Keywords: **Sol-Gel, Nanoparticles, α-Fe_2O_3, γ-Fe_2O_3, TEM, X-ray diffraction**

1. Introduction

Nanoparticles of magnetic metals and oxides have attracted great interest in recent years because of their unique physical and chemical properties, especially maghemite nanoparticles due to their technological and fundamental importance, such as information storage. For example, there are many active efforts to develop magnetic and optical storage components with critical dimensions as small as tens of nanometers, magnetic resonance imaging contrast agents, superparamagnetism, bioprocessing, gas sensitive materials, macroscopic quantum tunneling associated with size quantization, electronic quantum confinement effects and the transformation of γ–Fe_2O_3 phase into more stable α–Fe_2O_3 phase in the range of 773-873 K [1-7]. Different methods have been reported in the literature for the preparation of nanosized iron-oxide nanoparticles, via chemical routs [5] with enhanced surface to volume to ratio and novel properties better than micro-sized/bulk [6]. The sol-gel technique is largely used for the production of metal oxide thin films, allowing the accurate control of the film morphology and purity [7].

In this study we have scrutinized the effect of urea concentration on particle size, by decreasing urea concentration we can reduce the particle size of iron-oxide nonparties.

2. Experimental and Results

In this typical synthesis iron and urea were used as a reagents. The synthesis of nanoparticles were done by dissolving 4.04 g (0.01M) of Fe $(NO_3)_3 9H_2O$ into double distilled water and then urea was added to the solution. The reaction was carried out at 80°C for 24 hrs. Finally resulting brown precipitate obtained was centrifuged at 5000 rpms for 5 minutes. To transform the (γ-Fe_2O_3) into (α-Fe_2O_3) phase samples were calcinated at 400 to 500 °C in furnace for 3 hrs. Some additional experiments were carried out to drease the size of nanoparticles by dreasing concentration of urea.

To analyze the phase composition and average size of the dried powders X-ray diffraction (XRD) analysis was carried out .The morphology of the samples was inspected in Field emission scanning electron microscopy (FE-SEM). To investigate the size and morphology of the nanoparticles transmission electron microscopy (TEM) was used. TEM samples were prepared by the addition of pure acetone to the powder and then dispersed by ultrasonication for 15 mins. One drop of the suspension was put on a copper grid that subsequently was dried in open-air prior to use in the TEM apparatus. The surface area of the solid material was measured from Brunaur-Emmett-Teller (BET) analysis. Particle size distribution was also measured by Microtrac -UPA 150.

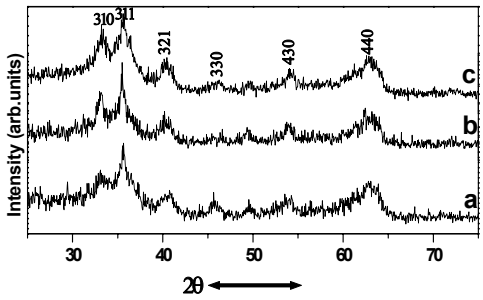

Fig. 1. X-ray diffraction patterns of sample (a) to (c) (indicating γ-Fe_2O_3 phase)

The The X-ray diffraction spectra (Figs.1 abc) shows decrease in the particle size which is clear evident by the changes in peak width (broadness and narrowness) indicating that aging affects the size of particles. Moreover the intensity of 311 peak is increased (Fig.1b) which shows that crystallinity is also enhanced. The XRD spectra (Fig.1) represents maghemite (γ-Fe_2O_3) with different aging periods. The phase identification was performed by comparing the measured diffraction peaks with peaks of JCPDS cards (39-1346). The particles appear agglomerated because of the various kinds of attractive forces between the fine particles. The average size of γ-Fe_2O_3 nanoparticles calculated by Scherer formula were 2 nm. With decreasing particle size, attractive forces increased leading to enhanced agglomeration of nanoparticles.

The XRD spectra Fig.2, represents only the hematite phase (α-Fe_2O_3) which is due to the transformation of γ-Fe_2O_3 (metastable phase) into α-Fe_2O_3(stable) on heating at 500˚C.The particles were agglomerated due to annealing of the samples for 3 hrs at 500˚C ,and the average size of particles as per Scherer formula were 8 nm.

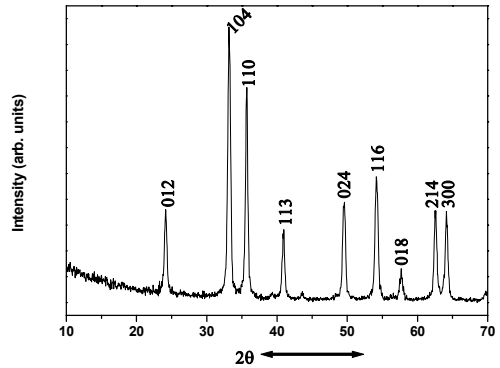

Fig. 2. X-ray diffraction patterns of sample (d) (indicating α-Fe_2O_3 phase).

The scanning images show a uniform distribution of particle size. The particles appear to be spherical and agglomerated because of various kinds of attractive forces, which consists of hydrogen bonds, vander waal forces, coulomb forces and physical friction between the fine nanoparticles. The particles appear elongated due to calcinations at 500˚C for 3 hrs. Moreover size of the particles is shown in table 1.

TEM analysis was done to observe the mean size and distribution of the spherical and agglomerated particles. Due to calcinations at 500^{o}C for 3 hrs γ–Fe_2O_3 was transformed into α–Fe_2O_3.It is interesting to note that the particle size increased considerably during calcinations.By TEM images the mean size of nanoparticles was 15 nm, while as on calcinations for 500^{o}C for 3 hrs size of elongated particles was 22 nm.

Table 1. Surface area, Particle size and Molar Concen-tration of Feric Nitrate/Urea

Sample No.	BET surface Area	Langmuir Surface Area	Particle size	Feric Nitrate/urea Concentration
a	122.303 (m2/g)	336.02 (m2/g)	7.2 nm	1:4.5
b	168.303 (m2/g)	336.02 (m2/g)	6.4 nm	1:1.5
c	200.303 (m2/g)	336.02 (m2/g)	4.9 nm	1:1

3. Summary

Maghemite and hematite iron-oxide nanoparticles with average sizes of 2 nm and 10 nm were synthesized by the Sol-gel method. X-ray diffraction analysis confirmed two phases corresponding to γ-Fe_2O_3 and α-Fe_2O_3. Particle size measurement from X-ray diffraction patterns show that the particle size decreases with decrease in urea concentration. BET and pore size analysis shows that surface area increased while particle size decreased. The particle size distribution shows uniform distribution.

4. References

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