Synthesis and Characterizations of Nanosized Fe_3O_4 for Biomedical Applications

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Introduction

Magnetite (Fe_3O_4), a ferrimagnetic compound, is the oldest magnetic material known to humankind. Due to its interesting magnetic properties, Fe_3O_4 has important applications. Magnetite (Fe_3O_4) is a common magnetic iron oxide, and it has a cubic inverse spinel structure with oxygen forming a FCC closed packing and Fe cations occupying the interstitial tetrahedral sites and octahedral sites. The electrons can hop between Fe^{2+} and Fe^{3+} ions in the octahedral sites at room temperature, rendering magnetite an important part of half-metallic materials.

Magnetite nanoparticles have been widely studied because of their applications in ultrahigh density magnetic storage media, biological labeling, tracking, imaging, detection, and separations, and Ferro fluid. In particular, magnetite nanoparticles have attracted great attention for many important biomedical applications such as magnetic separation, drug delivery, cancer hyperthermia and magnetic resonance imaging (MRI) enhancement, due to their non-toxicity property and high chemical stability. Superparamagnetic magnetite nanoparticles find clinical applications. In MRI, contrast enhancement. The magnetite nanoparticles produce an enhanced proton relaxation in MRI. The superparamagnetic magnetite can be incorporated into embolic materials to enable MRI detection and thus find a practical application in embolotherapy. In this respect, magnetite nanoparticles are required to be water-soluble, monodisperse, superparamagnetic and easily to produce in large scale. To date, various techniques but most often chemical syntheses methods for preparing magnetite nanoparticles already have been reported, such as co-precipitation, micro-emulsions, solvothermal processing, and high-temperature organic phase decomposition. However, it has been demonstrated that the physical and chemical properties of magnetite nanoparticles greatly depend upon the synthesis route, and the synthesis of magnetite particles that can meet all above mentioned requirements remains a challenge.

In this article, magnetite nanopowder was synthesized by thermal decompositions of iron salts in the presence of ethylene diamine tetra acetic acid (EDTA). The iron chloride underwent complex formation reaction with (EDTA) followed by thermal decomposition of the iron complex by boiling to produce the magnetite powder. The prepared powders were investigated by high magnification SEM and XRD to identify the powder shape, size and the chemical composition.

The produced magnetite powder has spherical like particle shape with around 30 nm particle sizes and has the chemical composition of magnetite. The magnetic properties were measured using vibrating sample magnetometer (VSM) method for characterizing the produced magnetite. It was observed that the prepared magnetite has ferrimagnetic properties with a magnetization saturation 88.1 emu/g, coercivity 214 Oe and retentivity 12.7 with squareness ratio of 0.14.

Experimental

Solution consists of mixture of ferric chloride and ferrous chloride were prepared as a source of iron by dissolving the respective chemicals in distilled water under vigorous stirring using a magnetic stirrer of 500 rpm at room temperature (25 ◦C). As a second step, solution contains EDTA was combined with the first solution and a homogenous mixture was formed. An aqueous dispersion of particles was obtained just after adjusting the pH by hydroxide and boiling of the contents. We obtain black particles (Fe_3O_4; magnetite). These black particles were washed free of anions with deionized water.

Scanning electron microscopy of the model (JEOL, JSM-T20) was used for investigating shape and size of the prepared powder. The phase structures of magnetite nanoparticles were characterized by powder X-ray diffraction using (BRUKER D8 ADVANCE) at room temperature.

The magnetization loops for magnetite nanoparticles were measured at room temperature using a vibrating sample magnetometer of model VSM 9600-1 LDJ. The measured properties included magnetization saturation, retentivity and coercivity. Fe_3O_4 nanoparticles were washed with distilled water several times before being analyzed for the magnetic properties. Because the magnetization saturation and the retentivity changes with weight and volume of the sample, the results were divided on sample's weight. Magnetization versus applied field was measured at room temperature for all samples. The magnetometer was calibrated with standard nickel sphere. Analyses were carried out using a maximum applied field of 1.5 T.

Results and Discussion

The magnetite nanoparticles were prepared by thermal decomposition of iron chloride / EDTA complex solution. The reaction parameters were systematically modified to produce designed nanoparticles. The mixture was heated at different temperatures for a period. The solution turned black upon the formation of Fe nanoparticles and then the

Fe nanoparticles oxidized to magnetite nanoparticles. For such synthesis, the chemical reaction is expected as follows:

$$Fe^{2+} + 2Fe^{3+} + 8\,OH^- \rightarrow Fe_3O_4 + 4H_2O$$

Figure (1) shows the XRD patterns of magnetite nanoparticles obtained by boiling of the iron chloride/ EDTA complex solution. It was observed that there is only one physical phase due to the presence of magnetite. In addition, there is no any other diffraction peaks corresponding to ferric chloride or other iron oxide, such as Fe_2O_3, was detected. This reveals that the resultant particles are mainly pure Fe_3O_4.

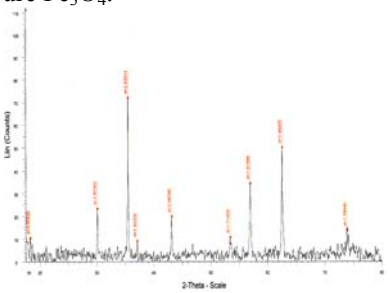

Figure (1) XRD pattern for the prepared magnetite powder by thermal decomposition of iron chloride/EDTA complex solution.

Figure (2) illustrate the SEM micrograph for the produced magnetite powder, which has a spherical like particle shape with around 30 nm particle size. By this unique and fine size, it can be used as biomaterial in the form of magnetic powder for several applications such as magnetic separation, drug delivery, cancer hyperthermia and magnetic resonance imaging (MRI).

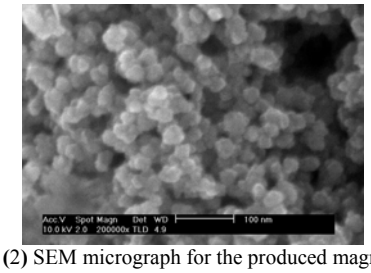

Figure (2) SEM micrograph for the produced magnetite powder.

The magnetic properties were measured by using vibrating sample magnetometer. The hysteresis loops of the Fe_3O_4 nanoparticles measured at room temperature are illustrated in Figure (3) and the magnetic properties values were listed in table (1). The saturated magnetization values of Fe_3O_4 nanoparticles obtained at magnetic field of 1.5 T was 88.1 emu/g. The coercivity values H_c are to be 214 Oe. This means the prepared magnetite readily displayed magnetization when subjected to a magnetic field. It was found that there was a small retentivity of 12.65 emu/g. One possible mechanism for this unique form is the independent thermal fluctuation of small ferrimagnetic domains inside the particles. The boundaries of the small crystallites within the particles may contain lattice defects that impede the propagation of the magnetic order.

Table (1) The magnetization saturation (B_s), retentivity (Br), and the coercivity (H_c) for the magnetite powder synthesized by thermal decomposition of the iron chloride/EDTA complex solution.

Magnetic Properties	Value
Magnetization Saturation (Bs), emu/g	**88.10**
Retintivity (Br), emu/g	**12.65**
Coercivity (Hc), Oe	**214.00**

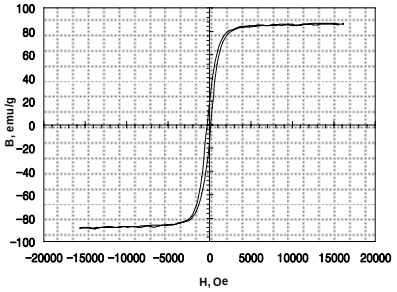

Figure (3) The B-H hysteresis loop for the produced Fe_3O_4 powder.

Conclusion

In summary, Fe_3O_4 nanoparticles have been prepared by thermal decomposition method. The XRD and SEM results show that homogeneous sized Fe_3O_4 nanoparticles with spherical shape can be obtained simply. The procedure in the present study offers several very important advantageous features for preparation Fe_3O_4 nanoparticles. First, the synthetic process is economical, medical and environmentally friendly, because it involves inexpensive and less toxic iron salts.

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