Synthesis and Characterization of Fe_3O_4 Nanoparticles for Engineering Applications

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ABSTRACT

In the past few decades, Magnetite (Fe_3O_4) nanoparticles have attracted growing research interest and have a great impact in the applications of wave absorbing material, paste for inducing magnetism, magnetic fluid ,adsorbent and many engineering applications. In this paper, Magnetite (Fe_3O_4) nanoparticles were prepared by the chemical co precipitation method from the solution of ferrous/ferric mixed salt-solution in alkaline medium. Magnetite nanoparticles was characterized by X-ray powder diffraction (XRD) analysis, Scanning Electron Microscopy (SEM).

1. INTRODUCTION

Researchers pay increasing attention to the production of iron-based magnetic nanopowders and their structure and properties. Compared to atomic or bulky counterparts, nano-sized Fe_3O_4 materials owe superior physical and chemical properties due to their mesoscopic effect, small object effect, quantumsize effect and surface effect.Recently, Fe_3O_4 MNPs have been intensively investigated because of their superparamagnety, high coercivity and low Curie temperature[1–4]. In addition to these characters, Fe_3O_4 MNPs are also non-toxic.

Nano- Fe_3O_4 find applications as (a) Wave-absorbing Materials : Fe_3O_4 is highly absorbent of magnetic waves and thus can be used in military high- performance micro meter and millimeter stealth materials,visible light-IR stealth materials ,constructive stealth materials and radiation-shielding materials for cell phones.(b)Paste for Inducing Magnetism : Due to it's high saturated magnetic strength and high magnetic induce ratio, Fe_3O_4 can be made into paste for inducing magnetism to be used in adhesive structures of very fine magnetic heads[5].(c)Magnetic Fluid : Due to it's excellence in producing magnetic fluid, Fe_3O_4can be widely used in sealing, anti-shock, medical instruments and equipment, volume tuning, light displaying, etc[6].(d)Adsorbent Nanotechnology is also involved in the removal of toxic metal pollutants from waste water. Industrialization has brought about an ever-increasing exposure to toxic heavy metals such as lead, mercury or arsenic where they are irresponsibly dumped into rivers and oceans. A process using iron oxide nano particles as adsorbent to attract these heavy metals has been developed. It has been widely used in a variety of technological applications, such as a catalyst for ammonia, ceramics, energy storage, magnetic data storage, ferrofluids, and bioapplications[7-9]. Keeping view of applications and importance of nano Fe_3O_4 in diversified fields,we synthesized magnetite by one of the cheap and convenient method.

Magnetite (Fe_3O_4) is a black, ferromagnetic mineral containing both Fe(II) and Fe(III). In stoichiometric magnetite Fe(II)/Fe(III)= 0.5 but magnetite is often non-stoichiometric resulting in a cation deficient Fe(III) layer. The crystal structure of magnetite is inverse spinel with a unit cell consisting of 32 oxygen atoms in a face-centered cubic structure and a unit cell edge length of 0.839 nm. In this crystal structure Fe (II) ions and half of the Fe (III) ions occupy octahedral sites and the other half of the Fe(III) occupies tetrahedral sites. Divalent iron atoms prefer to occupy octahedral sites to have a higher Crystal Field Stabilization Energy (CFSE), whilst the trivalent iron atoms has a CFSE=0 in both octahedral and tetrahedral sites. The crystal forms of magnetite include octahedron and rhombodecahedron and the specific surface area ranges from 4 - 100 m2/g[10].

There are various ways to prepare Fe_3O_4 nanoparticles, chemical coprecipitation method has the potential to meet the increasing demand for the direct preparation of welldispersed (water-base) Fe_3O_4 nanoparticles and offer a low-temperature alternative to conventional powder synthesis techniques in the production of nanopaticles, and the sizes of nanopaticles can be well controlled[11].

2. EXPERIMENTAL

2.1. Materials

All chemicals in this work were of analytical grade and were used without further purification. Ferric chloride hexa-hydrate (FeCl3·6H2O), ferrous chloride tetra-hydrate (FeCl2·4H2O), and ammonium hydroxide (NH4OH, 26% of ammonia) were purchased from Aldrich.

2.1.1. Synthesis of magnetite nanoparticles. This method may be the most promising one because of its simplicity and productivity . It is widely used for engineering applications because of ease of implementation and need for less hazardous materials and procedures .

Ferric chloride and ferrous chloride were mixed in 2:1 molar ratio. The solutions of Fe2+ and Fe3+ were prepared by making their aqueous solutions in distilled water and this solution containing both ions was then heated up to 50°C for 10 min. After heating, the solution was precipitated by ammonia solution with continuous stirring on the magnetic stirrer at50°C. Black colored particles of iron oxide were precipitated. These particles were then separated from the solution by using a strong magnet and then were washed many times with distilled water .The precipitated magnetite is black in color .The powder was then dried in hot air oven at 100°C for overnight.The overall reaction can be written as

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

Scanning electron microscopy of the model was used for investigating shape and size of prepared powder.The phase structure of magnetite nanoparticles were characterized by powder X-ray diffraction(XRD)

3. RESULTS AND DISCUSSION

The magnetite nanoparticles were prepared by co-precipitation method .The reaction parameters were systematically balanced to produce designed nanoparticles.For accurate determination of lattice parameters database power diffraction file JCPDF PDF release 1997,International Center for Diffraction Data (ICDD) was used.

XRD technique was adopted to identify the desired nanoparticles prepared with ferric and ferrous salts ,over 2θ range from 20° to 80°.Particle size was determined by Schere formula:

$$D=k\lambda/b\cos\theta \qquad (1)$$

The XRD of the sample(fig.1) shows the formation of Fe_3O_4 ,based on the comparison of their XRD patterns with the standard pattern of Fe_3O_4 (JCPDS 75-0033).The diffraction peaks corresponding to (220),(311),(400),(511),(440) are quite identical to characteristic peaks of the Fe_3O_4 crystal with the cubic spinal structure .In addition of the peaks of Fe_3O_4 some peaks of γ-Fe_2O_3 were also found in the prepared powder.The application of Scherrer's formula to the (311) reflection peak indicated the formation of nano Fe_3O_4 with approximately 10 nm in diameter. Lattice constant of synthesized Fe_3O_4 is found to be 8.357 A. & matches well with earlier reported data[7].
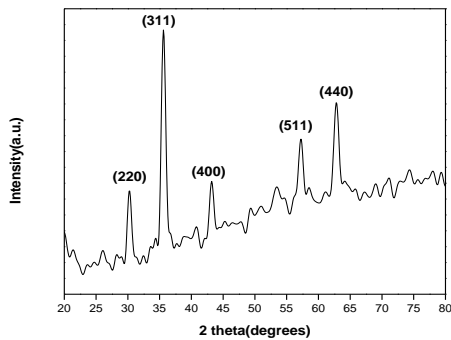

Figure 1. XRD of nano iron oxide powder.

Fig.2 illustrates the SEM micrograph for the produced magnetite powder ,which has a spherical like particle shape with around less than 15 nm particle size. By this unique and fine size ,it can be used in many engineering application as high density magnetic recording ,radar signal adsorbing ,magnetic fluid ,optical devices ,solar cell and so on.
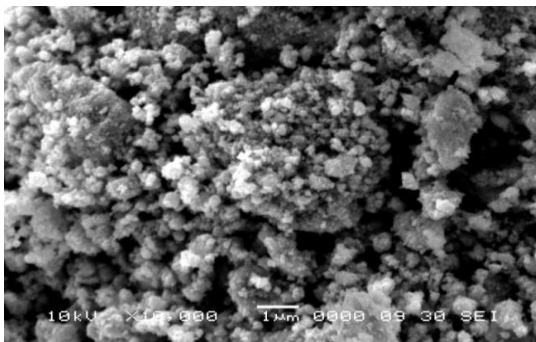

Figure 2. SEM of nano iron oxide powder.

4. CONCLUSION AND DISCUSSION

In summary, we reported chemical co-precipetation approach,which is simple and effective technique for preparing magnetite nanoparticles .The XRD and SEM results show that homogeneous sized Fe_3O_4 nanoparicles with spherical shape can be obtained . SEM of Fe_3O_4 MNPs show spherical shape with diameter about 10-15 nm, which is in agreement with the results of the XRD analysis. Lattice constant of Fe_3O_4 is 8.357,which is matching with a=8.384 of the standard (JCPDS 75-0033).The synthetic process used is economical and environmentally friendly ,as it involves inexpensive and less toxic iron salts ,which make Fe_3O_4 MNPs as a promising material for engineering applications.

5. REFERENCES

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