ABSTRACT

In the last decade, development of magnetite (Fe₃O₄) nanoparticles have been done intensively. The attractiveness of this nanoscale material is caused by its unique properties that different from its corresponding bulk, especially in magnetic aspect. By its superparamagnetic behavior the nanoparticles may be controlled by an external magnetic field for special purpose in many applications. The usual problem in any synthesis method is how to create high purity of monodisperse magnetite nanoparticles. For this reason many magnetite synthesis methods have been developed. The purpose of this research is to study the synthesis of magnetite nanoparticles by electrochemical method.

First of all, the synthesis was conducted by electro-oxidation of iron in plain water under various of current densities and inter-electrodes distances by Continuous Direct Current (CDC). All of the synthesis were conducted at room temperature. However some impurities were detected. In order to enhance purity of magnetite, the preparation was carried out in dilute solution of sodium silicate under CDC. Another innovative way to improve the purity of magnetite nanoparticles was also done using Pulsed Direct Current (PDC) in plain water. The nanoparticles generated by the above methods were black powder precipitated around the anode. The particles were then characterized by X-ray diffraction (XRD) and Fourier Transform Infrared Spectrophotometry (FTIR) for component identification; Brunauer-Emmet-Teller (BET) for indirectly measured particle size; Scanning Electron Microscopy (SEM) to observe the morphology; and Vibrating Sample Magnetometry (VSM) for evaluating its magnetic properties.

Both XRD pattern and FTIR spectra of the particles show that the particles are mainly magnetite. The generated particles are nearly spherical and monodispersed with a mean size between 10–30 nm depending on synthesis conditions. They show a ferromagnetic behavior with a relatively high saturation magnetization (~70 emu/g). However some impurities in the form of FeOOH still remain in the particles. FeOOH was assumed to be an intermediate product during magnetite formation. Based on magnetite formation mechanism deduced, two methods were proposed to enhance the purity of magnetite. In the first method, the impurities were reduced by isolating the magnetite via in-situ silica coating with sodium silicate as the source of silica that causes the transformation of FeOOH to magnetite proceeds completely. High purity magnetite with sizes ranging from 6–10 nm were successfully prepared by this method using sodium silicate solution with concentration of about 150–200 ppm. In the second way, FeOOH was eliminated by applying PDC that was assumed to interfere diffusion of OH⁻ produced by water reduction in cathode. The OH⁻ ion has a very important role in FeOOH formation. High purity magnetite nanoparticles having ferromagnetic properties with high Mₛ were successfully synthesized by PDC.

This study demonstrated that magnetite nanoparticles can be prepared using a simple electrochemical method. These results also demonstrate the possibility of using this technique as an environmentally friendly synthetic route to producing the very applicable magnetite nanoparticles.

Key words: magnetite, electrochemical, silica, pulsed