Contents lists available at ScienceDirect

Advanced Powder Technology

journal homepage: www.elsevier.com/locate/apt

Original Research Paper

Synthesis of magnetite nanoparticles by surfactant-free electrochemical method in an aqueous system

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ARTICLE INFO

Article history: Received 1 November 2010 Received in revised form 27 March 2011 Accepted 12 April 2011 Available online 27 April 2011

Keywords: Magnetite nanoparticles Electrochemical synthesis Aqueous system Surfactant-free Magnetic properties

ABSTRACT

A simple surfactant-free electrochemical method is proposed for the preparation of magnetite nanoparticles using iron as the anode and plain water as the electrolyte. This study observed the effects of certain parameters on the formation of magnetite nanoparticles and their mechanism in the system, including the role of OH^- ions, the distance between electrodes and current density. We found that OH^- ions play an important role in the formation of magnetite nanoparticles. Particle size can be controlled by adjusting the current density and the distance between electrodes. Particle size increases by increasing the current density and by decreasing the distance between electrodes. Particle formation cannot be favored when the distance between electrodes is larger than a critical value. The magnetite nanoparticles produced by this method are nearly spherical with a mean size ranging from 10 to 30 nm depending on the experimental conditions. They exhibit ferromagnetic properties with a coercivity ranging from 140 to 295 Oe and a saturation magnetization ranging from 60 to 70 emu·g⁻¹, which is lower than that of the corresponding bulk Fe₃O₄ (92 emu·g⁻¹). This simple method appears to be promising as a synthetic route to producing magnetite nanoparticles.

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1. Introduction

Magnetite (Fe₃O₄) is one of the most important magnetic materials and is widely used in industry. In recent years, monodispersed magnetite nanoparticles have attracted increasing attention due to their good biocompatibility, strong superparamagnetic properties, low toxicity and easy preparation process [1]. They have considerable potential for use in the biomedical industry, such as targeted drug delivery, hyperthermia treatment, cell separation, magnetic resonance imaging, immunoassays and the separation of biochemical products [2-6]. They are also useful for environmental processes, such as the treatment of water and wastewater [7–9]. Among the methods available for the preparation of magnetite nanoparticles, the co-precipitation of ferrous and ferric ions in aqueous solutions is attractive because of its simplicity and low cost [10]. Producing monodispersed magnetite nanoparticles using this conventional method is difficult due to instantaneous reactions in the mixture that make it difficult to control the process of crystallization [11]. Moreover, the size of the generated particle is typically in the range of 5–12 nm, which tends to decrease its magnetic properties [10,12]. Alternatively, sol-gel and hydrothermal techniques may effectively control the morphology and chemical composition of the prepared magnetite nanoparticles [13,14]. However, the sol-gel technique typically requires expensive alkoxide reactants, high temperature calcinations, and a long time to produce the final product. The hydrothermal technique requires a high temperature and a long time period to obtain the final product (about 25 h). This method is typically performed in the presence of a surfactant to avoid the aggregation of particles. The combination of surfactants with magnetite may not be firm enough, and they may severely interfere with certain applications, such as biomedical uses [15].

The electrochemical method is beginning to emerge as an option for the preparation of magnetite nanoparticles [16–19]. It offers many advantages over the above-mentioned methods for the production of magnetite nanoparticles. Using this method, the particle size can be easily controlled by adjusting the imposed electro-oxidation current density or potential to the system. Marques et al. prepared magnetite nanoparticles with sizes ranging from 4.4 to 9 nm by the electrochemical method, using $Fe(NO_3)_3$ as the iron precursor in ethanol media [16]. However, the purity of the final product is quite vulnerable to the synthesis conditions, i.e., iron concentration and current density. These parameters must be controlled very carefully to prevent the formation of iron hydroxide, which contaminates the magnetite particles. Moreover, this method requires a relatively water-free bath to obtain pure magnetite particles, which causes the generated particles to be

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