



Fine tuning of magnetite nanoparticle size distribution using dissymmetric potential pulses in the presence of biocompatible surfactants and the electrochemical characterization of the nanoparticles



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ABSTRACT

The effects of varying the surfactant concentration and the anodic pulse potential on the properties and electrochemical behaviors of magnetite nanoparticles were investigated. The nanoparticles were synthesized with an electrochemical method based on applying dissymmetric potential pulses, which offers the advantage that can be used to tune the particle size distribution very precisely in the range of 10 to 50 nm. Under the conditions studied, the surfactant concentration directly affects the size distribution, with higher concentrations producing narrower distributions. Linear voltammetry was used to characterize the electrochemical behavior of the synthesized nanoparticles in both the anodic and cathodic regions, which are attributed to the oxidation of Fe^{2+} and the reduction of Fe^{3+} ; these species are part of the spinel structure of magnetite. Electrochemical impedance spectroscopy data indicated that the reduction and oxidation reactions of the nanoparticles are not controlled by the mass transport step, but by the charge transfer step. The sample with the highest saturation magnetization was that synthesized in the presence of polyethylene glycol.

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1. Introduction

Magnetite (Fe_3O_4) is a material with diverse applications: in the environmental field it is widely used in the separation of metals from wastewater, and in industry it is extensively used in magnetic ink [1]. In recent years, magnetite has also been used in biomedical applications such as cell targeting, cell separation, drug delivery, and hyperthermia treatment [2–6].

Several methods have been described for synthesizing magnetite nanoparticles: for the chemical co-precipitation and microemulsion approaches, poor control of the particle size and the particle size distribution are unsolved problems [7,8], but the tight control of particle size and the particle size distribution is essential for some applications [7,9]. The addition of a surfactant in these methods addresses this problem to a certain extent, but the precise potential regulation that is possible in electrochemical methods enables the fine control of the magnetite particle size and particle size distribution [7,9–16].

It is common to coat magnetite nanoparticles with a surface active agent, such as ethylene glycol [7], to prevent agglomeration, and at the same time to protect the particles from oxidation by oxygen [13, 17,18]. The surfactants that have been employed in the synthesis of

magnetite nanoparticles include sodium oleate [14,19], polyethylene glycol [9,19,20], oleic acid [19,21,22], polyvinylpyrrolidone [23], and others [24]. For biomedical applications, the nanoparticles and their coatings must be biocompatible and have a suitable size and size distribution.

Previously, we proposed a new method for the synthesis of magnetite nanoparticles that uses dissymmetric potential pulses, and demonstrated that this method is useful in aqueous media [25]. In this method, the effects of mass transport during synthesis [3,8] are minimized by the selection of the pulse duration. We have previously established that the anodic potential and the duration of the pulse determine whether magnetite or maghemite is favored.

In this study, by using the novel method which offers indicated advantages [25], we assessed the effects of the presence of three different biocompatible surfactants at various concentrations in the synthesis of magnetite nanoparticles on their electrochemical behaviors and properties.

2. Experimental

2.1. Electrochemical system

A three-electrode glass cell with a capacity of 50 mL was employed. Pieces of high purity iron (Goodfellow, 99.5%) with a geometric area of

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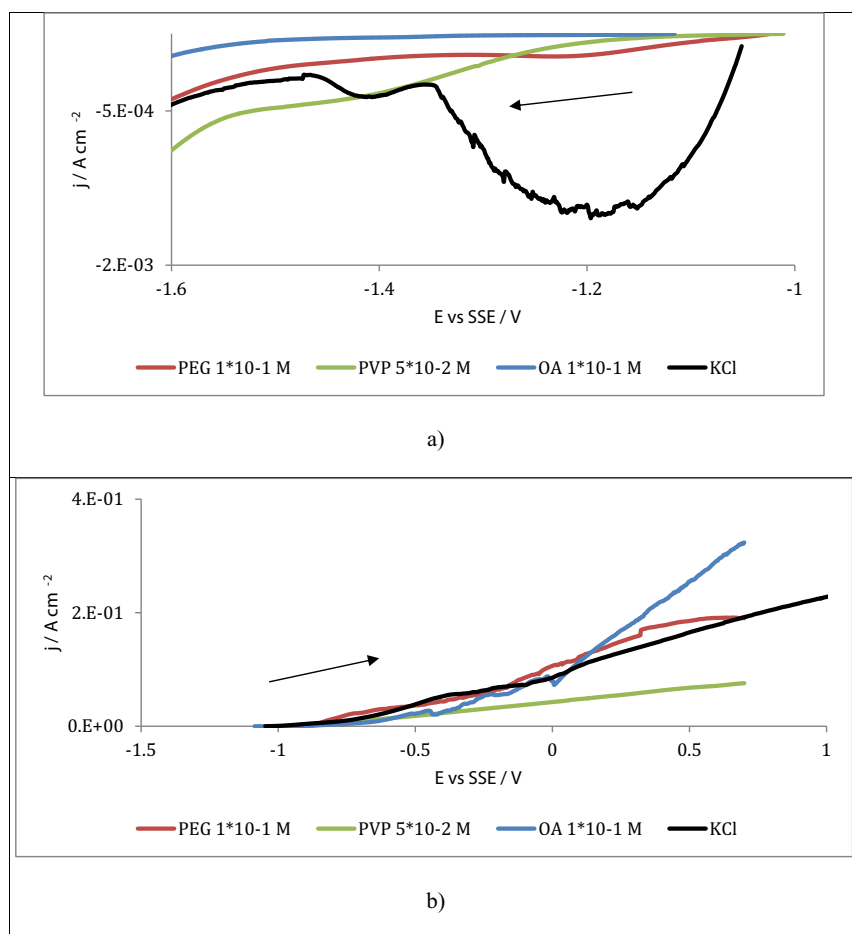


Fig. 1. Linear voltammetry in the cathodic (a) and anodic (b) directions, obtained in KCl 0.5 M and in the presence of three different surfactants. Scan speed 2 mV s^{-1} .

4 cm^2 were used as the working and counter electrodes. The reference electrode was a mercury–mercurous sulfate saturated electrode (SSE). The distance between the working and counter electrodes was set at 3 cm.

Polyethylene glycol (PEG, molecular weight 1000), polyvinylpyrrolidone (PVP molecular weight 10,000), and oleic acid (OA) were dissolved at three different concentrations in KCl 0.5 M. The solutions were prepared with ACS grade reagents and deionized water. The experiments were performed at environmental temperature and atmospheric pressure.

2.2. Syntheses

Before each synthesis, the system was stabilized for 3 to 5 min. The criterion for stabilization was 10 mV per minute as the maximum derivative. The solution was maintained without stirring. Slow linear voltammetry (2 mV/s) was carried out in order to identify and select the reduction and oxidation potentials of the iron ions and to evaluate the electrochemical stability of the medium. In order to select the duration of the potential pulses, chronoamperometric measurements were performed. We applied the design of experiments (DOE) method with

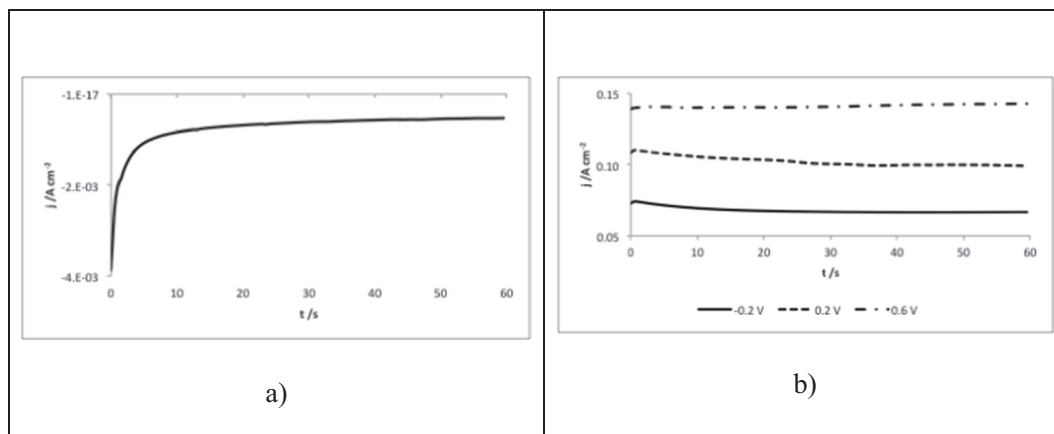


Fig. 2. Current behaviors at various pulse potentials: a) cathodic at -1.5 V vs SSE, and b) anodic potentials vs SSE.

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