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Sonochemical preparation of magnetite nanoparticles by reverse precipitation method

Tatsuya Shuto[1], Osamu Nakagoe[2] and Shuji Tanabe[1,*]
[1]*Graduate School of Science and Technology,*
[2]*Department of Materials Science and Engineering, Faculty of Engineering,*
Nagasaki University, 1-14 Bunkyo-machi, Nagasaki 852-8521, Japan
**Tel: 095-819-2659, Fax: 095-819-2661, E-mail:s-tanabe@nagasaki-u.ac.jp*

Abstract

Magnetic iron oxide nanoparticles were successfully prepared by reverse precipitation method with the assistance of ultrasound. Obtained nanoparticles were identified as magnetite (Fe_3O_4) by XRD measurement. It was found that obtained magnetite nanoparticles have small sizes (about 10.7 ±2.9 nm in diameter) and spherical shape by TEM observations. In reverse precipitation method, the dropping conditions of aqueous $FeSO_4$ solution affect on the sizes and uniformity of the products.

Introduction

In recent years, magnetic nanoparticles are expected to be applied for biotechnology and have been extensively studied. For these practical uses, however, they are needed to have small sizes, uniform morphologies and soft magnetisms. Magnetic iron oxides nanoparticles are generally prepared by oxidation of iron hydroxides, which obtained by the addition of alkaline solution into iron salts (normal precipitation method :NP method). In this method, however, changes of pH value with the addition of alkaline solution are rapid and local, so that products with low uniformity are obtained. The objective of this study is to develop a new preparation method of magnetic nanoparticles, reverse precipitation method(RP method) with the assistance of ultrasound. Sonication is expected to inhibit rapid and local changes of pH value by the effective agitation, and to form of active chemical species via cavitational collapse.

Experimental

An aqueous solution of $FeSO_4$ was dropped into an aqueous solution containing NaOH and polyethylene glycol monostearate (PEG-MS). Dropping rates were controlled by a micro feeder and ultrasound irradiation (200 kHz, 6 W/cm^2) was started simultaneously with the beginning of the addition of $FeSO_4$ aqueous solution. In all experiments, the time of ultrasound irradiation and the final concentrations and total volumes at the end of the addition were the same each others. Products were collected by magnet and washed with distilled water. The crystal structures of the particles were determined by XRD measurement, and the morphologies were observed by TEM.

Results and Discussions

XRD mesurements indicated that the crystal structures of all products were magnetite

(Fe_3O_4). TEM images and particle size distributions of the products are shown in Fig. 1. The preparative conditions and the sizes of obtained magnetite nanoparticles are summarized in Table 1. In the case of the constant dropping rate (5 ml/min, Fig. 1(a)~(c)), the sizes of magnetite nanoparticles decreased with decreasing the concentration of $FeSO_4$. For the constant concentration (0.067 mol/l, (c)~(e) shown in Fig. 1), slower dropping rate gave us magnetite nanoparticles with smaller sizes and a narrower distribution. Although we prepared magnetite nanoparticles by NP method, the obtained magnetite nanoparticles had 39.3 nm in diameter, which was larger than magnetite nanoparticles prepared by RP method. Magnetization of the particles prepared with the constant concentration of $FeSO_4$ were measured by SQUID (Superconducting QUantum Interference Device). The results showed that both of the coercivity and the residual magnetization were almost zero at room temperature.This means that obtained magnetite has soft magnetism.

Conclusions

The combination of RP method and sonication gave us magnetic nanoparticles with smaller sizes and a narrower distribution. Since obtained particles have soft magnetism and were prepared with “safe” materials such as $FeSO_4$, they are expected to be applied for biotechnologies.

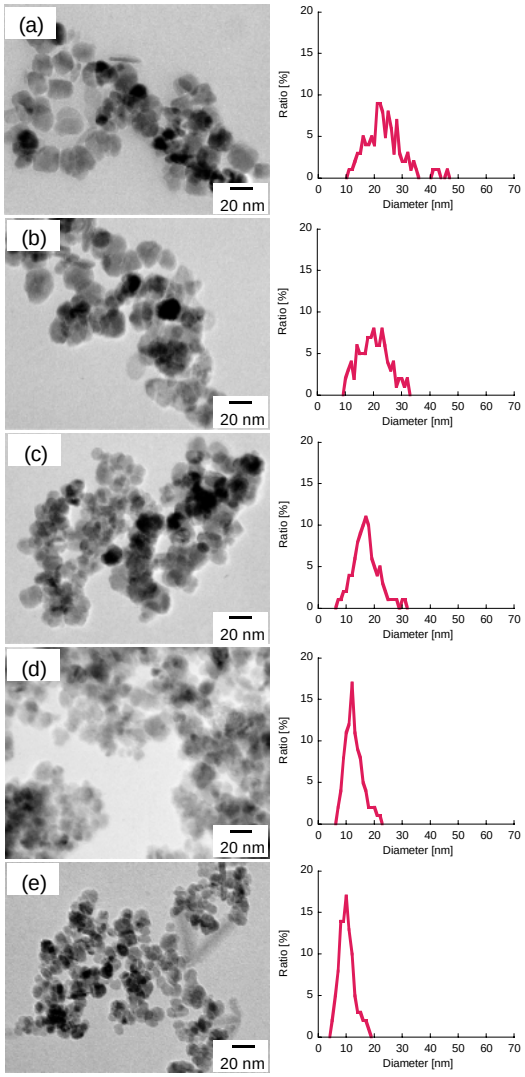

Fig. 1 TEM images and particle size distributions of magnetite nanoparticles prepared under various conditions. (a)~(e) are corresponded to those of table 1.

Table 1 The dropping conditions of $FeSO_{4aq}$ and average sizes of obtained magnetite nanoparticles

Conditions	Conc. of added $FeSO_4$ /mol･l^{-1}	Dropping rate /ml･min^{-1}	Average diameter /nm	Standard deviation /nm
(a)	0.2	5	23.8	6.0
(b)	0.1	5	20.8	5.6
(c)	0.067	5	17.8	5.9
(d)	0.067	3	13.1	3.1
(e)	0.067	1	10.7	2.9
Prepared by NPM			39.3	10.9