

A Novel Approach for Preparation of γ -Fe₂O₃ Nanoparticles: Successive Reduction-Oxidation in Reverse Micelles

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Nanometer-sized magnetic particles are very attractive for its unique properties including single domain structure and superparamagnetism, and so expected to be applied in many fields such as highly dense magnetic recording media and medical imaging. Thus, the specific properties of magnetic nanoparticles receive much attention, and there is also great interest in the fashion for the preparation of these characteristic magnetic nanoparticles. Especially for the iron-oxide, one of the most representative magnetic materials, several methods for the synthesis such as sol-gel method and alkalization of metal salt solution, as well as thermal decomposition of precursor in the nonaqueous solvent have been reported [1,2]. Here, we report a novel approach for the preparation of iron-oxide nanoparticles [3].

In this study, γ -Fe₂O₃ nanoparticles were synthesized via successive reduction-oxidation of ferrous or ferric ions within the reverse micelles dispersed in AOT/heptane system. Two heptane solutions containing 0.25 mol/dm³ of sodium bis(2-ethylhexyl)sulfosuccinate (Aerosol OT; AOT) were prepared separately. An aqueous solution of FeSO₄ (0.5 mol/dm³) was added to one heptane solution, while an aqueous solution of NaBH₄ (1.0 mol/dm³) was added to the other in order to give the molar ratio of $W = [\text{H}_2\text{O}]/[\text{AOT}] = 11$ for both the solution. After the addition of reducing agent-containing micellar solutions to the metal salt containing one, yellowish micellar solution turned light brown, via dark-blue intermediate state. This suggests the formation of iron-oxide within the nanometer-sized reverse micelles accompanied by the reduction of ionic iron species followed by the immediate oxidation due to the frequent contacts with oxygen in the air. Further, stabilization of nanoparticles with modifier molecules was carried out by the addition of 1,6-hexanediamine into the reverse micellar solution, and dark brown precipitate was filtrated, rinsed with copious amount of heptane succeeded by acetone. The crystallinity and magnetic property of the iron-oxide nanoparticles were characterized by X-ray diffractometer and super-conducting quantum interference device (SQUID) magnetometer, respectively.

X-ray diffraction (XRD) pattern of the dark brown powder collected from the micellar solution is shown in Fig. 1. The formation of γ -phase iron-oxide with relatively high crystallinity was indicated, and the mean diameter of obtained γ -Fe₂O₃ nanoparticles was estimated as ≈ 4 nm by the application of Scherrer formula to the (311) reflection peak observed at 2θ of 36°. Additionally, characteristic magnetization curves for nano-sized magnetic particles showing superparamagnetism were observed for the γ -Fe₂O₃ nanoparticles prepared by this method on the Fig. 2. The sharp maximum of the ZFC curve at T_{max} of ≈ 25 K and the splitting between ZFC and FC curves just above T_{max} indicate a narrow particle size distribution.

As described above, a novel approach for the preparation of γ -Fe₂O₃ nanoparticles was reported. We expect that the application of this method to other metal ions makes the preparation of various oxide nanoparticles easy, which is known to be synthesized with some complicated controls. In addition, the effect of several conditions for each reduction and oxidation process on the crystallinity and magnetic property of the product will be also discussed.

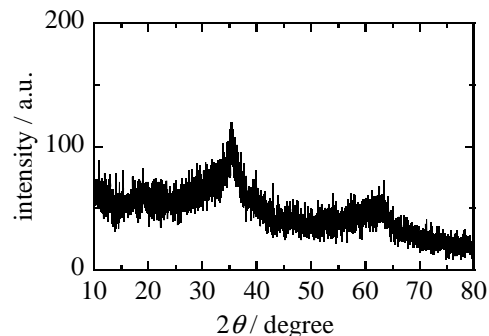


Fig. 1. XRD pattern of the sample prepared via successive reduction-oxidation in reverse micelles.

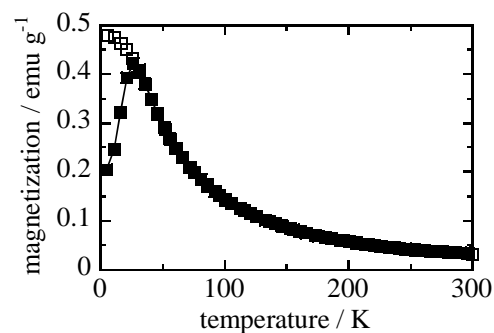


Fig. 2. Temperature dependence of the magnetization curves under ZFC; zero field cooling (solid) and FC; field cooling (open).

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