Effects of preparation conditions on the synthesis of nano-sized Ag metal particles by the wet-process using 3-mercapto-propionic acid

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Abstract—By the addition of sodium borohydride as a reducing agent into an aqueous solution of AgNO₃ mixed with 3-mercapto-propionic acid as a protective agent, nano-sized Ag metal particles could be synthesized. Using this advanced wet-process the synthesis of an aqueous Ag colloid system with a high density became possible, because the surfaces of the synthesized nano-sized Ag metal particles were covered and protected by the adsorbed 3-mercapto-propionic acid. By changing the mix ratio of AgNO₃ to 3-mercapto-propionic acid, the particle size of synthesized nano-sized Ag metal particles could be controlled; a higher 3-mercapto-propionic acid/AgNO₃ ratio was preferable in the synthesis of smaller Ag metal particles. From the relationship between the Ag metal particle size and the residual S content adsorbed on the metal particles, the mechanism of dispersion of Ag particles can be proposed as that the 3-mercapto-propionic acid having a thiol group adsorbs on Ag particles by forming Ag–S bonds as a protective agent.

Keywords: Nano-sized Ag particle; Ag colloid; mercapto-propionic acid; wet-process.

INTRODUCTION

Nano-sized particles containing metals such as silver and copper, which can perform various unique phenomena, are very attractive materials [1–8]. To synthesize large amounts of size-controlled nano-sized metal particles at low cost, a simple wet-
process is preferable. Because the stability of nano-sized metal particles is not so high and they aggregate easily, the control of particle size is very important to increase their usefulness and the development of a technique to design stable functionalized nano-sized metal particles is very attractive.

In the previous studies [9, 10], the synthesis of nano-sized Ag metal particles has been carried out using sodium borohydride as the reducing agent and 3-mercaptopropionic acid (3-MPA) as a protective agent for the metal particles. The surfaces of nano-sized Ag metal particles should be protected by the adsorbed 3-MPA in order to realize a unique aqueous colloid system with a high density of metal particles. In this study, the effects of preparation conditions on the particle size of nano-sized Ag metal particles has been investigated especially changing the ratio of AgNO₃ as starting material of Ag metal and 3-MPA as protective reagent. The characterization of samples was carried out using XRD and TEM measurements and quantitative analysis of residual S species adsorbed on metal particles.

EXPERIMENTAL

The nano-sized Ag particles and their aqueous colloid solution were synthesized by the procedure shown in Fig. 1. AgNO₃, NaBH₄ and 3-MPA were used as the source of the Ag metal, reductant agent and protective agent, respectively. As the Ag precursor solution (A), a mixture of AgNO₃ (0.1 mol), 14 mol/l aq. NH₄OH (26.0 ml), 3-MPA (4–16 mmol) and H₂O (15 ml) was prepared. As the reduction solution (B), a mixture of NaBH₄ (0.02 mol), 14 mol/l aq. NH₄OH (2.0 ml), and H₂O (15 ml) was prepared. For the reduction of Ag ions into the nano-sized Ag

![Figure 1. Scheme for the synthesis of Ag colloids.](image-url)