In-situ Observation of Formation of Ag Particles in Tween20/Water/Ionic Liquid Microemulsions

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**Introduction**

Microemulsions consisting of the ionic liquid (IL), surfactant and water have been intensively investigated by Gao et al [1-3]. For example, the ternary system consisting of Tween20/water/1-butyl-3-methylimidazolium hexafluorophosphate ([bmim][PF6]) was prepared, and three regions of the microemulsions, i.e., water-in-[bmim][PF6], bicontinuous, and [bmim][PF6]-in-water, were identified. Tween20/water/[bmim][PF6] microemulsions was found to solubilize salt species into the microemulsion droplets [3]. In this study we have synthesized Ag particles by the photoreduction of silver perchlorate (AgClO4) in the microemulsions, Tween20/water/1-octyl-3-methylimidazolium tetrafluoroborate ([omim][BF4])/CO2 system, and have investigated the electronic structure of Ag particles by in-situ EXAFS measurements. Here we have applied the high pressure CO2 to the ternary system in order to improve the mobility of ions and particles.

**Experimental**

Ag particles were synthesized in a high-pressure SUS 316 cell (inner volume of ca. 10 mL) equipped with four optical windows: two of them were CVD diamond windows for in-situ EXAFS measurements and the other two were quartz windows for the irradiation of Hg lamp. Tween20/water/[omim][BF4] microemulsions containing AgClO4 were prepared by adding CO2 into the cell which contained a mixture of AgClO4 aqueous solution, Tween20, [omim][BF4], and benzoin for photoreduction. The weight fraction of Tween20 was 0.33, and the [omim][BF4]-to-Tween20 molar ratio (R) and the water-to-Tween20 molar ratio (w) was fixed to 8.7 and 0.62, respectively. The concentration of [Ag+] in the ternary system was 4.4 or 21.1 mM. The cell was kept at 35°C and 25 MPa for 1 h with continuous stirring to form microemulsions. After stirring, the microemulsion was photo-irradiated for 5 h to investigate the reduction of Ag+ and the formation of Ag particles.

The in-situ EXAFS measurements were carried out in a fluorescence mode at NW10A beam line in PF-AR. EXAFS spectra at Ag-K edge were collected by a Ge solid-state detector to evaluate the electronic state and the coordination number of Ag particles. Data analysis was performed by REX2000 ver. 2.0.7 (Rigaku Co.).

**Results and Discussion**

Fig. 1(a) shows the EXAFS oscillations of Ag colloidal solutions prepared by 5h photoreduction for (a) the Ag solutions prepared by 5h photoreduction and (b) the reference of Ag foil and AgClO4 aqueous solution.

**References**


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