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Synthesis of Silver Particles in Water-in-Ionic Liquid Microemulsions

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Introduction

Room-temperature ionic liquids (RTILs) are attracting interest in many fields of chemistry and industry due to their potential as a "green" recyclable alternative to the traditional organic solvents [1]. Recently we synthesized Ag particles by the photoreduction of silver perchlorate ($AgClO_4$) in water-in-[OMIm][BF_4] microemulsions in the presence of Tween 20 and elucidated the formation mechanisms of Ag particles [2, 3]. In this study we have investigated the size of Ag particles as well as the water droplets containing Ag particles synthesized by the photoreduction in various microemulsions consisting of nonionic surfactants and RTILs, such as Tween 20(or Triton X-100)/water/[OMIm][PF_6] microemulsions by means of *in-situ* SAXS measurements.

Experimental

Colloidal dispersions of Ag particles were synthesized by the photochemical reduction of $AgClO_4$ in the presence of Tween 20 or Triton X-100 in water-in-[OMIm][PF_6] microemulsions. For example, 2 mL of Tween 20 was added to 4 mL of [OMIm][PF_6], followed by the addition of 20 mg benzoin and mixed vigorously. Just before the irradiation of a 500W super-high-pressure mercury lamp, 20 μL of 1.32 M $AgClO_4$ aqueous solution was added to the mixture solution with the simultaneous ultrasonication. Subsequently, the Ag^+-containing water-in-[OMIm][PF_6] microemulsions obtained were poured into a quartz cell, and the irradiation of UV-light was started with continuous stirring using a magnetic stirrer. In this case, the weight fraction of Tween20 was 0.31, and the [OMIm][PF_6]-to-Tween20 molar ratio (R) and the water-to-Tween20 molar ratio (w) was 8.1 and 0.62, respectively. SAXS measurements were performed at BL-15A. The scattering data was collected by a position sensitive proportional counter (PSPC).

Results and Discussion

Fig. 1 shows the SAXS profiles of the colloidal dispersions of Ag particles in the water-in-[OMIm][PF_6] microemulsions in the presence of Tween 20 and TX-100 before and after the photoirradiation. The SAXS intensity at a small q range ($0.3 < q < 1.2$ nm^{-1}) increases with increase of the reduction time up to 15 min. The increase of the scattering intensity indicates the formation of Ag particles during the UV-irradiation. By means of the

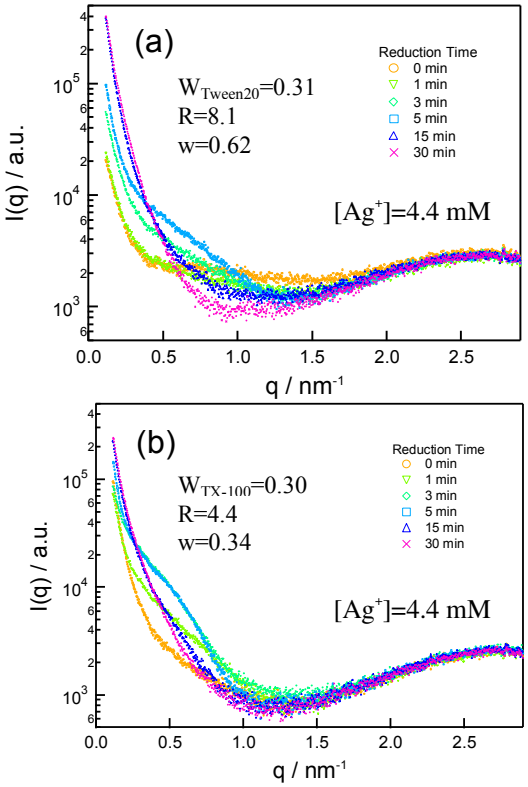

Fig. 1. SAXS profiles of the Ag colloidal solutions prepared in (a) Tween20/water/[OMIm][PF_6] and (b) TX-100/water/[OMIm][PF_6] before and after the photoirradiation.

SAXS analysis previously described [2], the number-averaged particle diameter is determined as 5.6-9.0 nm during the photoirradiation. From their Guinier plots, the average diameter of the water droplet, which consisted of ionic precursors of $AgClO_4$ and Ag particles, is estimated to be about 30-40 nm. In the Ag particle formation, aggregation of Ag atoms rapidly occurs in the water droplets of TX-100/water/[OMIm][PF_6] rather than in Tween20/water/[OMIm][PF_6] microemulsions, resulting in the increase of the particle formation rate in the case of TX-100. The detailed analysis is in progress.

References

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