

Oleic Acid as the Capping Reagent in the Synthesis of Noble Metal Nanoparticles in Imidazolium-Based Ionic Liquids

[Supporting Information]

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Chemicals. Silver trifluoroacetate (99.99+%), oleic acid (99.99%), oleylamine (70%, tech. grade), 1,2-hexadecanediol (90%, tech grade), hexane (anhydrous, 95+%), and lithium bistrifluoromethanesulfonimide ($\geq 99.95\%$) were purchased from Aldrich. Pt(acac)₂ (49.3-49.8% Pt) was provided by Gelest. Acetone (HPLC grade), chlorobutane (99.5+%), and 1-methylimidazole (99%) were provided by VWR. All reagents and chemicals were used as received. The colorless [BMIM][Tf₂N] ionic liquid was made in-house and reported elsewhere.¹ The water content in [BMIM][Tf₂N] was about 0.03%, determined by Karl Fisher coulometry method.

Synthesis of silver nanoparticles. Freshly dried colorless [BMIM][Tf₂N] (3 mL) was mixed with oleic acid (160 μ L or ~ 0.50 mmol) and silver trifluoroacetate (20 mg, ~ 0.09 mmol or 40 mg, ~ 0.18 mmol) in a 15 mL three-neck flask. This mixture was heated with a heating mantle under the protection of argon and stirred vigorously by a magnetic stirrer. The color of the reaction mixture turned into brown once the temperature reached ~ 150 °C. The temperature of the reaction mixture was raised from room temperature to reaction temperature of 160 °C or 200 °C in 90 min. The mixture was kept at this temperature for another 40 min before the reaction was terminated by removing the heating source. The ionic liquid in the reaction vessel was collected using a pipette. The product, formed in the form of brownish solid on the flask wall, was extracted by washing with 6 mL of hexane.

Synthesis of platinum nanoparticles. Freshly dried [BMIM][Tf₂N] (5 mL) was mixed with Pt(acac)₂ (25 mg or 0.07 mmol), oleic acid (40 μ L or ~ 0.13 mmol), oleylamine (43 μ L or 0.09 mmol) and 1, 2-hexadecandiol (98 mg or 0.34 mmol) in a 15 mL three-neck flask. This mixture was heated with a heating mantle under the protection of argon and was stirred vigorously by a magnetic stirrer. This mixture turned into a transparent bright yellowish solution after 1, 2-hexadecandiol and Pt(acac)₂ dissolved at ~ 75 °C. The color of the mixture turned into brown once the reaction temperature reached 200 °C, and became dark black at ~ 230 °C. The reaction was kept this temperature for another hour before it was terminated. The subsequent steps were the same as those for making Ag nanoparticles.

Characterizations. TEM and PXRD specimen were made directly from the suspensions of particles in hexane. The TEM images and SAED patterns were recorded on a JEOL JEM 2000EX microscope at an accelerating voltage of 200 kV. PXRD spectra were recorded on a Philips MPD diffractometer with a Cu K α X-ray source ($\lambda = 1.54056$ Å). The thermal analyses of the ILs and nanostructured materials were

conducted using a Q600 SDT DSC-TGA system from TA Instrument. The test runs begun at a heating rate of 10 °C/min in a nitrogen flow at a rate of 100 mL/min till 800 °C. The onset thermal decomposition temperature was picked up in the Universal Analysis (TA Instruments) manually using 300 °C and 500 °C as the start and end points, respectively.

Reference

1. Y. Wang and H. Yang, *J. Am. Chem. Soc.*, 2005, **127**, 5316.

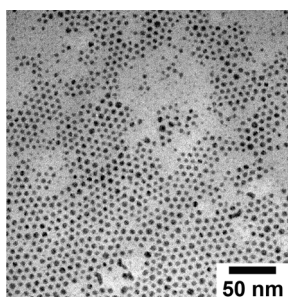


Fig. S1 TEM image of silver nanoparticles made at silver triflouracetate/oleic acid molar ratio of 1 : 3. The reaction was conducted at 200 °C for 40 min.

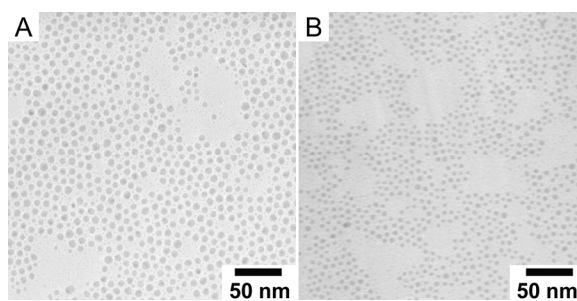


Fig. S2 TEM images of silver nanoparticles made at silver triflouracetate/oleic acid molar ratio of (A) 1 : 3 and (B) 1 : 6, respectively. The reaction was conducted at 160 °C for 40 min.

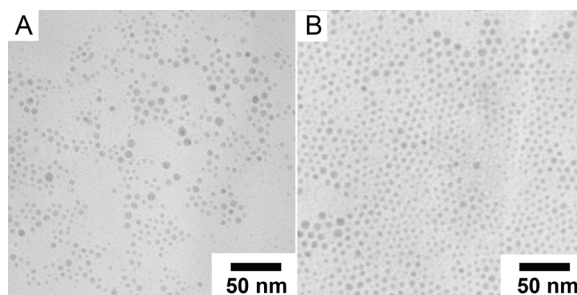


Fig. S3 TEM images of silver nanoparticles made at silver triflouracetate/oleic acid molar ratio of 1 : 3 at (A) 200 °C and (B) 160 °C for 120 min.