

Preparation of imidazolium-group-containing cyclic siloxane indicating ionic liquid nature

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Abstract

Ionic liquids have been widely studied for their remarkable potential. However, little has been reported regarding the preparation of ionic liquids containing inorganic frameworks. Recently, cage-like oligosilsesquioxane (POSS) with an ionic liquid nature was developed by Chujo et al.¹ More recently, we have also reported the preparation of ionic liquids containing random- and cage-structured oligosilsesquioxanes by the hydrolytic condensation of quaternary ammonium-group-containing and imidazolium-group-containing organotrialkoxysilanes using superacid catalysts, such as bis(trifluoromethanesulfonyl)imide (TFSI).²

In this study, as a new siloxane based ionic liquid, imidazolium-group-containing cyclic siloxane was prepared by the hydrolytic condensation of 1-[3-(dimethoxymethylsilyl)propyl]-3-methylimidazolium chloride (DSMIC) using superacids, such as TFSI and trifluoromethanesulfonic acid (TFSA), as catalysts.³

Imidazolium-group-containing cyclic siloxane with TFSI anion (Im-CyS-IL-TFSI) was prepared by the following procedures: DSMIC was stirred in water/methanol mixed solvent of TFSI at room temperature. The resulting solution was heated in an open system until the solvent completely evaporated. The resulting crude product was heated at 100 °C for 2 h, washed with water, and then dried to obtain Im-CyS-IL-TFSI. ¹H NMR, ²⁹Si NMR, and MALDI-TOF MS results of Im-CyS-IL-TFSI indicated that this product was a mixture of cyclic tetra- and pentasiloxanes. Im-CyS-IL-TFSI showed fluidity at 0 °C.

On the other hand, when the hydrolytic condensation of DSMIC was performed using aqueous TFSA, we also found that ionic liquid containing cyclic tetra-, penta-, and hexasiloxanes (Im-CyS-IL-TFSA) was obtained. Im-CyS-IL-TFSA showed obvious fluidity over 20 °C.

References

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