


Experiment title:

SAXS/WAXS characterization of new family of liquid Ionic crystals

Experiment number:
CH-3679

Beamline:

ID02

Date of experiment:

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Shifts:

9

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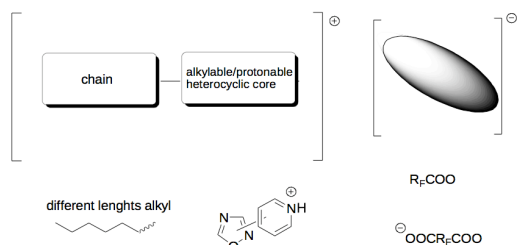
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Report:

The samples were heated until free flowing and put inside a 1 mm-thick glass capillaries. The capillaries were put inside a Linkam hot stage apparatus to control the temperature with approximately 0.1 °C precision. The angular range was set to cover a scattering vector range of approx. 0.009 to 0.5 Å⁻¹ in the SAXS region. A WAXS detector was also used to cover the wide angle part up to 4 Å⁻¹.



The protic ionic liquids (PILs) had the following structure. The cation was characterized by the presence of an elongated or angular oxadiazolyl-pyridinium (3- or 5-substituted regioisomer with an alkyl chain, either C₇H₁₅ or C₁₁H₂₃). The counterion was one of the following: perfluorooctanoate, or a perfluorinated dicarboxylate, either [OOC-(CF₂)₆-COO]²⁻ or [OOC-(CF₂)₈-COO]²⁻. The total number of

samples was therefore 24. Each samples underwent two complete temperature cycles from 10 to 150 °C and back at 10 °C/min speed: no hysteresis was shown in both SAXS and WAXS patterns. In general, the low-temperature form displayed diffraction peaks in both the SAXS and WAXS regions, indicative of crystalline structures. On heating, these evolved into viscous ionic liquid forms, with only one broad correlation peak appearing in the SAXS region. In the WAXS region, the diffraction peaks were progressively broadened and lowered

until disappearing completely into broad oscillations. This general behavior was displayed by all samples. The different nature of substituent chains, spacers and counterions affected the transition temperature.

In one group of samples, with angular pyridinium and long alkyl chains, recrystallization upon cooling was delayed: the effect was less pronounced for the monovalent salt, which recrystallized after about 1' and much more pronounced for the two divalent salts with C_6F_{12} and C_8F_{16} chains (see figure on the right hand side), which recrystallized after 30' and 50', respectively.

The complete data analysis is currently underway. The results on the group of samples referenced above which does not recrystallize immediately at room temperature will be included in a manuscript under preparation to be submitted to Chem. Comm.

