Experimental report

07/12/2021

Proposal:	**6-03-459**	**Council:**	4/2021
Title:	INFLUENCE OF WATER ON THE DYNAMICSOF A PROTIC IONIC LIQUID		
Research area:	Physics		

This proposal is a new proposal

Main proposer:	**Filippa LUNDIN**
Experimental team:	Filippa LUNDIN
Local contacts:	Peter FALUS

Samples: 1-butylpyrrolidinium bis(trofluoromethanesulfonyl)imide + 3.8 wt% D2O
1-butylpyrrolidinium bis(trofluoromethanesulfonyl)imide + 2 wt% D2O
1-butylpyrrolidinium bis(trofluoromethanesulfonyl)imide

Instrument	Requested days	Allocated days	From	To
WASP	5	5	16/06/2021	21/06/2021

Abstract:

We propose to investigate the dynamics in a new class of electrolytes know as water-in-protic ionic liquids (water-in-PILs). In these systems a nano-segregated structure (length scale 1-10 nm) is formed where aqueous domains coexist with apolar and polar domains of the ionic liquid. On the macroscopic level an increase in transport properties is found and is proposed to be linked to local ion-ion interaction. The aim of this experiment is to address the influence of water on the local cation ion dynamics and its link to the macroscopic dynamics. With the WASP instrument we directly access the relevant time and length scales (ps-ns and Å-nm) and with selective deuteration we can directly focus on the cation dynamics.

Influence of water on the dynamics of a protic ionic liquid

Experimental report

Beamtime 16-21/6 - 2021

Proposal number: 6-03-459
Main proposer: Filippa Lundin
Co-proposer: Aleksandar Matic, Timo Stettner, Andreas Balducci
Local contact: Peter Falus
Instrument: WASP

In this experiment we investigate the dynamics in a new class of electrolytes known as water-in-protic ionic liquids (water-in-PILs). In these systems a nano-segregated structure (length scale 1-10 nm) is formed where aqueous domains coexist with apolar and polar domains of the ionic liquid. On the macroscopic level an increase in transport properties is found and is proposed to be linked to local ion-ion interactions. The aim of this experiment is to address the influence of water on the local cation dynamics and its link to the macroscopic dynamics.

The investigated system consist of the protic ionic liquid $Pyr_{H4}TFSI$ (1-butyl-pyrrolidinium bis(trofluoromethanesulfonyl)imide) diluted with 0, 2.0 and 3.8 wt% D_2O. Due to the deuteration of the water, the cation, Pyr_{H4}, is the only hydrogen containing component and therefore dominates the signal (out of the total scattering >89% is incoherent scattering from the cation). Two wavelengths, 5Å (detector angle 68º) and 7Å (detector angle 38º), were used to cover a larger time/momentum transfer (Q) range. Measurements were done in a temperature range of 280-360 K using the orange cryostat. Resolution was measured with TiZr and a measurement of an empty and a cadmium covered annular can was used for background subtractions.

Figure 1 shows the temperature dependence of the normalised intermediate scattering function for the neat protic ionic liquid $P_{H4}TFSI$ measured at 0.82 $Å^{-1}$ using the two wavelengths 5 and 7Å. The overlap from the two wavelengths is good, which indicates that we can directly combine the data measured at the different wavelengths. It is clear that the WASP spectrometer provides good data quality and that we can clearly capture the dynamics in all samples. As expected for liquids, a strong temperature dependence is found for the dynamics, with faster dynamics at higher temperatures.

Figure 2 shows the normalised intermediate scattering function obtained in the experiment for the samples measured at 300 K at various Q-values. A strong Q-dependence is found, a signature of a diffusive nature of the dynamics. Looking at the different D_2O-concentrations it can be seen that the relaxation is faster in the samples with more D_2O, this is especially clear at low Q-values. From a first preliminary analysis we can quantitively correlate the local dynamics with the macroscopic conductivity, i.e. faster dynamics with the introduction of the D_2O.

A detailed analysis of the spectral shape, i.e., the nature of the relaxation function, will be performed to identify the detailed nature of the relaxation processes contributing to the scattering. The results from the WASP experiment will be combined with conductivity data in order to span from local molecular/ionic relaxations to macroscopic ion conductivity to better understand ion transport in the water-in-PILs.

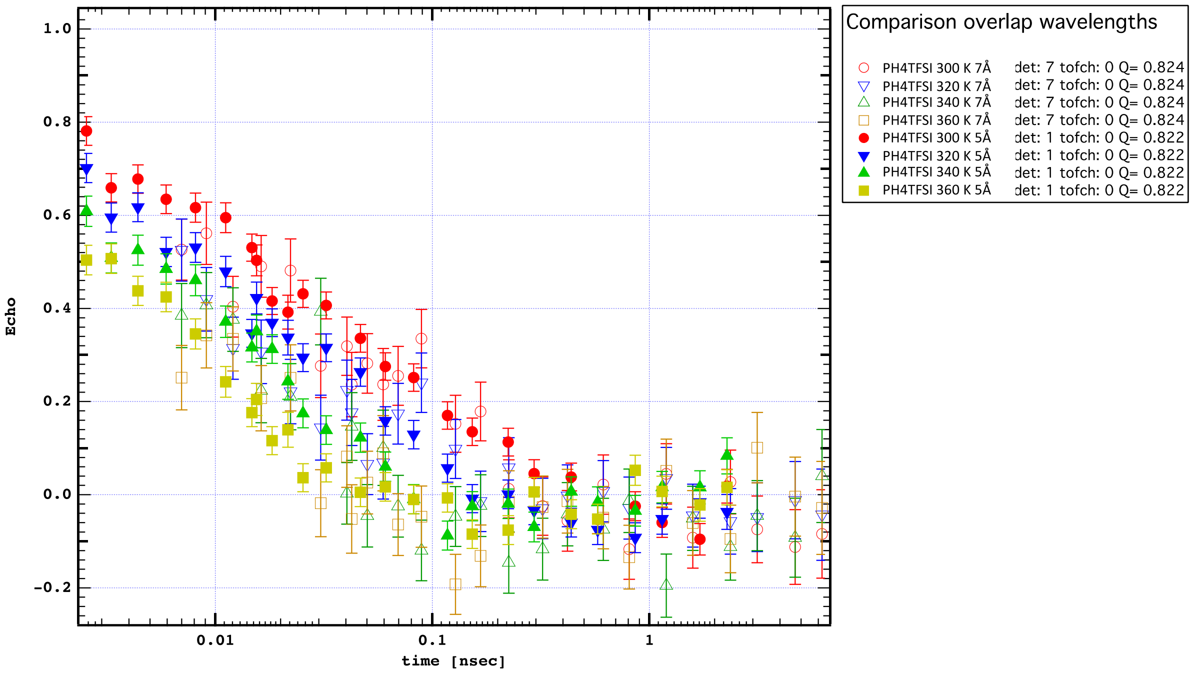

Figure 1 Normalized intermediate scattering function for P$_{H4}$TFSI at 0.82 Å^{-1} as a function of temperature measured at wavelengths 5 and 7Å.

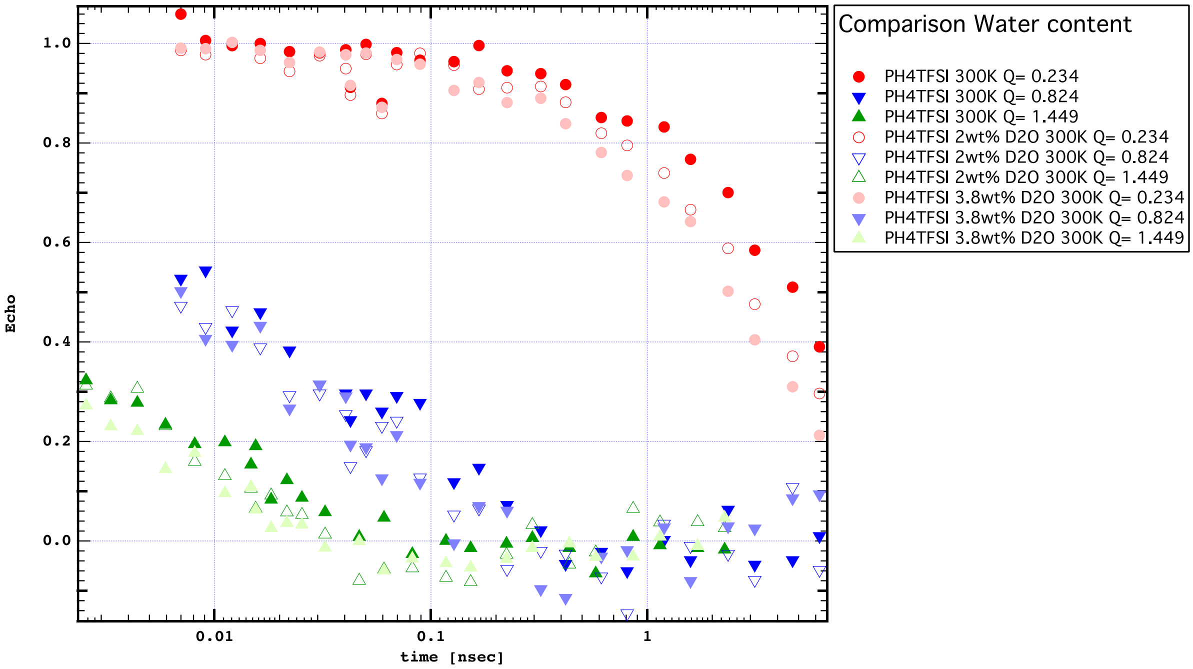

Figure 2 Normalized intermediate scattering function for different D_2O concentrations at 300 K measured at Q=0.23, 0.82 and 1.45 Å^{-1}.