Water-induced mica/ionic liquid interfacial nanostructure switches revealed by AFM

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Experimental section:

AFM force curve measurements were performed on an Agilent 5500 SPM. Gold-coated silicon probes (NT-MDT) with a typical force constant 0.01 N/m and a typical curvature radius 35 nm were employed. BMPTFSI were purchased from IoLiTec in the highest available quality (99%) and kept in the glove box filled with high-purity argon (99.999%, Linde Industrial Gas). All ILs were vacuum dried at 80 degrees Celsius to further remove the trace water and argon, then ultra-pure water (18.2 MΩ cm, Merck Millipore) was added and stirred to produce different water-contained ILs. Mica was purchased from EMS at the highest Tedpella V-1 level. Before each measurement, the top-most mica layer was peeled off to expose fresh surface, and the ILs were dropped onto the mica surface rapidly. AFM force curves were obtained within 30 min to avoid a significant change of the water content[1]. Afterwards, the water concentration was measured by Karl Fischer Coulometer (KF-831, Metrohm). To avoid intake of water from other sources, AFM probes and scanner were vacuum dried for at least 4 h, and all measurements were conducted in the argon-filled glovebox.

Statistical section:

F-test was adopted to test the goodness of fit.

The null hypothesis:

$$H_0 : A_H = W' = 0 \quad (i.e. F = 0)$$

and the alternative hypothesis:

$$H_A: A_H \neq 0, W' \neq 0 \; and \; \kappa^{-1} \neq 0 \; (at \; least \; one \; hold)$$

The statistics results were listed in following table:

Water content (ppm)	A_H (nN nm2)	W' (nN)	κ^{-1} (nm)	χ^2	***F***	***p (Prob>F)***	***R-square***
43	0.0456±0.0092	2.9607*	0.02*	4.55×10^{-6}	8.17**	2.72×10^{-5}	0.00355
`478	0.0557±0.1059	0.0059±0.0051	13.53±7.6930	2.08×10^{-5}	19.22**	1.17×10^{-11}	0.01576
2356	0.0811±0.0791	0.0048±0.0023	40.58±36.406	1.65×10^{-5}	60.06**	4.77×10^{-32}	0.05386

*: not given; **: at significant lever of 0.01

The test statistics *F*, following the *F* distribution with degree of freedom 3 and 398, are 8.17, 19.22 and 60.06. All *p*-value are smaller than 0.0001. Therefore, we reject null hypothesis at the significant level of 0.01. That is, we accept our model parameters at the significant level of 0.01.

Note that R-squares are close to zero and the derivations of the parameters are not considerably small, these are mainly because our data are not in the same scale: dependent variable(Force) is at the scale of 0.01 and varies weakly, while the independent variable (Separation) is at the scale of 10, which is three orders of magnitude larger than the former one; another reason is that the force in this region is weaker and more sensitive, which cause a low Signal-Noise-Ratio, increasing the variation of measured data, reducing the R-square value. We tried binned-averaging (as we mentioned in the text) to reduce the noise, but it remains.

1. H.-W. Cheng, P. Stock, B. Moeremans, T. Baimpos, X. Banquy, F. U. Renner and M. Valtiner, *Adv. Mater. Inter.*, 2015, **2**, 1500159.