	Experiment title:	Experiment number:
	Studies of drying process of proton exchange membrane for fuel cell: reflectivity measurements	SC985
Beamline: ID10b	**Date of experiment**: from: 03/04/02 7h00 to 11/10/02 7h00	Date of report: 03-05-05
Shifts: 18	**Local contact(s)**: N. Galatanu	*Received at ESRF:*

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Report:

The casting of charged polymer, sPI ionomer, was studied using X-Rays Reflectivity. The thickness loss of sPI ionomer solution in m-cresol was recorded in the course of time. In a first step, it decreases linearly with time and the process is ruled by the vapor pressure of m-cresol. The results indicate that the models developed for non-charged polymer drying process may be suitable for ionomer. During this first period, the X-Rays reflectivity signal can be recorded, but when approaching the end of this period, we observed a rapid loss of the X-Rays reflectivity signal. This loss signs a degradation of the surface of the drying film (roughness). A correlation occurs between the drying process of the bulk sample and the structuration of the membrane surface. Moreover, we evidenced a strong influence of the quantity of charges carried by the ionomer chains: the higher quantity, the faster is the drying kinetic.

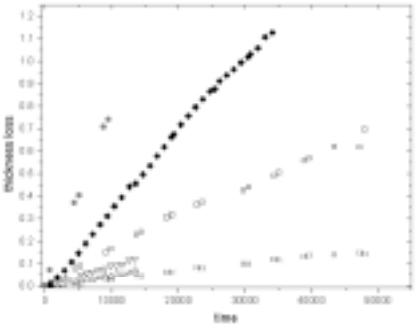

Figure 1: Variation of the thickness loss versus time of sPI (EW=792g/equiv and X=5) ionomer solution of 8wt% for several temperatures: (open circle) 20°C, (up triangle) 42.3°C, (down triangle) 50.9°C, (cross) 53.2°C, (open square) 58°C. The result for a sPI (EW=504g/equiv and X=5) ionomer solution at 55°C has been also plotted (black diamond). For comparison, the water is represented with the grey circle.

a) b)

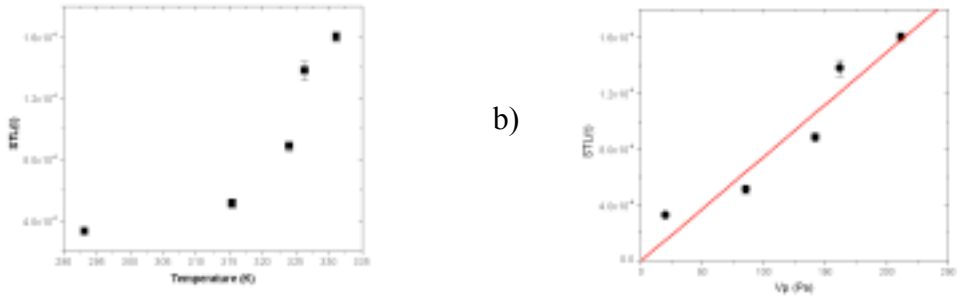

Figure 2: (a) Variation of the slope thickness loss in the course of time of sPI (EW=792g/equiv and X=5) ionomer solution of 8wt% as a function of the temperature. (b) Variation of the slope thickness loss with time of sPI (EW=792g/equiv and X=5) ionomer solution of 8wt% as a function of the vapour pressure of m-cresol.

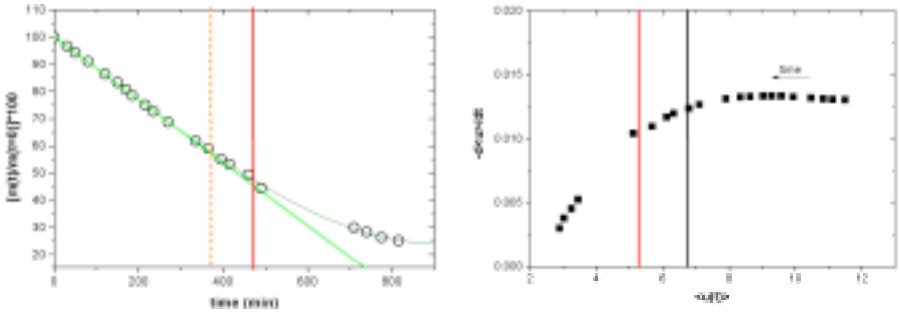

Figure 3: Variation of the weight loss versus time of sPI (EW=792g/equiv and X=5) ionomer solution of 8wt% at 52°C and variation of –d<u>/dt versus <u>. A line is plotted to help the reader to see when the decrease is not linear anymore. The dash line represents the beginning of the degradation of the X-ray signal and the full line represents the limit when reflectivity signal cannot be measured anymore.

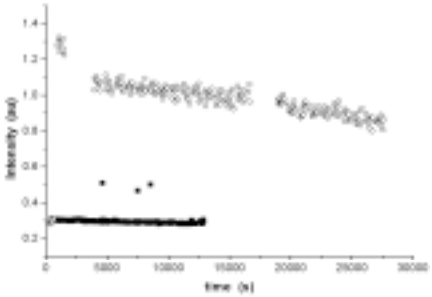

Figure 4: Variation of the intensity of X-ray reflectivity peaks of sPI (EW=792g/equiv and X=5) ionomer solution of 8wt% at 50.9°C (open circle) and 42°C (black square) versus drying time.

Then all these results where correlated with transport measurements performed using NMR and radiotracers techniques and published in J. Chem. Phys. B.