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Measuring SANS and NMR simultaneously: a new tool for transient physics

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Specific properties of soft condensed matter result from the control of subtle interactions which trigger macroscopic variations. In such (macro)molecular assemblies, tuning molecular interactions via external chemo/thermo/photo (…) stimuli is often the key point to control self-aggregation mechanisms and the visible properties (optical, viscosity, color …). Analysis of such multi-scale transformations requires usually a multi-technique approach to probe both structural and dynamical changes. To this respect, Small Angle Neutron Scattering (SANS) is a central tool to probe nanometric structures while Nuclear Magnetic Resonance (NMR) is a versatile technique which can easily probe dynamical information such as local reorientation mechanisms (relaxation times) and self-diffusion coefficients of molecules.

In this talk, we report on an **original setup** which allows to measure simultaneously *in-situ* SANS and NMR using a low-field spectrometer[1]. We illustrate the capabilities of alliancing these experimental methods by following the critical temperature-induced phase separation of a concentrated Poly(Methacrylic Acid) (PMAA) solution at its Lower Critical Solution Temperature (LCST). The characteristic size related to the domain growth of the polymer-rich phase of the gel is monitored by the evolution of the SANS spectra, while the dynamics of the sol phase (H_2O and polymer) is simultaneously characterized by NMR by measuring T_1, T_2 and the diffusion coefficient. A specific cell was carefully deisigned to optimize thermalization of the sample and in particular its equilibration time. The acquisition time needed to reach good signal-to-noise ratios, for both NMR and SANS, match: it is of the order of one hour. Altogether, we show that *in-situ* low-field NMR/SANS coupling the NMR is meaningful and is a promising experimental approach. Such multimodal approach is of major interest when a sample experiences transient physical states or evolves rapidly and/or irreversibly.

a) *b)*

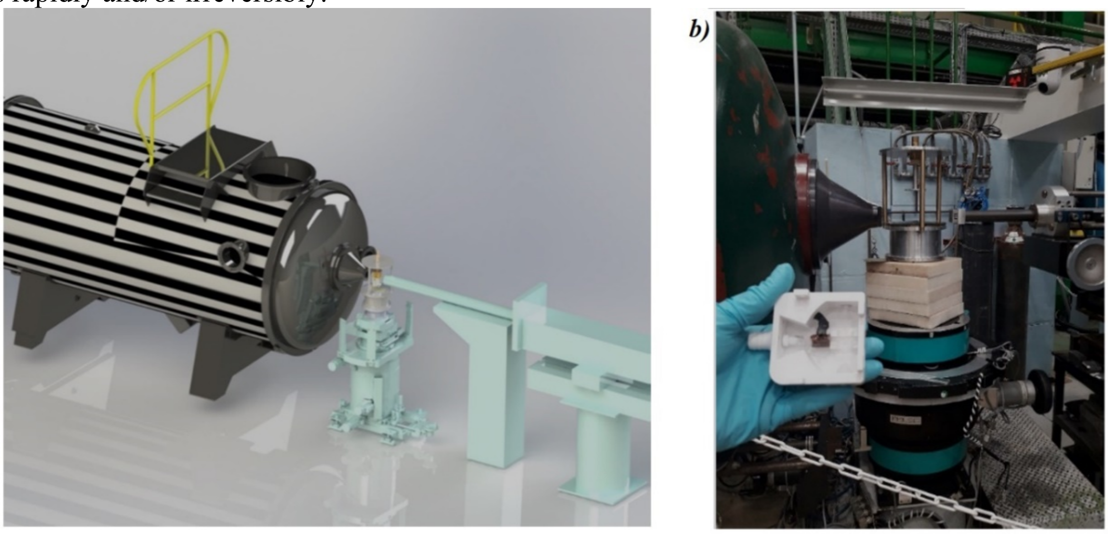

Figure 1 : a) 3D model of the implementation of a low field NMR spectrometer on the LLB SANS instrument PAXY – the one-sided NMR magnet is seen in the middle of the drawing between the neutron guide (in turquoise blue)and the neutron detector box (zebra) (courtesy P. Lavie). b) Real picture of the central part and the NMR-Neutron probehead.

[1] R. de Oliveira-Silva, A. Bélime, C. Le Coeur, A. Chennevière, A. Helary, F. Cousin, P. Judeinstein, D. Sakellariou, J.-M. Zanotti, *J. Neutron Research*, (2019) in print.