ESRF	**Experiment title: SAXS Investigation of Phase Transitions in Liquid Crystal Polymers confined in Silica Networks**	**Experiment number**: 02 01 640
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Shifts: 6	**Local contact(s)**: Françoise Bley	*Received at ESRF:*

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Report:

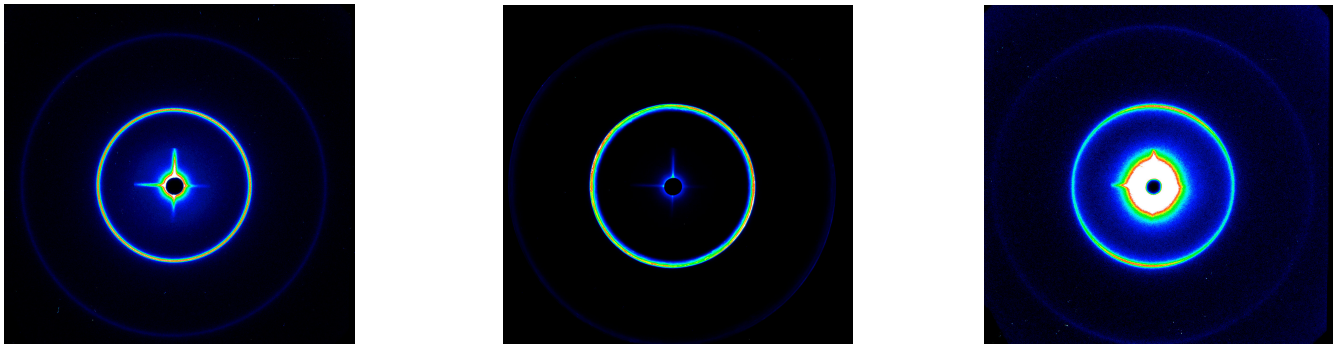
The aim of this SAXS experiment was to investigate the structure of a series of side chain liquid crystalline polymers (SCLCP) at different temperatures and under different conditions:
- as received SCLCP, i.e., polymerised in solution (AIBN) and dried (*initial SCLCP*)
- SCLCP that have been heated above their isotropic temperature (near 160°C) before being placed in the measuring cell (*heated SCLCP*)
- SCLCP confined in silica aerogel by impregnation in the isotropic phase (*aero-SCLCP*)
- SCLCP confined in silica xerogel: SCLCP is solubilized in the gelling solution of silica (solvent THF) and the final gel is dried under subcritical conditions yielding a xerogel (*xero-SCLCP*).

In order to measure the Bragg peaks of the SCLCP with a high resolution, the following experimental arrangement was used:
- the energy is set to 12.398 keV (λ=1 Å)
- the sample to detector distance is close to 82 cm
- vertical and horizontal gap of slit 1 were set to 1 and 3.5 mm, respectively.
An indirect illumination CCD camera (Princeton) with image size 1340x1300 pixels^2 was used.

Typical 2d patterns (Figure 1) display rings characteristic of a powder pattern. This feature reveals randomly oriented crystalline domains. Meanwhile, in some cases, the intensity along the ring goes through two maxima that indicates a better orientation. In these conditions, the degree of ordering can be obtained by determining the variation of the peak intensity as a function of the azimuthal angle (Figure 2). To this end, the software used for radial averaging needed to be modified. Furthermore, it was necessary to improve some corrections already used in the software aiming to take into account imperfections inherent in these CCD

detectors. This task has been performed by Jean-François Berar. Analysis of the data is in progress. An example of a first level typical information is given below.

Figure 1 and Figure 2 show the 2d patterns and their azimuthal analysis respectively, obtained for a given SCSCP sample, PCL2, which molecular structure in indicated in Figure 3. DSC measurements have shown that the isotropic temperature of this sample is quite high (near 150°C). At 60°C, PCL2 is in a smectic crystalline phase. For the sample obtained by impregnation of a silica aerogel (*aero-SCLCP*, not shown), only the second peak is clearly visible, the first peak being partly hidden by the silica aerogel scattering. These figures indicate that the ordering of the liquid crystalline phase of PCL2 grown from melt (*heated SCLCP*) is poorer than when it is grown from a solution (*initial SCLCP*, *xero-SCLCP*).



heated SCLCP (isotropic) after heating above T_{Sm-I} (170 °C)

initial SCLCP (anisotropic)

xero-SCLCP (anisotropic) added in the sol-gel reaction bath

Figure 1. SAXS patterns of PCL2 at 60 °C

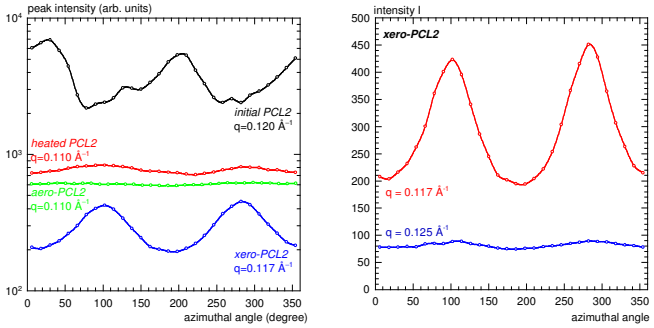


Figure 2. Azimuthal analysis of the patterns.

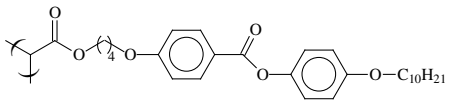


Figure 3. Molecular structure of PCL2 (Mw = 13531)

<u>Preliminary conclusions (working hypotheses)</u>

1) It is possible to confine SCLCP in a silica network by adding SCLCP in the gelling solution
2) It seems that ordering of the LC phase is more pronounced when it is formed in solution than from the melt.