ESRF	**Experiment title:** Multiscale characterisation of organic gels, aerogels and carbon aerogels by SAXS.	**Experiment number**: SC-677
Beamline: BM2	**Date of experiment**: from: January 28, 2000 to: February 1, 2000	**Date of report**: August 2000
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Report:

These experiments aimed to characterize multiscale morphology of organic gel precursors and supercritically dried aerogels, before and after pyrolysis, in the context of a CNRS supported project (Programme Matériaux 1999, Projet N°20c) and as a part of the PhD thesis of Olivier Barbieri. The influence of solvent (water or organic solvent) and catalyst (acidic or basic) on the structure of gels of resorcinol-formaldehyde (RF) or phenol-furfural (PF) was investigated. The second series of parameters investigated dealt with aging and conditions of supercritical drying and pyrolysis. In the case of aerogels, information obtained by SAXS was compared to that deduced from classical methods of measuring surface area and pore size distribution. For some RF samples, the sol-gel reaction and the gel structure were also investigated by quasi-elastic light scattering.

USAXS measurements were made at the BM2 beamline. The incident beam section was limited by the defining slits at 10 m to 200µm×200µm, the curvature of the second mirror and the monochromator being adjusted to provide a mildly focused beam in both the sagittal and the vertical directions to 100µm×100µm at the sample. The incident energy was 7.8 keV. To reduce unwanted scattering from windows, a differential pumping system was employed, with a 10m long window-free path for the beam between the second mirror and the sample. Specimens were held in cells fixed on a sample changer open to the atmosphere, the air gap being less than 5 cm. In order to measure the scattered intensity over a broad range of q values, two distances between sample and detector were used (2.3 m and 0.54 m). The beamstop, a small pillar of 1 mm diameter gold wire, was fixed to a Kapton foil just upstream of the flight tube exit window. An indirect illumination

CCD detector (Princeton Instruments), cooled by a Peltier effect device, with 42.5 µm pixel size was used. In this configuration the minimum value of the transfer wave vector *q* was close to 2×10^{-3} Å^{-1}.

Among the many experimental results and new and important information which was already deduced from them, one may quote the following.

1) Comparison of the texture of the gel and of the aerogel (before and after pyrolysis yielding a carbon aerogel) was performed for several series. The figures shown below summarize the results obtained for RF samples prepared in water and base catalysis (WB series) or in acetone and acid (AA series) or base (AB series) catalysis. The concentration of catalyst is determined by the ratio R/C (molar ratio between resorcinol and catalyst). It was equal to 200 for the WB and AA series and to 50 for the AB series (in order to have similar gelation times). The mass percent M was equal to 5% for the former and to 15 % for the latter (gelation did not occur at a lower mass percentage). In all cases, pyrolysis induces the appearance of nanopores to which the change of regime in the high-*q* range can be attributed.
This part of the experimental work will be presented orally at the 6th International Symposium on Aerogels (Albuquerque, 8-11 October, 2000) and submitted as a publication in the Proceedings.

2) The influence of the mass percentage on the structure of the gels and of the subsequent polymer and carbon aerogels was investigated. It appears that the most dramatic changes appearing for larger M values are observed for the AA series, for which the fractal features remain only in the gel but disappear in the aerogel.

3) Analysis of the nanoporosity in the pyrolyzed samples is under progress. An interesting part will be the evolution of the nanoporosity as a function of the pyrolysis temperature and its comparison with information deduced from gas adsorption measurements.

4) Analysis of the data obtained for other series of gels and aerogels (*e.g.* phenol-furfural gels and aerogels) is under progress as well as analysis of the reproducibility of the structure for different batches.