



Experiment title: Effect of surface functionalization on a well defined activated carbon

Experiment number:
SCII73

Beamline:	Date of experiment: from: 06 February 2003 to:11 February 2003	Date of report:
Shifts:15	Local contact(s): E. Geissler	<i>Received at ESRF:</i>

Names and affiliations of applicants (* indicates experimentalists):

*László, Krisztina, Department of Physical Chemistry, Budapest University of Technology and Economics, Budapest

*Kosik, Katalin Department of Physical Chemistry, Budapest University of Technology and Economics, Budapest

*Ehrburger-Dolle, Françoise, Laboratoire de Spectrométrie Physique, Université J. Fourier de Grenoble

*Rochas, Cyrille, Spectro, Université J. Fourier de Grenoble

*Geissler Erik Spectro, Université J. Fourier de Grenoble

*Livet Frédéric. LTPCM. INPG. Grenoble

Report: Measurements of the scattering pattern from activated carbon samples were made at two different incident energies, 8 keV and 18 keV, and with several sample detector distances, 30 cm, 1m60 and 2m10. The carbon samples, previously functionalized to different degrees with nitric acid, were bathed in various organic solvents: hexane, toluene, nitrobenzene and bromobenzene. The resulting data covered the q range from about $7 \cdot 10^{-4} \text{ \AA}^{-1}$ to 1.3 \AA^{-1} . Exposure times ranged from 0.4 s to 200 s. To avoid the unwanted fluorescence from bromine, measurements were also conducted at 13 keV. In spite of this precaution, it turned out that the signal-to-noise ratio from the bromobenzene samples was too weak to allow a proper comparison with the other spectra. For the other samples, however, the signals were strong and consistent.

All samples displayed an extended region at low q with a slope of about -3.6, characteristic of scattering from rough surfaces. Comparison of the signal from each sample immersed in the different solvents leads to the conclusion that the effects of multiple scattering are small and confined to the lowest values of q , where an apparent tendency toward plateau behaviour is visible in the dry specimens. This means that the size of the entities giving rise to the low q surface scattering is larger than the experimental resolution, i.e. >0.5 micrometers. A typical combined data set is shown in figure 1 for an activated sample previously subjected to 3 hours of nitric acid treatment at room temperature.

The unactivated carbon (KPET) in the dry state showed a structure similar to that of the activated samples (APET). In presence of the solvents, or even when simply exposed to hexane vapour, the intensity of the shoulder feature in the intermediate q range ($q > 0.1 \text{ \AA}^{-1}$) is strongly reduced. This indicates that soluble residues in the micropores, such as tars, dissolve and the contrast between the carbon structure and the liquid decreases.

In the APET samples, however, the micropore structure is maintained in the presence of solvent.

When hexane vapour is introduced, the scattering intensity remains similar to that of the dry sample in the low q region, i.e. the surface scattering from the large structural units is unchanged. At higher q the scattering curve from the hexane vapour-containing sample falls below that of the dry sample and merges with that from the sample in contact with liquid hexane. It follows that the hexane vapour has condensed into all the available space in the micropores. At the highest values of q explored, all the curves tend to converge, showing that the scattering contrast from the smallest cavities, where the solvent molecules fail to penetrate, is insensitive to the presence of solvent molecules. The apparent radius of gyration (ca 5-6 Å) deduced from the shoulder around $q=0.3 \text{ \AA}^{-1}$ decreases as the electron density of the solvent increases. This is probably due to loss of signal from the pore structure as it fills partially with the solvent molecules. Comparison between the scattering intensities shows that, on the resolution scale of 0.1 \AA^{-1} (i.e. the plateau), the effective density of the carbon is close to 2 g/cm^3 , in agreement with macroscopic helium density measurements on the same samples.

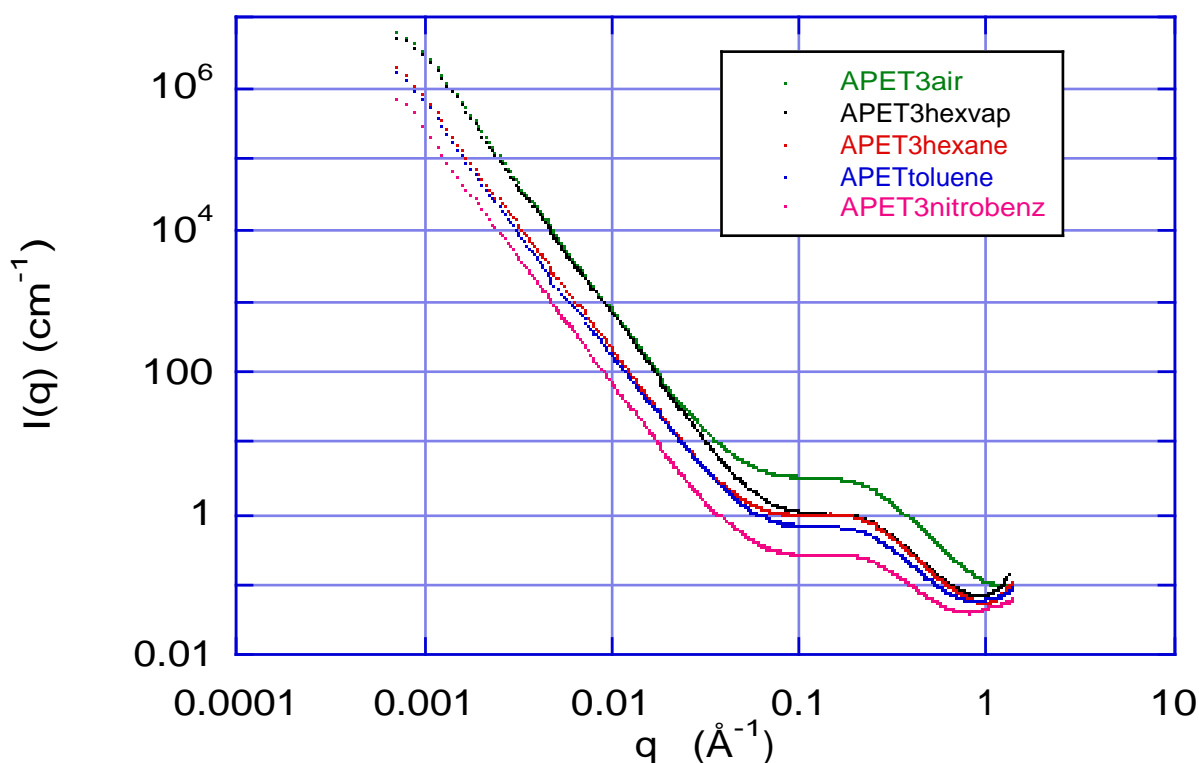


Figure 1 Combined SAXS curves from sample APET3, an activated carbon prepared from poly(ethyleneterephthalate), exposed to HNO₃ at room temperature for 3 hours, in different solvents. The low q response of the sample in contact with hexane vapour (black curve) coincides with that of the dry material (green), while at $q > 0.1 \text{ \AA}^{-1}$, it coincides with that of the sample immersed in liquid hexane (red).