	Experiment title: Water vapour adsorption on surface-functionalized microporous carbons	**Experiment number**: 02 01 696
Beamline: BM2	**Date of experiment**: from: 19-04-2006 to: 23-04-2006	**Date of report**: 07-08-2006
Shifts:12	**Local contact(s)**: Cyrille Rochas	*Received at ESRF:*

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Report:

Measurements were made of the scattering response of activated carbons derived from poly(ethylene terephthalate) with three different degrees of surface oxidation (O/C atomic ratios: 6%, 9% and 26%), as a function of partial water vapour pressure. The wave vector range explored was 8 $10^{-3} \leq q \leq 6$ Å^{-1}. A preliminary analysis of the data is made in the pseudo-binary approximation in which the water phase is assumed to be continuous in the micropores of the carbon. The intensity ratio $u(q)=I_{wet}(q)/I_{dry}(q)$, where $I_{dry}(q)$ is the signal from the dry reference sample, yields the occupation probability $p(q)=(\rho_C/\rho_{H2O})[1-u^{1/2}]$, where ρ_C and ρ_{H2O} are the electron densities of the carbon and of water.

Figure 1

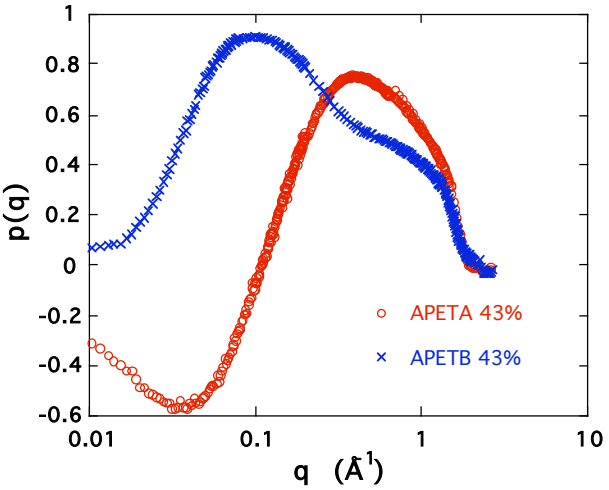
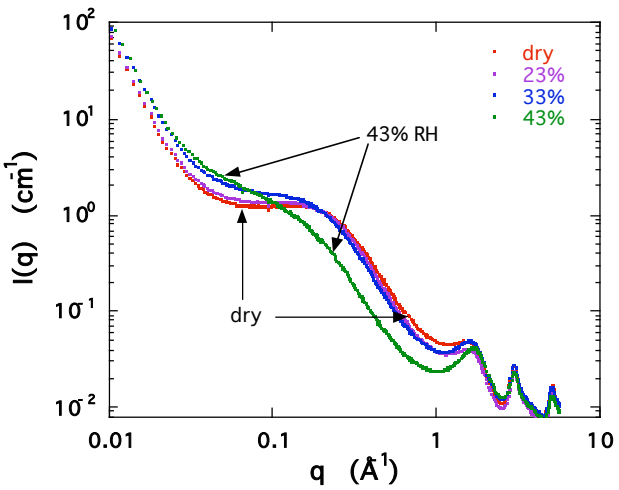


Figure 2



The results depend strongly on the degree of surface oxidation of the samples.
Figure 1 compares the calculated values of $p(q)$ for the intermediate oxidized sample (APETA) at 43% relative humidity with that for the most highly oxidized sample (APETB) at the same partial pressure. While APETB shows incomplete filling in the smallest pores (ca. 50%) and improved but still incomplete filling (ca 90%) in the larger pores, for APETA the values of $p(q)$ become negative in the lower q range, a result that is inconsistent with the model.
Figure 2 shows the explanation for this phenomenon: with increasing partial vapour pressure the scattering intensity $I_2(q)$ decreases at $q>0.2$ Å^{-1} in this sample, but below this value of q the intensity exceeds that of the dry sample. The extra scattering demonstrates the presence of water clusters, i.e., the system becomes ternary with significant water-air interfaces in the lower q-range. The deviation of the scattering curves from that of the dry sample show that a broad distribution of cluster sizes is present, with an average of approximately 30 Å.

The above results were the subject of a keynote lecture given at the Conference *Carbon 2006*, Aberdeen July 2006. (László, K., Geissler E. SAXS characterization of solid/vapor interfaces in microporous carbons with different surface chemistry, lecture 4D1).