



Experiment title: Distribution of transition metals in carbon aerogels

Experiment number:
CH2636

Beamline:
BM2

Date of experiment:
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07-09-2008

Shifts:12

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Received at ESRF:

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Report:

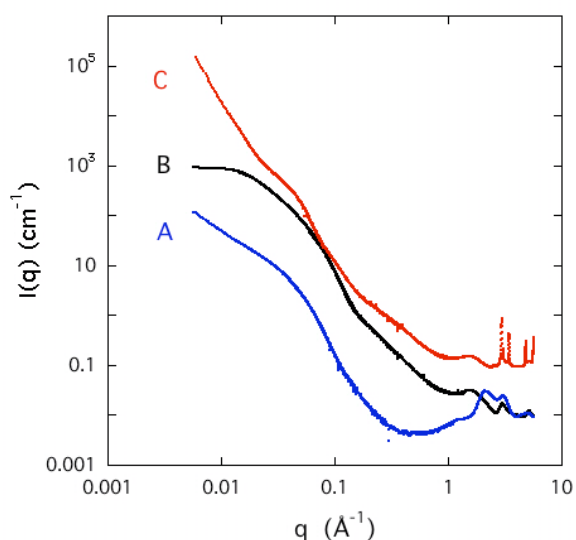


Figure 1. SAXS/WAXS spectra of copper-doped RF networks; A: wet RF gel catalysed with CuAc; B: catalysed with CuAc, then carbonized; C: carbonized after adding CuAc to network.

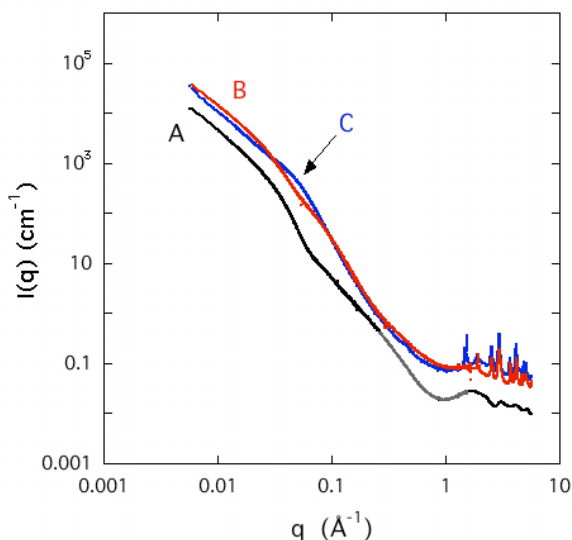


Figure 2. SAXS/WAXS spectra of titanium-doped RF networks; A: carbonized RF network with added TiOx; B: network catalysed with TiOx then carbonized; C: RF network with added TiOx, then carbonized.

The objective of this experiment was to determine the structure of carbonized resorcinol-formaldehyde (RF) aerogels doped with transition metal compounds with useful

catalytic properties. Comparisons were made between networks prepared with sodium carbonate as a polymerization catalyst and those with copper acetate (CuAc) and titanium oxalate (TiOx). These compounds were also added at different stages of the process, before and after carbonization. As it was not feasible to operate conveniently in ASAXS conditions at the time of this experiment, we worked at fixed energy 16 keV. Some of the missing ASAXS information could be compensated for by analysis of the WAXS scattering region.

Figures 1 and 2 show samples of the spectra obtained. When CuAc is used as a catalyst (Figure 1), it modifies the underlying carbon structure compared to catalysis with sodium carbonate, particularly in the region of short range order, but does not remain in the network after washing. Metallic copper is formed if the acetate is introduced before carbonisation. From the width of the diffraction peaks, an approximate size of 20 Å was estimated for the copper particles.

With titanium oxalate (Figure 2), titanium hydroxide is formed immediately in the polymerisation process. Upon carbonization, titanium oxide is generated, principally as rutile, with a small admixture of anatase. The ratio of these two forms is expected to depend on the temperature of carbonization. Unlike copper, titanium modifies the network structure only weakly.

Some of these results have already been communicated in conference proceedings

1 Czakkel O., Székely E., Szilágyi I.M., Geissler E., László K. SAXS study of transition metals in resorcinol - formaldehyde derived carbon aerogels *10th International Symposium on Particle Size Analysis, Environmental Protection and Powder Technology*, August 27—29, 2008 Debrecen, Hungary (oral presentation)

2 O. Czakkel, E. Székely, I.M. Szilágyi, E. Geissler, K. László Distribution of transition metals in carbon aerogels *22nd Conference of the European Colloid and Interface Society* August 31 - September 5, 2008 Cracow, Poland (poster)