ESRF	**Experiment title:** USAXS analysis of long range structure in weakly aggregating and network forming dispersions	**Experiment number:** SC78
Beamline: ID2 - BL4	**Date of experiment:** from: 15:00,28/8/95 to: 23:00,29/8/95	**Date of report:** 8/96
Shifts: 4	**Local contact(s):** Peter Bösecke	*Received at ESRF:* 28 FEV. 1997

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Report:

1. Purpose of measurements

The purpose of the proposal was to investigate in one experiment the complex aggregate structure of industrial carbon materials from the nanometer scale up to the micron scale. Two types of materials were investigated with the 10 m pin-hole camera: (1) polymers with a dilute second continuous phase of aggregated conductive carbon black (0.08-2.0 wt%), and (2) heavy-oil dispersions, with colloidally aggregated asphaltene molecules.

2. Results.

Typical data sets from the polymer/carbon-black compounds are shown in figure 1. Three main features are clear. (a) There is a clear signal equivalent to d=30 nm in all curves, reflecting the size of the monodisperse primary carbon black particles, accurately known from transmission electron microscopy. (b) The intensity scales at all lengths exactly with carbon-black concentration, which suggests that aggregates of at least ca. 300 nm are the real primary building blocks of the network. Moreover, this intensity closely follows that of the polymer background beyond 30 nm. The concentration-dependent build-up of the percolating network apparently will only be visible around the micron scale. This is in line

with ideas on the network structure as derived from AC impedance spectroscopy (to be published). Imaging of that build-up with USAXS needs the use of the Bonse-Hart camera.

Figure 2 shows the data for asphaltenes in toluene, at two representative concentrations. As with the carbon blacks, the intensity scales with concentration, so it reflects the individual stable colloidal aggregates. In the high-q regime the slopes of the logarithmic I vs. q plots tend to a value -2; this value is actually seen with normal SAXS around 1 nm^{-1}, and shows the presence of the individual flat polycyclic aromatic asphaltene molecules. The intensity in the low-q regime contains information on how the asphaltene unit sheets are packed together in aggregates. We are in the process of developing models to accurately derive this information. However from the present data it is already clear that to obtain information on the structure at the upper length scales the 300 nm resolution is not enough, and that again the use of the Bonse-Hart camera is essential.

3. Conclusion.

The experiments performed so far on aggregated carbon have been very useful to show the presence of structures at several length scales. This kind of information is industrially important since it reflects both the conditions under which the structures were formed, and the material properties of the aggregates. However, to obtain sufficient quantitative information a camera with micron-scale resolution has to be available.

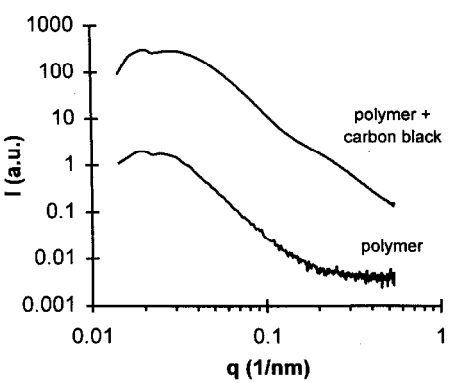

Figure 1. Carbon black/polymer data

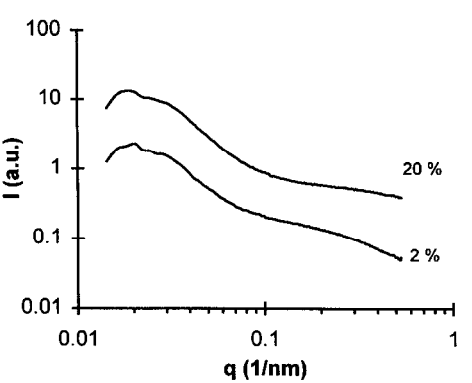

Figure 2. Asphaltenes in toluene