ESRF	**Experiment title:** Characterization of activated carbon fibers, carbon molecular sieves and superactivated carbons for gas storage and pollutant removal, by small-angle x-ray scattering.	**Experiment number**: ME-93
Beamline: ID13	**Date of experiment**: from: 13-sept-00 to: 16-sept-00	**Date of report**: 6-July-01
Shifts: 9	**Local contact(s)**: Dr. Martin MÜLLER	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr. Diego Cazorla-Amorós*

Dr. Encarnación Raymundo-Piñero*

Miss Dolores Lozano-Castelló*

Miss Mª Ángeles Lillo-Ródenas*

Departamento de Química Inorgánica

Universidad de Alicante

E-03080, Alicante, SPAIN

Report:

The porous texture characterization of activated carbons is a very important subject due to the growing interest in the preparation of activated carbon with well defined pore structures and high adsorption capacities. The characterization of porosity is an essential task to foresee their behaviour in a given use. Gas adsorption (mostly N_2 adsorption at 77 K) is the most widely used technique for characterizing porous materials. However this technique has significant limitations as it is only sensitive to the accessible porosity and it is strongly dependent on the experimental conditions used (i.e., temperature and pressure). Significant effort has been already done through the use of other techniques. For example, our research group has worked on characterization of porous texture of diverse carbon materials by techniques like SAXS, SANS, PALS [1-4]. The characterization of the porosity is very difficult and a combination of different techniques is necessary for the assessment of all range of the porosity.

In the work at the ESRF, we have focused on analysing the development of porosity in isotropic pitch-based carbon fibers derived from petroleum pitch. The previous studies showed that the activated carbon fibers (ACF) had a different evolution of porous structure with burn-off depending on the activating gas used (steam and CO_2) [5]. The confirmation of the structural changes produced in the activation process using both activating agents, could be carried out analyzing, with a suitable technique, the porosity development along the fiber diameter. SAXS technique provides information about the structure of matter on a mesoscopic scale. Usually, SAXS experiments in carbon fibers are carried out packing the sample in a cell or using a parallel fiber bundle, depending on the experimental system and the beam diameter. For analyzing the different regions of a single fiber along its diameter, a beam size much smaller than the fiber diameter and with high intensity is needed. The availability at ESRF of X-ray microbeams with sizes down to 2 μm (Microfocus Beamline, ID13), together with a position-resolved X-ray scattering method makes this technique suitable for analyzing single fibers. Up to now, the use of this technique in fibrous materials has been focused on cellulose fibers [6], and carbon fibers [7]. In the work carried out with carbon fibers [7] the internal structure of single carbon fibers, from

differents precursors (PAN-based fiber and mesophase-pitch based fibers) have been investigated. In the literature there is not any report about the use of this technique for the characterization of activated carbon fibers. On the other hand, the use of an area detector (MAR-CCD, active diameter 130 mm) in microfocus beamline has the advantage of having a better angular resolution than a quadrant detector, where the measurements are carried out rotating the sample in the plane normal to the direction of the incident beam.

The experiments performed in ACF with different porosity and origin have been successfully obtained with a single fiber, and important conclusions on the porosity distribution along the fiber diameters have been obtained. As an example, Figure 1 presents the two dimensional scattering patterns corresponding to the original CF and ACF. The ACF included are: CFC29, CFC50 (that is ACF prepared by CO_2 activation to a 29 and 50 % burn-off) and CFS48 (that is ACF prepared by steam activation to a 48% burn-off). The scattering intensity is related to the porosity development; thus, a stronger scattering means higher porosity. Then. it can be observed that the scattering intensity increases with the burn-off degree, accordingly with the development of porosity (i.e., samples CF, CFC29 and CFC50) and that the activation with CO_2 develops more porosity than steam (i.e., samples CFC50 and CFS48). Addtionally, the isotropic SAXS patterns obtained indicate that the prosity is isotropically distributed.

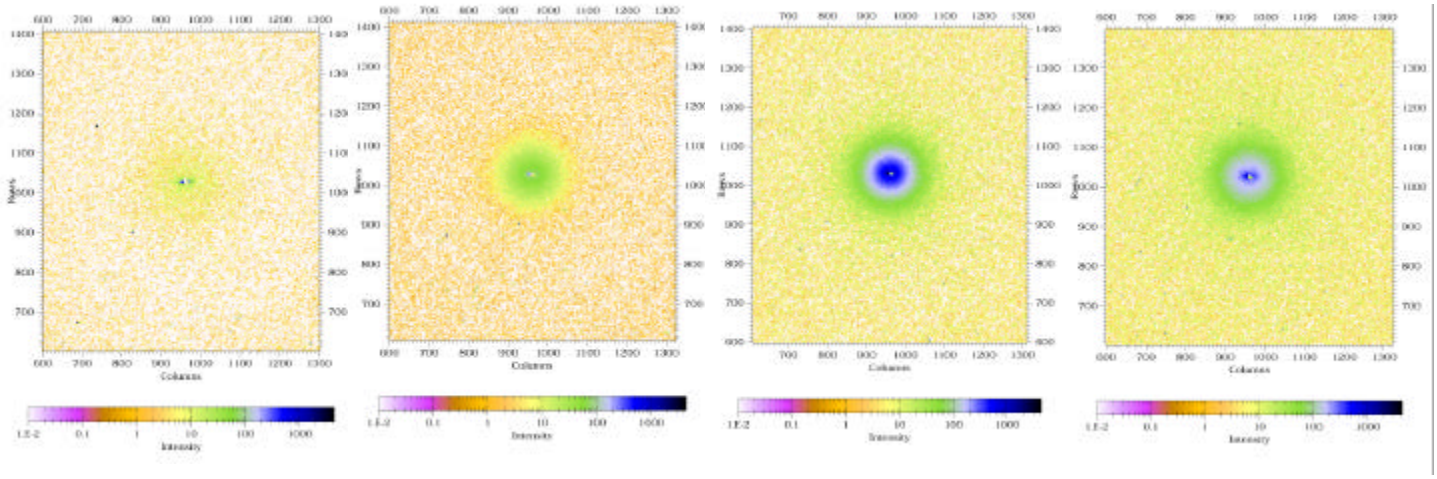

Figure 1.- *Two-dimensional scattering pattern for the CF and ACF. From left to right: CF, CFC29, CFC50, CFS48.*

The main conclusions from the experiments analyzed to the moment are:

1) Microbeam small-angle scattering is a technique useful for studying the porosity in different regions of single ACF. These experiments open new possibilities to study this type of carbon materials.
2) Porosity is isotropically developed along the fiber axis.
3) CO_2 develops porosity in the bulk of the fiber in more extension than steam, which localizes the activation mainly in the external surface.

These results are part of Dolores Lozano-Castelló's PhD. Thesis (which title is "Preparation and characterisation of advanced carbon materials for gas separation and storage of gases and energy", defended on July 3, 2001) and one paper is currently under preparation.

[1] D. Cazorla.Amorós, C.Salinas-Martínez de Lecea, J.Alcañiz-Monge, M.Gardner, A.North and J.Dore,*Carbon* **1998**, 36 309.

[2] D.Lozano-Castelló, D.Cazorla-Amorós, A.Linares-Solano, P.J. Hall, J.J. Fernández. *Characterization of Porous Solids V,* **2000**, 128, 523. K.K.Unger G. Kreysa, J.P. Balset (Eds.)

[3] D. Lozano-Castelló, D.Cazorla-Amorós, A.Linares-Solano, Peter J. Hall, David Gascon, Carmen Galan. *Carbon* **2001**, 39, 1343.

[4] M.A.Gardner, A.N.North, J.C.Dore, C.Salinas-Martinez de Lecea and D.Cazorla-Amorós. *Characterization of Porous Solids III*, **1994**, 87, 273. J.Rouquerol, F.Rodríguez-Reinoso, K.S.W.Sing and K.K.Unger (Eds.)

[5] J.Alcañiz-Monge, D.Cazorla-Amorós, A.Linares-Solano,S.Yoshida, A.Oya, *Carbon* **1994**, 32, 7.

[6] Paris, O.; Loidl, D.; Peterlik, H.; Müller, M.; Lichtenegger, H.; Fratzl, P. *Journal of Applied Crystallography*, **2000**, 33, 695.

[7] Müller, M.; Burghammer, M.; Riekel, C. *ESRF Newsletter*, **1999**, 12.