POROSITY DEVELOPMENT IN CARBON FIBERS AS EXPLORED BY μSAXS

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Activated carbon fibers (ACF) are typically microporous fibrous adsorbents, which have some advantages compared to granular and powdered adsorbents. ACF can be prepared from PAN fibers, cellulose fibers, phenolic fibers, and pitch fibers by "physical" or chemical activation of the carbon fibers. Both the nature of the precursors and the method of activation have a strong influence on the porous structure and adsorption properties of the resulting samples.

A deep characterization of ACF having large micropore volume is strongly desired to develop advanced technologies based on this property. In our research group, an important effort has been dedicated to analyze the development of porosity in isotropic pitch-based carbon fibers during "physical" activation with CO_2 and steam as activating agents. Among the different techniques, the use of an X-ray microbeam of 2 μm diameter (μSAXS) at beamline ID13 (ESRF) allowed us the characterization of different regions of the same fiber with microscopic position resolution [1,2].It was seen that in the case of CO_2 activation, ACF presented scattering patterns with high intensity in everywhere across the fiber diameter, confirming that CO_2 activation takes place within the fibers, generating a quite homogeneous development of porosity. On the other hand, in the case of steam activation, ACF presented scattering patterns with much higher intensity in the external zones of the fibers than in the bulk, which means that steam focuses the activation at the outer parts of the fibers.

Recently, we have extended this type of study to ACF prepared by chemical activation with KOH and NaOH, using as raw material, not only isotropic pitch-based carbon fibers, but also anisotropic PAN-based carbon fibers (fiber diameter in some cases less than 8 μm). For these measurements a sub-micrometer size beam (0.5 μm diameter) has been used, in a position-resolved X-ray scattering experiment at ID13 (ESRF). We have analyzed the development of porosity by chemical activation at different regions of a single fiber. The advantage of the SAXS technique to be sensitive to shape and orientation of the scattering objects (pores), has allow us to obtain additional information on the structural features already existing in the raw fibers or being created during the activation process, as well as in anisotropic studies in oriented samples. The results show that depending on the precursor, the chemical activation process produces isotropic or anisotropic development of porosity. It has been observed that chemically ACF prepared from isotropic carbon fibres present an isotropic development of the porosity and that a high micropore volume is developed not only in the external region of the fiber, but also in the core. On the other hand, in the case of anisotropic PAN-based carbon fibres the existence of two regions with different structure was detected by μSAXS measurements across the fiber diameter: an anisotropic external ring and a more isotropic fibre core. The results showed that these two regions remain after chemical activation and that the activating agents are reaching the fiber core. It seems that the more isotropic fibre core is activated easier by NaOH than KOH.

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References

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