Microporous carbon nanostructures derived from $Cr(C_5H_5)_2$ and $Cr(C_6H_6)_2$

P. González-García[1] , E. Urones-Garrote[2], A. Gómez-Herrero[2], D. Ávila-Brande[1] and L.C. Otero-Díaz[1]

[1]Departamento de Química Inorgánica, Facultad de Ciencias Químicas, Universidad Complutense, E-28040, Madrid, Spain. *E-mail:* pegonzal@quim.ucm.es
[2]Centro de Microscopía y Citometría, Universidad Complutense, E-28040, Madrid, Spain

Organometallic compounds have been widely used as precursors to produce shaped carbon materials because their chemical composition provides the carbon source and the metal catalyst [1,2]. Their final characteristics (purity, shape, surface area and pore size distribution) make them potential candidates for hydrogen and methane storage materials, supercapacitors and catalysts. In this work, we present the synthesis and characterisation of microporous carbon nanostructures obtained by chlorination of $Cr(C_5H_5)_2$ (chromocene) and $Cr(C_6H_6)_2$ (chromobencene). Precursors, powder purity of 97% Aldrich, were heated in a tubular furnace at 900 ºC (heating rate of 50 ºC/min) during 30 min, in a continuous flow of high purity chlorine gas (25 mL/min) following the next possible reactions:

$$Cr(C_5H_5)_{2(s)} + (13/2)Cl_{2\,(g)} \rightarrow 10C_{\,(s)} + CrCl_{3\,(g)} + 10HCl_{(g)}$$

$$Cr(C_6H_6)_{2\,(s)} + 7Cl_{2\,(g)} \rightarrow 12C_{\,(s)} + CrCl_{2\,(g)} + 12HCl_{(g)}$$

Transmission Electron Microscopy (TEM) images were done with a JEOL 3000 F (acceleration voltage of 300 kV) microscope (point resolution of 1.7 Å) equipped with an ENFINA spectrometer for Electron Energy Loss Spectroscopy (EELS) measurements. Solid and hollow carbon nanospheres were found in both samples (Figure 1a, 1b); additionally, amorphous carbon nanotubes-like (α-CNT) structures were observed in the sample prepared from chromocene. On the High Resolution-TEM images we observe that the structures are formed by highly disordered graphene layers. Quantification of bonding type, obtained by EELS, indicates sp^2/sp^3 content higher than 95 %. The mass-density (1.3 – 1.8 g/cm^3), obtained from the low-loss region of the EEL spectra, shows values bellow graphite density. The N_2 adsorption measurements at 77 K showed, isotherms shape which can be assigned to Type I (Figure 1c) due to small contributions of mesoporosity and textural porosity ($p/p_0 > 0.8$); however, high surface areas were developed (694 and 1761 m^2/g) and pore size distribution in the range of 0.58 – 1.35 nm (see inset in Figure 1c).

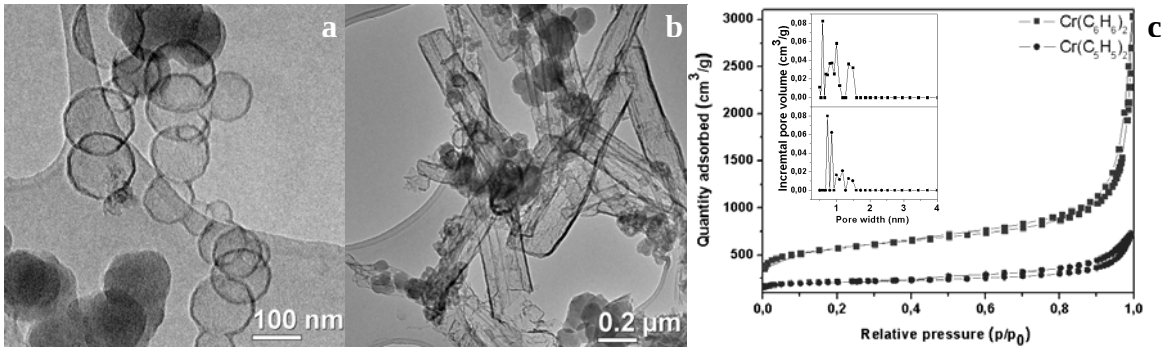

Figure 1. Shaped carbon nanostructures derived from (a) $Cr(C_5H_5)_2$ (b) $Cr(C_6H_6)_2$ and (c) BET measurements.

References

1. Nyamori V, Mhlanga S, Coville N, *J. Organomet. Chem.* (2008) 2205.
2. González-García P, et al., *Carbon* (2010) In Press.