Synthesis and Characterization of Novel Nanoporous Carbon (NCCR-1)

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Ordered nanoporous carbons with various structures, e.g., CMK-*n* (*n* = 1–5), have been synthesized by carbonization of sucrose, furfuryl alcohol and other suitable carbon sources utilizing mesoporous silicates/aluminosilicates as templates for their preparation.[1,2] In the present study, for the first time, we have prepared novel nanoporous carbon, designated as NCCR-1, employing a nanoporous silica (hexagonal MCM-41) template having one-dimensional pore structure, and sucrose as the carbon precursor. The nanoporous silica[3,4] (Fig. 1a) was first impregnated with aqueous sucrose solution containing sulfuric acid as per the procedure reported elsewhere[1,2]. The impregnated sample was placed in an air oven for 6 h at 373 K. Subsequently, the temperature was raised to 433 K and maintained at the same temperature for 6 h. The partially polymerized dark-brown composite was again impregnated with the acidified sucrose solution and heat treated as before at 373 K and 433 K for 6 h. The final carbonization was completed by pyrolysis of the polymerized composite at 1173 K under nitrogen atmosphere. The silica-carbon composite (Fig. 1b) thus obtained was treated with 10 wt. % hydrofluoric acid at room temperature. The template(silica)-free nanoporous carbon material was then filtered, washed with ethanol, and dried at 393 K for 6 h. The resulting nanoporous carbon is referred as NCCR-1 (Fig. 1c).

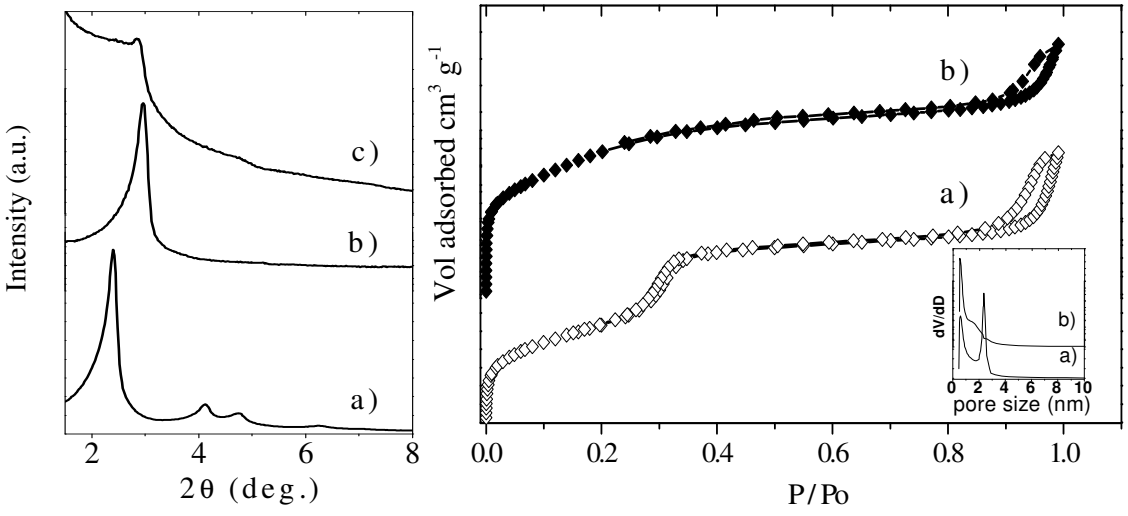

Figure 1. (Left) XRD patterns of: (a) Nanoporous Silica (MCM-41); (b) Silica-sucrose composite; (c) Nanoporous carbon (NCCR-1).

Figure 2. (Right) N_2 adsorption-desorption isotherms of: (a) Nanoporous Silica (MCM-41); (b) Nanoporous carbon (NCCR-1). Inset: (a) Pore size distribution of nanoporous silica (b) Pore size distribution of Nanoporous carbon (NCCR-1).

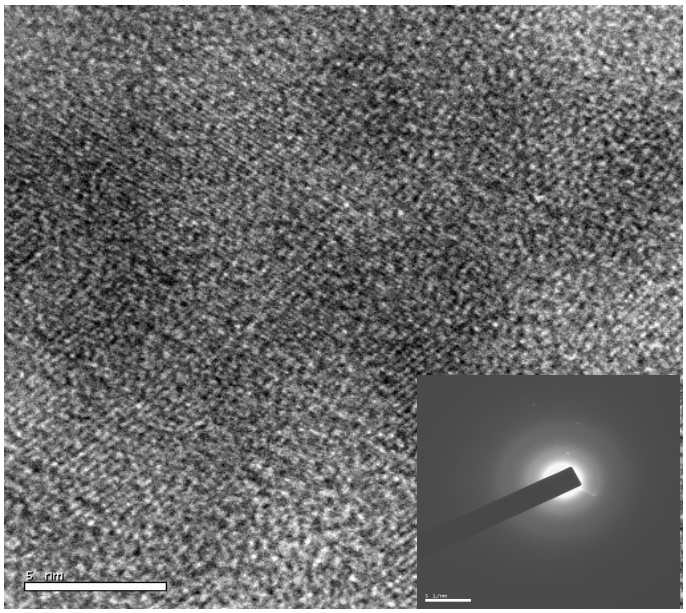

Figure 3. TEM of microporous containing mesoporous carbon (NCCR-1).

Figure 2 depicts the N_2 adsorption-desorption isotherms of both silica and carbon nanoporous materials. The isotherms are of type IV and typical characteristic of mesoporous materials with surface areas of 1034 and 1080 m^2g^{-1}, and pore volumes of 1.26 and 0.83 cm^3g^{-1}, respectively. However, as can be seen from Fig. 2 as well as the inset, the NCCR-1 carbon contains a large amount of micropores with a narrow pore size distribution and a broad pore size distribution in mesoporous region which is in contrast to the silica template. This is well supported by the TEM which shows the presence of micropores and small amount of mesopores (Figure 3). The electron diffraction pattern also confirms the XRD findings. It is, therefore, concluded from these studies that the NCCR-1 carbon is just an inverse replica of silica template and the presence of micropore in the silica template which inter-connect the carbon after replication.

[1] R. Ryoo, S.H. Joo, and S. Jun, J. Phys. Chem. B **103**, 7743 (1999).

[2] S. Jun, S.H. Joo, R. Ryoo, M. Kruk, M. Jaroniec, Z. Liu, T. Ohsuna, and O. Terasaki, J. Am. Chem. Soc. **122**, 712 (2000).

[3] C.T. Kresge, M. E. Leonowicz, W. J. Roth, J. C. Vartuli, and J. S. Beck, Nature **359**, 710 (1992).

[4] S. E. Dapurkar, S.K Badamali, and P. Selvam, Catal. Today **68**, 63(2001).

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