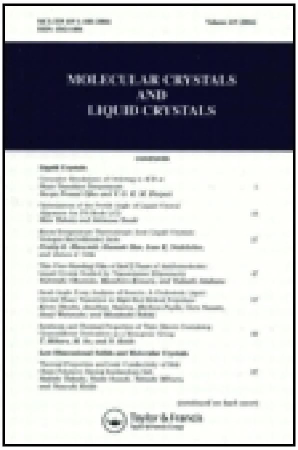

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Template-Directed Synthesis of Nanoporous Carbons

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Template-Directed Synthesis of Nanoporous Carbons

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Ordered porous carbons were synthesized by replication of colloidal crystal templates made from silica spheres 50 nm in diameter. The pores in the templates were filled with carbon precursor solution of divinylbenzene (DVB) and a free radical initiator, azobisisobutyronitrile (AIBN). Polymerization, carbonization, and subsequent dissolution of the silica templates leave a polycrystalline network of carbon with interconnected uniform pores.

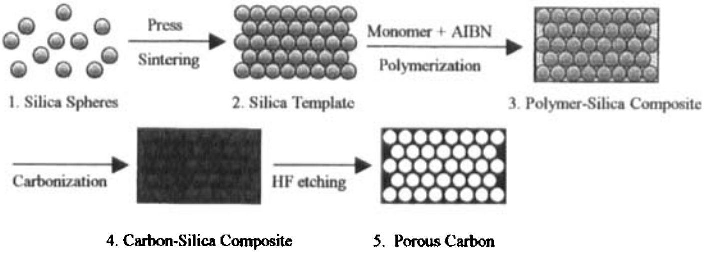
Keywords: carbon with uniform pore size; colloidal crystal template; pore replication

INTRODUCTION

Enormous effort has been given to synthesis of new ordered nanoporous materials mainly due to fundamental and technological interests.[1] The synthesis of porous carbons with uniform pore sizes is extremely important for their applications to adsorbents in many separation and purification processes.[2] Recently, a few studies have used colloidal silica spheres as templates to make three dimensional porous carbon replicas.[3,4] Interestingly, the meso- and macroscopic regimes of 30 to 100 nm still represent a largely unfilled gap for three dimensionally ordered porous carbon and thus are of immediate interest for the preparation of mesoscopic devices such as ordered arrays of metal or semiconductor quantum particles and for various sorption and separation processes.[5] In this work, we report the novel synthesis of nanoporous carbons with three-dimensionally interconnected uniform pores 30 nm to 100 nm in diameter through colloidal silica templates.

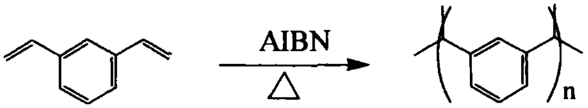
EXPERIMENTAL

Several monodisperse silica particles from 30 nm to 100 nm in diameter were synthesized based on sol-gel method (Step 1).[6] To create inorganic templates, dry monodisperse particles were pressed into pellets at a pressure of 1×10^4 KPa. The particles were then sintered slightly at their points of contact by heating the pallets to 800 °C in order to provide pore connectivity in the carbon replica through which the silica was removed after infiltration with carbon precursor (Step 2). Polymer precursor solution of divinylbenzene (DVB) and a free radical initiator, azobisisobutyronitrile (AIBN) with 10 : 1 mole ratio was added dropwise to the silica pellets, and the polymers were first formed by heating to 60 °C for 12 h to produce a polymer-silica composite (Step 3) according to the reaction



Scheme 1. Synthetic strategy for uniform porous carbons of tunable pores. Number indicates each of the synthetic steps explained in the Experimental.

shown in Fig. 1. The polymer and silica composite was then carbonized by heating the composite at 850 °C in dry inert gas flow to obtain a carbon-silica composite (Step 4). The silica was subsequently dissolved in aqueous HF and resulting porous carbon pellets were washed and dried in air (Step 5). Overall scheme for the synthesis of ordered porous carbons is shown in scheme 1.



DVB(Divinylbenzene) PDVB(Polydivinylbenzene)

Figure 1. Polymerization of divinylbenzene.

RESULTS AND DISCUSSION

Fig. 2a shows the transmission electron microscope (TEM) images of dried silica particles 50 nm in diameter prepared in this work. Note that the silica particles are uniform spheres. The particles then formed close-packed polycrystalline aggregates in colloidal crystal template. The volume fraction of space filled by close-packed spheres in such arrangement is approximately 75%. Fig. 1b shows the TEM image of a thin edge of porous carbon replicas synthesized in this work. The replica consists of pores close to 50 nm in diameter with a volume-filling fraction up to about 25 %. The pores are close-packed, roughly spherical, and interconnected. This shows that the degree of order of the polycrystalline template is faithfully reproduced in the carbon replica.

Elemental analysis of porous carbon replica shows C:H mole ratio of about 27 (C, 98.18 wt% and H, 0.33 wt%). Brunauer-Emmett-Teller (BET) surface area of the porous

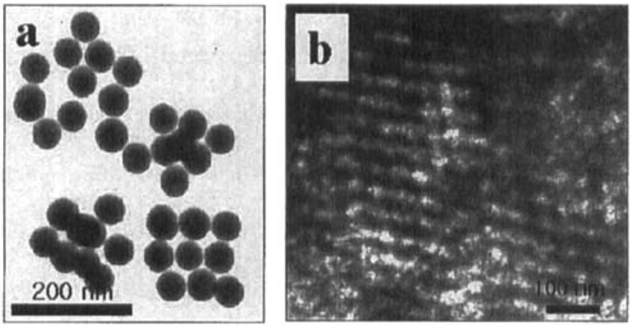


Figure 2. TEM images (a) of silica particles (50 nm in diameter) used for colloidal crystal templates and (b) of the thin edge of porous carbon replica.

carbon, calculated from nitrogen adsorption isotherms, was found to 484 m^2/g. The N_2 adsorption and desorption isotherms exhibited a curve characteristic of the presence of macropores. Silica spheres with different diameters in the range from 30 to 100 nm can be prepared by the same methods and thus we anticipate that the replication method cover an even smaller or larger range of pore sizes. After all, the uniform pore sizes of porous carbon materials can be easily controlled through template synthesis by monitoring the sizes of silica spheres.

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