Hierarchically ordered CdS doped nanoporous membrane

Weon-Sik Chae,^a Sang-Wook Lee,^a Sung-Jae Im,^a Sik-Won Moon,^b Wang-Cheol Zin,^b Jin-Kyu Lee^c and Yong-Rok Kim^a

 ^a Department of Chemistry, Yonsei University, Seoul 120-749, South Korea. Fax: +82 (0)2 364 7050; E-mail: yrkim@yonsei.ac.kr
^b Department of Materials Science and Engineering, Pohang University of Science and Technology, Pohang 790-784, South Korea. Fax: +82 (0)54 279 2399; E-mail: wczin@postech.ac.kr
^c School of Chemistry, Seoul National University, Seoul 151-747, South Korea. Fax: +82 (0)2 882 1080; E-mail: jinklee@snu.ac.kr

Experimental Details: The porous alumina was purchased from Whatman (Anodisc 13), and the nanochannel surface of the porous alumina was organically modified with octadecyltrichlorosilane (Sigma-Aldrich Chemical Co.; Catalogue No. 104817-25G, 90+%, St. Louis, USA) through reflux in benzene for 10 h in order to enhance the filling of the lyotropic precursor solution. The lyotropic precursor solution consisted of the silica and cadmium precursors and the triblock copolymer of Pluronic F127 (BASF Chemical Co., New Jersey, USA) that were previously utilized as a supramolecular template for mesoporous materials¹ in ethanol: 1 g of Pluronic F127 was homogeneously mixed with 2.1g of tetraethylorthosilicate (TEOS, Aldrich Chemical Co.; Catalogue No. 13190-3, 98%, Milwaukee, USA), 0.03g of cadmium nitrate tetrahydrate (Kanto Chemical Co.; Catalogue No. 07022-00, 98%, Tokyo, Japan), and 0.54 g of 0.3 M nitric acid (Matsunden Chemical Co., 60%, Osaka, Japan) in 2.3 g of ethanol (Merck KGaA; Catalogue No. 111727, 99.9>%, Darmstadt, Germany). In this lyotropic system, the molar ratio of CdS to SiO₂ was 0.01 (1 mol%), and the lyotropic system with increased molar ratio of 3 mol% was also studied for the structural and optical properties. The porous alumina support was dipped into the lyotropic precursor solutions and slowly stirred for 12 h in order to have the porous alumina channels completely filled with the precursor solutions. The filled porous alumina membranes were then allowed to gellation at 60 °C for 12 h. The resulting porous alumina including the lyotropic supramolecular precursors was exposed to the excess H₂S gas (10 ml) for 10 h, which immediately resulted in the characteristic yellow color of the CdS. Some of the bulk remains deposited on the membrane surface was cleaned out and subjected to calcination at 350 °C for 2 h in order to remove the organic template molecules incorporated in the mesoporous composite nanofiber confined in the porous alumina.

Instrumentations: The resulting membranes were studied by FE-SEM (JEOL, JSM-6700F), TEM (JEOL, 2010), nitrogen adsorption (Micromeritics, ASAP 2010), diffuse reflectance (Jasco, V-550) equipped with an integrating sphere (Jasco, ISV-469), and fluorescence spectrophotometer (Hitachi, F-4500). The small-angle X-ray scattering measurement was performed in a transmission mode with synchrotron radiation at the 3C2 X-ray beam (λ =0.1542 nm) line at Pohang Accelerator Laboratory, POSTECH, South Korea. The X-ray beam was vertically irradiated to the plane of the nanoporous membranes.



Fig. S1 FE-SEM image of the 3 mol% CdS doped mesoporous nanostructure confined in the porous alumina: (A) top and (B) side views. (C) TEM image of a single mesoporous silica nanofiber including the CdS, which was obtained after removal of the porous alumina template by 1 M of aqueous NaOH.



Fig. S2 Small-angle X-ray scatterings for (A) the bare porous alumina and (B) the confined CdS doped mesoporous nanofibers within the porous alumina with different doping level: 1 and 3 mol%. (C) Typical X-ray beam direction for the structural analysis of the mesoporous nanofiber and (D) graphical drawing of the onion-like structure in cross-sectional view for the mesoporous nanofiber. The d_{100} -spacing is estimated to be 10.64 nm from the X-ray scattering analysis.

Reference

1. D. Zhao, Q. Huo, J. Feng, B. F. Chmelka and G. D. Stucky, J. Am. Chem. Soc., 1998, 120, 6024.