Supporting information

Facile synthesis of mesoporous silica by CO$_2$/N$_2$ switchable templates using convenient compound

Jianzhong Jiang, a* Jinchao Yin, a‡ Li Dai, a‡ Rukuan Liu b and Zhihong Xiao b

a The Key Laboratory of Food Colloids and Biotechnology, Ministry of Education, School of Chemical and Material Engineering, Jiangnan University, 1800 Lihu Road, Wuxi, Jiangsu 214122, P. R. China. Email: jzjiang@jiangnan.edu.cn

b Institute of Bioenergy, Hunan Academy of Forestry, Changsha 410004, China.

Experiment reagents:
Tetraethoxysilane (TEOS) and Ethylenediaminetetraacetic acid disodium salt (Na$_2$EDTA) were purchased from Sinopharm Chemical Reagent Co., Ltd; N$_2$ (>98%) and CO$_2$ (>98%) were obtained from Wuxi Xinnan Chemical Gas Co., Ltd; N,N-Dimethyldodecylamine; N,N-Dimethyltetradecylamine and N,N-Dimethylhexadecylamine were purchased from Tokyo Chemical Industry Co. Ltd.

Experiment procedure:

Synthesis of mesoporous silica

N,N-Dimethyldodecylamine (0.8536g, 0.004mol) and distilled water (36g, 2 mol) were added into a bottle with stirring. The bottle was connected with CO$_2$ cylinder (0.1Mpa). Na$_2$EDTA·2H$_2$O (3.6114 g, 0.01mol) was then added into the bottle and TEOS (4.166g, 0.02mol) was dropped into the bottle another hour later. The reaction was kept in a static condition at a certain temperature for 6 days. N$_2$ was bubbled into product suspension at 70 °C for an hour when the reaction was completed. The
product was ultrasonically washed by distilled water and acetone for 6 times (5 minutes per time) at room temperature. Mesoporous silica product was obtained after vacuum dry at 70 °C.

**Reuse of soft template**

The solutions after bubbling N₂ and washing were collected, evaporated and then extracted with diethyl ether. The layer of diethyl ether was evaporated and viscous liquid was then obtained. The viscous liquid was weighted and added into a bottle with distilled water. The bottle was connected with CO₂ cylinder (0.1Mpa). The transparent solution was then used as template for the mesoporous silica as described above.
Figure S1. (a) conductivity of pure water after bubbling CO₂ (30 mL/min) and N₂ (100 mL/min) at 20 °C; (b) conductivity of the aqueous acetone solution (50 vol.%) of 8 mM N,N-Dimethyldodecylamine as a function of the time of bubbling with CO₂ (30 mL/min) followed by bubbling with N₂ (100 mL/min) at 20 °C alternately (three cycles).
Figure S2. IR transmission spectra of silica product. (a) as-made sample; (b) washing with distilled water and acetone 20 times; (c) bubbling N₂ to remove templates; (d) calcination.

Figure S3. Recovery percent of N,N-Dimethyldodecylamine in different recycle times.
Figure S4. SEM images of silica product synthesized by tertiary amines with different carbon number in alkyl chain (a:12, b:14, c:16).

Figure S5. SEM images of silica product synthesized at different temperature (a:10 °C, b:20 °C, c:30 °C).