

Surfactant-Assisted Synthesis of Mesoporous Silica/Ceria-Silica Composites with High Cerium Content under Basic Conditions

Eun-Bum Cho,*^a Seunghyuk Yim,^a Dukjoon Kim*^b and Mietek Jaroniec*^c

^a Department of Fine Chemistry, Seoul National University of Science and Technology, Seoul 139-743, Korea

^b Department of Chemical Engineering, Sungkyunkwan University, Suwon, Gyeonggi-do 440-746, Korea

^c Department of Chemistry and Biochemistry, Kent State University, Kent, Ohio 44242, USA

Table S1. Physicochemical properties of 3D bicontinuous cubic (*Ia3d*) silica/ceria-silica composites prepared by using a cationic surfactant under basic conditions.^a

sample	$f_{\text{Ce/Si}}$	S_{BET} ($\text{m}^2 \text{g}^{-1}$)	V_t ($\text{cm}^3 \text{g}^{-1}$)	V_{textural} ($\text{cm}^3 \text{g}^{-1}$)	D_{KJS} (nm)	d -spacing (nm)	a (nm)	w (nm)
CCS-20	0.2	637	0.64	0.46	3.4	3.685	9.03	1.2
CCS-30	0.3	543	0.56	0.38	3.5	3.685	9.03	1.2
CCS-40	0.4	455	0.50	0.30	3.4	3.689	9.04	1.2
CCS-50	0.5	356	0.42	0.23	3.4	3.672	9.00	1.2

^aNotation: $f_{\text{Ce/Si}}$ - molar ratio of cerium hydroxide to tetraethylorthosilicate used in the synthesis gel; S_{BET} - BET specific surface area determined in the range of relative pressures from 0.04 to 0.2; V_t - single-point pore volume at $P/P_o = 0.99$; V_{textural} - volume of textural pores obtained by t -plot analysis; D_{KJS} - mesopore diameter at the maximum of the PSD curve obtained by the improved KJS method;⁵² d -spacing - Bragg's spacing ($= 2\pi/q^*$; q^* is q value at maximum (211) peak for *Ia3d* mesostructure); a - unit cell parameter ($=\sqrt{6}d_{211}$ for *Ia3d* mesostructure); w - pore wall thickness ($= a/3.0919 - D_{\text{KJS}}/2$ for *Ia3d* mesostructure).⁵³

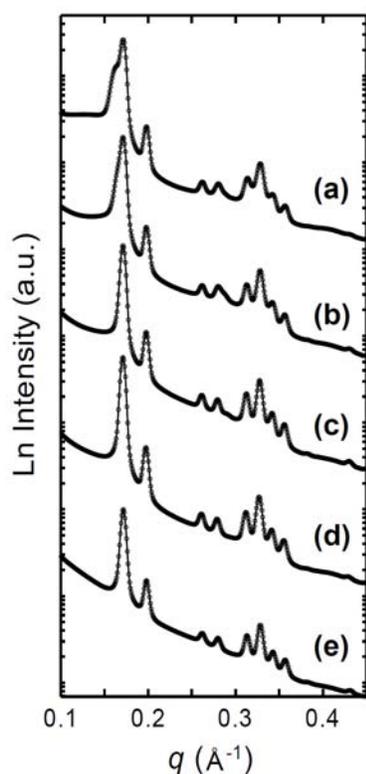


Figure S1. Synchrotron SAXS patterns of bicontinuous cubic (*Ia3d*) mesoporous silica/ceria-silica composite samples prepared under basic conditions. The sequence of the SAXS patterns from (b) to (e) corresponds to the list of samples in Table S1: (b) CCS-20, (c) CCS-30, (d) CCS-40, and (e) CCS-50, respectively. The pattern (a) is for the mesoporous silica/ceria-silica sample corresponding to the Ce/Si ratio in the synthesis gel = 0.1.

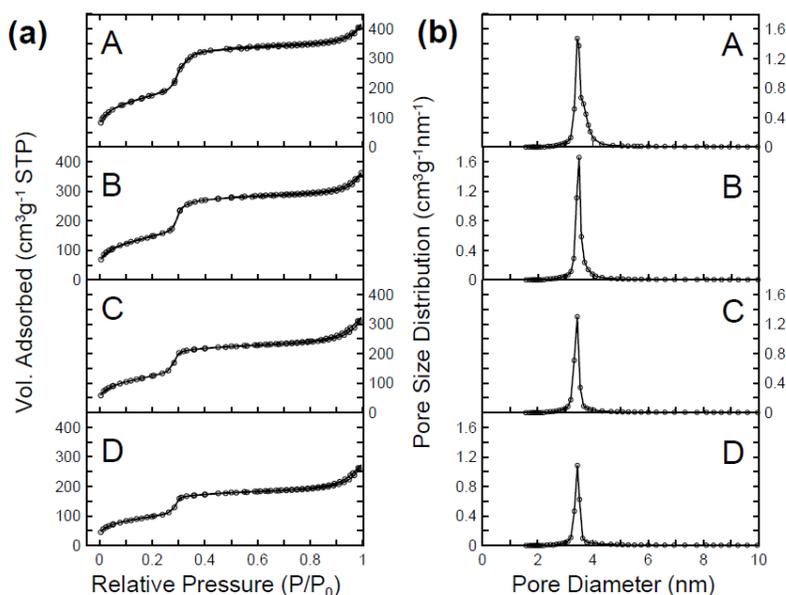


Figure S2. Nitrogen adsorption-desorption isotherms (a) and the corresponding pore size distributions (b) for bicontinuous cubic (*Ia3d*) mesoporous silica/ceria-silica composites prepared under basic conditions. The sequence of the isotherms from A to D refers to the following samples: (A) CCS-20, (B) CCS-30, (C) CCS-40, and (D) CCS-50.

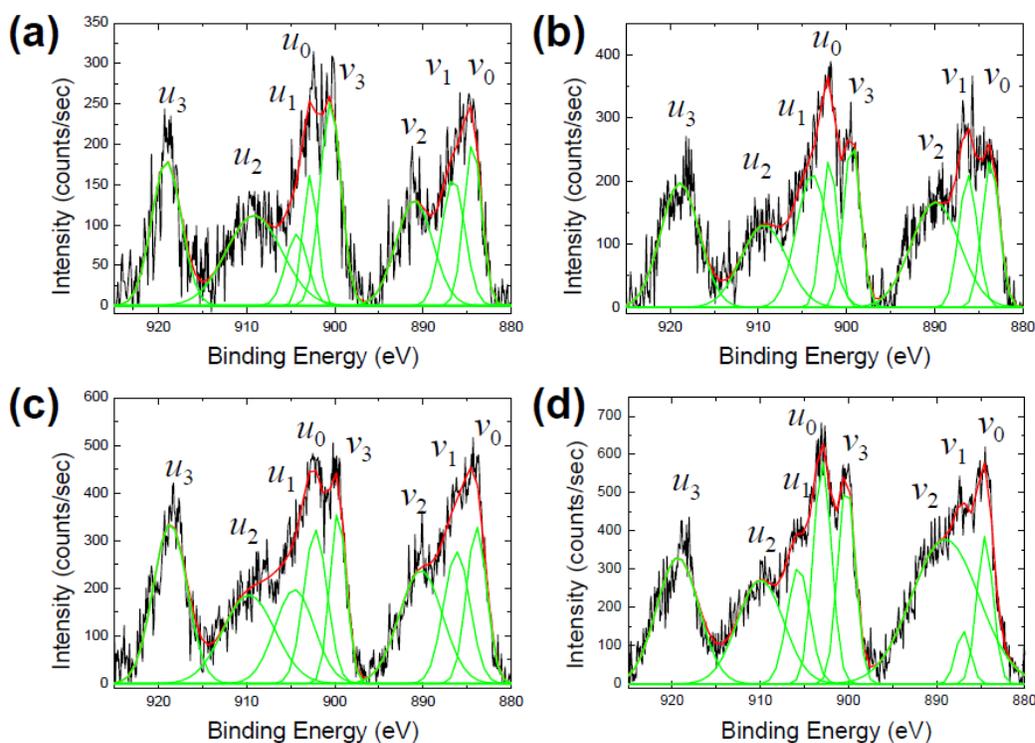


Figure S3. XPS patterns of the Ce 3d core level spectra and the deconvoluted eight peaks by a Gaussian fitting method for mesoporous silica/ceria-silica composites prepared under basic conditions. The XPS patterns are (a) HCS-20, (b) HCS-30, (c) HCS-40 and (d) HCS-50, respectively. The series of *v* denotes Ce 3d_{5/2} and *u* denotes Ce 3d_{3/2}, where the peaks of *v*₁ and *u*₁ belong to Ce³⁺ ion and the other peaks belong to Ce⁴⁺.

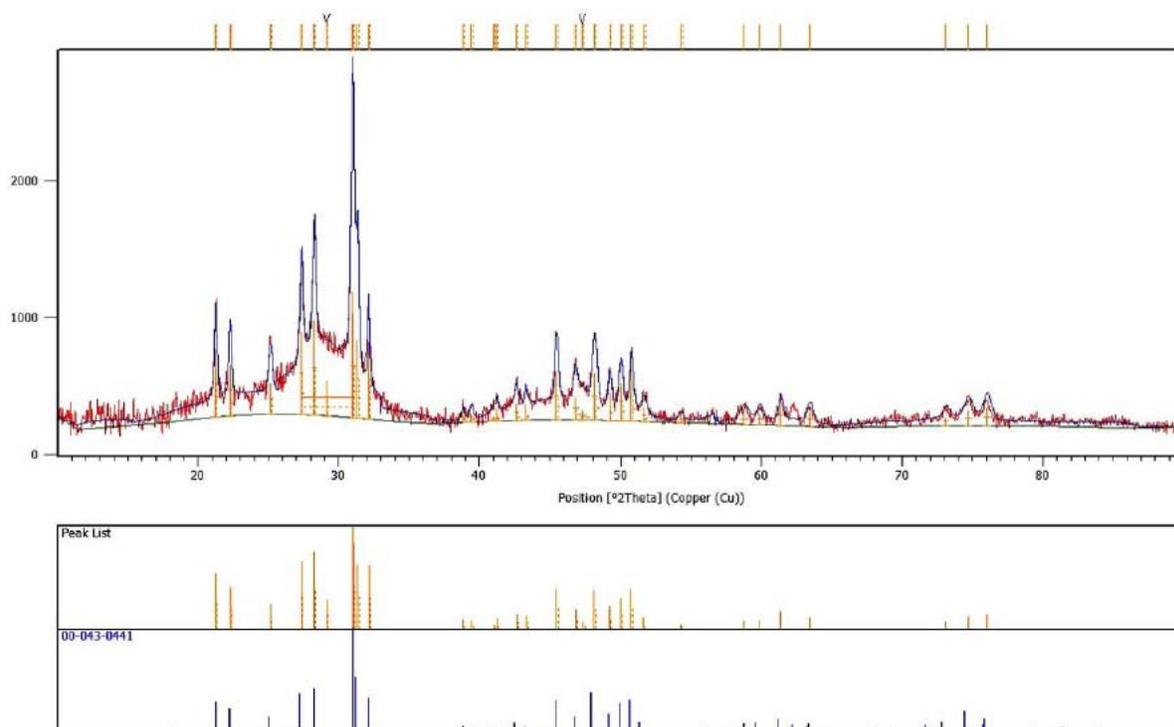


Figure S4. Wide angle X-ray diffraction patterns of the reduced HCS-50HR sample and the corresponding JCPDS reference.

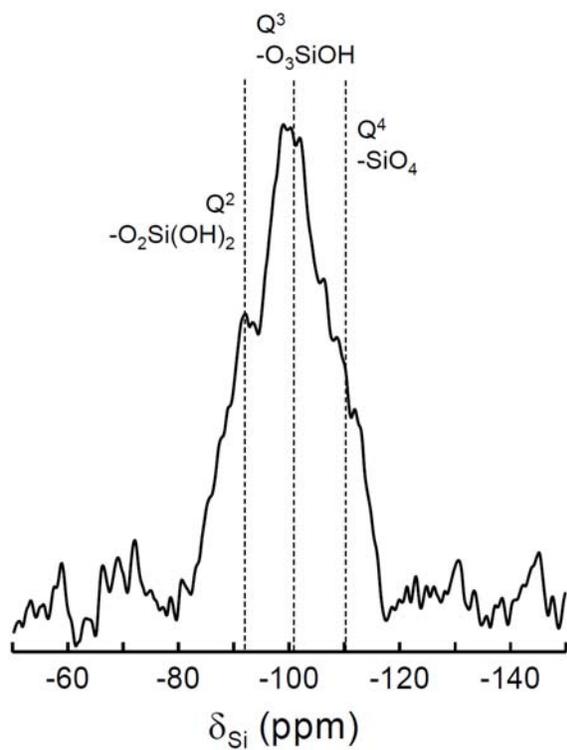


Figure S5. Solid state ^{29}Si CP MAS NMR spectra of the reduced HCS-50HR sample.