

Electronic Supplementary Information for

A P123/benzyl alcohol/TEOS/HCl(aq.) templating system for preparation of KIT-6 type mesoporous silica with morphological and structural control

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Experimental section

Chemicals

Concentrated HCl (37%) and tetraethoxysilane (TEOS, AR) were purchased from the Sinopharm Chemical Reagent Co., Ltd. Pluronic PEO20-PPO70-PEO20 (P123 with Mw = 5800) was purchased from Aldrich, Benzyl alcohol (Bz, AR) was purchased from Aldrich. All the chemicals were used as received.

Synthesis of samples

The chosen synthesis procedure was the partitioned cooperative self-assembly (PCSA) process. Typically, 2.0 g P123 was dissolved in 78.0 g de-ionized water and 10.5 g conc. HCl at 33.5 °C to form a clear solution. To this solution, 0.7 g benzyl alcohol (Bz) was added under vigorous stirring. After string for 10 min, 3.0 g TEOS (1st part, 7/10 of total TEOS) was dropped into above synthesis mixture into which the rest of 1.28 g TEOS (2nd part, 3/10 of total TEOS) was then added after 4 h. The mixture was stirred at 33.5 °C for 24 h and then sealed within a Teflon autoclave for hydrothermal treatment at 100 °C for 24 h. The product was collected by filtration, washing and then dried at 70 °C overnight, followed by calcination at 550 °C for 4 h.

Characterization

The low-angle powder XRD patterns at 2 θ angles from 0.6° to 3.0° were recorded at an interval of 0.01° on a SmartLab diffractometer (Rigaku, Tokyo, Japan) with Cu K α radiation (40 kV, 30 mA). Low-angle diffraction peaks were used to determine the d-spacings according to the normal Bragg relationship. FE-SEM images were recorded using Field-emission Scanning Electron Microscope (Hitachi S4800). Transmission electron microscopy (TEM) images were examined using a JEM F2100 transmission electron microscope. N₂ adsorption-desorption measurement was measured using a Tristar 3020 analyzer (Micromeritics) at 77 K. The Brunauer-Emmett-Teller (BET) surface area was calculated from the adsorption data in the relative pressure range from 0.04 to 0.20. Before adsorption measurements, all samples were degassed under vacuum condition at 200 °C for 2 h before analysis.

Table 1 Physical properties of samples prepared in the presence of Bz based on PCSA process and conventional process

Sample	S _{BET} (m ² /g)	V _{total} ^a	V _{mi} ^b	V _{me} (cm ³ /g) ^b	a (nm) ^c	D _{B_{JH}} (nm) ^d	W(nm) ^e	Structure
10-0	730	1.00	0.01	0.99	11.7	9.0	2.7	2D hexagonal
7-4h-3	774	1.14	0.07	1.07	23.7	9.3	2.6	Ia3d
7-4h-3 (120)	661	0.93	0.08	0.85	25.1	10.3	2.3	Ia3d
7-4h-3 (140)	426	1.08	0.01	1.07	24.5	10.6+	1.7	deteriorated Ia3d

- a V_{total} is the total pore volume calculated from the amounts of N_2 adsorbed at relative pressure 0.995.
- b Micropore volumes (V_{Mi}) and mesopore volumes (V_{Me}) calculated according to a_s -plots analysis.
- c Unit cell size of SBA-15 = $2d_{(100)}/3^{1/2}$, Unit cell size of KIT-6 is equal to $6^{1/2}d_{(211)}$.
- d D_{BJH} is the pore diameter calculated using the BJH method.
- e W , wall thickness, W of 10-0 is the difference between a and D_{BJH} , W of 7-4h-3 is equal to $a/2 - D_{BJH}$

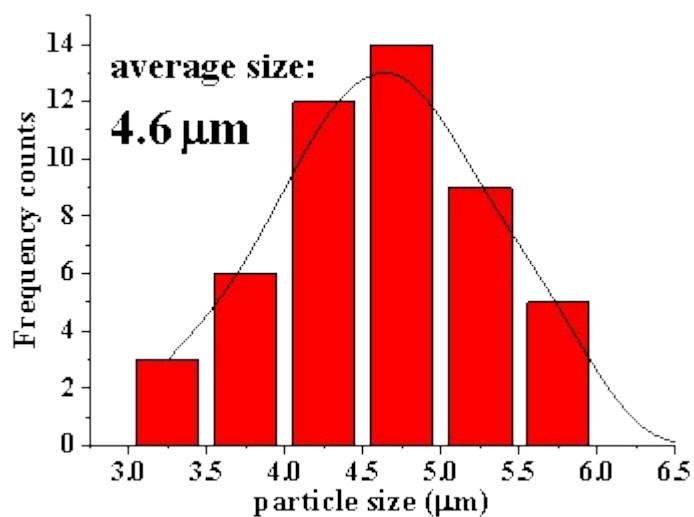


Fig.S1 The diagram of particle size distribution of 7-4h-3

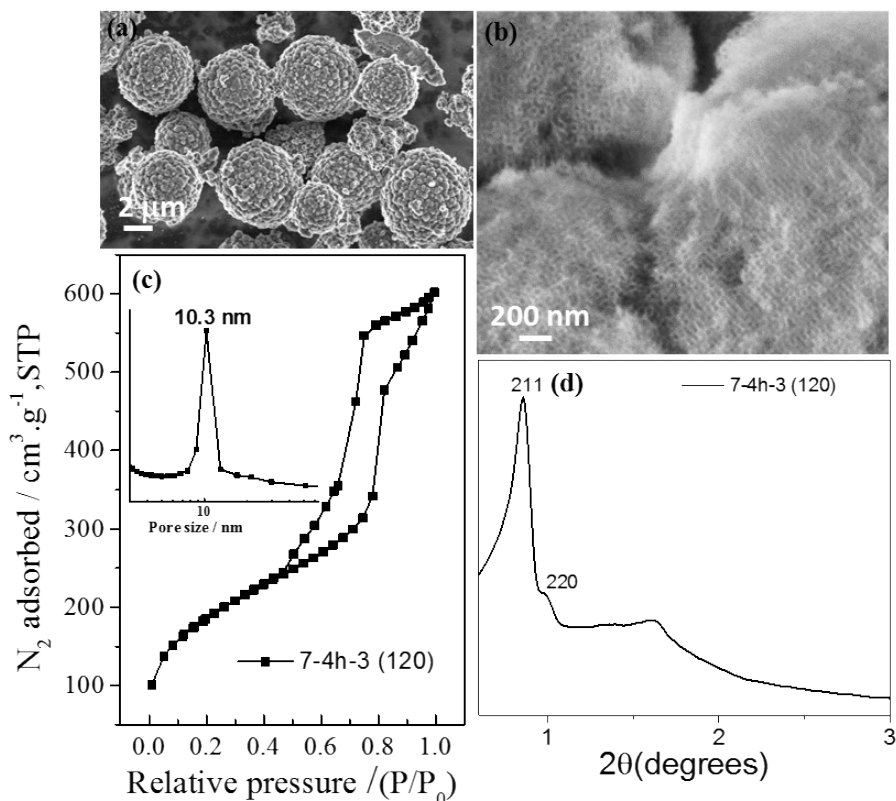


Fig.S2 SEM image (a, b) and of 7-4h-3(120) (c) N_2 adsorption-desorption isotherm and mesopore size distributions of 7-4h-3(120). (d) Low-angle XRD patterns of sample 7-4h-3 (120)

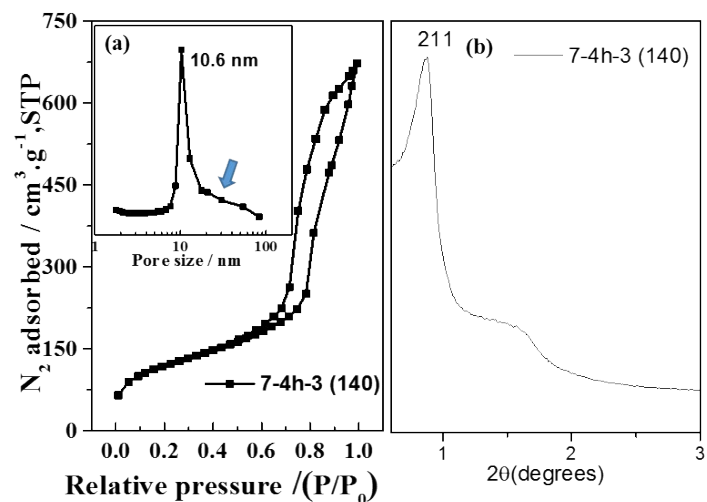


Fig.S3 (a) N_2 adsorption-desorption isotherm and mesopore size distributions of 7-4h-3 (140). (b) Low-angle XRD pattern of sample 7-4h-3 (140) lacking resolving high-angle XRD reflections

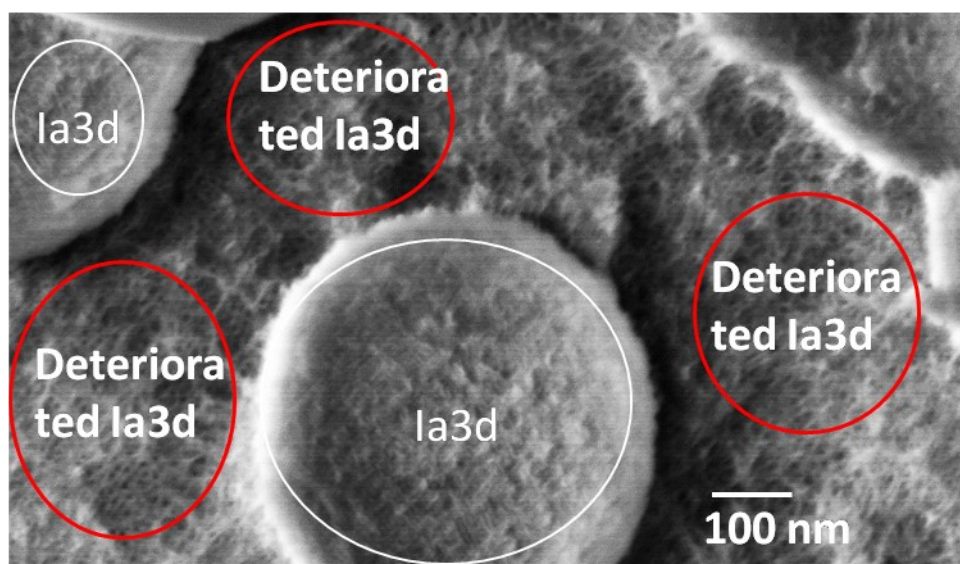


Fig.S4 FE-SEM image of 9-4h-1 at 0.7 Bz, showing the presence of ordered Ia3d phase and deteriorated Ia3d phase.

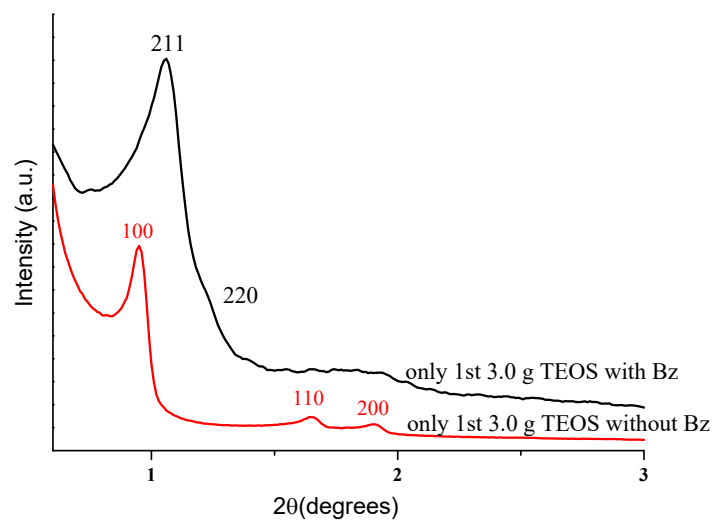


Fig.S5 The Low-angle XRD pattern of sample prepared with only 1st 3.0 g of TEOS with addition of Bz and a control sample prepared without the addition of Bz

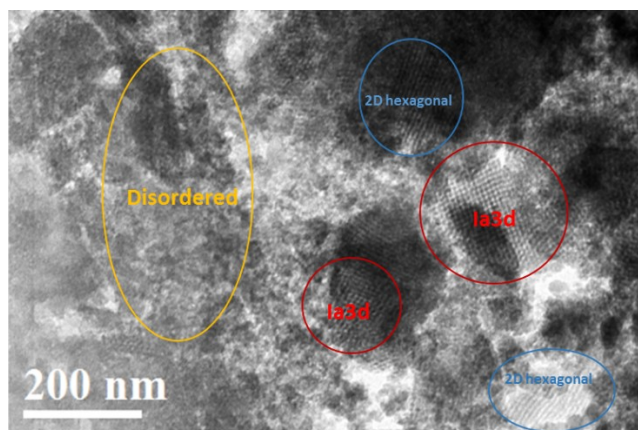


Fig.S6 TEM image of sample prepared with only 1st part of TEOS (3.0g TEOS, i.e., 7/10 of total TEOS)

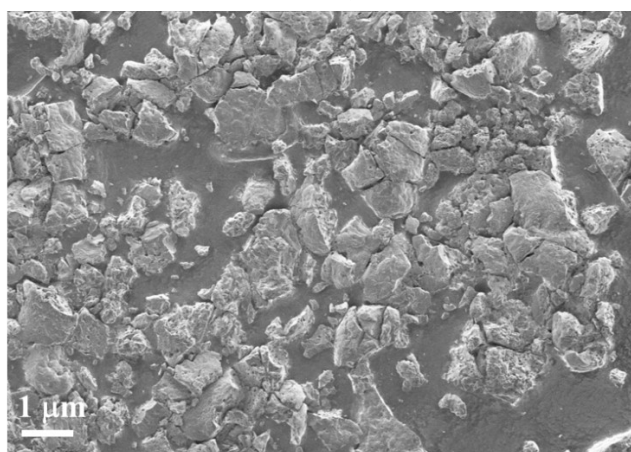


Fig.S7 SEM image of KIT-6 prepared by conventional process (10-0) using n-butanol as co-solvent.