Supplementary material (ESI) for Journal of Materials Chemistry

Synthesis of Periodic Mesoporous Phosphorus-Nitrogen Frameworks by Nanocasting from Mesoporous Silica Using Melt-infiltration

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Experimental details

Chemicals

Triblock copolymers $EO_{20}PO_{70}EO_{20}$ Pluronic P123 and $EO_{106}PO_{70}EO_{106}$ Pluronic F127 (BASF, USA), tetraethyl orthosilicate (TEOS, Sigma-Aldrich), phosphonitrilic chloride trimer (PNC) (Alfa-Aesar), 48-50 wt% HF in water (Fisher Chemicals), nitrogen (Airgas, Inc, 99.998 %) and ammonia (GT & S, Inc, 99.999 %) gas. All the chemicals were used as-received without further purification.

Synthesis of SBA-15

Mesoporous hexagonal silica SBA-15 was synthesized by using P123 as a structure directing agent and TEOS as silica source according to the procedure reported previously.[31,32] In a typical synthesis, 3.0 g of P123 was dissolved in 114.0 ml of 1.6 M hydrochloric acid. To it, 6.6 g of TEOS was added at 35 ºC under stirring with a magnetic stirrer until TEOS was completely dissolved. The mixture was placed in an oven for 24 h at 35 ºC for the mesostructure formation, and subsequently for 6 h at 100 ºC for hydrothermal treatment. The product was filtered, dried at 100 ºC without washing, and then calcined in air at 550 ºC for 5 h.

Synthesis of SBA-16

Mesoporous body centered cubic silica SBA-16 was synthesized according to the reported procedure.[32-34] In a typical synthesis, 5.0 g of F127 was dissolved in 240 g of distilled water and 10.5 g of hydrochloric acid (35 wt %). To it, 15.5 g of butanol was added at 45 ºC. After stirring for 1 h, 24 g TEOS was added and the stirring of the mixture was continued for 24 h for the mesostructure formation. The hydrothermal treatment was carried out by aging the mixture for another 24 h in static condition. The product was filtered, dried at 100 ºC without washing, and then calcined in air at 550 ºC for 5 h. The molar composition of the starting reaction mixture was 0.0035 F127/1 TEOS/1.8 Butanol/0.91 HCl/117 H_2O.

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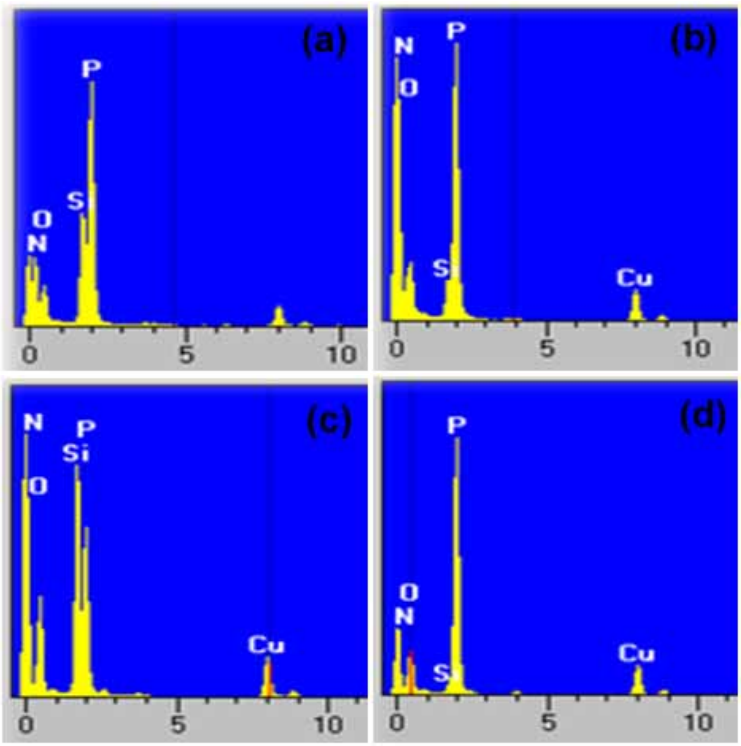

Fig. S1 EDS spectra of (a) LUM-1/SiO_2, (b) LUM-1, (c) LUM-2/SiO_2 and (d) LUM-2, respectively.

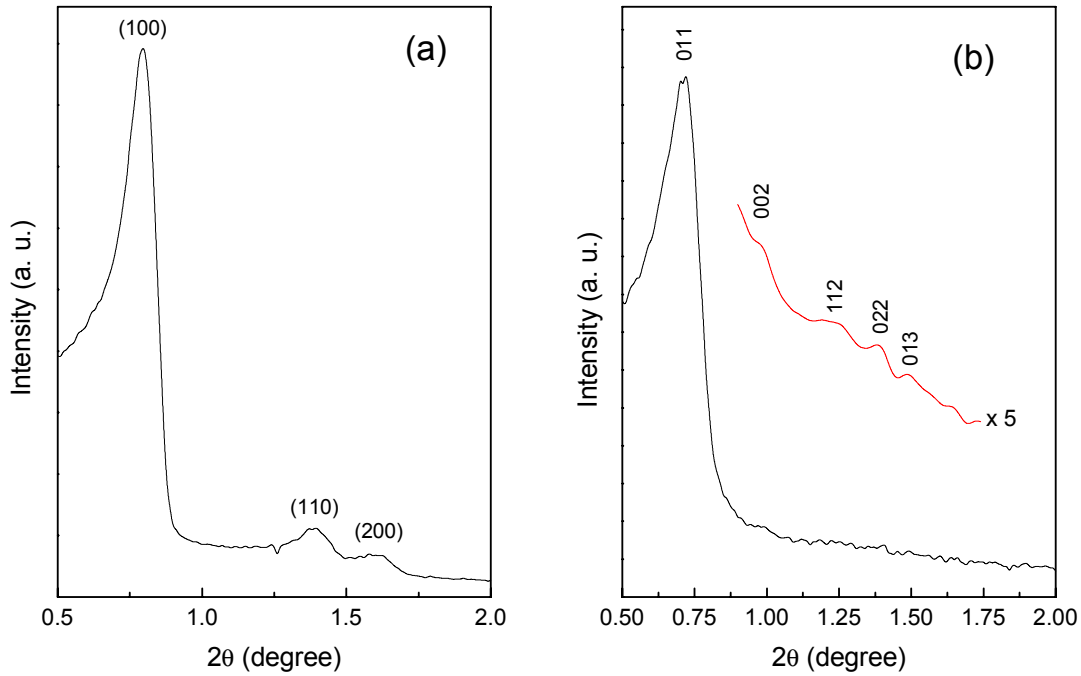

Fig. S2 SAXS patterns of (a) *p6mm* hexagonal silica SBA-15 and (b) body centered cubic $Im\bar{3}m$ silica SBA-16.

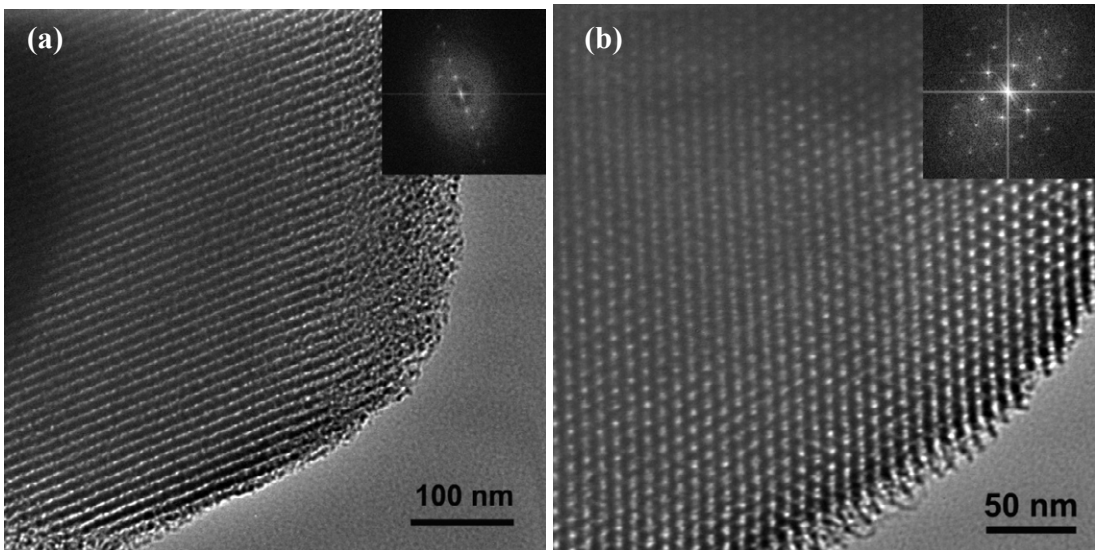

Fig. S3 TEM images of SBA-15 along (a) [110] and (b) [001] projections. The insets are the corresponding FFT images.

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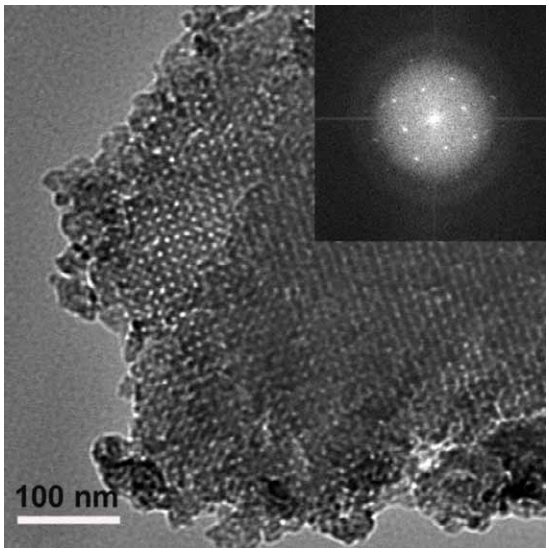

Fig. S4 TEM image of $Im\bar{3}m$ LUM-2 along [100] projection. The FFT image is given in the inset.

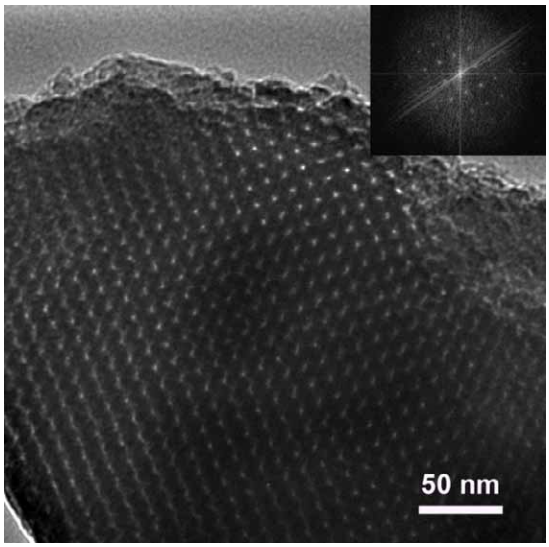

Fig. S5 TEM and the FFT images of $Im\bar{3}m$ c-silica along [100] projection.

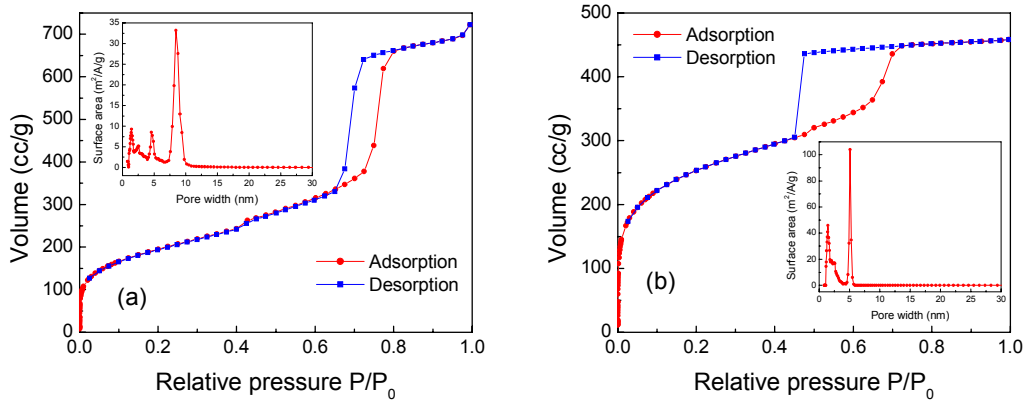

Fig. S6 N_2 adsorption-desorption isotherm of (a) SBA-15 and (b) SBA-16 measured at -196 ºC. The pore size distribution (PSD) calculated by DFT & Monte-Carlo method are given in the inset.

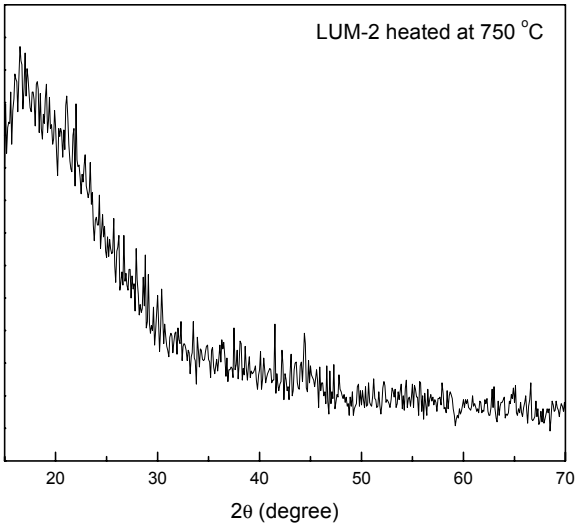

Fig. S7 XRD pattern of LUM-2 heated at 750 °C for 1 h in nitrogen atmosphere.