Supporting Information

Highly Ordered Periodic Mesoporous Organosilica Nanoparticles with Controllable Pore Structures

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Preparation of highly ordered PMO nanoparticles

In a typical synthesis of ethylene-PMO nanoparticle, CTAB (0.021 g), water (9.35 ml) and ammonia (0.652 ml, 27 wt.%, aqueous solution) were mixed and stirred for 10 min to form a clear solution at room temperature. This solution was heated in an oil bath to 50 °C, to which of bis(triethoxysilyl)ethane (0.034 g) was added under stirring. The mixture was cooled to room temperature after 6 h, and then aged overnight without stirring.

In a typical synthesis of ethenylene-PMO nanoparticle, CTAB (0.021 g), water (9.74 ml) and ammonia (0.36 ml, 27 wt.%, aqueous solution) were mixed and stirred for 10 min to form a clear solution at room temperature. This solution was heated in an oil bath to 50 °C, to which bis(triethoxylsilyl)ethylene (0.033 g) was added under stirring. The mixture was cooled to room temperature after 6 h, and then aged overnight without stirring.

In a typical synthesis of phenylene-PMO nanoparticle, CTAB (0.021g), water (8.74 ml), *n*-propanol (1 ml) and ammonia (0.26 ml, 27 wt.%, aqueous solution) were mixed and stirred for 10 min to form a clear solution at room temperature. This solution was heated in an oil bath to 50 °C, to which 1,4-bis(triethoxysilyl)benzene

(0.038 g) was added under stirring. The mixture was cooled to room temperature after 6 h, and then aged overnight without stirring.

All the samples were then collected by centrifugation and washed with ethanol several times. Template extraction was performed with hydrochloric acid/ethanol mixture. The compositions of these extraction solutions were HCl (1 mL, 36 wt.%, aqueous solution) in the ethanol (50 mL). The sample (1 g) was treated two to three times with 100 mL of the extraction solutions by stirring at 50 °C for 6 h.

Preparation of the mesoporous silica nanoparticles (MSNs)

The detailed synthetic procedure for the preparation and purification of MSN was described in the literature.[1]

References

[1] K. Moeller, J. Kobler, T. Bein, *Adv. Funct. Mater*. **2007**, *17*, 605.

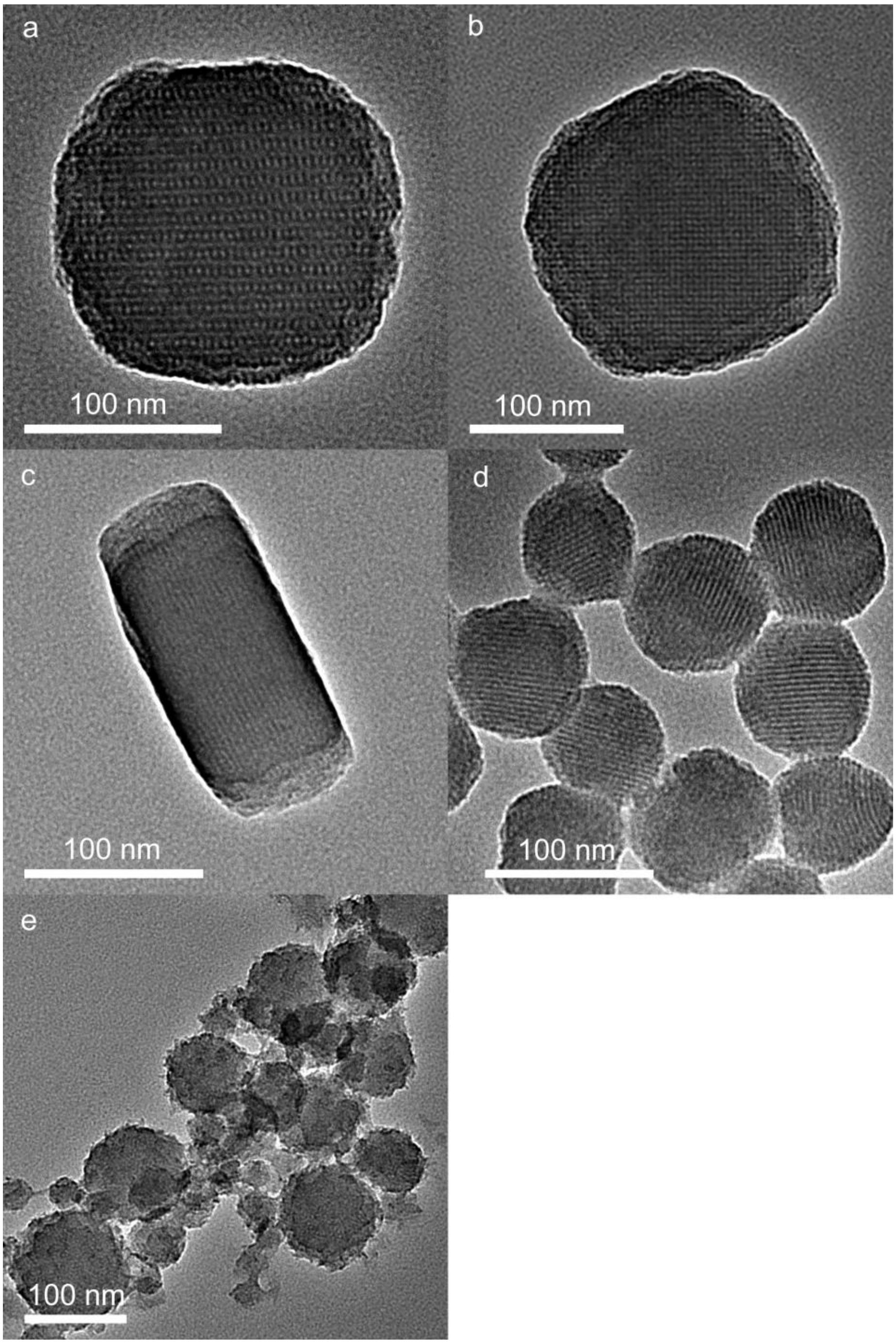

Fig. S1 TEM images of (a) ethylene-PMO nanoparticle at [210] projection of *Pm*3*n*, (b) ethylene-PMO nanoparticle at [110] projection of *Pm*3*n*, (c) methylene-PMO nanoparticle at $[10\bar{1}0]$ projection of $P6_3/mmc$, (d) ethenylene-PMO nanoparticle perpendicular to the channel direction, (e) phenylene-PMO nanoparticle prepared in the absence of cosolvent.

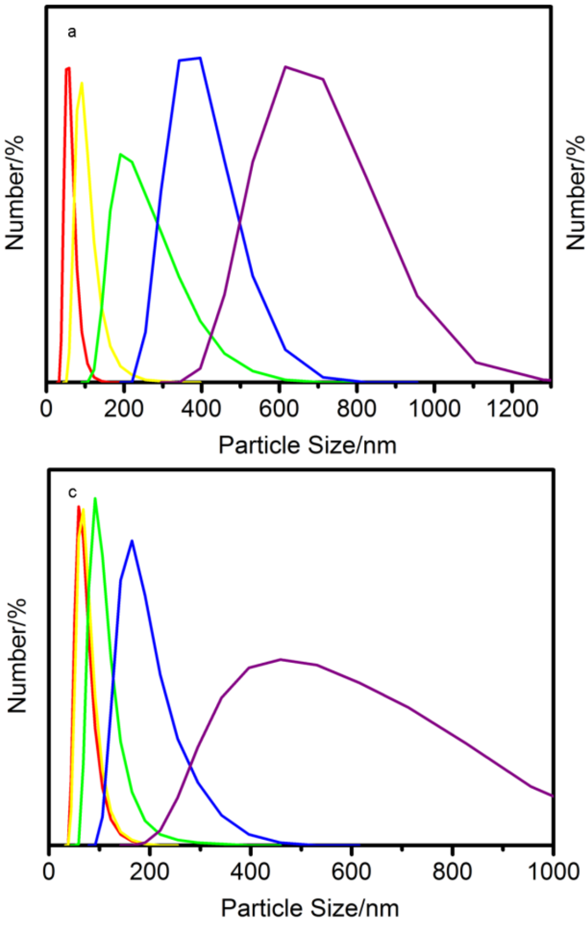

Fig. S2 DLS measurements of PMO nanoparticles synthesized with organosilica units including (a) methylene, (b) ethenylene, and (c) phenylene-bridged silanes. For each material, the five curves are shown corresponding to the particles prepared with the concentration of ammonia from 0.088 M (red), 0.14 M (yellow), 0.35 M (green), 0.88 M (blue) to 2.2 M (purple).

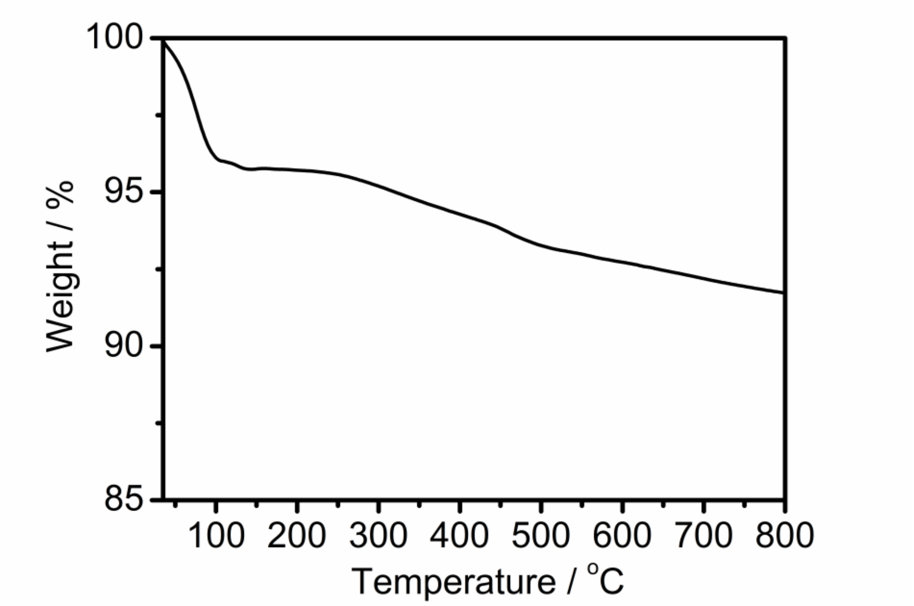

Fig. S3 The thermogravimetric analysis (TG) of methylene-PMO nanoparticle after extraction in an acidic ethanol solution.