

Supplementary Material (ESI) for New Journal of Chemistry
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One-Pot Reaction Cascades Catalyzed by Base and Acid Confined Inside Pores of Mesoporous Silica Nanospheres

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1. Synthesis of mesoporous silica nanosphere (MSN) containing organic acid confined inside pores (SAMSN) and MSN containing organic base confined inside pores (APMSN)

SAMSN and APMSN were synthesized via the co-condensation reaction from our group.¹⁻³ A typical procedure is as following: A mixture of cetyltrimethylammonium bromide (CTAB, 5.49 mmol), 2.0 mol / L of aqueous NaOH (7 mL, 14 mmol) and water (480 mL, 26.67 mol) was heated at 80 °C for 30 min. Into this clear solution, tetraethylorthosilicate (TEOS, 10 mL, 51.40 mmol) and 1.0 mmol of 2-(4-chlorosulfonylphenyl)ethyltrimethoxysilane for SAMSN (or 3-aminopropyltrimethoxysilane for APMSN) were added rapidly and sequentially via injection with vigorous stirring. Within few minutes, the white solid precipitate was observed. After 2 hours, the as-synthesized material was separated by hot filtration, washed with copious amount of methanol, dried under vacuum overnight. The surfactant (CTAB) was removed by acid extraction at 60 °C with vigorous stirring by placing 1.0 gram of as-made solid in 100 mL methanol including 1.0 mL concentrated hydrochloride acid for 6 hours. The resulted surfactant-removed solid SAMSN (or APMSN) was collected by hot filtration, washed with copious amount of water and methanol, dried at 90 °C under vacuum overnight.

2. Characterization of MSB-acid and APMSN

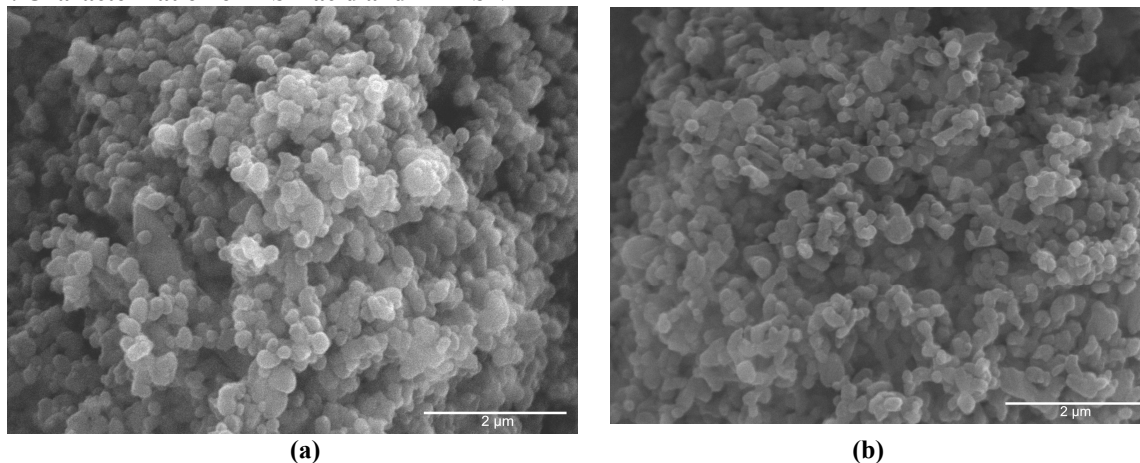


Figure S1. Scanning electron microscopy (SEM) of SAMSN (a) and APMSN (b).

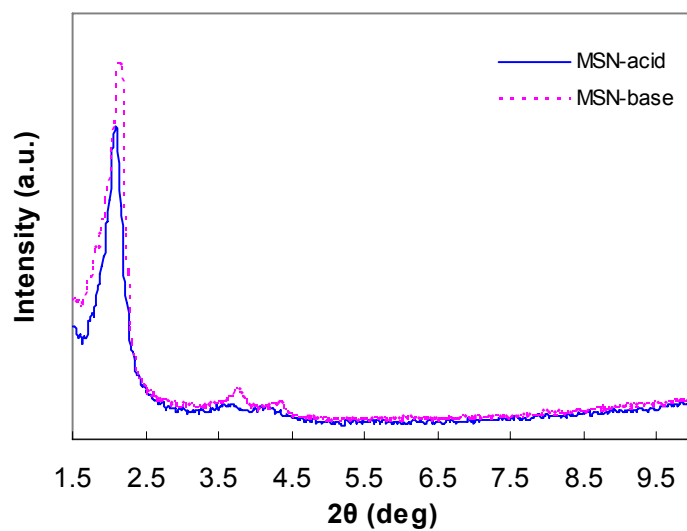
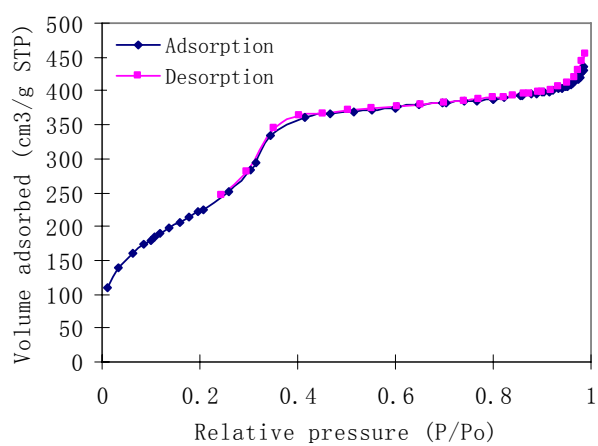
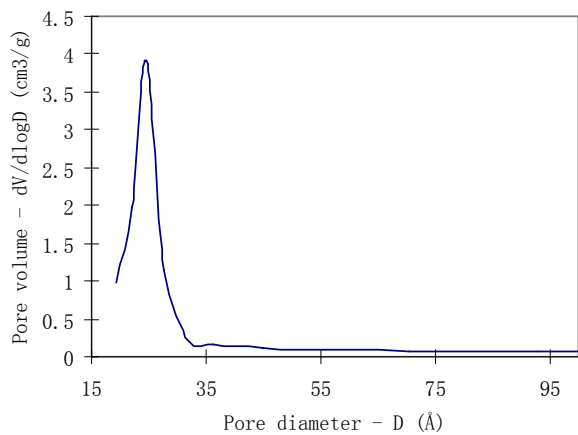
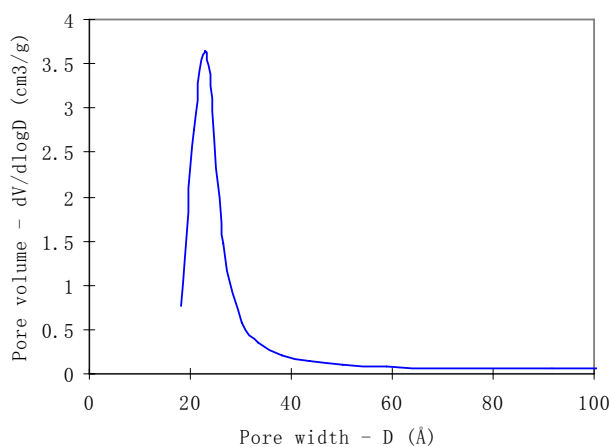
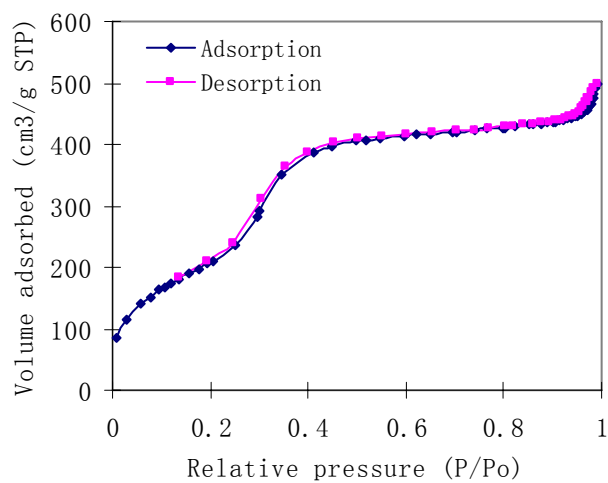


Figure S2. Powder XRD data of SAMSN and APMSN.





(a) SAMSN



(b) APMSN

Figure S3. BET isotherms and BJH pore diameter distribution curves of SAMSN and APMSN.

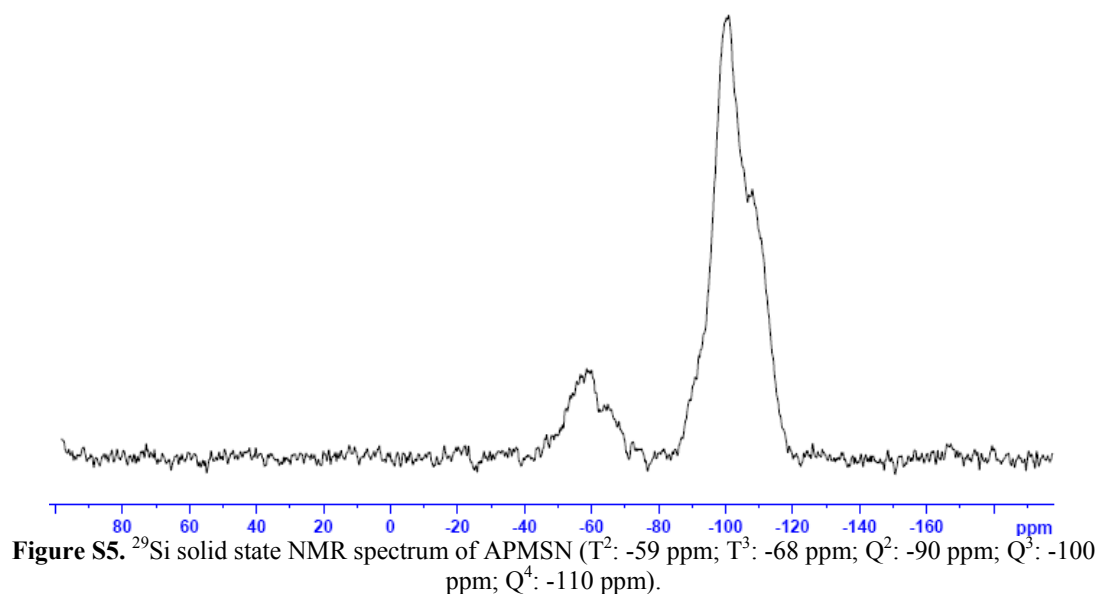
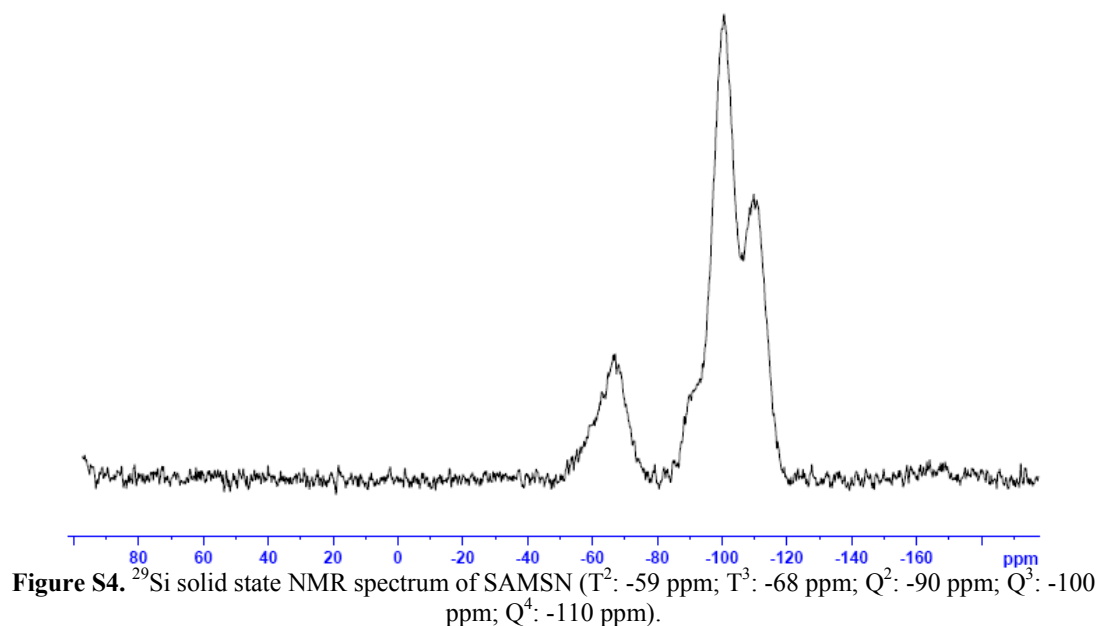


Table S1. Structural data of SAMSN and APMSN.^a

Samples	d_{100} (Å)	S_{BET} ($\text{m}^2 \text{g}^{-1}$)	V_p ($\text{cm}^3 \text{g}^{-1}$)	D_{BJH} (Å)
SAMSN	42.5	827.9	0.67	25.4
APMSN	40.9	789.0	0.74	22.3

[a] The BET surface area (S_{BET}), the mesopore volume (V_p), and the mean mesopore diameter (D_{BJH}) were obtained from the nitrogen absorption / desorption data in Figure S3. The d_{100} data represent the d-spacing corresponding to the (100) peaks in XRD (Figure S2).

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3. One-pot reaction cascades

4-nitrobenzaldehyde dimethyl acetal (0.75 mmol) and water (0.75 mmol) were mixed with SAMSN (or free acid, and the acid quantity is decided by the reaction conditions) and APMSN (or free base, and the base quantity is decided by the reaction conditions) at room temperature under N₂ and sealed in a glass vessel. The as-made slurry was heated to 80 °C with vigorous stirring. After 24 hours, the reaction was stopped by cooling to room temperature. The product was analyzed by GC-MS with capillary column (HP-5, 30 m x 0.319 mm x 0.25 μm).

(1) Huh, S.; Wiench, J.W.; Yoo, J.C.; Pruski, M.; Lin, V.S.Y. *Chem. Mater.* **2003**, 15, 4247 – 4256.

(2) Huh, S.; Wiench, J.W.; Trewyn, B.G.; Song, S.; Pruski, M.; Lin, V.S.Y. *Chem. Commun.* **2003**, 2364.

(3) Radu, D.R.; Lai, C.Y.; Huang, J.; Xu, S.; Lin, V.S.Y. *Chem. Commun.* **2005**, 1264 – 1266.