Electronic Supplementary Material (ESI) for RSC Advances


# Supporting Information

## Controllable Synthesis of Hollow Mesoporous Silica Nanoparticles Templated Kinetic Self-Assembly of Gemini Surfactant

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### 1. Synthesis of mesoporous silica nanoparticles (MSNs)

**General Methods.** Materials obtained commercially were used without further purification. N,N,N',N'-tetramethylethylenediamine (TMEDA) (AR, SCRC), bromotetradecane(AR, Alatin), bromohexadecane(AR, Alatin), bromodecane(AR, Alatin), acetonitrile(AR, SCRC), chloroform(AR), acetone(AR, SCRC), sodium hydroxide(AR, SCRC), tetraethyl orthosilicate (TEOS, 99%, Alfar Aesar). NMR spectra were measured on a Bruker AV400 spectrometer. Field-emission scanning electron microscopy (FE-SEM) images were obtained on a FEI Sirion 200 instrument. Transmission electron microscopy (TEM) images were obtained on a FEI Tecnai G220 instrument. X-Ray powder diffraction (XRD) patterns were recorded on X'Pert PRO X-ray diffractometer with Cu-K radiation with a wavelength of 0.1542 nm. The nitrogen adsorption experiments were performed at 77 K on a Micromeritics ASAP 2020 instrument. Samples were degassed at 573 K for 4 h prior to the measurements.

**Synthesis of gemini surfactants $C_{14\text{-}2\text{-}14}$ and $C_{16\text{-}2\text{-}16}$[S1]**

The TMEDA (0.1 mol) and 1-bromotetradecane or 1-Bromohexadecane (0.4 mol) were refluxed in MeCN for 2 days. After evaporation, the residue was re-crystallized from $CHCl_3$ /acetone yielding the $C_{14\text{-}2\text{-}14}$ or $C_{16\text{-}2\text{-}16}$.

$C_{14\text{-}2\text{-}14}$: Yield, 80.1%. $^1$H NMR ($CDCl_3$, 400MHz): δ, 0.88 (t, 6H, *J*=6.8Hz), 1.26-1.38(m, 44H), 1.82(s, 4H), 3.52(s, 12H), 3.71(t, 4H, *J*=8.0Hz), 4.77(s, 4H).

$C_{16\text{-}2\text{-}16}$: Yield, 69.6%. $^1$H NMR ($CDCl_3$, 400MHz): δ, 0.88(t, 6H, *J*=7.2Hz), 1.26-1.37(m, 52H), 1.81(s, 4H), 3.49(s, 12H), 3.69(t, 4H, *J*=7.6Hz), 4.60(s, 4H).

**Synthesis of gemini surfactants $C_{14\text{-}2\text{-}10}$ [S1]**

The TMEDA (0.1 mol) and 1-bromotetradecane (0.08 mol) reacted in MeCN (125 ml) at 40 °C for 3 days. After evaporation and crystallization from $Et_2O$, the corresponding pure $C_{14\text{-}2}$ was isolated in 42.0% yield. $C_{14\text{-}2}$ (0.1mol) and the 1-bromodecane (4 equiv.) were refluxed in EtOAc (125 ml) for 2 days, and purified by re-crystallization from $CHCl_3$/acetone yielding $C_{14\text{-}2\text{-}6}$ in 52.3%.

$C_{14\text{-}2\text{-}10}$: $^1$H NMR ($CDCl_3$, 400MHz): δ, 0.88(t, 6H, *J*=7.2Hz), 1.26-1.38(m, 36H), 1.82(s, 4H), 3.52(s, 12H), 3.72(t, 4H, *J*=7.6Hz), 4.78(s, 4H).

**Synthesis of mesoporous silica nanoparticls(MSNs) templated with $C_{14\text{-}2\text{-}14}$**

To solution of $C_{14\text{-}2\text{-}14}$ (0.218g) in distilled water (240ml), sodium hydrate solution (2M, 1.75ml) were added at 80 ºC and stirred. After a period of pre-assembly time (*t* = 10, 30, 60 and 120 min), TEOS (2.5ml) was dropped slowly into the mixture solution and then kept stirring for 2h. The MSNs were obtained by filtration. Finally, the surfactant template ($C_{14\text{-}2\text{-}14}$) was removed by refluxing in a mixture of methanol and hydrochloric acid.

**Synthesis of HMSNs templated with $C_{14\text{-}2\text{-}10}$**

Following the similar procedure of MSNs templated with $C_{14\text{-}2\text{-}14}$, used the template of $C_{14\text{-}2\text{-}10}$, **HMSNs** were obtained.

**Synthesis of HMSNs templated with $C_{16\text{-}2\text{-}16}$**

Following the similar procedure of MSNs templated with $C_{14\text{-}2\text{-}14}$, used the template of $C_{16\text{-}2\text{-}16}$ with a longer pre-assembly time of *t* = 180 min, **HMSNs** were obtained.

## 2. Characterization of MSNstemplated with $C_{14\text{-}2\text{-}14}$

### 2.1 TEM images of MSNs templated with $C_{14\text{-}2\text{-}14}$

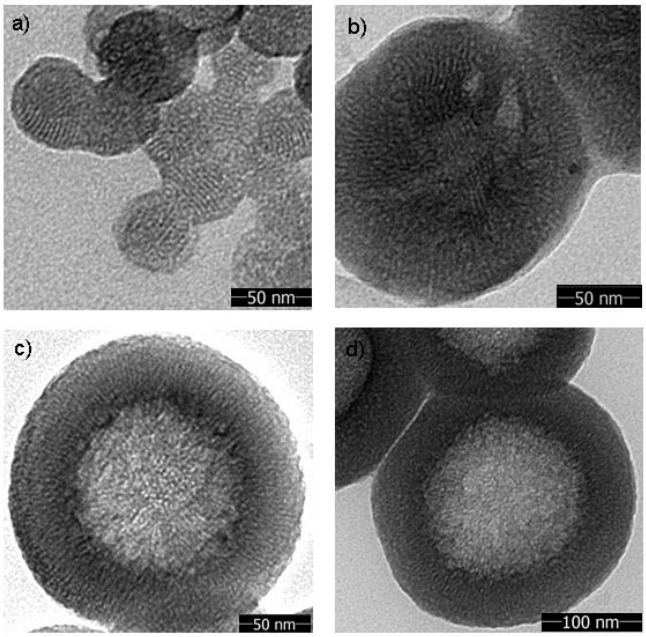


**Fig. S1**. TEM images of MSNs templated with $C_{14\text{-}2\text{-}14}$ from different pre-assembly time of $t$ = 10 min (a), 30 min (b), 60 min (c) and 120 min (d).

### 2.2 SEM images of MSNs templated with $C_{14\text{-}2\text{-}14}$

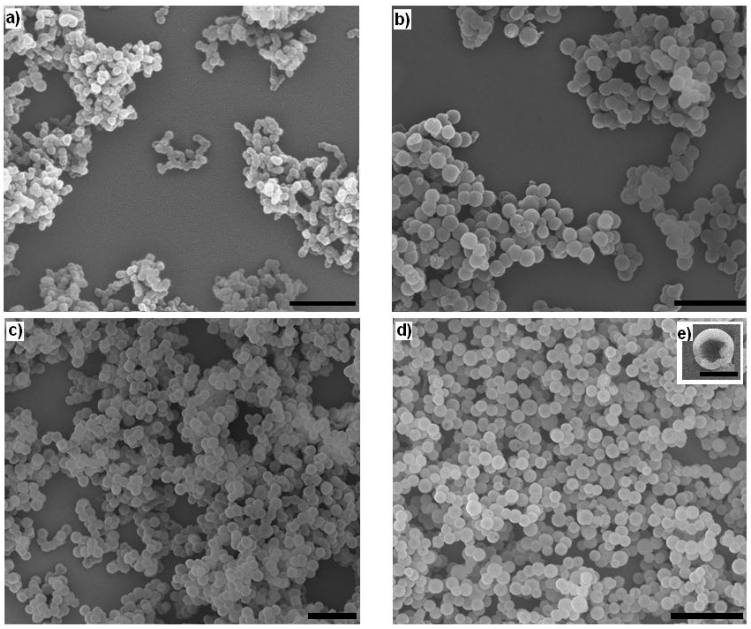


**Fig. S2**. SEM images of MSNs templated with $C_{14\text{-}2\text{-}14}$ from different pre-assembly time of $t$ = 10 min (a), 30 min (b), 60 min (c) and 120 min (d). The SEM image of broken HMSNs of $t$ = 120 min (e). Scale bar: 500 nm (a), 1μm (b and c), 2μm (d) and 200 nm (e).

### 2.3 XRD spectra of MSNs templated with $C_{14\text{-}2\text{-}14}$

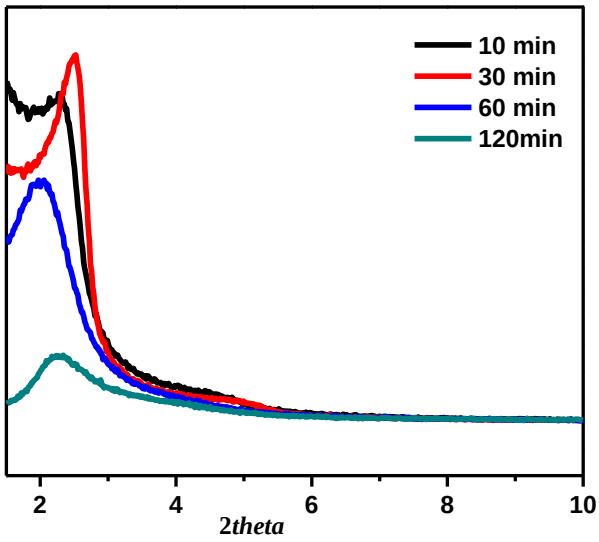


**Fig. S3**. XRD spectra of MSNs templated with $C_{14\text{-}2\text{-}14}$.

### 2.3 Nitrogen sorption isotherms and pore size distribution of MSNs templated with $C_{14\text{-}2\text{-}14}$

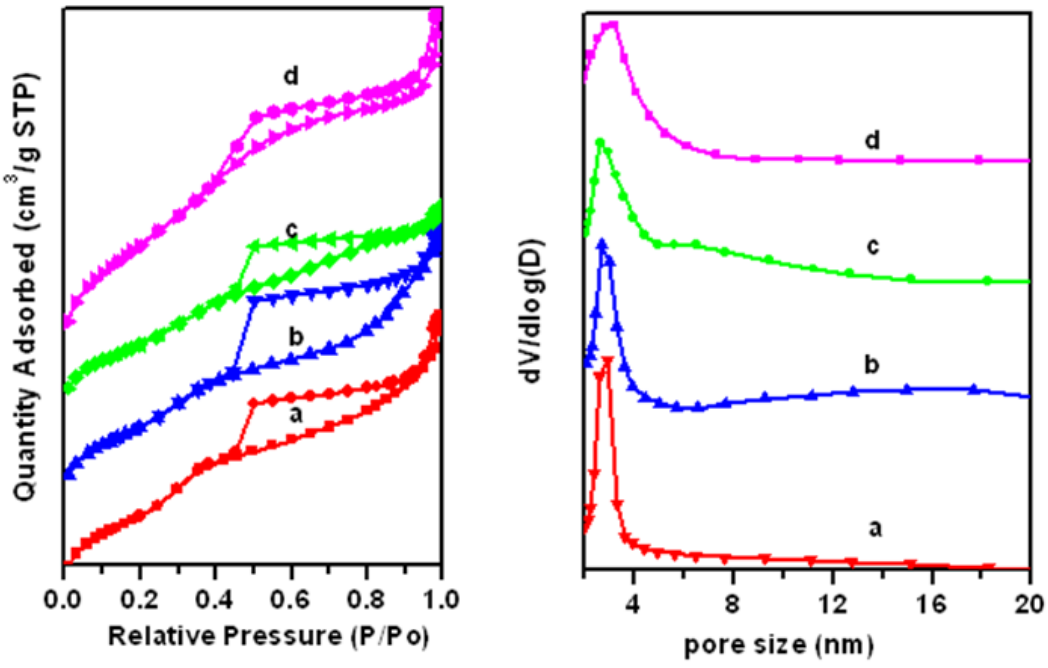


**Fig. S4**. Nitrogen adsorption isotherms (left) and Barett-Joyner-Halenda (BJH) pore size distribution (right) of HMSNs templated with $C_{14\text{-}2\text{-}14}$ from different pre-assembly time of $t$ = 10 min (a), 30 min (b), 60 min (c) and 120 min (d).

## 3. Characterization of HMSNs templated with $C_{14\text{-}2\text{-}10}$

### 3.1 SEM and TEM images of HMSNs templated with $C_{14\text{-}2\text{-}10}$

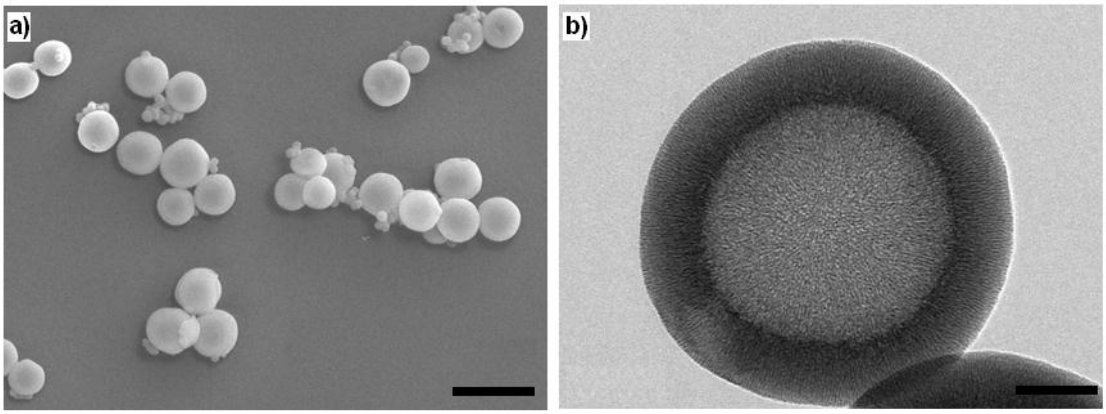


**Fig. S5**. SEM (a) and TEM (b) images of HMSNs templated with $C_{14\text{-}2\text{-}10}$. Scale bar: 500 nm (a), 50nm(b).

### 3.2 XRD spectrum of HMSNs templated with $C_{14\text{-}2\text{-}10}$

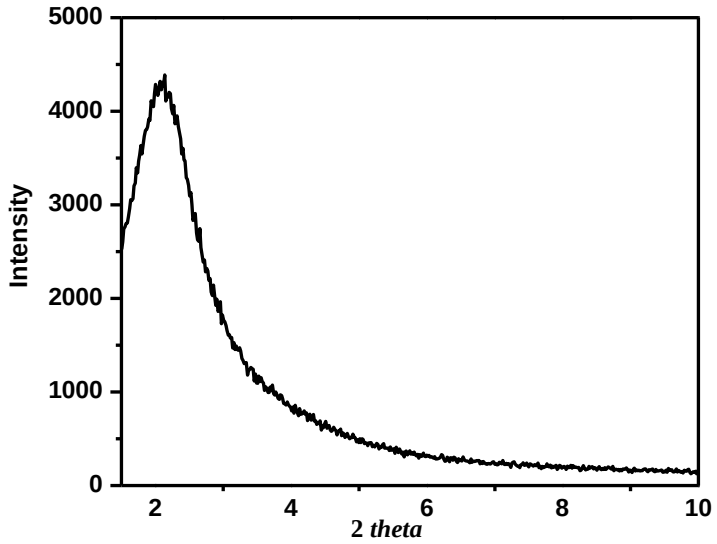


**Fig. S6**. XRD spectrum of HMSNs templated with $C_{14\text{-}2\text{-}10}$

### 3.3 Nitrogen sorption isotherms and pore size distribution of HMSNs templated with $C_{14\text{-}2\text{-}10}$

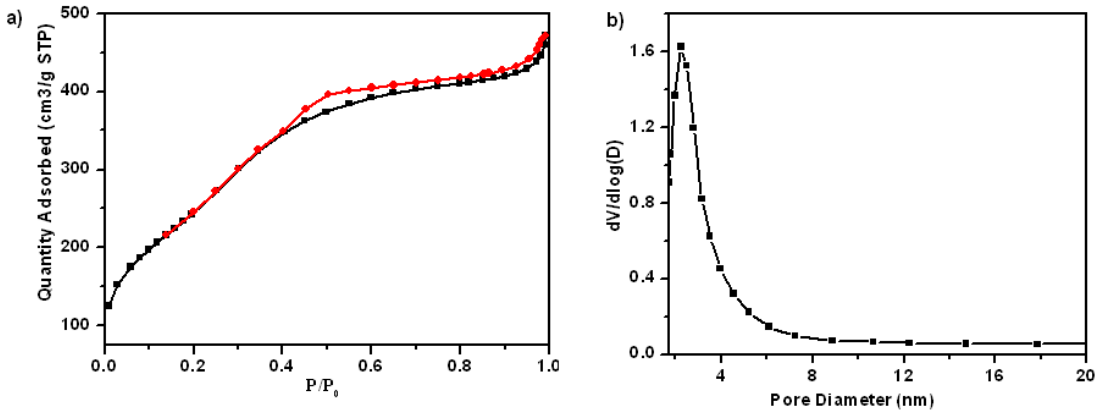


**Fig. S7**. Nitrogen adsorption isotherm (a) and BJH pore size distribution (b) of HMSNs templated with $C_{14\text{-}2\text{-}10}$.

### 4. Characterization of HMSNstemplated with $C_{16\text{-}2\text{-}16}$

### 4.1 SEM and TEM images of HMSNs templated with $C_{16\text{-}2\text{-}16}$

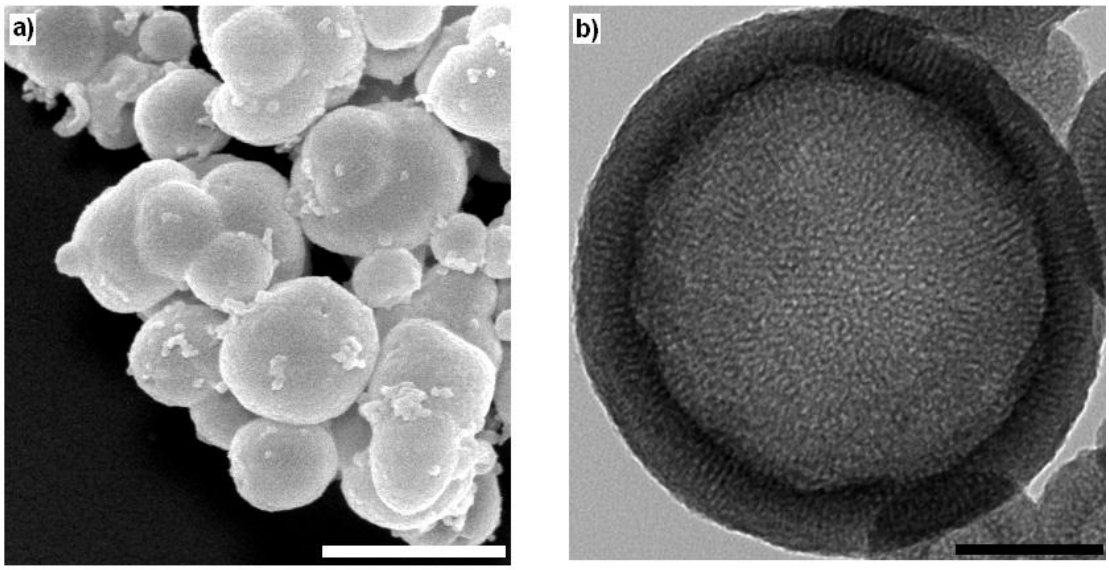


**Fig. S8**. SEM (a) and TEM (b) images of HMSNs templated with $C_{16\text{-}2\text{-}16}$. Scale bar: 500 nm (a), 100nm (b).

### 4.2 XRD spectrum of HMSNs templated with $C_{16\text{-}2\text{-}16}$

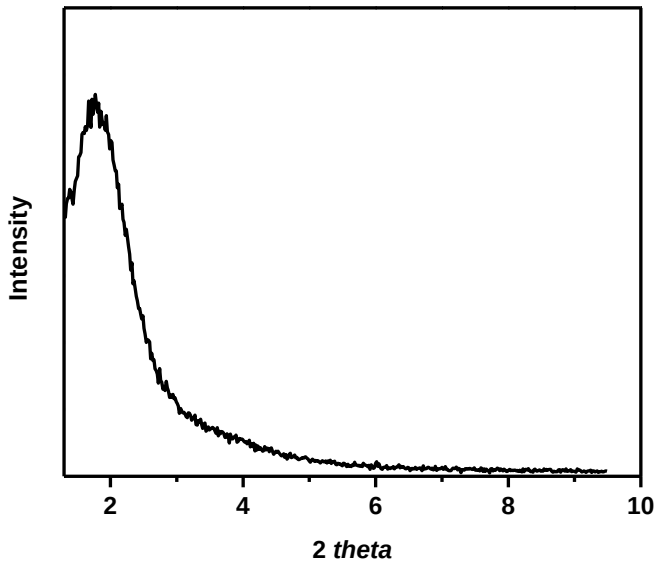


**Fig. S9**. XRD spectrum of HMSNs templated with $C_{16\text{-}2\text{-}16}$

### 4.3 Nitrogen sorption isotherms and pore size distribution of HMSNs templated with $C_{16\text{-}2\text{-}16}$

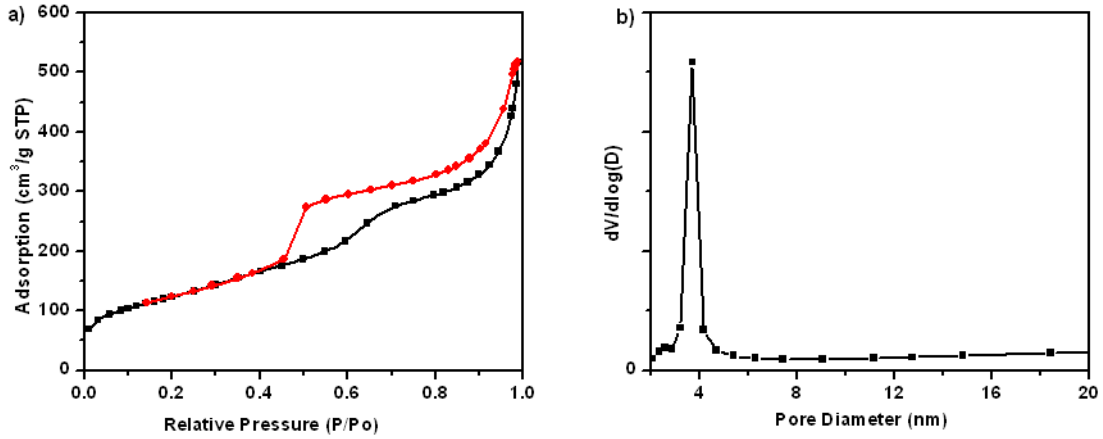


**Fig. S10**. Nitrogen adsorption isotherm (a) and BJH pore size distribution (b) of HMSNs templated with $C_{16\text{-}2\text{-}16}$.

### Reference

[S1] R. Oda, I. Huc, S. J. Candau, *Chem. Commun.* ***1997***, 2105.