

**Single-micelle-templated Synthesis of Hollow
Silica Nanospheres with Tunable Pore Structures**

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Supporting Table S1. Summary of synthesis conditions for Pluronic-F108-templated silicas.^a

Sample	Initial synthesis temperature (°C)	Swelling agents (mL)	Precursors	Hydrothermal treatment temperature (°C)
HSN01	14			
HSN02	18			
HSN03	20			
HSN04	22	toluene (3 mL)	TEOS (1.5 mL)	100
HSN05	23			
HSN06	25			
HSN07	r.t. (~25)			
HSN08		N/A		
HSN09		TIPB (3.4 mL)		
HSN10	25	cyclohexane (3.06 mL)	TEOS (1.5 mL)	100
HSN11		<i>m</i> -xylene (3.46 mL)		
HSN12		ethylbenzene (3.44 mL)		
HSN13		toluene	TEOS (2.2 mL)	
HSN14	25	(3 mL)	TMOS (1.0 mL)	100
HSN15				r.t. (~25)
HSN16				60
HSN17	25	toluene (3 mL)	TEOS (1.5 mL)	80
HSN18				120
HSN19				130

^a Taken in part from Li, Y. *Ph.D. Dissertation*, City University of New York, 2014.

Supporting Table S2. Structural parameters for calcined silicas synthesized at different initial temperatures.^{a,b}

Sample	Initial synthesis temperature (°C)	w _{KJS} (nm)	S _{BET} (m ² /g)	V _t (cm ³ /g)
HSN01	14	44	838	1.23
HSN02	18	37	744	1.57
HSN03	20	30	681	1.44
HSN04	22	28.3	789	1.95
HSN05	23	22.7	771	1.81
HSN06	25	16	761	1.65
HSN07	r.t. (~25) ^c	16.1	807	1.75

^a Notation: w_{KJS}, pore diameter calculated using BJH-KJS method; S_{BET}, BET specific surface area; V_t: total pore volume. ^b Li, Y. *Ph.D. Dissertation*, City University of New York, 2014. ^c Synthesized at room temperature without temperature control.

Supporting Table S3. Structural parameters for calcined silica samples synthesized at different initial synthesis temperatures by using either Pluronic F108/ethylbenzene or F108/*m*-xylene pair as the template.^{a,b}

Template	Initial synthesis temperature (°C)	w_{KJS} (nm)	S_{BET} (m ² /g)	V_t (cm ³ /g)
F108/ethylbenzene	14	27.9	942	0.64
	18	31.0	875	0.94
	22	30.7	805	1.37
	25	25.6	713	1.65
F108/ <i>m</i> -xylene	14	27.7	823	0.55
	18	30.7	652	0.68
	20	30.8	729	1.06
	22	27.3	688	1.38
	25	21.6	710	1.58

Notation^a: w_{KJS} , pore diameter calculated by using BJH-KJS method for cylindrical mesopores; S_{BET} , BET specific surface area; V_t : total pore volume. ^b Data for samples prepared at 25 °C and from Li, Y. *Ph.D. Dissertation*, City University of New York, 2014.

Supporting Table S4. Structural parameters of calcined silicas synthesized at 25 °C using different swelling agents.^{a,b}

Sample	swelling agent	w_{KJS} (nm)	S_{BET} (m ² /g)	V_{t} (cm ³ /g)
HSN08	N/A	N/A	730	0.36
HSN09	TIPB	13.1	840	0.58
HSN10	Cyclohexane	15.6	884	0.58
HSN06	Toluene	16.0	761	1.65
HSN11	<i>m</i> -Xylene	21.6	710	1.58
HSN12	Ethylbenzene	25.6	713	1.65

^a Notation: w_{KJS} , pore diameter calculated by using BJH-KJS method; S_{BET} , BET specific surface area; V_{t} : total pore volume. ^b Li, Y. *Ph.D. Dissertation*, City University of New York, 2014.

Supporting Table S5. Structural parameters for calcined silica samples synthesized with different amount or kind of precursor.^a

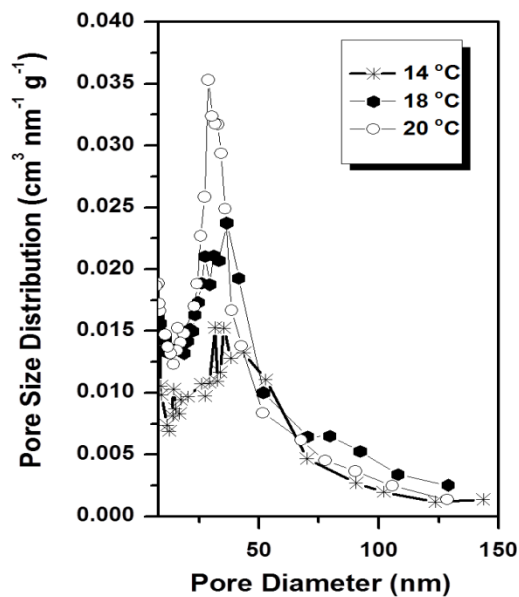
Sample	Silica Precursor (mL)	w_{KJS} (nm)	S_{BET} (m ² /g)	V_{t} (cm ³ /g)
HSN06	TEOS (1.5)	16.0	761	1.65
HSN13	TEOS (2.2)	17.8	707	1.68
HSN14	TMOS (1.0)	20.7	757	1.67

^a Notation: w_{KJS} , pore diameter calculated by using BJH-KJS method; S_{BET} , BET specific surface area; V_{t} : total pore volume. Adapted from Li, Y. *Ph.D. Dissertation*, City University of New York, 2014.

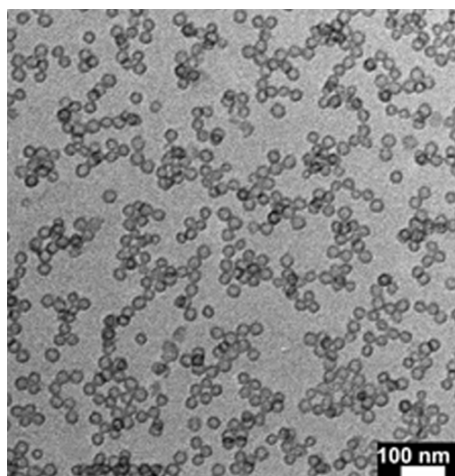
Supporting Table S6. Structural parameters for calcined silicas synthesized at different hydrothermal treatment temperatures.^{a,b}

Sample	Hydrothermal treatment temperature (°C)	w_{KJS} (nm)	S_{BET} (m ² /g)	V_t (cm ³ /g)
HSN15	room temperature (~25)	9.5	481	0.43
HSN16	60	10.7	655	0.60
HSN17	80	13.1	775	0.77
HSN06	100	16.0	761	1.65
HSN18	120	16.7	658	1.87
HSN19	130	23.2	585	2.19

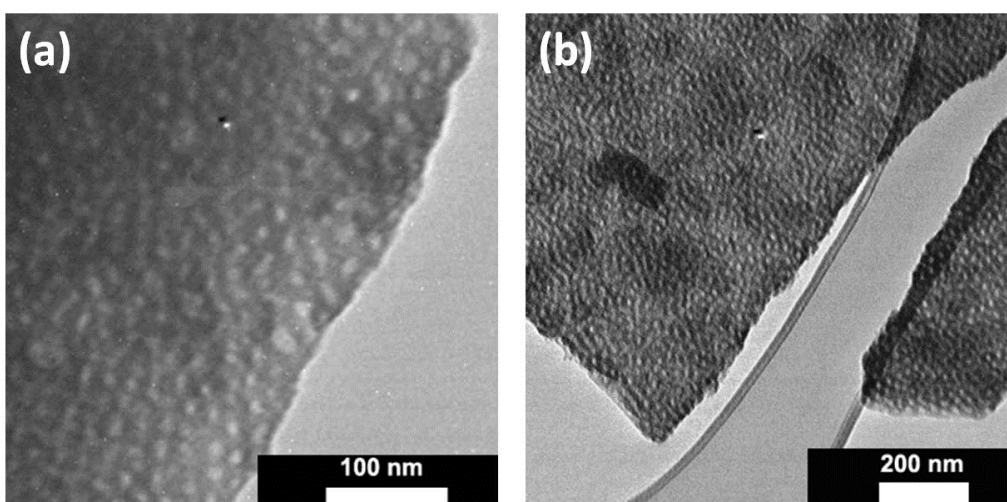
^a Notation: w_{KJS} , pore diameter calculated by using BJH-KJS method; S_{BET} , BET specific surface area; V_t : total pore volume. ^b Adapted in part from Li, Y. *Ph.D. Dissertation*, City University of New York, 2014.



Supporting Figure S1. The pore size distribution of the samples of calcined hollow silica nanospheres synthesized with Pluronic F108/toluene pair at 14, 18 and 20 °C.

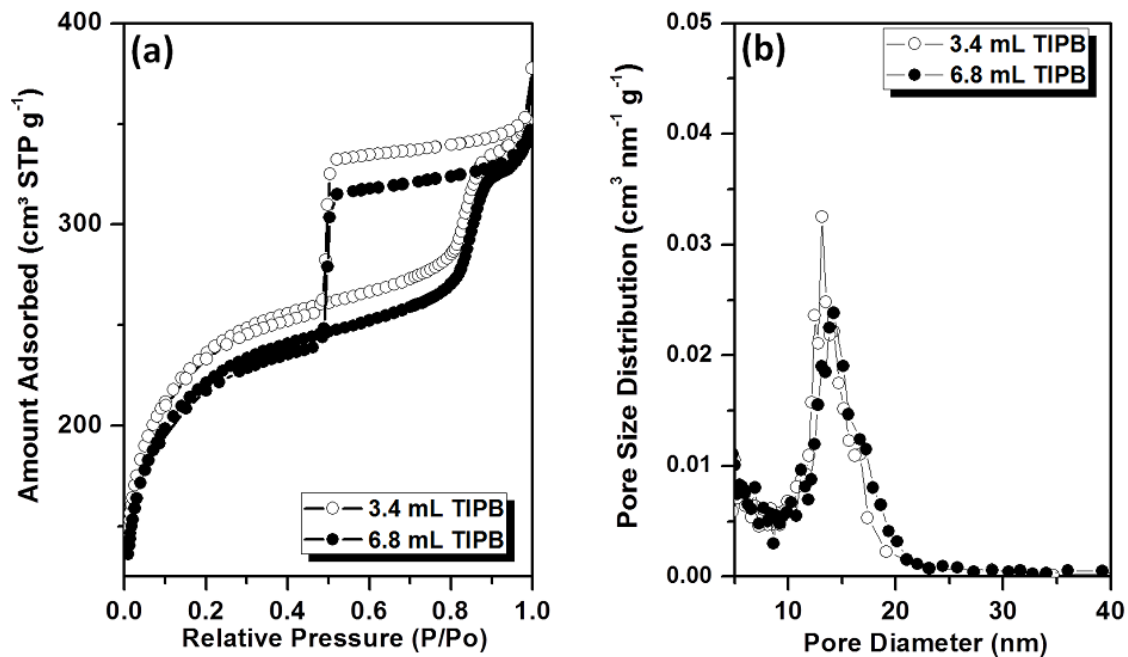


Supporting Figure S2. Transmission electron microscopy image of hollow silica nanospheres synthesized at 25 °C for ethanol extracted HSN13.



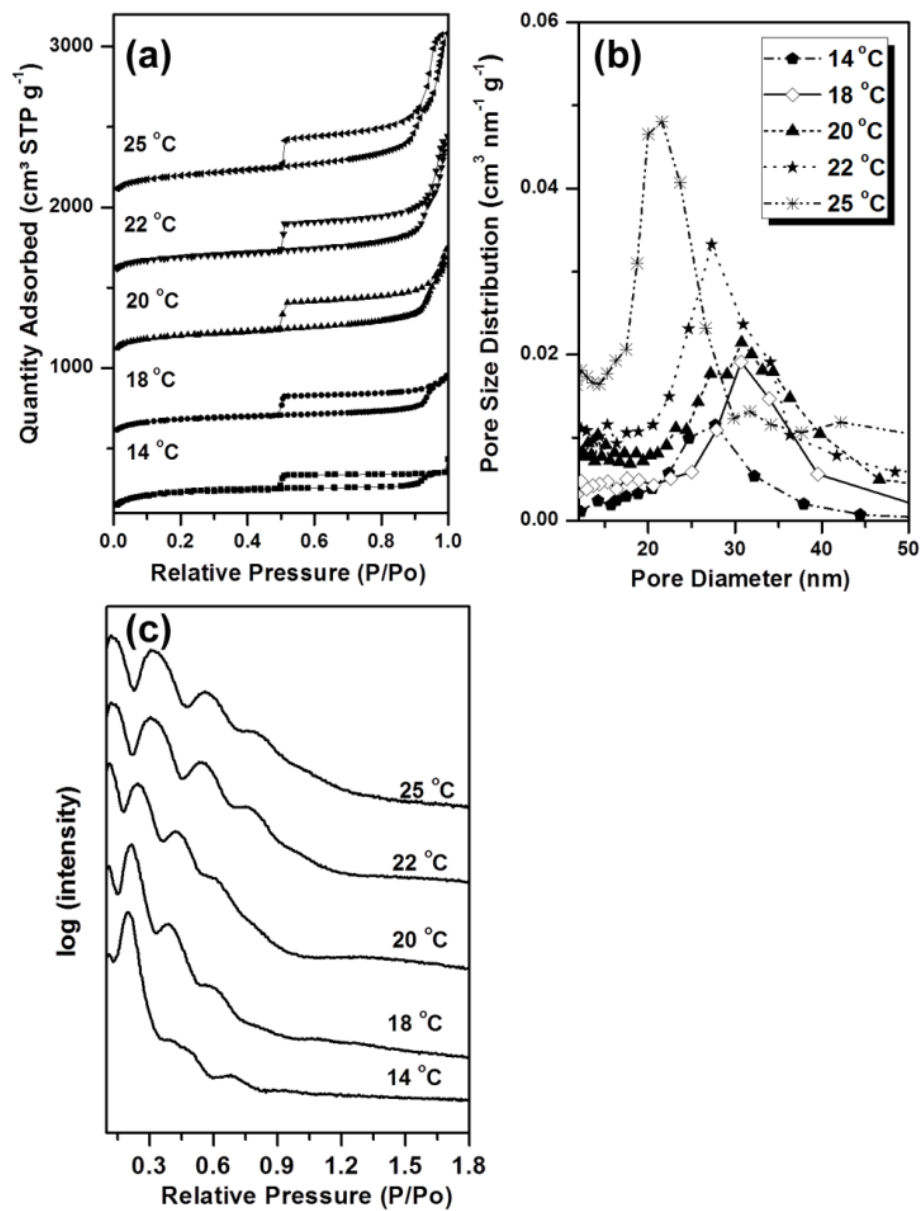
Supporting Figure S3. TEM images of silica samples synthesized at 25 °C with (a) TIPB and (b) cyclohexane swelling agents (samples were hydrothermally treated at 100 °C). Li, Y. *Ph.D.*

Dissertation, City University of New York, 2014.

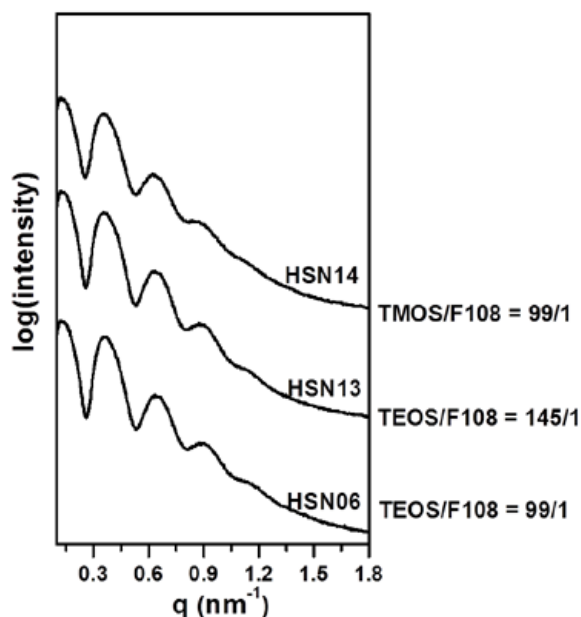


Supporting Figure S4. Nitrogen adsorption isotherms (a) and pore size distributions (b) for calcined silicas synthesized at the initial temperature of 25 °C with different amounts TIPB.

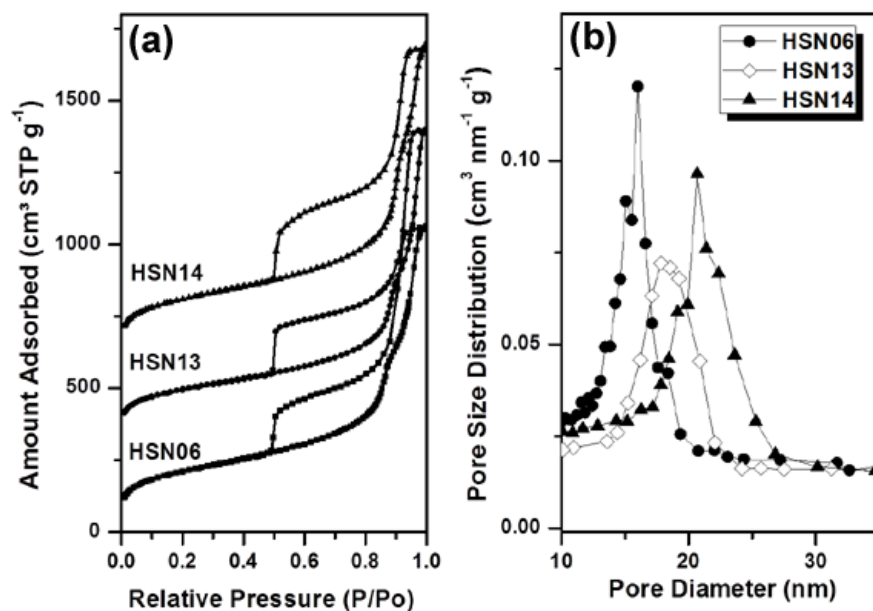
Adapted in part from Li, Y. *Ph.D. Dissertation*, City University of New York, 2014.



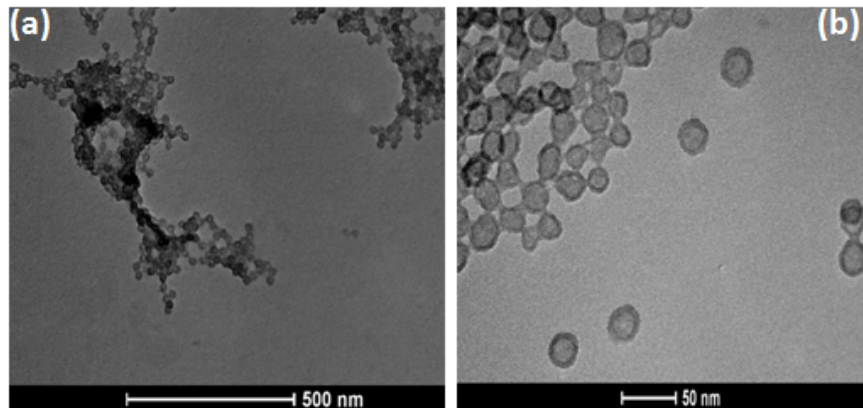
Supporting Figure S5. (a) Nitrogen adsorption isotherms, (b) pore size distributions and (c) SAXS patterns for calcined silicas initially synthesized at different temperatures using F108/*m*-xylene pair. Li, Y. *Ph.D. Dissertation*, City University of New York, 2014.



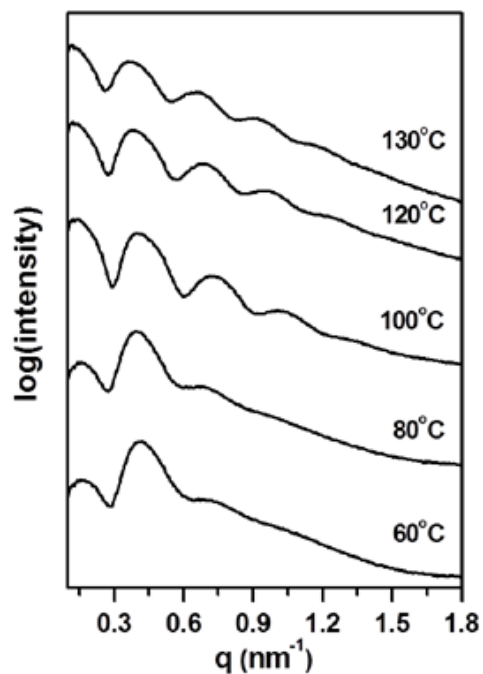
Supporting Figure S6. SAXS patterns of HSNs samples prepared with different silica precursors using F108/toluene pair as the template, with hydrothermal treatment at 100 °C. Adapted from Li, Y. *Ph.D. Dissertation*, City University of New York, 2014.



Supporting Figure S7. (a) Nitrogen adsorption isotherms and (b) pore size distributions for calcined silicas synthesized with different amount or kind of the framework precursor using toluene at 25 °C. Adapted from Li, Y. *Ph.D. Dissertation*, City University of New York, 2014.



Supporting Figure S8. TEM images of the as-synthesized samples of (a) HSN15 that was not hydrothermally treated, and (b) HSN17 that was hydrothermally treated at 80 °C. Taken from Li, Y. *Ph.D. Dissertation*, City University of New York, 2014.



Supporting Figure S9. XRD patterns of samples prepared at an initial synthesis temperature of 25 °C using Pluronic F108/toluene pair as the template, but with aging at different temperatures. Adapted in part from Li, Y. *Ph.D. Dissertation*, City University of New York, 2014.