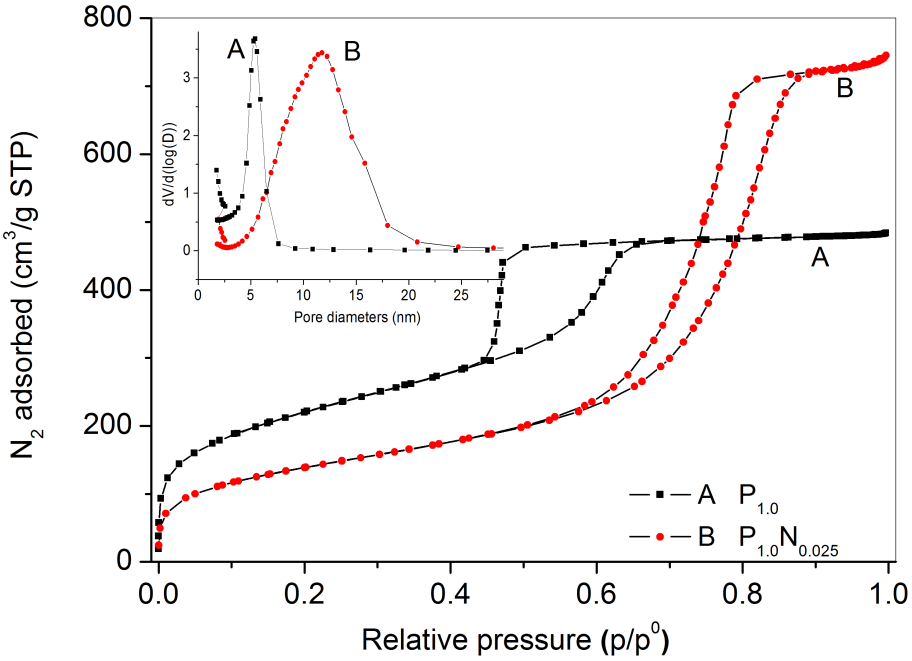
Electronic Supplementary Material (ESI) for RSC Advances

Water-deficient templating system: a general, versatile and efficient synthetic approach for mesoporous silicas

Electronic Supplementary Information

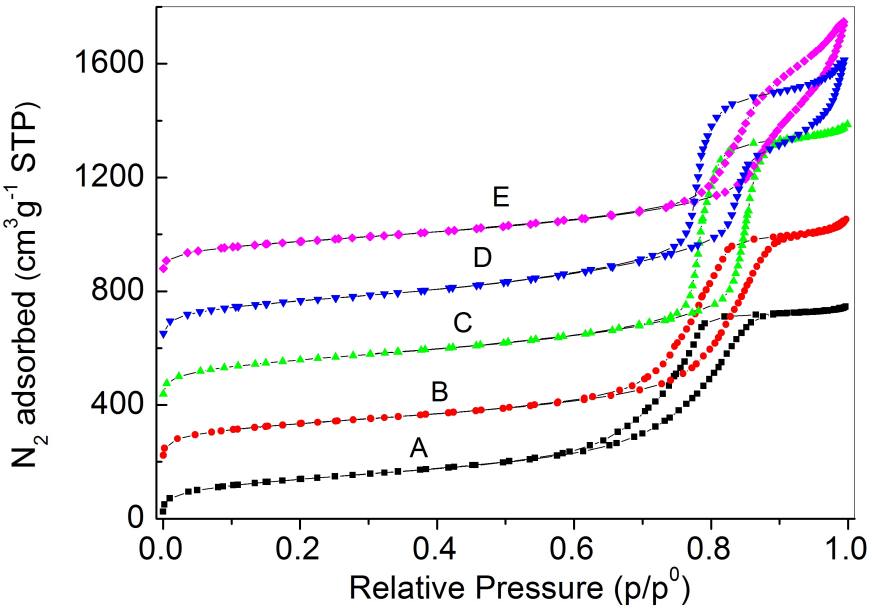
S1



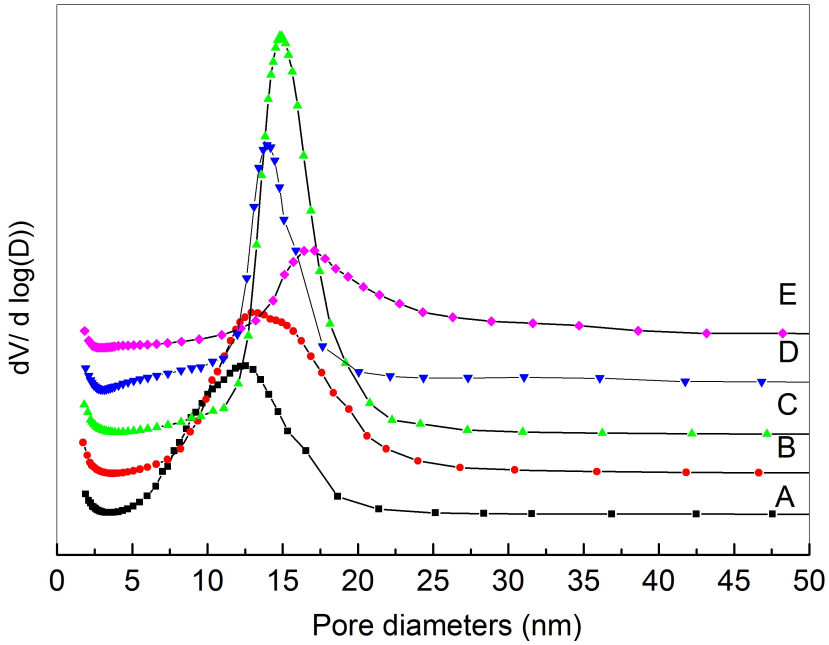
Nitrogen sorption results and corresponding pore size distributions (BDB) (inset) of wormhole-like meso-silicas prepared *via* the WDT system using 1.0 g P123.

Physico-properties of meso-silica prepared

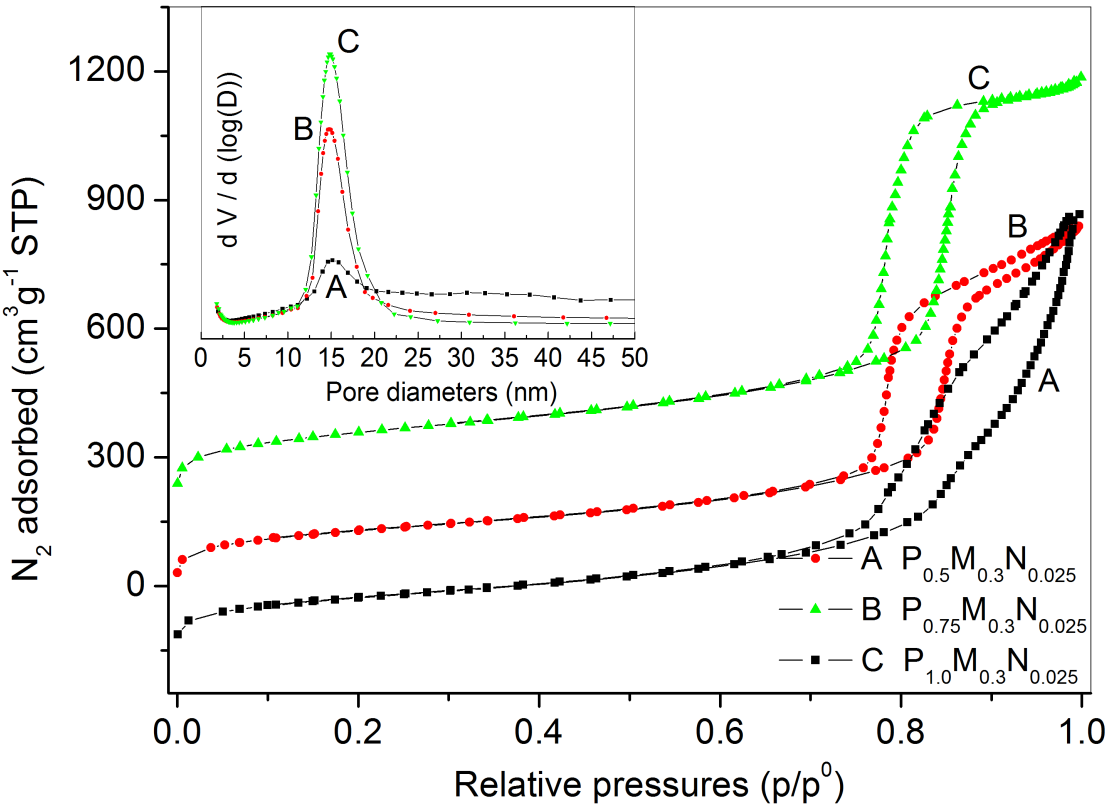
Sample No.	BET /m^2g^{-1})	V_{total} (cm^3g^{-1})	D_{pore} /nm
$P_{1.0}N_{0.025}$	489	1.15	11.7/9.3



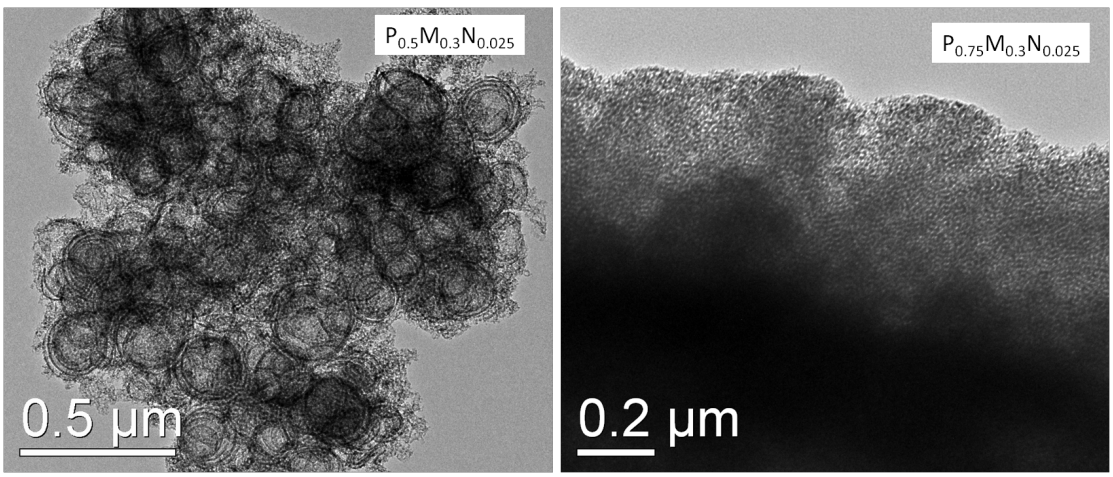
Nitrogen adsorption-desorpion curves of (A) $P_{1.0}N_{0.025}$, (B) $P_{1.0}M_{0.15}N_{0.025}$, (C) $P_{1.0}M_{0.3}N_{0.025}$, (D) $P_{1.0}M_{0.45}N_{0.025}$. and (E) $P_{1.0}M_{0.6}N_{0.025}$. $P_{1.0}M_{0.3}N_{0.025}$ was included here for comparison purpose.



Pore size distributions (KJS) of (A) $P_{1.0}N_{0.025}$, (B) $P_{1.0}M_{0.15}N_{0.025}$, (C) $P_{1.0}M_{0.3}N_{0.025}$, (D) $P_{1.0}M_{0.45}N_{0.025}$. and (D) $P_{1.0}M_{0.6}N_{0.025}$. $P_{1.0}M_{0.3}N_{0.025}$ was included here for comparison purpose.

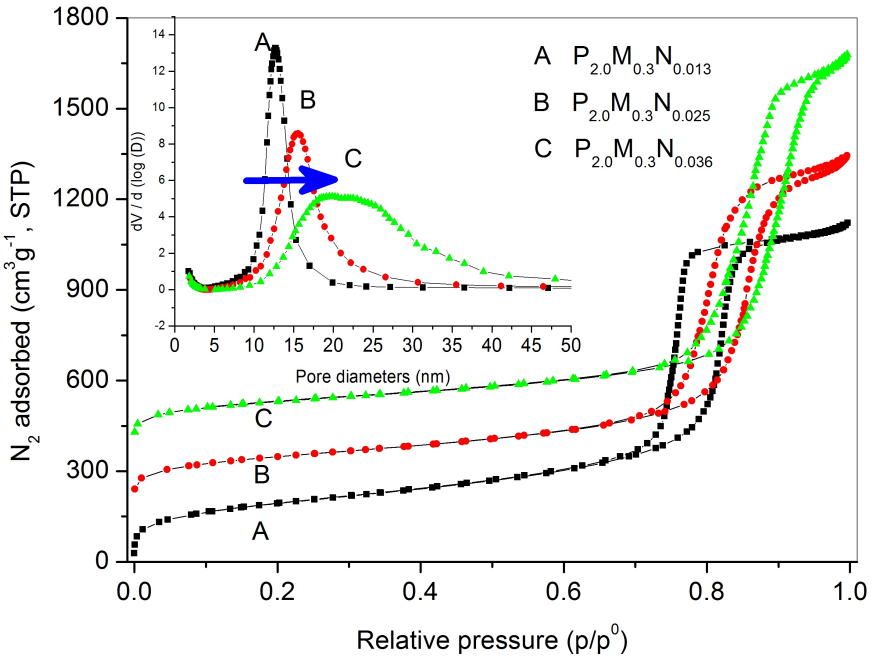


Nitrogen sorption results of meso-silicas prepared using 0.3g TMB but with varying P123 amounts. Inset shows corresponding pore size distributions (KJS). $P_{1.0}M_{0.3}N_{0.025}$ was included here for comparison purpose.



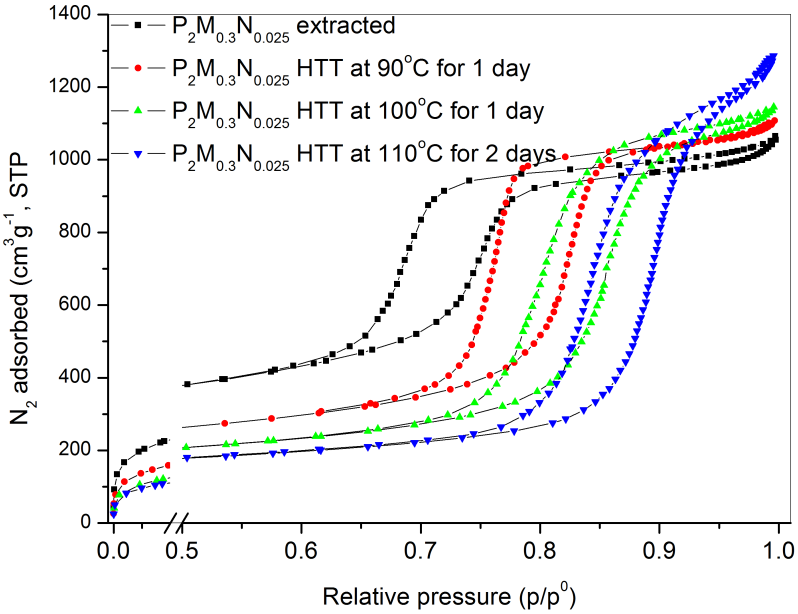
TEM of $P_{0.5}M_{0.3}N_{0.025}$ & $P_{0.75}M_{0.3}N_{0.025}$

Note: the broadened main peak and the loss of higher angle XRD peaks of $P_{0.75}M_{0.3}N_{0.025}$ indicate the formation of disordered mesostructures, as confirmed by the TEM results. Further reduction in the P123 led to disordered mesostructures full of spherical pores.

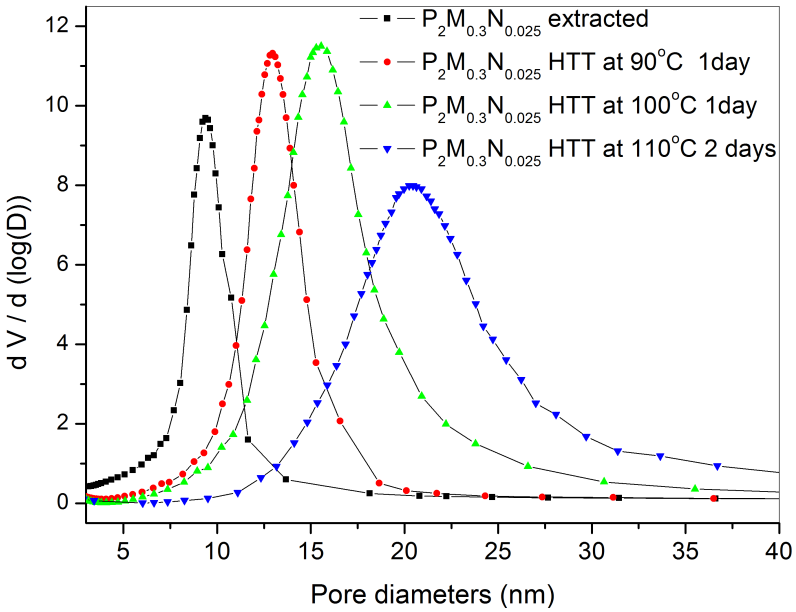


Nitrogen sorption results and corresponding pore size distributions (inset, KJS) of (A) $P_{2.0}M_{0.3}N_{0.013}$, (B) $P_{2.0}M_{0.3}N_{0.025}$, (C) $P_{2.0}M_{0.3}N_{0.036}$. The isotherms are subject to vertical shifts for clarity.

Note: the enlarged mesopores and more broadening in the PSDs can be seen from the meso-silicas prepared with increasing amounts of NH_4F.



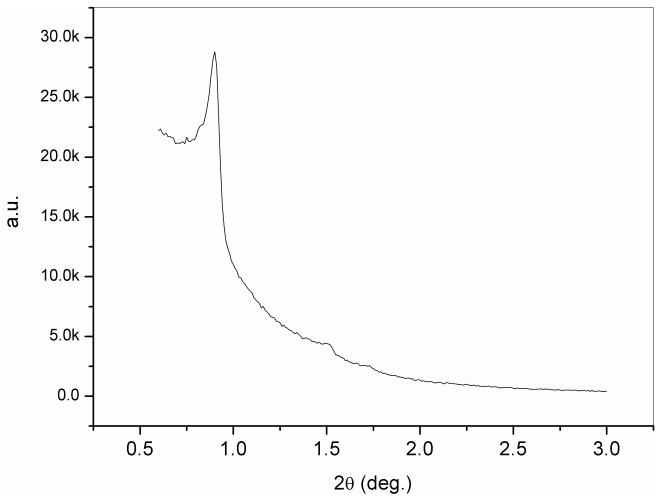
N_2 adsorption-desorpion curves of $P_{1.0}M_{0.3}N_{0.025}$ prepared without (extracted one) or with different HTT conditions after low-temperature step (40°C) (from left to right): 90°C 1d, 100°C 1d, 110°C 2d.



PSDs of extracted one, and ones with different HTTs (from left to right): 90°C 1d, 100°C 1d, 110°C 2d.

Physico-properties of meso-silica prepared with varying HTTs

Sample No.	BET /m^2g^{-1})	V_{total} (cm^3g^{-1})	D_{pore} /nm	i-KJS/nm
extracted	941	1.63	9.4	8.8
HTT 90°C/1d	661	1.70	13.0	10.6
HTT 100°C/1d	554	1.51	14.9	11.8
HTT 110°C/2d	456	1.97	20.5	14.0



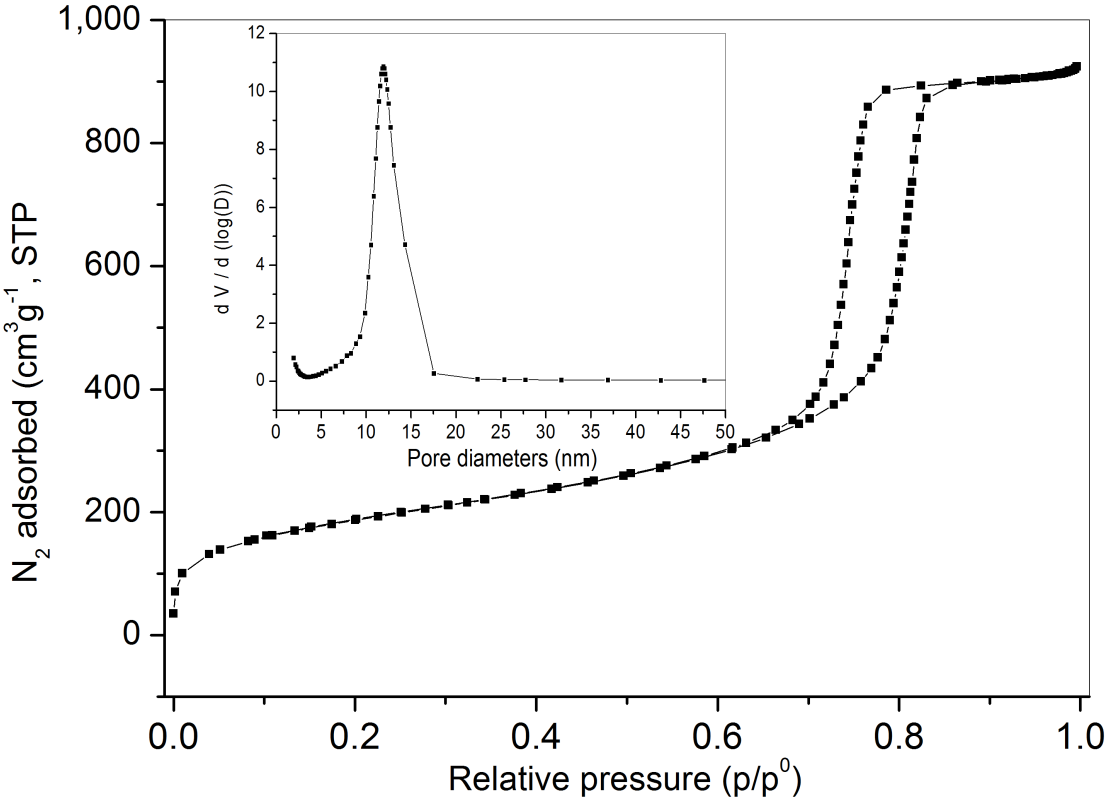
Low-angle XRD pattern for $P_{2.0}M_{0.3}N_{0.025}$ prepared with HTT at 110°C for 2 days, indiating the preservation of meso-ordering.

Control 1#

Synthesis

1.0 g P123 and 0.3g TMB were added in 4.25g tetraethoxysilane under stirring (800 rpm) at 40 ^{o}C to form a clear solution, to which 0.33g c-HCl diluted by 7.67g de-ionized water containing 0.025g NH_4F was added. The rest procedures see the Experimental in the manuscript.

N_2 sorption results



Nitrogen sorption results and corresponding pore size distributions (inset) of $P_{1.0}M_{0.3}N_{0.025}$ using total 8.0g diluted HCl with half amount of c-HCl.

Sample No.	BET /m^2g^{-1})	V_{total} (cm^3g^{-1})	*a* /nm	D_{pore} /nm	i-KJS/nm	Mesophase
control 1#	653	1.43	11.9	11.9	10.6	p6mm

Contol 2#

Synthesis

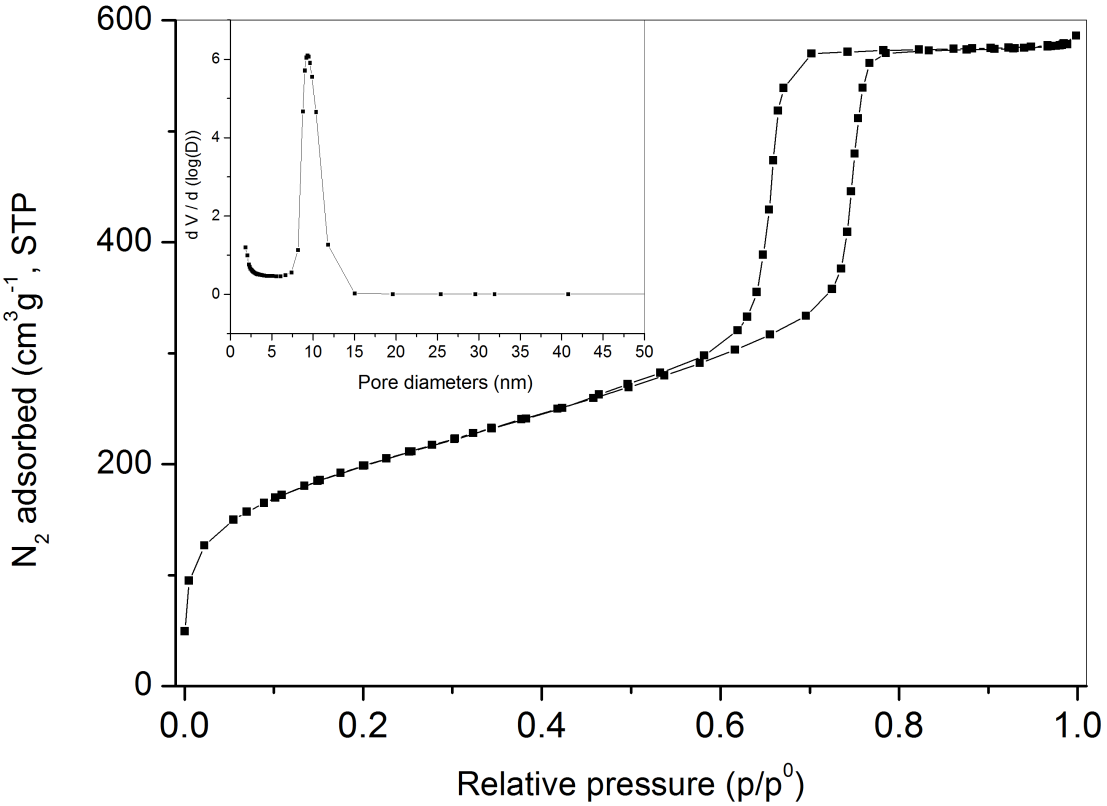
1.0 g P123 and 0.3g TMB were added in 4.25g tetraethoxysilane under stirring (800 rpm) at 40 ^{o}C to form a clear solution, to which 0.5g c-HCl diluted by 5.5g de-ionized water containing 0.025g NH_4F was added. The rest procedures see the Experimental in the manuscript.

Contol 3#

Synthesis

10 g P123 and 3g TMB were added in 42.5g tetraethoxysilane under stirring (800 rpm) at 40 ^{o}C to form a clear solution, to which 6.67g c-HCl diluted by 73.3g de-ionized water was added. The rest procedures see the Experimental in the manuscript.

N_2 sorption results



Nitrogen sorption results and corresponding pore size distributions (inset) of $P_{10.0}M_{3.0}$.

Sample No.	BET /m^2g^{-1})	V_{total} (cm^3g^{-1})	*a* /nm	D_{pore} /nm	i-KJS/nm	Mesophases
control 3#	693	1.03	11.8	9.3	8.7	p6mm

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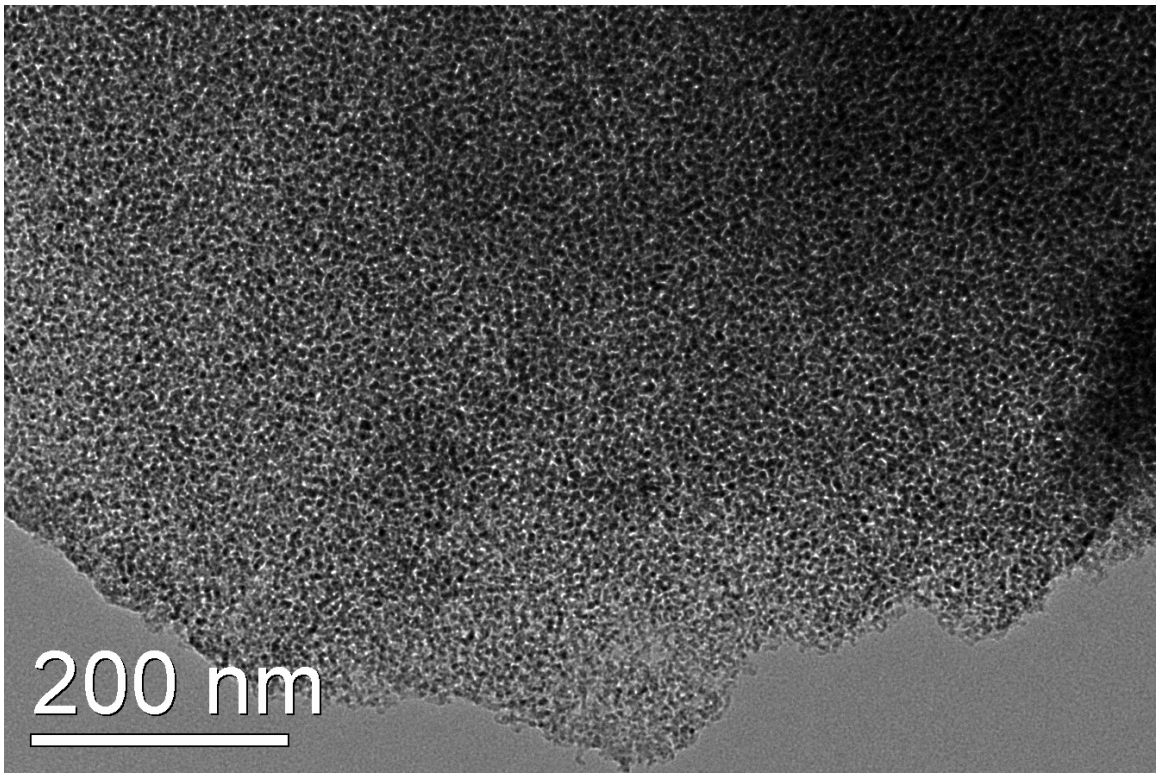
Contol 4#

Synthesis

1.0 g P123 and 0.23g n-hexane (the same volume as 0.3g TMB) were added in 42.5g tetraethoxysilane under stirring (800 rpm) at 40 ^{o}C to form a clear solution, to which 0.67g c-HCl diluted by 7.33g de-ionized water was added. The rest procedures see the Experimental in the manuscript.

TEM result



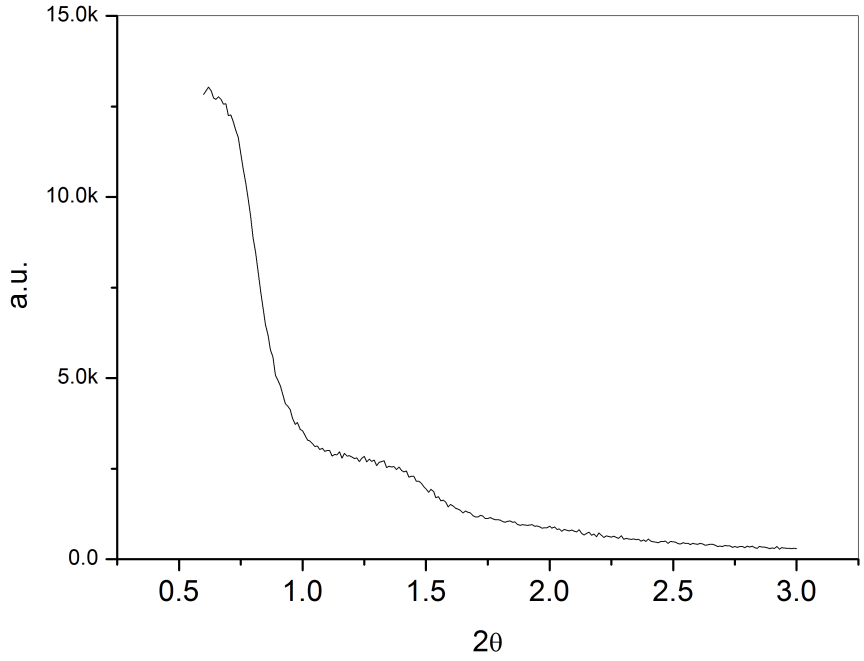
Note: TEM image show the wormhole-like mesostructures rather than the 2D hexagonal one for the mesosilica prepared via the WDT system using n-hexane as swelling agent.

Contol 5#

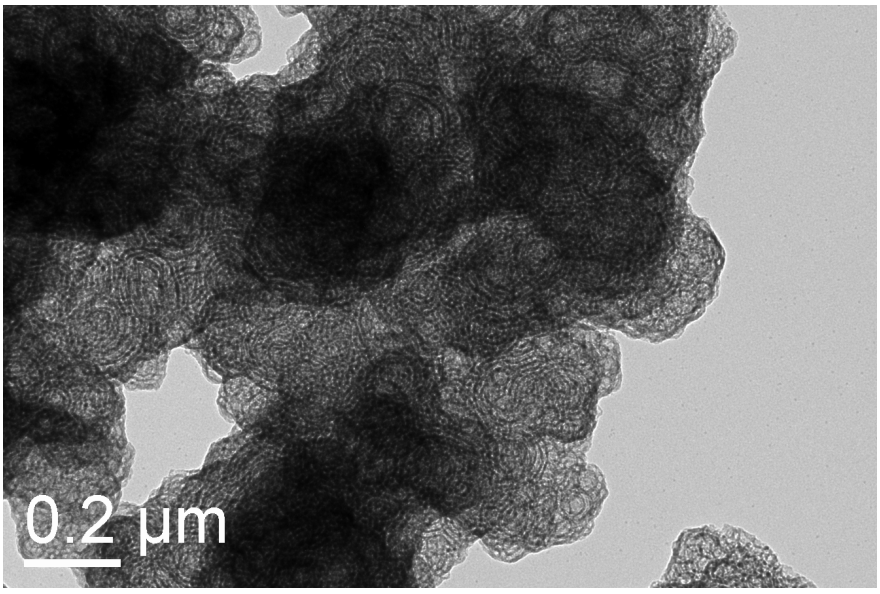
Synthesis

1.0 g P123 and 0.3g TMB was added to 60 g containg 11.9g c-HCl at 40 ^{o}C to form a clear solution, to which 4.25 TEOS was added. The rest procedures see the Experimentat in the manuscript.

Low-angle XRD result

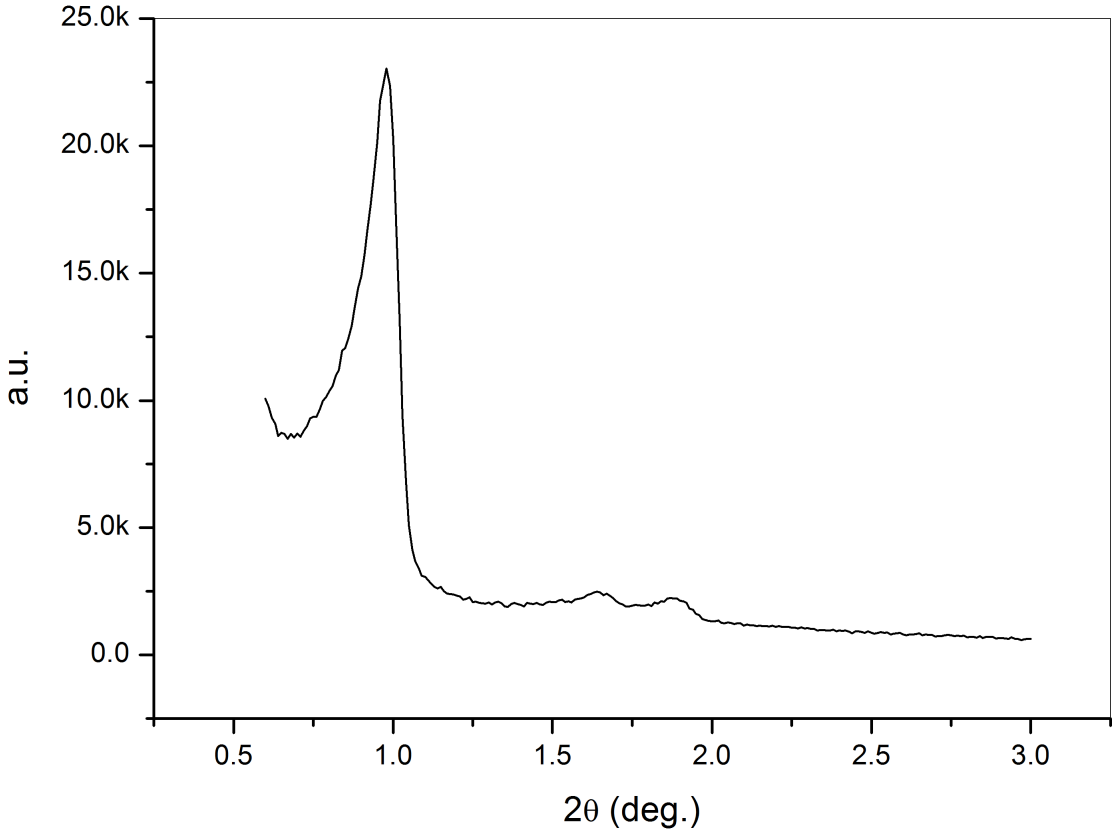


TEM result



Note: low angle-XRD and TEM results both demonstrated that the mesostructures are lack of ordering and show to be a mixture of MCF and wormhole-like structures.The

disordering arised from the use of TMB, because the meso-silica prepared via the procedure shown above but without adding TMB showed to be hexagonally ordered according to the low-angle XRD shown below.



Low-angle XRD pattern of meso-silica prepared **without using TMB** *via* the DSS approach as shown in the Synthesis.