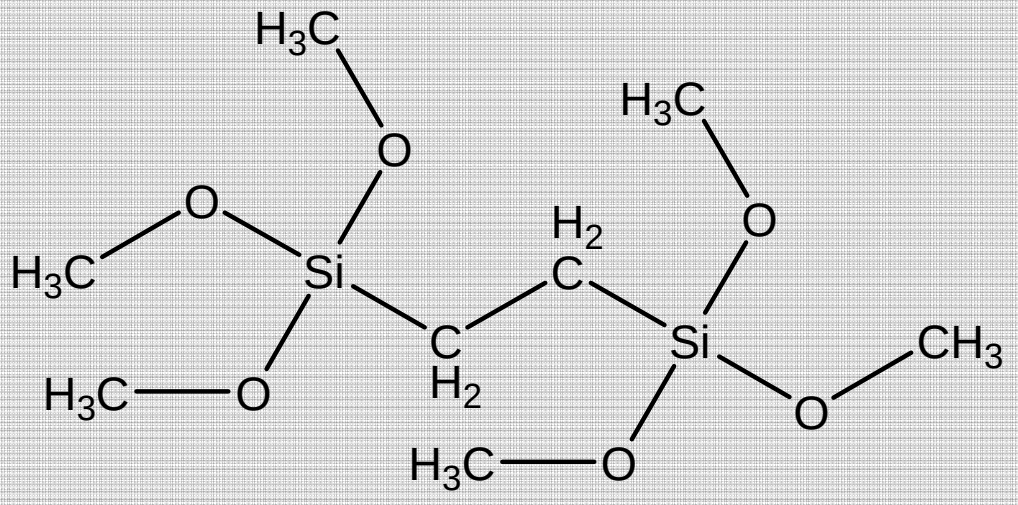
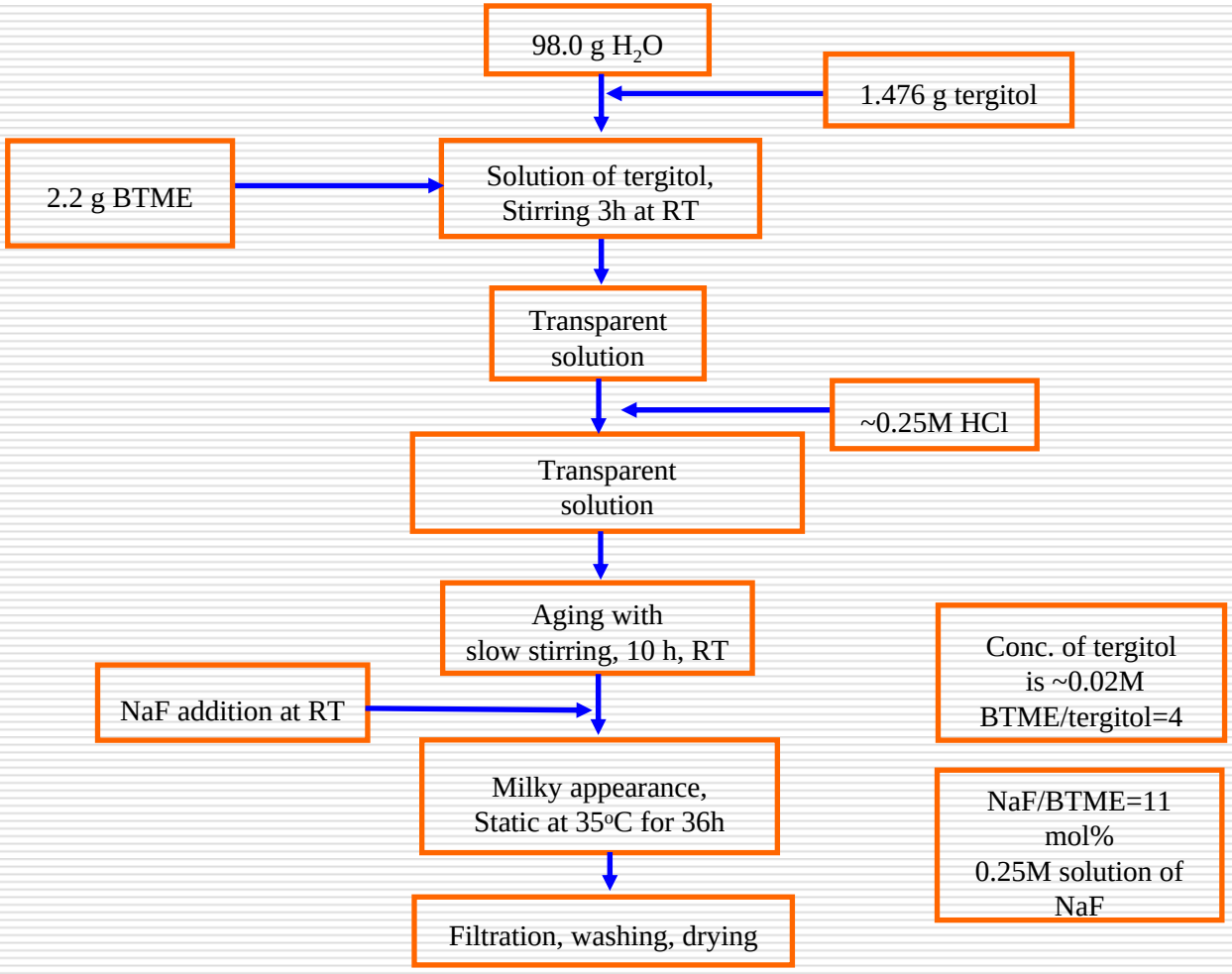
Synthesis of spherical hybrid mesoporous silica of MSU-1 type using BTME by the two-step process

BTME (1,2-Bis(trimethoxysilyl)ethane)

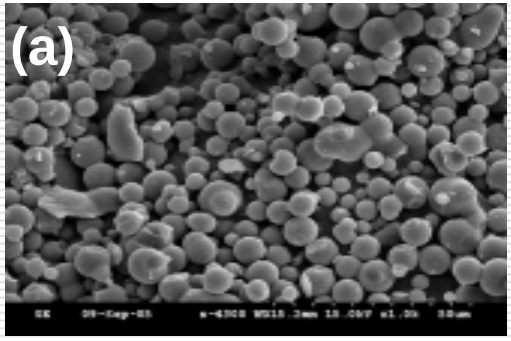


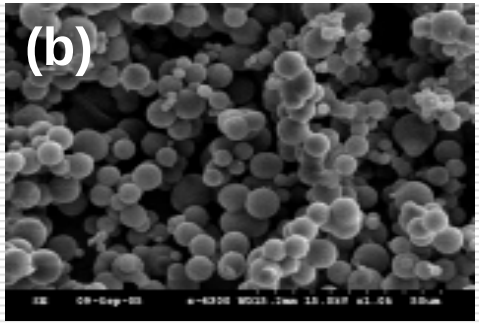
Synthesis steps

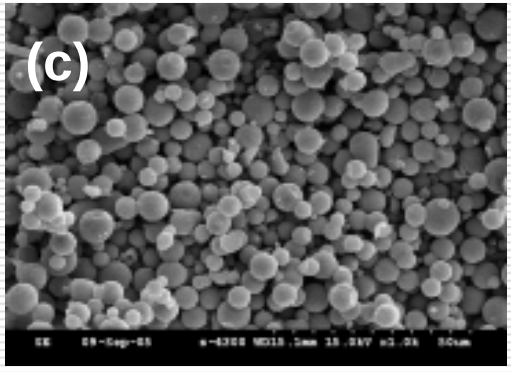


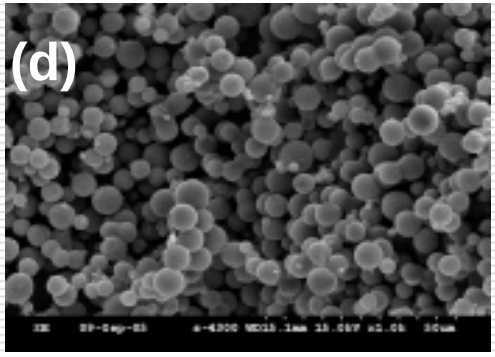
Effect of mol% of NaF (I)

Effect of NaF mole percent over BTME
(a) 8 mol%, (b) 9 mol%, (c) 10 mol% and (d) 11 mol%





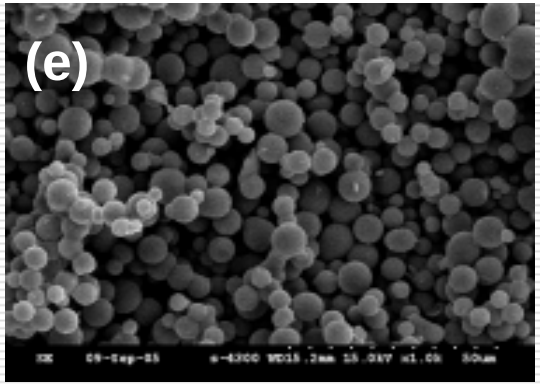


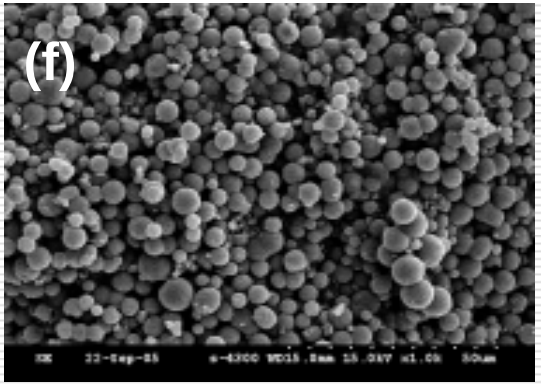


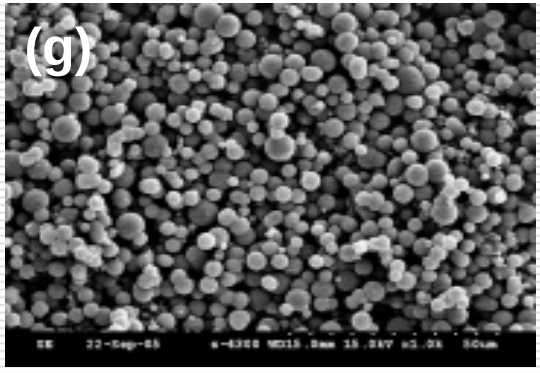
Comment
1)Similar morphology for 8-15 mol% of NaF
2)Compare with TEOS (2mol%) as a silica source, substantially larger amount of F^- is required for BTME.

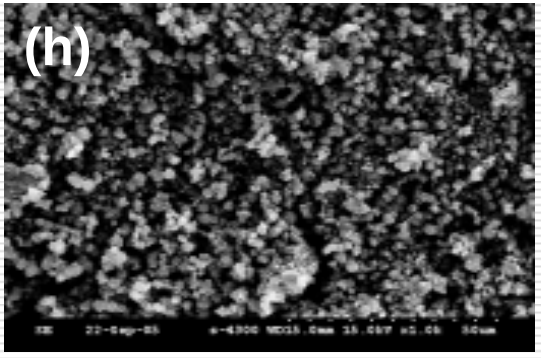
Effect of mol% of NaF (II)

SEM images of the samples prepared with varying mol% of NaF
(e) 12 %, (f) 14 %, (g) 15 % and (h) 20 %





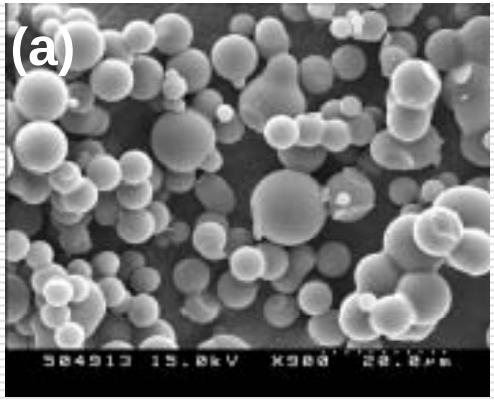


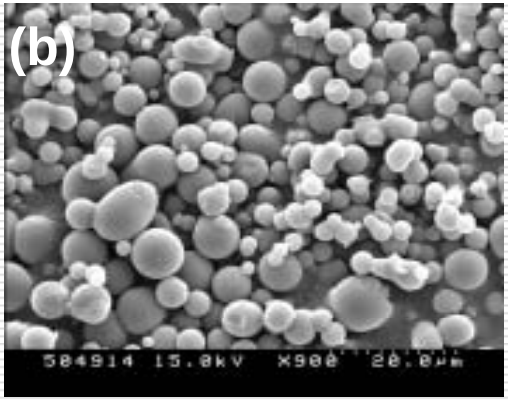


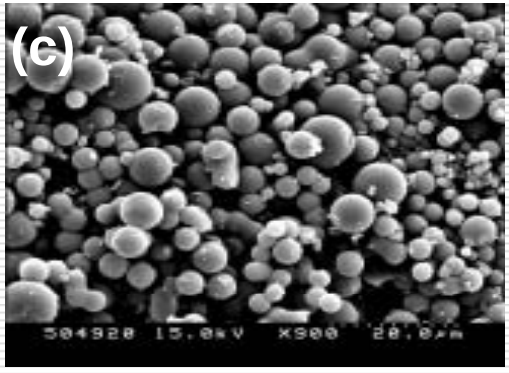
For 20 mol% of NaF
No good morphology

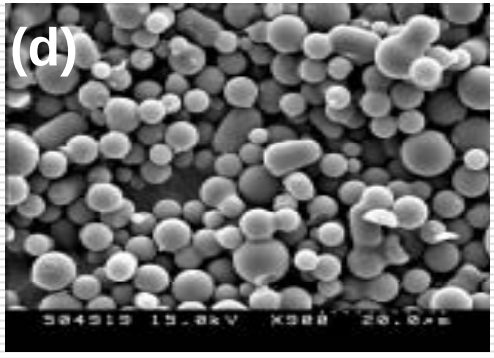
Effect of temperature

Hydrolysis/Condensation temperature effect
(a) RT/35℃, (b) 35/35℃, (c) 35/45 ℃, and (d) 35/55 ℃





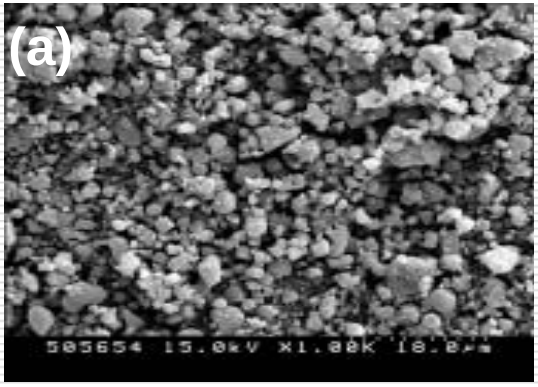


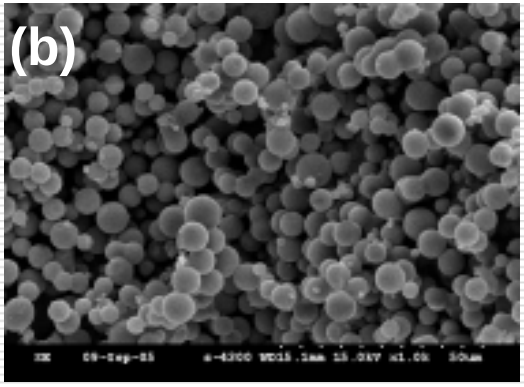


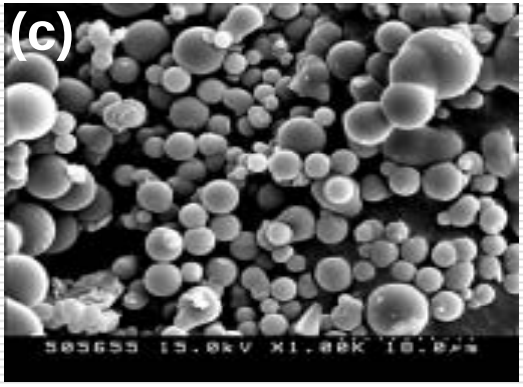
Hydrolysis at RT and condensation at 35 ℃ is better for the desired spherical morphology with size distribution in the range of 2 to 8 μm.

Effect of BTME concentration

Effect of different BTME over surfactant mol ratios
(a) 2, (b) 4, and (c) 6



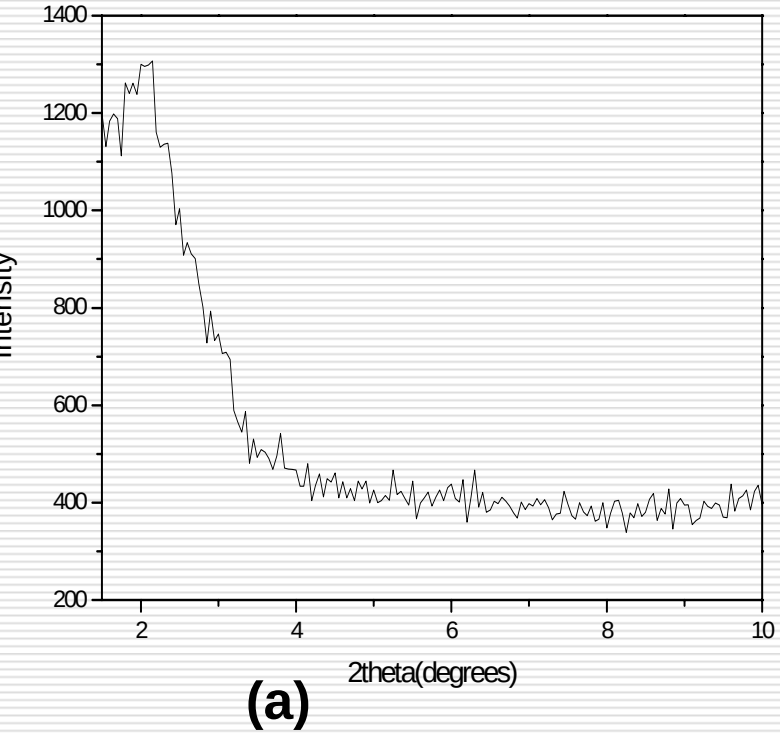




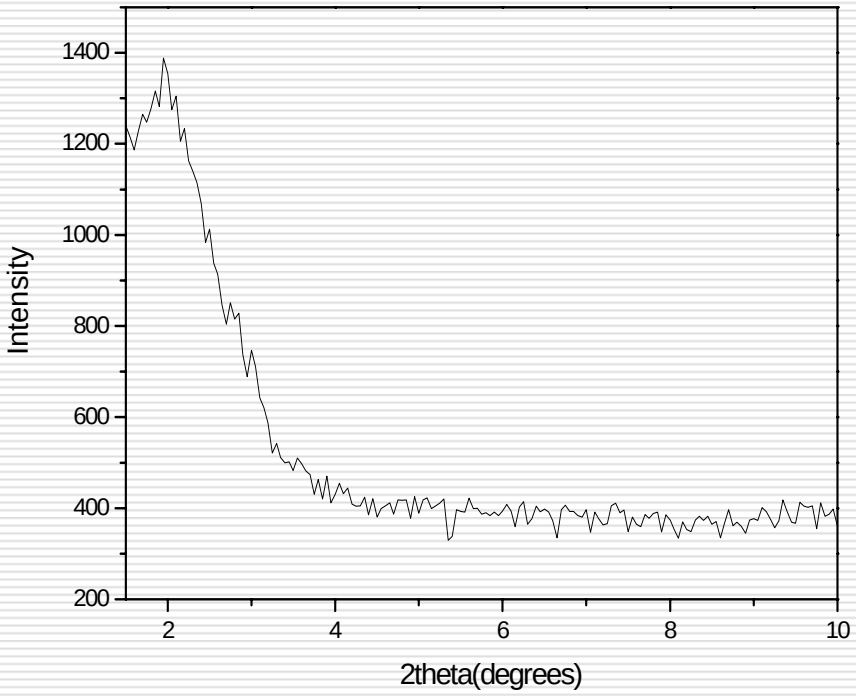
To obtain desired morphology the optimum mol ratio of BTME: surfactant = 4, whereas in the case of TEOS the optimum mol ratio of TEOS: surfactant = 8.

XRD patterns of the as-made sample

Small angle XRD peaks of (a)10 mol% of NaF, (b) 15 mol % of NaF



(a)



(b)

Similar XRD patterns are obtained for all other samples

Conclusion

To obtain a narrow particle size distribution in the range of 2-8 μm, optimum temperature of hydrolysis and condensation steps are RT and 35 ℃, respectively. Spherical morphology is obtained for 8-15 mol% of NaF. The optimum mol ratio of BTME: surfactant was 4.