Supporting Information

Scaling-up Continuous production of Mesoporous Silica particles at Kg Scale: Design & Operational strategies

Rajashri B. Jundale [a, b, ‡], Jayesh R. Sonawane [a, b, ‡], Anil V. Palghadhmal [a], Hemant Kumar Jaiswal[a], Hital S. Deore[a] and Amol A. Kulkarni[, a, b]*

[a]Chemical Engineering & Process Development Division, CSIR-National Chemical Laboratory, Pune 411008, India

[b]Academy of Scientific and Innovative Research (AcSIR), Ghaziabad, 201002, India

E-mail: aa.kulkarni@ncl.res.in

List of Figures:

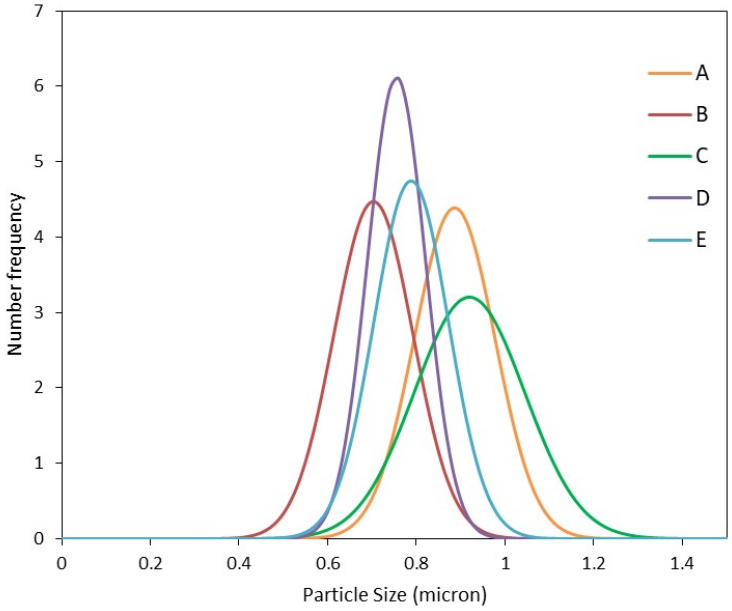

Figure S1: Particle size distribution of MSPs obtained at pilot scale operation, A) $T_{N=}$ 35 °C, T_G=55 °C, 0.0045 M NaOH, no intermediate pulsation, B) $T_{N=}$ 55 °C, T_G=55 °C, 0.0045 M NaOH, 5 s pulse at 88 ml/min flow rate, C) $T_{N=}$ 45 °C, T_G=45 °C, 0.0045 M NaOH, 5 s pulse at 88 ml/min flow rate, D) $T_{N=}$ 35 °C, T_G=65 °C, 0.003 M NaOH, 10 s pulse at 88 ml/min flow rate, E) $T_{N=}$ 35 °C, T_G=65 °C, 0.003 M NaOH, 15 s pulse at 88 ml/min flow rate.

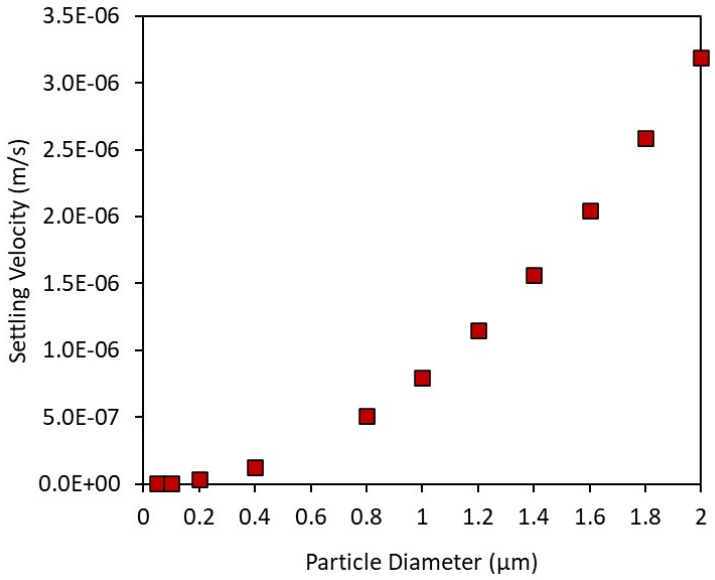

Figure S2: Settling velocity vs particle size of silica particles.

The following co-relation was used for calculating settling velocity of particles

$$v = gd^2(\rho_p - \rho_m)/18\mu$$

Where,

ρ_p = Particle density, kg/m^3

ρ_m = Density of reaction mixture, kg/m^3

μ = Viscosity of reaction mixture

d = Particle diameter, m

g= Acceleration of gravity, m/s^2

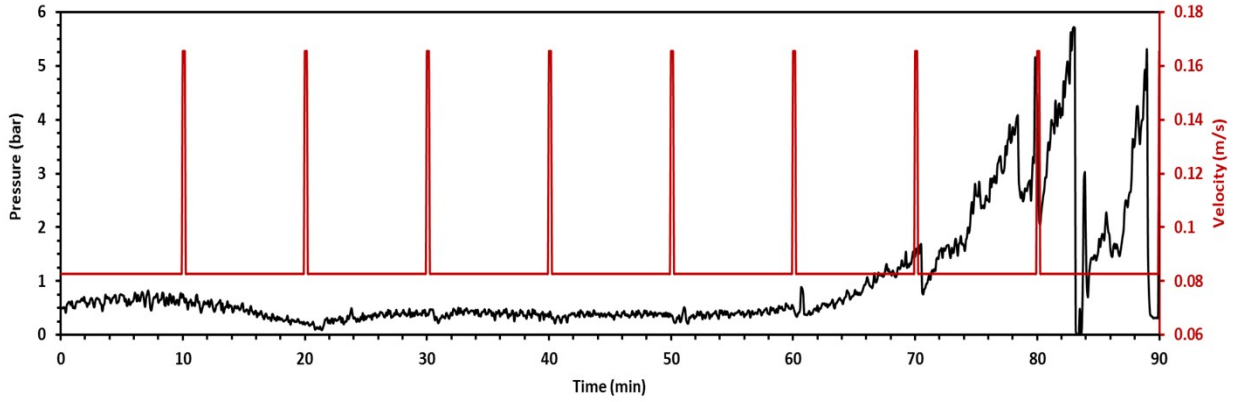

Figure S3. Pressure and velocity profile for the pulsation experiment under given conditions, 20 min residence time, 0.003 M NaOH concentration, pulsation for 10 s after every 10 min, T_N= 35 ˚C and T_G= 65 °C.

The plot of velocity vs. time shows how the velocity changes with an instant change in flow rate during the pulsation for a short time. Thus, the residence time also changes for that short time.

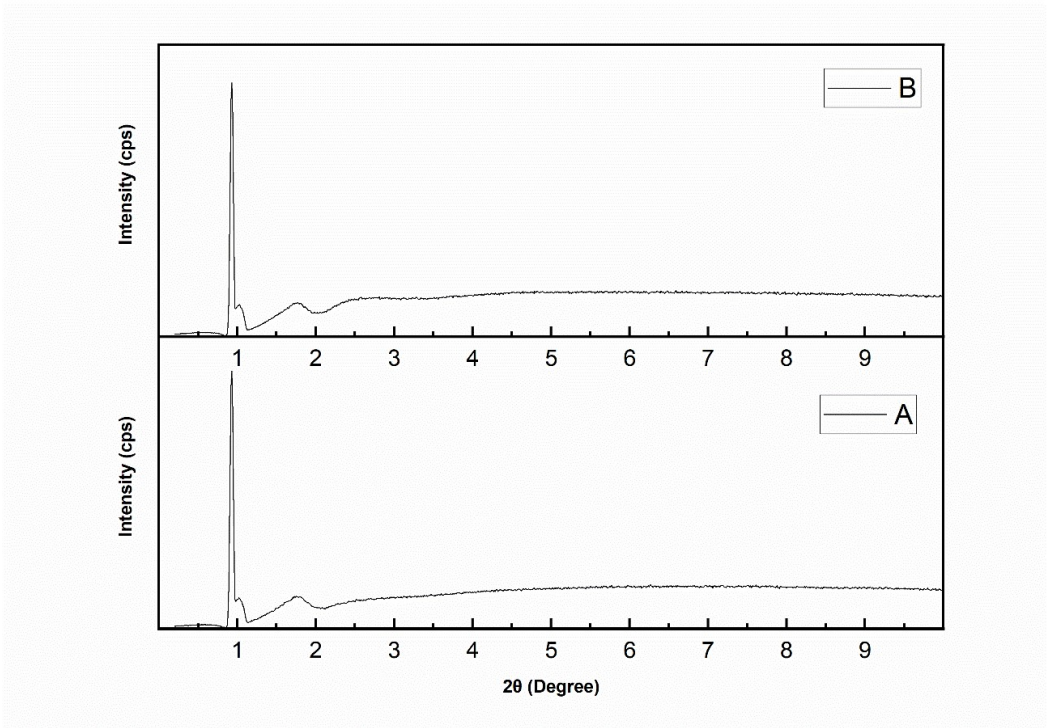

Figure S4. XRD spectra of mesoporous silica particles were synthesized using, A) slug flow 25%, 0.003 M NaOH concentration, $T_{N,G}$ = 55 °C, and B) slug flow 25%, 0.0045 M NaOH concentration, T_N = 55 and T_G= 35 °C.

Mesostructural ordering was also confirmed through wide angle X-ray Diffraction pattern. Particles exhibited one sharp diffraction peak at 2θ = 0.92 due to the (210) plane, and relatively broad peaks appeared at 2θ = 1.02 and 1.7 which indexed to (211) and (400) reflection respectively with cell parameter 210.54 Å, which are characteristic of cubic mesophase.

Variation in the viscosity in the reaction mass:

The flow rates and their ratios were fixed in all the experiments. The resultant change in the volume fraction of silica particles depended entirely on the concentration of the base and reaction temperature (hence, particle yield). In addition to this, the volume fraction changes with particle diameter and the mass of particles along the length of the reactor. While it was not possible to remove intermediate samples (along the length or at different residence time inside the reactor) from the pilot-scale reactor (as presence of any fitting or sampling valve in the reactor flow path would lead to particle deposition), time-dependent data is not reported. However, based on the lab scale data (for ¼" tubing reactors), the approximate change in volume fraction of particles with respect to time along the length of the reactor was estimated for the pilot scale reactor. It is obvious that the volume fraction increases with an increase in particle diameter and yield.

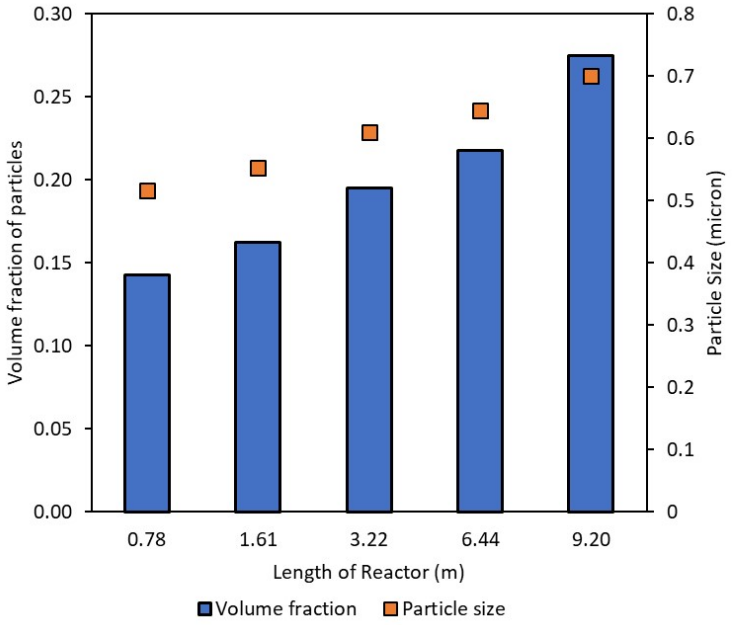

Figure: Change in volume fraction of particles with respect to reactor length and particle size

Particles in a liquid act as obstacles, hindering the liquid's flow and therefore it is obvious increasing the viscosity of suspension. Particle fraction (mass fraction) and particle size are also influence the change in viscosity of suspension. Here the figure shows the change in viscosity of suspension along the length of reactor.

Viscosity calculated by considering the viscosity of the reaction mixture (Methanol: water 1:1) and the mass fraction of the dispersed particles using following formula.

Where, ϕ mass fraction of particles and η0 viscosity of reaction mixture. $\eta=\eta0(1+2.5\phi+6.2\phi2)$

Density of suspension also calculated by using volume fraction of particles

$$\rho_{suspension} = \rho_{np} \times \emptyset_{np} + (1 - \emptyset_{np})\rho_{medium}$$

Where $\emptyset_{np}$ Volume fraction of particles, ρ_{np} Density of particles

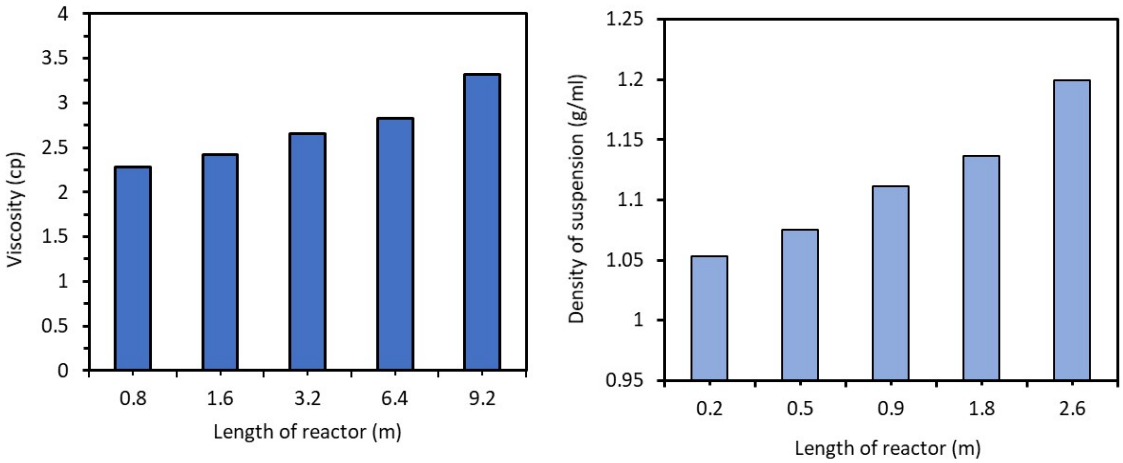

Figure: Change in viscosity and density of suspension along the length of reactor.

From the graph it can see that both the viscosity and density of suspension increases along the length of the reactor. Although the increase in viscosity is just 30%, the % length over which it changes is significant and hence for laminar flow, the overall impact will be large.