Supporting Information

Kinetics of fluoride-catalysed synthesis of organosilica colloids in aqueous solutions of amphiphiles

Teh-Min Hu,¹,²* Chien-Yu Lin,¹ Meng-Ju Wu¹

¹. Faculty of Pharmacy, School of Pharmaceutical Sciences, National Yang-Ming University, Taipei 112, Taiwan, ROC.
². Center for Advanced Pharmaceutics and Drug Delivery Research, National Yang-Ming University, Taipei 112, Taiwan, ROC.

*Corresponding author: Teh-Min Hu, Ph.D. E-mail: tehmin@ym.edu.tw

1. Supporting video clips

Video S1. Time-lapse video captures the changes of reactions over 90 min.
Reaction solution: 2 mL aqueous solution containing 100 mM MPTMS, 5% Tween 20, 0–160 mM NaF.
Room temperature.
Please find the video file online using the following DOI: 10.1039/C9RA05509F.
2. Supporting Figures & Images

Figure S1. FE-SEM image of colloidal particles collected from the reaction solution (24 h, room temperature) containing 100 mM MPTMS, 0.625% Tween 20, and 160 mM NaF.
Figure S2. Fluoride-concentration-dependent particle size evolution over time. (A) Hydrodynamic size. (B) Polydispersity index. Aqueous reaction solution containing 100 mM MPTMS, 5% Tween 20, and NaF at 40, 80, or 160 mM; room temperature.
Figure S3. Difference ATR-FTIR absorbance spectra showing the time evolution of O-H bending vibrations of water. (A) The difference spectra (relative to the absorbance at 1 min) at various sampling times. (B) Quantitative absorbance changes at 1640 cm$^{-1}$ over the reaction time course. The spectra and data were extended from Figure 6.
Figure S4. Kinetic ATR-FTIR spectra for the reaction system of Tween 40 (A), Tween 60 (B), and Tween 80 (C). Similar to Figure 5 in the main text, each curve represents the differential absorption spectrum (i.e. absorption at time t – absorption at 1 min) at a specific time (from 2 to 15 min). Mean of three independent experiments. Generally, each trace is moving upward over time at major peaks (e.g. 1260, 1120, 1040, and 900 cm$^{-1}$, as indicated in Figure 5A in the main text)
Figure S5. Hydrodynamic sizes of colloidal particles generated in the reaction system containing amphiphilic polymers (PVA: polyvinyl alcohol; PVP: polyvinylpyrrolidone). Reaction solution: 100 mM MPTMS, 160 mM NaF, various concentrations of polymers (as indicated), room temperature, measured at 24 h.
Figure S6. Hydrodynamic sizes of colloidal particles generated in the reaction system containing SDS or CTAB. Reaction solution: 100 mM MPTMS, 160 mM NaF, various concentrations of surfactants (as indicated), room temperature, measured at 24 h.
Figure S7. Kinetic ATR-FTIR absorption spectra for the reaction system containing CTAB (0.625%). Reaction solution: 100 mM MPTMS, 160 mM NaF, 0.625% CTAB, water. ATR-FTIR spectra measured every minute up to 15 min at room temperature. Note that unlike the system with other amphiphiles, the CTAB system shows small or insignificant spectra changes over time at the two major wavenumber ranges. When the concentration of CTAB was further reduced, more significant spectral evolutions can be observed (see text).
Figure S8. Effect of adding HCl to the reaction system.

Reaction solution: 0.2 mL aqueous solution containing 100 mM MPTMS, 160 mM NaF, and 5% Tween 20, room temperature.

Turbidity (absorbance at 800 nm) was continuously measured. At different time points (A, 0 min; B, 3 min; C, 10 min; D, 20 min, as indicated by the red arrow), 5 μL of HCl solution (0.4, 1, and 5 M) or blank water was added to the solution. The final HCl concentrations in the reaction solution are 0, 9.8 mM, 24 mM, and 122 mM.

As can be seen, the reaction system is very sensitive to the addition of HCl at initial time points (e.g. 0–3 min), i.e. acidification inhibits colloidal formation; however, once the colloidal solution is formed, the system is less perturbed upon adding HCl.