Sustainable Nanotechnology Organization

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UtahStateUniversity

Self-assembly of tri-functional and di-functional alkane silanes into hydrophobic silica nanoparticles in aqueous media

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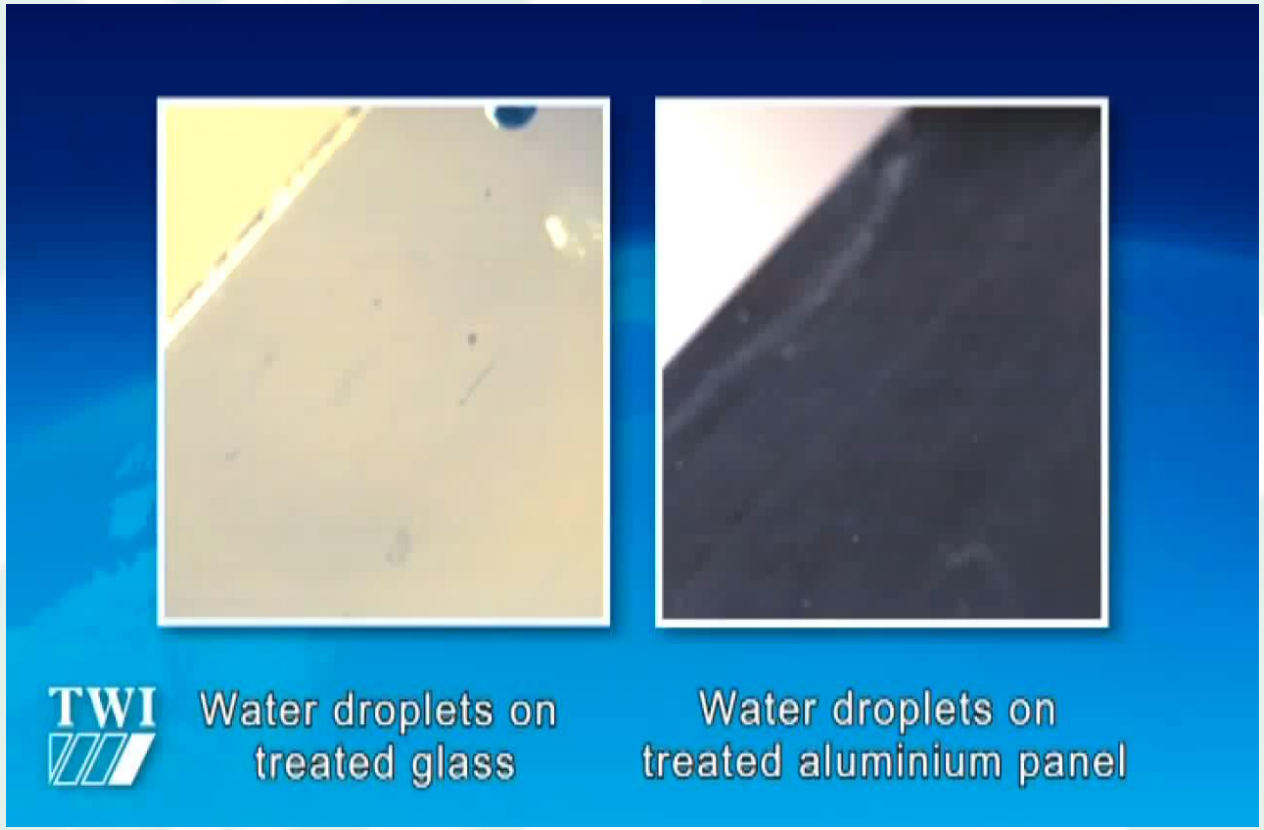
Outline

- Background
- Stöber method silica nanoparticles (NPs)
  - Synthesis
  - Hydrophobic coating
- Concept of 1-step, aqueous synthesis of silica NPs from tri-functional silane
- Hydrophobic silica NPs from tri- and di- functional alkane silanes
  - DLS (kinetics of NP growth, final sizes)
  - AFM (hydrophobic silica NP pathway elucidation)
- Conclusion

Background

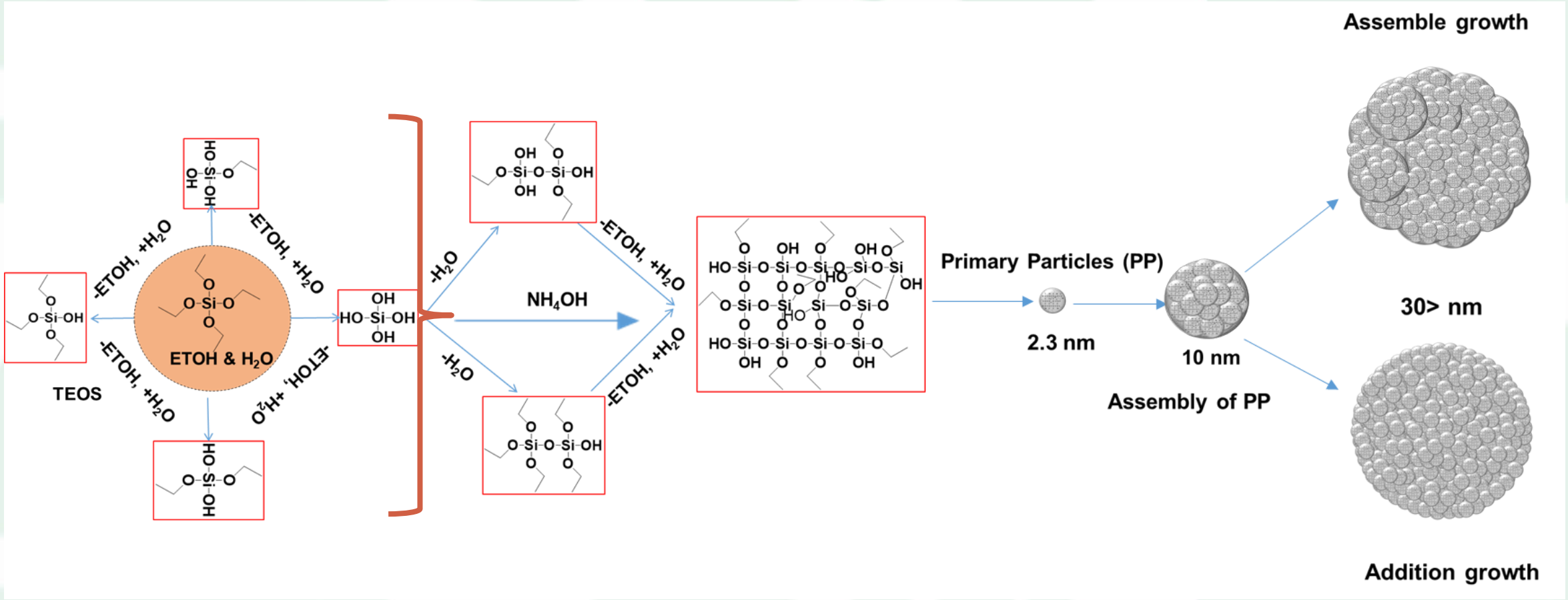
- **Hydrophobic silica nanoparticles (NPs) are widely used in self-cleaning materials, waterproof textiles, oil separation, and anticorrosive industrials parts**

- **Silica based NPs with hydrophobic functionality also have potential applications as carriers for hydrophobic drugs**



Vitolane® technology (hydrophobic silane coatings)
150° water contact angles

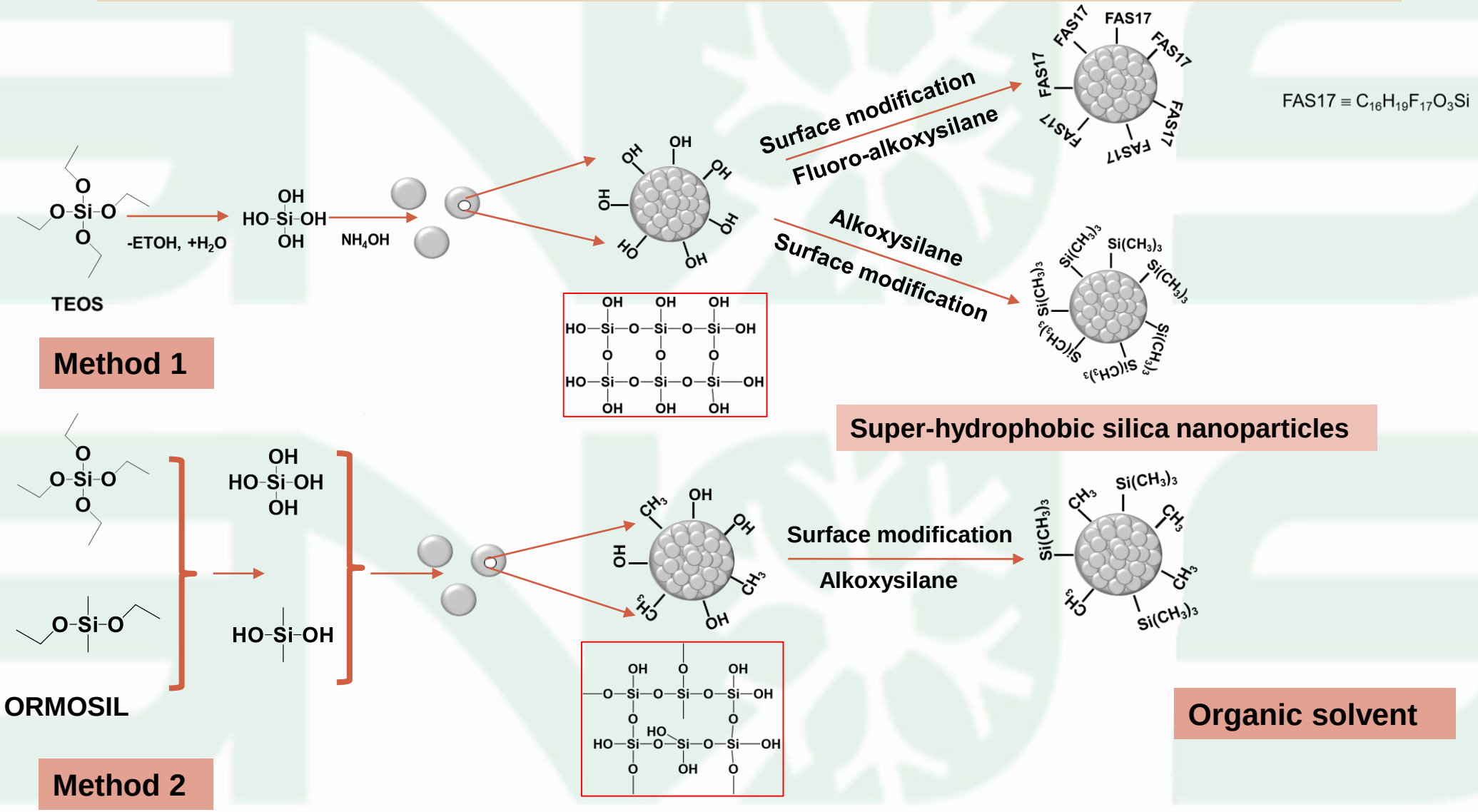
Synthesis of silica NPs: Stöber method



Stöber et al., *J. Colloid Interface Sci.*, 1968, **26**, 62–69

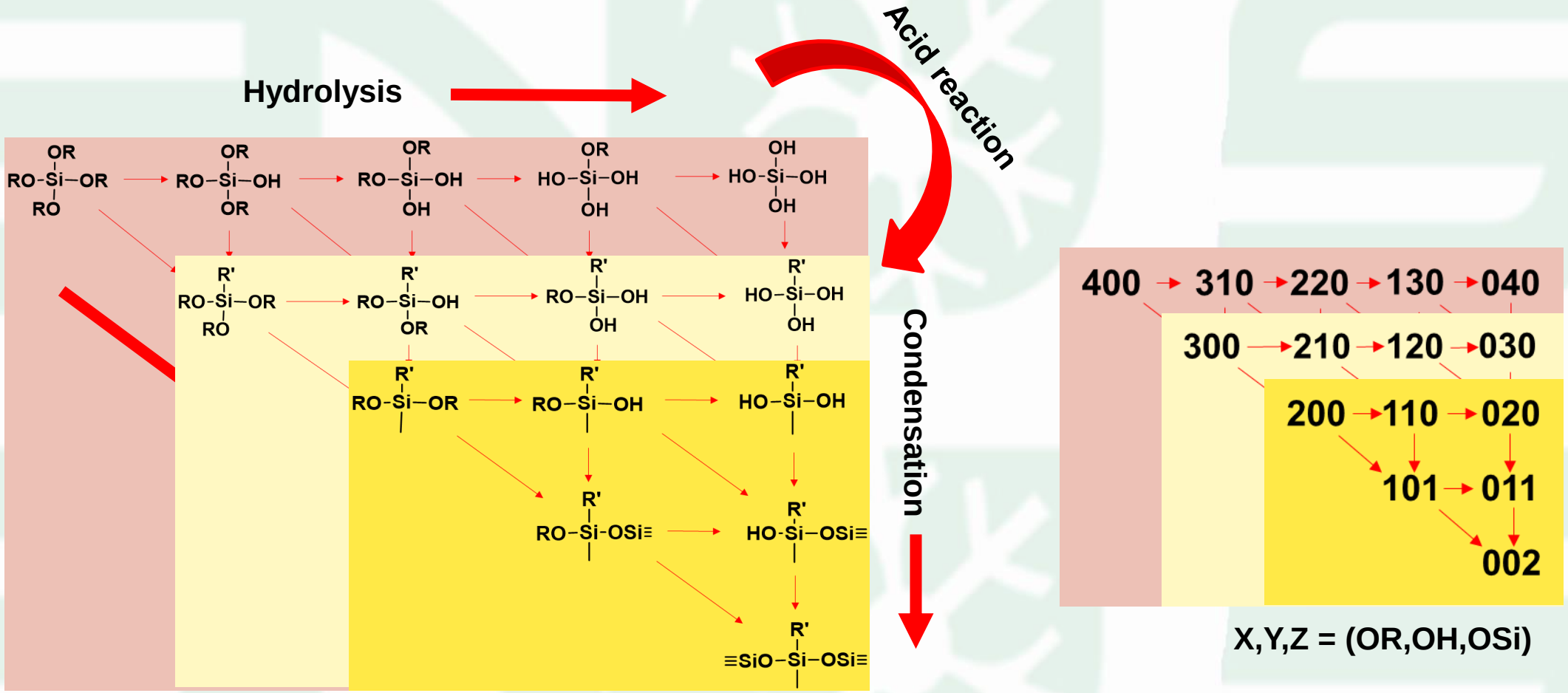
Carcouet et al., *Nano Lett.* **2014**, 14, 1433– 1438

Hydrophobic coating on silica NPs



J. *ACS Appl. Mater. Interfaces* **2011**, 3, 3583
RSC Adv. **2014**, 4, 9838-41

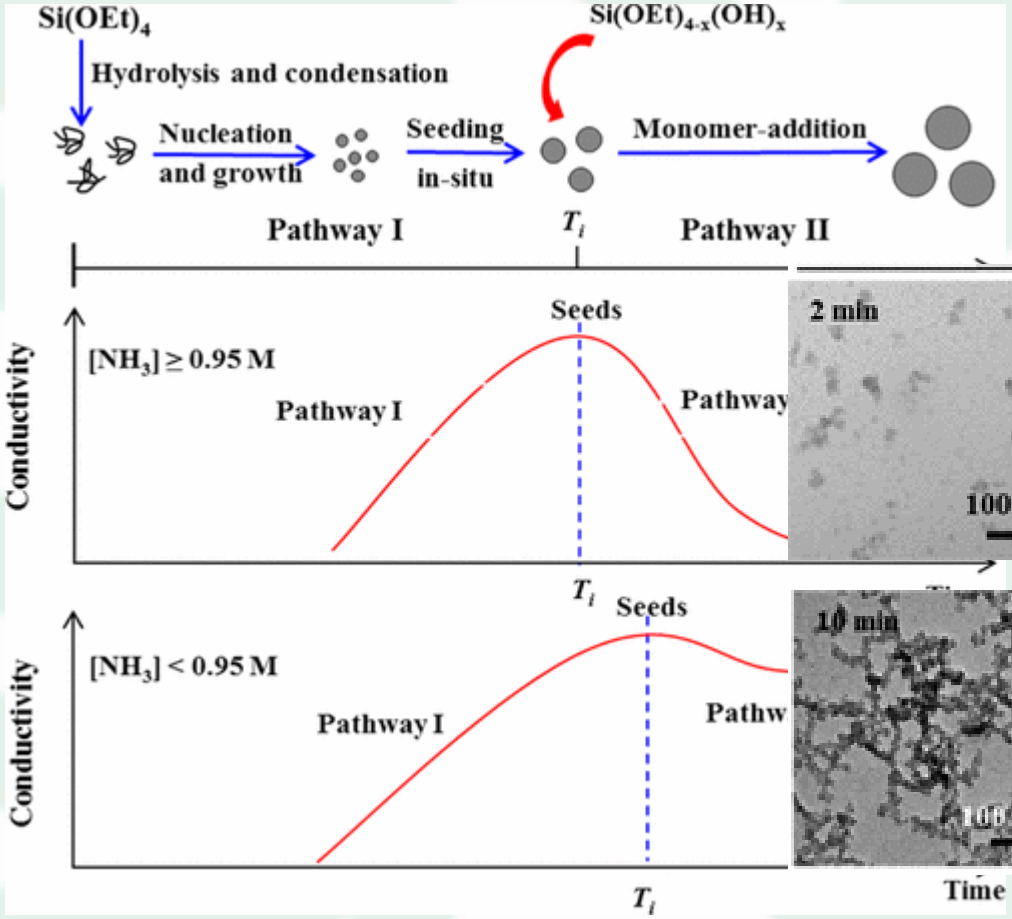
Hydrolysis and condensation pathways

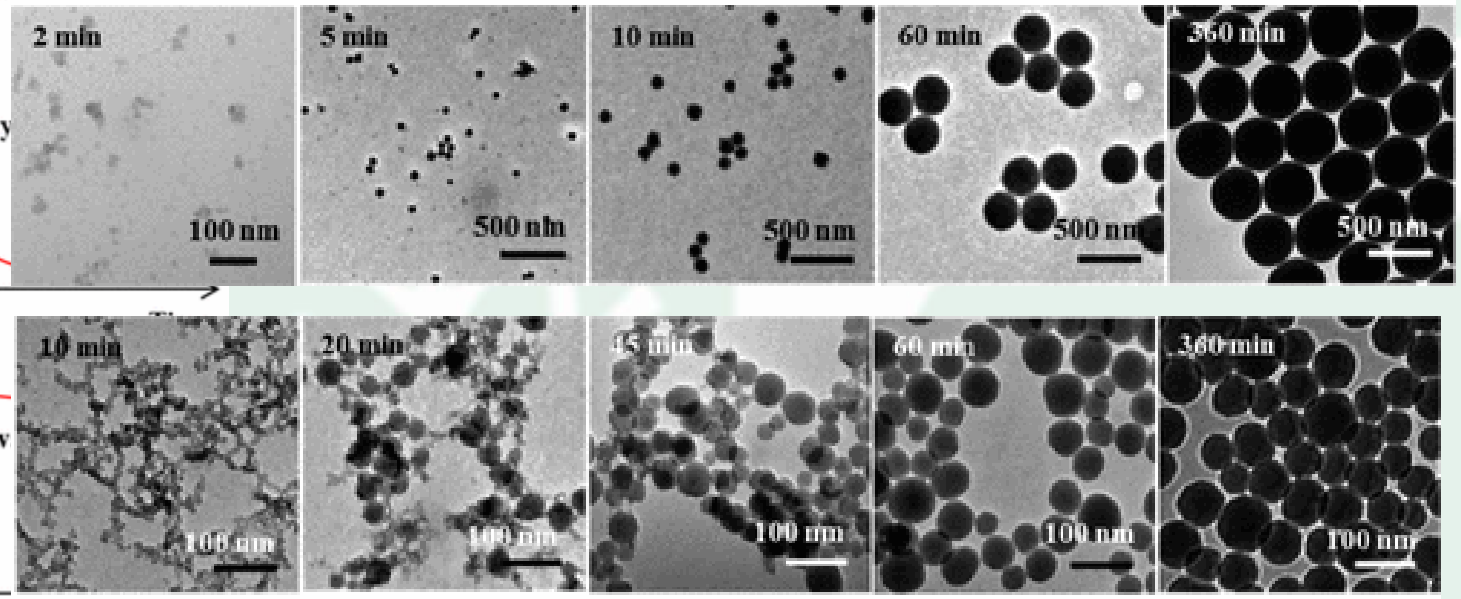


R/R' = CH_3/CH_2CH_3/$CH_2CH_2CH_3$…

B. Arkles et al., Silanes and Other Coupling Agents, 91-104, 1992

Controlling size of the silica NPs

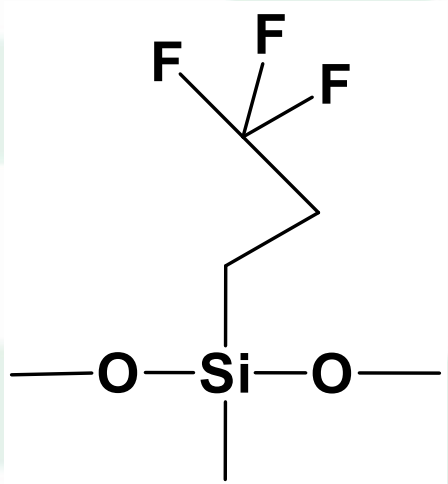




Size of NPs strongly dependent on catalyst (NH_4OH) concentrations, and solvent (Ethanol, Methanol)

Han et al., *Langmuir*, **2017**, *33* (23), pp 5879–5890

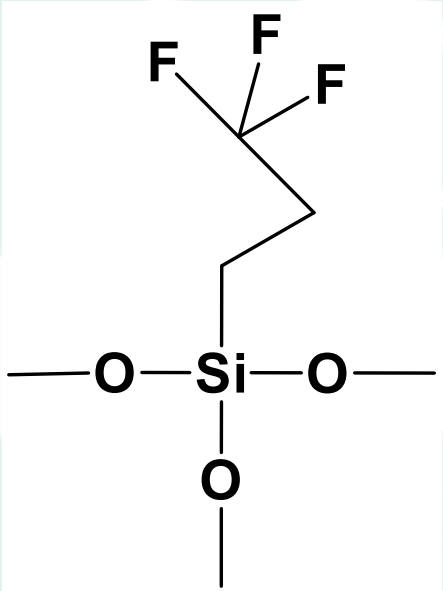
Silanes used in this study



D3F (Methyl(3,3,3-trifluoropropyl)dimethoxysilane)

ρ: 1.089 g/mL

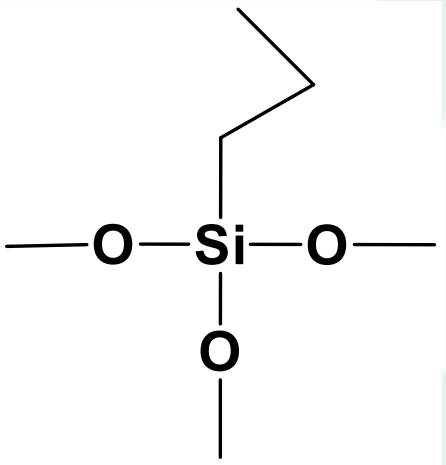
Purity: >95%



3F (3,3,3-trifluoropropylmethoxysilanol)

ρ: 1.142 g/mL

Purity: 98%

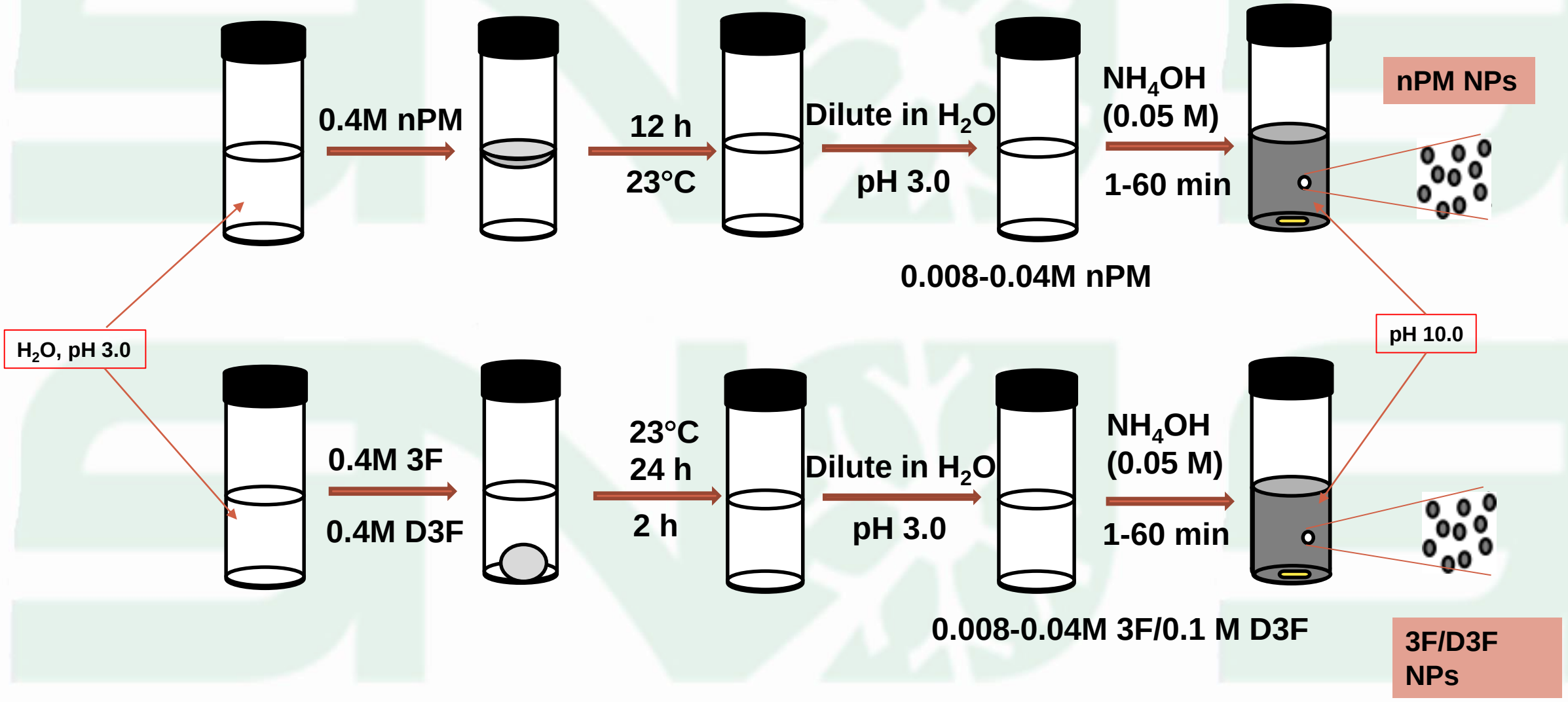


nPM (n-propyltrimethoxysilanol)

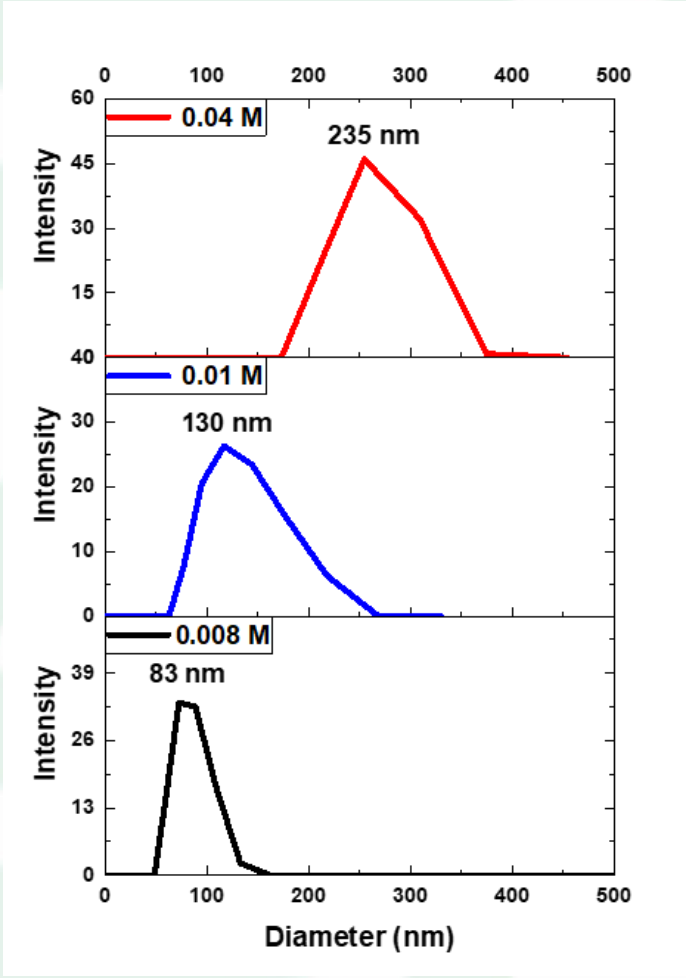
ρ: 0.932 g/mL

Purity: >95%

Experimental procedure



Controlled growth of 3F and nPM NPs: DLS



3F NPs

PDI: 17%

PDI: 27%

1 h

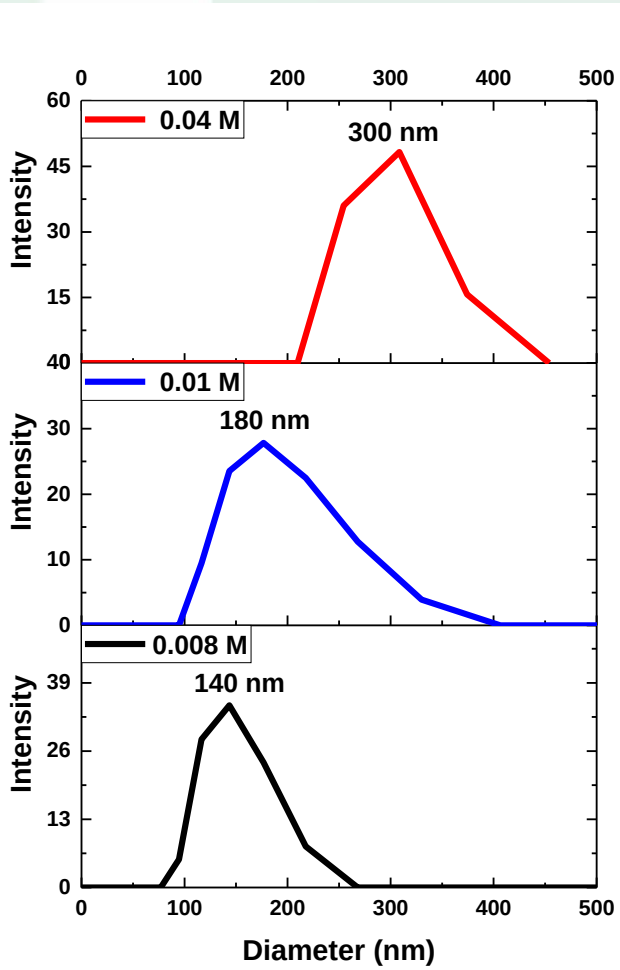
6 h

PDI: 13%

PDI: 35%

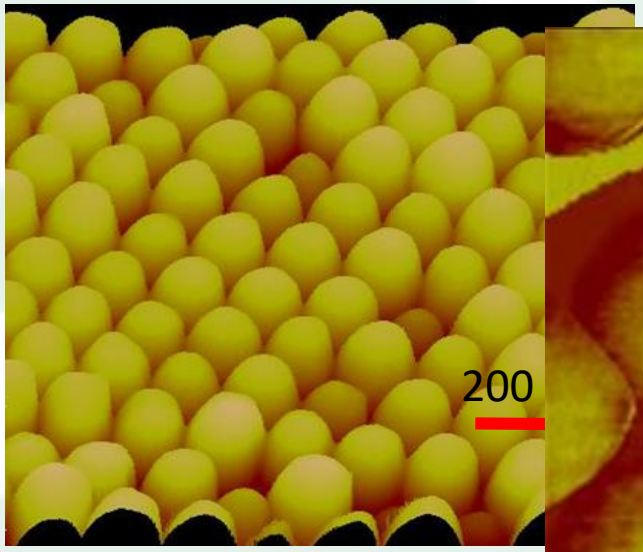
12 h

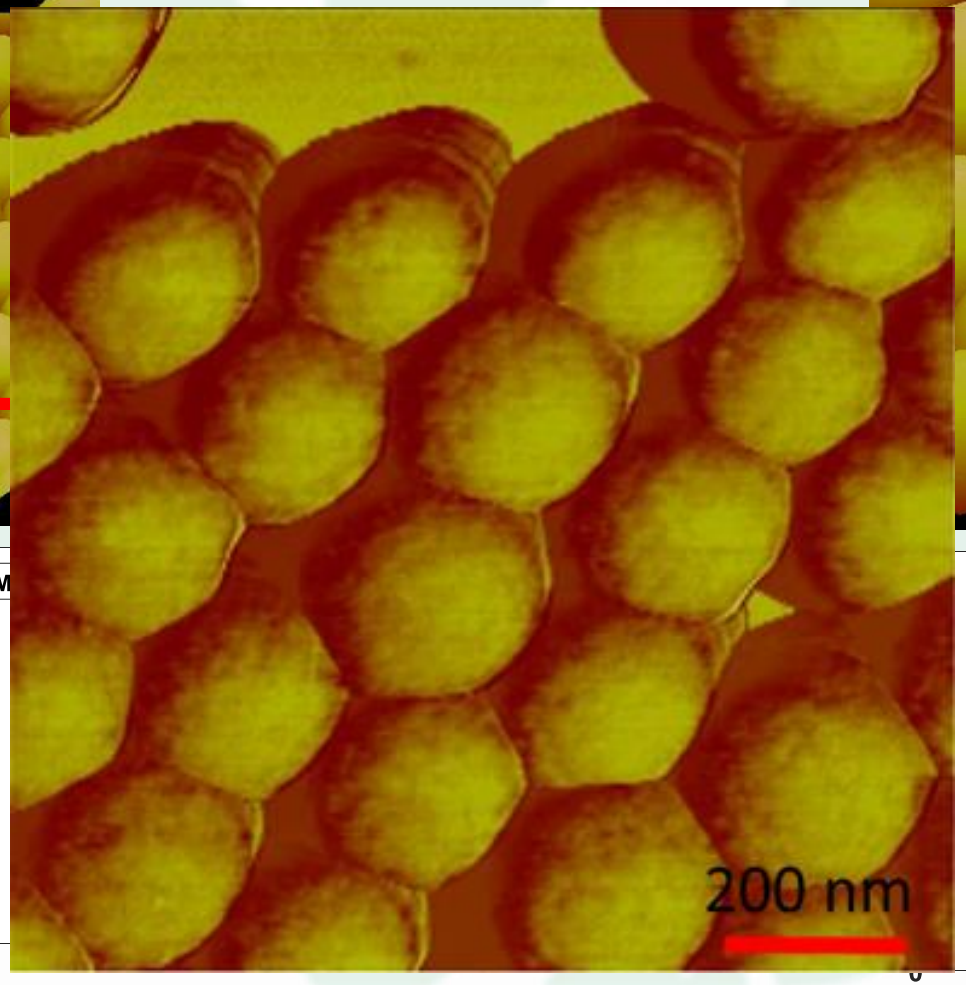
Sizes of the NPs were stable after 48 hours

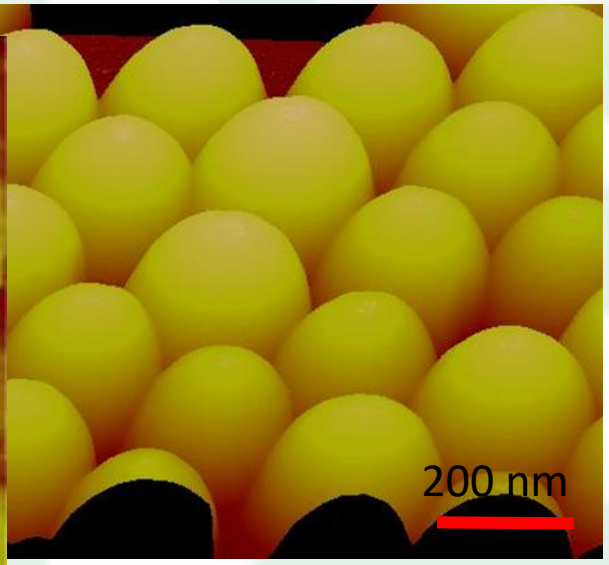


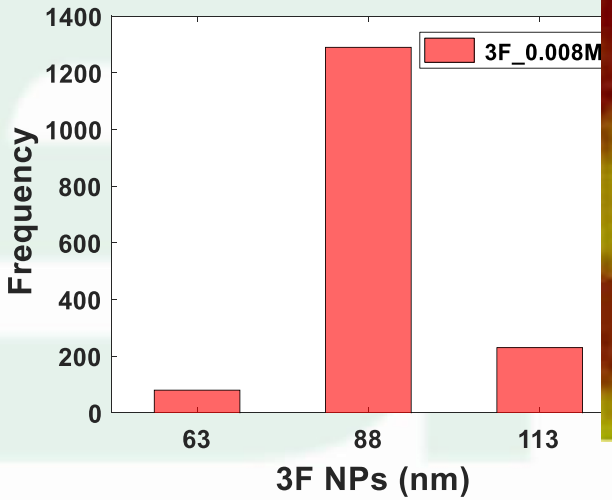
nPM NPs

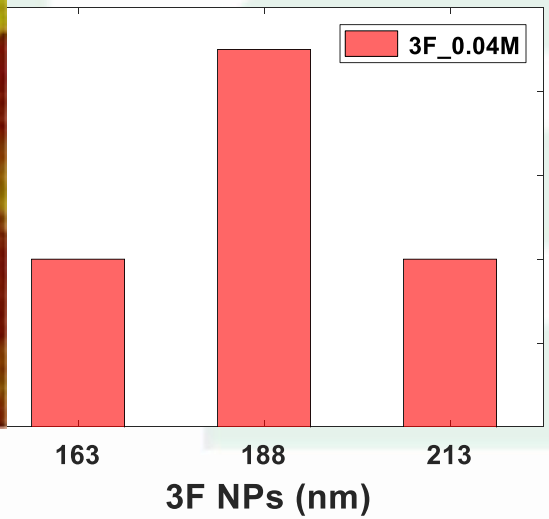
Controlled growth of 3F NPs: AFM



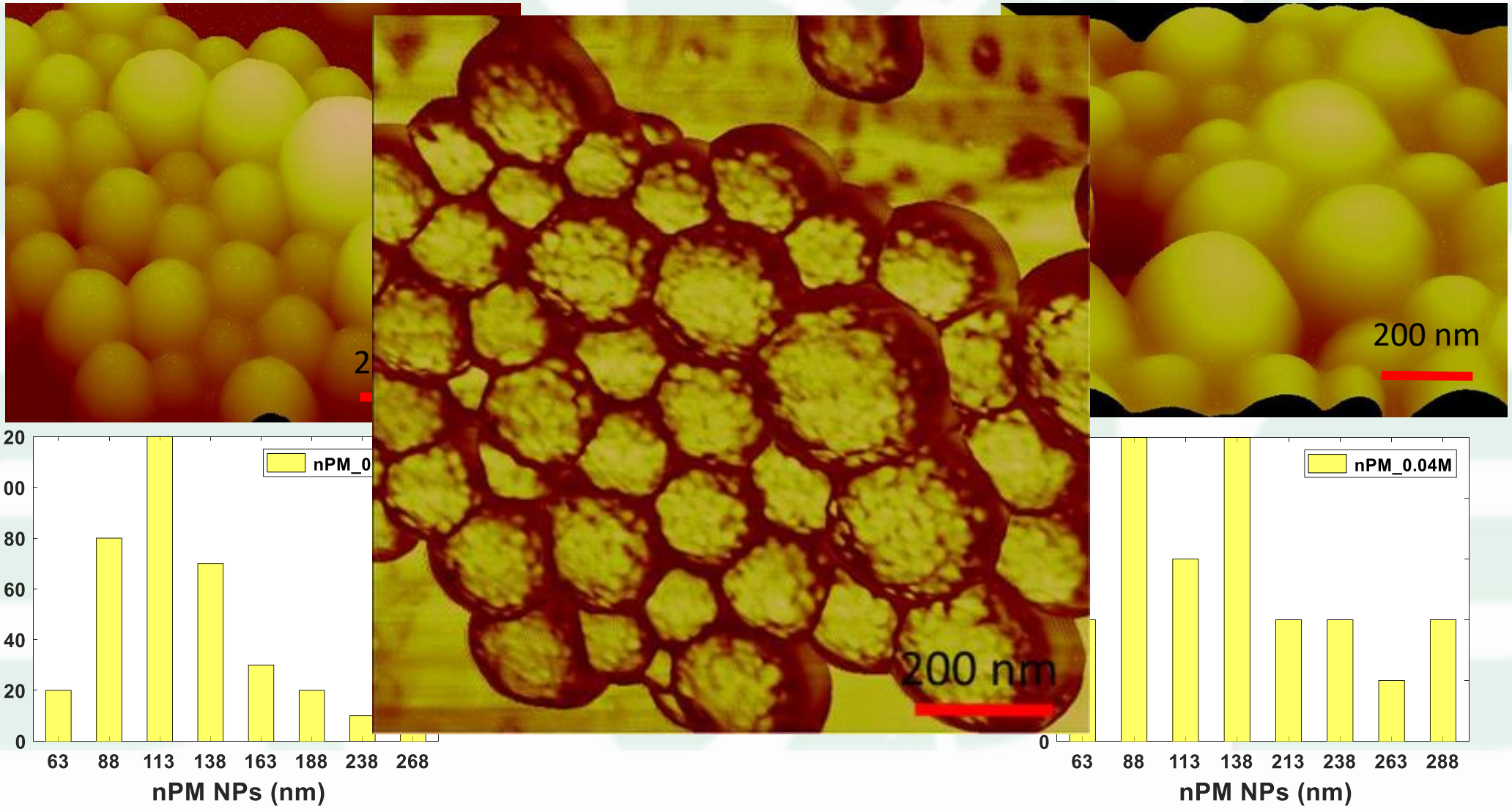




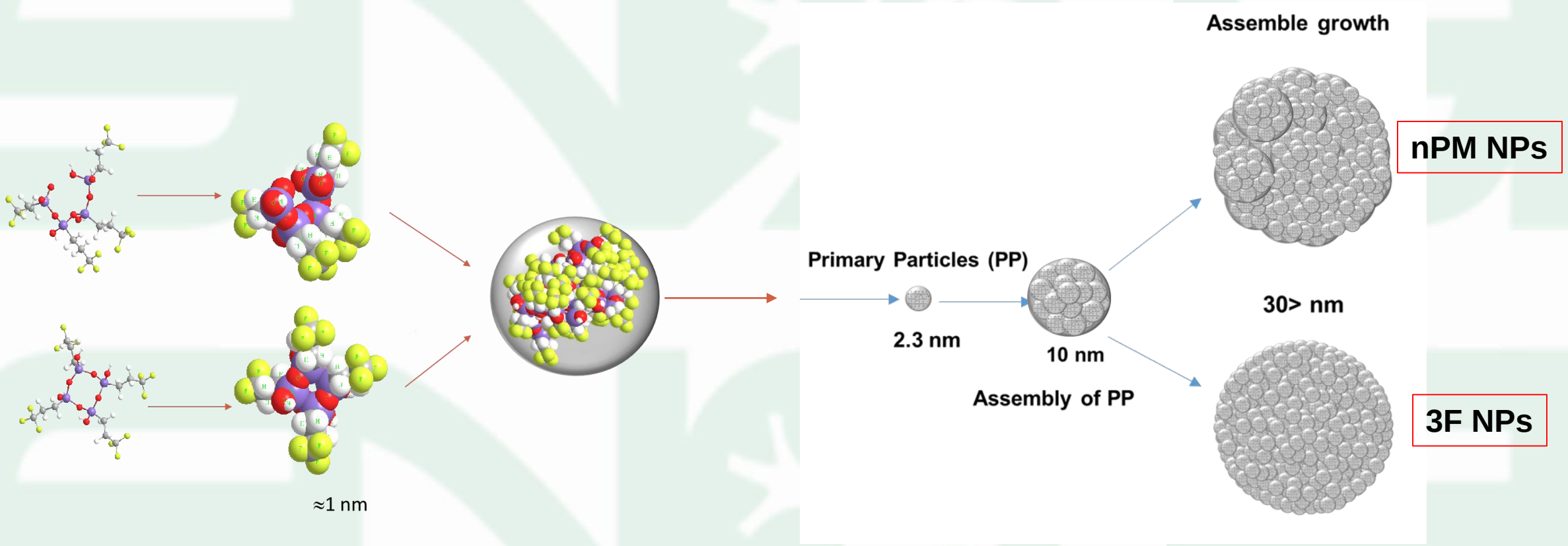




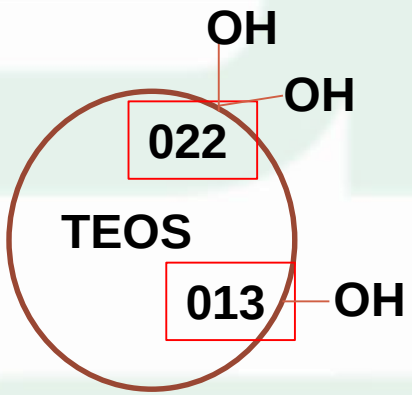
Controlled growth of nPM NPs: AFM

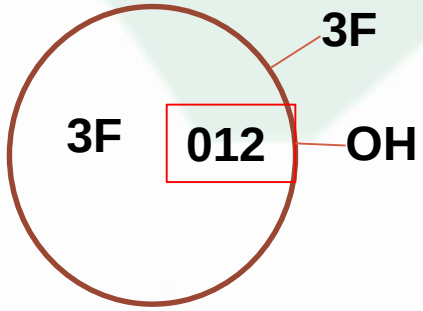


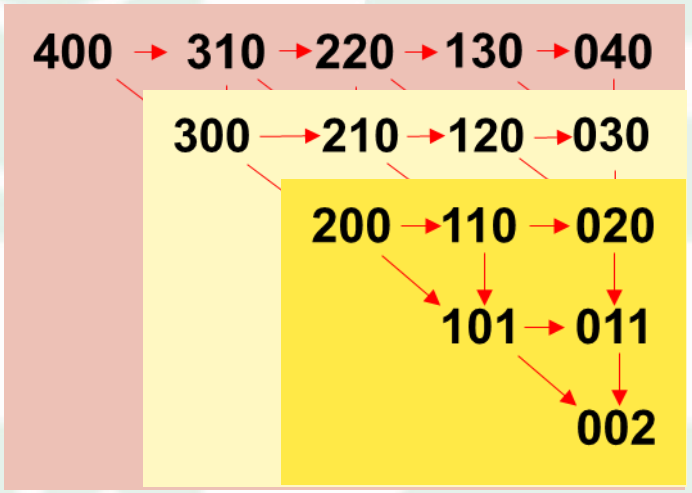
3F and nPM NPs growth model

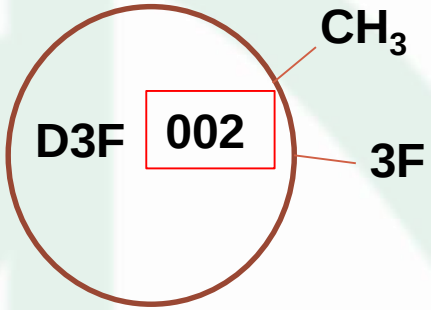


NPs from di-functional silanes



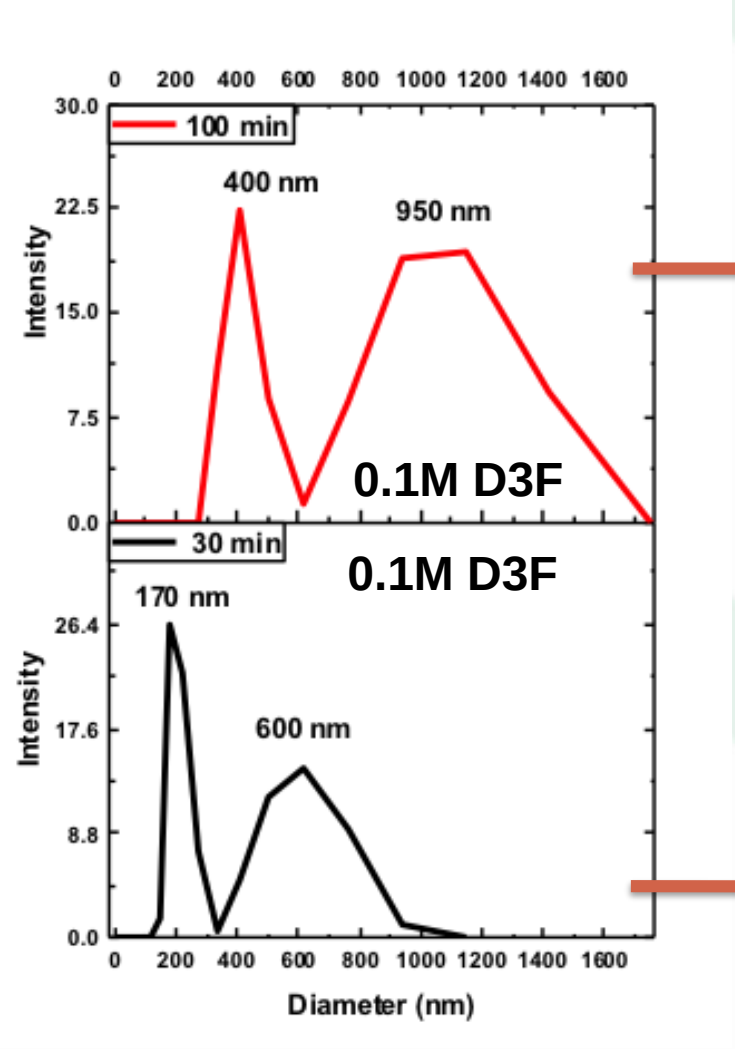


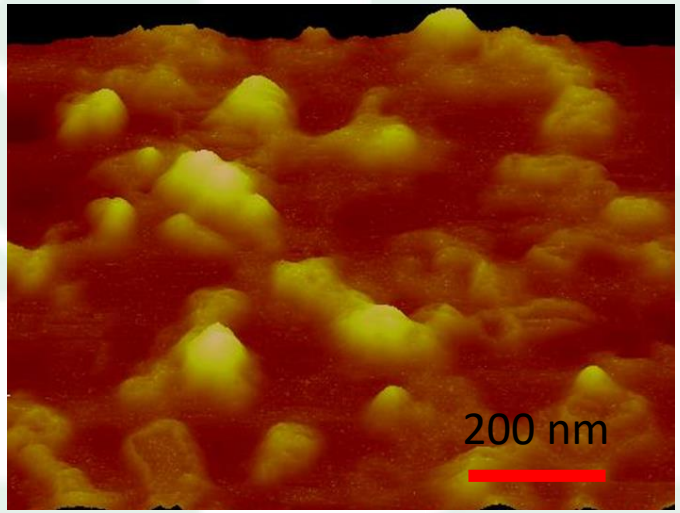




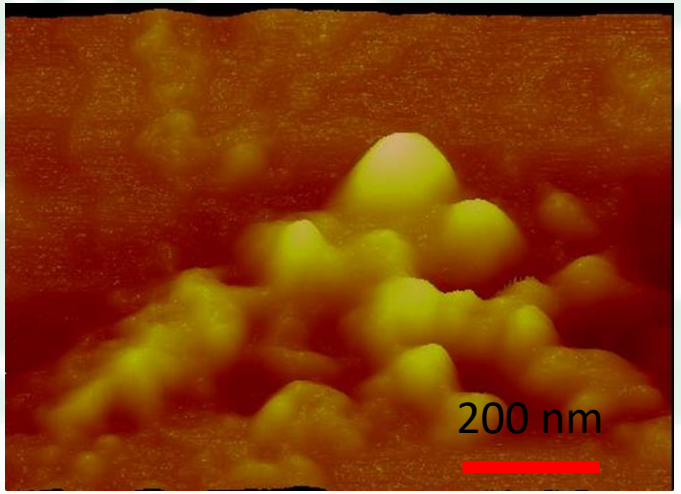
Di-functional analog of 3F

D3F NPs: DLS and AFM analysis

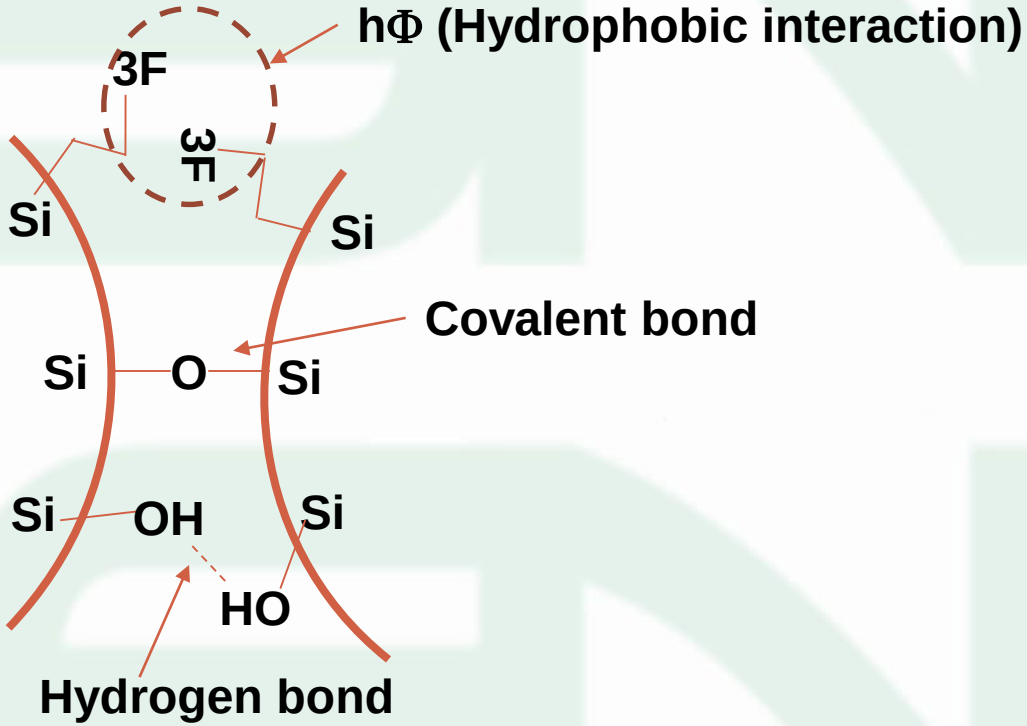


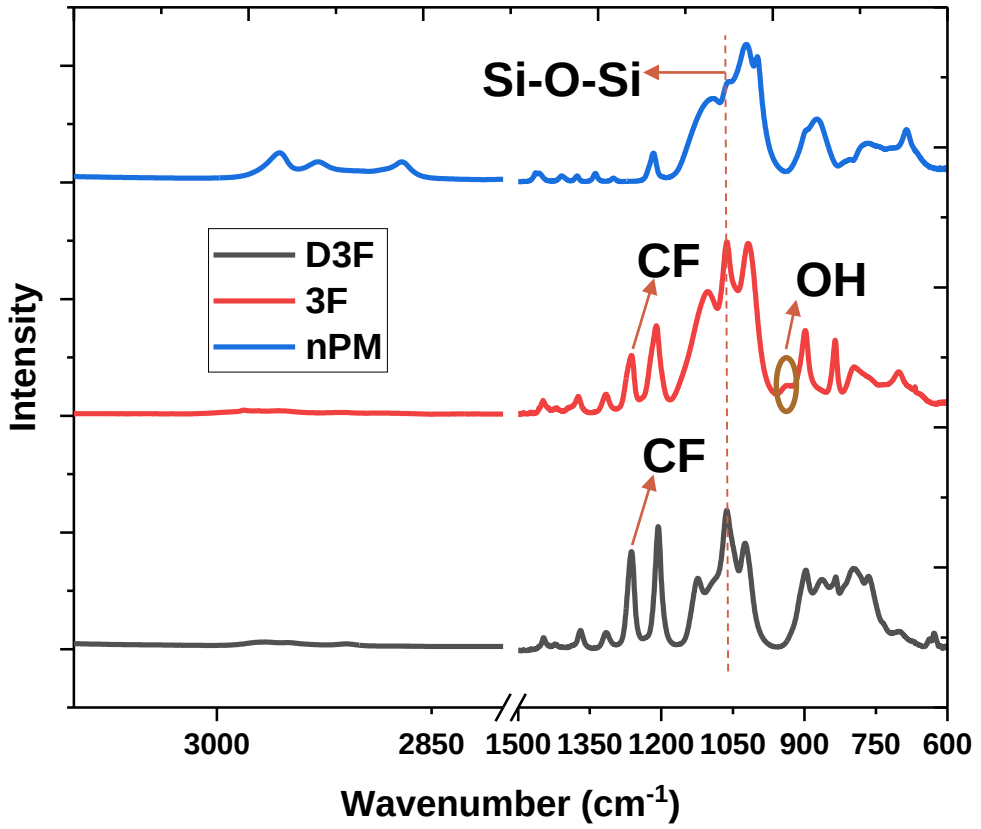


Di-functional analog of 3F does not form any NPs (nanoaggregates)



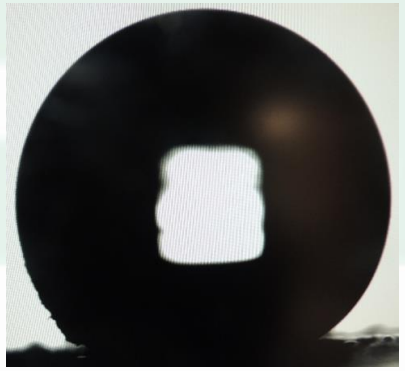
3F and nPM NPs formation mechanism



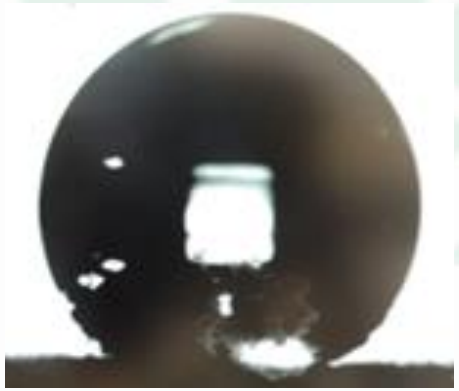


Super-hydrophobicity of 3F and nPM NPs

a)



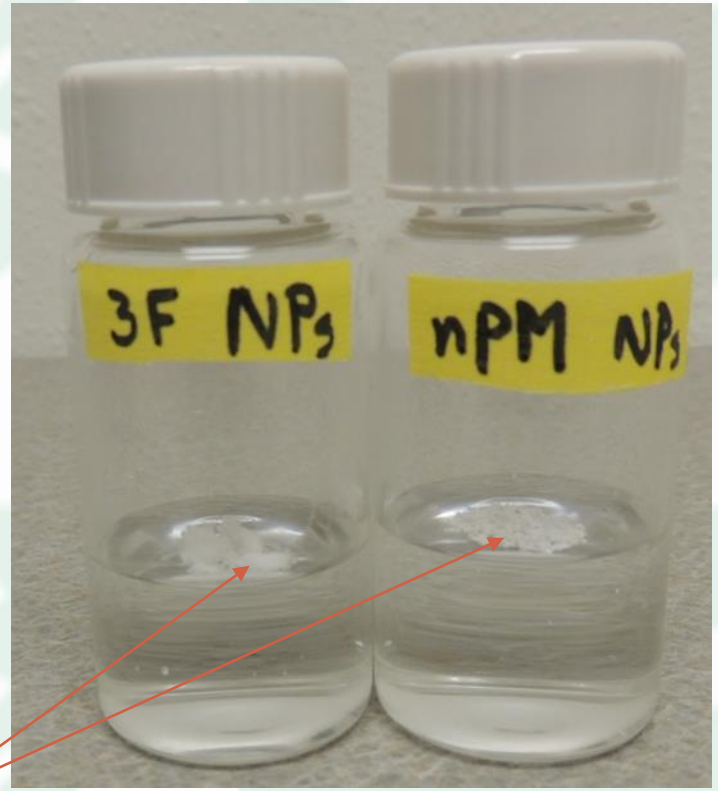
b)



3F NPs: 151°

nPM NPs: 153°

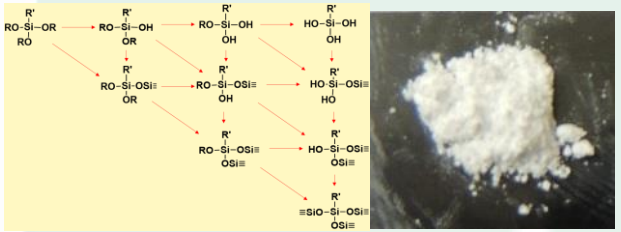
Static water contact angle on a) fluorinated silica NPs (3F NPs), and b) methylated silica NPs (nPM NPs) prepared as thin films on double-stick tape

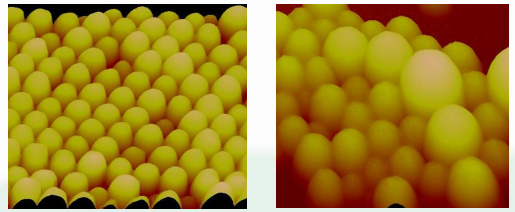


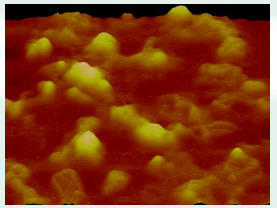
Floating NPs on water

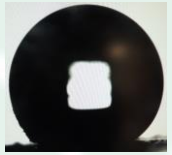
Conclusion

- Successful aqueous 1-step synthesis of fluoro- (3F) and methyl- (nPM) silica NPs
- 3F based NPs are monodisperse spheres while the methyl-silane nPM NPs were polydisperse under same reaction conditions
- Dimethoxy 3F control confirmed cross-linking necessary for NP formation
- Water contact angles confirmed super hydrophobicity









Thanks & Question?