Pickering Emulsions using a Fumed Silica and a Silica Sol - The Effect of Microfluidization

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Background

The ability of colloidal silica to stabilize nano-oil-in-water emulsions prepared using a Microfluidizer® has previously been demonstrated [1]. A vast amount of work has been performed and reported on fumed silica-stabilized emulsions. Nevertheless, the possibility to reduce the emulsion droplet size in these emulsions by means microfluidization has to our knowledge not been tested.

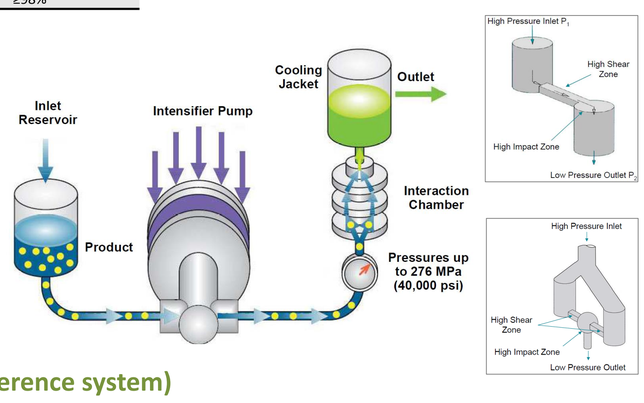
➢ Objective: **investigate the smallest emulsion droplet size attainable in fumed silica-stabilized emulsions** by means of microfluidization.

Materials

	Supplier	Comment
Levasil CC301	Nuyryon	Silica wt% 28 Average particle size 7 nm 2,5% ethanol
Fumed Silica	Wacker Chemie AG	Fumed silica powder, primary particle diameter is 25-30 nm.
Squalene	Sigma Aldrich	≥98%

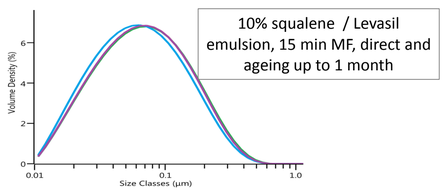
Microfluidization

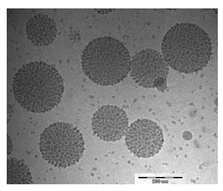
A M-110Y Microfluidizer processor (Microfluidics, USA), with a F2OY 75 µm interaction chamber (Y type) with a H30 Z 200 µm auxiliary chamber (Z type) placed inline and pressure of 600 bar was used.



Emulsification using a Silica Sol (Reference system)

Emulsions stabilized with sols of hydrophobically - modified silica (Levasil CC301) had a droplet size of 0.092 µm (volume mean diameter). No variations in droplet size were observed for at least a month (previous work indicates that these type of emulsions remain stable for years).

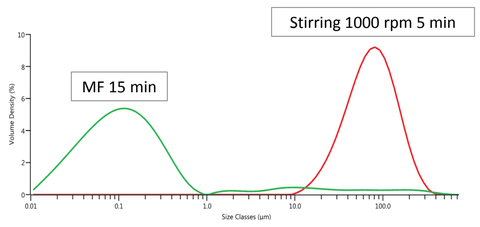




CRYO-TEM of 5% oil nano emulsion stabilized with a silica sol. The scalebar is 200 nm. From previous work. Acknowledgement Jonas Gustafsson

Deagglomeration of Fumed Silica

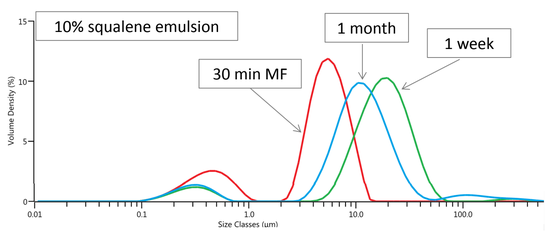
➢The Microfluidizer® was efficient for deagglomeration of fumed silica dispersion.

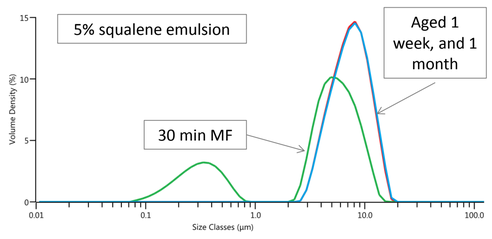


Emulsification using Fumed Silica

Method 1) 10 wt% oil-in-water, pre-emulsification step, microfluidized for 30 min.
Method 2) 5 wt% oil-in-water, silica pre-dispersed in microfluidizer, microfluidized for 30 min.

- ➢ Method 2 provides the most stable emulsion. Droplet size is constantly 2.5-20 µm between 1 week and 1 month.
- ➢ Bimodal size distribution. The smaller peak may be silica.

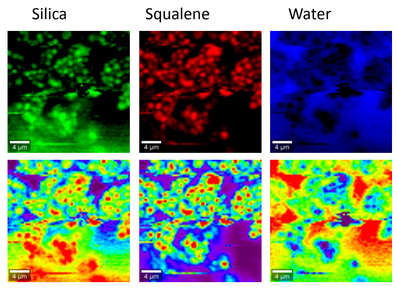


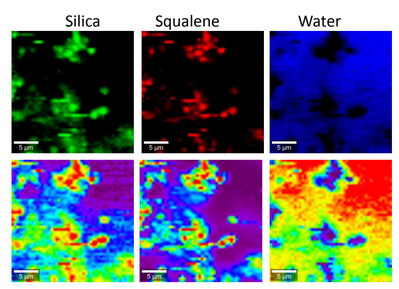


Morphology of Fumed Silica-stabilized Emulsions using Confocal Raman

Sample: 10 wt% squalene with fumed silica
The measurements were performed with a WITec alpha300 RAS system in combination with a 532 nm laser for excitation. -10°C cooling from below. 60x using a water immersion objective.

➢ The droplets are not spherical.



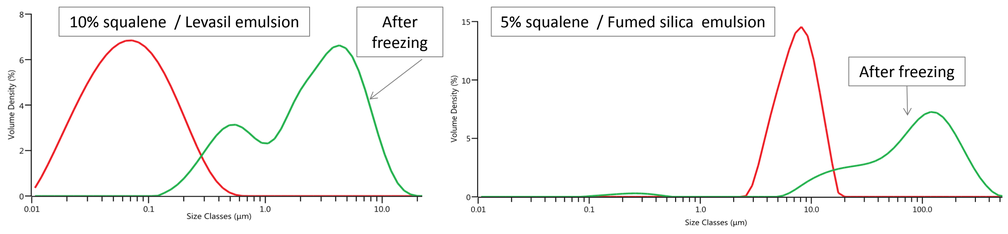


Freeze-thaw Stability of Silica-stabilizedd Emulsions

Emulsions stabilized with starch granules have been reported to exhibit high freeze-thaw stability [2].

The freeze-thaw stability of the fumed silica and Levasil CC301-stabilized emulsions was tested. The emulsions were placed in a freezer at -18°C for a week and allowed to thaw at RT before their size distribution was measured.

➢ The silica stabilized emulsions are not stable to freezing, and a significant increase in droplet size of the emulsions occurs.



Summary

Levasil CC301

- ✓ Levasil CC301 is a good stabilizer for nano-oil-in-water-emulsions.
- ✓ Emulsions with droplet size of 0.092 µm (volume mean diameter) were obtained with Levasil CC301.
- ✓ These emulsions are stable at least one month

Fumed Silica

- ✓ Fumed silica is good for stabilization of emulsions with droplets sizes larger than 2 µ, but it is not suitable for stabilization of nano-emulsions.
- ✓ Fumed silica can stabilize a 10% oil-in-water emulsions however, the droplet size of these are larger (2.5-10 µm), and they grow with time.
- ✓ Using less oil (5%) and pre-dispersing the fumed silica in the Microfluidizer results in an emulsions with a droplet size of 2.5-20 µm.
- ✓ The 5% emulsion was stable for at least one month

Freezing

- ✓ The silica stabilized emulsions were not stable to freezing/thawing.

[1] *Persson, K.H., Blute, I.A., Mira, I.C., Gustafsson, J. Creation of well-defined particle stabilized oil-in-water nanoemulsions, 2014, Colloids and Surfaces A: Physicochemical and Engineering Aspects, 459, pp. 48-57.*
[2] *A Marefati et al. Colloids and Surfaces A: Physicohem. Eng. Aspects 436 (2013) 512-520*

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