



Physico-chemical separation process of nanoparticles and nanostructured materials of cosmetic formulations

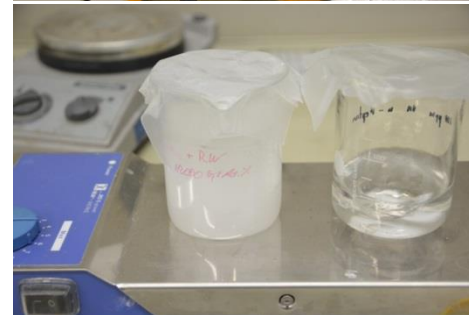
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SOP-development for characterisation of NM

- Wetting/Suspending
- Dispersion
- Sampling
- Separation the NM
- Measurement/Interpretation



Motivation & objective

- Synthetic amorphous silica (SAS) = nanostructured material
 - used in a wide range of food and cosmetic emulsions
 - wide range of dispersion energies when preparing or applying such products
 - submicron aggregate and/or nanosized particles
- behaviour of the nanomaterial (NM)
 - dispersibility in aqueous and lipid phases
 - adsorption at fluid-fluid interfaces or on the surface of large particles
- Objective
 - separation or extraction of SAS of lipid phases by means of organic solvents

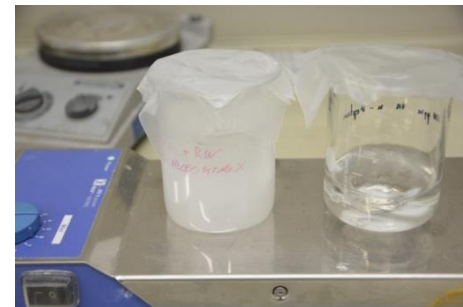
Instruments, materials and preparation

Materials and instruments

- Materials
 - Nanomaterials (NM) in emulsions-based
 - 10 wt.-% pyrogenic synthetic amorphous silica (SAS)
 - 24 wt.-% lipid phase
 - dispersion media:
 - de-ionised water (18.3 MΩcm), filtered at 0.2 μm
 - lipophilic solvent n-heptane (99%), filtered at 0.1 μm
- Characterisation techniques
 - laser diffraction (LD) – HELOS KR (Sympatec/Germany)
 - dynamic light scattering (DLS) – HPPS (Malvern/UK)
 - scanning electron microscopy (SEM) – Gemini DSM 980 (Zeiss/Germany)

Preparation

- Preparation emulsion sample
 - 1.0 g emulsion in 49.5 g water and 49.5 g n-heptane
- Sampling:
 - after sedimentation
 - after centrifugation (4500 rpm)
- Dispersion and homogenisation of emulsion sample:
 - magnet stirring (Fa. IKA)
- Filtration grade
 - syringe filter (PTFE membrane) for different pore size 0,1 μm , 1 μm und 5 μm





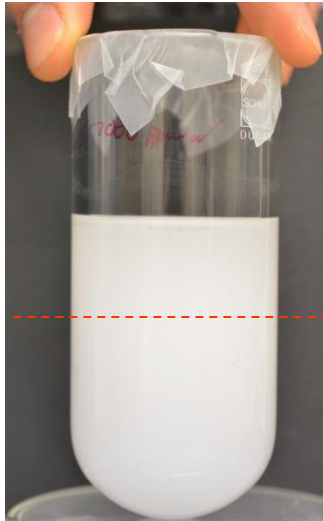
Results

Methodological procedure

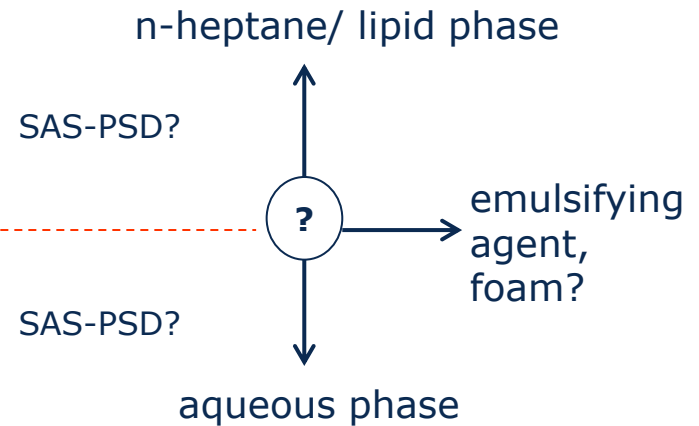
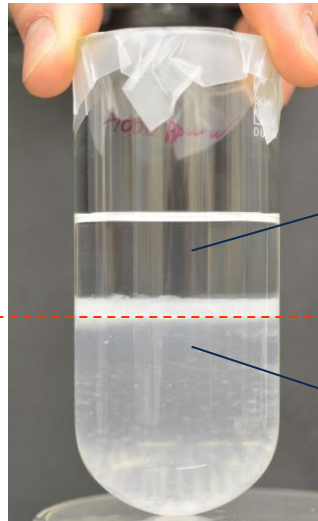
- Question:
How can SAS in cosmetic emulsion (SAS in oil-water-emulsifying agent) be characterised and separated better?
- Procedure of solution:
Interaction of physico-chemical separation process
→ Extraction
 - Oil with organic lipophilic solvent (Hansen,2007)
 - Separation of SAS-particles (sedimentation/centrifugation)
 - Mass balance (preparation of emulsion)
 - Filtration (PTFE membrane)

Evolution of extraction process

after 20 h

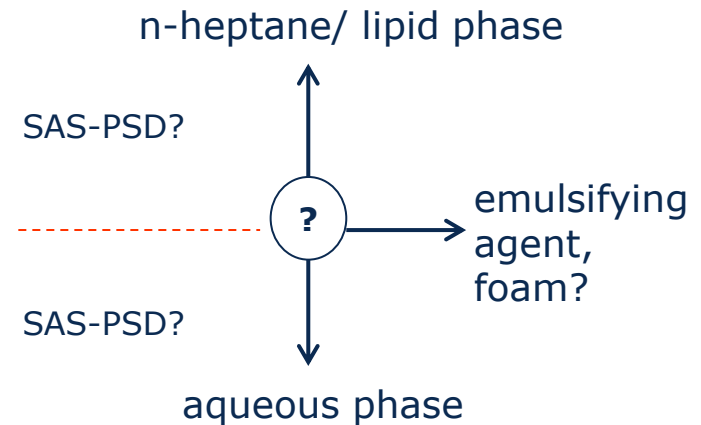
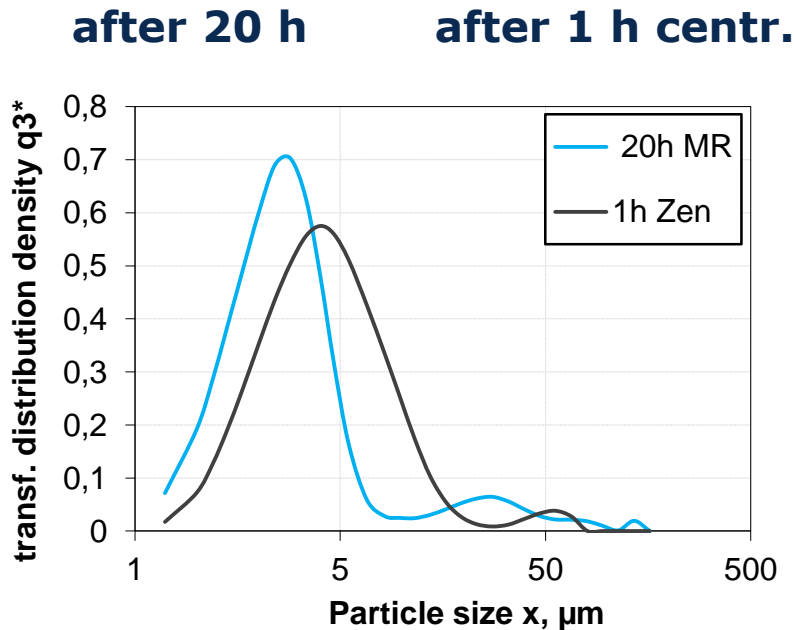


after 1 h centr.



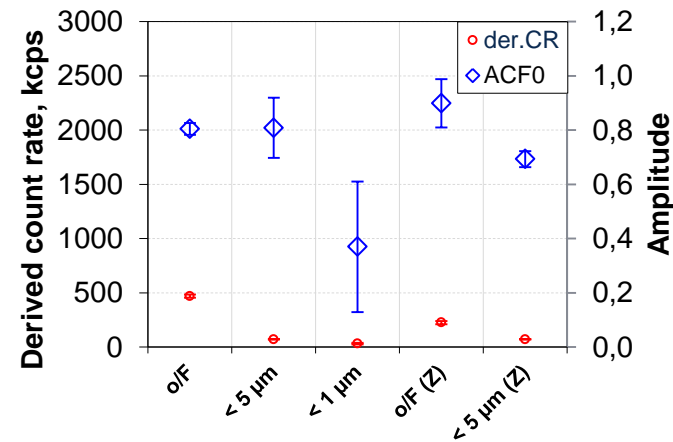
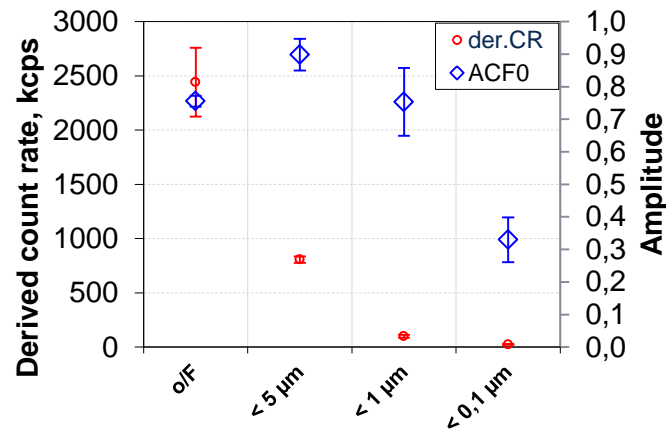
- Hydrophobic fumed SiO_2 in water and n-heptane (mix ratio 1:1)

LD - Evolution of extraction process



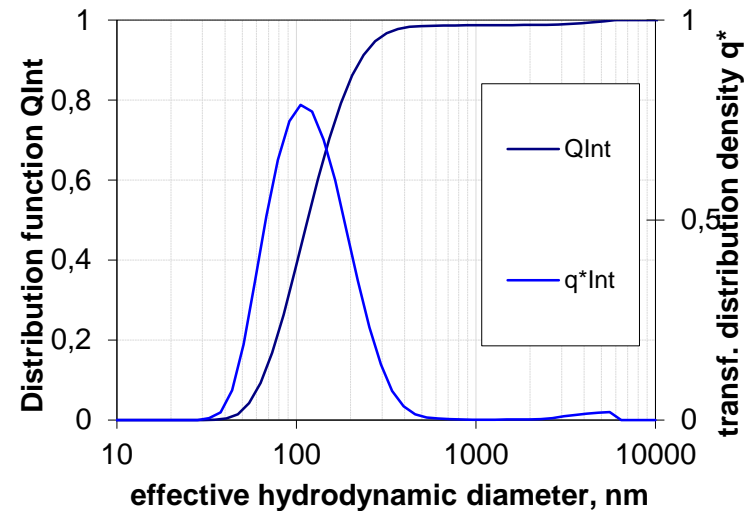
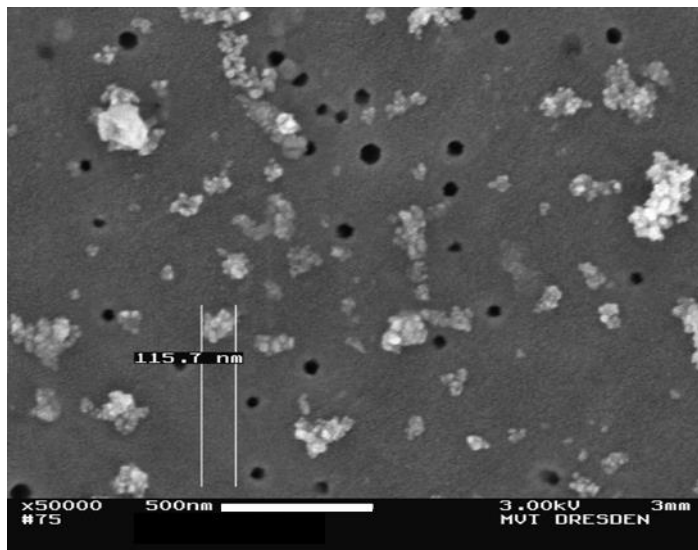
- Hydrophobic fumed SiO_2 in water and n-heptane (mix ratio 1:1)
- Change of size distribution (determined with LD) of a SAS cosmetic emulsion within water phase, realised by magnetic stirrer (MR) and centrifugation (Zen)

DLS - Effectivity of separation



- Separation = filtration with membrane
- DLS-result for the detection of SiO₂ nanoparticles in cosmetic formulation; water phase left: with, right: without SiO₂
- Clear trend: count rate ↓ with cut size ↓ of separation degree of derived count rate (der.CR) for filtration grade (syringe filter; PTFE membrane)

SEM analysis



- Existence of SAS-particles in n-heptane phase?
- Left: weighted intensity distribution for SiO_2 in n-heptane phase; right: SEM image with high magnification („finest particles found“); sample carrier: track-etch-membrane with 50 nm pore

Conclusion

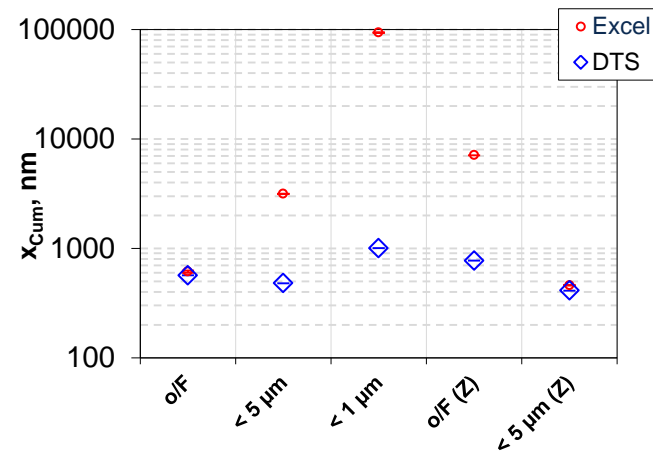
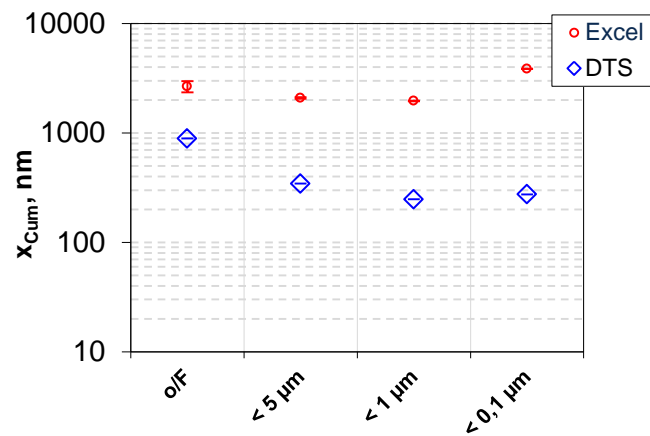
- This investigation verified an alternative procedure to separate particles with regard to the polarity of solvent and solute in such complex media such as cosmetic emulsions
 - This procedure is applicable to O/W and W/O emulsions and it can be employed for hydrophilic as well as hydrophobic SAS
- Interaction of physico-chemical separation process
 - no presence of sediment after centrifugation
 - stable phases (aqueous phase and lipophilic solvent)
 - presence of a liquid foam (oil-water-emulsifying agent) at the interface between water and heptane
 - discrepancy of density and polarity of solvent play an important role of separation and homogenisation

Thank you for your attention!

References

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Effectivity of separation - DLS



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