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

R. R. Retamal Marín, F. Babick, M. Stintz
Research Group Mechanical Process Engineering, TU Dresden

Nanosafe, 8th of November 2016
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

- 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 SiO$_2$ nanoparticles in cosmetic formulation; water phase left: with, right: without SiO$_2$
- **Clear trend**: count rate $\downarrow$ with cut size $\downarrow$ 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


Effectivity of separation - DLS

- Separation = filtration with membrane
- DLS-result for the detection of SiO$_2$ nanoparticles in cosmetic formulation; water phase left: with, right: without SiO$_2$
- Clear trend: count rate ↓ with cut size ↓ of separation degree of derived count rate (der.CR) for filtration grade (syringe filter; PTFE membrane)