Encapsulation in double emulsions
Fundamental analysis of stability

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Formula X I Manchester I 24-27th June 2019
Double emulsions: promising structures to encapsulate hydrophilic active ingredients

Potential applications - Encapsulation of...
- ...enzymes, proteins or peptides for detergents
- ...hydrophilic bioactive ingredients (e.g. vitamins) for cosmetic and food applications
- ...hydrophilic crop protecting agents and active ingredients in pharmaceuticals

Benefits
- Stability/protection of active ingredients
- Triggered or retarded release
- Taste/smell masking
- Drift and washing-out prevention
Double emulsions – example “Hollow microcapsules”

Concept
- Filling of empty, porous capsules with active material
- Pores of capsules to be closed after filling

Benefits
- Universal capsules for various active ingredients
- Biodegradable capsule matrix

Porous capsule

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Active loading

Curing

@customer

Closed capsule
Double emulsions – example “Hollow microcapsules”

**Step 1: W₁ in O emulsification**
- Lipophilic surfactant (W/O-emulsifier)
- High energy input (e.g. gear rim dispersing device)

**Step 2: (W₁/O) in W₂ emulsification**
- Hydrophilic surfactant (O/W-emulsifier)
- Low energy input (e.g. stirred vessel)

Distillation of solvent

Filtration

Washing
Double emulsions: challenges

Advantages
- Various different applications
- Preparation with common equipment

BUT
- Big challenge to keep active inside
- No guidelines for process and product development
Double emulsions: challenges
Analysis of coalescence- and diffusion phenomena in $W_1/O/W_2$-double emulsions

- New analytical approaches for investigating instability mechanisms
- Influence of process parameters
- Identification of structure/property-relationships
- Guidelines for faster formulation and process development

Formulation and process development based on molecular understanding
Methods to investigate instability mechanisms

- Diffusion and coalescence at interfaces: single drop experiments & interfacial tension measurements

- Characterization of interfaces via nonlinear spectroscopy (SFG, SHG)

- Supported by molecular modeling (BASF)

- Analysis of double emulsions in different scales

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Diffusion and Coalescence Time Analyzer*
Influence of emulsifier systems

Diffusion and coalescence at interfaces
Influence of emulsifier system

Oil phase: Miglyol® 812
W/O-emulsifier: PGPR
O/W-emulsifier: Lutensol® TO8

O/W-emulsifier disturbs stability
Analysis of emulsifier diffusion
Interfacial tension measurements

Oil phase: Miglyol® 812
O/W-emulsifier: Lutensol® TO8

Determination of emulsifier diffusion via interfacial tension
Characterization of interfaces
Nonlinear spectroscopy (SFG, SHG)

- Second Harmonic Generation (SHG): amount of molecules at interface (intensity)
- Sum Frequency Generation (SFG): type and orientation of molecules (spectra)

Planar setup

Scattering setup

- Fundamental analysis
- Learning about systems

Analysis of real emulsions

Interfacial emulsifier composition

Experimental SFG setup
Experimental SHG setup
Characterization of interfaces
Planar SFG spectra I Influence of O/W-emulsifier

- Distinction between Miglyol® 812 and Lutensol® TO 8 is possible
- Lutensol® TO 8 dominates at interface
- Ordered and covering layer of surfactant

✔ Detection of O/W-emulsifier at interface
Characterization of interfaces
Scattering SHG analysis \& Adsorption of Malachite green

Emulsion production: Ultrasound
Dispersed phase: Miglyol® 812 (φ=1 %)
Continuous phase: water + surfactant
Addition of malachite green

- Miglyol® 812 I Texapon® NSO (24 mM, d = 182 nm)
- Miglyol® 812 I SDS (0.3 mM, d = 190 nm)
- Hexadecane I SDS (0.3 mM, d = 180 nm)

✓ Different types of adsorption depending on emulsion properties
?
Surfactant molecules: replacement, binding on, relocation…
Double emulsions - Summary & Outlook

Advantages

- Various different applications
- Preparation with common equipment

Challenges

- Keeping the active inside → stability issues
- No guidelines for process and product development

Analysis of instability mechanisms

- New technical approaches to analyze instability mechanisms and for the characterization of interfaces
- Applicability of analytical approaches shown
- Next steps: screening of different emulsifiers and transfer of gained knowledge to real systems
We create chemistry