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Encapsulation in double emulsions
Fundamental analysis of stability

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Formula X I Manchester I 24-27th June 2019

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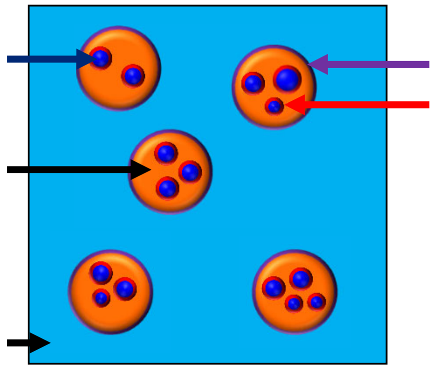
LVT Lebensmittelverfahrenstechnik
LFG Erlangen
FAU

Double emulsions: promising structures to encapsulate hydrophilic active ingredients



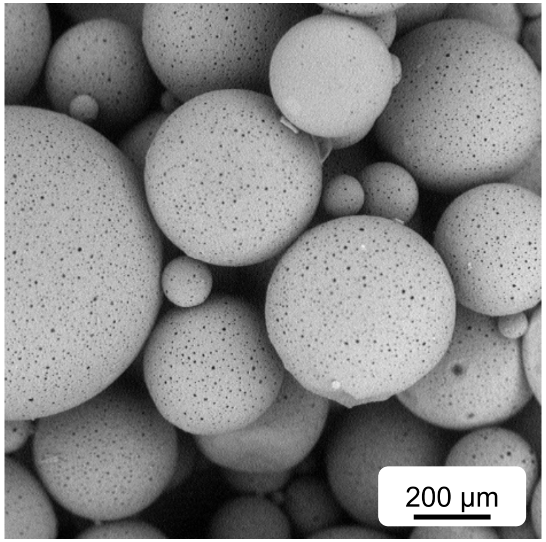
Potential applications - Encapsulation of…

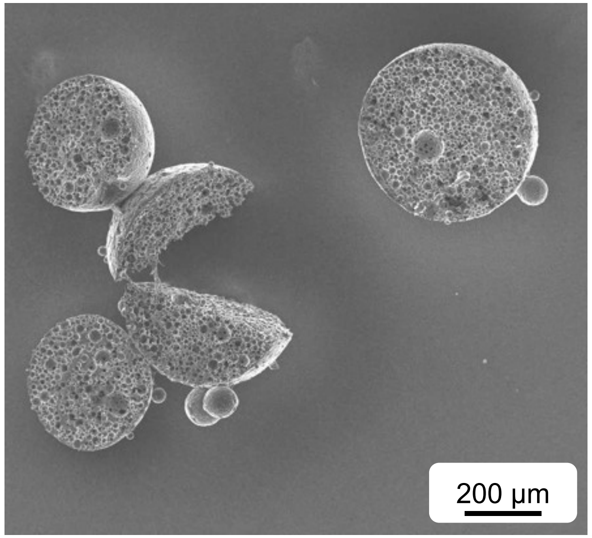
- ✓ …enzymes, proteins or peptides for detergents
- ✓ …hydrophilic bioactive ingredients (e.g. vitamins) for cosmetic and food applications
- ✓ …hydrophilic crop protecting agents and active ingredients in pharmaceuticals

Benefits

- ✓ Stability/protection of active ingredients
- ✓ Triggered or retarded release
- ✓ Taste/smell masking
- ✓ Drift and washing-out prevention

Double emulsions – example "Hollow microcapsules"



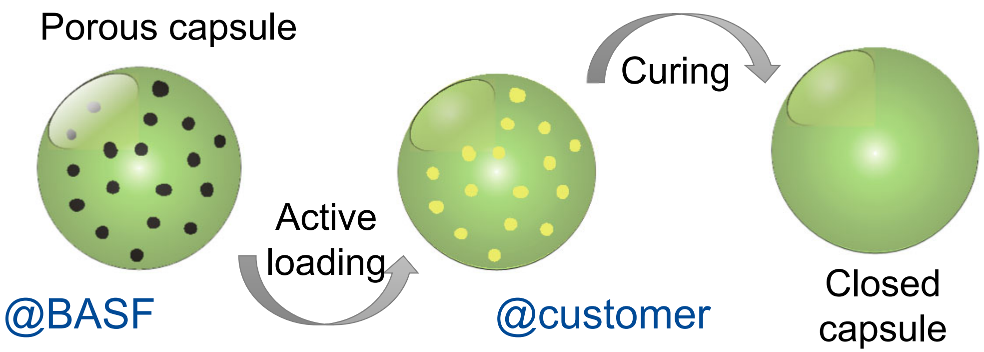


Concept

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

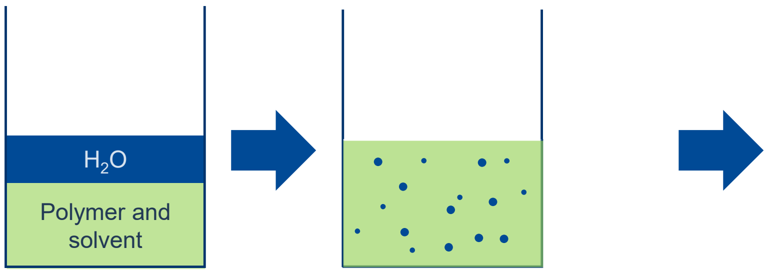
Benefits

- Universal capsules for various active ingredients
- Biodegradable capsule matrix



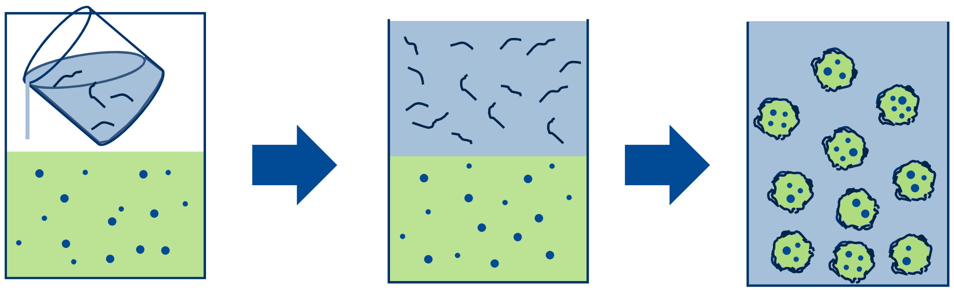
Double emulsions – example "Hollow microcapsules"

Step 1: W_1 in O emulsification

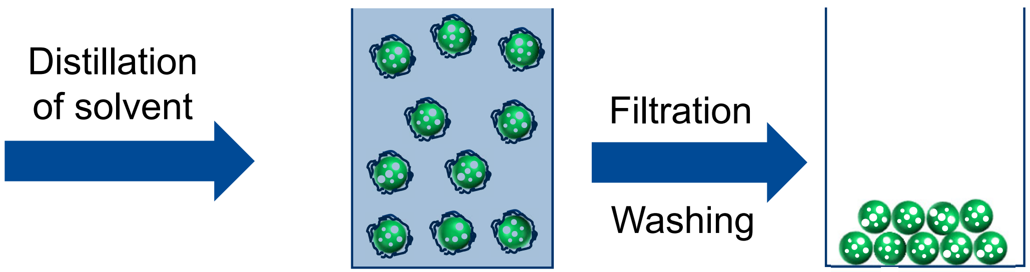


- Lipophilic surfactant (W/O-emulsifier)
- High energy input (e.g. gear rim dispersing device)

Step 2: (W_1/O) in W_2 emulsification



- Hydrophilic surfactant (O/W-emulsifier)
- Low energy input (e.g. stirred vessel)



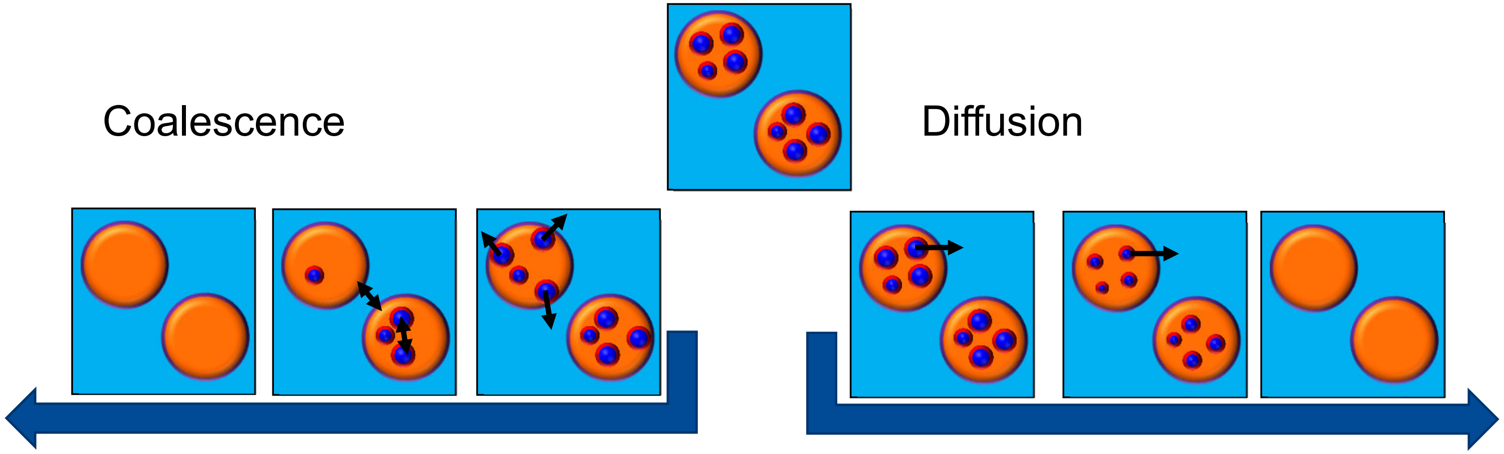
Double emulsions: challenges

Advantages

- Various different applications
- Preparation with common equipment

BUT

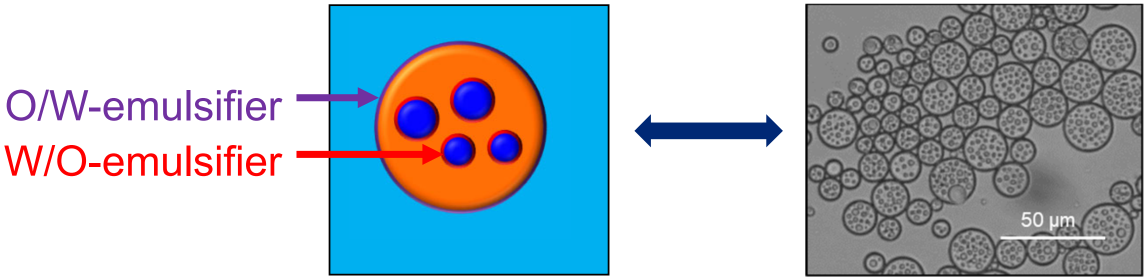
- Big challenge to keep active inside
- No guidelines for process and product development



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

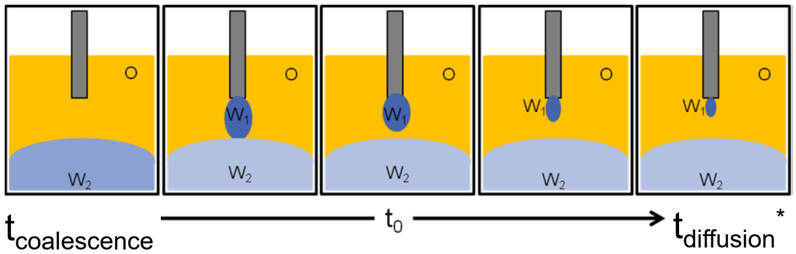


- Identification of structure/property-relationships
- Guidelines for faster formulation and process development

Formulation and process development based on molecular understanding

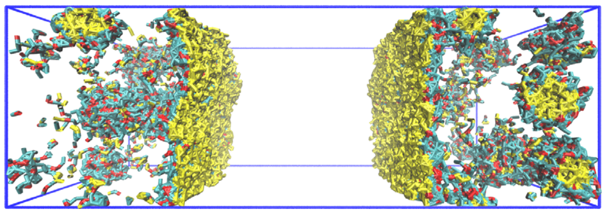
Methods to investigate instability mechanisms

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

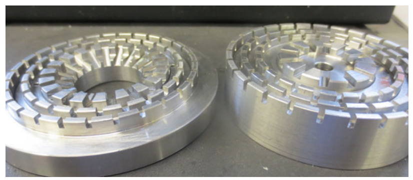


- Characterization of interfaces via nonlinear spectroscopy (SFG, SHG)

- Supported by molecular modeling (BASF)



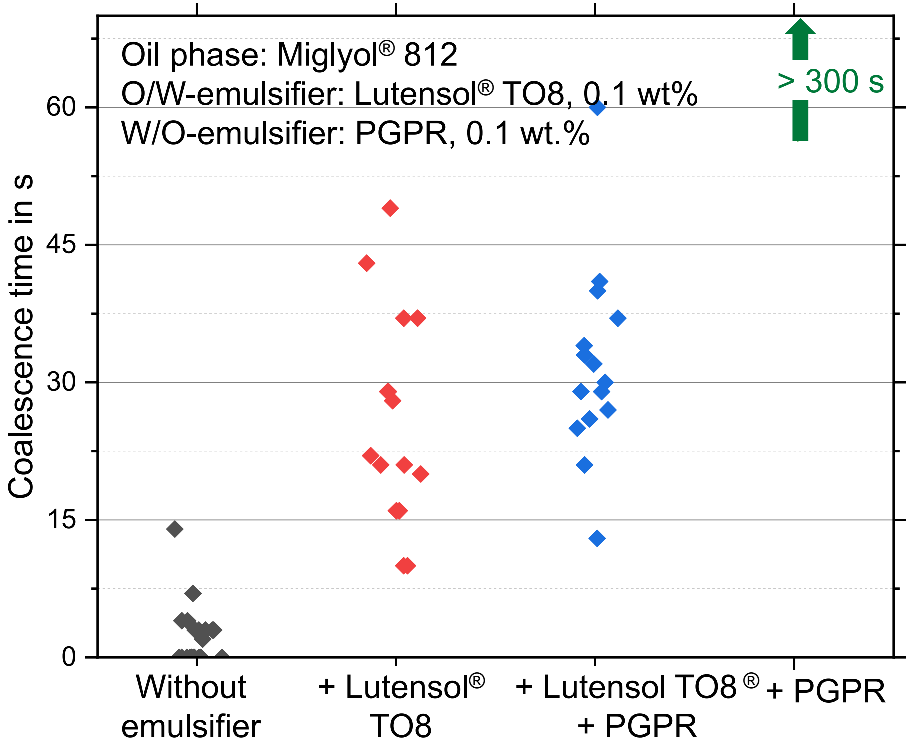
- Analysis of double emulsions in different scales



*S. M. Neumann, CC BY

Diffusion and Coalescence Time Analyzer*

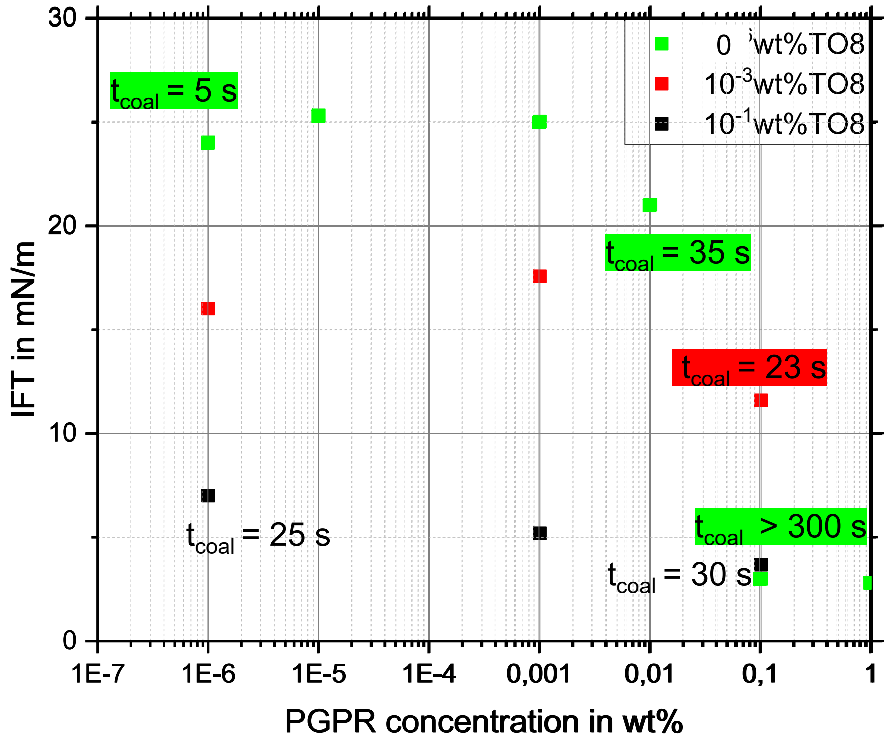
Influence of emulsifier systems



S. M. Neumann, U. van der Schaaf, H.P. Schuchmann: The Diffusion and Coalescence Time Analyzer (DCTA): A novel Experimental setup for investigating instability phenomena in double emulsions. Food Structure 12 (2017) 103 – 112.

Diffusion and coalescence at interfaces

Influence of emulsifier system

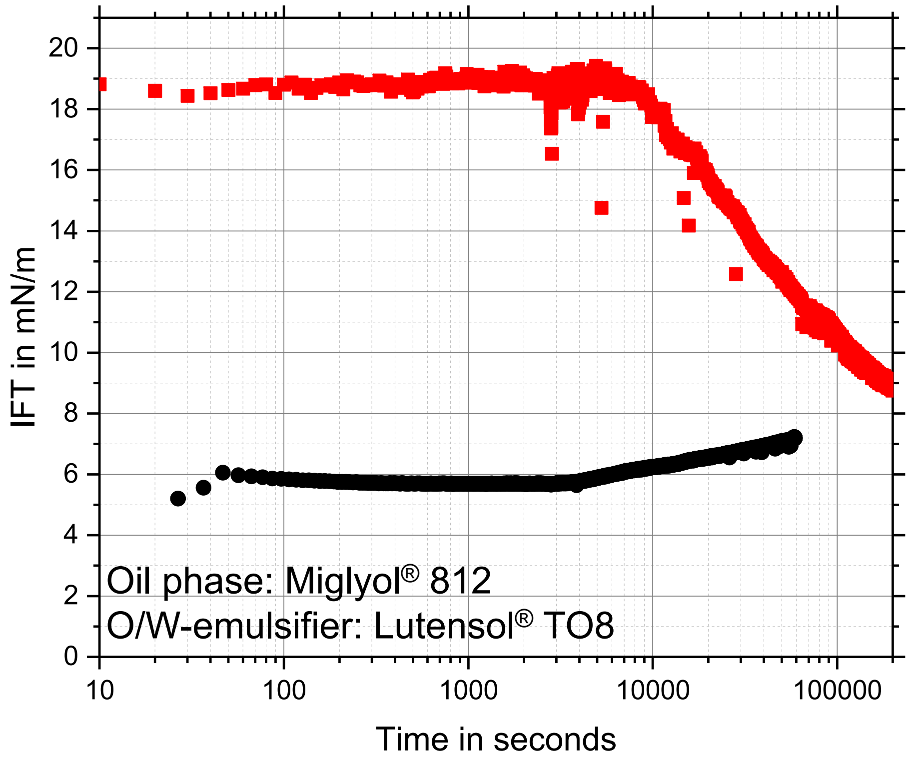


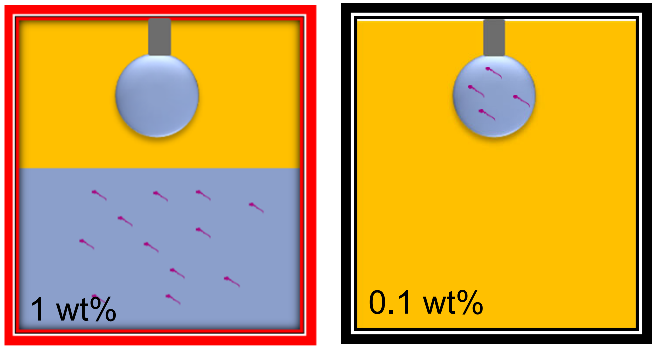
Oil phase: Miglyol® 812
W/O-emulsifier: PGPR
O/W-emulsifier: Lutensol® TO8

O/W-emulsifier disturbs stability

Analysis of emulsifier diffusion

Interfacial tension measurements





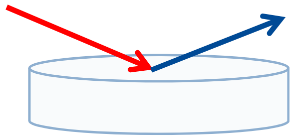
Determination of emulsifier diffusion via interfacial tension

Characterization of interfaces

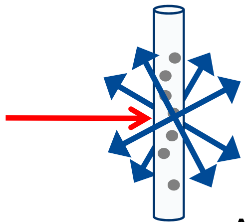
Nonlinear spectroscopy (SFG, SHG)

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

Planar setup



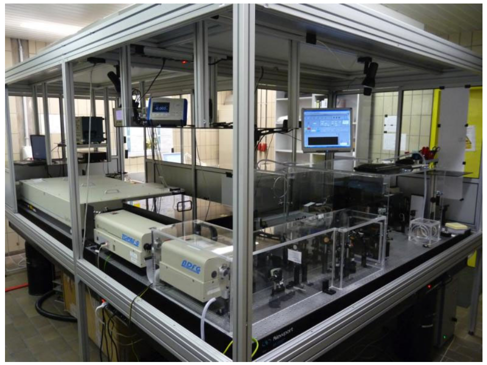
Scattering setup



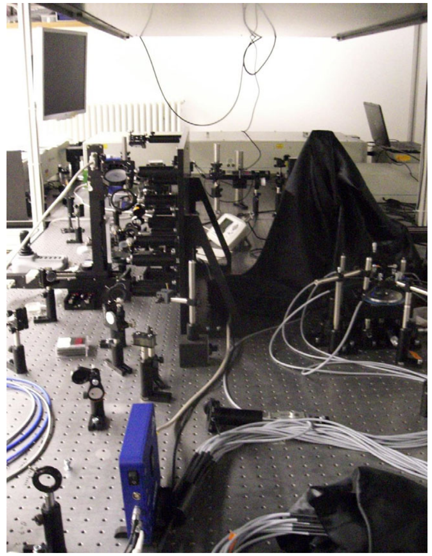
✓ Fundamental analysis
✓ Learning about systems

Analysis of real emulsions

Interfacial emulsifier composition



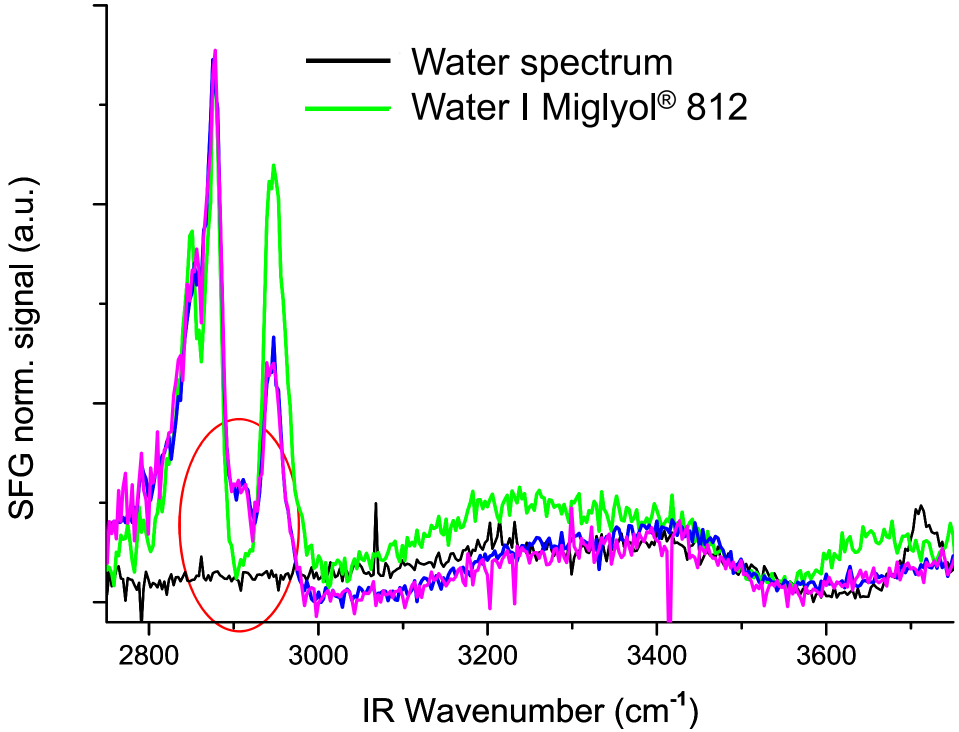
Experimental SFG setup



Experimental SHG setup

Characterization of interfaces

Planar SFG spectra I Influence of O/W-emulsifier

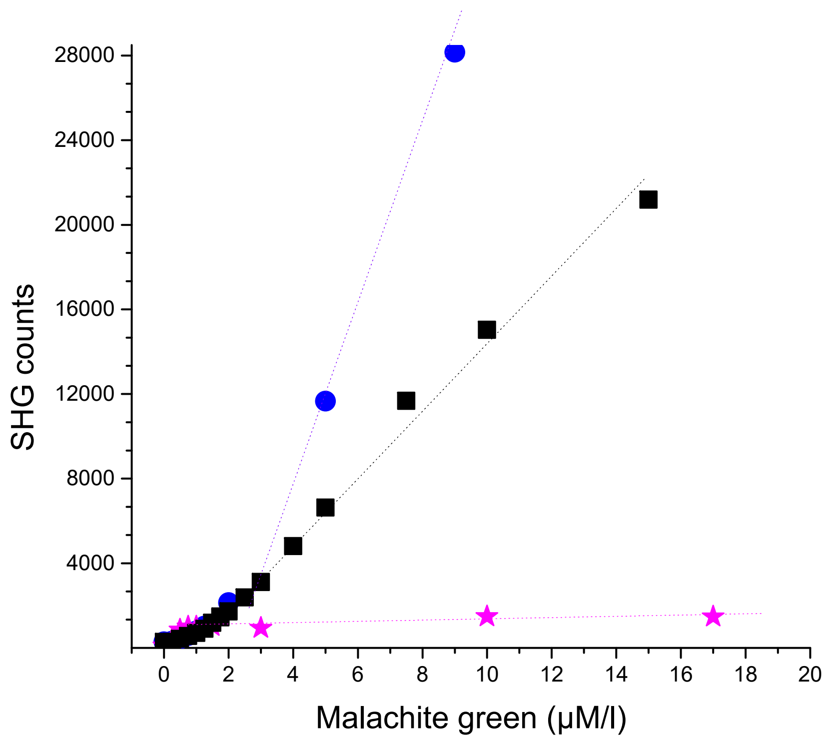


- Distinction between Miglyol® 812 and Lutensol® TO 8 is possible
- Lutensol® TO 8 dominates at interface
- Ordered and covering layer of surfactant

✓ **Detection of O/W-emulsifier at interface**

Characterization of interfaces

Scattering SHG analysis I 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…

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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

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