Droplet Size Distribution of Technical Macroemulsions

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Motivation

• Emulsion-based precipitation of tailor-made nanoparticles [1]

⇒ Important control parameter: droplet size and size distribution

<u>w/o-microemulsion</u>
• Nano-sized droplets
• Narrow distribution
• **But:** small fraction of the dispersed phase

Increase of the water fraction ⇒

<u>Technical mini- and macroemulsion</u>
• Kinetically stabilized
• Droplet size and size distribution depend on different process conditions

⇒ Investigation of droplet size variation of technical emulsions

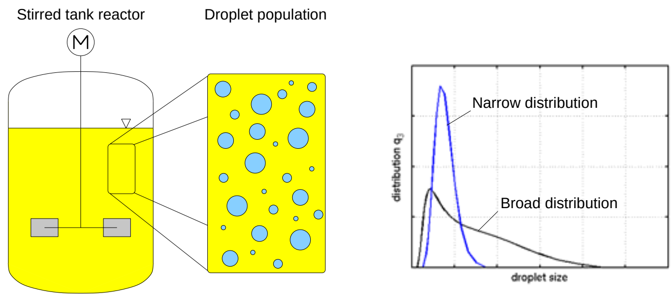

Fig. 1: Process scheme (left) and droplet size distributions (right).

Experimental Setup

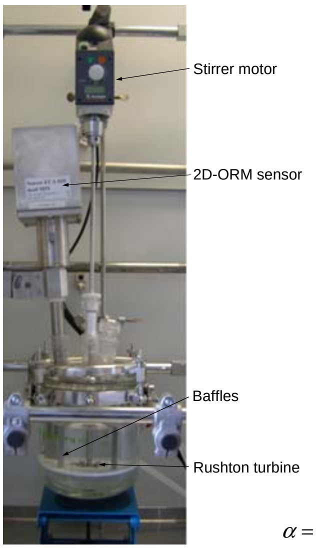

Fig. 2: Reactor setup.

<u>Reactor setup</u>
• 2 L stirred tank reactor with heat jacket
• 4 baffles
• Rushton turbine

<u>Technical emulsions</u>
• Water, n-decane
• Nonionic surfactants:
  NP4 – nonylphenol ethoxylate
  Span20 – sorbitan ester

<u>Process control parameters</u>
• Mass fraction parameters of the emulsion, α and γ
• Stirring speed
• Temperature
• Hydrophilic-lipophilic balance (HLB) of the surfactant

$$\alpha = \frac{m_{oil}}{m_{oil} + m_{water}} \qquad \gamma = \frac{m_{surfactant}}{m_{oil} + m_{water} + m_{surfactant}}$$

Measuring Equipment

• Optical reflectance measurement (ORM) technique

⇒ In-situ and on-line measurement even in high concentrated systems [2]

⇒ Obtaining a chord length distribution over thousands of droplets per second

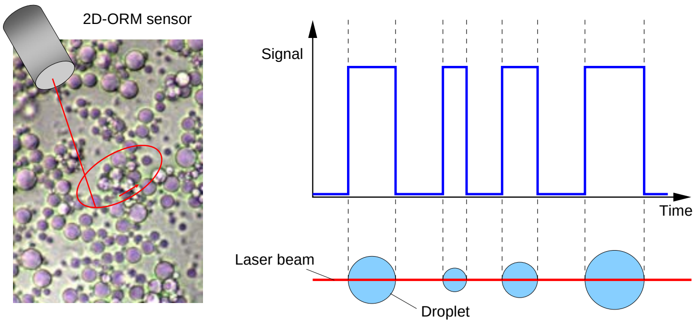

Fig. 3: Circular path of the rotating laser beam (left) and relation between scanned droplets and reflected signal (right).

Experimental results

Process parameters

• Oil mass fraction: α = 0.6, 0.7
• Surfactant mass fraction: γ = 0.005, 0.015
• HLB value: 9.75 (NP4) and 8.6 (Span20)
• Stirring speed: 200 - 400 rpm
• Temperature: 25 - 75 °C

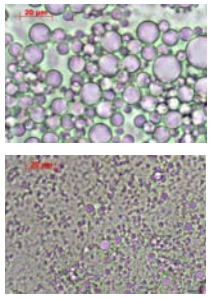

Fig. 4: Microscope pictures of w/o emulsion stabilized with NP4 α = 0.7 and γ = 0,015 (top) and with Span20 α = 0.6 and γ = 0.005 (bottom).

Variation of stirring speed

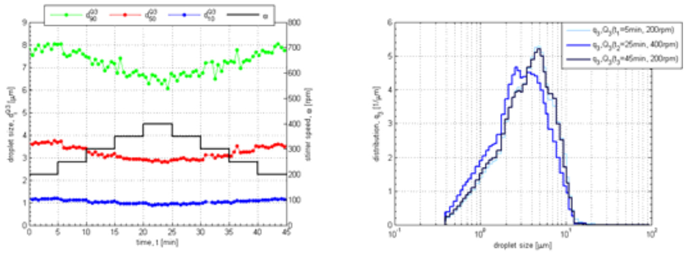

Fig. 5: Volume-based mean droplet sizes vs. stirring speed over time for a water, n-decane and NP4 emulsion (α=0.7, γ=0.015 and T=25°C) (left) and the droplet size distributions for ω=200rpm and ω=400rpm (right).

Variation of temperature and surfactant

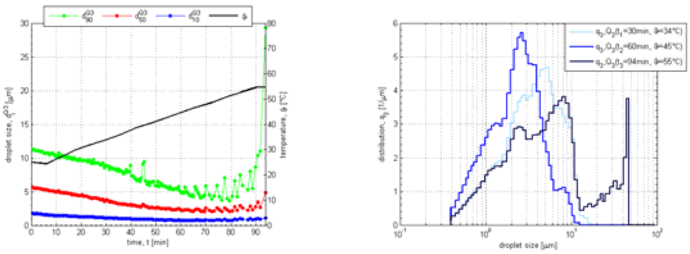

Fig. 6: Volume-based mean droplet sizes vs. temperature over time for a water, n-decane and NP4 emulsion (α=0.7, γ=0.015 and ω=400 rpm) (left) and the droplet size distributions for T=34°C, T=45°C and T=55°C (right).

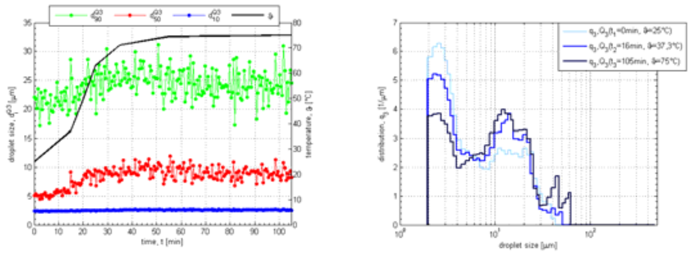

Fig. 7: Volume-based mean droplet sizes vs. temperature over time for a water, n-decane and Span20 emulsion (α=0.6, γ=0.005 and ω=200 rpm) (left) and the droplet size distributions for T=25°C, T=37.3°C and T=75°C (right).

Conclusions

• Droplet size distribution might be influenced by type of surfactant, stirring speed and temperature.
• Stirring speed marginally influences the droplet size and distribution.
• Temperature changes have a significant impact on the drop size.
• Insufficient energy input to homogenize emulsions containing Span20.

Publications

[1] D. Adityawarman, A. Voigt, P. Veit and K. Sundmacher. Precipitation of $BaSO_4$ nanoparticles in a non-ionic microemulsion: Identification of suitable control parameters. *Chemical Engineering Science* 60 (12): 3373-3383, 2005

[2] J. Lovick, A.A. Mouza, S.V. Paras, G.J. Lye, P. Angeli. Drop size distribution in highly concentrated liquid-liquid dispersions using a light back scattering method. *Journal of Chemical Technology and Biotechnology* 80: 545-552, 2005

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