



# From micrometre scale insights to novel process design

March 31<sup>st</sup> 2014, Karin Schroën

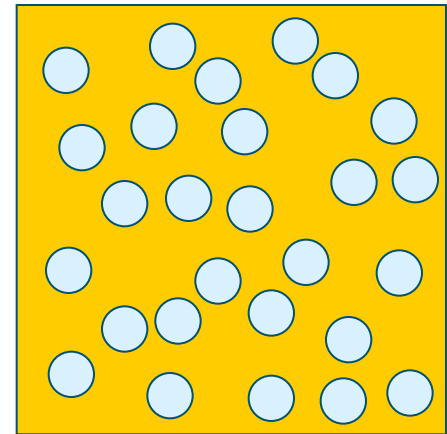
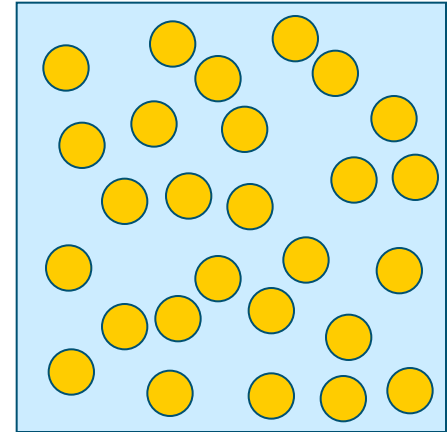


# Overview

- Emulsions and emulsification methods
  - Energy
  - Product properties
- Findings in the field of membranes & micro technology
  - Cross-flow membrane emulsification
  - Spontaneous emulsification (EDGE)
  - (Hybrid) pre-mix emulsification
- How to match these findings with large scale production?
- Conclusions and link to conference theme

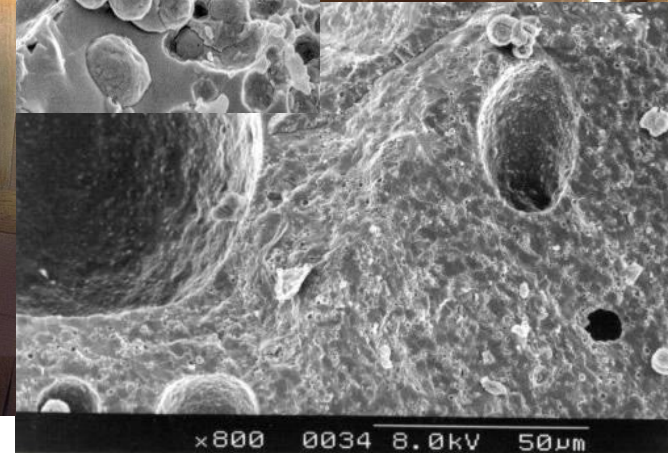
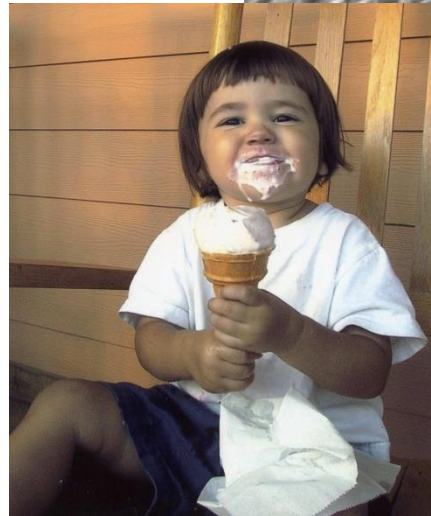
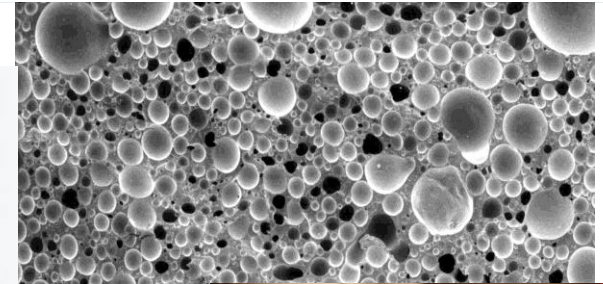
# Emulsions

- Dispersion of one immiscible (fluid) phase into another
- Oil in water
- Water in oil
  
- Gas and water or oil: foam / droplets



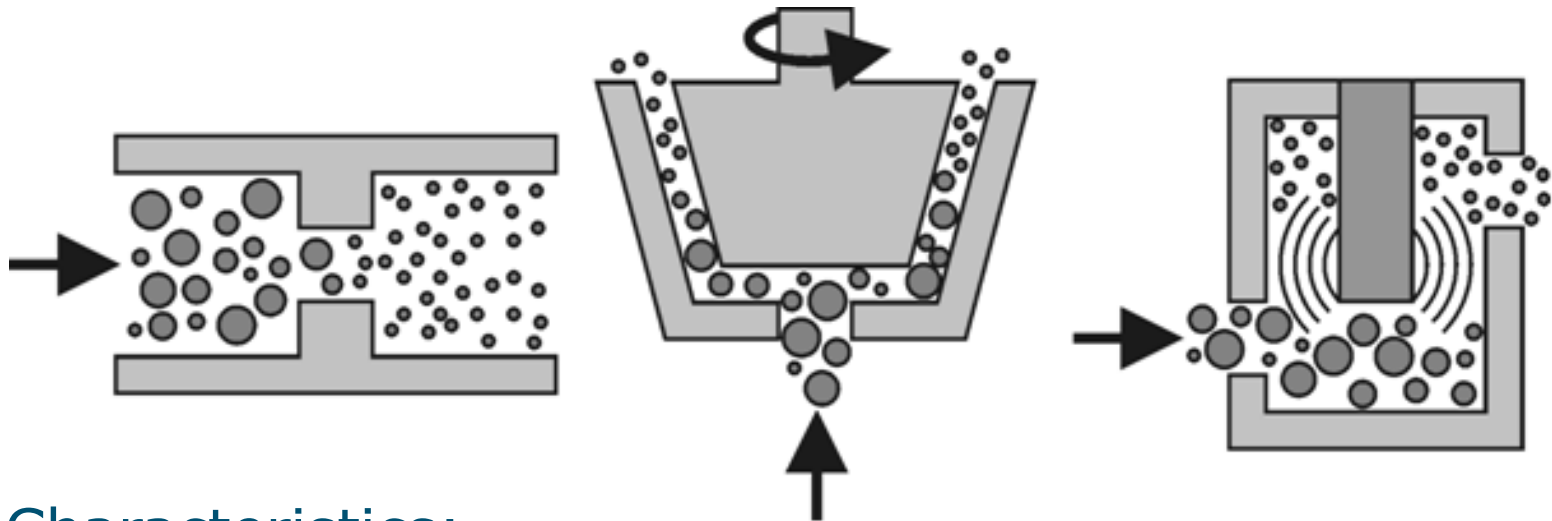
# Emulsions

- Paint
- Bitumen
- Mayonnaise
- Salad dressing
- Egg yolk
- Sausage
- Beer
- Milk
- Butter, margarine
- Bread
- Cream
- Ice cream



# Emulsification

- Mechanical methods
  - High-pressure homogenisers
  - Rotor-stator systems
  - Ultra sonifiers



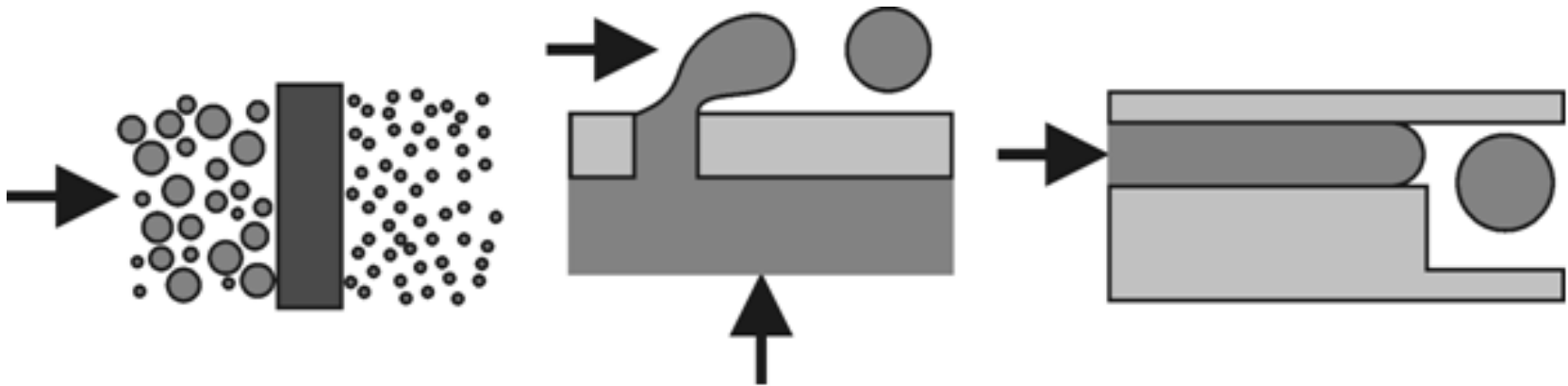
Characteristics:

High energy input, Temperature effects, Multiple passes



# Microstructured systems

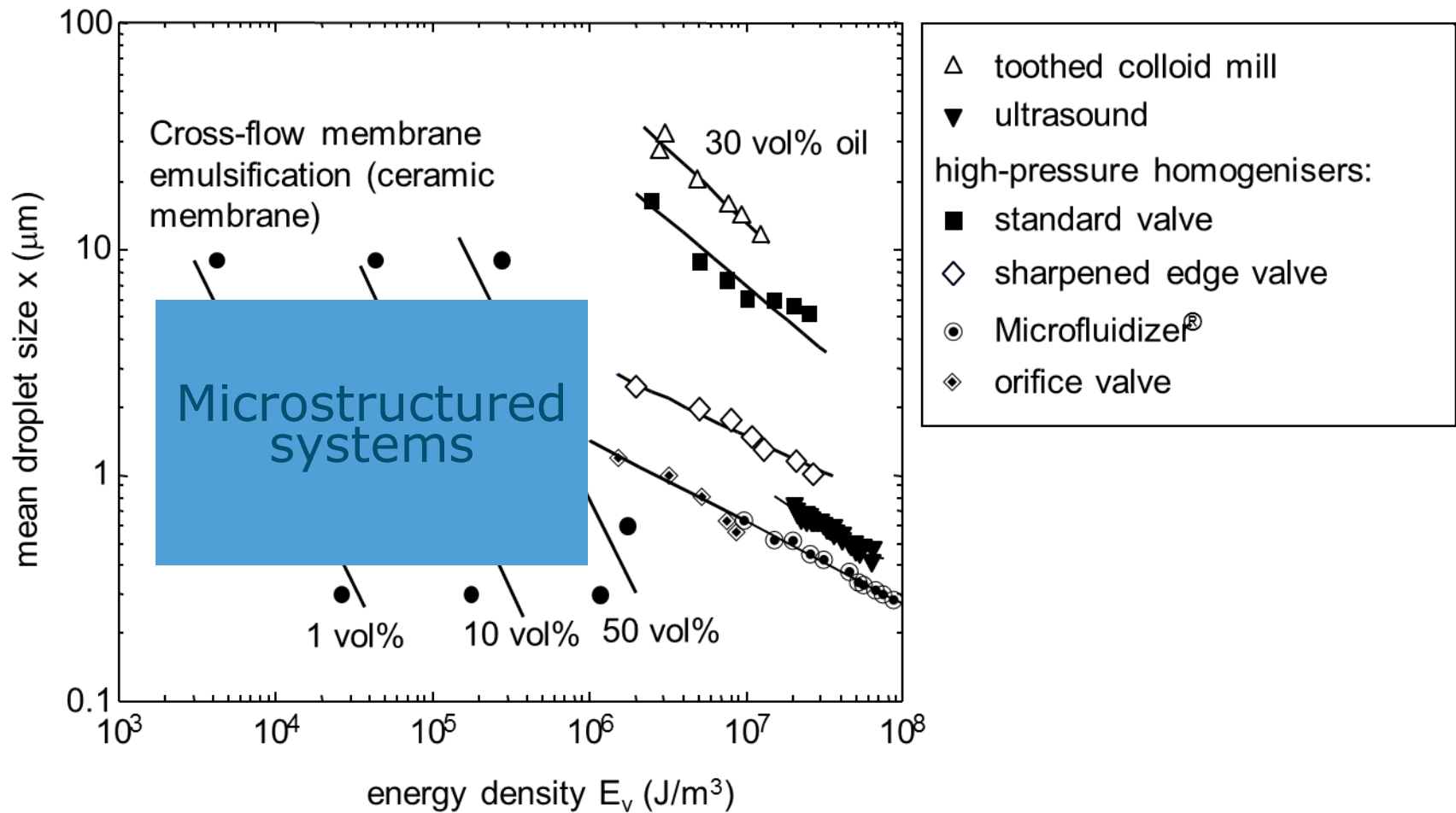
- Premix membrane emulsification
- Cross-flow (membrane or microchip)
- Spontaneous emulsification



Characteristics:

Low energy input, No temperature effects, Single pass

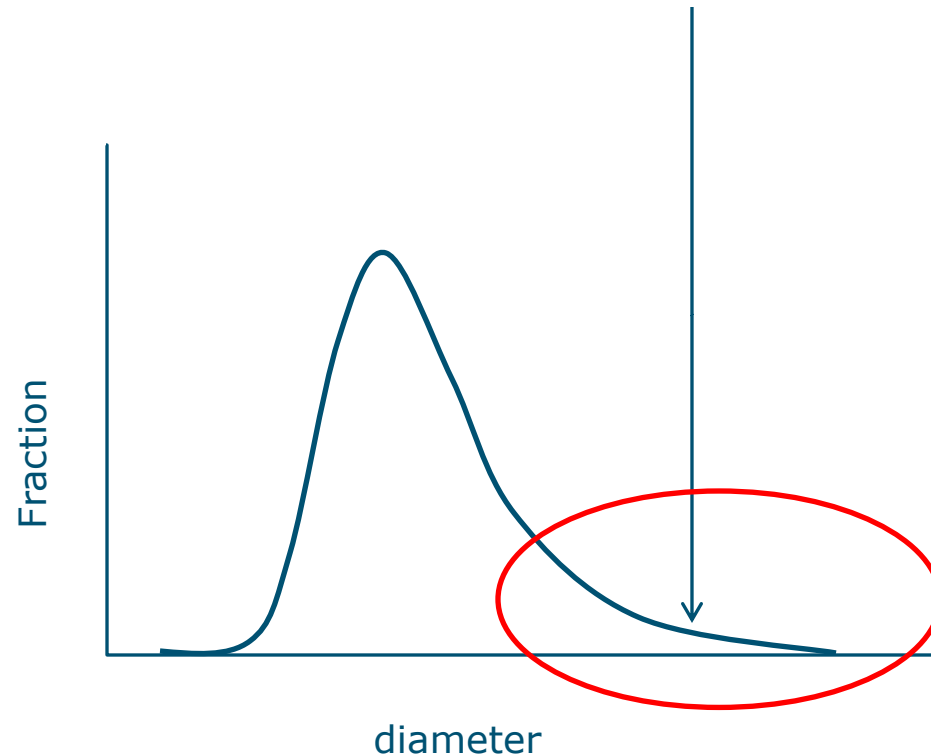
# Comparison of emulsification techniques



# 'Industrial' demands put on alternative technology

- High through-put
- Small particle size (narrow size distribution)

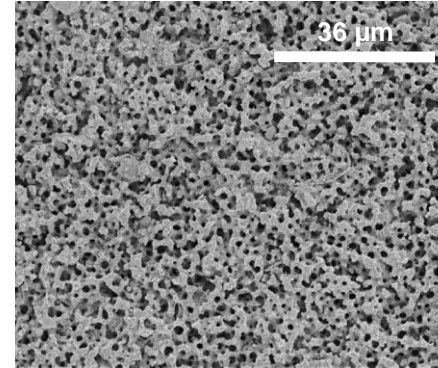
- Cheap!





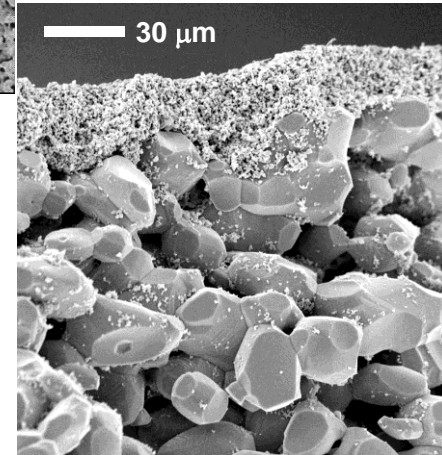
# Microstructures starting point: membranes

- Shirazu porous glass (SPG)

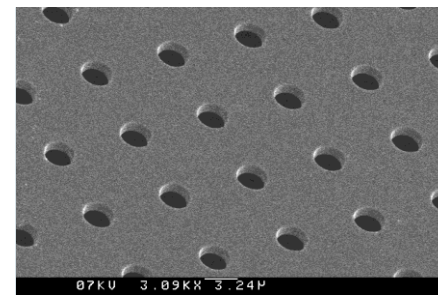


- Ceramics

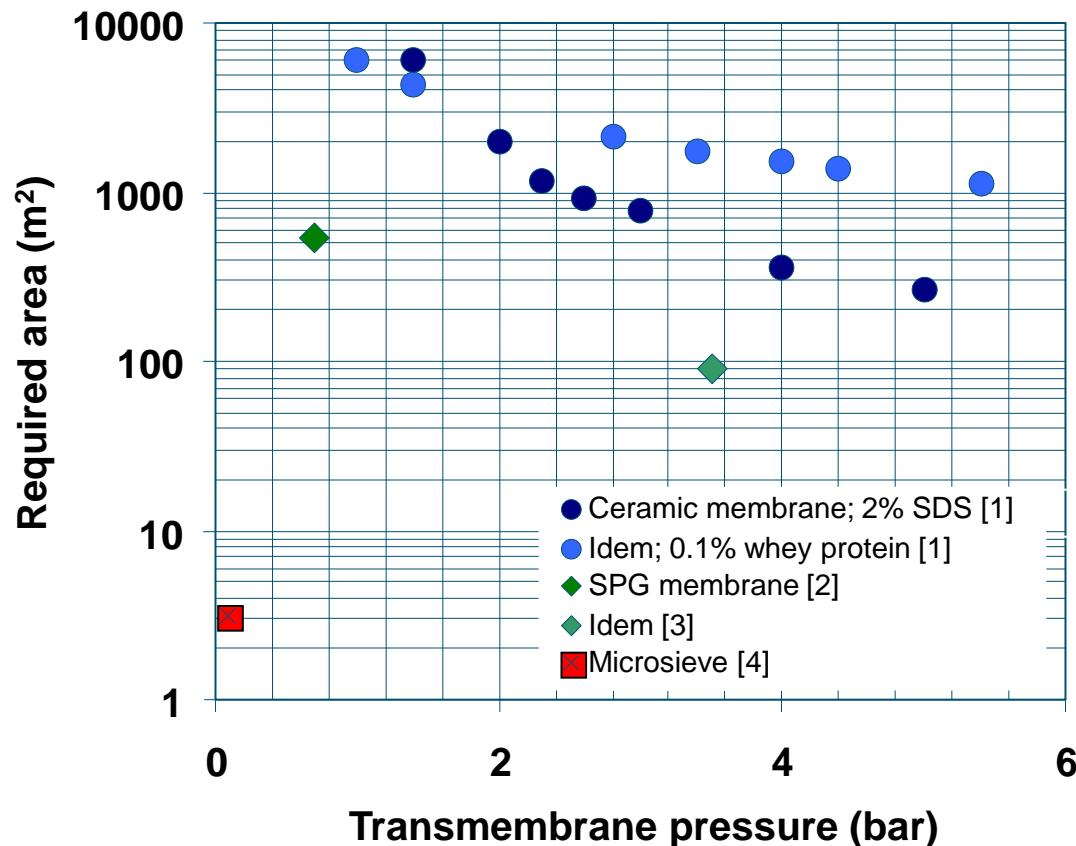
- Pore size distribution



- Microsieves

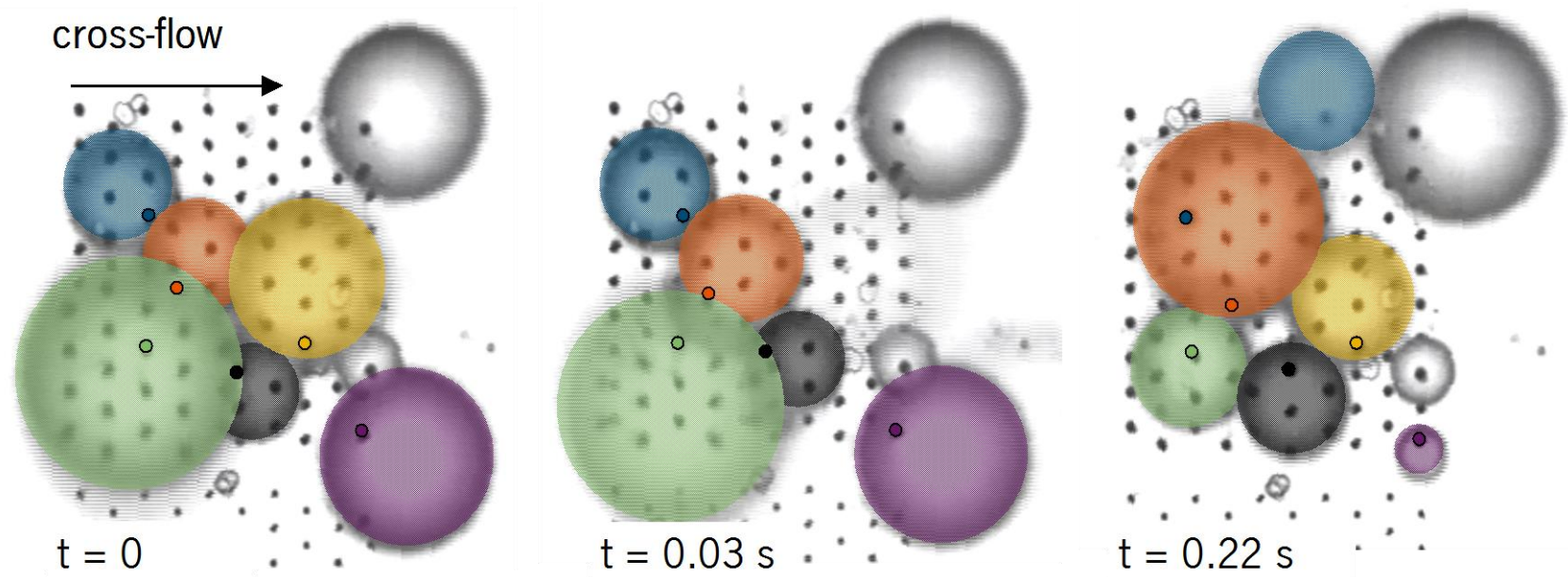


# Literature/experimental studies



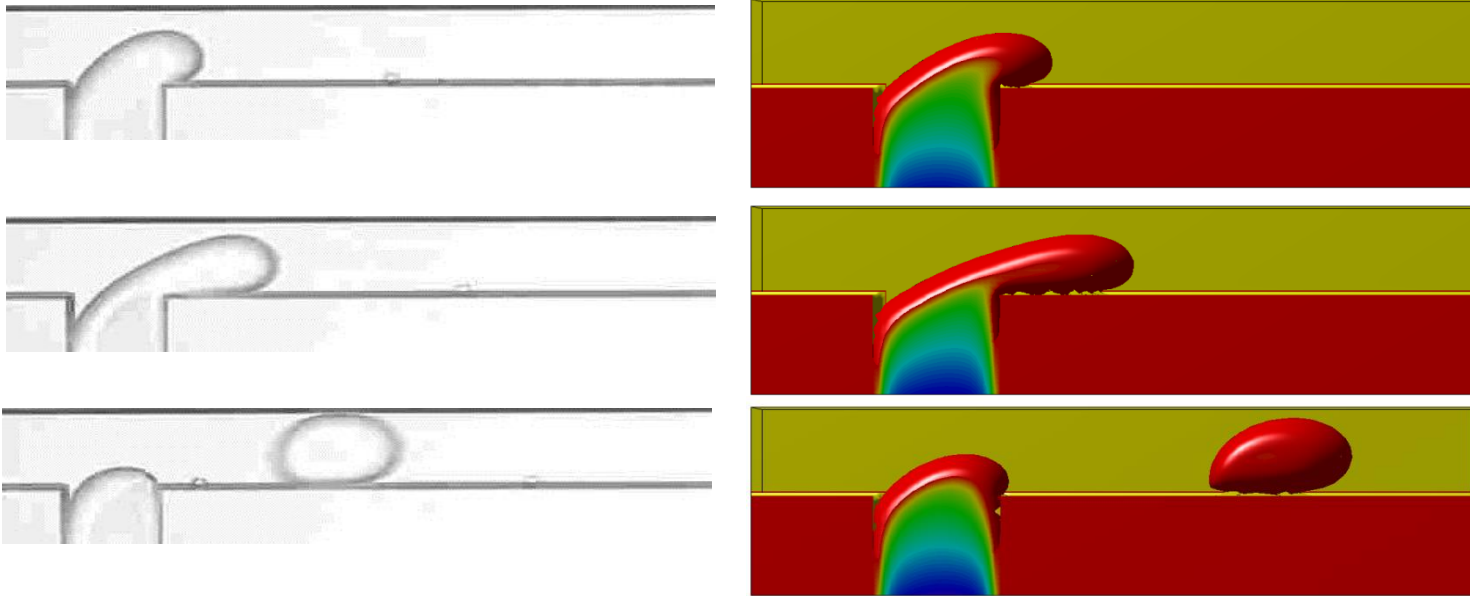
- Culinary cream
- 30% volume fraction fat
- Vegetable oil
- 20 m³/h

# Microsieves in practice: no monodispersity



- Highly porous membrane: steric interaction (exaggerated because of low flow rates)
- Not all pores are active! Cross-talk.

# Cross-flow mechanism



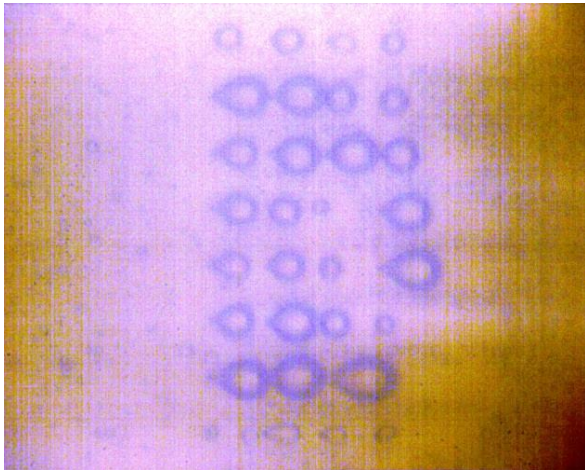
Scaling relation:

$$V_{\text{droplet}} = V_{\text{crit,ref}} Ca^{-0.75} + t_{\text{neck,ref}} Ca^{-0.75} \varphi_d \quad Ca = \frac{\gamma_{sh} r_h \eta}{\sigma}$$

Two steps: *Interfacial tension, viscosity, shear rate, design!*

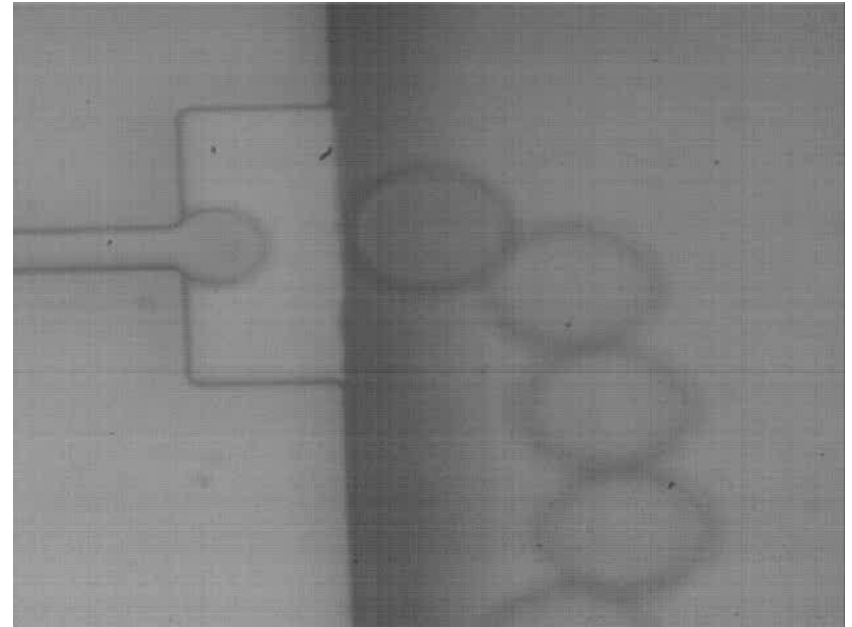
# Shear-based microfluidics

- Design of the sieves
  - thickness to prevent cross-talk
  - pore positioning
- Control on continuous phase + dispersed phase needed
- Pore size distribution!



*Difficult to control droplet size!*

# Spontaneous droplet generation

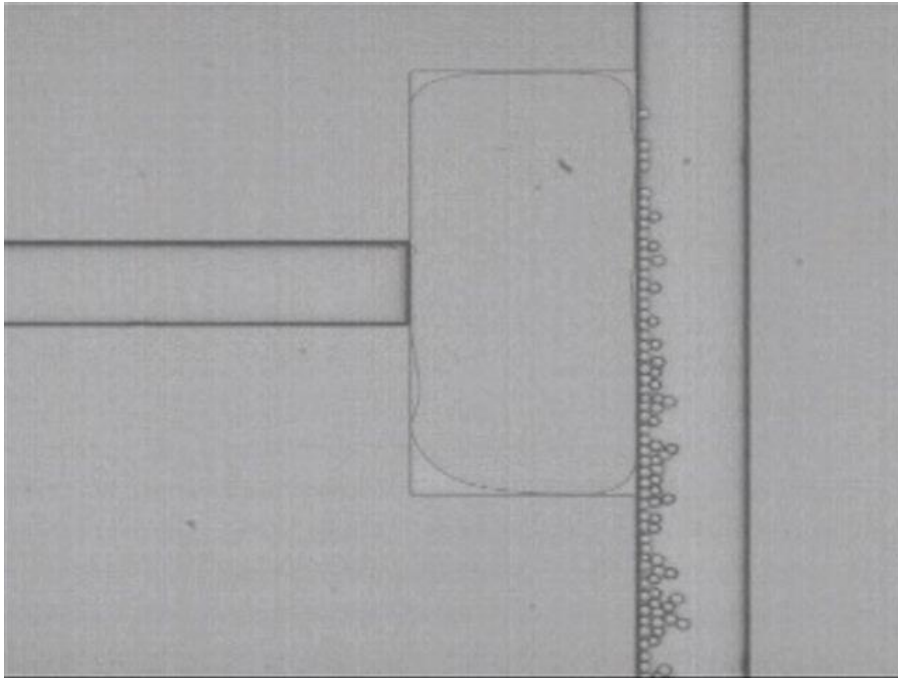


Laplace pressure differences  
Dispersed phase, terrace design

*Up-scaling?*

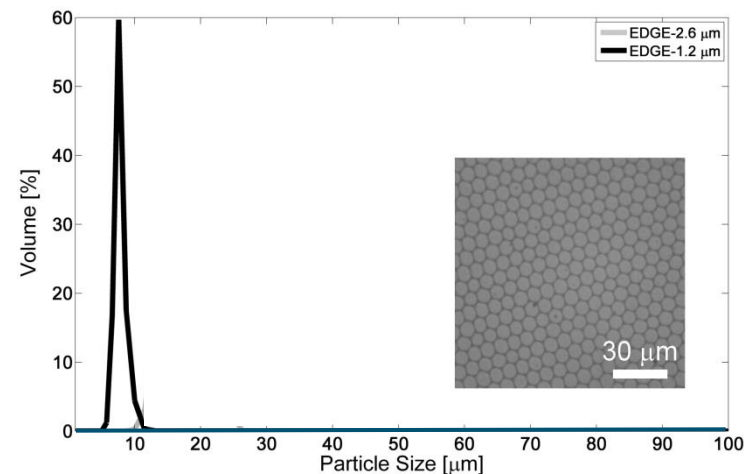
# Edge-based Droplet Generation (EDGE)

Simultaneous formation of droplets from **one** microstructure



slowed down 100x

Droplet  $\sim 6-8 \times$  plateau depth  
Dispersed phase flow

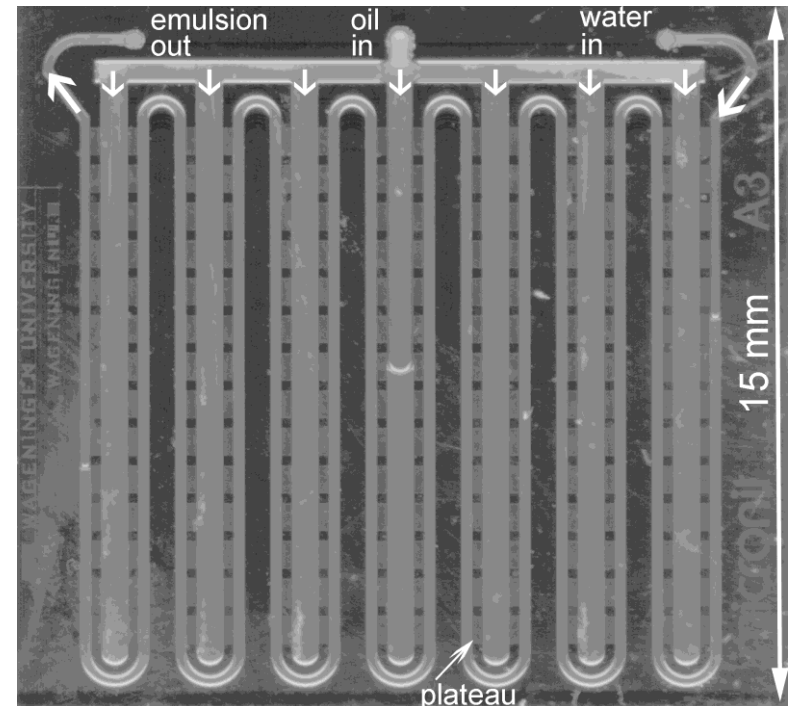
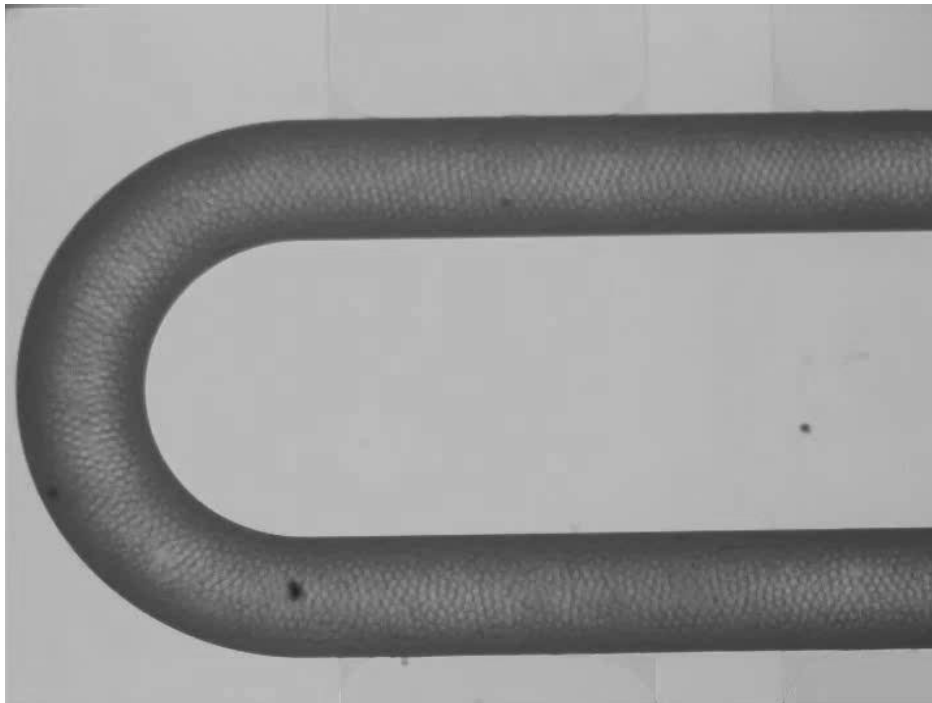




# 'Up scaled' EDGE system

- ✓ Small droplets
- ✓ All plateaus fill
- ✓ Food ingredients
- Scaling: single chip

*Simple & stable!*



*Productivity?*

# Pre-mix emulsification

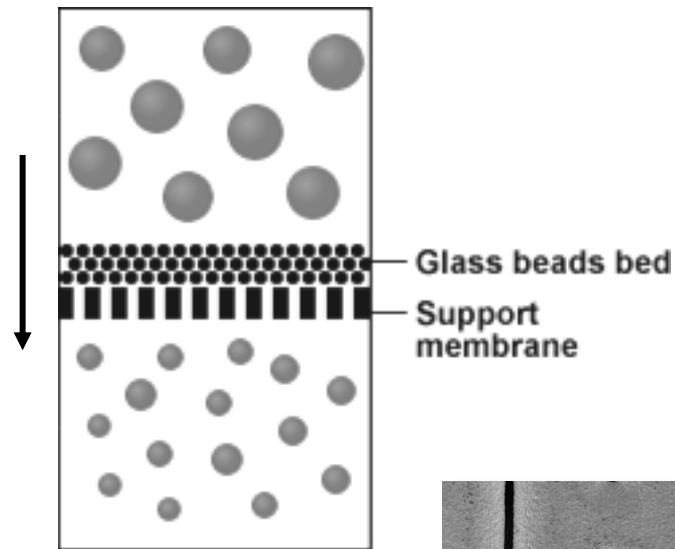
- Regular membranes: fouling → flux loss

## ➤ Dynamic membrane

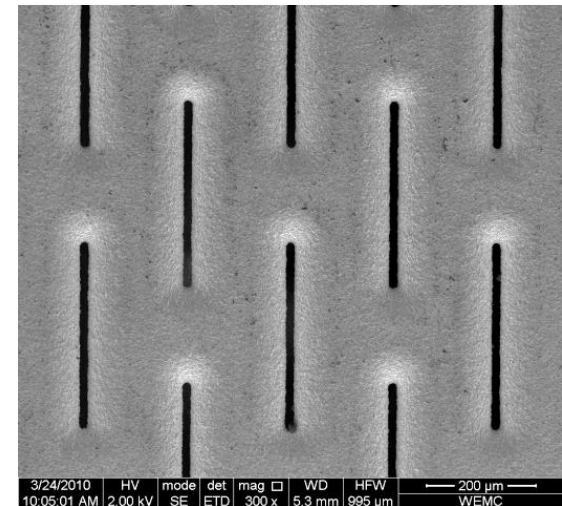
Break-up bed

Clean

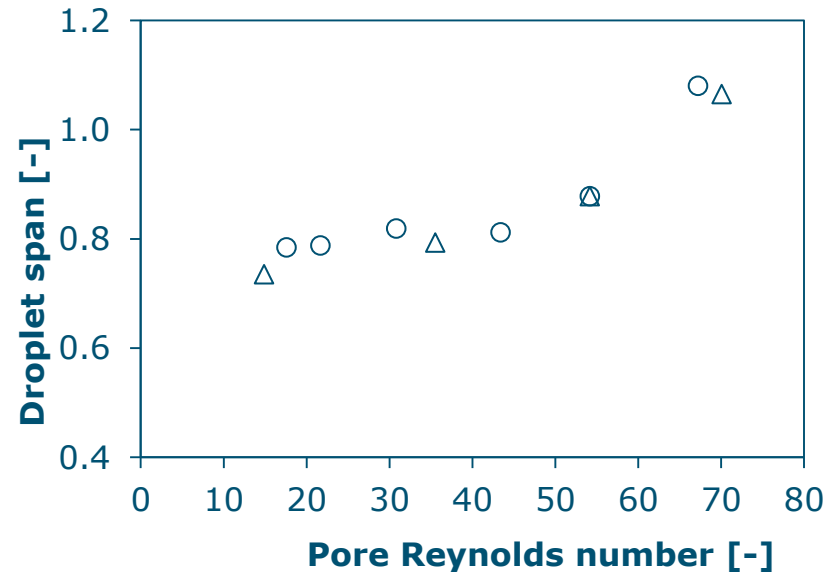
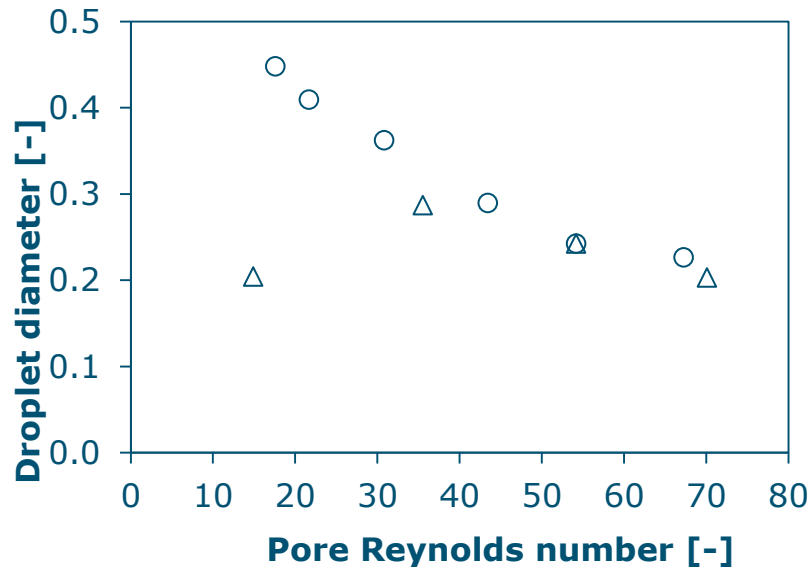
Use again



Support metal sieve (width  $\sim 10$  micron)



# Characterisation



← Decreasing  $d_p$   
← Increasing  $H_{bed}$

Fluxes: 100-800 m<sup>3</sup>/m<sup>2</sup>/h

Reasonable monodispersity

'Droplets < voids'

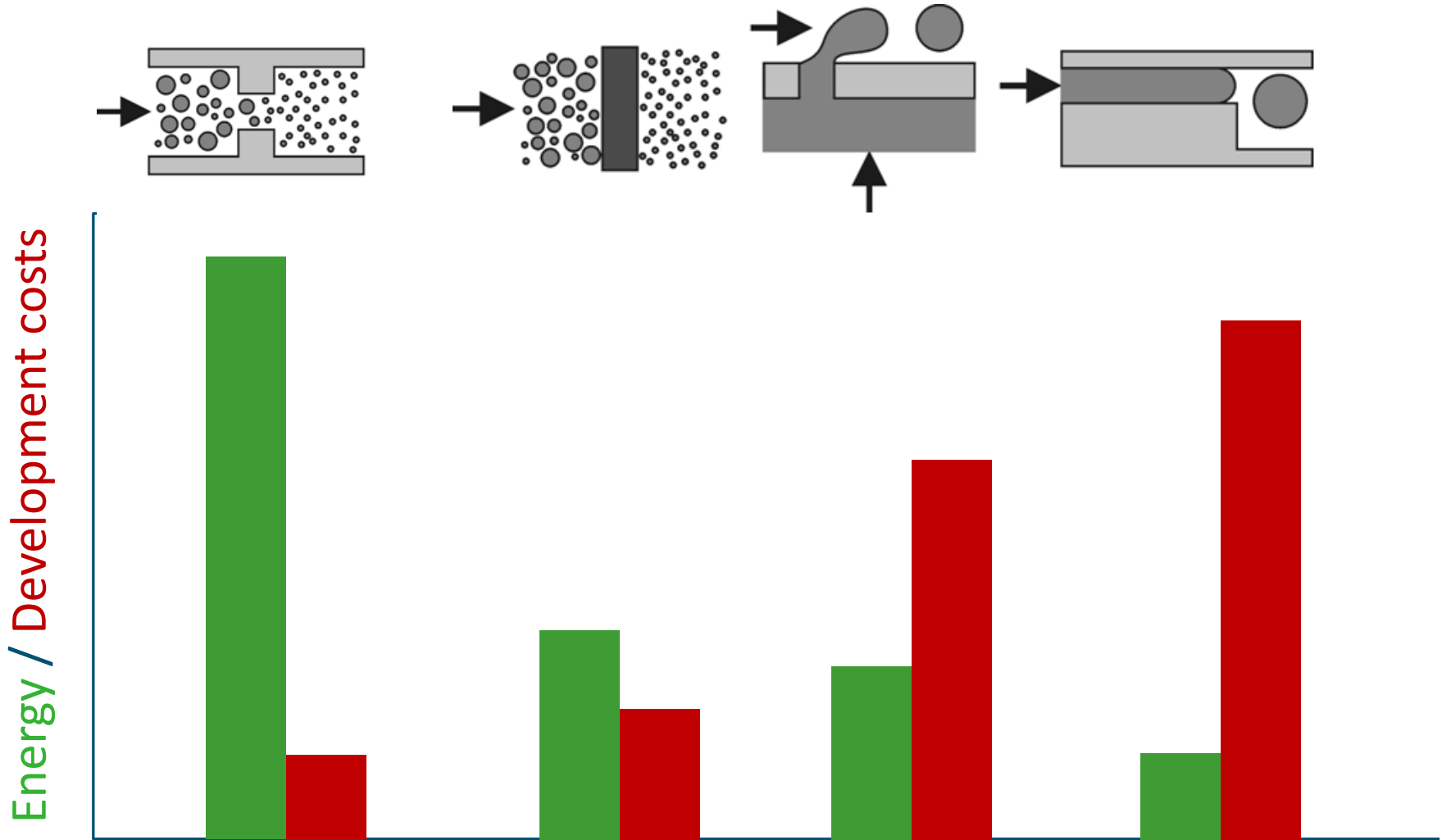
High volume fractions are possible

# Comparison characteristics microsystems

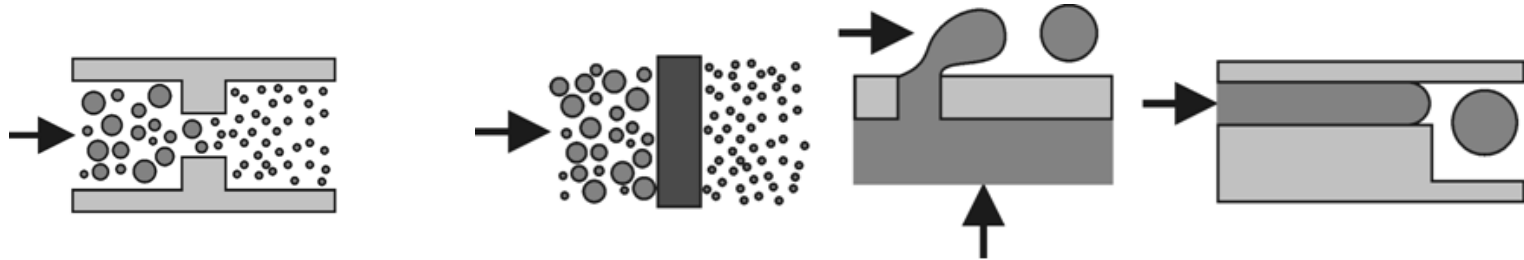
	<i>Shear based</i>	<i>Spontaneous</i>	<i>Hybrid systems</i>
<i>High through-put</i>	reasonable	no(t yet)	yes!
<i>Small particle size /</i>	$d_d > d_p$	$d_d = 6h_p$	$d_d < d_p$
<i>Narrow size distribution</i>	reasonable	yes	reasonable

For 1 micro droplets, design reaches limits of microfluidics resolution shear / spontaneous

# Comparison with large scale demands



# Which applications?



Various oil  
fractions

Various oil  
fractions

Max 10%

Various oil  
fractions

Shear /  
temperature  
sensitive  
ingredients

Shear /  
temperature  
sensitive  
ingredients

Shear /  
temperature  
sensitive  
ingredients

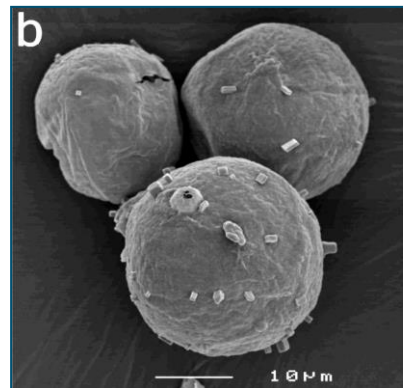
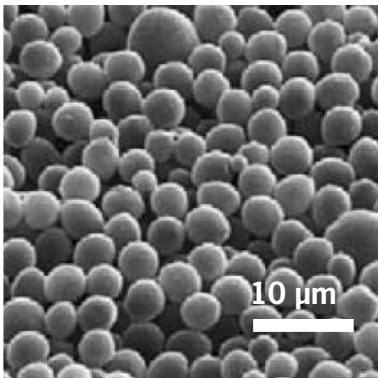
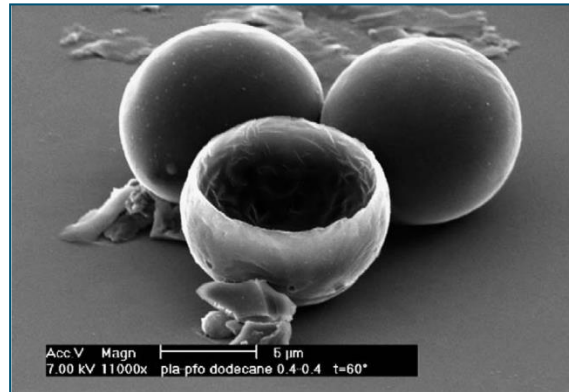
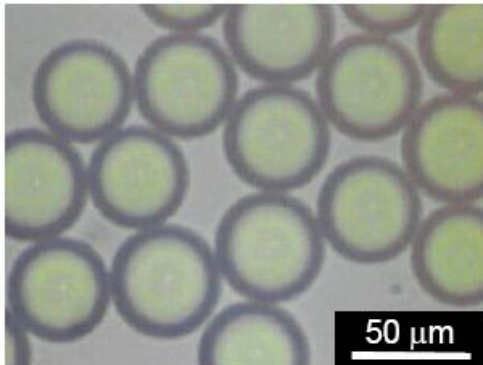
Specialty / bulk  
product

Specialty /  
bulk product

Specialty  
product

Specialty  
product

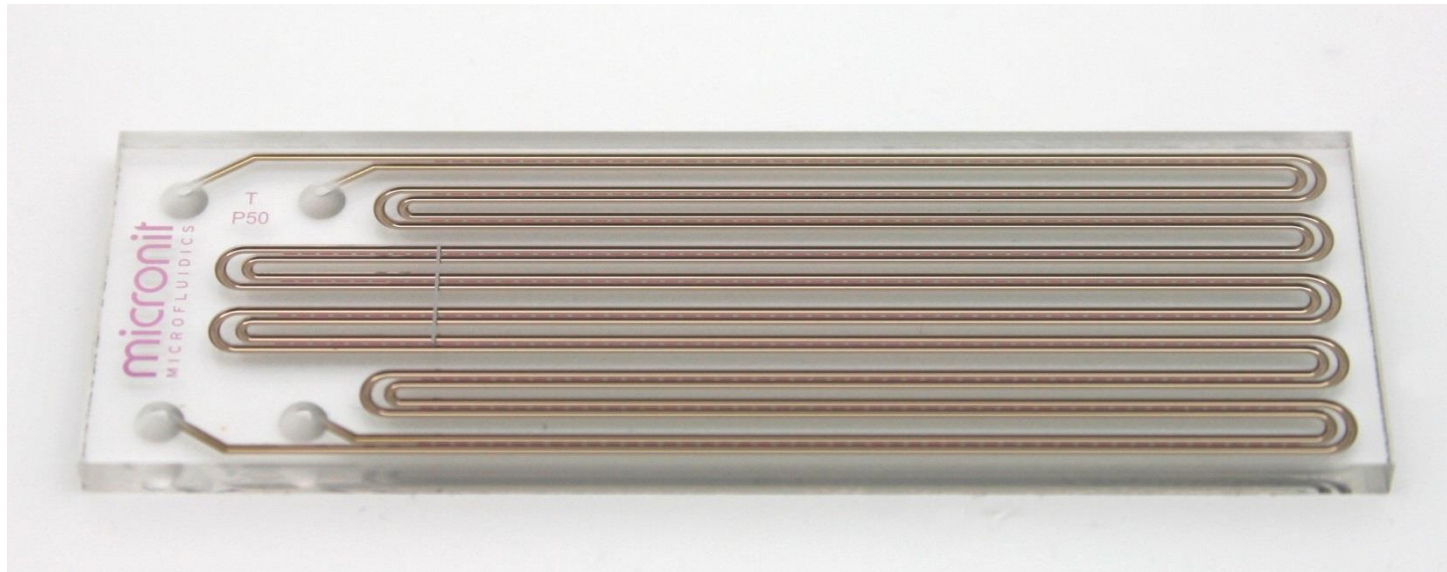
# Beyond emulsions and beyond food





# Remaining industrial questions

- Construction materials: metal ideally steel
  - Roughness
  - Wettability (modification)
  - Construction limitations in microfluidics



# Conclusions:

## New emulsification techniques:

- Membranes
- Microfluidics (up-scaling)
- ✓ Hybrid systems (flux, energy & droplet size)

## Construction material:

- Metal (+ modification)

## Combination with modelling

On-going: various products + tools!

