



Multiple emulsions for confectionery fillings

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Dept. of Food Technology

closed since October 2007





Agenda

- 1. What are multiple emulsions?**
- 2. Preparation methods**
- 3. Influences on emulsion stability**
- 4. Influences on droplet sizes**
- 5. Emulsions with high sugar content**
- 6. Summary**



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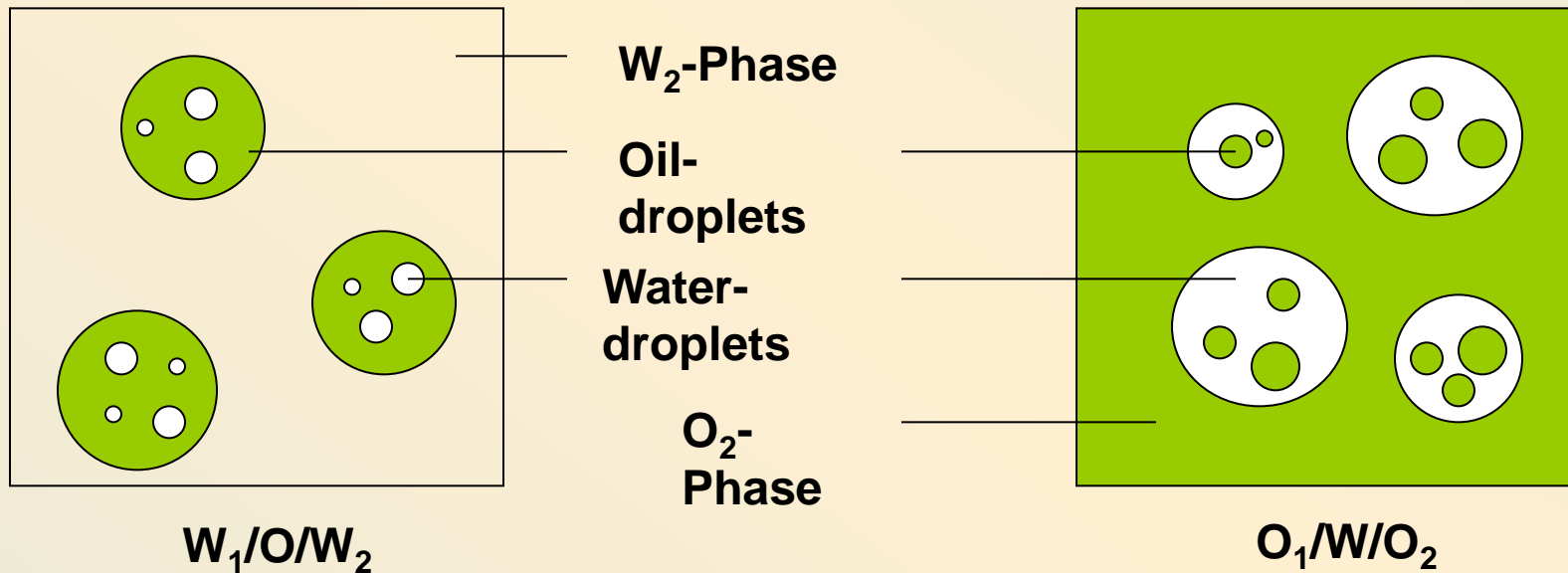
What are multiple emulsions?



Multiple emulsions

Definition:

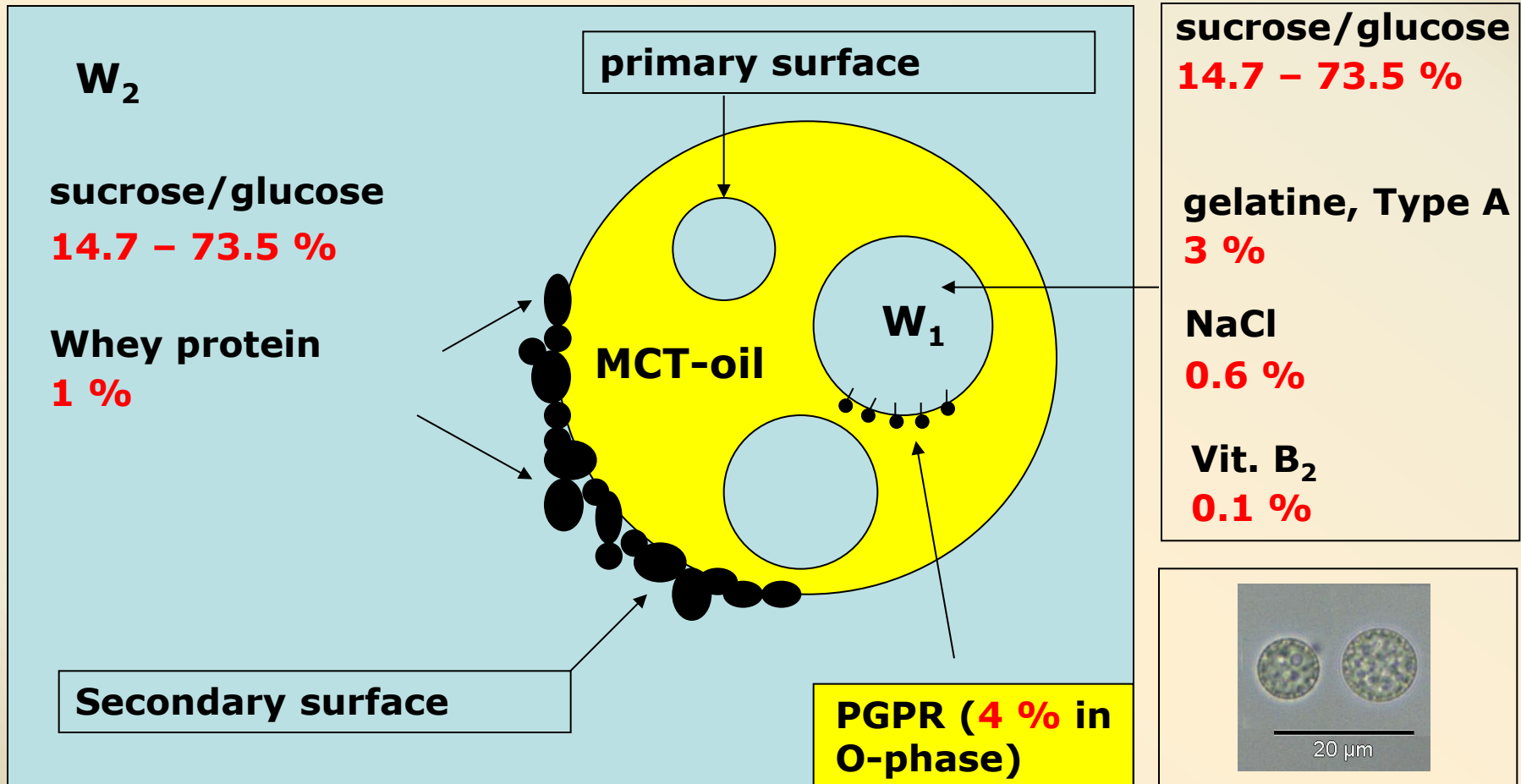
Droplets of a dispersed phase (O or W) contain small droplets of another phase



Special types: O/W/O/W/O and W/O/W/O/W



Structure of confectionery W/O/W





Research questions

- **How can confectionery W/O/W emulsions be prepared?**
- **How influence emulsification conditions the droplet sizes?**
- **Which sugar content (water activity) can be attained in W/O/W emulsions?**
- **What are the encapsulation properties for bioactive substances (e.g. Vit. B₂)?**



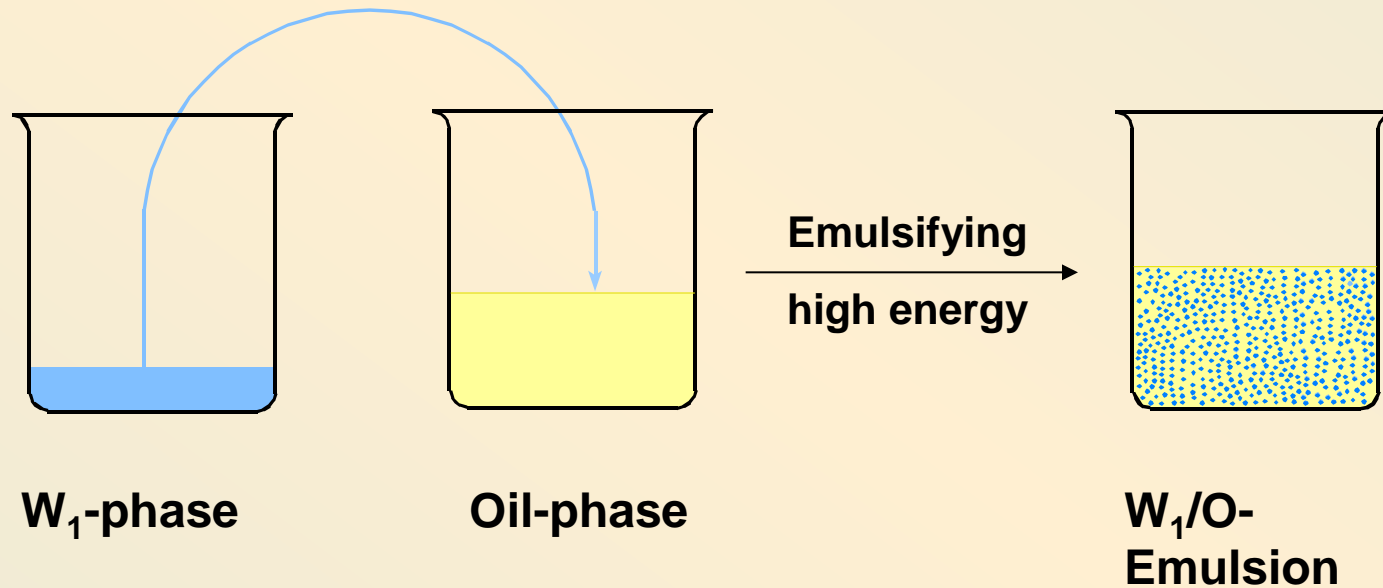
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Methods of emulsion preparation



Principle of formation multiple emulsions

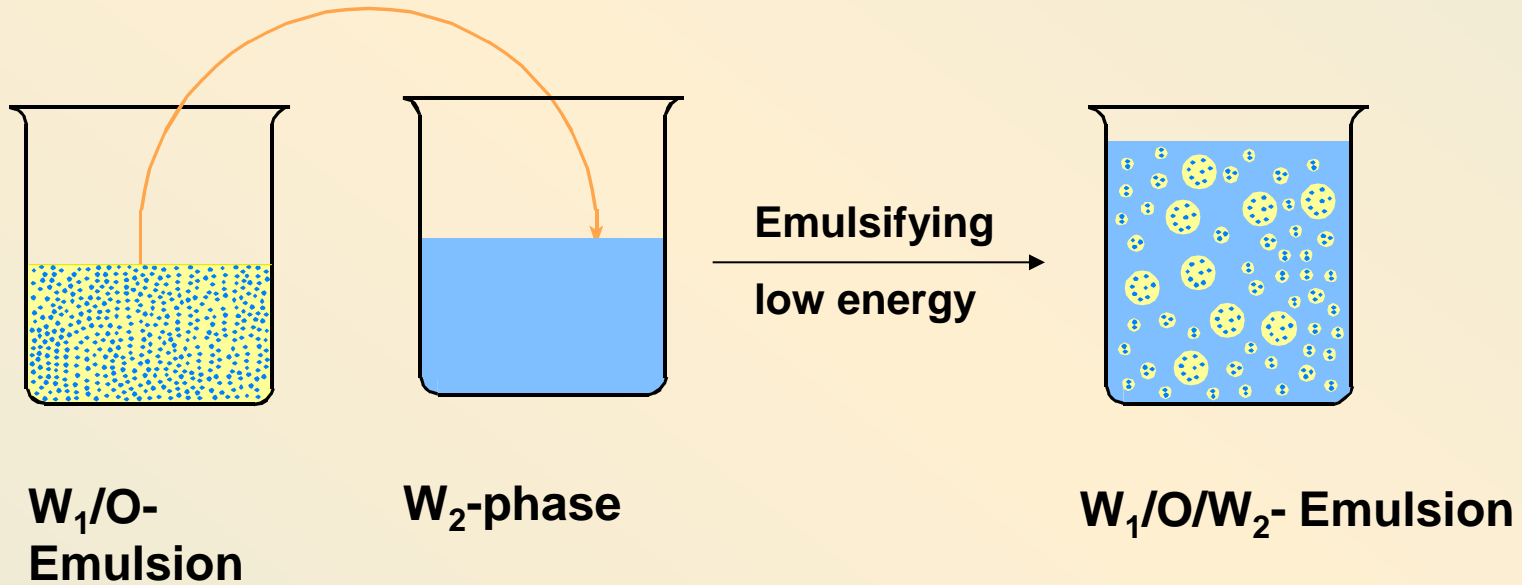
1. W_1/O - Emulsion





Principle of multiple emulsion preparation

2. $W_1/O/W_2$ - Emulsion





Methods for emulsion preparation (emulsification of W_1/O in W_2)

Prevention of:

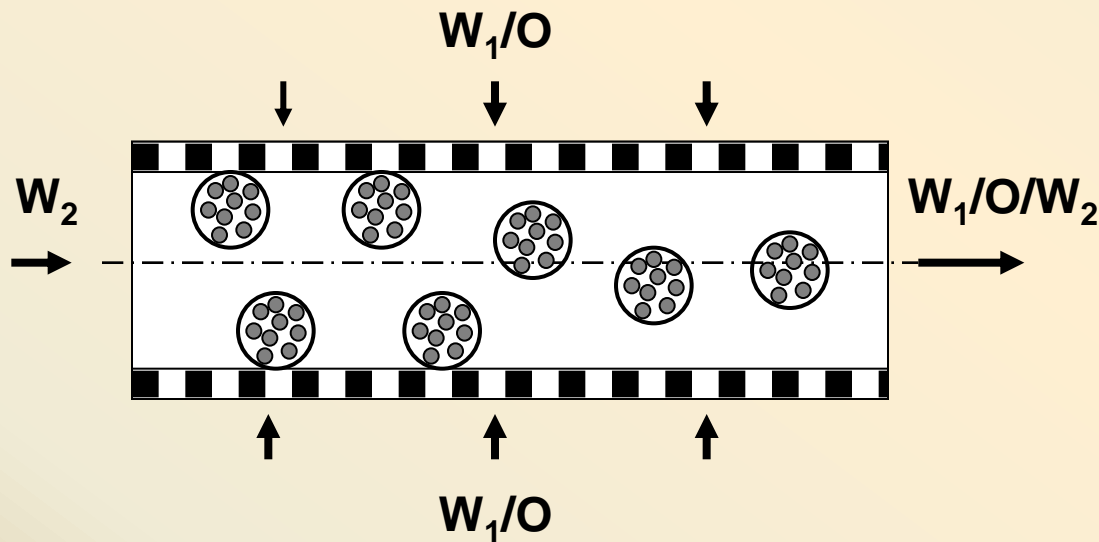
Disruption of internal emulsion droplets (W_1) and coalescence with the external phase (W_2)

Preferable emulsification methods:

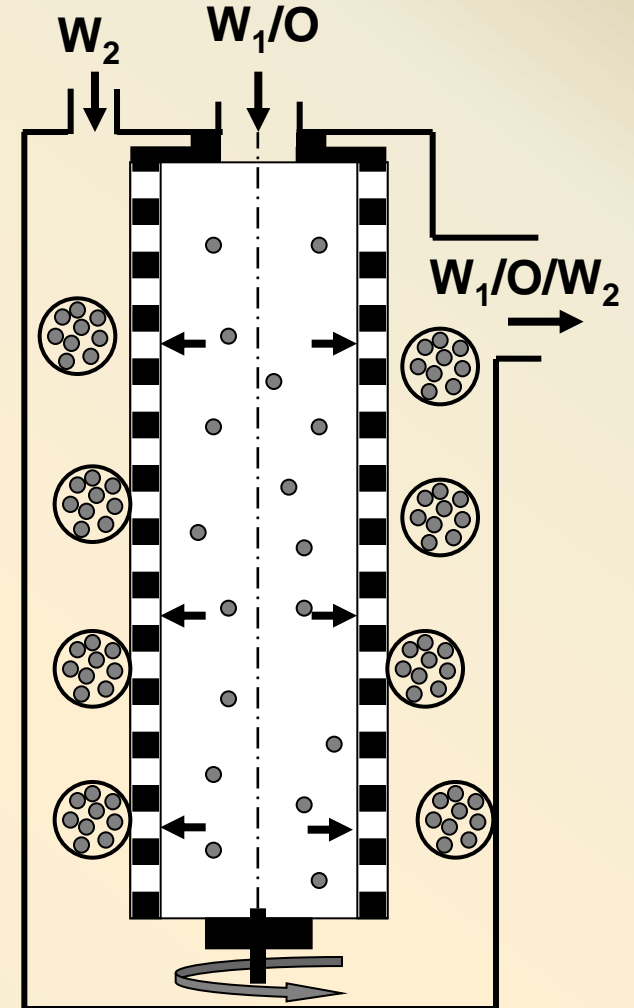
- Membrane emulsification
- Combi-orifice-homogenizer
(modified low pressure emulsification)



Membrane emulsification



Fixed membran tube



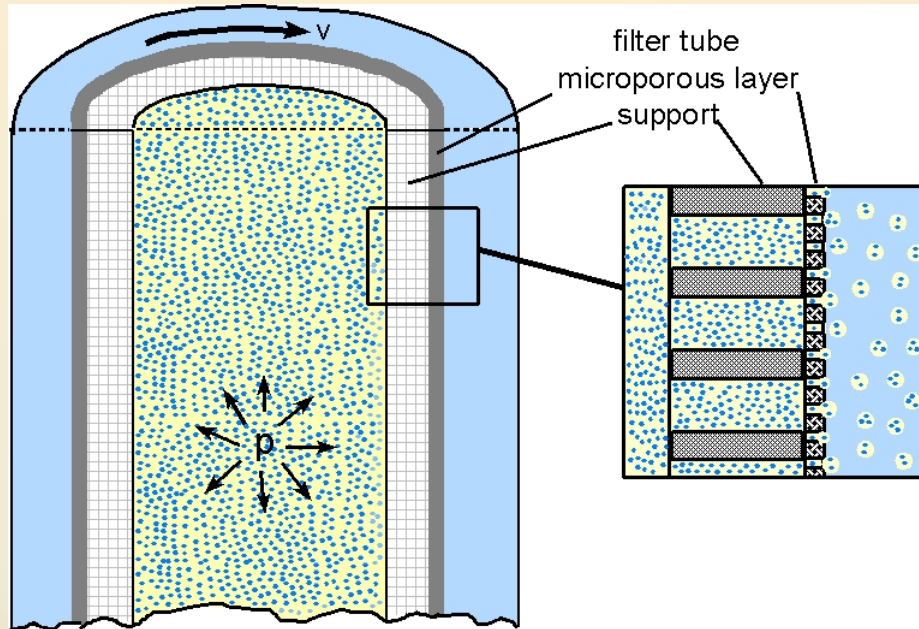
Roating membrane tube



Pilot plant for rotating membrane emulsification (DIL e.V.)

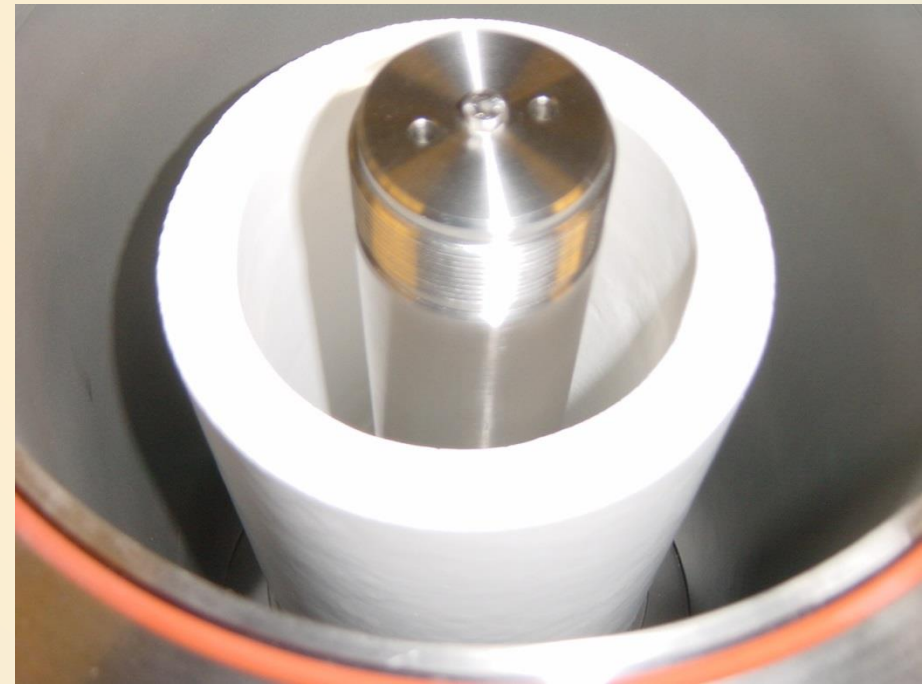
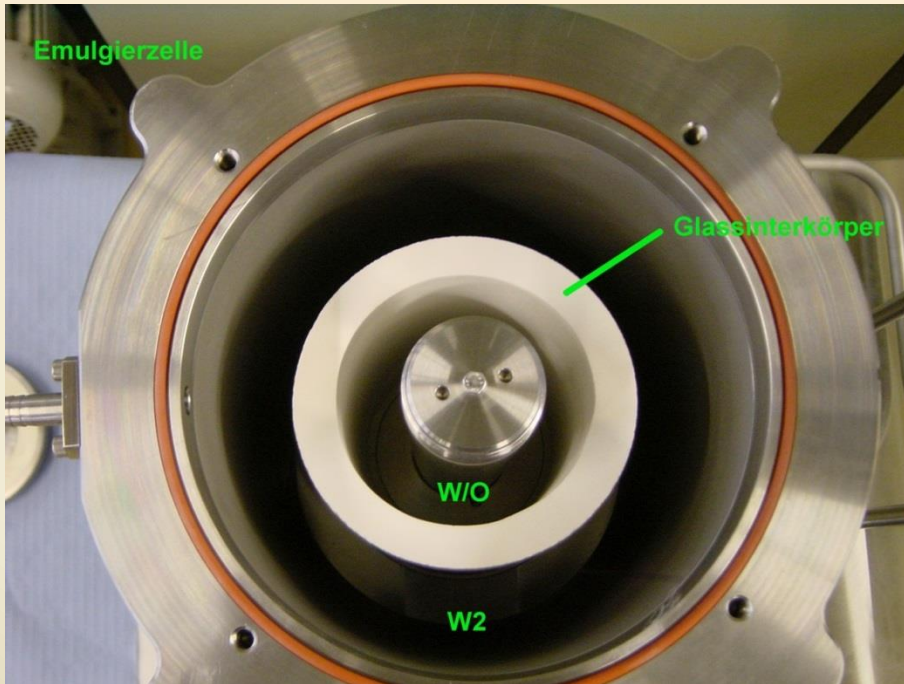
Principle of emulsification:

Rotation of sintered glass support with microporous layer outside



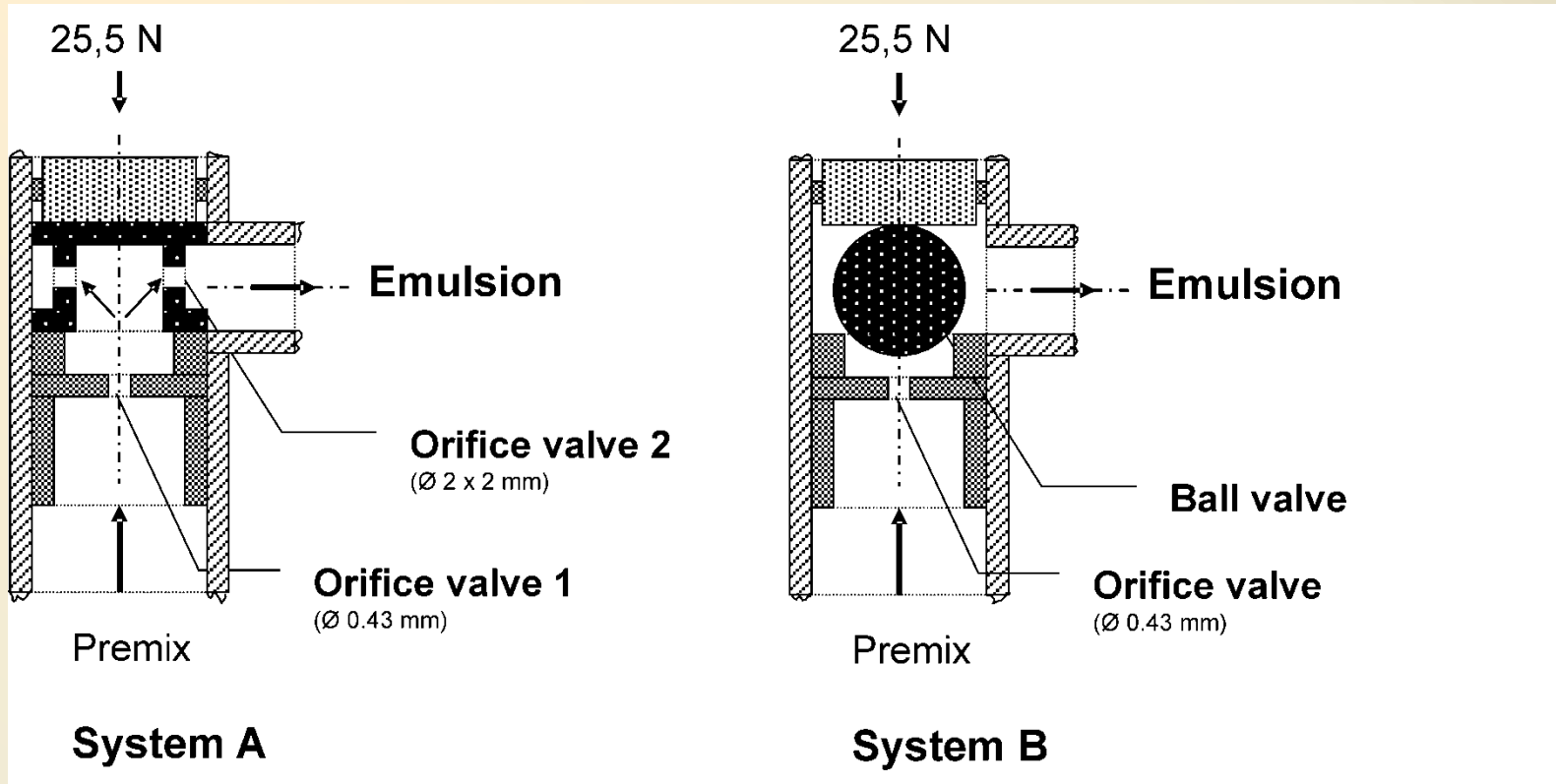
DE 103 06 259 A1, 2003

Pilot plant for rotating membrane emulsification (DIL e.V.)



Modified low pressure homogenizer

Muschiolik et al., 1995



„Combi orifice system“

Premix
W/O/W
0.5-1.0 MPa

„Orifice-ball-system“

Premix W/O
7 – 8 MPa



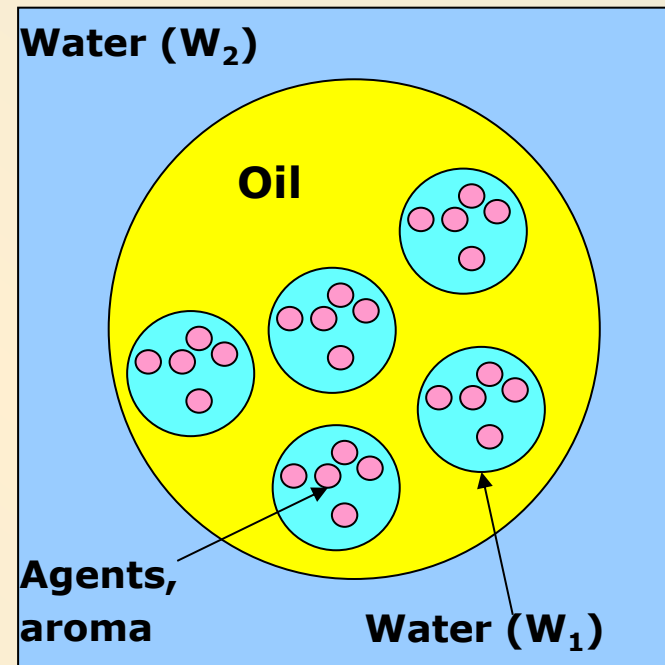
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Influences on emulsion stability



Stability of W/O/W is influenced by:

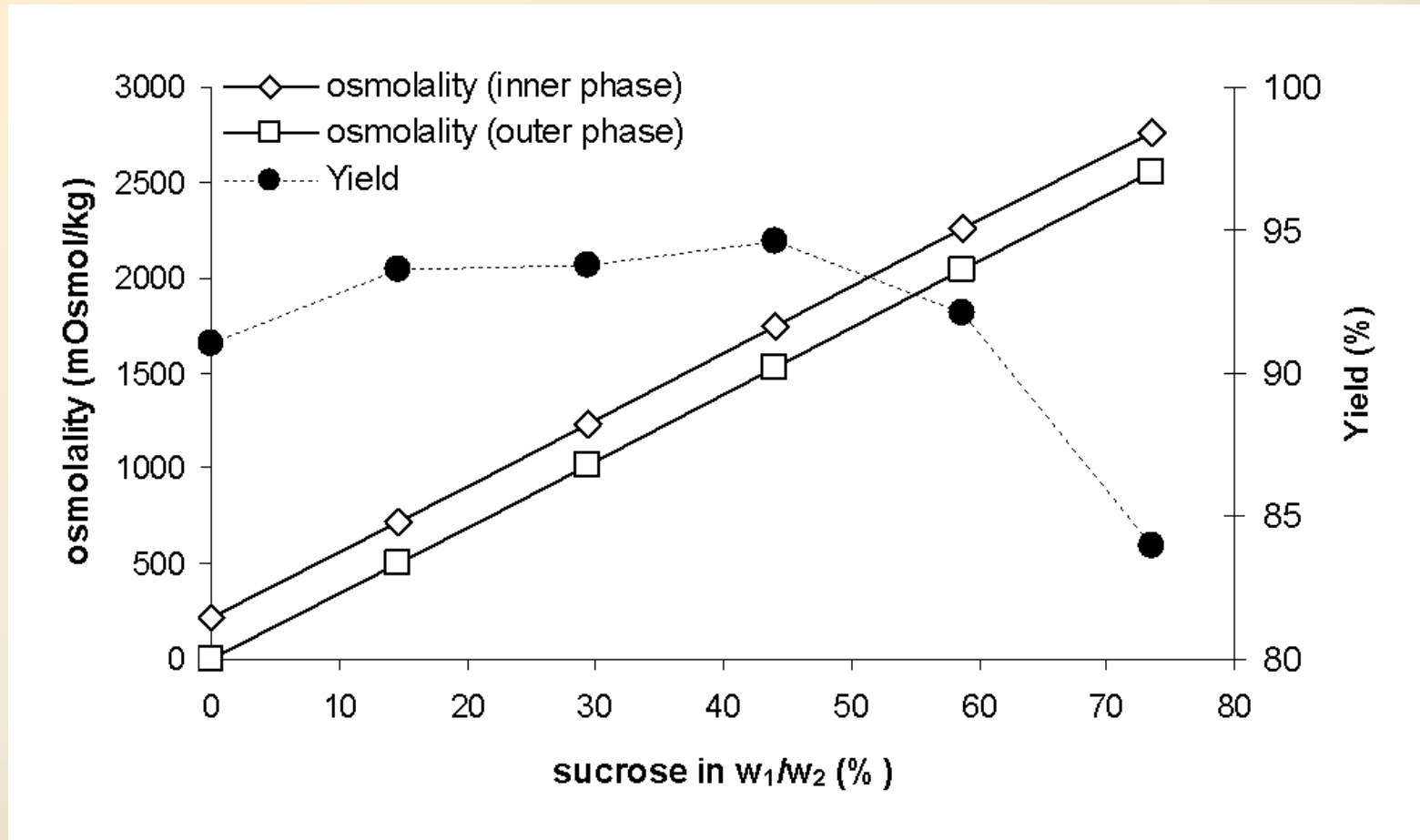
- Size of W_1 and W_1 -stability
- Size of O and O-stability
- **Osmotic gradient between W_1 and W_2**
- Water flow between W_1 and W_2 (influenced by osmotic gradient)
- Viscosity of the emulsion phases





Influence of sugar content on osmotic gradient between the aqueous phases

Yield: encapsulation of Vitamin B₂



The osmotic gradient between W_1 and W_2 should be constant at different sucrose contents (osmolality in the W_1 phase should be higher)



Stability of W/O/W is influenced by:

Process conditions:

- Shear force and emulsification pressure (high for W/O, low for W/O in W)
- Temperature (important for phospholipids*)
- pH (important for phospholipids*)

*as W/O-emulsifier

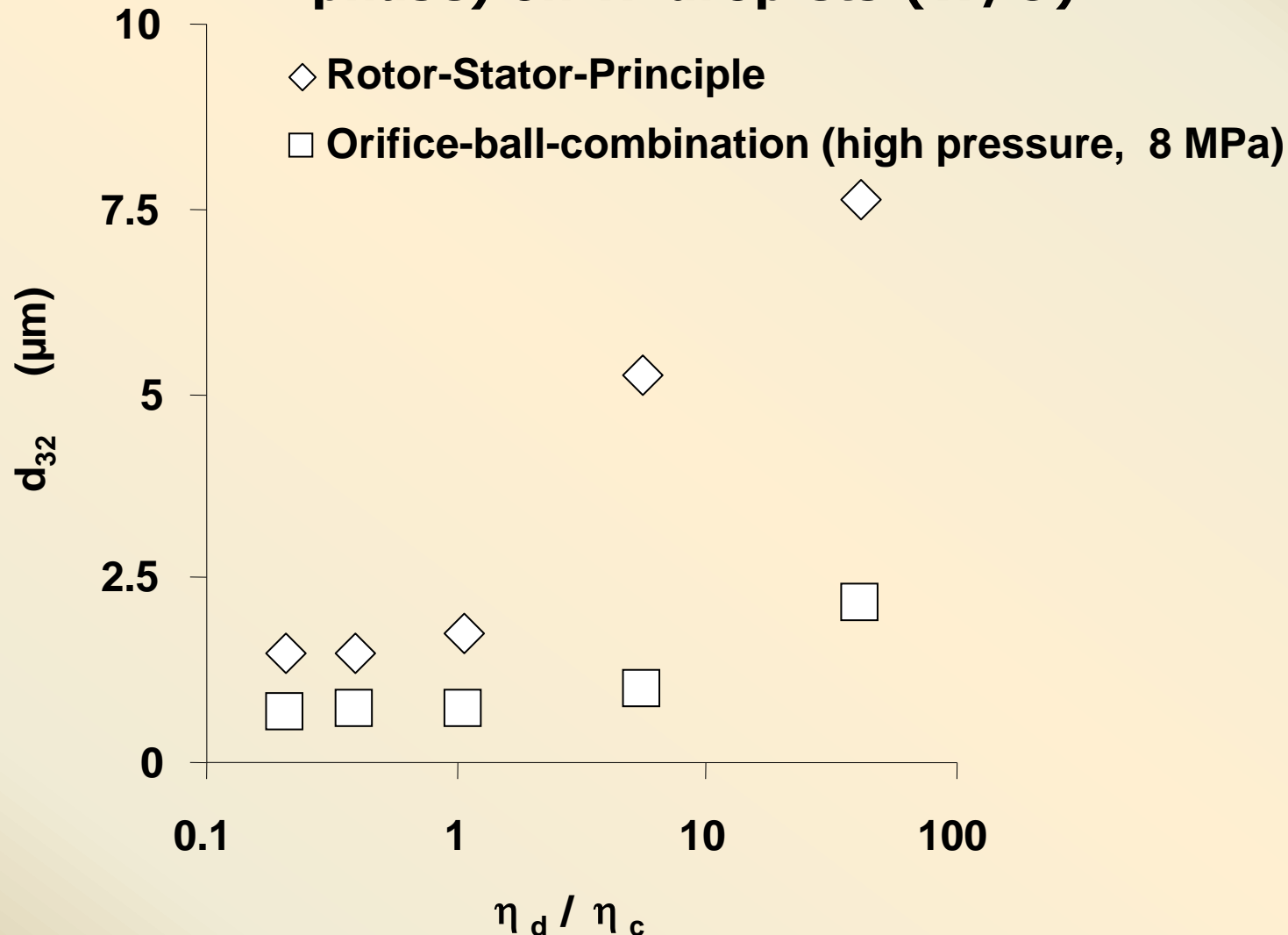


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Influences on droplet sizes



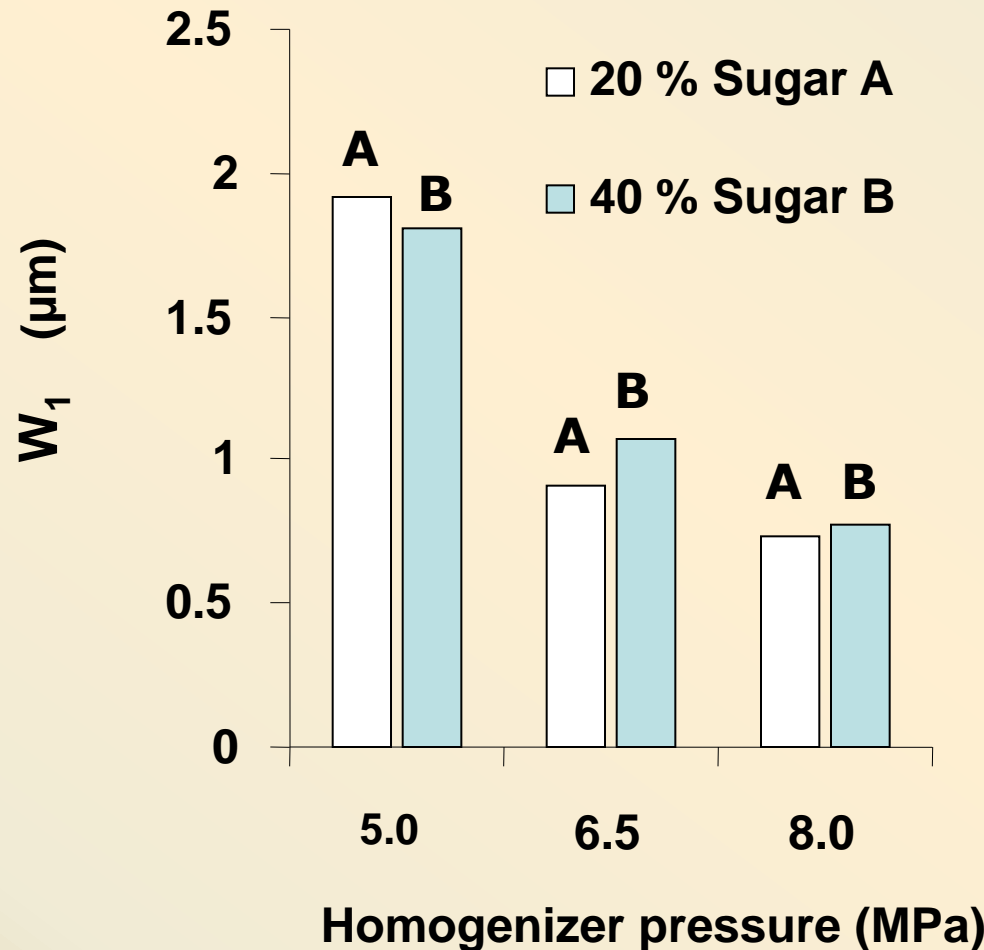
Influence of viscosity relation (disperse/continuous phase) on W-droplets (W/O)



A special emulsification system is necessary to get fine W-particles with a high sugar content



Influence of homogenizer pressure (orifice-ball-combination) on W-particles



With the „orifice-ball-homogenizer“ can be prepared fine W/O emulsions



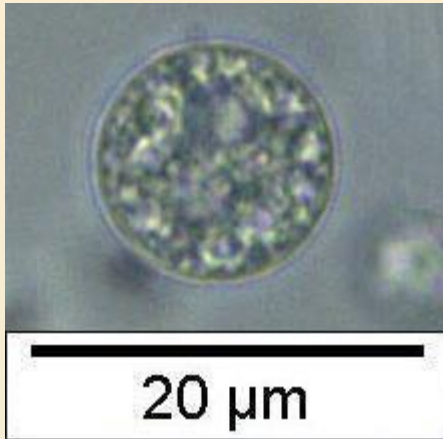
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Emulsions with high sugar content

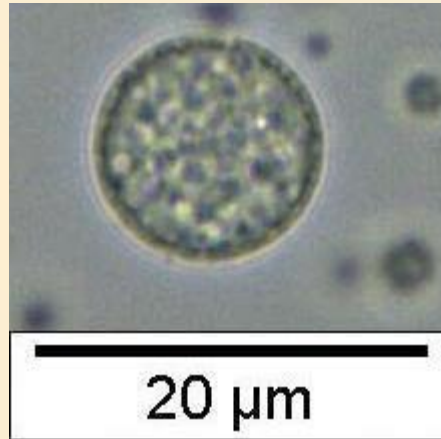


Images of sugar containing multiple emulsions (O-droplets), diluted

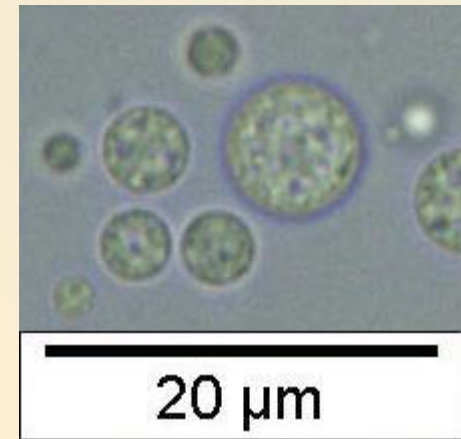
0 % Sucrose



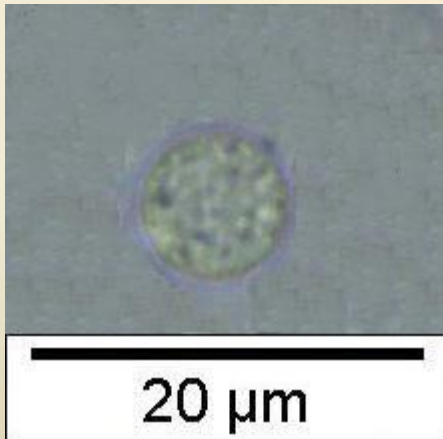
10 % Sucrose



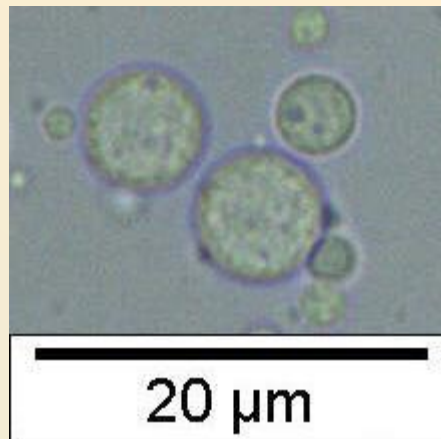
20 % Sucrose



30 % Sucrose



40 % Sucrose

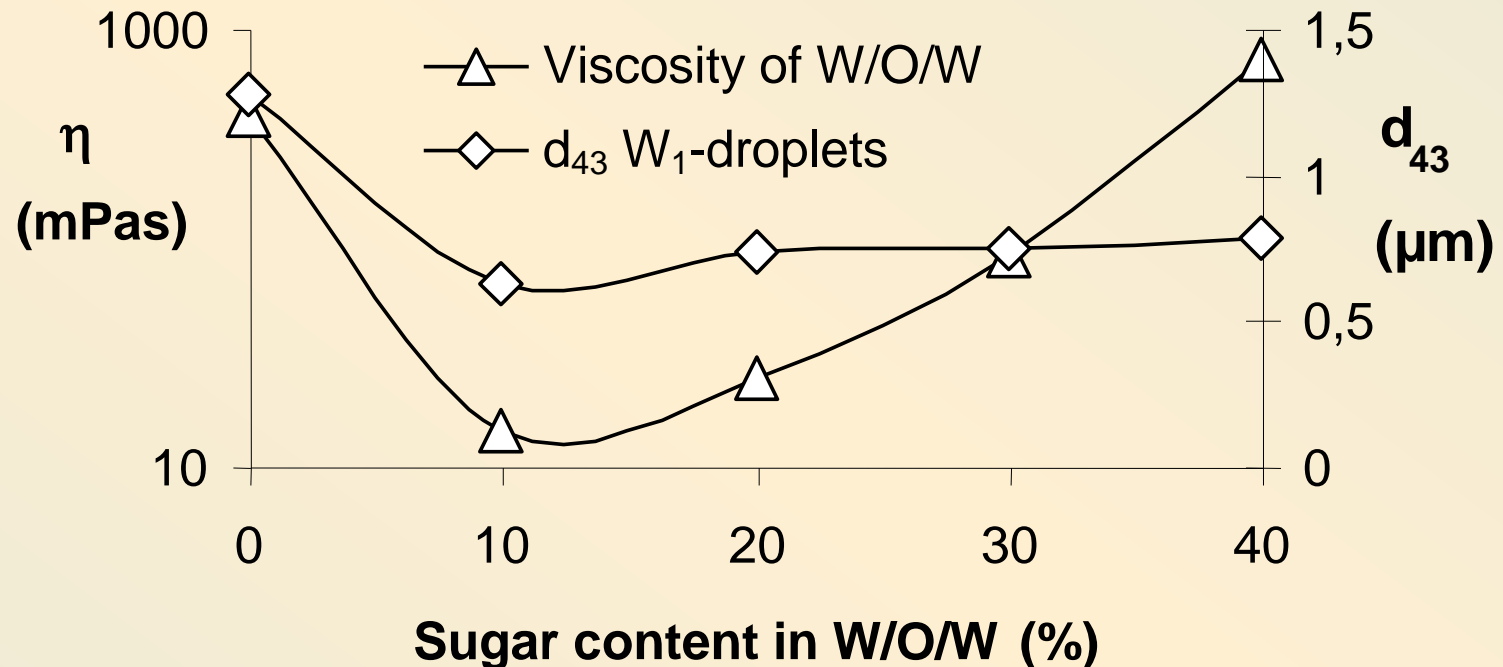


> 50 % Sucrose

**Refractive index
of oil and
sugar solution
get similar**



Influence of sugar on W_1 -droplets and W/O/W viscosity (100 s^{-1} , $50 \text{ }^\circ\text{C}$)

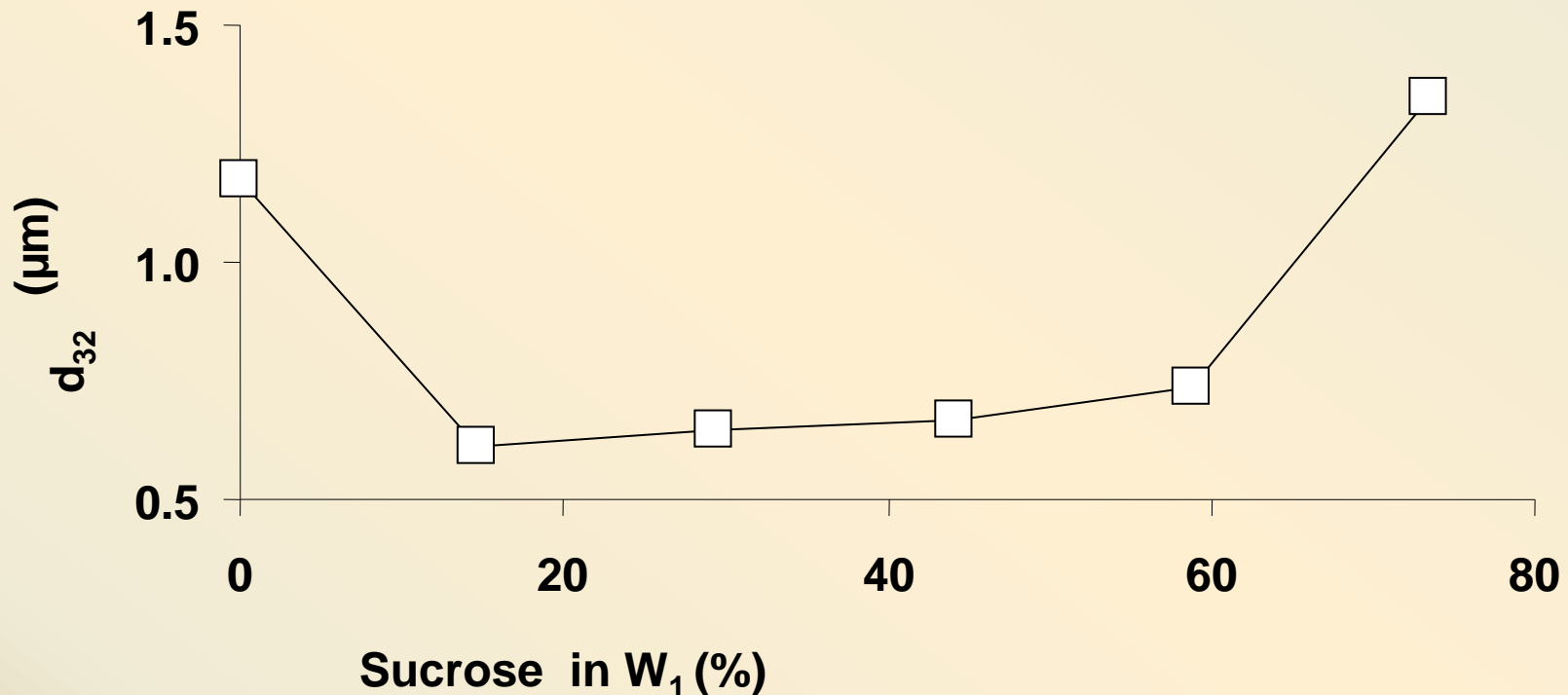


W_1 -droplet sizes decrease from 0 to 10 % sugar

The viscosity of W/O/W decreases from 0 to 10 % sugar and increases with higher sugar content



Influence of sugar content on W_1 -droplets at ~ 8 MPa

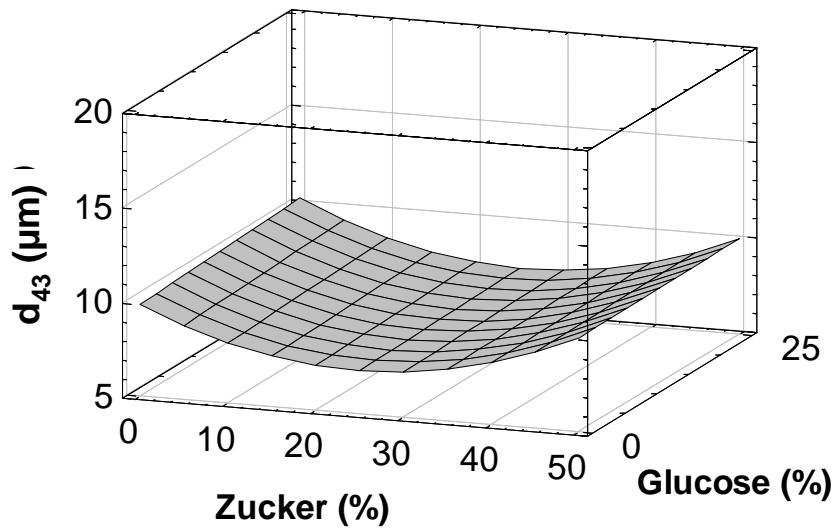


The W_1 -droplet size increases rapidly with more than 50 % sucrose in W



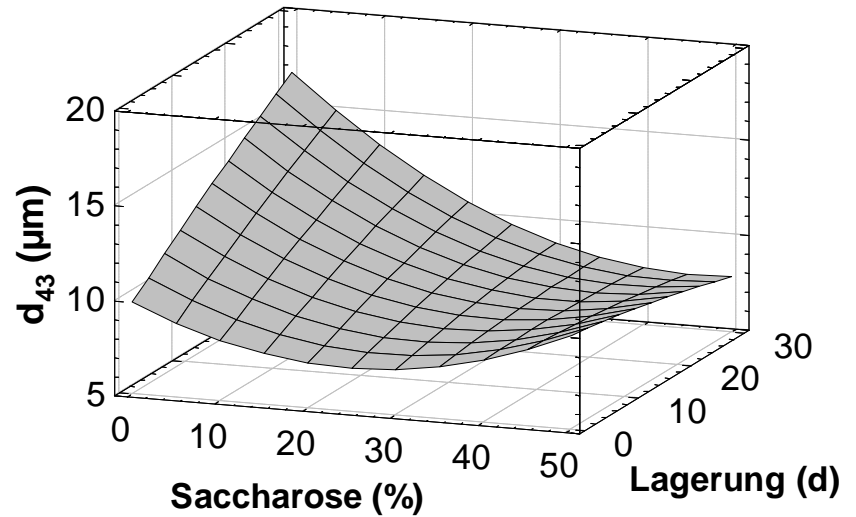
Particle size of O-droplets (d_{43}) depending on sugar content

(W/O/W preparation at ~ 1 MPa)



**Sugar
%**

**Glucose
%**



**Sucrose
%**

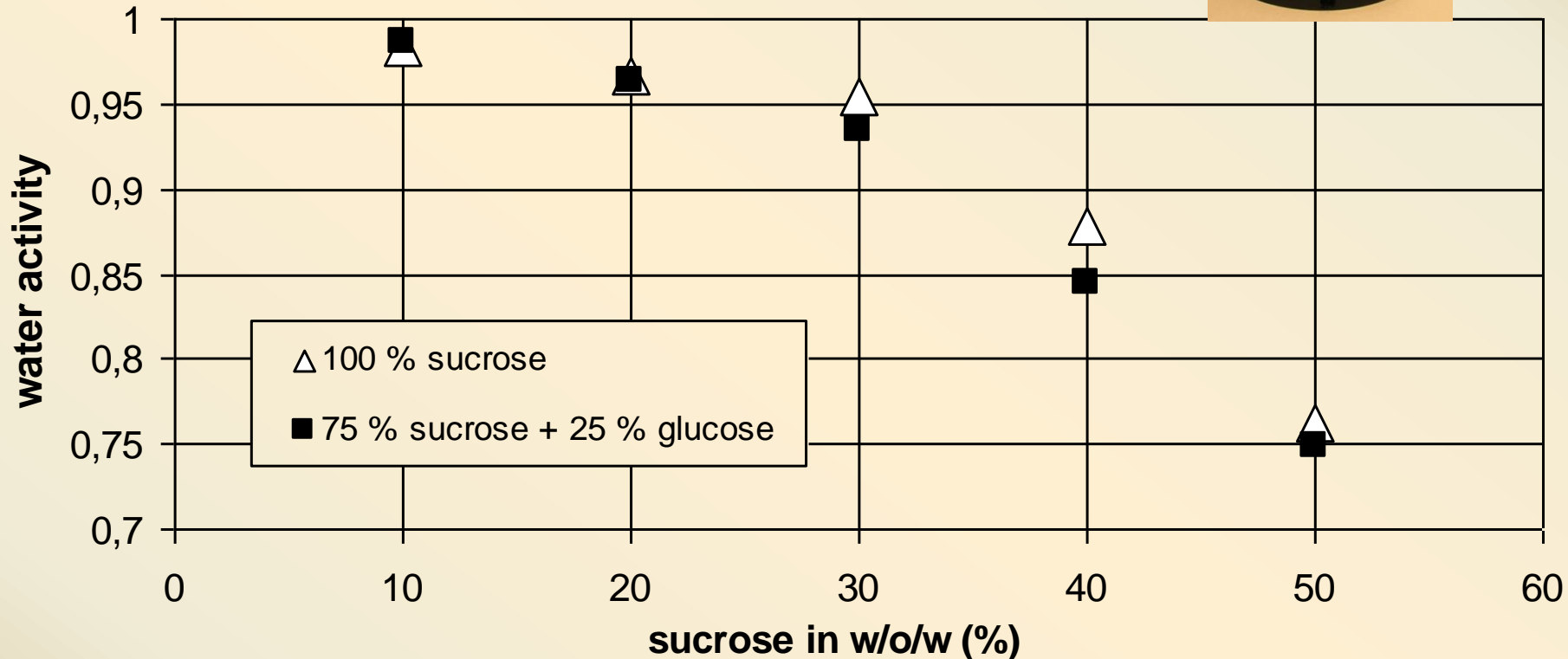
**Storage,
days**

The storage stability increases up to a sucrose content of ~ 40 %



Multiple emulsions with sucrose water activity

FA-st,
Fa. GBX

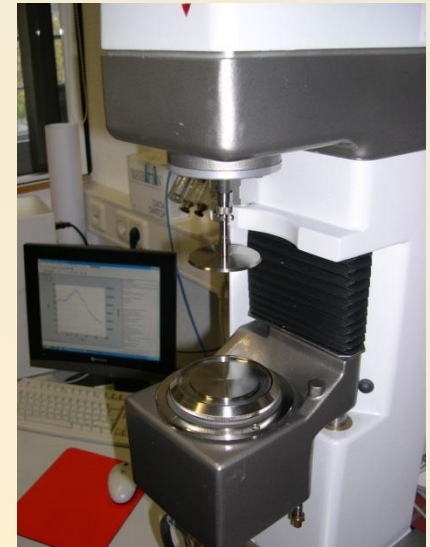


**Preparation W/O/W with ~ 59 % sugar in water
phases is possible ($a_w > 0,75$)**



Viscosity of W-phases

<i>Sugar conc.</i> <i>(%)</i>	<i>Sucrose</i> <i>(mPa s)</i>	<i>Sucrose/Gluc.</i> <i>(mPa s)</i>
14.7	3.0 ± 0.06	3.1 ± 0,3
29.4	5.8 ± 0,5	6.8 ± 2.5
44.1	16.0 ± 1.5	21.2 ± 13.7
58.8	83.5 ± 42.,7	44.0 ± 1.8
73.5	626.9	624.7 ± 237.0



O-Phase (continuous phase)

13.5 mPa s

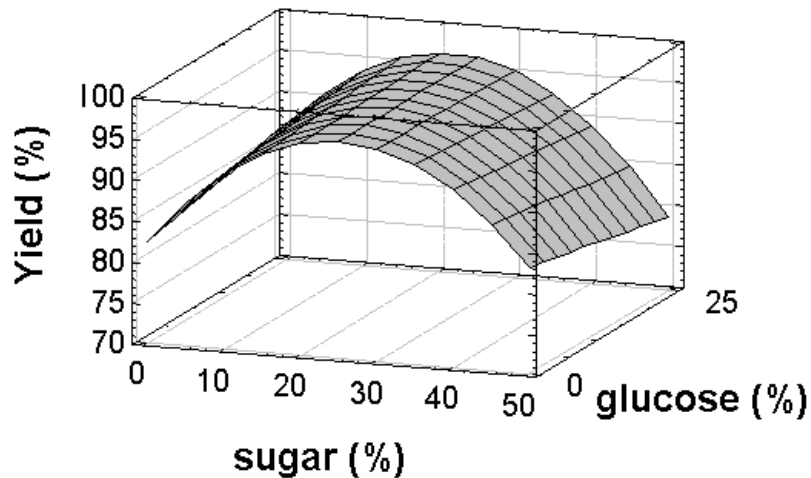
Shear rate: 100 s⁻¹, 60 °C,
Cone-plate 60 mm, 1°



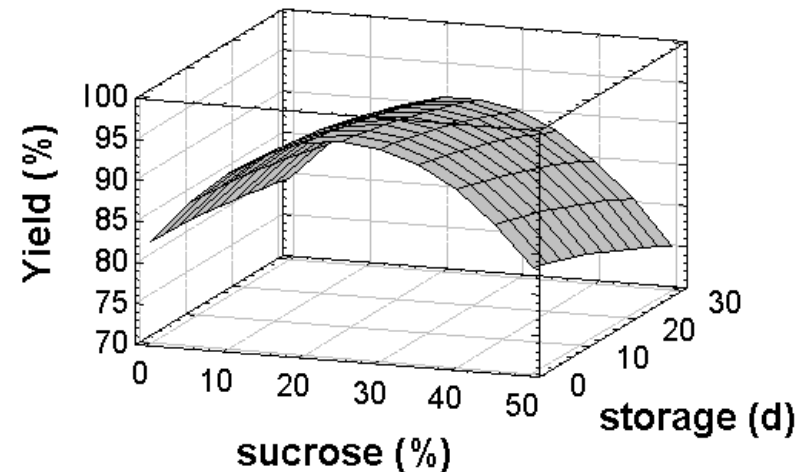
Multiple emulsions with sucrose

Encapsulation behaviour

A Effect of sugar on yield



B Effect of sugar and storage on yield



**Yield: Encapsulation of vitamin B₂ in W₁ > 80 %
at a sugar content of 20 – 40 % in W**



Sucrose content in $W_1/O/W_2$ (O-phase Miglyol)

Phase relations

W_1 to O = 20/80 W_1/O to W_2 = 40/60

Phases	Sucrose concentration (%)				
	W_1 and W_2	14.7	29.4	44.1	58.8
$W_1/O/W_2$	10	20	30	40	50

Transparency of emulsions increases with sugar content



Nutritional value of W/O/W

Composition of W/O/W per 100 g
 50 % sugar in W-phases
 (70 % sugar in W-phases)

W/O	W/O in W	kcal	Sugar %	Fat %	Protein %	H₂O %
20/80	40/60	439 (495)	34 (47.6)	32	0.68	33
30/70	40/60	410 (469)	36 (50.4)	28	0.72	34.8

Food calories can be reduced by increasing the W₁-phase



Conclusions

- W/O and W/O/W phases can be prepared with a high sugar content in W (tested up to 73.5 %)
- Droplet sizes of W/O emulsions containing up to 59 % sugar of $< 1 \mu\text{m}$ ($\eta_d/\eta_c \sim 5$) are possible
- High encapsulation rate ($> 90 \%$) can be realised in emulsions with a sugar content up to 59 %



Conclusions

- The next research should be concentrated on multiple confectionery emulsions prepared with **lecithin** (PC depleted) or **sugar esters** and with different **fat phases**



Main publications

- Muschiolik/Bunjjes,
Multiple Emulsionen
Behr's Verlag, 2007
- Muschiolik, G.
Multiple emulsions for food use,
Current Opinion in Colloid & Interface Science 12
(2007) 213-220
- Preissler, P.
Süßwarenfüllmassen auf Emulsionsbasis
Logos Verlag Berlin, 2006



Acknowledgement

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