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Comparison of O/W emulsions produced using cross-flow SPG membranes and a microfluidizer

Goran T. Vladislavljevic^{a,b*}, Helmar Schubert^b

^aInstitute of Food Technology and Biochemistry, Faculty of Agriculture, University of Belgrade, P.O. Box 127, YU-11081 Belgrade-Zemun, Serbia, Yugoslavia; Tel: +381 11 615-315/327, Fax: +381 11 199 711, E-mail: gtvladis@afrodita.rcub.bg.ac.yu

^bInstitute of Food Process Engineering, Faculty of Chemical Engineering, University of Karlsruhe (T.H.), Kaiserstrasse 12, D-76128 Karlsruhe, Germany; Tel: +49 0721 608-2497, Fax: +49 0721 694-320, E-mail: Helmar.Schubert@lvt.uni-karlsruhe.de

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Abstract

The oil-in-water emulsions consisting of vegetable (rape seed) oil as the dispersed phase and 2 wt % Tween 80 dissolved in demineralized water as the continuous phase were produced using Shirasu-porous-glass (SPG) membranes and Microfluidizer[®] (Microfluidics Corp., Newton, MA, USA). The mean pore size of the membranes used was in the range of 0.4-6.6 μm and the wall porosity 53-60 %. The hydraulic membrane resistance was inversely proportional to the square of the mean pore size according to the equation: $R_m = 0.056d_p^{-2}$, where R_m and d_p are in m^{-1} and m , respectively. The shear stress at the membrane surface was 8 Pa and the transmembrane pressure was 10 % higher than the capillary pressure. Using SPG membranes under these conditions, the emulsions with a mean droplet size 3.5 times larger than the mean pore size and the span of the droplet size distribution of 0.26-0.45 were produced. Therefore, the SPG technology is very suitable for producing emulsions with a narrow droplet size distribution over a wide range of mean droplet sizes (0.2-30 μm) using small mechanical stresses. On the other hand, a Microfluidizer[®] is more appropriate device for producing emulsions with a very low mean droplet size (0.08-0.2 μm). However, the span of the droplet size distribution curves for the emulsions produced using Microfluidizer[®] typically range between 0.91 and 2.7.

1. Introduction

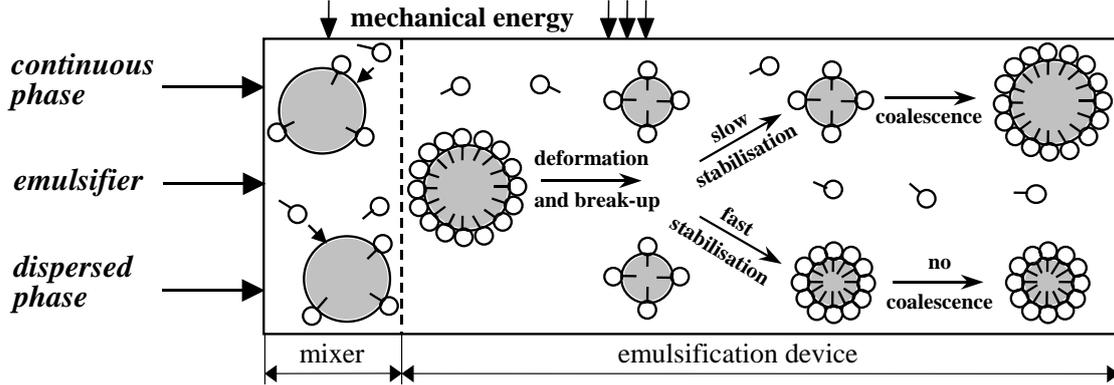
Mechanical emulsification can be performed using high-pressure homogenizers, rotor-stator systems, ultrasound homogenizers [1] or novel microporous emulsification systems based on a microporous membrane [2] or a microchannel plate [3]. In high-pressure homogenizers and rotor-stator or sonicated systems, interfacial area is increased by droplet disruption in a premix using relatively high energy inputs (Fig. 1a). However, in a membrane or a microchannel emulsification system, small droplets are directly formed by permeation of dispersed phase through the micropores or microchannels into moving (flowing or stirring) continuous phase (Fig. 1b). Accordingly, interfacial area is increased by direct formation of small droplets, rather than by disruption of larger droplets. In that way, droplet size can be more effectively controlled and the required shear stress is much smaller. The additional advantage over conventional emulsification methods is the possibility to obtain uniform droplets over a much wider range of mean droplet sizes.

Membrane emulsification (ME) is a suitable technique for the production of all types of single and multiple emulsions, including novel M/W (liquid metal-in-water) [4] and S/O/W (solids-in-oil-in-water) [5] emulsions. The main disadvantage of ME technology is a low dispersed phase flux through the membrane, which is required to obtain a narrow droplet size distribution. Due to this limitation, ME is primarily used for the production of small quantities of relatively expensive

*Corresponding author

special products containing highly uniform droplets or particles of controlled mean sizes. These applications include the preparation of drug delivery systems, monodispersed polymer microspheres and microcapsules, silica hydrogel particles [6], solder particles for surface mount technology [4] (an example of M/W emulsion application), etc. The polymer microspheres prepared using ME find applications as packings for GPC and HPLC columns [7-8], carriers of enzymes [9], spacers for liquid crystal displays [10], core particles for toner application [11], etc. DDS prepared using ME have been used for arterial injection chemotherapy of liver cancer [12], oral insulin preparation [13], etc. A promising application of ME in food industry is the production of super low fat spreads, introduced by Morinaga Milk Industry Co. Ltd., Japan [14].

(A) Droplet disruption in conventional emulsification systems



(B) Droplet formation at membrane pores

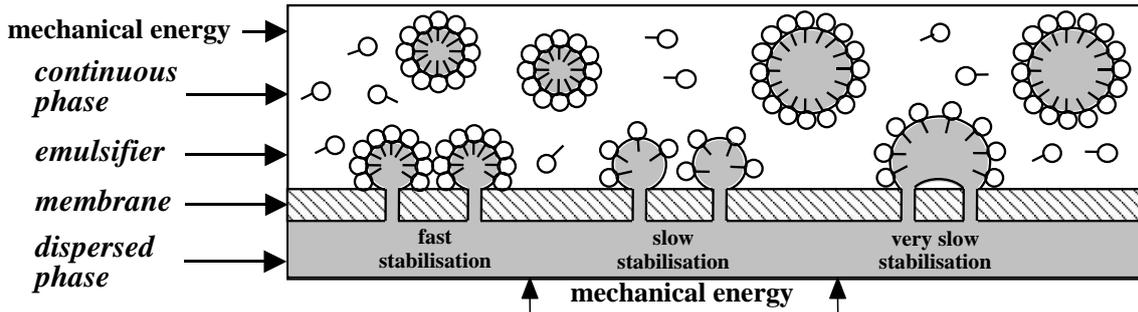


Fig. 1. Principles of droplet formation in emulsification

The most commonly used membrane for the preparation of emulsions is a Shirasu porous glass (SPG) membrane developed by Nakashima and Shimizu [15]. This membrane is characterized by uniform pores, a wide spectrum of available mean pore sizes (0.05-30 μm), and the possibility of surface modification. The O/W emulsions have been also produced using ceramic $\alpha\text{-Al}_2\text{O}_3$ and Zr_2O_3 membranes [16], perforated stainless steel plates [17], teflon membranes [18], silicon nitride microsieves [19], etc. The W/O emulsions have been produced using a hydrophobic or a pretreated hydrophilic SPG membrane [20], polypropylene hollow fibers [21], etc.

2. Theory of ME

The permeation of pure water through a membrane with a thickness of δ_m whose pores are capillaries of diameter d_p and length l_p , can be explained in terms of the Hagen-Poiseuille law [22]:

$$\Delta p_{tm} = J_w \eta_w R_m = 32 \eta_w l_p v_w / d_p^2 \quad (1)$$

where Δp_{tm} is the transmembrane pressure, R_m the hydraulic membrane resistance, η_w the water viscosity, J_w the water flux through the membrane, and v_w the mean water velocity in the pores. The substitution of $v_w = J_w / \varepsilon$ and $l_p = \delta_m \xi$ into Eq. (1) gives:

$$R_m = \Delta p_{tm} / (J_w \eta_w) = 32 \delta_m \xi / (\varepsilon d_p^2) \quad (2)$$

where ξ is the mean tortuosity factor of the pores and ε the membrane wall porosity.

During ME process droplets are usually formed simultaneously at only 2-40 % of the pores, while the remaining pores are unactive [23]. The fraction of active pores at any moment is given by:

$$k = J_d \eta_d R_m / \Delta p_{tm} \quad (3)$$

where J_d is the dispersed phase flux through the membrane and η_d is the dispersed phase viscosity. If the active pores are arranged over the membrane surface in a regular square array, the distance between the centers of adjacent active pores can be expressed as:

$$z = (d_p/2)(\pi/k\varepsilon)^{0.5} \quad (4)$$

Neglecting droplet deformation in the direction of continuous phase flow, droplets do not touch each other at the pore openings if $z < d_{drop}$. Thus, the condition for unhindered droplet growth in the case of uniform pore arrangement and rigid droplets reads:

$$d_{drop}/d_p < 0.5(\pi/k\varepsilon)^{0.5} \quad (5)$$

In order to avoid contact between two neighboring droplets at the pore openings, the fraction of active pores must be kept below $k_{max} = (\pi/4\varepsilon)(d_{drop}/d_p)^{-2}$.

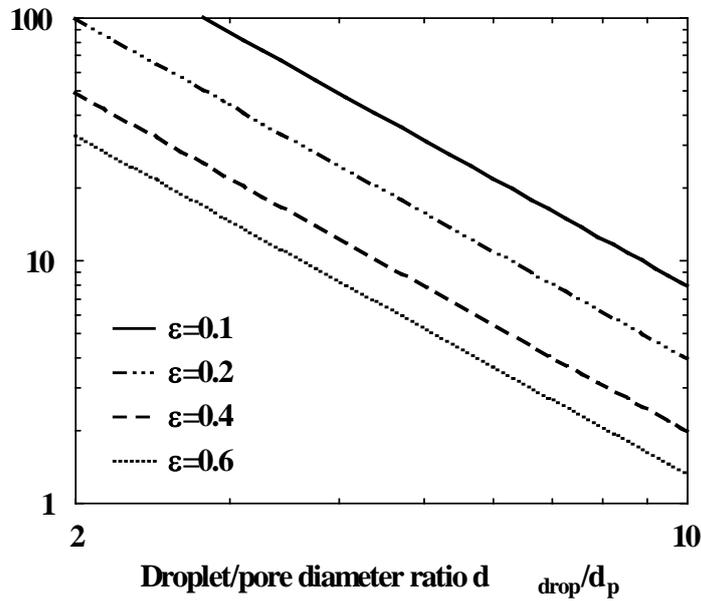


Fig. 2. Maximum percentage of active pores k_{max} for unhindered droplet growth as a function of droplet/pore diameter ratio d_{drop}/d_p and membrane wall porosity, ε

The porosities of SPG membranes are in the range of 0.5-0.6 [20] and for O/W emulsions d_{drop}/d_p ranges typically from 2.5-8 [24]. Therefore, to ensure that no coalescence can occur at the surface of the SPG membrane, the percentage of active pores must be kept below 2-25 %, depending mostly on the droplet/pore diameter ratio (Fig. 2).

The average droplet formation time t_f can be calculated as [16, 23]:

$$t_f = (2/3)(k\varepsilon/d_p^2)(d_{4,3}^3/J_d) \quad (6)$$

Although the above expressions are only approximate, they are useful in an attempt to give some quantitative explanations of the experimental results.

3. Experimental

3.1. Materials

Vegetable (rape seed) oil (Floreal GmbH) with a density of 920 kg m^{-3} and a viscosity of 58 mPa s was used as the dispersed phase. The continuous phase was 2% (w/w) Tween 80 (Polyoxyethylene

(20) sorbitan monooleate, Merck GmbH) dissolved in demineralized water. The viscosity of the continuous phase at 298 K was 1.42 ± 0.01 mPa s and density 1005 kg m^{-3} .

3.2. Experimental set-up and procedure for membrane emulsification

The Shirasu porous glass (SPG) membranes were supplied from SPG Technology Co., Ltd (Miyazaki, Japan) with a mean pore size of 0.4, 1.4, 2.5, 5, and $6.6 \mu\text{m}$, determined by a Shimadzu model 9320 mercury porosimeter. The SPG membrane ($125 \text{ mm length} \times 10 \text{ mm OD} \times 0.7 \text{ mm wall thickness}$) was installed inside a laboratory made stainless steel module. The effective length of the membrane tube was 116 mm and the effective membrane area 31.3 cm^2 .

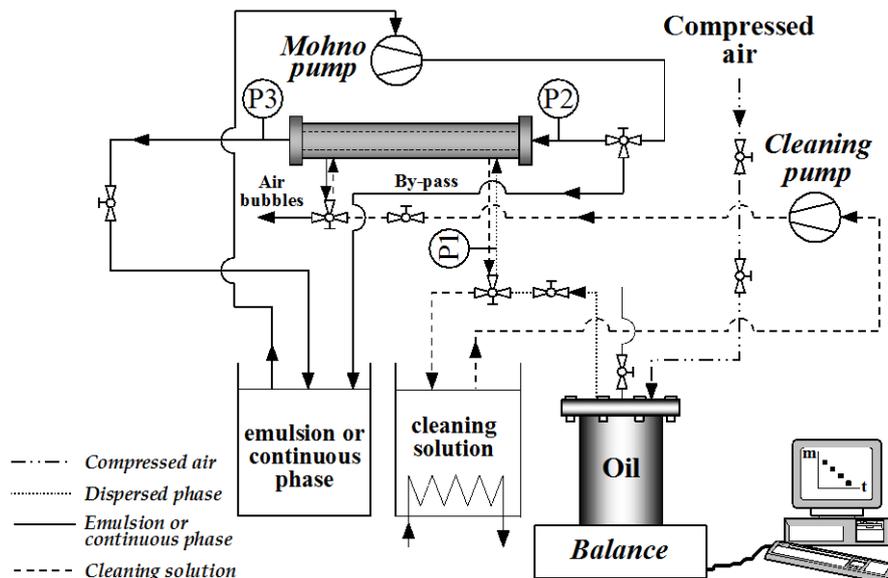


Fig. 3. Experimental set-up for cross-flow membrane emulsification

The continuous phase was recirculated in a closed loop between the membrane module and a continuous-phase reservoir using a Netzsch model NL 20 Mohno-pump (Fig. 3). In the most experiments, the continuous phase flow rate was 305 l h^{-1} , which corresponds to a mean velocity in the membrane tube of 1.4 m s^{-1} and tube Reynolds number of 8500. Under these conditions the shear stress at the membrane surface was 8 Pa. The oil phase was placed in the pressure vessel and was introduced at the module shell side with compressed air. The weight of oil permeated through the membrane was measured by a digital balance on which the pressure vessel rested.

3.3. Experimental set-up and procedure for emulsification using Microfluidizer

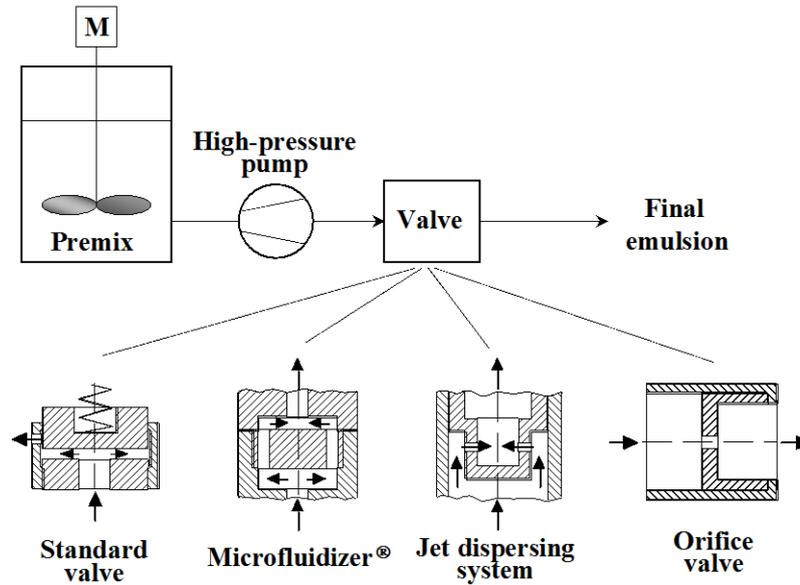


Fig. 4. Experimental set-up for emulsification using high-pressure homogenisers

The Microfluidizer (Microfluidics Corp., Newton, MA, USA) is a special kind of high-pressure homogenizer, allowing the generation of homogenizing pressures of up to 2750 bar. Inside the Microfluidizer emulsification chamber the stream of premix is separated in two streams which collide into a single stream afterwards (Fig. 4). In our experiments, raw emulsion (premix) flowed in a single pass through the homogenizing valve (no recirculation of the emulsion was applied).

The droplet size distribution for all emulsions was determined by a light scattering particle size analyser using PIDS technology (Coulter LS 230).

4. Results and discussion

4.1. Properties of the SPG membranes used in this study

The hydraulic and morphological properties of the SPG membranes used are listed in Table 1. The hydraulic membrane resistances were calculated by measuring the pure water flux through the membrane and using Eq. (2). The membrane resistance was inversely proportional to the square of the mean pore size (Fig. 5):

$$R_m = 0.056d_p^{-2} \quad (7)$$

where R_m and d_p are in m^{-1} and m , respectively. Eq. (7) is in accordance with Eq. (2). The wall membrane porosities ε determined by the pycnometric method [25] and the mean pore tortuosity factors calculated from Eq. (2) were independent on the mean pore size (Tab. 1). The ε values listed in Tab. 1 are within the range of 50-60 %, reported earlier for a typical SPG membrane [20]. As a comparison, the porosity of active layer of ceramic membranes ranges typically from 30-60 % [23].

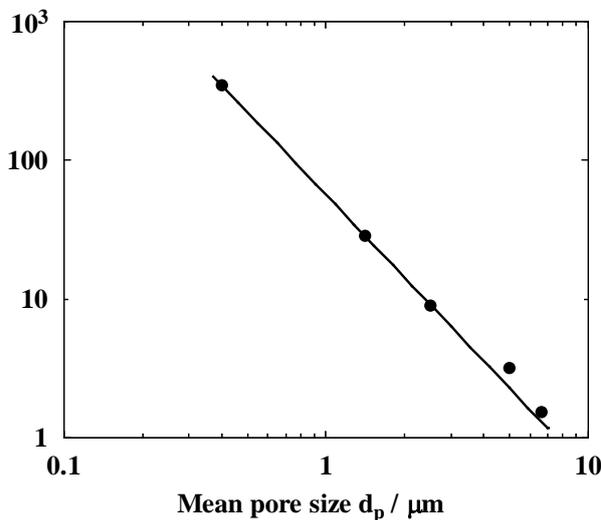


Table 1. The hydraulic resistances, R_m , the wall porosities, ε , and the mean tortuosity factors, ξ for the different SPG membranes used in our experiments

Pore size, d_p (μm)	1.4	2.5	5.0	6.6
$R_m \times 10^{-9}$ (m^{-1})	29	9.0	3.2	1.5
Porosity, ε (-)	0.60	0.53	0.60	0.58
Tortuosity, ξ (-)	1.7	1.3	2.1	1.7

Fig. 5. Hydraulic resistance of the SPG membrane as a function of mean pore size

4.2. Preparation of O/W emulsions using the SPG membranes

The experimental results obtained using SPG membranes at $\Delta p_{tm} \approx 1.1 p_{cap}$ are given in Tab. 2. Except for the 6.6 μm membrane, the experimentally determined p_{cap} values given in Tab. 2 are higher than the theoretical values calculated from the Laplace equation. Under given conditions the emulsions with a very narrow droplet size distribution were prepared over a wide range of mean droplet sizes (Fig. 6). The span of the droplet size distribution of 0.26-0.45 (Tab. 2) was much lower than that reported for ceramic membranes. As an example, Joscelyne and Trägårdh [26] obtained the span of 0.89-1.6 using ceramic membranes with a mean pore size of 0.1-0.5 μm . However, the dispersed phase flux in their experiments was 15-270 $\text{kg m}^{-2} \text{h}^{-1}$. Williams et al. [27] obtained the span of 0.83 at the oil flux of 8 $\text{l m}^{-2} \text{h}^{-1}$ using ceramic membrane with a mean pore size of 0.5 μm .

As a comparison, two droplet size distribution curves for the emulsions with the same dispersed phase content prepared using Microfluidizer (MF) are also given in Fig. 6. At both homogenizing pressures, very broad droplet size distribution curves are obtained with a span of 2.3 and 2.1 at 50 and 1100 bar, respectively. However, at the homogenizing pressure of 1100 bar the emulsion with a mean droplet size of only 0.16 μm was obtained, which is much lower than 0.76 μm at 50 bar and 1.4 μm obtained using the 0.4 μm -SPG membrane. The span of the droplet size distribution at 1100 bar can be decreased from 2.1 to 1.1 by applying two passes through Microfluidizer. In that case the final emulsion obtained after the first run is used instead of premix in the next run.

Table 2. The experimentally found capillary pressures, p_{cap} , and emulsification results at $\Delta p_{tm} \approx 1.1 p_{cap}$ and $\phi = 1-2 \text{ vol } \%$ for different mean pore sizes.

Pore size, d_p (μm)	0.4	1.4	2.5	5.0	6.6
p_{cap} (kPa)	185	48	24	10	5
$d_{3,2}$ (μm)	1.4	4.6	8.5	14.7	23.9
Span (-)	0.45	0.30	0.44	0.37	0.26
J_d ($\text{l m}^{-2} \text{h}^{-1}$)	0.7	2.3	2.9	4.3	6.6

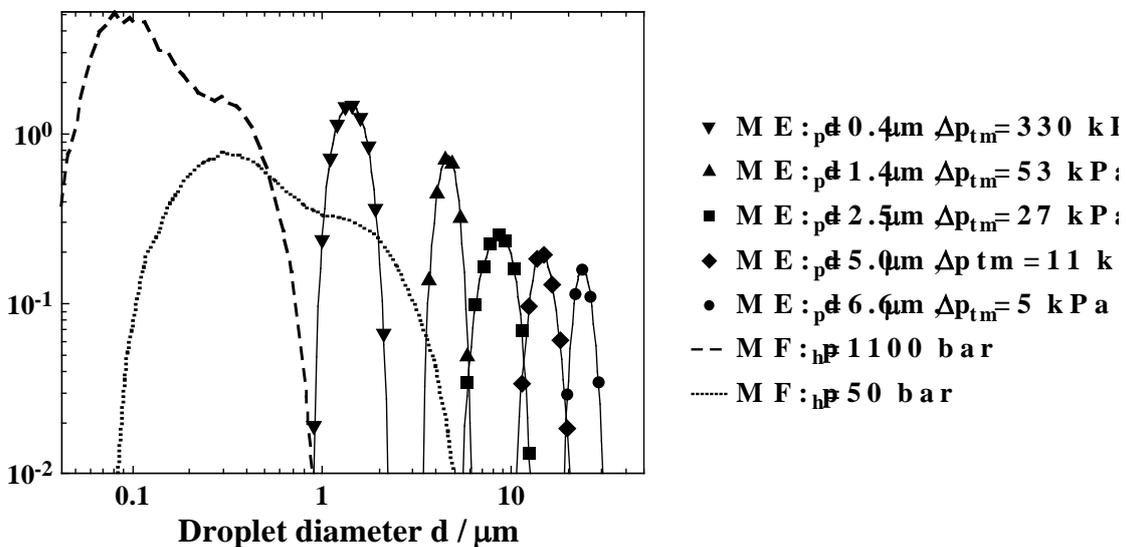


Fig. 6. Droplet size distribution curves for the emulsions obtained using membrane emulsification (ME) and Microfluidizer (MF) at the dispersed phase content $\varphi = 1-2\%$.

The mean droplet size at $\Delta p_{tm} \approx 1.1p_{cap}$ was found to be 3.5 times larger than the mean pore size (Fig. 7). The droplet/pore diameter ratio of 3.5 is similar to 3.25 reported by Nakashima et al. [2] for SPG membrane. Using Eq. (3) it was found that droplets were formed simultaneously at only 1.6-2.6 % of the pores (Tab. 3). Taking a mean ε value of 0.58 and $k = 0.02$, one obtains from Eq. (5) the ratio $d_{drop}/d_p < 8$. Thus, the condition for unhindered droplet growth is satisfied.

Table 3. Calculations of the fraction of active pores, k and the average droplet formation time, t_f at $\Delta p_{tm} \approx 1.1p_{cap}$ and $\varphi = 1-2\%$ for different mean pore sizes

Pore size, d_p (μm)	0.4	1.4	2.5	5.0	6.6
k (-)	0.019	0.020	0.016	0.020	0.026
t_f (s)	0.6	0.7	0.8	0.9	1.8

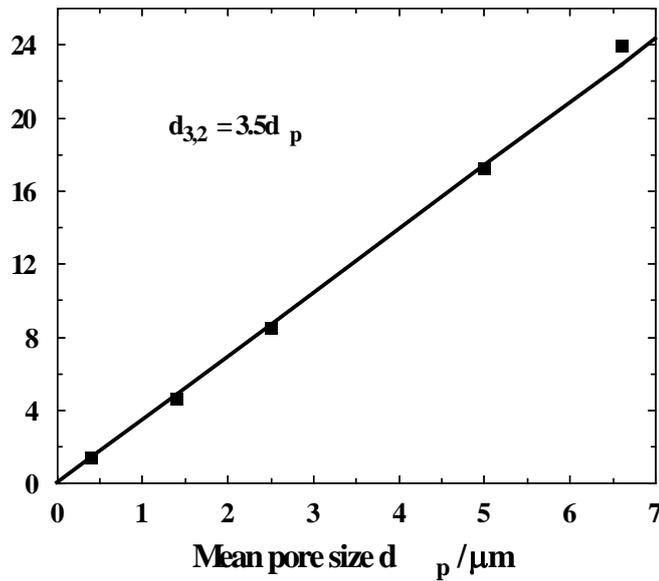


Fig. 7. Relationship between mean pore size and mean droplet size at $\Delta p_{tm} \approx 1.1p_{cap}$

The average droplet formation times of 0.6-1.8 s (Tab. 3) were similar to 1-1.5 s found by Schröder and Schubert [28] for Tween 20 and a ceramic membrane. The longest droplet formation time for the 6.6 μm membrane is a consequence of the largest droplet volume in that case.

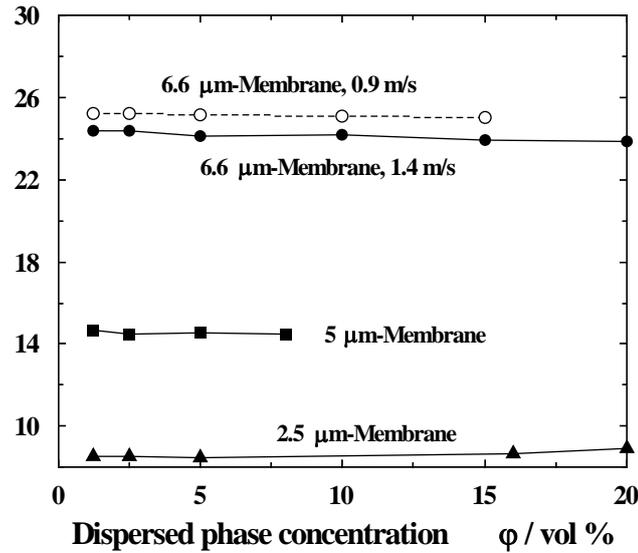


Fig. 8. The influence of dispersed phase content on mean droplet size at $\Delta p_{tm} \approx 1.1 p_{cap}$

The mean droplet size was almost independent of the dispersed phase content up to 20 vol % (Fig. 8), although the dispersed phase flux through the membrane increased with time. This trend was also found by Katoh et al. [29] in the preparation of food emulsions using SPG membranes. The small decrease of the mean droplet size in the experiments with the 6.6 μm membrane can be explained by the disruption of droplets during recirculation. At the smaller cross-flow velocity of the continuous phase (0.9 m s^{-1}), the disruption was less pronounced but the mean droplet size was higher.

4.3. Preparation of O/W emulsions using the Microfluidizer

The mean droplet size in the emulsions produced using Microfluidizer primarily depends on the dispersed phase content, ϕ and the consumption of energy per unit emulsion volume, i.e. energy density, E_v . Here, E_v is defined as: $E_v = P/Q_v = p_h$ [30] where P is the power input, Q_v the volumetric flow rate of the emulsion and p_h the homogenizing pressure. For continuous droplet disruption (avoiding re-coalescence), the mean droplet size can be described as a power function of the energy density: $d_{3,2} = C(E_v)^{-b}$. In Fig. 9, the mean droplet size is plotted against the energy density for different dispersed phase concentrations (2.5-40 vol%). Obviously, the previous equation is valid and the constant b decreased with increasing the dispersed phase content. However, the mean droplet size was in a relatively narrow range between 0.16 and 2.1 μm .

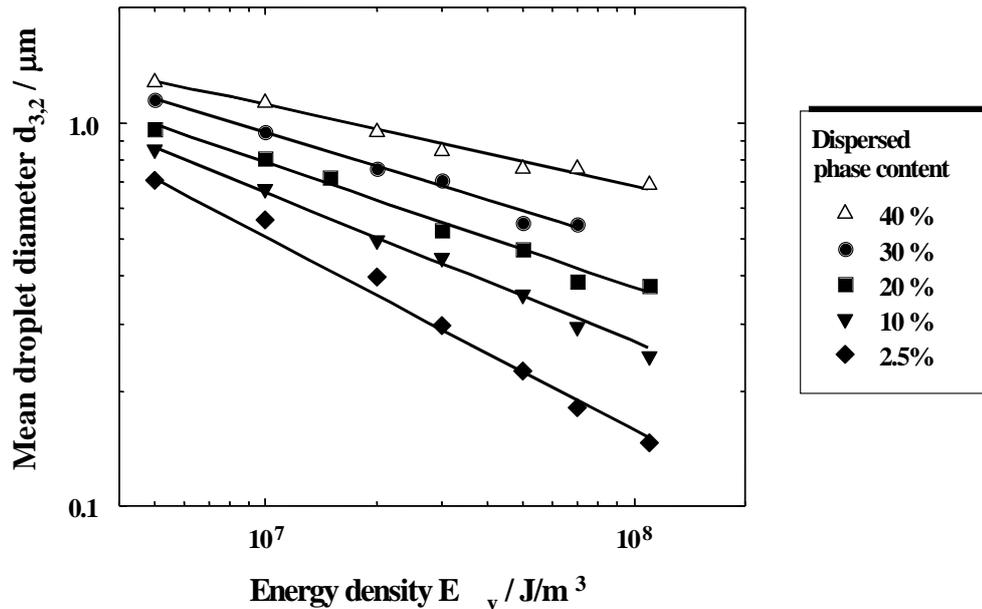


Fig. 9. Mean droplet size as a function of energy density for droplet disruption in Microfluidizer

5. Conclusions

The SPG membrane can be successfully used to produce O/W emulsions with a very narrow droplet size distribution, on the condition that the transmembrane pressure is not much higher than the capillary pressure. Under these conditions the mean droplet size is 3.5 times larger than the mean pore size and not significantly affected by the dispersed phase content up to 20 vol %. However, the transmembrane flux is very low, because at any moment only about 2% of the pores are active. In addition to that, SPG membranes are not suitable for producing emulsions with the mean droplet size below 0.2 μm . For such emulsions, a special type of high-pressure homogenizer called Microfluidizer can be successfully used. However, the span of the droplet size distribution for the emulsions produced using Microfluidizer was in the range of 0.9-2.7.

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List of symbols

d_p	mean pore size of the membrane, m
d_{drop}	droplet diameter, m
$d_{3,2}$	mean Sauter diameter, m
$d_{4,3}$	volume-weighted mean droplet diameter, m
E_v	energy density, J/m^3
J_d	dispersed phase flux through the membrane, $m\ s^{-1}$
J_w	pure water flux through the membrane, $m\ s^{-1}$
k	fraction of active pores, (-)
l_p	pore length, m
P	power input, W
p_{cap}	capillary pressure, Pa
p_h	homogenizing pressure, Pa
R_m	hydraulic membrane resistance, m^{-1}
t_f	average droplet formation time, s
v_w	mean water velocity in the pores, $m\ s^{-1}$
z	distance between the centres of adjacent active pores, m
Δp_{tm}	transmembrane pressure, Pa
δ_m	membrane thickness, m
ε	membrane porosity, (-)
η_d	viscosity of dispersed phase, Pa s
η_w	viscosity of water, Pa s
ξ	mean tortuosity factor of pores, (-)
φ	dispersed phase concentration in emulsion, vol %