Oscillating membrane emulsification

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Conventional methods for preparing of emulsions (high pressure homogenizers, colloid mills, rotor–stator systems ultrasonic or microfluidizers) apply more energy then needed for production of droplets giving the droplets with wide range distribution. Membrane emulsification is a membrane-assisted dispersion process to produce an emulsion of one liquid phase (such as oil) in a second immiscible liquid phase (such as water)[1].

In this work alternative method to generate the shear at the membrane surface, that can minimize the risk of break up of the drops previously formed, is applied. The technique is based on the low frequency oscillation of the membrane surface in a direction perpendicular to the flow of the injected phase through the membrane. The advantage of oscillating membrane technique is that it can be scaled up by providing a larger membrane area what makes it very interesting for industrial application. The technique is applicable to the generation of larger drops (ion exchange resins, food and flavor encapsulation, controlled release depots under the skin, electronic ink capsules, medical diagnostic particles, high value fillers and other species with particle, or drop, size greater than approximately 20 μm) than can be reliably achieved by a crossflow membrane emulsification process, where drop breakage after formation occurs.

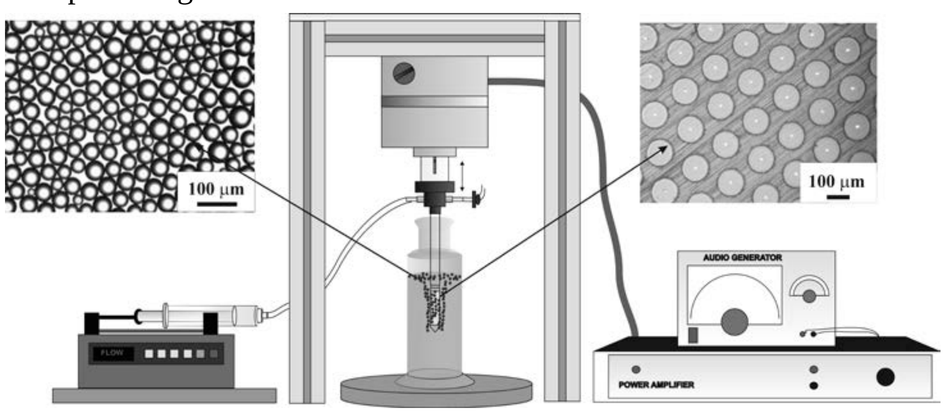


Figure 1. *Oscillating membrane system, surface of used membrane and image of produced droplets for condition* τ_{max} ***=3.****6P and f=15 Hz*

The shear stress required for droplet detachment from the membrane surface in used system was provided varying the frequency, or the amplitude of oscillation. The oscillation signal was provided by an audio generator which fed a power amplifier driving the electro-mechanical oscillator on which the inlet manifold was mounted. The oscillating membrane system used is illustrated in Figure 1 and was supplied by Micropore Technologies Ltd. The membrane was in the form of a candle with external diameter of 15 mm and a working length of 57 mm, 10 μm pore size and 180 μm pore spacing.

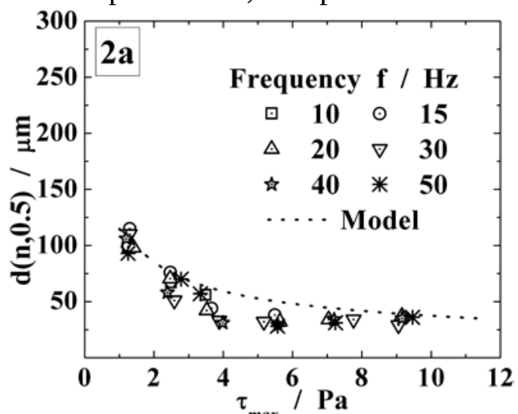
The dispersed phase for the oil in water emulsions was commercially available food grade sunflower oil. The continuous phase was purified water (obtained from a reverse osmosis system) containing 2% vol/vol Tween 20 surfactant (polysorbate). The continuous phase volume was 150 cm^3 and 10 cm^3 of dispersed phase was injected for each experiment. The oscillation frequency ranged from 10 to 50 Hz. In all experiments a very low flow rate of 30 liters of dispersed phase injected per square meter of membrane area per hour (L m^{-2} h^{-1}) was maintained in order to minimize any push-off effect which was observed in previous work [2]. Influence of applied maximal shear stress ($\tau_{\max}$) on droplet size is presented in Figure 2a.

$$\tau_{\max} = (2\pi)^{3/2} (\mu\rho)^{1/2} a f^{3/2} \text{ Eq. (1) [3]}$$

The data on Figure 2a records the frequency used to achieve the given peak shear stress and the corresponding amplitude can be estimated from Eq. (1). Under identical conditions of shear stress, a higher frequency is compensated by using lower amplitude of oscillation, where amplitude is half of the peak-to-peak displacement of the membrane motion. The number distributions for the o/w emulsions were obtained using a Malvern Mastersizer (model S) and droplets were in range between 29 and 115 μm (while span was between 0.40 and 0.55). Apart from the very low frequency of 10 Hz, there does not appear to be any significance in the combination of the frequency and amplitude used: the resulting drop size is apparently a function of the peak shear stress only and not the frequency used to achieve it. On increasing peak shear the droplet size decreases sharply and for most frequencies reaches a constant value after the peak shear is 4 Pa. Also the data were compared with existing model Eq. (2) and model predictions are presented in Fig 2a.

$$x = \frac{\sqrt{18\tau_{\max}{}^2 r_p^2 + 2\sqrt{81\tau_{\max}{}^4 r_p^4 + 4 r_p^2 \tau_{\max}{}^2 \gamma^2}}}{3\tau_{\max}} \text{ (2) [4]}$$

The data on Figure 2b records the frequency used to achieve the given peak shear stress. A peak-shear-event will take place twice for every cycle: once during upward movement and again during downward movement. Hence, for the slowest frequency used, 10 Hz, the number of peak-shear-events is 20 per second; or a peak shear every 50 milliseconds.



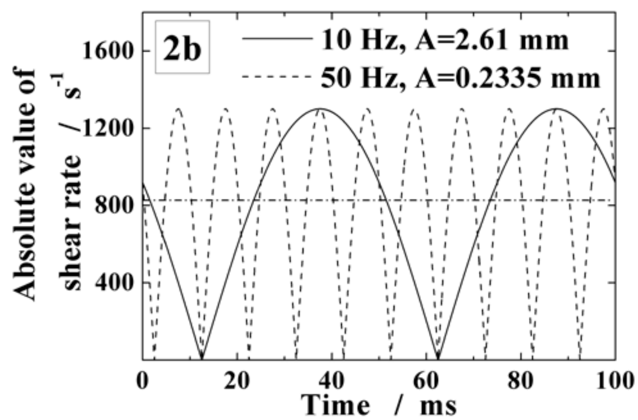


Figure 2a *Comparison of experimental drop diameters produced in Oscillating Membrane Emulsification and predicted values using max shear stress model [4]: the values of d(n,0.5) were obtained at 30 L m^{-2} h^{-1}.*

Figure 2b *Shear rate with time where the maximal peak shear for both frequencies was 1.3 Pa, the dashed/dot line represents the average shear rate of 828 s^{-1} which is the same for both frequencies.*

Under constant peak shear stress at the membrane surface, the drop size was essentially independent of the frequency of oscillation, because the effect of an increasing frequency was compensated by a decreasing amplitude. With increasing of peak shear the droplet size decreased sharply and for most frequencies reached a constant value at the peak shear stress of about 4 Pa. It was shown that oscillating membrane system is applicable for generating of larger drops than can be reliably achieved by a crossflow membrane emulsification process, where drop breakage after formation occurs. Very important advantage of oscillating membrane technique is that it can be scaled up by providing a larger membrane area in the oscillating membrane assembly.

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