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Preparation of multiple emulsions using SPG membranes: Factors influencing droplet size distribution, dispersed phase flux and encapsulation yield

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Membrane emulsification (ME) involves the permeation of pure dispersed phase through a porous membrane into continuous phase (direct ME) or the passage of pre-emulsion through the membrane (premix ME [1]). In direct ME, fine droplets are directly formed at the pore openings. In premix ME, large droplets of a premix are disrupted into fine droplets inside the pores. The most suited membrane for ME is a Shirasu porous glass (SPG) membrane developed by Nakashima and Shimizu [2].

Preparation of multiple W₁/O/W₂ emulsions using multi-pass premix ME has been studied in this work. The oil phase was 5 wt. % PGPR dissolved in soybean oil. The inner aqueous phase contained 2 wt. % Ca (II)-EDTA as a marker for the determination of the encapsulation yield and 5 wt. % glucose as the osmotic additive. The outer aqueous phase contained 0.5 wt. % Tween 80, 1 wt. % sodium alginate, and 5 wt. % glucose. The primary W₁/O emulsion was prepared by means of a homomixer at 24,000 rpm. The W₁/O emulsion was then mixed with the outer aqueous phase by means of a stirring bar to prepare a W₁/O/W₂ premix. The premix was homogenized by permeation through the SPG membrane with a mean pore size of 10.7 µm and an effective membrane area of 3.75 cm². Centrifugal cells with a mean pore size of 0.2 µm were used to separate the prepared W₁/O drops from the outer aqueous phase. The Ca content in the filtrate was measured by the ICP method. The total Ca content in the internal droplets was found by breaking a prepared emulsion with acetone. The encapsulation yield was determined as the percentage of Ca which was left in the internal droplets after emulsification.

The transmembrane flux increased and asymptotically approached a constant maximum value, as the number of passes through the membrane increased (Fig. 1). The flux was higher at the smaller volume ratio of W₁/O drops, φ₀. At φ₀ = 0.3-0.5 and the volume ratio of internal droplets, φ₁ = 0.3-0.5, the mean size of W₁/O drops was independent on φ₀ and φ₁ and decreased with the number of passes. After 3 transmembrane passes, very uniform W₁/O drops were obtained (δ = 0.33-0.36), combined with a high encapsulation yield (84-88 %). At φ₁ = 0.5 the yield was smaller due to larger internal droplets.

Fig. 1 (left): The effect of number of passes on transmembrane flux. Fig. 2 (right): The effect of number of passes on mean size of W₁/O drops, encapsulation yield of internal droplets and span.

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Literature