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The effects of membrane composition and morphology on the rotating membrane emulsification technique for food grade emulsions

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ABSTRACT

The effects of using different membrane materials and morphologies in the membrane emulsification process were observed using similar operating parameters and system geometry, allowing a direct comparison of not only the membranes themselves but also between both a stationary cross-flow membrane emulsification device and a rotated membrane emulsification device. Each membrane type tested had distinct characteristics, and the droplet sizes produced responded differently to changes in operating conditions.

The rotating membrane produced similar droplet sizes to the cross flow membrane system, but at a much lower shear rate. This suggests that the detachment of the droplets occurs sooner due to the additional centrifugal force and system vibration. The Shirasu porous glass (SPG) membrane produced the smallest droplet sizes (<1 μm from a 1 μm membrane), however the stainless steel membrane produced the lowest droplet size to pore size ratio ($\sim$0.5:1) due to its cylindrical pore geometry as opposed to the tortuous geometries of the other membranes used. The droplet sizes produced at different pressures are similar between rotated and cross-flow membrane emulsification, with increases in pressure increasing droplet size and size distribution. The viscosity of the continuous phase has an effect on the droplet size; increasing the viscosity decreases the droplet size by increasing the applied shear, allowing fine tailoring of the size produced, with a more viscous continuous phase reducing the droplet size from $\sim$4 μm to $\sim$1 μm with an increase in viscosity of 100 mPa s.

Rotating membrane emulsification has properties with potential to produce shear sensitive emulsion microstructures with small droplet sizes. Emulsion microstructures such as duplex emulsions, core/shell structures beads etc. can be used in the production of novel food structures.

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1. Introduction

The manufacture of emulsions is an important part of many processes across many industry sectors; however, the emulsification process often still relies on traditional droplet break-up systems during which droplets are produced by repeatedly breaking large droplets into smaller ones until the desired size range is reached [1]. The last few decades have brought many advances in producing droplets with very tightly controlled size distribution spans, which find uses in high value products such as spacers for liquid crystal displays [2] and packing beads for chromatography columns [3]. These droplets are produced by more careful emulsification techniques, producing the droplets at the size that is required rather than breaking them up from pre-existing larger droplets [4,5]. One such technique is that of membrane emulsification, and this has been explored with the aim of producing near mono-disperse droplets [6,7].

Membrane emulsification does allow a degree of control over the size distribution of the droplets produced, however there are other made to measure droplet production techniques capable of far better monodisperse droplet production, such as micro and nanochannel devices [8,9], edge emulsification [10,11] and microsieve emulsification [12]. The ability to control the microstructure of the droplets is perhaps a more appealing possibility of this technique, as well as minimising the amount of shear applied to droplets as they form [13]. It also has the advantage of easier scaling (whilst not without challenges) than the slower microchannel devices, which require complicated parallelisation to scale production [8].

The idea of applying shear to break forming droplets from the surface of the membrane has been previously explored, using both a flow of continuous phase along the membrane surface [14–16] and by rotating a tubular membrane [6,17] as well as vibrating/

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sonicated [18] membrane devices. The forces that result in the eventual detachment of a droplet as it grows at a pore have been identified as shear, pressure/inertia of the dispersed phase, interfacial tension, and buoyancy [19]. Since buoyancy is usually several orders lower than the others it is usually disregarded [20]. The interfacial tension is dependant on the emulsifier present, and the initial interfacial tension between the two immiscible liquids making up the two phases. The pressure of the dispersed phase through a pore, and the resultant inertia is a factor of the transmembrane pressure applied. The shear force is provided by flowing the continuous phase across the surface of the membrane, or by rotating the membrane in a vessel of the continuous phase [6,21].

The application of a perpendicular flowing (rather than static) continuous phase was shown to reduce the droplet size [22] with greater shear (faster flow) producing greater reduction in average droplet size [23]. Changes to the pores size and morphology has been varied and shown to have a great effect on the detachment of droplets [24,25], with faster, more consistent detachment from structured uniform pores (varying slightly with shape [26]) and slower more uneven detachment from unstructured pores such as porous glass membranes [22].

Vibration of the membrane was studied [18] and found to detach droplets from pores sooner, producing smaller droplets, but only at low vibrational frequencies < 100 Hz. In this study, Zhu and Barrow (2005) also suggest a detachment mechanism in membranes of low pore separation due to steric hindrance between forming droplets, where droplets are 'pushed' from the membrane surface by those beginning to form behind them.

Rotation of the membrane has been studied with metal membranes and shown to enhance membrane detachment, with large droplets produced at low shear rates [6] and very small droplets produced in a high shear system [17]. Both of these studies used metal membranes, which were not studied in a comparison cross flow system.

The aim of this study was to investigate rotating membrane emulsification with different membrane types and morphologies, and to directly compare the same membrane size and type in the two hydrodynamic configurations of the cross-flow and rotating membrane techniques for the preparation of food grade emulsions.

2. Experimental

2.1. Membrane emulsification systems

For this research two membrane emulsification systems were used, both accepting the same diameter membrane tube, one capable of rotating the membrane whilst dispersed phase is pressurised through it from inside (Fig. 1), and the other capable of allowing cross-flowing continuous phase to flow over the membrane whilst dispersed phase is pressurised through from inside (Fig. 2).

The membrane tubes used had an outside diameter of 10 mm, and in the rotating system the vessel used to house the continuous phase in which the membrane was rotated had a inside diameter of 30 mm, giving a gap to the membrane of 10 mm.

2.2. Materials

Emulsions produced were made with commercial sunflower oil (it was important that no special oil was used to give an accurate representation of what would normally be used in food production), and deionised filtered (milliQ reverse osmosis) water. Several different emulsifiers were used (Tween®20 (polyoxyethylene

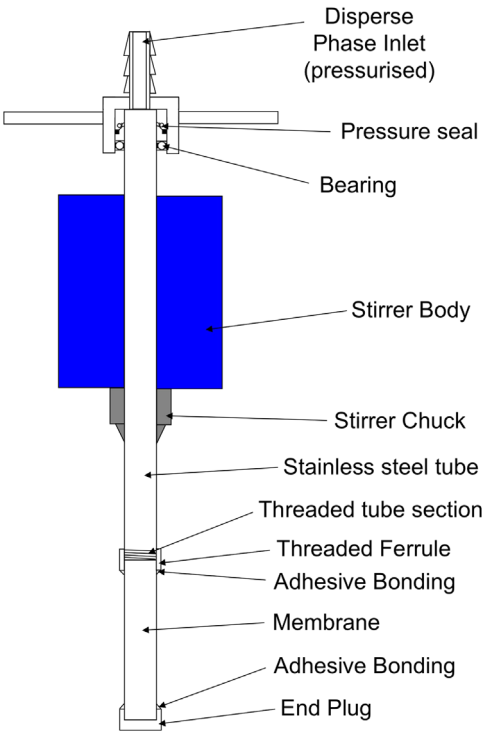

Fig. 1. The rotating membrane emulsification system employed a rotating fluid coupling to allow pressurised fluid to flow into the shaft and on into the rotating tubular membrane. The continuous phase was housed in a suitable vessel in which the membrane was submerged.

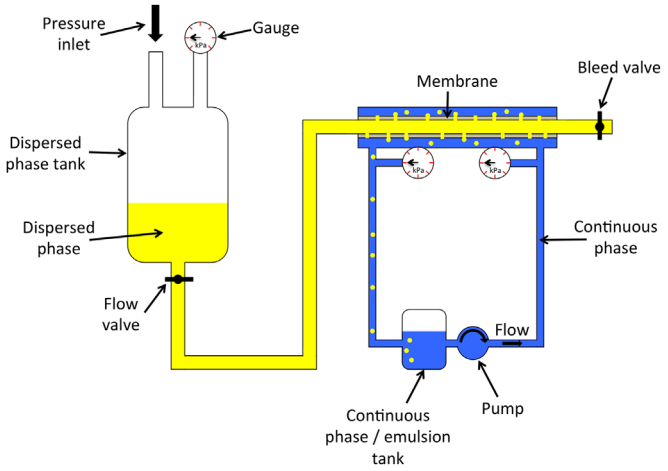

Fig. 2. The cross-flow membrane emulsification system used in this study was setup as shown. The membrane module houses the interchangeable membranes such that they separate the dispersed and continuous phases, with the continuous phase flowing along the outside of the membrane tube.

20 sorbitan monolaurate), Tween®80 (polyoxyethylene 80 sorbitan monolaurate), SDS (Sodium Dodecyl Sulphate), Soya Lecithin (phosphatidylcholine), sodium caseinate) and were all purchased from Sigma Aldrich, UK.

2.3. Emulsion analysis

The resultant emulsion droplets were analysed using a Malvern Mastersizer 2000 with an attached hydro 2000 small volume sample dispersion unit. Droplet diameters are given in $D_{[4,3]}$ (volume weighted mean) and size distributions are given in % by volume. Relative span is calculated by $D_{0.9}-D_{0.1}/D_{0.5}$ where D is the diameter in microns, below which the subscript proportion of

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