



Process optimisation of rotating membrane emulsification through the study of surfactant dispersions



David M. Lloyd*, Ian T. Norton, Fotis Spyropoulos

Centre for Formulation Engineering, School of Chemical Engineering, University of Birmingham, Edgbaston, Birmingham B15 2TT, UK

ARTICLE INFO

Article history:

Received 13 October 2014

Received in revised form 21 May 2015

Accepted 7 June 2015

Available online 19 June 2015

Keywords:

Membrane emulsification

Rotor–stator

Surfactant

Diffusion

Interfacial tension

Energy density

ABSTRACT

In this study, a rotating membrane emulsification setup incorporating a 6.1 μm pore diameter Shirasu porous glass membrane was used to produce oil-in-water emulsions. The processing conditions varied between 0.2 and 1.5 bar for the transmembrane pressure and shear rates at the membrane surface between 0.6 s^{-1} and 104.6 s^{-1} were generated. All emulsions consisted of 10 vol.% of sunflower oil stabilised by one of four different surfactants (Tween 20, Brij 97, lecithin and sodium dodecyl sulphate) of either 0.1 wt.% or 1 wt.% concentration. A novel approach for emulsification processing was introduced which incorporates high hydrophilic–lypophilic balance, non-ionic surfactants within the dispersed phase rather than the continuous phase. A reduction in droplet size by at least a factor of 3 for the same formulation can be achieved without significant hindrance on disperse phase flux. This therefore suggests a possible strategy for further process optimisation.

© 2015 The Authors. Published by Elsevier Ltd. This is an open access article under the CC BY license (<http://creativecommons.org/licenses/by/4.0/>).

1. Introduction

Formulating dispersions of one liquid phase within another immiscible liquid (i.e. an emulsion) remains an important area of research since these are readily incorporated within many foods, pharmaceutical, agrochemical and cosmetic products. Commonly cited examples from within the food industry include ice cream, mayonnaise and salad dressings, all of which are supplied to a global marketplace in large quantities. As such, there is increasing focus on the development of emulsification processes either to deliver improved product characteristics (e.g. greater stability, increased flavour perception) or to match expectation of current product quality but in more sustainable manner (e.g. lower energy consumption). Emulsions require the use of a surfactant to stabilise the droplet interface and as such, selection of an appropriate one is a key consideration for producing a microstructure with the desired droplet size distribution.

There are two philosophies that can be adopted to create an emulsion. The majority of emulsification processes focus on the breaking down droplets into smaller entities through subsection to mechanical energy e.g. homogenisers, rotor–stator mixers, colloid mills. A number of disadvantages are associated with forming droplets in this way, primarily associated with a wide droplet size range due to non-uniform energy dissipation and low energy

efficiency due to repeated droplet break up and re-coalescence (Joscelyne and Tragardh, 2000; Charcosset, 2009; Gijsbertsen–Abrahamse et al., 2004; Jafari et al., 2008). In the latter instance, surfactant concentration is often overcompensated in order to achieve favourable processing kinetics. It is widely accepted that surfactants are some of the most costly components within many formulations. Processes that require both excessive use of energy and costly ingredients are neither environmentally nor economically sustainable and thus attention is shifting towards alternative processes that can minimise their use. More recent approaches look to build up droplets individually and then add them to the continuous phase in a controlled manner until the desired volume fraction of the phase to be dispersed is obtained (Nakashima et al., 1991; Yuan and Williams, 2014). This is the basis of membrane emulsification in which droplets are produced at individual membrane pore outlets, only detaching when the force holding the droplet at the membrane surface (primarily interfacial tension) is overcome by a combination of forces determined by operating parameters such as transmembrane pressure (inertial) and shear (drag) as well as by the physical properties of the phases e.g. density difference (buoyancy) Peng and Williams, 1998; De Luca and Drioli, 2006. With careful operation of the membrane emulsification process, droplets can be eloquently crafted and as such narrow droplet size distributions are achievable which may improve functionality of an emulsion based product e.g. stability against Ostwald ripening or ensure uniform release rate of an active ingredient throughout the system

* Corresponding author.

E-mail address: DML066@bham.ac.uk (D.M. Lloyd).

(Kobayashi et al., 2003). In combination with this benefit, the energy consumption is at least an order of magnitude lower than when adopting a droplet break down approach (Gijbetsen-Abrahamse et al., 2004; Schubert et al., 1997; Walstra and Smulders, 1998). With the current rising costs of energy and negative environmental consequences associated with excessive energy consumption, this therefore increases the appeal of low energy, sustainable processes such as membrane emulsification.

Up until now, a number of drawbacks associated with membrane emulsification have perhaps held back the process from being implemented industrially. It is widely documented that the primary limitation is the low dispersed phase flux achievable (Kukizaki and Goto, 2007; Vladisavljevic and Schubert, 2003). Attempts to maximise the flux through application of high pressure driving force lead either to coalescence (Lepercq-Bost et al., 2010) or jetting of the dispersed phase (Kobayashi et al., 2003; Pathak, 2011), both of which reduce the level of control on the droplet size produced. Alternatively, a pre-mix membrane emulsification approach is used in which a coarse emulsion is passed through a membrane to break down droplets within pore channels (Surh et al., 2008; Vladisavljevic et al., 2004; Nazir et al., 2011, 2013). Whilst higher fluxes are achievable due to the generally lower viscosity (than pure dispersed phase), the requirement of multiple passes to ensure droplet uniformity negatively impacts the time and energy savings in comparison to the conventional approach. Furthermore, it is likely that fouling will occur as the mixture of oil, water and surfactant is broken down within the internal structure of the membrane (Trentin et al., 2009). If one aimed to maximise the level of control over droplet formation (at the expense of high dispersed phase flux), the advantages of energy saving are lost due to the long operating time. It is therefore very difficult to produce small, mono-dispersed droplets at a rate that is competitive with current emulsion production technologies. The key to solving this challenge is by ensuring rapid adsorption of surfactant to ensure early droplet detachment and stabilisation of the interface against coalescence. However, conventional approaches lead to membrane coalescence in the majority of cases irrespective of the surfactant type(s) and concentrations used (Wagdare and Marcelis, 2010; Abrahamse et al., 2002).

The aim of this study is to investigate the coupled behaviour between the droplet size of oil-in-water (O/W) emulsions and either the applied transmembrane pressure or the shear rate for a range of surfactant systems. Furthermore, a novel approach to ensure the rapid adsorption of surfactant is presented namely through positioning high hydrophilic–lyophobic balance (HLB), non-ionic surfactants within the dispersed phase rather than their common positioning within the continuous phase. This is subsequently compared with a pre-mix membrane emulsification approach as well as a rotor–stator high shear mixer both in terms of the emulsion droplet size produced but also the rate of production. The study will further understanding of membrane emulsification, enabling process optimisation to reduce droplet size, energy and surfactant consumption whilst maximising production rate simultaneously.

2. Experimental

2.1. Materials

Oil-in-water (O/W) emulsions containing 10 vol.% (unless otherwise stated) of commercially available sunflower oil (SFO) were produced. The aqueous phase was passed through a reverse osmosis unit and then a milli-Q water system. The emulsions were stabilised by a single surfactant in each case. The surfactants

investigated were Tween 20 (polyoxyethylene 20 sorbitan mono-laurate, Sigma Aldrich), Brij 97 (polyoxyethylene 10 oleoyl ether, Sigma Aldrich), SDS (sodium dodecyl sulphate, Fisher Scientific) and hydrolysed lecithin (Cargill). These were either dissolved within the aqueous continuous phase (w) or organic dispersed phase (o). The concentrations are expressed as weight percentages of the whole emulsion system.

2.2. Setup and procedure

2.2.1. Rotating membrane emulsification (RME)

The experiments were performed using a tubular, hydrophilic SPG membrane of 6.1 μm mean pore size (SPG Technology Co. Ltd., Miyazaki, Japan). The membrane dimensions were 10 mm outer diameter and 45 mm length, corresponding to an effective membrane surface area of 14.1 cm^2 . The wall thickness of the membrane was approximately 1 mm. The membrane was mounted on an IKA Eurostar digital overhead stirrer and positioned in the processing vessel. This vessel was interchangeable allowing for two different sizes (inner diameters 20 mm and 60 mm) to be used in order to vary the shear applied at the membrane surface (0.6–12.0 s^{-1} and 52.4–104.7 s^{-1}). This altered the amount of continuous phase within the vessel since the membrane had to be submerged during process operation. Emulsion batch sizes between 25 and 110 g were produced. The membrane rotational speed in each experiment was varied between 100 and 2000 RPM. The transmembrane pressure (TMP) was also investigated in the range of 0.2–1.5 bar (gauge).

The schematic of the RME equipment setup is shown within an earlier publication (Lloyd et al., 2014). For typical emulsification operation, the oil phase (or oil/surfactant blend) was introduced to the inside of the membrane tube at the beginning of the experiment with the opening of the dispersed phase valve. Pressurisation of the dispersed phase storage tank with compressed air enabled the oil to permeate through the membrane to the outer continuous phase. Once the required mass of oil was added, the experiment was stopped by closing the dispersed phase valve.

In the case of pre-mix rotating membrane emulsification, a TMP of 0.5 bar (gauge) was used along with a membrane surface shear rate of 6.0 s^{-1} (1000 RPM and 60 mm diameter vessel). An initial 20 vol.% sunflower oil in water emulsion stabilised by 1 wt.% Tween 20 was formed (denoted Pass 1) and then subsequently passed through the membrane three times into an equal volume of distilled water. Observation of the droplet size decrease with each pass could therefore be observed but not without inadvertently diluting the dispersed phase volume fraction each time (to a minimum of 2.5 vol.% after Pass 4).

2.2.2. High shear mixer (HSM)

Emulsions were also produced using a rotor–stator high shear mixer (Silverson, model L4RT with 21 and 22 mm impeller and screen diameter respectively and 1 mm diameter screen holes). The two phases were introduced within the 60 mm diameter vessel prior to emulsification. The emulsion batch size was 110 g in all experimental runs. The amount of energy input during processing was varied by altering the rotational speed of the impeller between 2000 and 10,000 RPM for 1.5 min, which roughly corresponds to the time required to add the dispersed phase during the membrane emulsification process at 0.5 bar.

2.3. Droplet size measurements

Droplet size distribution of all emulsion samples were measured using a Malvern Mastersizer (United Kingdom) with a hydro 2000 small volume sample dispersion unit. Droplet sizes were

Download English Version:

<https://daneshyari.com/en/article/6665221>

Download Persian Version:

<https://daneshyari.com/article/6665221>

[Daneshyari.com](https://daneshyari.com)