

# Production of sub-micron emulsions by ultrasound and microfluidization techniques

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## Abstract

The purpose of this study was to produce an oil-in-water nano-emulsion by microfluidization and ultrasonication for spray drying encapsulation. Maltodextrin combined with a surface-active biopolymer (Hi-Cap) at a ratio of 3:1 were used as the continuous phase, while dispersed phase consisted of D-limonene. Results showed that microfluidization was an efficient emulsification technique producing small emulsion droplets with narrow distributions compared with conventional emulsifying devices. The main problem was that increasing the microfluidization energy input beyond moderate pressures (40–60 MPa) and cycles (1–2) lead to “over-processing” of emulsion droplets due to re-coalescence. In general, it was not possible to decrease emulsion droplet size below 0.5  $\mu\text{m}$  by microfluidizer. For ultrasound emulsification, increasing the energy input through improving sonication time helped to reduce emulsion size with minimum re-coalescence of new droplets, but the results were depending on the coarse emulsion preparation method.

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## 1. Introduction

It has been well documented that emulsion droplet size<sup>1</sup> (EDS) plays an important role in the retention of volatiles and surface oil content of encapsulated powders during spray drying (Liu, Furuta, Yoshii, & Linko, 2000, 2001; Risch & Reineccius, 1988; Soottitantawat et al., 2005; Soottitantawat, Yoshii, Furuta, Ohkawara, & Linko, 2003). It has been proved that the lower the emulsion size, the higher is the encapsulation efficiency. Accordingly, many emulsion properties such as stability, rheology, and colour, depend on the EDS and size distributions (Becher,

2001; McClements, 2005). Based on EDS, emulsions can be divided into micro- (10–100 nm), mini (nano)- (100–1000 nm) and macro-emulsions (0.5–100  $\mu\text{m}$ ) (Windhab, Dressler, Feigl, Fischer, & Megias-Alguacil, 2005). Nano- (submicron) emulsions are kinetically stable systems that can be transparent (EDS < 200 nm) or “milky” (EDS  $\approx$  500 nm) (Izquierdo et al., 2002; Tadros, Izquierdo, Esquena, & Solans, 2004), and because of their very small EDS and high kinetic stability, they have been applied in various industrial fields, for example, personal care and cosmetics, health care, pharmaceuticals, and agrochemicals (Schulz & Daniels, 2000; Sonnevile-Aubrun, Simonnet, & L’Alloret, 2004).

Production of nano-emulsions by “low-energy emulsification” methods like PIT (phase inversion temperature) technique involves transitional inversion induced by changing factors that affect the HLB of the system, such as temperature, electrolyte concentration, etc., or catastrophic inversion induced by increasing the dispersed phase volume

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<sup>1</sup> In rest of the discussion, instead of using different terms such as droplet diameter, droplet size, emulsion size, etc. which may become confusing, emulsion droplet size or simply EDS will be used.

fraction (Forgiarini, Esquena, Gonzalez, & Solans, 2002; Izquierdo et al., 2002; Solans, Izquierdo, Nolla, Azemar, & Garcia-Celma, 2005; Sole, Maestro, Gonzalez, Solans, & Gutierrez, 2006). These methods have several limitations such as requiring a large amount of surfactants and a careful selection of surfactant–cosurfactant combination, and are not applicable to large scale industrial productions (Seekkuarachchi, Tanaka, & Kumazawa, 2006). On the other hand, “high-energy emulsification” methods such as microfluidization are applicable because of flexible control of EDS distributions, and the ability to produce fine emulsions from a large variety of materials. Stang, Schuchmann, and Schubert (2001) Schultz, Wagner, Urban, and Ulrich (2004), and recently Urban, Wagner, Schaffner, Roglin, and Ulrich (2006) and Seekkuarachchi et al. (2006) provide some good overviews of the high-energy emulsification techniques.

In the interaction chamber of the “microfluidizer”, two jets of crude emulsion from two opposite channels collide with one another (Olson, White, & Richter, 2004; Schultz et al., 2004). The process stream is delivered by a pneumatically powered pump that is capable of pressurizing the in-house compressed air (150–650 kPa) up to about 150 MPa (Microfluidics, 2003). Forcing the flow stream by high pressure through microchannels toward an impingement area creates a tremendous shearing action, which can provide an exceptionally fine emulsion. In general, inertial forces in turbulent flow along with cavitation are predominantly responsible for droplet disruption in microfluidizer (Dalglish, Tosh, & West, 1996; Maa & Hsu, 1999; Robin, Blanchot, Vuillemand, & Paquin, 1992; Schultz et al., 2004). There are many studies regarding the application of microfluidizer in the homogenization of milk and dairy model emulsions (Dalglish et al., 1996; McCrae, 1994; Olson et al., 2004; Robin et al., 1992, Robin, Remillard, & Paquin, 1993, 1996; Strawbridge, Ray, Hallett, Tosh, & Dalglish, 1995). Some workers believe microfluidization is superior because, EDS distributions appeared to be narrower and smaller in microfluidized emulsions than in the traditional emulsifying devices (Dalglish et al., 1996; Pinnamaneni, Das, & Das, 2003; Robin et al., 1992; Strawbridge et al., 1995). It is shown, however, microfluidization is unfavourable in specific circumstances such as higher pressures and longer emulsification times, as it leads to “over-processing”, which is re-coalescence of emulsion droplets and an increase in EDS (Jafari, He, & Bhandari, 2006, 2007; Lobo & Sverika, 2003; Olson et al., 2004).

By the advent of modern emulsification systems and their potential application in encapsulation of food ingredients, understanding the mechanisms of emulsification and the behaviour of emulsion components along with the knowledge of factors affecting the emulsion properties during emulsification is essential. Also, there has been a limited work to produce emulsions in sub-micron area with a narrow distribution for nano-particle encapsulation. In fact, most of the published works in the emulsion territory are dealing with pure emulsions consisting water, oil and emul-

sifier. While in emulsification for subsequent encapsulation purposes, there is another constituent involved, the so-called wall material or encapsulation matrix, which is mainly a biopolymer and has some direct and indirect influences on the emulsion properties. Therefore, the objectives of this work are to determine the optimum emulsification conditions and investigate the emulsion properties during extreme emulsification conditions of microfluidization and ultrasonication.

## 2. Materials and methods

### 2.1. Materials

D-Limonene ( $\rho = 840 \text{ kg/m}^3$ , RI = 1.487) was supplied by Quest International (NSW, Australia). Modified starch (Hi-Cap 100, waxy corn starch-modified, 5% moisture, solubility > 90%) and maltodextrin (DE = 16–20, 5% moisture, bulk density =  $600 \text{ kg/m}^3$ ) were purchased from National Starch and Chemical (Sydney, Australia), and Penford Limited (NSW, Australia), respectively. Distilled water was used for the preparation of all solutions. All general chemicals used in this study were of analytical grade.

### 2.2. Coarse emulsion preparation

All emulsions were produced in two stages, as described in our previous study (Jafari, He, & Bhandari, 2007): (a) pre-emulsions were obtained with a high-speed blender (RW 20.n, IKA Works, Malaysia), or a rotor-stator system (L2R, Silverson Machines Ltd., UK). Silverson is a typical colloid mill with a stator composed of a metal grating in which, 2 mm holes are drilled. (b) The coarse emulsions were then further emulsified using a microfluidizer or an ultrasound probe. Sodium azide (0.02 wt%) was added to the emulsions as an antimicrobial agent. The concentration of dispersed phase in emulsions was expressed in terms of dispersed-phase volume fraction ( $\phi$ ).

### 2.3. Microfluidization

Previously prepared coarse emulsions (at room temperature) were passed through an air-driven microfluidizer (Model M-110 L, Microfluidics, USA), as described in our previous work (Jafari et al., 2007). Pre-emulsion was fed to the system through a 200 mL glass reservoir. The device splits the pre-emulsion feed into two opposing channels in a stainless steel block (a ceramic interaction chamber); these channels narrow to approximately  $75 \mu\text{m}$  in width, and the two jets of pre-emulsion are forced to collide head-on at high pressure, creating extreme shear. Through mechanical amplification of  $\times 232$ , the typical pressure of the liquid jets flowing through the channels is about 120 MPa when the air pressure at the regulator is 530 kPa. The volume flow rate of the emulsions was measured and it was approximately  $4 \times 10^{-6} \text{ m}^3/\text{s}$  at 60 MPa for one cycle. The experiments were duplicated.

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