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Development of microfluidization methods for efficient production of concentrated nanoemulsions: Comparison of single- and dual-channel microfluidizers

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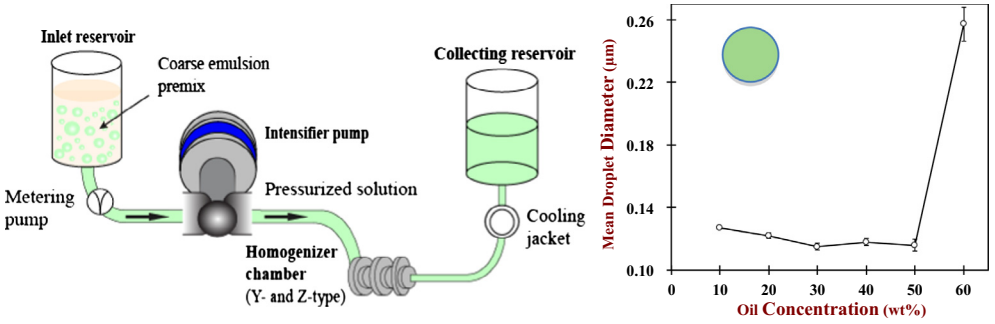
GRAPHICAL ABSTRACT



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ABSTRACT

Nanoemulsions are being increasingly utilized within the pharmaceutical, food, personal care, and chemical industries because of their unique physicochemical properties and functional performances: high optical clarity; prolonged stability; enhanced bioavailability; and novel rheology. For commercial applications, it is important to be able to produce nanoemulsions containing small droplets using efficient homogenization processes. In this study, we compared two microfluidization methods for fabricating nanoemulsions: (i) single-channel microfluidization and (ii) dual-channel microfluidization. The influence of emulsifier concentration, homogenization pressure, disperse phase volume fraction, and initial emulsifier location (oil *versus* water phase) on particle size was examined. For both devices, the mean particle diameter decreased with increasing emulsifier concentration and homogenization pressure, and there was a linear log–log relationship between mean particle diameter and homogenization pressure. At a similar emulsifier level and homogenization pressure, dual-channel microfluidization produced smaller droplets and narrower distributions than single-channel microfluidization. This effect was attributed to a higher droplet disruption efficiency and/or lower droplet recoalescence rate for the dual-channel system. The dual-channel method could successfully produce nanoemulsions even at high oil concentrations (50%), whereas the single-channel method was only effective at producing nanoemulsions at relatively low oil concentrations (10%). This study demonstrates that dual-channel microfluidization is an efficient means of producing fine nanoemulsions with high oil loading levels, which may be advantageous for many commercial applications.

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1. Introduction

Homogenization is a powerful technique for incorporating non-polar functional components, such as hydrophobic drugs, bioactive lipids, antioxidants, antimicrobials, preservatives, flavors, or colors, within an aqueous medium [1]. Indeed, oil-in-water emulsions produced by homogenization are central components in a diverse range of aqueous-based products, including foods, beverages, pharmaceuticals, and personal care products [2]. Recently, there has been a growing interest in the utilization of nanoemulsions as delivery systems for non-polar functional components because their small particle size and high surface-to-volume ratio leads to prolonged stability, higher optical clarity, and increased bioavailability [3–5]. Oil-in-water nanoemulsions contain small oil droplets (radius < 100 nm) dispersed within a continuous aqueous phase, with each oil droplet being surrounded by a protective coating of emulsifier molecules [6,7]. Nanoemulsions can be fabricated using either low-energy or high-energy approaches, which can be classified based on the physicochemical mechanisms involved [8]. Low-energy approaches utilize the intrinsic properties of the emulsifier, oil, and water system to spontaneously form nanoemulsions based on changing system composition or environmental conditions in a specific way [9–11]. Low-energy approaches, such as the spontaneous emulsification, emulsion phase inversion, and phase inversion temperature methods, are inexpensive and simple to implement because they require no specialized equipment, and they are also highly effective at producing small droplets for certain combinations of oils and emulsifiers [12]. However, there are many oil and emulsifier types that cannot be utilized in this approach, and the high levels of surfactant typically required to produce nanoemulsions lead to concerns about ingredient costs, off flavors, and safety, especially in food and beverage applications [13].

High-energy approaches, such as those based on high-pressure homogenization, allow more flexibility in nanoemulsion formulation [14,15]. Typically, these approaches use mechanical devices capable of generating intense disruptive forces to break up the oil and aqueous phases, leading to the formation of fine oil droplets dispersed in water [16,17]. Of the various high-energy approaches available, microfluidization has been shown to be one of the most efficient at producing small droplets with uniform particle size distributions [13,17]. Nanoemulsions are typically produced using a two-step procedure when using the conventional single-channel microfluidization method [18]. Initially, a high shear mixer is used to form a coarse oil-in-water emulsion by blending oil, emulsifier, and water together. Then, this coarse emulsion is forced through an interaction chamber in the microfluidizer using pneumatic pressure (Fig. 1). After the coarse emulsion enters the homogenizer it is divided into two streams that flow through narrow channels toward an impingement area at high velocities [19]. The channels are designed so that the two streams of coarse emulsion collide with each other, thereby generating intense disruptive forces (cavitation, turbulence, and shear) that lead to production of very fine oil droplets [20]. Previous studies have shown that the mean particle size and size distribution produced by a microfluidizer tend to be smaller and narrower than those produced by other homogenization devices [21].

Conventional microfluidizers can be described as two-step single-channel devices because the premixed coarse emulsions are fed into the microfluidizer from a single inlet reservoir [22,23]. Despite their effectiveness at producing nanoemulsions, there are a number of potential limitations associated with this type of microfluidizer [24]. First, the manufacturer needs to premix the oil and water phases using a high-shear mixer to prepare the initial coarse emulsion [25], which requires additional equipment, time, and expense. Second, in a batch microfluidization process, some of the coarse emulsion is wasted because the microfluidizer typically needs to be rinsed before collecting the final product, thereby lowering the yield. Third, the maximum amount of oil that can be incorporated into a nanoemulsion is often limited in single-channel microfluidizers because the high viscosity of concentrated coarse emulsions makes them difficult to force through the device [26]. Therefore, it would be useful to develop a more efficient microfluidization method to produce nanoemulsions.

In this research, a one-step dual-channel microfluidization method is used to efficiently produce nanoemulsions by separately feeding oil phase and aqueous phase into the microfluidizer. This approach is capable of not only saving the time, expense, and labor associated with producing a coarse emulsion premix, but also in reducing the amount of material wasted. In addition, this technique only requires a single pass of material through the homogenizer (rather than the multiple passes often required for conventional homogenizers), which could reduce time and energy costs. Furthermore, it may be possible to produce nanoemulsions with high final oil contents because it is not necessary to pass a highly viscous emulsion premix through the device.

The objective of the present study was to compare the single- and dual-channel microfluidization methods for fabricating nanoemulsions with similar compositions. The influence of emulsifier concentration, homogenization pressure, and oil concentration on the mean particle size and particle size distribution were investigated to better understand the difference between these two methods. The information obtained from this study will prove useful for the design and fabrication of functional nanoemulsions suitable for utilization within the pharmaceutical, personal care, food, chemical and other industries.

2. Experimental

2.1. Materials

Medium chain triglycerides (MCT, Miglyol 812N) were purchased from the Warner Graham Co. (Cockeysville, MD). Non-ionic surfactant, polyoxyethylene (20) sorbitan monooleate (Tween®80), and sodium phosphate monobasic were purchased from Sigma–Aldrich Co. (St. Louis, MO). Double distilled water (Milli-Q) was used to prepare all solutions and nanoemulsions.

2.2. Emulsion preparation

Oil-in-water nanoemulsions were prepared by homogenizing oil phase (MCT, 10–60 wt%) with aqueous phase (40–90 wt%, containing 0.5–6 wt% emulsifier). The aqueous phase consisted of emulsifier (Tween®80) and buffer solution (10 mM sodium phosphate, pH 7.0). Two different methods were used to manufacture nanoemulsions: single- and dual-channel microfluidization (Fig. 1).

Single-channel method: A coarse emulsion premix was prepared by blending lipid phase and aqueous phases together using a high-speed mixer (Bamix, Biospec Products, Bartlesville, OK) for 2 min at room temperature. Nanoemulsions were formed by passing the coarse emulsions through an air-driven microfluidizer with a Y- and Z-type interaction chamber (Microfluidics PureNano, Newton, MA, USA). The coarse emulsions were fed into the microfluidizer through a 100 ml glass reservoir (pressurized by an intensifier pump, flow rate 500 ml per min), and were passed through the homogenization unit for one pass at various homogenization pressures (9–19 kpsi).

Dual-channel method: The lipid phase and aqueous phase were fed into two different 100 ml glass reservoirs. Fine emulsions were formed by simultaneously forcing the lipid phase and aqueous