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Invited review paper

Precision emulsification for droplet and capsule production

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ABSTRACT

Emulsions and microcapsules are typical structures in various dispersion formulations for pharmaceutical, food, personal and house care applications. Precise control over size and size distribution of emulsion droplets and microcapsules are important for effective use and delivery of active components and better product quality. Many emulsification technologies have been developed to meet different formulation and processing requirements. Among them, membrane and microfluidic emulsification as emerging technologies have the feature of being able to precisely manufacture droplets in a drop-by-drop manner to give subscribed sizes and size distributions with lower energy consumption. This paper reviews fundamental sciences and engineering aspects of emulsification, membrane and microfluidic emulsification technologies and their use for precision manufacture of emulsions for intensified processing. Generic application examples are given for single and double emulsions and microcapsules with different structure features.

Contents

1. Introduction

Droplets, particles and capsules sized in the range of micro- and nanometres are popularly used for delivery of functional or active components in a wide variety of products or as construction elements for more complex structures [1]. Droplets in emulsions are most common structure in pharmaceutical, food and cosmetic products for controlled delivery of drug or nutrition for the amount and pattern desired. Simple or complex solid particles/capsules manufactured from liquid droplets are often found in personal and household care products for delayed or triggered (such as self-healing) releases. Precise manufacture of these particulates is in the centre of designed delivery for better quality of products and of intensified processing.

The manufacture of emulsion droplets involves a bulk liquid phase breaking up and being stabilised in another immiscible liquid phase. Conventional methods of emulsification create

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droplets through vigorous shear forces exerted through either strong turbulence driven by high speed/pressure or cavity generated by ultrasounds. The disperse and continuous phases are mixed together before the operation and the disperse phase deforms and breaks randomly in the continuous phase. The force field, physical properties of the disperse, continuous phase and interface govern the droplet formation and further on the size and size distribution, among them the interfacial tension and viscosities are the most sensitive properties. Prolonging the emulsification time or generating a more vigorous hydrodynamic shear force field is the immediate solution used to achieve smaller and more uniform emulsion droplets. The vigorous hydrodynamic force is highly randomly distributed at a micrometre range in terms of direction and intensity for droplet size reduction, so that these methods often exhibit limitations in the control of emulsion droplet size, size distribution, reproducibility and with very high energy consumption. They can also pose serious limitations if thermal or shear-sensitive materials have to be processed.

This review addresses to the challenge of precision and intensified manufacture of droplets through drop-by-drop methods at high energy efficiency. Droplet formation and stabilisation and process scale-out are discussed.

2. Droplet formation

2.1. Droplet formation in a drop-by-drop manner [2]

A minimum energy required for an emulsification process, theoretically, could be the sum of surface energy change of droplet formation of an emulsion if the droplet were manufactured in the most efficient way. Droplet manufacture from a bulk liquid composes two key events: disperse phase deformation and breakup into smaller droplet. The process is normally carried out in the presence of a stabiliser in order to prevent coalescence of the droplets formed. In the last one or two decades, many methods have gradually been developed or under development to produce droplets in a highly ordered drop-by-drop manner to precisely manufacture the liquid droplets with desired sizes, structures and minimum energy consumption.

The drop-by-drop ordered feature is distinctly different from random deformation and breakup in a disordered force field of conventional methods. Fixed geometries of pores, capillary or gaps in micrometres, called microfluidic devices, are used to shape or confine the disperse phase before meeting the continuous phase and forming a droplet. The microfluidic devices such as membranes (Fig. 1a, with a large number of micropores) and microchannel fluidics (Fig. 1b, with a single pore [3]) provide control mechanisms for the flow of both the disperse and continuous phases. Such well controlled flows make it possible subscribe the size and size distribution of a population of droplets repeatedly at a low energy supply.

The flow rate and relative flow of the disperse and continuous phases are often the major operational control mechanisms for the droplet formation. As the flow rate increases, the disperse phase flow in a small space (capillary) can be categorised as three distinct regimes of squeezing, dripping and jetting of formation of droplets as capillary number (C_a) of the flow increases. Capillary number represents a relative effect of viscous force to interfacial tension:

$$C_a = \frac{\mu V}{\gamma} \tag{1}$$

where μ is the viscosity of the liquid, V a characteristic velocity and γ interfacial tension.

In the squeezing and dripping regimes, the flow velocity is slow so that capillary numbers (a rule of thumb says less than 10^{-5}) are dominated by interfacial tension of the fluid. The droplet formation follows the drop-by-drop mechanism, while the jetting regime occurs only at very high flow rates, or with very low interfacial tension similar to the conventional jetting process. This region is out of interest of this review.

In the squeezing and dripping regions, the drop-by-drop formation process is normally composed of the breakthrough of the disperse phase into and out of the pore/orifice, droplet growth through the influx of the disperse liquid and detachment/breakup of a droplet from pore and the feeding disperse liquid stream. Droplet growth is dominated by the influx of the disperse liquid when the flow rate is constant. If the flow is driven by a constant

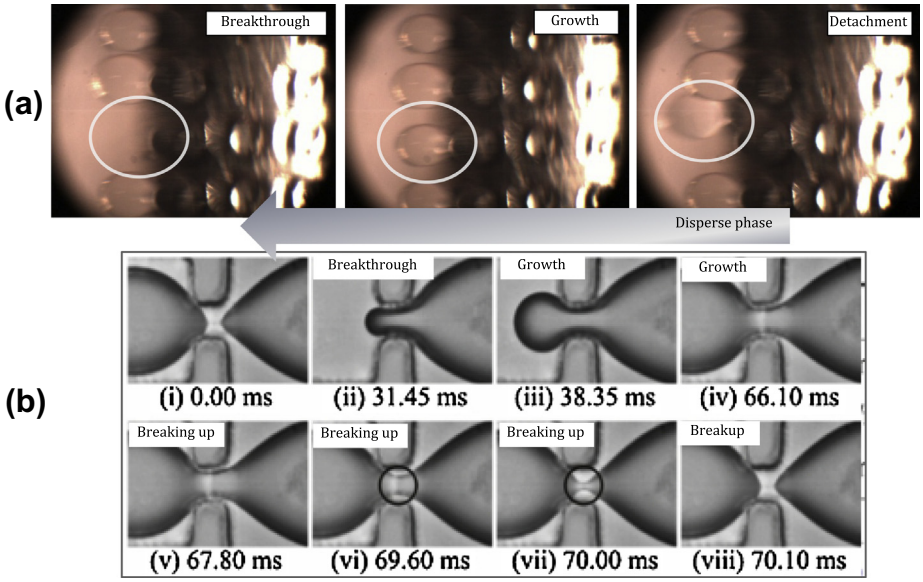

Fig. 1. Three stages of droplet formation process of breakthrough, growth and detachment (breakup) in a drop-by-drop manner in (a) membrane and (b) focused-flow microfluidic [3] emulsification. Reproduced from Ref. [3] with permission from Springer.