Emulsification Using EDGE (Edge based Droplet GEneration) Devices

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Abstract

Spontaneous emulsification through microchannels, in which droplets are formed due to Laplace pressure difference, is a relatively new technique for preparing monodisperse emulsions. The technique is extremely suited for products containing temperature and shear sensitive ingredients and for products that require high monodispersity; however, industrial application is still not possible due to low production rates. We have recently introduced a new spontaneous droplet formation technique called EDGE (Edge-based droplet generation) with which multiple monodisperse droplets can be produced simultaneously from a single droplet formation unit (DFU). An EDGE-unit is a slit like structure called plateau located between an oil and a continuous phase channel. Monodisperse droplets are successfully produced at several locations along the entire length of the plateau. The system is robust and stable under acceptable pressure range. Investigations on an up-scaled version (chip of 1.5 cm^2) showed that all the DFUs were active, further scale-up is currently being investigated.

Keywords

Spontaneous emulsification, EDGE systems, monodispersed emulsions

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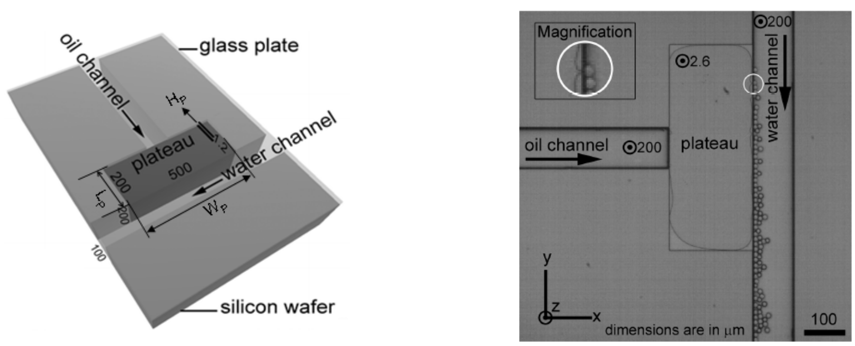
1. Introduction

Emulsions play an important role in various industries including pharmaceutical, petrochemical, cosmetics and food industries (Egidi et al., 2008). Characteristics of the emulsions including rheology, appearance, chemical reactivity and physical stability are all affected by the size of the droplets and distribution of droplet sizes (Sugiura et al., 2002c, Sugiura et al., 2002b). Traditional equipments used for emulsions preparation (high pressure homogenizers, ultrasound homogenizers and rotor-stator systems) produce polydisperse emulsions with a wide range of droplet sizes. Furthermore, they apply a high shear stress to deform and disrupt the large droplets which can cause the shear sensitive ingredients (starch, proteins etc.) to lose their functional properties (Charcosset et al., 2004, Sugiura et al., 2002a).

Microchannel emulsification is a relatively new technique for preparing monodisperse emulsions. The distinguishing feature of this technique is that no shear forces are needed to form droplets, and further, the size of droplets can be controlled by microchannel geometry. Droplet formation takes place spontaneously due to Laplace pressure differences and less energy (a factor of 10-100 less) as compared to the conventional techniques is needed (Sugiura et al., 2001), for the production of various products such as O/W emulsions, W/O emulsions, lipid microparticles, polymer microparticles or microcapsules (Sugiura et al., 2004). Industrial application of microchannel emulsification is still not possible due to low production rates.



(a) **(b)**

Figure 1: (a) Schematic three dimensional representation of a typical EDGE system (b) droplet formation through a typical EDGE device (van Dijke et al., 2010a).

We have recently introduced a new spontaneous droplet formation technique called EDGE (Edge based Droplet GEneration) (van Dijke et al., 2010c) with which we are able to produce multiple monodisperse droplets simultaneously from a single droplet formation unit. An edge unit is a large slit like structure called plateau located between an oil phase and a continuous phase channel (see figure 1a). The droplet formation from a typical edge device is shown in figure 1b. On applying pressure, the dispersed phase flows through the oil channel and spreads over the plateau. On reaching the edge of the plateau, it forms monodispersed droplets at several locations along the entire length of the edge. This paper aims to provide an overview of EDGE emulsification systems including their working principle and suitability for scale-up of spontaneous emulsification.

2. Theory

The pressure needed for dispersed phase to flow onto the plateau is determined by Laplace's law given as van Dijke *et al*., (2010c):

$$\Delta P = \sigma \cos\theta \left(\frac{1}{R_1} + \frac{1}{R_2} \right) \approx \frac{\sigma}{R_1} \qquad (1)$$

Where σ is the interfacial tension, R_1 *and* R_2 are the radii of curvature of interface and θ is the contact angle. For simplicity reasons we assume contact angle to be 0° and because the curvature R_2 is very large as compared to R_1, the minimum pressure needed here is determined by the smallest curvature in equation 1. If applied pressure exceeds this value, the oil flows over the plateau and droplet is formed at the edge; which remains connected to the plateau through a neck. Growth of the droplet results in a lower Laplace pressure in the droplet (ΔP=2 σ/R_{drop}). As long as the amount of oil flowing into the droplet does not exceed the amount of oil flowing into the neck from the plateau, the droplet remains attached otherwise the neck breaks and droplet flows into the continuous phase channel.

3. Methodology

Emulsification process is observed under a microscope attached to a high speed camera. EDGE unit is etched with the Deep Reactive Ion Etching (DRIE) on a silicon microchip 1.5×1.5 cm. An EDGE unit consists of a dispersed and a continuous phase channel (200 µm in width and depth). In between the channels, a large flattened area called plateau (with 200 µm length, 500 µm width and 1.2 µm depth) is placed as shown in figure 1a. Microchip is fixed in a chip holder and placed under the microscope. Continuous phase containing surfactant, is supplied through a syringe pump and dispersed phase is supplied through a digital pressure controller (Bronkhorst, The Nethelands). The emulsification is visualized using a high speed camera connected to the microscope. Captured images are then analyzed with image analysis software (Image Pro Plus).

4. Results and discussions

Oil-in-water emulsions were prepared using hexadecane as a dispersed phase and SDS (1%) in MilliQ ultra pure water as continuous phase. The resultant droplet diameters (d) and coefficient of variation (CV) are shown in figure 2. At low pressures, droplet diameter and CV remain constant. When applied pressure exceeds certain critical value, both droplet size and CV increase rapidly. Plateau, having the highest hydrodynamic resistance is the main factor which determines the droplet size in pressure independent range. The droplet size scales with the height of the plateau and typically the droplets are 6 times the plateau height. The size of the droplets is not sensitive to small pressure changes and this is an important advantage over other micro emulsification devices. Each unit can be considered as self regulating. The operation of the system was straightforward; plateaus filled with oil smoothly even in the presence of disturbing factor (e.g., dust particles). Overall the system is very stable and is suitable for practical applications (van Dijke et al., 2010c).

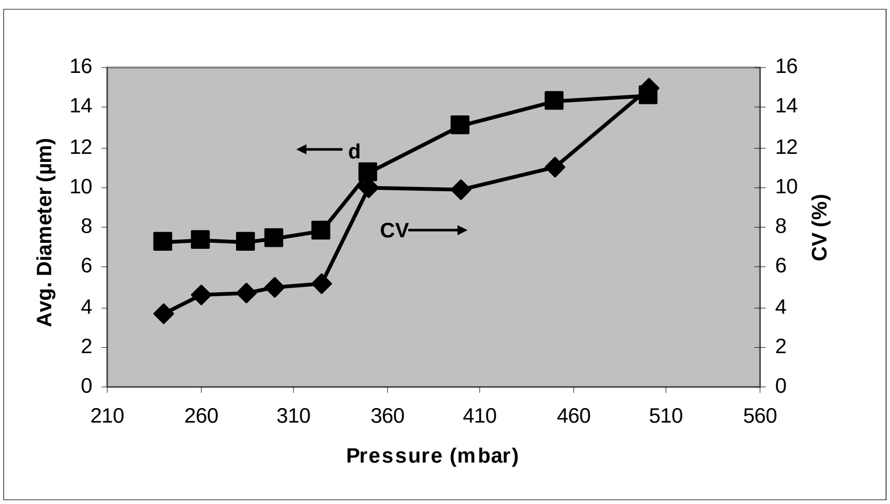
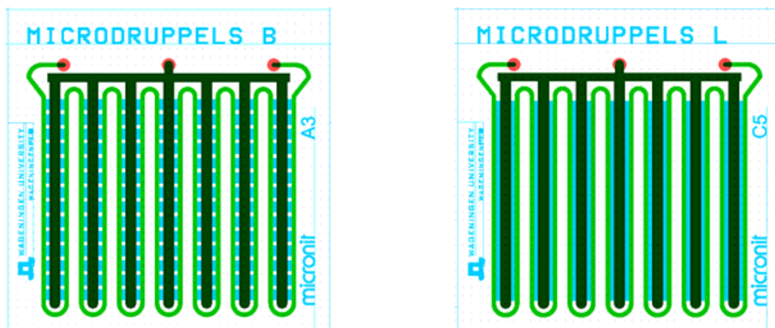


Figure 2: Average droplet diameter and CV as a function of applied pressure.

In order to scale-up the process of emulsification edge units were parallelized in two different designs (see figure 3). In first design (EDGE-R) 196 DFUs (rectangular shaped) with dimensions 200 × 500 × 1.2 μm (length × width × depth) were placed parallel to each other along seven rows of oil channels (dark green). In second design, (EDGE-W) 14 elongated plateaus (200 × 9500 × 1.2) were placed along seven rows of oil channels in order to increase the droplet formation length and thus the productivity. Plateaus are surrounded by water channels (light green) in which water flows from one end, takes droplets from the plateaus, and leaves the system on the other end.



a **b**

Figure 3: Parallelized EDGE systems (1.5 × 1.5 cm microchip) (a) EDGE-R (b) EDGE-W

In both designs, activity of DFUs was 100 %, and also the long plateaus gave droplets over the entire length of the structure. All the units filled completely with oil when minimum required Laplace pressure was reached and regular sized droplets were produced along the edge. The systems were stable to pressure changes within the pressure independent range. The system was subjected to long term operation by running for three consecutive days and we did not observe any change in droplet size as a function of time (van Dijke et al., 2009).

Conclusion and outlook

We are now able to produce multiple monodisperse droplets simultaneously from a single droplet formation unit using EDGE systems. The system is simple in design (only the height of the structure is essential), and operation and can be successfully parallelized. Stability under pressure changes and suitability for long term operation make it an appropriate candidate for scale up of spontaneous emulsification.

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