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Millimetric core–shell drops via buoyancy assisted non-confined microfluidics

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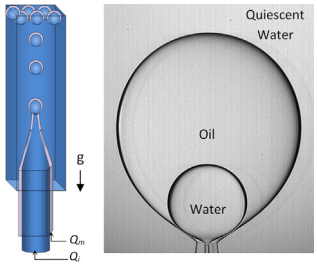
HIGHLIGHTS

- Uniform millimetric core–shell drops were produced with the assistance of buoyancy.
- A model was presented which was validated against surfactant-free conditions.
- The presence of surfactants reduced the effect of buoyancy on drop formation.
- Drops with thinner shells were obtained in the presence of surfactants.
- The transitions to jetting could be monitored by the deviation from $We+Bo \approx 1.0$.

GRAPHICAL ABSTRACT



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ABSTRACT

Core–shell drops having millimetric dimensions have attracted a wide range of applications. Such drops cannot be achieved in typical microfluidic devices due to buoyancy-driven phase separation difficulties at low flow rates. A buoyancy assisted approach based on two-phase flow (core and shell phase) with the outer phase as quiescent and non-confined, is presented for producing large core–shell drops. Buoyancy was found to be the dominant force for drop formation in the surfactant-free system, but considerably less effective in the presence of surfactants. The drop formation was mostly limited to the dripping regime when the surfactant concentrations were low. A simple force-balance model is developed for the prediction of the core-shell and the core drop sizes, which are validated against the data obtained in a surfactant-free system. The suggested method gives highly monodisperse (coefficient of variation smaller than 3%) core–shell drops (radius $R \sim 800$–$3000\ \mu m$) with a wide range of absolute ($t \sim 30$–$1000\ \mu m$) and relative ($t/R \sim 0.03$–0.80) shell thickness.

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1. Introduction

Uniform core–shell drops made by micro-capillary based techniques (Utada et al., 2005; Chang et al., 2009) have found numerous applications within biomedical and pharmaceutical fields such as micro-reactors (Shum et al., 2009), delivery vehicles in the form of liposomes (Shum et al., 2008) or microcapsules (Chen et al., 2012), and controlled content release (Abbaspourrad et al., 2013) using structures having ultra-thin shells (Kim et al., 2011). The size of the drops decides its viability for any application. For certain purposes such as cell manipulation (Chang 2004) and encapsulation (Uludag et al., 2000; Lewinska et al., 2008; Bremond et al., 2010), large core–shell drops are required. Typical confined microfluidic approaches for the formation of core–shell drops via co-flow (Chang et al., 2009; Saeki et al., 2010) or flow-focused (Shum et al., 2009; Vladisavljevic et al., 2012; Erb et al., 2011) techniques produce rather small-sized drops. In

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such approaches, the drop size becomes less dependent on the inner and middle phase flow rates and is mainly controlled by the outer phase flow (Chang et al., 2009), due to a high drag. To produce large drops, a small outer phase flow rate is usually required. In both co-flow and flow-focusing approaches, application of low outer phase flow rates becomes non-feasible due to the problems associated with drop accumulation in the dead zones resulting mainly from gravity effects. To avoid this drawback, capillaries should be set up vertically to allow for continuous generation and buoyancy-driven transportation of resulting large drops.

Vertically orientated co-axial capillary setups have previously been employed to produce core–shell structures. Berkland et al. (2007, 2004) used a coaxial setup in which the inner and middle phases were jetted to create a coaxial jet stream in a carrier phase, which was then acoustically disturbed to create core–shell droplets. This technique was reported to give high control over drop size and shell thickness, but produced small droplets. Manukian et al. (2008) also used a concentric nozzle setup, oriented vertically, to produce a core–shell compound jet in the outer phase, which then ruptured without external disturbance to form core–shell drops. This process was used to commercially produce large core–shell structures, known as Hydrocapsules, ranging from 200 μm to 10 mm in diameter. However, drop formation from a jetting stream of liquid is usually associated with a low degree of droplet uniformity (Chen et al., 2013). A vertically orientated co-flow microfluidic device with outer phase flow has also been employed to produce millimetre-scaled core–shell structures (Shao et al., 2013). The presence of high outer phase flow, however, limited the drop size to a considerable degree. In all the cases discussed above, either the effect of buoyancy was suppressed (due to either the external disturbances or the drag force caused by the outer phase flow), or the monodispersity was compromised because the drops were formed due to the uncontrolled rupture of a compound jet.

Gaitzsch et al. (2011) explored the formation of pendant water-in-oil-in-water droplet under the effect of buoyancy. However, the focus of this study was on the mechanism of expulsion of the water core from the pendant double emulsion due to partial coalescence in a surfactant-free system. A similar vertical setup has also been used to form multiple emulsions for studying their behaviour via electrophoretic manipulation (Schoeler et al., 2014), which is an easy and efficient method of transporting an oil phase through an oil environment. Recently, Schmit et al. (2014) reported the formation of millimetre-sized double emulsions via a pendant drop method. This method, where the core was first formed at the tip of the inner nozzle, allowed a good control over the number of core droplets (Chang et al., 2009). The core droplets then entered a pendant drop, which later detached from the outer tip (kept far ahead of the inner nozzle tip) under the influence of gravity, as shown in Fig. 1a.

However, using such a two-step device prevents the formation of drops with large core and ultra-thin shelled drops, especially on the millimetre scale (Shao et al., 2013). Furthermore, keeping the inner capillary tip far behind the outer tip usually leads to a lower uniformity of the core droplets due to the difficulties involved in the frequency matching of the core and the core-shell drop formation (Shao et al., 2013).

In this work, we report the use of a vertically oriented non-confined microfluidic device where buoyancy effect is significant. Generally the term “microfluidic” applies to devices that have at least one or more components (drop size, capillary tip size, etc.) below a millimetre scale (Nguyen and Wereley, 2006). This justifies the use of the term “non-confined microfluidics” for our system that employs two sub-mm dimensions (glass capillaries) and a third dimension (cuvette) that can vary between the diameter of drops and infinity. We kept the inner and the outer capillary tips at the same level to facilitate the formation of millimetric water-in-oil-in-water core–shell drops (Fig. 1b) with better control over shell thickness. The capability of accommodating multiple

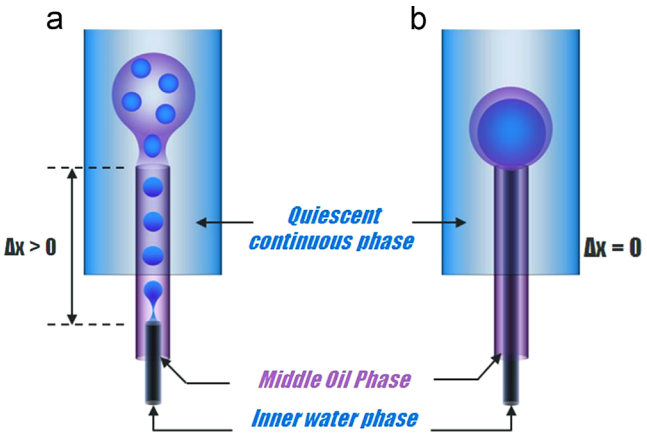

Fig. 1. The schematic illustration of the difference in the mechanism of core shell formation under the influence of gravity when (a) the inner capillary tip is placed far behind the outer capillary tip, and when (b) both are kept at the same level.

capillaries in a non-confined environment that eliminates the difficulties associated with micro-capillary alignment in a restricted space as experienced in common microfluidic devices (Chang et al., 2009; Vladisavljevic et al., 2012; Shao et al., 2013), the elimination of the third pumping mechanism required for the outer phase due to a non-confined environment, and the simplicity of buoyancy assisted transport of drops and their facile collections makes this design easy and economical for scale up.

In the proposed system, drag acts as a cohesive force, albeit its magnitude has been found to be negligible as compared to the dominant interfacial and buoyancy forces (Chaurasia et al., 2015b). This extends the flow conditions (thus increases the productivity) under which uniform droplet formation via dripping occur, which is associated with high uniformity of droplets. It is also shown that the core-shell drop and the core sizes can be predicted by a force-balance model, which is validated against a surfactant-free system. We demonstrate the production of core-shell drops having large sizes, between 1.6 and 6.0 mm in diameter.

2. Experimental

2.1. Materials

Octane (Sigma-Aldrich, 98%, density: 702 kg/m^3, viscosity: 0.52 mPa s) was used as received as the middle oil phase. Sodium dodecyl sulphate (SDS; 98.5%, Sigma-Aldrich) and Span85 (Sigma-Aldrich) were used as surfactants in the outer water and middle oil phase, respectively. De-ionised water (density: 998 kg/m^3, viscosity: 1 mPa s) was used as the inner water phase.

2.2. Device

The schematic of the non-confined microfluidic device is shown in Fig. 2a. Two glass capillaries, circular (ID: 0.55 mm, OD: 1 mm) and square (IL: 1 mm, OL: 1.5 mm), were pulled using a pipette puller (P-1000, Sutter Instrument, Novato, USA). The tapered tips were cut to the desired sizes. The cross-sectional shape of the outer square capillary tip turned circular (ID: 290 μm, OD: 335 μm) after being pulled (Chaurasia et al., 2015a), while the inner capillary tip retained its circular cross-section (ID: 46 μm, OD: 70 μm). The outer capillary's inner surface was made hydrophobic by treating it with *n*-Octadecyltrimethoxysilane, while its outer surface was kept hydrophilic by plasma cleaning (Femto Plasma cleaner, Diener). The inner capillary was treated to be hydrophobic throughout.

The inner capillary was introduced in the outer capillary and was axially aligned with both the tips placed at same level (Fig. 2b).

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