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VISUALIZATION OF EMULSION GENERATION MECHANISM IN PORE-NECK GLASS MODELS

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Abstract. *Conjoint water and oil flow during petroleum production operations in oil reservoirs is frequently accompanied by emulsion co-production at the surface, affecting well productivity, artificial lift and separation process, depending on emulsion's morphology. Flow conditions responsible for generation of emulsions present at the surface are uncertain. The present work focuses on the study of oil-in-water emulsion generation in porous media. Constricted circular capillaries with a converging-diverging section were used to physically simulate a pore body-neck-body section. The snap-off of oil drops was visualized, using an optical microscope. Low Reynolds Numbers were set in experiments. Three drop/neck diameter ratios, three oil/(water+surfactant) systems and three drop/continuous phase viscosity ratios (k), were used. Visualization results indicate that drop break up and produced drop diameters depend upon parameters such as oil drop/continuous phase viscosity ratio, capillary number (Ca), continuous phase fraction, capillary's geometry, and drop/capillary diameter ratio. In some cases, produced drops were smaller than 10 micrometers. This results suggests that emulsions with very small drops (a problem in separation process) may be produced in porous media, i.e. before the fluids enter in contact with choke valves or are exposed to turbulent flow down hole.*

Keywords*. Emulsion generation, oil production, porous media, snap-off, constricted capillaries, visualization.*

1. Introduction

Dispersions are systems made up of minimum of two immiscible phases, where the least one of the phases is dispersed within a second, continuous phase. There are 3 different types of dispersions that can be formed with a liquid as the continuous phase and one dispersed phase: foams (gas-liquid), emulsions (liquid-liquid), and suspensions (solid-liquid). Dispersions may be classified according to the features size of the dispersed phase (droplets, bubbles or grains) in molecular solutions, colloids and dispersions. In this work, we concentrate on emulsions, i.e. liquid-liquid systems. Emulsions are generally stabilized by addition of a surfactant, which acts by reducing the interfacial tension between the two liquid phases involved, and by creating repulsive (steric) forces between droplet, to limit coalescence. Emulsions are frequently encountered in industrial processes such as manufacture of adhesives, inks, foods, cosmetics, controlled dosage medicines and in practically all production stages in the oil petroleum.

Emulsions can be produced spontaneously or in controlled ways. Controlled emulsion production involves shear or physico-chemical processes, separately or combined. Spontaneous emulsion production may occur, for instance, during events of oil production, because oil and water phases of certain composition enter in contact. Such phase components are some times associated with the generation of natural surfactants, from long chain of carbonated acid present in oil that reacts with ions present in water. According to Davies G. A. *et al.* (1996) and Sarbar, M.A. *et al.* (1997), such emulsions may be stabilized, additionally, by the presence of other oil components, typically heavy oil fractions such as asphaltenes and possibly suspended solids.

The emulsion formation during oil production may take place in the flow inside the reservoir or in the two-phase flow down hole in production wells, and through tubes and valves in surface facilities. Emulsions cause a number of operational problems such as tripping of equipment in gas-oil separations plants (GOSPs) and high pressure drops in flow lines (Kokal *et. al*., 1999). Uncontrolled and undesired emulsion production causes problems and production cost increment, related to frequent equipment shut down for maintenance, and need for demulsifying processes. In some cases, water cut may be in a range of 0-70%, or more (Kokal *et. al*., 1999; Janssen *et al.*, 2001; Sjöblom *et al.*, 2003). Separation of emulsions is found to be extremely sensitive to the size of the droplets, and it is controlled by settling of drops in the order of 30-60μm. Separation time is usually greater than residence time of fluids in the separation equipment (Davies, G.A. *et al.*, 1996). The separation step may be critical in some cases for emulsions having drop diameters less than 30 micrometers in the case of plate separator, and for diameter less than 5 micrometers in the case of separation by centrifugation (Van der Zande, M.J. *et al.*, 1999).

Flow conditions responsible for generation of emulsions present at the surface, going from porous media, turbulence down hole, through pumps, or in the surface facilities, are uncertain, and their effects on the drop size, are not very well understood. Knowledge of the morphology of the resultant emulsions is important input data in the design of process conditions and separation equipments. Better knowledge of emulsification in porous media and in surface facilities is required to achieve good predictability of the produced emulsions morphology and behavior, and to improve production planning. This is the focus of the present work.

Porous media emulsion generation occurs because of the flow of the two-phase fluid systems through a complex interconnected pores-neck system, by means of the so-called snap-off mechanism, which is a type of drop breakup mechanism. This mechanism may be described as an invasion of the wetting phase flowing adjacent to the pore-neck wall, into a constriction occupied by a non-wetting phase. The wetting film grows like a collar, until it creates a discontinuity in the non-wetting phase (drop breakup).

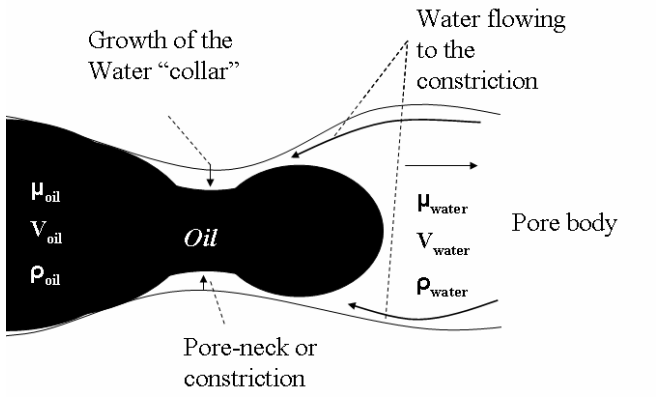

Figure 1. Snap-Off mechanism in a porous media for a system having water as continuous phase.

The snap-off mechanisms have been extensively studied to explain foam and emulsion generation in oil reservoirs. Roof (1970) studied the effects of channel geometry on snap-off of a slug of non-wetting phase using an oil/water system. Results of his experiments agree with his proposed theory, which suggests that depending on the constriction geometry, a minimum protrusion of the non wetting phase through the pore-neck is required for snap-off to occur. According to Roof, snap-off happens only in the case of a tube/constriction diameter ratio of at least 2:1. Rossen (2002) completed a review on the snap-off mechanism of foam generation. He concluded that the pressure drop for snap-off to occur depended on the pore-neck geometry, and that snap-off, in agreement with Roof's criterion, stops when the downstream pore body is filled with one o more bubbles or drops.

According to Janssen *et al.* (2000), wettability also influences the diameter of drops in emulsion produced from a reservoir: relatively big drops diameter in oil-wet porous media and small drops, of the order of the pore body scale diameter, in water-wet porous media.

Snap-off phenomena in a pore-neck inside a porous media may be physically simulated by flowing a two-phase liquid system through a constricted capillary. Several variables may influence the breakup of drops inside a constricted channel: The Capillary number (*Ca*), capillary geometry, pressure drop along the constriction, viscosity ratio and density ratio, drop-to-neck diameter ratio, wettability of the capillary wall, surfactant concentration gradient and chemical nature of the phases. The influencing variables that were studied in the present work are discussed in what follows.

The capillary number Ca expresses the ratio of viscous to interfacial forces in the system, and is defined by:

$$Ca = \frac{\mu V}{\sigma}. \tag{1}$$

Where μ is the viscosity of the continuous phase; $V \equiv Q/A$ is the average velocity; and σ is the interfacial tension between the two liquids.

The capillary number influences significantly the deformation and snap-off of drops flowing through capillaries. It represents the ratio between the viscous flow resistence of the wetting phase adjcent to the capillary walls and the capillary pressure gradient that drives the flow of the wetting phase to the neck of the constricted capillary. Below a critical value of the capillary number, referred here as the critical capillary number, drop breakup occurs.

The present paper focuses on the physical simulation and visualization of emulsion generation at the pore scale, using a glass constricted capillaries. The study was done with circular constricted capillaries with three different constriction-to-capillary diameter ratio and three different oil/water systems.

2. Materials and Experimental Procedure

2.1 Materials

Properties of liquids used are listed in Tab.(1). In the experiments reported here, water is the continuous phase that wets the capillary wall. The experiments were done with three oil-water+surfactant emulsions, with different viscosity ratio, k. Mineral oils were selected to avoid chemical interferences of foreign components. The surfactant used was Sodium Dodecyl Sulfate (SDS), at a concentration of 0.03M (three times the critical micelle concentration).

Table 1. Properties (@ 20°C) of liquids used in the experiments

Fluid	Viscosity (cp)	Density (g/ml)	Interfacial tension Oil/ (Water+SDS) 0,03 M(mN/m)
OP3 Mineral Oil	3.5	0.7876	45.1
OP10 Mineral Oil	13.36	0.8361	42
Shell Mineral Oil	620	0.900	41.6
Water+SDS 0,03 M	1	1.0006	-------

Borosilicate glass constricted capillaries were used to simulate a pore passage in a porous medium. Three different geometries consisting of different neck-to-straight section diameter ratios were considered in the study. The geometries and flow conditions analyzed are listed in Tab.(2) and showed in Fig.(2).

Table 2. Studied velocities and capillary numbers (*Ca*)

Studied Geometries (constriction / straight section diameter ratio)	Velocity in the straight section (cm/s)	Velocity at the constriction (cm/s)	*Ca* in the straight section	*Ca* at the constriction
50/200	$0.884 - 8.84*10^{-2}$	0.141 - 1.41	$0.185 - 1.85*10^{-5}$	$0.3 - 3*10^{-4}$
20/200	$0.884 - 8.84*10^{-2}$	0.884 - 8.84	$0.185 - 1.85*10^{-5}$	$0.185 - 1.85*10^{-4}$
50/100	$0.353 - 3.53*10^{-1}$	0.141 - 1.41	$0.74 - 7.4*10^{-5}$	$0.3 - 3*10^{-4}$

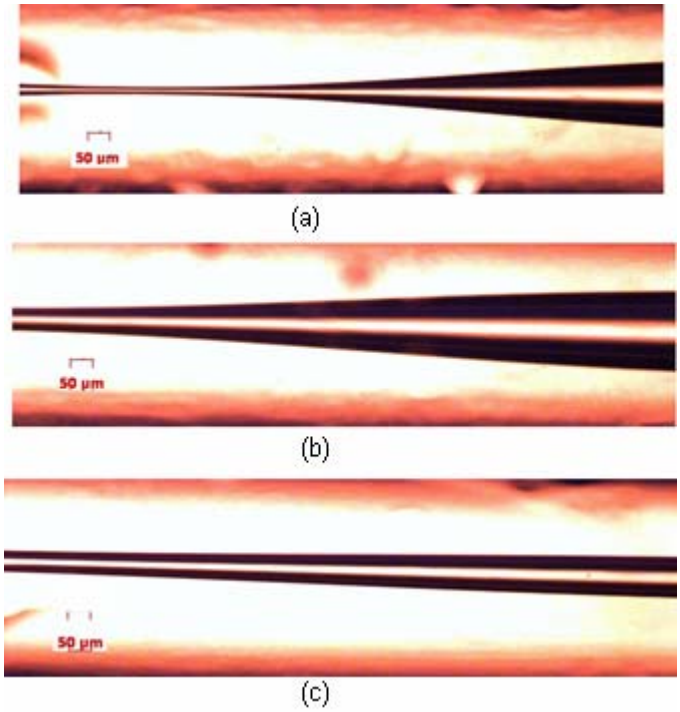

Figure 2. Capillaries used in the study. (a) 20/200 (constriction/straight section diameter ratio) geometry; (b) 50/200 and (c) 20/200.

2.2 Experimental Procedure

Because of the technical difficulties related to injecting controlled volumes of small drops at a micro scale, emulsions with large drops were injected into the constricted capillary. The emulsions were prepared simply by hand-shaking the oil-water-surfactant mixture to obtain the large drops. The drop size distributions of the emulsions before they flow through the constriction were determined by visualizing them in a microscope. The range of drop size of each emulsion prepared is presented in Tab.3. The surfactant solution, oil and water were filtered before mixing to avoid blocking the capillaries at the constriction, caused by impurities present in the liquids. Effects of the surfactant concentration were not conducted in this study. The concentration of oil phase was 33.33 % v/v in all of the cases.

A low-Reynolds number regime, corresponding to volume rates between 0.01 ml/h and 0.1 ml/h, was studied for each capillary geometry and oil/(water+SDS) system. The flow rate was controlled by using a syringe pump (Colle Parmer). The flow parameters explored are listed in Tab.2, they represent typical values for flows in reservoirs.

The visualization setup is shown in the Fig.(3). Undesirable refractive index contrasts were mitigated by submerging the capillaries in glycerin, for the visualization of the flow of drops flow through the capillaries. The flow images were captured using a CCD camera coupled to an Axiovert 40 MAT Inverted Microscope (Carl Zeiss). The capturing rate of the CCD camera was of 15 frames per second. 5X and 10X lenses were used to observe the constriction region. An objetive of 10X was used to provided details of the snap-off, while an objective of 5X was used to gather wider field of view for the constriction where drop breakup occurs. The video camera was connected in parallel with a video recorder (for long-time video recording purposes) and to a computer (for still pictures or frame grabbing and short-time period video recording). Videos were converted to a digital format using a LabView interfaces, and the images were analyzed using the Axiovision software (Carl Zeiss), in order to measure details of the experiments, i.e. droplets size and distances into the constriction.

Table 3. Drop size ranges of the prepared emulsions.

Emulsion	Diameter ranges of the drops (μm)
OP3 /(water+SDS)	10-200
OP10/(water+SDS)	10-160
Shell/(water+SDS)	5-130

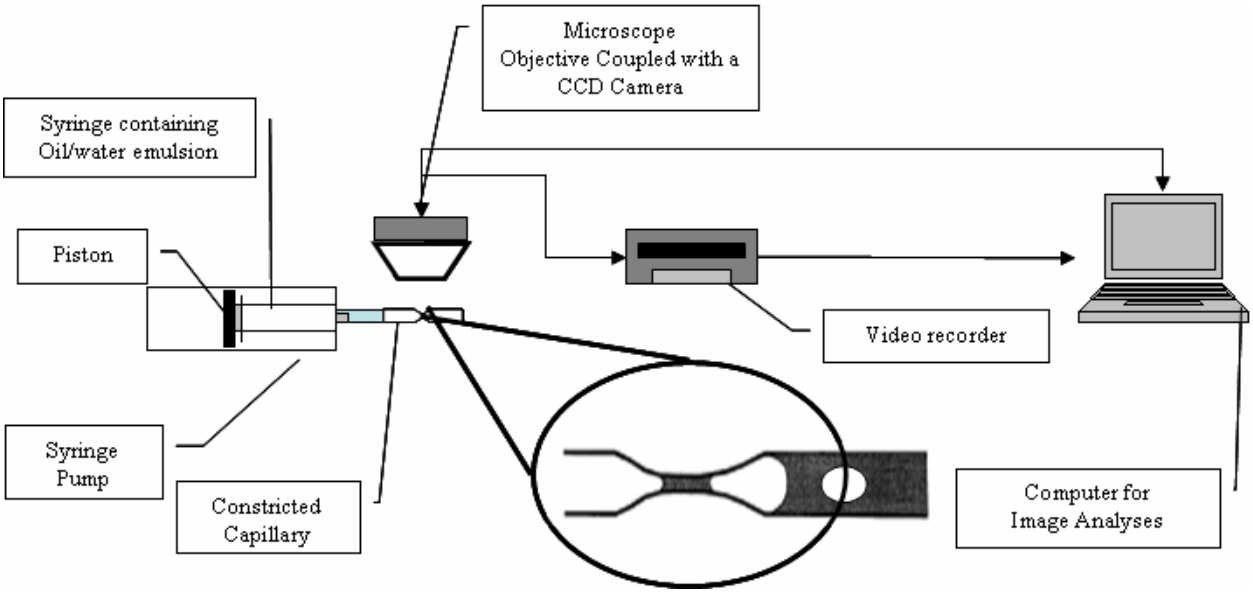

Figure 3.Visualization experimental Set-up

3. Results

As the oil drop flows through the constriction, a thin film of water is left attached to the capillary walls. This thin film flow is unstable, because a small disturbance on the oil-liquid interface creates a capillary pressure gradient that drives water into the disturbance, creating a collar structure that grows until it breaks the oil drop. Different stages of this process were visualized and are presented in Fig.5. In the images, the oil phase appears clear and the water phase, dark. The oil drop is moving from left to right in the figure. A interface non-uniformity, located at a distance d1 from the constriction, can be observed if Fig.5(a). This collar structure grows with time leading to a drop breakup at a position d2 downstream from the constriction – Fig,5(d).

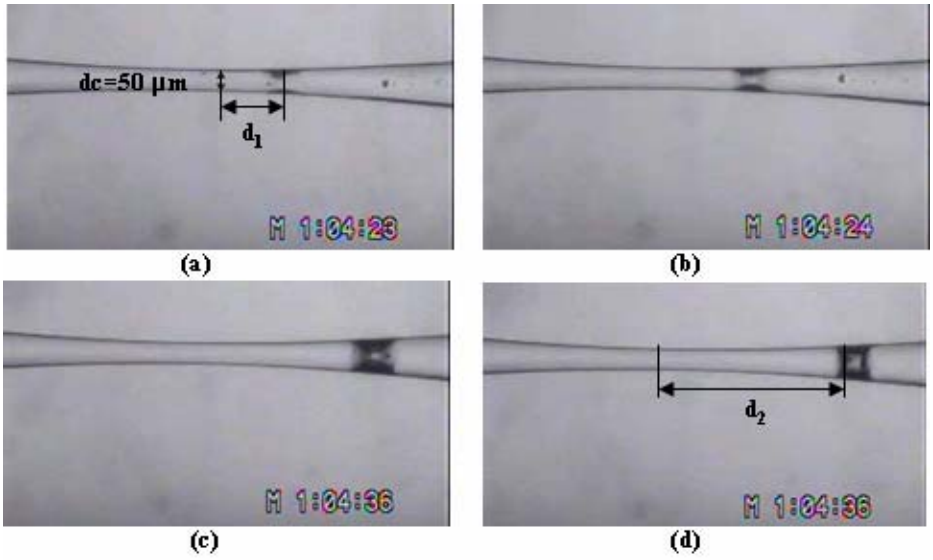

Figure 5. Snap-off of an oil drop in a (water+SDS) continuous phase.
Flow rate (Q)=0,02 ml/h. Emulsion: OP3/(water+SDS). Actual times are showed in the photography sequence (a) to (d). Constriction diameter (dc)=50 µm. d_1=2,33*dc in picture (a) and d_2=8*dc in picture (d)

The main parameter that determines the drop break up is the ratio between the driving force of the water flow through the thin film (the capillary pressure gradient) and the viscous resistance to the flow, e.g. the capillary number. The intensity of the capillary pressure gradient is a function, not only of the interface tension between the liquids, but also of the geometry of the capillary tube. An abrupt contraction will lead to a higher capillary pressure gradient, favoring the drop breakup. It is clear that if the oil drop is small enough, the disturbance will not have enough time to grow and to break the oil drop. The mechanism visualized here and depicted in Fig.5 agrees with that observed by Roof (1970), who stated that in order to snap-off occurs, the drop had to protrude pass the capillary constriction.

Because the broad distribution of the drop diameter of each of the oil-water emulsions tested, the effect of the drop size on the snap-off mechanism was not studied here. However, as explained in the previous paragraph, it is clear that this variable plays an important role in the process.

The occurrence of snap-off mechanism was mapped as a function of capillary number for the three emulsions prepared (three viscosity ratio) and the three different capillary geometries (different constriction-to-capillary diameter ratio). The results for viscosity ratio of $k = 3.5$ are shown in Fig.6. The open symbols indicate experimental conditions at which snap-off was not observed, even for the largest drop of the emulsion. The filled symbols indicate experimental conditions at which snap-off occurs. At high capillary number, the capillary pressure gradient was not strong enough to lead to drop breakup for the three geometric configuration tested, represented by the constriction-to-capillary diameter ratio. The critical capillary number at the onset of the drop breakup was approximately $Ca = 4 \times 10^{-2}$, and was virtually independent of the geometry configuration of the constricted capillary. The results for viscosity ratios of $k = 13.3$ and $k = 620$ are shown in Fig.7. At $k = 13.3$, the critical capillary number at the onset of drop break up is a function of the geometry of the constricted capillary. It falls as the contraction-to-capillary diameter ratio rises. When the geometry with the weaker contraction (highest contraction-to-capillary diameter ratio) was used, snap-off was not observed in the range of capillary number explored. Snap-off was also not observed in the experiments using the emulsion with the highest viscosity ratio, e.g. $k = 620$. This result also agrees with those reported by Olbrich (1996) and Tsai et al. (1994), who state that snap-off only occurs for low viscosity drops (low viscosity ratio).

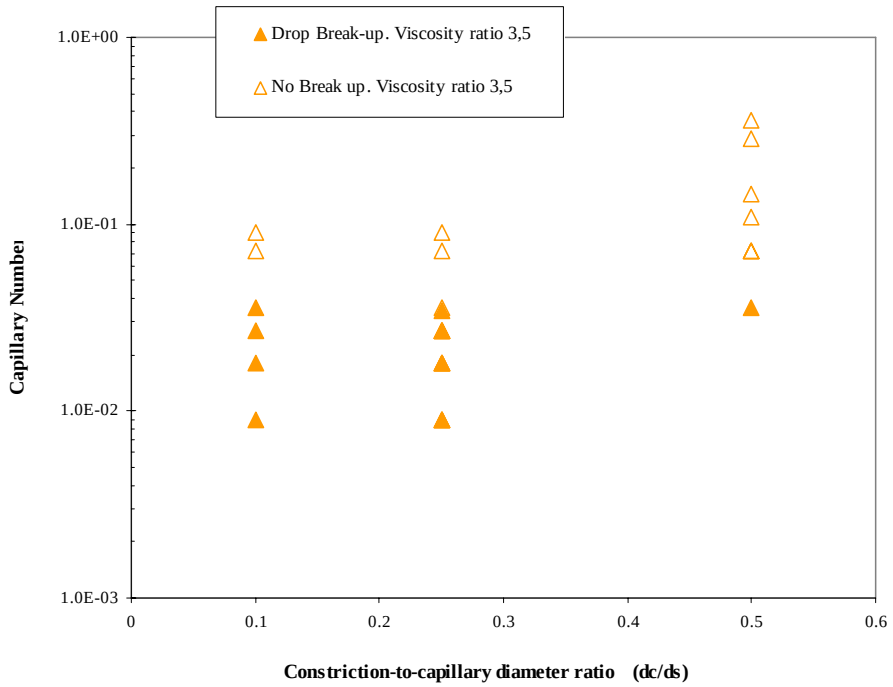

Figure 6. Regions of Snap-off in circular constricted capillaries for geometries studied. Viscosity ratio: 3.5 cP.

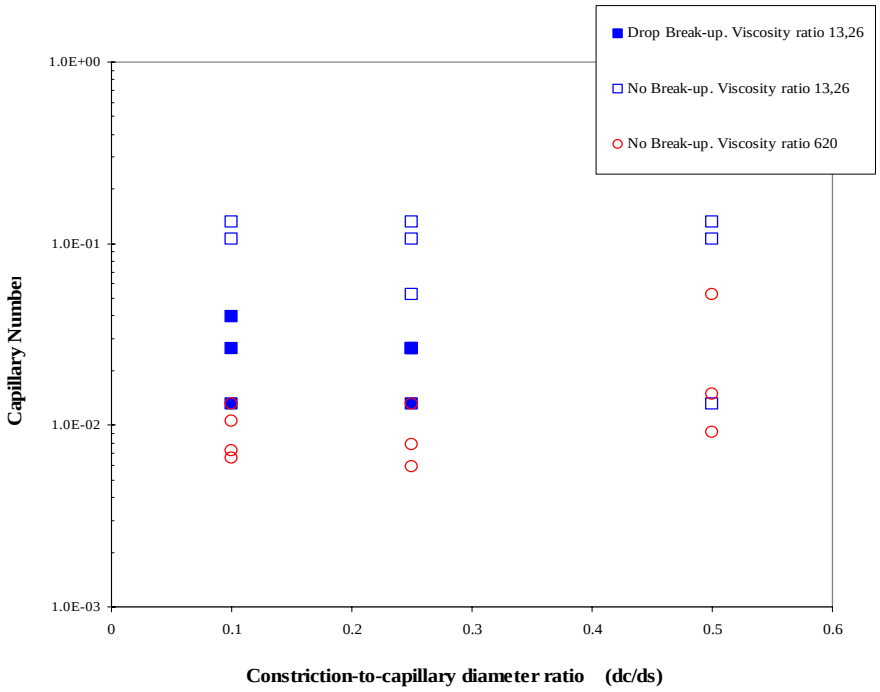

Figure 7. Regions of Snap-off in circular constricted capillaries for geometries studied. Viscosity ratio: 13.26 cP

Accidentally, during one of the experiments, the capillary tube was clogged by unfiltered surfactant solution, creating an irregular and very abrupt contraction. This enabled an analysis of drop breakup in the flow through very small constriction. The actual size of the constriction caused by the accidental clogging could not be measured. Figure 8 shows a sequence of images showing the oil drop break up. Small drops are formed at the conditions shown, with an average size of approximately $12\mu m$ and a very narrow distribution of drop diameter. As interesting phenomena was

observed. As the oil drop breaks into small drops, the volume of the oil in the large drop upstream the constriction falls. As the volume of the source drop falls, the size of the produced drops, downstream of the constriction, rises, until a point at which the oil drop upstream the constriction is small enough that the snap-off phenomena is not observed. At this point, the oil drop just flows through the constriction without being broken.

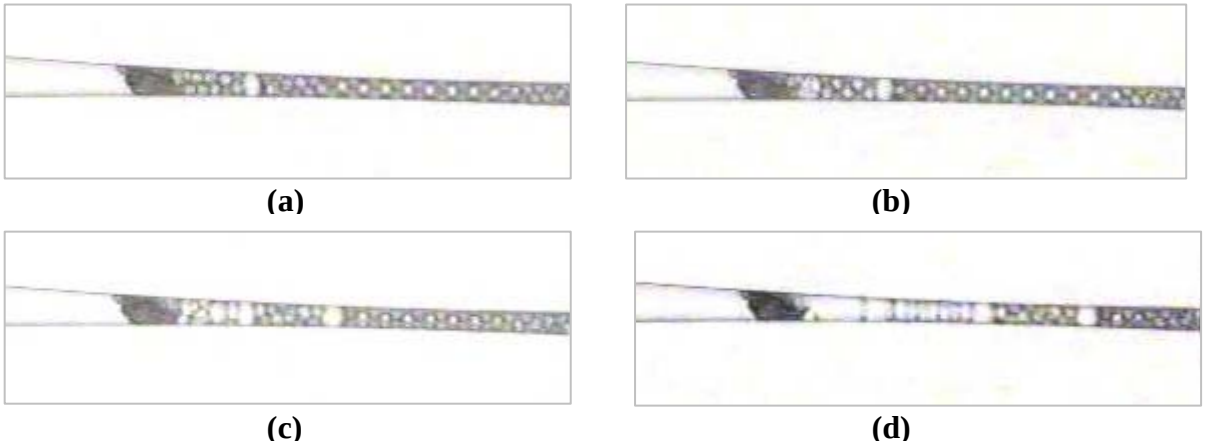

Figure 8. Snap-off during continuous phase running out.
Q=0.02 ml/h. Emulsion: OP3/(water+SDS). Average diameter of the produced drops (dp) are: (a)dp=11.57 μm; (b)dp=13.33 μm; (c)dp=20 μm and (d)dp=32 μm.

4. Final Remarks

Visualization of drop breakup through the snap-off mechanism at the micron scale was achieved by flowing oil droplets through circular glass-constricted capillaries.

A dependence of snap-off on experimental parameters such as contraction-to-capillary diameter ratio, viscosity ratio between the two liquid phases, and capillary number was explored. The set of flow parameters at which drop breakup occurs was determined for different capillary geometries.

It was observed that produced drops could be smaller than 10 micrometers for a very small pore neck. This result suggests that emulsions with very small drops may be produced in porous media, i.e. before the fluids enter in contact with choke valves or are exposed to turbulent flow down hole. Those small drops represent a challange for the separation process.

5. Acknowledgement

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