OIL-IN-WATER EMULSION FORMATION IN LAMINAR FLOW THROUGH CAPILLARIES

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Abstract. *Emulsion formation is usually a natural consequence of the recovery process during oil production. During this process, the oil flows through the porous space of the reservoir that also contains or is filled with water. After leaving the reservoir, the oil-water mixture flows through pipes, pumps and valves. During all stages, large drops of the dispersed phase may break leading to the formation of an emulsion. It is important to be able to predict the characteristics of the formed emulsion in order to properly design the pumping and separating systems. The main challenge is to understand how the different operating flow parameters affect the break up process.*
In this paper we conducted a parametric analysis of oil-water emulsion formation in laminar flow through capillaries. The experiments were carried out using two syringe pumps connected by a double-hubbed needle. The oil-water emulsion is forced back and forth through the needle. The drop size distribution was obtained as a function of the capillary diameter, shear rate of the flow (flow rate), residence time and liquid properties. As expected, with everything else fixed, the mean diameter of the dispersed phase falls with the shearing time, reaching an asymptotic value, which was a strong function of the shear rate at the capillary wall.

Keywords: *emulsions, break up process, capillaries, shear rate*

1. INTRODUCTION

An emulsion can be defined as a heterogeneous dispersion of at least two immiscible liquids (aqueous and oily phase) stabilized by an emulsifier. The internal phase is dispersed in the continuous external one, while the emulsifier (surfactant) molecules containing hydrophilic and hydrophobic sections align themselves surrounding the oil-water interface reducing the interfacial tension.

Emulsions have widespread applications in numerous industrial areas including cosmetics, beverage and food formulations, printing and paint industries as well as in pharmaceutical and agricultural products among other applications (Becher and Schuster, 1985).

Besides being abundant in daily life products, emulsions are usually formed in many other processes, particularly in oil field exploitation. During the oil production by primary or advance recovery methods, the reservoir pressure release and the fluids movement in the porous media promote emulsion formation by the mixture of the oil and water phases in contact within the sandstone formation. This mixture process is further intensified in the oil production stages through the surface facilities. During the two phase flow through pipelines, pumps and chokes valves, the phases undergoes a significant agitation and shear giving rise to the break-up of small drops of one phase inside the other. In the emulsification process the natural lipophilic surfactants of the oil migrate to the water-oil interface forming a stabilizing film around the dispersed water drops preventing its coalescence and promoting stable water-in-oil (W/O) emulsions type. The production of such stable emulsions in oil production operations becomes a costly problem since it requires the implementation of additional chemicals and treatment stages during the oil processing to break the emulsions and separate the produced water (Becker ,1997; Myers, 2005; Rondon et al., 2006).

To control the production of emulsions it is important to investigate and understand the process of emulsion formation, initially in simpler multiphase systems. Unfortunately even in laminar flow of simplified emulsion systems, this characterization is not yet fully accomplished because of the complexity of the emulsion formation mechanism and the numerous variables involved (Becher, 1967; Walstra P. 1993).

In a broad sense, the making of an experimental emulsified system involve several choices as the selection of the formulation variables (emulsion's constitutive materials), the composition variables (amount of the constitutive substances) and the emulsifying procedure. These three key features will define important properties of the resulting emulsions as the type of emulsion (water-in-oil W/O, oil-in-water O/W or water-in-oil-in-water W/O/W) mainly determined by the type of surfactant, and the drop size distribution.

In this work we prepare oil in water (O/W) emulsions in a laminar flow through capillaries at different experimental formation parameters, such as residence time, capillary diameter and flow rate. Parametric analyses of these variables were done to study their influence in the emulsion formation process on the break-up mechanism and the resulting average drop diameter.

2. EXPERIMENTAL SETUP

The continuous phase (CP) of the emulsion consisted of a mixture of 15% distilled water, 85% Glycerin, and Sodium Dodecyl Sulfate (SDS) surfactant in a concentration of 6.9×10^{-3} Kg/L, amount equivalent to three times the critical micelle concentration. The distilled water and the surfactant were previously mixed, homogenized through a magnetic stirrer (Fisatom, Model 754A) and then filtered before the glycerin addition.

For the disperse phase (DP) we used the mineral oil Shell Tivela S460 already characterized and filtered ($\rho = 994.9$ Kg/m^3 and $\mu = 0.9736$ Pa.s). The filtration process of the SDS solution and the oily phase were fulfilled using two 0.45 micrometers sterile analytical filter units (Nalgene®, Model 130-4045) and a double stage vacuum pump (Quimis™, Model Q355D).

Both phases were poured in two 10ml double-hubbed syringes in a DP/CP proportion of 1/10. One of the syringes was attached to a micro-emulsifying needle (Popper and Sons, Inc.) and before the entrapped air been expelled from both syringes they were connected through the needle creating a closed system with both syringes opposed. Emulsification was accomplished with a programmable emulsifier syringe pump (Cole-Parmer®). The two syringes-needle system was inserted in the syringe holders of the pump and the operation conditions were programmed by selecting the continuous mode of injection and by entering the flow rate, the syringe's diameter and the volume to be dispensed. The emulsifying process was performed automatically by the pump, forcing the mixture of fluids in the syringes back and forth through the needle for a time-lapse. To analyze the effect of the operation variables on the produced emulsion morphology we used the described emulsator system (see Fig. 1) and follow the former procedure for three flow rates, five residence times and three needle diameters. The selected values for the experimental conditions are summarized in Tab. 1.

Figure 1. Experimental Setup: a) Emulsator system assembly, b) Syringe and micro-emulsifying needles.

Table 1. Experimental conditions for the emulsion formation.

Experiment #	Flow Rate, Q (m^3/s)	Needle specifications	
		Nominal ID, ϕ (m)	Gage
1	1.67×10^{-8}	1.194×10^{-3}	16
2	6.67×10^{-8}		
3	1.17×10^{-7}		
4	1.33×10^{-7}		
5	1.67×10^{-7}		
6	2×10^{-7}		
7	1.33×10^{-7}	8.38×10^{-4}	18
8	1.67×10^{-7}		
9	2×10^{-7}		
10	1.33×10^{-7}	5.84×10^{-4}	20
11	1.67×10^{-7}		
12	2×10^{-7}		

Individuals experiments were performed for each flow rate and micro-emulsifying needle listed in Tab. 1 for residence times of 1800 s, 3600 s, 5400 s, 7200 s and 9000 s.

At each elapsed time the drop diameter distribution of the produced emulsion was measured by using the laser diffraction based particle size analyzer Mastersizer 2000 (Malvern Instruments, Ltd.).

3. EXPERIMENTAL RESULTS

Emulsions formed with the described experimental procedure were relatively monomodal and uniform. The experimental results show a good agreement inside each parameter evaluation, so for simplicity, we show one result obtained for each experimental condition.

Figure 2 shows the drop size distribution at different residence time for the experimental condition # 10. At short time, most drops are large. As time goes by, the large drops are broken as they flow through the capillary. After a critical residence time, all the large drops are broken and the drop size distribution does not vary with time.

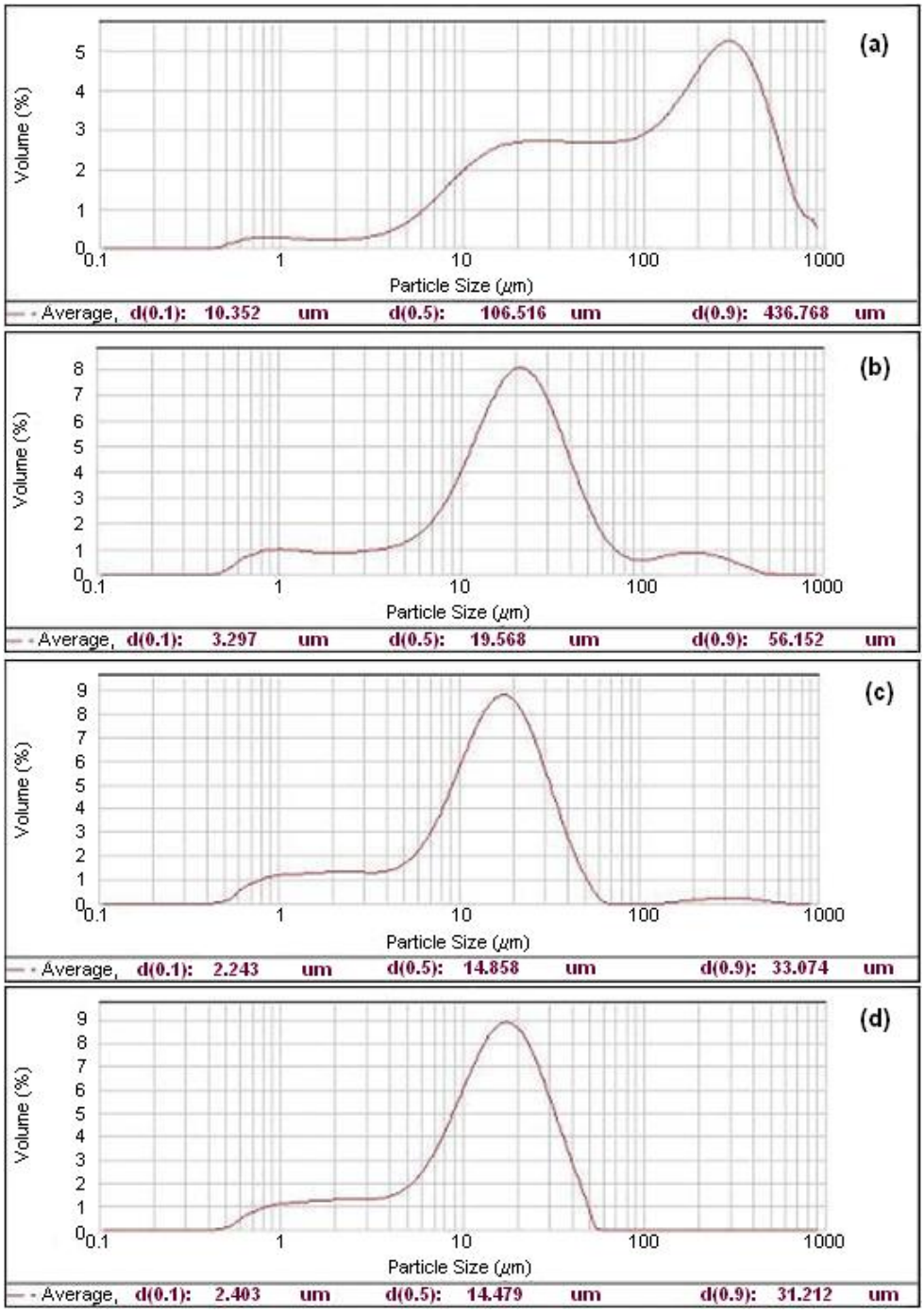

Figure 2. Drop size distribution for the experimental condition # 10 at residence times of 1800 s (a), 5400 s (b), 7200 s (c) and 9000 s (d).

In what follows we present the experimental results for the average drop size of the produced emulsions as function of the critical preparation parameters evaluated: effect of the residence time, the flow rate and the size of micro-emulsifying needle. Figure 3 shows the average drop diameter of the formed emulsions as function of the residence time for a flow rate of 1.66×10^{-7}m^3/s and the 16 gage micro-emulsifying needle.

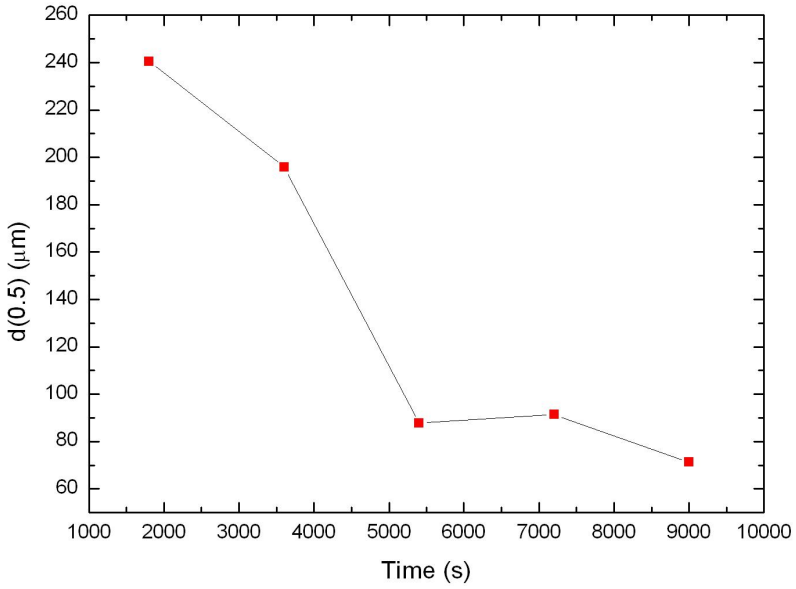

Figure 3. Effect of the residence time in the emulsion average drop diameter. Q= 1.67×10^{-7}m^3/s, Needle gage: 16.

As it was expected the emulsion average drop diameter decreases with the emulsification residence time. The micro-emulsifying needle can be seen as a narrow capillary in which the immiscible mixture, initially in the syringes reservoir, is forced to continuous cyclic passages back and forth. During this flow both phases undergo large shear stress, compression and elongations, leading to the deformation of the oil drops until their break up. The longer the two phases remain mixing in the emulsification system, the more is the mixture subjected to shear forces during its passage through the needle and consequently the smaller the drop size of the disperse phase in the emulsions.

In Figure 4 we present the average drop diameter of the emulsions produced varying the flow rate of the emulsifying process from 1.33×10^{-7}m^3/s to 2×10^{-7}m^3/s for a fixed residence time of 9000 s and the 18 gage micro-emulsifying needle.

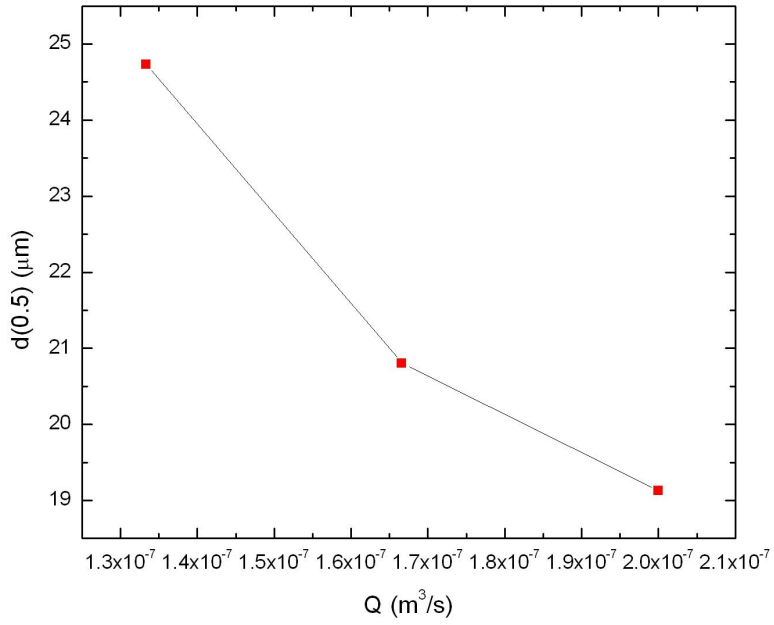

Figure 4. Effect of the flow rate in the emulsion average drop diameter. Time= 9000 s, Needle gage: 18.

Similarly to the effect of the residence time obtained in Fig. 3, the behavior of the emulsion drop size observed in Fig. 3 can be addressed to the shear forces exerted on the mixture of fluids in the micro-emulsifying needle. As the flow rate increases, the oil drops deformation becomes more intense increasing the drop break-up process and hence reducing the drop size of the dispersed phase.

Figure 5 shows the average drop diameter of emulsions prepared with the three micro-emulsifying needles for the same residence time and flow rate. As can be noted, there is a strong reduction in the average drop diameter between the emulsions produced with the 16 and 18 gage needles, being this difference less remarkable when compared with the drop sizes obtained with the needles gages 18 and 20.

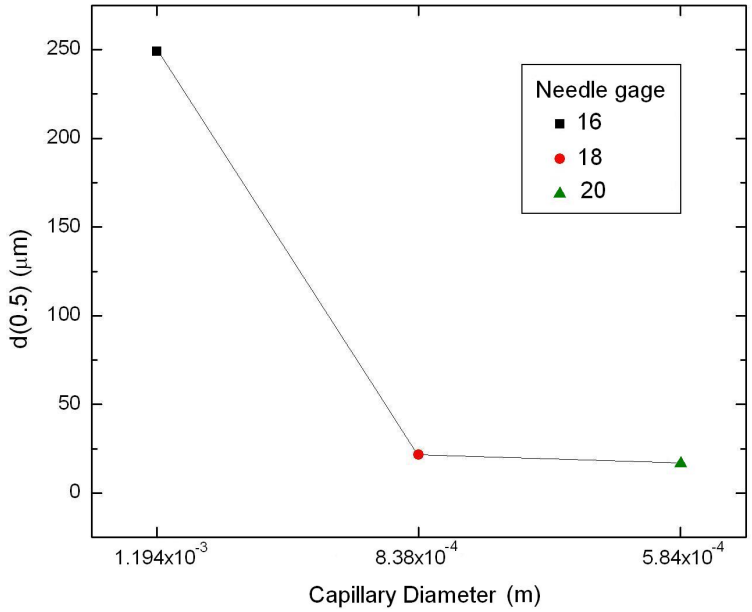

Figure 5. Effect of the needle diameter in the emulsion average drop diameter. Time= 1800 s, Q= 2×10^{-7}m^3/s.

A dramatic reduction in the diameter of the capillary at a constant flow rate reduces the effective area of the flow through the needle, bringing as a consequence an increase on the exerted shear rate and shear stress on the internal drops, increasing their break-ups.

In order to study the experimental average drop diameters in term of the flow properties and the capillaries dimension, we compute the shear rate ($\dot{\gamma}$) for each needle and flow rate assuming Newtonian fluid and laminar flow regime. Under these conditions the shear rate in a capillary tube is given by

$$\dot{\gamma} = \frac{4Q}{\pi R^3} \tag{1}$$

where R is the radius of the capillary. Figure 6 shows the log-log plot of the emulsions average drop size as function of the shear rate for the three micro-emulsifying needles at a fixed residence time. It can be seen that the results can be approximated by a power-law relation between the drop diameter and shear rate: $d \approx 28506\dot{\gamma}^{-0.8373}$. This result suggest that it's possible to predict an average droplet size by using the shear rate and the variables involved in its calculation as control experimental parameters.

4. RESEARCH PLANS

The characteristics of the produced emulsions depend on the preparation parameters as those here evaluated (residence time, flow rate and capillary diameter). The mechanical droplet break-up phenomena in capillaries results from a competition between the shear stress exerted at the fluids interface to deform it and the interfacial tension and dispersed phase viscosity acting against the drop deformation. To analyze how interfacial properties of the fluids can affect the emulsion drop formation more comprehensive studies in that respect should be conducted.

Because the surfactant is one of the key components on the emulsion formation process, its nature and concentration effect on the drop particle diameter will be evaluated in future projects. Surfactant is adsorbed in the interface providing more stability to the emulsion, reducing the interfacial tension and then the energy required to form the particles interfacial area.

Although it is usual to think that the more energy supplied to the emulsification system the smaller the droplet diameter of the produced emulsions, some of the energy supplied for the emulsion formation is lost by viscous dissipation, hence

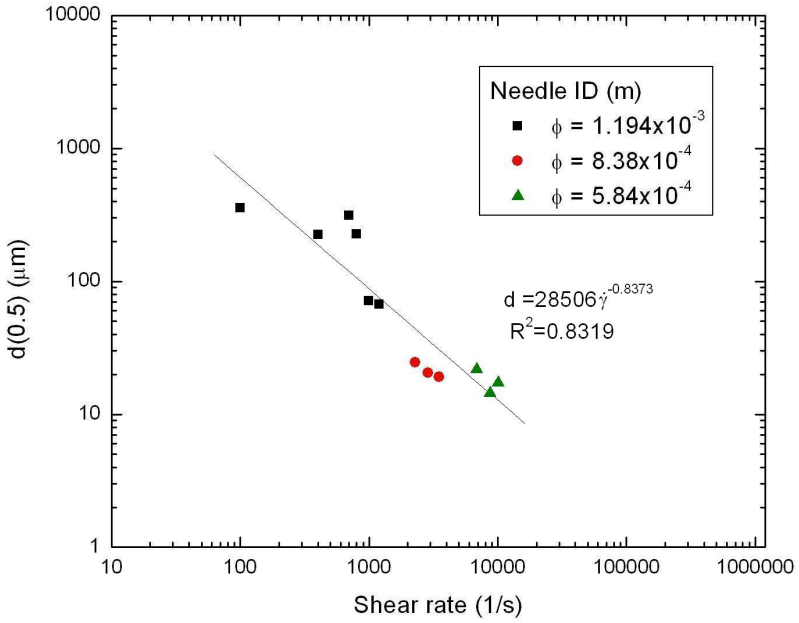

Figure 6. Average drop diameter of the emulsions as function of the shear rate. Time= 9000 s.

another factor that also will be evaluated is how the viscosity of the oily and the aqueous phases affect the break-up process and the drop formation of the emulsions.

5. CONCLUSIONS

Oil in water emulsions were prepared using a emulsification syringe pump to study the experimental parameters that affects the morphology of the produced emulsion. Parametric analyses were done for the preparation variables: flow rate, elapsed time and needle diameter. As expected, experimental results shows that the average drop size diameter of the emulsion produced becomes smaller with the reduction of the capillary diameter, showing an inverse proportional behavior with respect to the residence time of emulsification and flow rate. It was found a power-law relationship between the shear rates computed and the average drop size for each experiment. It suggests that by controlling the size of the capillary and the flow rate it is possible to obtain emulsions with a desired drop size. Moreover, future studies must be conducted to observe the effect of the emulsifier (nature and concentration) as well as the viscosity of the disperse and continuous phases on the morphology of the resulting emulsions.

6. ACKNOWLEDGEMENTS

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