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# CHARACTERIZATION OF OIL-WATER DISPERSIONS/EMULSIONS FLOWING THROUGH RESTRICTIONS

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## ABSTRACT

An experimental study of the characterization of oil-water dispersions/emulsions flowing through an orifice plate was carried out in the Dispersion Characterization Rig<sup>®</sup> (DCR), a state-of-the-art facility for studying the separation process of dispersions/emulsions. In this study, experiments with distilled water and mineral oil at different choke pressures, velocities, and different orifice sizes were conducted in order to find the phase-inversion zone and observe how the separation profile is affected by these variables. Bulk flow kinetic energy and water cut, are plotted against the pressure drop in the orifice plate to find the inversion point. Image processing technique is used to measure the coalescing and sedimenting profiles with respect to time. Results indicate a good agreement between the two methods used to find where phase inversion occurs and that this is affected by velocities, choke pressure and orifice plate size; also that emulsions become more stable when smaller size of orifice plates are used, as expected.

## INTRODUCTION

Dispersions are systems in which particles are dispersed in a continuous phase of a different composition or state. A dispersion may be classified in different ways, including how large the particles are relative to the particles of the continuous phase, whether or not precipitation occurs, and the presence of Brownian motion [1].

An emulsion is a dispersed system of two immiscible liquids such that the drops of one are suspended in the other. The liquid that forms the drops is called the dispersed phase and the liquid within which the drops are suspended is called the continuous phase. Thus, a stability criterion is required to show that the dispersed phase must exhibit resistance to coalescence in an emulsion [2].

In oil-water systems, like those of the Petroleum Industry, there are two major types of simple emulsions. These are oilin-water (O/W) and water-in-oil (W/O) which refer to the dispersed/continuous phase. While moving from one emulsion type to the other, properties of the mixture are going to change depending on the continuous phase, except in the inversion region where, with a small change in the operational conditions, the continuous and dispersed phase spontaneously invert. The inversion point is usually defined as the critical volume fraction of the dispersed phase above which this phase will become the continuous phase. Phase inversion is a major factor to be considered in the design of oil-water pipelines, since the rheological characteristics of dispersion and the associated pressure drop change abruptly [3].

Oil-water dispersions are a typical product of an oil reservoir especially nowadays when in order to avoid hydrates or wax precipitation emulsifiers are being added more frequently to the system. The non-continuous-phase tends to be broken into droplets and dispersed into the continuous-phase due to different active surfactants that are contained in the produced crude oil and once the fluid flows through restrictions in its way to the surface separator, these droplets break into even finer ones. Thus, the droplets are usually larger than those occurring upstream of the separator at the bottom of the oil well. The location where most of the droplet break-up takes place is the choke valve. In this valve, a large pressure drop is induced by forcing the fluids through a reduced flow area resulting in smaller droplets that are difficult to separate. The pressure drop in the choke is used to regulate the flow rate in the production system.

The water-phase in dispersion has to be separated by means of the separation equipment, which is an important part in field petroleum production industry. Traditionally, gravity separators are used to achieve this purpose. Since the restrictions in the tubing and pipe strings contribute considerably to the emulsification of the flow, it is necessary to understand the mechanisms by which chokes affect the properties of the dispersion. This will help in the development of predictive methods to optimize the separation process and provide better design tools for separators. To observe the coalescing and sedimenting behaviors of an emulsion system, the state-of-the-art Dispersion Characterization Rig<sup>®</sup> (DCR) facility at the University of Tulsa, is used to conduct the experimental work presented in this study [4, 5].

Several studies related to batch separators have been carried out. Nadiv and Semiat [6] developed a mathematical model for sedimentation based on Aris and Admunson [7] analysis for batch precipitation solids, and presented a study for a twophase liquid-liquid dispersion poured into a vertical tube studying the height variation with respect to time; results for O/W dispersion are shown in Figure 1, here the height of the dispersion band is reduced from the initial value (on the left side) into the interface between the two phases at the end of the separation (on the right side). The parameters that affect the separation process are [4]:

- 1. Fluid Properties.
- 2. Size and distribution of droplets in the continuous phase.
- 3. Droplet motion in the dispersion.
- 4. Droplet coalescence, into their own phase and binary coalescence between droplets.
- 5. Existence of different dispersion types (oil-in-water, water-in-oil, or a combination of both).
- 6. Geometry (diameter of the settling tube, height of the initial dispersion band, and insertions in the separator volume).
- 7. Mixing characteristics, such as mixer geometry, mixer type, impellers, impeller relative position to interface before initiation of mixing.
- 8. Existence of solids or gas-phase in the dispersion.

Figure 1 helps us to visualize how the separation process takes place in the DCR batch trap separation cell where, due to the difference in densities, the oil and water phases separate, creating coalescing and sedimenting interfaces which are dependent on time. Thus, the trapped sample is separated, by forming clean oil and clean water at the top and bottom of the cell, respectively, and leaving the mixture zone in between.

Galinat *et al.* [8] measured axial profiles of static pressure downstream of the orifice in dispersed two-phase flows of water and glycerin for various concentrations and Reynolds numbers. They plotted  $\Delta P_{max}$  against the global kinetic energy of the mixture, for six different concentrations using a fixed value of the restriction ratio; in all cases,  $\Delta P_{max}$  showed an increasing linear function of the global kinetic energy, with its slope depending on the case under consideration.

They observed that the presence of a small concentration of drops is sufficient to cause a significant decrease of the slope relative to the single-phase flow, but further decreasing of the water cut, no longer influences the slope. Also, that the slope of  $\Delta P_{max}$  for the water flow was lower than that of water-glycerin. This is a consequence of the decrease of Reynolds number as the flow viscosity is increased.



Figure 1: Schematic of Batch Separation Process in the DCR Cell for Oil-in-Water Systems.

## **EXPERIMENTAL SET-UP AND PROCEDURE**

Tests were conducted for this study using the Dispersion Characterization  $\text{Rig}^{\textcircled{B}}$  (DCR) located at the North Campus research building of The University of Tulsa [5]; a schematic is shown in Figure 2.

Distilled Water and Citgo Oil Crystex AF-L, for which a red colored dye was added in order to improve flow visualization between the phases, were used as the water and oil phases respectively, Tables 1 and 2 show the physical properties of the oil and water, respectively. Three orifice plate sizes ( $\beta$ = 0.3, 0.5, 0.7), two different pressures at the choke valve (0 psi and 50 psi), and three mixture flow rates (114756, 17200 and 22000 cc/h) that corresponds to the velocities of (0.89 ft/s, 1.34 ft/s and 1.71 ft/s) in the 0.15-in pipe where the orifice plate is installed.

#### **Table 1: Physical Properties of Mineral Oil**

Citgo Crystex AF-L				
Color/ Odor	Colorless/ Odorless			
Specific Gravity	0.85			
Solubility in Water	insoluble in cold water			
Density, lb/gal	7.09			
Gravity, °API @ 60°F	35			
Dynamic Viscosity, cP @104°F	11			

The DCR enables characterization of the separation behavior of oil-water dispersions under controlled conditions. The DCR control and data acquisition system allows the operation of the rig through the Falcon<sup>®</sup> interface software, developed by Sanchez Technologies, France.

Table 2:	Physical	<b>Properties</b>	of Distilled	Water
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Distilled Water				
Color/ Odor	Colorless/ Odorless			
Specific Gravity	1			
Density, lb/gal @ 60°F	8.33			
Dynamic Viscosity, cP @60°F	1.129			

The DCR is a high-temperature and high-pressure comprehensive test rig, where the separation process of oilwater dispersions, created by chokes and an orifice plate, can be evaluated in a batch test cell. This rig can operate under a range of temperatures between -20 to  $150^{\circ}$ C, pressures up to 300 psia, and viscosities ranging from 0.1 to 2,000 cP.

Currently, the injection system is divided into two sections: Pumping systems I and II. The pumping system I consists of two 2,000 cc positive displacement pumps filled with a hydraulic fluid (water) which hold a maximum pressure of 6,000 psia. They are operated with variable speed motors and can simultaneously pump controlled volumes of water to the sample bottles. In order to inject the required amount of each fluid to the mixing and separation section, from here the hydraulic fluid is displaced to the sample bottles which basically are high pressure cylinders with an internal moving plunger that isolates the hydraulic fluid utilized in the injection pump from the fluids to be tested contained within the bottles.

Bottles are made out of stainless steel and have a capacity of 600 cc. These bottles can be used at the same ranges of temperature and pressure of the DCR. The bottles are filled up in the lab using the Pumping System II composed of two positive displacement Pulsatron Series A Plus pumps rated at 30 gallons per day, with a maximum pressure of 100 psi, and able to handle a maximum solution viscosity of 1,000 cP. The mixing and separation sections are located inside a



Figure 2: DCR Schematic

temperature-controlled cabinet which regulates and provides isothermal environment for each test. The mixing section consists of a mixer valve, a choke valve and an orifice plate device.

Once the oil and water are mixed in the static mixer by being forced to flow through a 4 mm diameter section, the fluid mixture passes through the choke valve, with controlled aperture by adjusting the back-pressure, and the orifice plate device, which consists of a stainless steel porta-plate and an interchangeable orifice plate with available  $\beta$  ratios of 0.3, 0.5, and 0.7, undergoing through a pressure drop, which provides the necessary shear force between the oil and water phases, to form O/W or W/O dispersions/emulsions.

Following the mixing section is the separation section formed by two components: a buffer cell and a batch test cell. The buffer cell is made out of stainless steel and can tolerate pressures up to 6,000 psia has a capacity of 600 cc., and is meant to contain all the volume of the purge fluid so as to ensure that the fluid coming to batch test cell is only the test fluid.

The 400 cc batch test cell is made out of borosilicate to guarantee full visibility of the separation process, can hold pressures up to 300 psia, and has a stirrer which can go up to 500 rpm. The separation process is recorded in order to get the separation profile under each operational condition as a function of time. The videos taken from the batch test cell are edited using the FrameShots software and then the pictures are processed with the Visilog Software to measure the sedimenting and coalescing interfaces with time. [9, 10]

#### RESULTS

Since the restrictions in the tubing and pipe strings contribute considerably to the emulsification of the flow, it is necessary to understand the mechanisms by which chokes affect the properties of the dispersion. The discussion of the results is divided into two parts: Separation Results and Phase Inversion Results.

#### **Separation Results**

The water-phase in dispersion has to be separated by means of the separation equipment, which is an important part in the petroleum production industry. This will help in the development of predictive methods to optimize the separation process and provide better design tools for separators. To observe the coalescing and sedimenting behaviors of an emulsion system using the state-of-the-art Dispersion Characterization Rig<sup>®</sup> (DCR) facility at the University of Tulsa, parametric evaluation tests were conducted according to the following parameters:

- Water Cut: 25, 30, 50, 75%
- Choke Pressure: 0, 50 psi
- Orifice Size, β: 0.3, 0.5, 0.7
- Mixture Velocity: 0.89, 1.34, 1.71 ft/s

For each case a height against time graph was plotted to evaluate the behavior of the coalescing and sedimenting interfaces with respect to time.

**Water Cut Effect:** Four water cuts were studied, namely, 25, 30, 50 and 75%, with the orifice of  $\beta$ =0.3 orifice at a mixture velocity of 1.71ft/s, and the choke valve in completely open position i.e. no pressure.



Figure 3: Separation Profiles with Respect to Time for Different Water Cuts

Figure 3 shows the separation profiles for the four water cuts studied. Here, one can see that for the water cut of 25%, oil continuous case, complete separation is reached and at the fastest time, around 400 s, followed by the water continuous case, 75% WC, where complete separation is reached around the time of 700 s; finally, the intermediate water cuts of 30 and 50% where complete separation was not reached, due to the existence of drops and pockets present in the dense packed zone. It is hypothesized that this particular experiment is near the phase inversion region.

Evaluating the standard deviations, following methodology by Urdaneta [11], the 75% water cut shows the lowest standard deviation for both interfaces. 1.678 mm and 2.657 mm are the maximum values for the coalescing and sedimenting interface, respectively; and 0.756 mm and 0.261 mm the minimum values for the sedimenting and coalescing interfaces, respectively. The highest standard deviation for both cases are found for the 50% water cut where the maximum and minimum values for the coalescing interface are 3.787 mm and 0.465 mm, respectively; and 5.185 mm and 0.537 mm, respectively, for the sedimenting interface.

**Orifice Size Effect:** For this study, a mixture velocity of 1.71 ft/s, water cut of 75%, and no pressure at the choke valve (absolute value of 14.7 psi) were set constant for three different orifice sizes, corresponding to  $\beta$  ratios of 0.3, 0.5, and 0.7.

Figure 4 shows that for bigger orifice sizes the separation time will be less, due to the shear effect created by the restriction

which will be more significant at a smaller orifice diameter. Also, it is observed that for the orifice  $\beta$ =0.3 more stable emulsions are created which cannot be separated during the time studied.



Figure 4: Separation Profiles with Respect to Time for Different Orifice Plate Sizes

Evaluating the standard deviations, the  $\beta$ =0.3 orifice shows the lowest standard deviation for both interfaces. 3.569 mm and 1.079 mm are the maximum values for the coalescing and sedimenting interface, respectively; and 0.269 mm and 0.467 mm the minimum values for the sedimenting and coalescing interfaces, respectively. The highest standard deviation for both cases are found for the  $\beta$ =0.7 orifice, where the maximum and minimum values for the coalescing interface are 5.414 mm and 0.539 mm, respectively; and 2.472 mm and 0.467 mm, respectively, for the sedimenting interface.

**Choke Pressure Effect:** Pressure drop between the choke and the test cell was investigated with an orifice plate of  $\beta$ =0.5, keeping the water cut of 75% and a mixture velocity of 1.71ft/s, constant. Results in Figure 5 show that the liquid-liquid dispersion produced when the choke valve is set at a higher pressure, 50 psi (345 KPa), is more stable than the one obtained when no pressure is applied at the choke, as a result of higher shear force imposed on the fluids.

Evaluating the standard deviations, the case with no pressure shows the lowest standard deviation for both interfaces. 2.437 mm and 3.546 mm are the maximum values for the coalescing and sedimenting interface, respectively; and 0.372 mm and 0.286 mm the minimum values for the sedimenting and coalescing interfaces, respectively. The highest standard deviation for both cases are found for pressurized case, where the maximum and minimum values for the coalescing interface are 4.571 mm and 0.754 mm, respectively; and 6.238 mm and 0.983 mm, respectively, for the sedimenting interface.



Figure 5: Separation Profiles with Respect to Time at Different Pressures at the Choke Valve

#### **Phase Inversion Results**

Two methods were used to identify where phase inversion occurs for the three orifice sizes at three different velocities; these two methods were: pressure drop across the orifice vs. bulk flow kinetic energy and WC. Tests were run in a random mode to assure that the  $\Delta P$  read across the orifice was not being affected by the sequence in which the experiments were run.

It is important to recall Piela *et al.* [12] who mention that, there is an important difference between a continuous experiment and a direct experiment, and state that in a direct experiment, the two liquids are mixed from the start at a constant concentration of the phases and there is no preliminary structure, thus depending on the concentration, the liquids mix as a water-continuous mixture or an oil-continuous one, and at the phase inversion concentrations none of the regions is sufficiently strong to dominate the other, and so both regions flow downstream. These regions interact, entrap parts of the other continuous phase, break-up and coalesce, causing a large pressure gradient over the pipe.

Since the studies done with the DCR fall in the category of direct experiments, given that the two liquids are injected at a constant fixed concentration from the start and the rig design is such that experiments are run one at a time. This will cause a large pressure drop across the whole system, including the orifice plate. This pressure drop can be used to determine where phase inversion occurs.

Since the pressure drop across the orifice,  $\Delta P$ , is the relevant global parameter to characterize a flow downstream of the restriction [13] and that the behavior of the properties of a mixture is that starting from oil continuous to water continuous, there is going to be a high value for the oil continuous phase till a discontinuity (inversion point) is found and then the values drops down to the water continuous phase. Pressure drop across the orifice against the water cut was plotted to evaluate the existence of this behavior in the system as well as to identify the phase inversion and the inversion point. Figures 6, 8, and 10 show the referred plot for orifice

sizes of  $\beta$ = 0.3, 0.5, and 0.7, respectively; at 0.89, 1.34 and 1.71 ft/s.

The relationship studied by Galinat et al. [8] was modified in order to observe the phase inversion of the mixture since  $\Delta P$ increases as a linear function of the global kinetic energy, K, but its slope depends on the case under consideration.

The bulk flow kinetic energy equation is given by:

$$K = \frac{1}{2} \frac{\rho_m \cdot v_m^2}{g_c \cdot 144}$$

Where the density of the mixture,  $\rho_m$ , is defined as:

$$\rho_m = \rho_w \cdot WC + \rho_o \cdot (1 - WC)$$

Figures 7, 9 and 11 show the evolution of  $\Delta P$  versus the global kinetic energy of the mixture for orifice sizes of  $\beta = 0.3, 0.5$ , and 0.7, respectively, for eleven water cuts ranging from zero (pure oil) to 100 (pure water). Looking at the slopes one is able to identify where the water and oil continuous phases are since, for each phase, the slopes tend to be similar. Hence, from the figure one can deduce that the lines at the extremes represent the water and oil continuous phases, with water continuous phase having consistently lower  $\Delta P$ , and the region in the center of the graph represents the phase inversion.

Figure 6 shows that for the three studied mixture velocities, the oil continuous phase is present from water cut of 0% to 30% where the discontinuity that represents the inversion point is found, and then drops down till water cut of 50% where the water continuous region starts. This matches the separation profile results for the water cut effect, as presented in Figure 3 where the intermediate water cuts studied of 30 and 50% were identified as water cuts belonging to the phase inversion region. Here, one can see that they are within the phase inversion region and that indeed the 30% water cut is very close to the inversion point of the system.



Figure 6: Pressure Drop (β=0.3) as a Function of Water Cut

Comparing with Figure 7, the water continuous phase is easily identified in the lower half of the plot, represented by the lines of water cuts 50 to 100%. To identify the oil continuous phase from the phase inversion, it is necessary to look at the change of the slope, which is shown in the line of water cut of 30%. Thus, the oil continuous phase lies in the half of the graph for the water cuts of 0% to 25%. In a similar way Figures 8 to 11 are shown.



Figures 8 and 9 show the results for the three studied mixture velocities when a larger orifice is installed in the system,  $\beta=0.5$ , keeping the choke valve fully open.

Figure 8 shows  $\Delta P$  across the orifice against the water cut. Here, the  $\Delta P$  overall values are significantly smaller (by a factor of 8) than for the previous case,  $\beta=0.3$ . Starting from pure oil, the pressure drop remains almost constant around the values of 3, 2 and 1.5 psi for the 1.71, 1.34, and 0.89 ft/s cases, respectively; till the 35% water cut where the values drop down until the values representing the water continuous phase are reached from 65% onwards.



Figure 8: Pressure Drop (β=0.5) as a Function of Water Cut

Similar behavior was found in the pressure drop across the orifice versus bulk flow kinetic energy plot shown in Figure 9. The water continuous phase is clearly identified in the bottom-Copyright © 2010 by ASME

right corner of the plot, and the oil continuous phase in the center top. The phase inversion region can be identified once there is a change in the slope, which starts after the water cut of 35%. Thus, the phase inversion region is identified between the water cuts of 35 and 50%.



Figures 10 and 11 show the results for the three studied mixture velocities with the orifice of  $\beta=0.7$ , installed in the system, keeping once again the choke valve fully open.

 $\Delta P$  across the orifice,  $\Delta P_{orif}$ , is plot against water cut of the mixture to identify the inversion point and phase inversion. If compared with the  $\beta=0.3$  orifice plate size, the values of the pressure drop are smaller by a factor of 12.5.

Starting from pure water case in Figure 10, the pressure drop values remain almost constant in the water continuous phase increasing for the phase inversion between the water cuts of 50 and 40% where a discontinuity representing the phase inversion is found, and finally reaching the oil continuous phase.



Figure 10: Pressure Drop ( $\beta$ =0.7) as a Function of Water Cut

In Figure 11 the oil continuous phase, identified in red color, is located at the center top of the graph. If coming from pure oil case, a significant change in the slope could be noticed for water cut of 40% where the phase inversion starts taking place. The water continuous phase, identified in green, is found in the lower-right part of the graph starting with the water cut of 65%. Having a very good agreement with the plot shown in Figure 10.



**Kinetic Energy** 

Figures 6 to 11 show that  $\Delta P$  across the orifice plate is directly proportional to the flow rate and inversely proportional to the orifice size. The inversion region starts at higher water cut when a larger orifice is installed in the system.

For the oil continuous phase, the  $\Delta P$  across the orifice is higher than, that of water continuous phase due to the respective viscosities of the fluids.

Finding the inversion point using the  $\Delta P$  across the orifice against the bulk flow kinetic energy of the mixture plot is not as easy as plotting the  $\Delta P$  across the orifice versus the water cut but both methods showed a very good agreement with each other.

## CONCLUSIONS

- The main goal of this study, the characterization of oilwater dispersions/emulsions flowing through restrictions, was accomplished successfully, with the DCR data being reproducible as shown by the low standard deviation values.
- The liquid-liquid dispersion produced when the choke valve is set at a higher pressure will take longer time to separate as a result of higher shear forces imposed on the fluids. A similar result was observed reducing of the orifice plate diameter.
- Water cut also affects the separation time, especially near the phase inversion region where presumably the existence of a combination of smaller drops and dispersed phase pockets makes the separation process more difficult.
- The results of plotting  $\Delta P$  vs bulk flow kinetic energy and  $\Delta P$  vs WC are in very good agreement with each other.

 The inversion point of a mixture is not easy to identify by plotting ΔP across the orifice against the bulk flow kinetic energy of the mixture.

## NOMENCLATURE

β	Ratio	between	the	orifice and	the pipe	diameter, [-]
10	D	P	r			

- $\Delta P$  Pressure Drop, [psi]
- $\Delta P_{max}$  Maximum Pressure Drop, [psi]
- $\triangle Porif$  Pressure Drop across the orifice, [psi]
- $\rho_m$  Density of the Mixture, [lbm/ft<sup>3</sup>]
- $\rho_o$  Density of the Oil, [lbm/ft<sup>3</sup>]  $\rho_w$  Density of the Water, [lbm/ft<sup>3</sup>]
- $\rho_w$ Density of the Water, [lbm/ft<sup>3</sup>]  $g_c$ Acceleration due to gravity, [=32 lbm\*ft/lbf\*s<sup>2</sup>]
- *g<sub>c</sub>* Acceleration due to gravity, [=32 lbm\*ft/lbf<sup>2</sup> *K* Bulk Flow Kinetic Energy, [psi]
- $P_{ch}$  Pressure at the Choke Valve, [psi]
- $v_m$  Mixture Velocity, [ft/s]
- $q_m$  Mixture Flow Rate, [cc/h]
- WC Water Cut, [-]

# ABBREVIATIONS

- DCR Dispersion Characterization Rig DP Pressure Drop
- O/W Oil in Water
- W/O Water in Oil
- WC Water Cut

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