Effects of Aging of Oil-In-Water Emulsions On Flow Instabilities in A Hele-Shaw Cell

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ABSTRACT

The effects of aging on viscous fingering resulting from the displacement of Oil-in-Water (O/W) emulsions with water are analyzed. The flow is examined in a rectilinear Hele-Shaw cell which is an analogue of homogeneous porous media. The experimental measurements were conducted for two concentrations of the emulsions, 40% and 60% (v/v %) that were displaced at a constant injection rate of 0.21 ml/min. The flow instabilities were found to vary strongly with the elapsed time between the initial emulsion preparation and the start of the displacement. In particular, fresh emulsions resulted in smooth finger structures that are less complex than the aged ones which tend to exhibit strong ramified branching and needle-like fingers. Furthermore, it was found that the aged emulsions show a shorter breakthrough time and a smaller sweep efficiency than their fresh counterparts. Characterization of the emulsions revealed substantial changes in the rheological behaviour and microstructure of the emulsions with age, but no significant changes in the drop size distribution. It is speculated that as a result of aging, there is an increase in flocculation or adsorption of surfactant on the dispersed droplets, which affects the emulsions' rheological behaviour and in turn the finger structures.

1. INTRODUCTION

Flow displacements in porous media can result in the development of an interfacial instability between the two fluids involved in the displacement process. This instability, first reported by Hill (1952) manifests itself in the form of finger-shaped intrusions of the displacing fluid into the displaced one. It is commonly referred to as the Saffman-Taylor instability [1, 2], and can have a dramatic impact on the displacement process [3]. When the driving factor behind the instability is the viscous mismatch between the two fluids, the instability is referred to as the viscous fingering instability. These instabilities are encountered in a variety of natural and industrial applications and can have an important impact on the efficiency of those applications. In particular they are observed in a variety of oil recovery processes, solute transport in aquifers, chromatography and polymer processing.

There is a large body of literature dealing with the viscous fingering instability in porous media. However, due to the opaque nature of porous media that usually precludes detailed information about the flow, simpler and more practical flow geometries have been often used to analyze the instability. In particular, the Hele-Shaw cell geometry that consists of closely spaced parallel plates has been widely and successfully used to visualize and analyze the flow dynamics in homogeneous porous media. The analogy between this simple geometry and real porous media stems from the fact that the flow averaged across the gap of the cell is well described by Darcy's law.

Numerous studies have been conducted to understand the viscous fingering instability using the Hele-Shaw cell and other similar geometries. Most of these studies have focused on flows where both the displacing and displaced fluids are Newtonian. The main controlling parameters in the Newtonian displacements are the viscosity [4] or density ratio between the two fluids [5], injection rate [6, 7] interfacial tension between the two fluids [8] and the geometry of the cell [4]. The first studies to examine the viscous fingering instability date back to the 1950's with the original work of Hill [5] and of Saffman and Taylor [9]. These pioneering works were followed by numerous studies mainly in the 1980's that were discussed in the extensive review by Homsy [10]. Two subsequent reviews dealing with experimental perturbations [11] and numerical modeling [12], have also appeared.

The past two decades have witnessed an increased interest in understanding the mechanisms of this instability for non-Newtonian fluids such as polymer solutions and particle suspensions [13–16]. Experiments carried out using shear thinning fluids revealed that fingers grow mainly by shielding, spreading, tip splitting, dense-branching and skewering [17–20]. Among these studies, there is a particular focus on polymer solutions due to the complexity in their structure and rheo-logical behavior. A number of experimental studies have looked at the effects of the polymer structure, molecular weight and solution concentration on the viscous fingering instability [19, 21, 22]. These studies allowed understanding, at least qualitatively, the effect of the viscous and elastic behaviour on the viscous fingering.

A number of theoretical and numerical studies have also attempted to understand and model the onset and later development of the instability for non-Newtonian fluids [6, 23–25]. These theoretical investigations which were based on different types of rheological models, also revealed interesting morphological structures in the displacements of shear thinning fluids. The importance of different perturbations sources associated with cell anisotropy [26, 27], visco-elasticity [28] as well as yield stress behaviour [29, 30] has also been investigated.

In spite of the great wealth and growing number of studies dealing with the viscous fingering for non-Newtonian fluids, there are very few studies that have actually focused on the case of emulsions. Indeed, even though emulsions can exhibit very complex rheological behaviour, there is a real dearth in the literature of studies on emulsion flows in spite of their importance in many applications. Such fluids are of particular importance in the oil industry where they can occur naturally as a result of the displacement of oil by water or are actually injected in processes of enhanced oil recovery. Even though emulsions share some important characteristics with non-Newtonian fluids such as the shear-thinning or visco-elastic behaviour, they in fact represent a class apart due to the complexity of their microstructure and compositions.

A review of the current literature reveals that there are only two studies that have examined the viscous fingering instability in displacements involving emulsions [31,32]. In the first of these two studies, the authors examined the flow in a radial Hele-Shaw cell for emulsions consisting of Silicone oil dispersed in aqueous Hydroxyl Propyl Methyl Cellulose (HPMC) solution. They observed crack-like finger to ramified finger patterns when the emulsion is displaced by an immiscible fluid. The authors related qualitatively the pattern transitions to the changes in the rheological properties of the emulsions and osmotic pressure difference between the injected fluid and the dispersion medium of the emulsion. However, they did not give any correlation between the imposed pressure and the injection rate or relate the physical behaviour of emulsions with the flow dynamics. The second study [32] focused on analyzing the flow displacement in a rectilinear geometry. This geometry differs from the radial one adopted in [31] by the fact that radial Hele-Shaw cells involve a point source injection and a contact interface between the two fluids that expands as the flow evolves, as opposed to the rectilinear cell that has a constant width and hence a fixed initial interface defined by the cell width. The study analyzed the relationship between the qualitative and quantitative characteristic of the flow and the rheological behavior of the solutions which were related to the droplet size distribution.

The only existing two studies dealing with O/W emulsions' displacements have examined the dynamics for *fresh* emulsions, in the sense that the emulsions used in the experiments were prepared a short time before running the displacements. However in a variety of applications, the emulsions used in the process may not necessarily be *fresh*, and it is important to understand how the aging of the emulsions may affect the flow and the development of the instability. The present study examines the viscous fingering instability of Oil-in-Water emulsions displaced by water, with a particular focus on analyzing and understanding effects on the instability due to changes in the emulsions microstructure as a result of aging.

2. EXPERIMENTAL SETUP

2.1. Materials

A light mineral oil (trade name Marcol 7) supplied by Esso Imperial Oil, Canada Ltd and reverse osmosis de-ionized water were used to prepare the oil in water (O/W) emulsions. The density and viscosity of the oil at 23ºC were 862 kg/m^3 and 10.7 mPa.s, respectively. The surfactant used was

Tween−65, a commercially available non-ionic surfactant also known as polyethylene glycol sorbi-tan tristearate. It is water soluble and has a hydrophilic-lipophilic balance (HLB) value of 10.5 ± 1.0.

2.2. Emulsions preparation procedure

Two different concentrations of oil in water emulsions were prepared in batches of 200 ml each. The concentrations of oil were 40 and 60 vol% (surfactant free basis) and the emulsion compositions are listed in Table 1.

For the preparation of the emulsions, the surfactant was dissolved in the aqueous phase by heating for about 15 to 20 min. Once the surfactant was completely dissolved, oil was added to the aqueous phase and heated for 7 to 8 min. The heating of the components reduced the oil viscosity and assisted in dispersing the oil droplets in the continuous water phase. The above mixture was then mixed in a blender for 20 min at a fixed speed. The emulsions produced were stored at a temperature of 23 ± 1° C.

2.3. Experimental apparatus

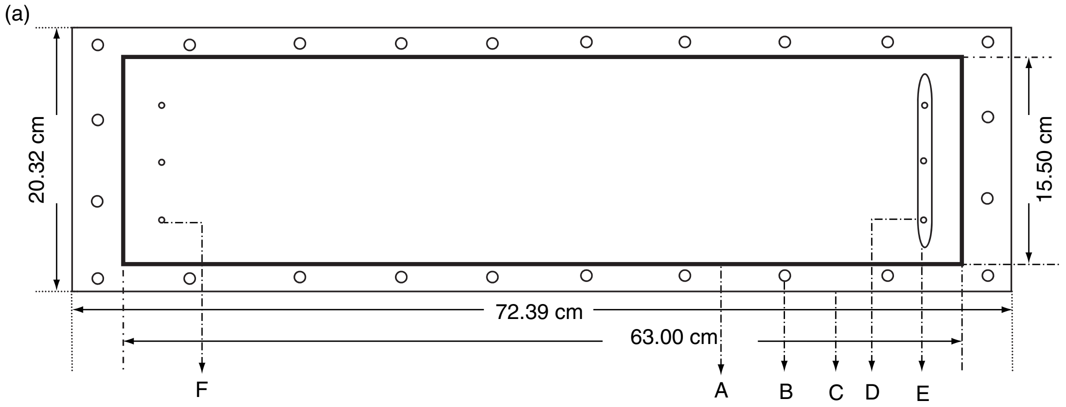
The flow displacements were carried in a rectangular Hele-Shaw cell shown in Figure 1. The cell is considered as a prototype for homogeneous porous media and is widely used to analyse flow instabilities in such geometries.

The cell consisted of two plane rectangular parallel plates made of plexiglass (72.39 × 20.32 × 5.08 cm^3). The plates were separated by a narrow gap using a soft rubber o-ring (Buna- N, diameter 0.3175 cm) along the cell perimeter and were fixed by 24 bolts and nuts.

On the top plate of the cell, three holes of a diameter = 0.3175 cm each were drilled evenly along each short edge of the plate inside the o-ring. The centre hole at one end of the cell was used for

Table 1. Details of emulsion samples.

Concentration of oil in emulsion (Surfactant free basis) (V/V %)	**Wt% of surfactant in continuous phase**
40	8
60	11



Symbol	Name	Number	Comments
A	O-Ring	1	Buna-N
B	Bolts	24	Stainless steel
C	Top plate	1	Plexiglass
D	Injection ports	3	
E	Channel		
F	Production ports	3	

Figure 1. Schematic and characteristics of the Hele-Shaw cell.

injecting fluids during displacements while the centre hole at the other end was used for collecting the fluids flowing out of the cell. The use of single line injection/ production avoided the effect of different pressures in the three lines. The other two holes on both sides of the cell were used for filling the cell with the emulsion as well as in the cleaning process.

The inner length, width and breadth of the channel were L = 63 cm, W = 15.5 cm and b = 0.034 cm respectively. A differential pressure transmitter was connected across the inlet and outlet ends of the cell to record the pressure drop during the flow. The inlet of the cell was connected to a syringe pump that provides constant flow rates. The error involved in the flow rate was less than 4%.

The cell was mounted on a light-table to assist in capturing images with better clarity. A digital camera (Sony digital camera, Model No: DSC–P93A) was used to capture images of the flow during the displacements. These captured images were analyzed using Image J software, which is a public domain Java image processing program that can run as an online.

2.4. Experiment procedure

The development of the viscous fingering instability was examined by carrying flow displacement experiments for the two emulsions at a constant injection rate. Each experiment followed a systematic procedure that consisted first of injecting the displaced fluid (emulsion) and then the displacing fluid (water). The emulsion was first injected into the Hele-Shaw cell with the syringe pump at very low injection flow rates to ensure that no air bubbles were trapped inside the cell. The water was then injected into the cell at constant injection rate. For easy contrast between the displacing and displaced fluid, water was dyed with a food colour. The development of the flow was recorded during the displacements using the camera and the captured images were analyzed using Image J software.

3. RESULTS AND DISCUSSION

3.1. Qualitative results of flow displacements

Results of flow displacements of the 40% and 60% emulsions are presented. The flow develops from the right side to the left side of the cell and the time is indicated in hh:mm:ss under each frame. All results are presented for a constant injection rate of 0.21 ml/min.

In the process of conducting different experiments, it was found that the finger structures and the flow development show important variations depending on the *age* of the tested emulsion. Indeed, emulsions that were tested after a short time from their preparation lead to results substantially different from those obtained with older emulsions. These differences are probably due to changes in the rheological behavior of the emulsions as they age. These changes and the possible reasons behind them will be investigated.

In what follows, qualitative and quantitative results for fresh and aged emulsions are presented and discussed.

3.1.1. Finger Structures for Fresh Emulsions

In this section results performed 3 hours (with a maximum deviation of±10%) after the completed preparation of the emulsions, are presented. These solutions will be referred to as the *fresh* emulsions.

Figure 2-a depicts results for the 40% *fresh* emulsion with an injection rate of 0.21ml/min. In the early stages of the flow, the interface exhibits three dense fingers. However, as the displacement progresses, only one finger ends up dominating the flow and suppressing the other two. This mechanism whereby one finger dominates its neighbours and out grows them is known as shielding [9]. Note that the fingers tend to have a rather diffuse structure which is particularly noticeable at later times.

Figure 2-b shows corresponding results in the case of a 60% *fresh* emulsion. In this case, a single finger dominates the flow throughout the whole displacement process. The finger is thinner than its counterparts in the case of the 40% emulsion and is more complex showing many side branches. Furthermore, when compared with the 40% emulsion, there is very little diffusion of the injected water into the emulsion. These results are expected since the viscosity of the 60% emulsion is much higher than that of the 40% emulsion, resulting in larger viscosity ratio with water and hence in more complex finger structures.

3.1.2. Finger Structures for Aged Emulsions

Results for aged 40% emulsions are shown in Figure 3-a for an aging time of 30 hours. The *aged* emulsions result in substantially different structures that are more reminiscent of those observed in the case of the *fresh* 60% emulsions. Unlike their *fresh* counterpart, this aged emulsion results in one

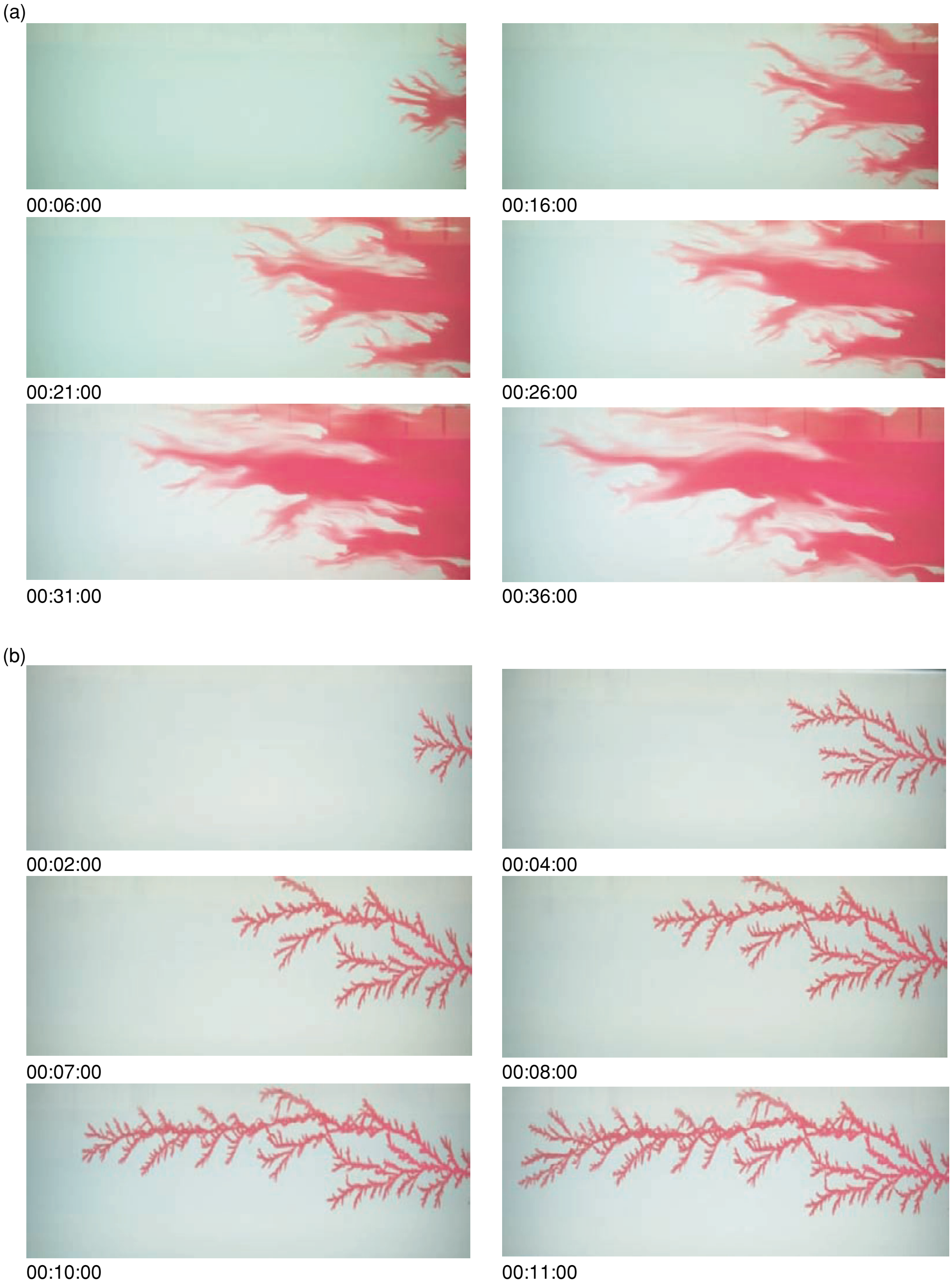


Figure 2. Displacement of *fresh* emulsions: (a) 40% emulsion, (b) 60% emulsion.

dominant finger with a lot of side- and multiple-branching and a sharper interface with no noticeable diffusion. It should be also noted that the injected fluid reaches the left side of the cell much faster than in the case of the fresh emulsion.

Results for the *aged* 60% emulsions are depicted in Figure 3-b. Here too the finger structures are different even though the changes are not as noticeable as in the case of the 40% emulsion. The finger tends to extend fast towards the production side with less side-branching than in the case of the corresponding *fresh* emulsion. It is worth noting that in this case water reached the left side faster than in the fresh case and that this breakthrough time is actually similar to that of the *aged* 40% emulsion.

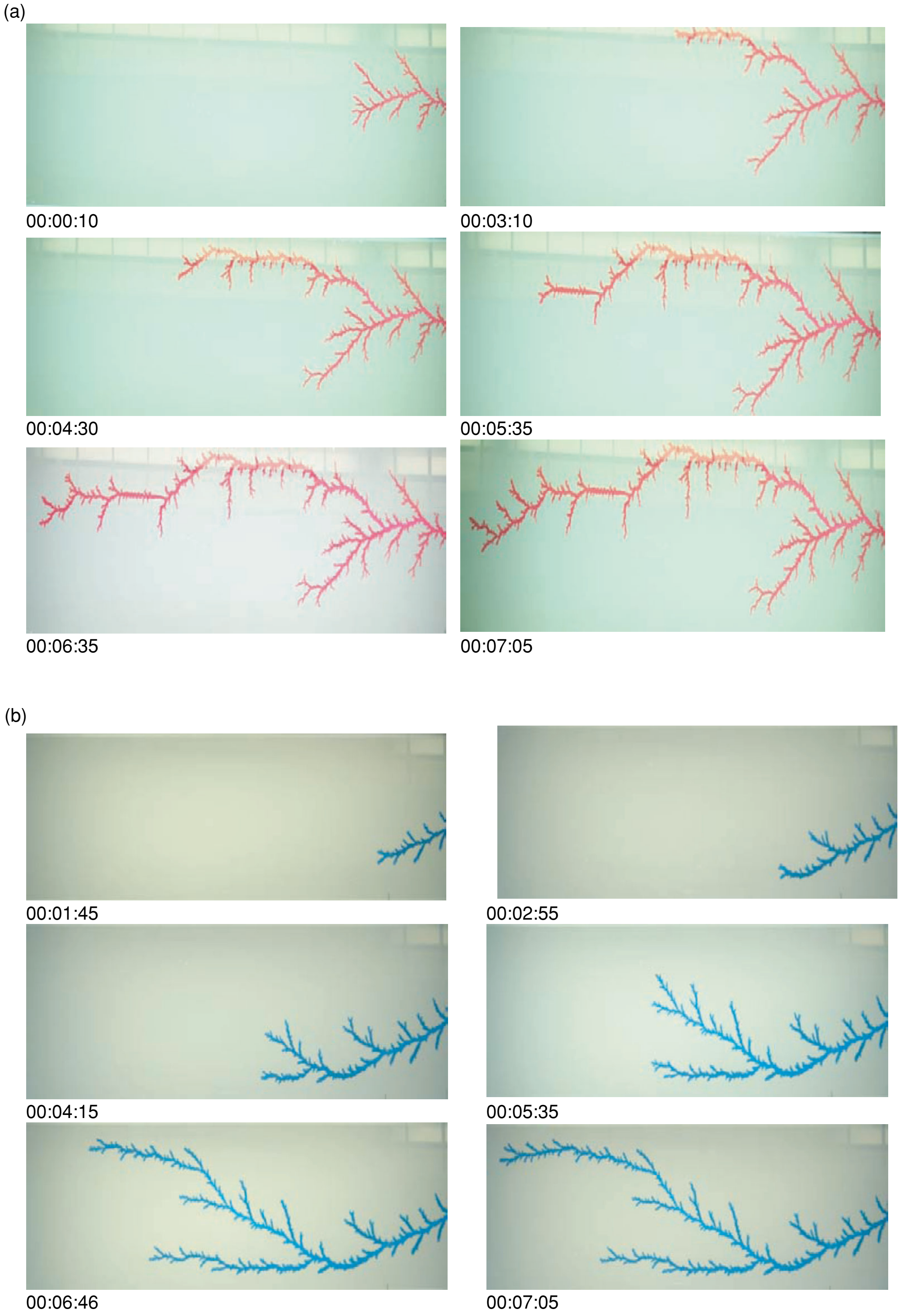


Figure 3. Displacement of *aged* emulsion: (a) 40% emulsion, (b) 60% emulsion.

3.2. Quantitative characterization of the displacements

In this section, a quantitative analysis of the displacements is presented. Image J software was used to analyze the images taken during the experiments and a number of important quantitative parameters were generated to characterize the flows. These parameters are the breakthrough time (t_{bt}), defined as the time where the injected water reaches the other side of the cell, and the sweep efficiency (E_V).

The latter property is widely used in reservoir engineering to characterize how effective the displacement process is, and is defined as the fraction of the area contacted by the displacing fluid to the total area behind the displacement front. Since the sweep efficiency is a time dependent variable, the average sweep efficiency (E_{Vavg}) obtained as the average over time of the different E_V and the breakthrough sweep efficiency (E_{Vbt}) corresponding to the sweep efficiency at the breakthrough time are presented.

Table 2 summarizes the values of the quantitative characteristics in the case of the 40% emulsion. Similar results and trends were obtained for the 60% emulsions but their discussion will be omitted and are not presented here for conciseness. The results in Table 2 indicate that the breakthrough time decreases with aging time, and that the decrease is very noticeable for the early aging but tends to be smaller for large aging times. The decrease in the breakthrough time can be easily explained from the change in finger structures from wide fingers to a single branched finger that extends rapidly towards the production side. Similar trends are observed in the case of the sweep efficiencies and can be also attributed to the changes in the finger structures that result in the displacing fluid not sweeping efficiently as the emulsions are aged.

We should close this section by noting that even though the previous results were based on the same injection rate of 0.21ml/min, similar trends were also obtained for other injection rates and will not be presented here for the sake of brevity.

The previous results indicate that the dynamics of the flow are different depending on the age of the emulsions. Since the viscous fingering instability is mainly driven by the difference in the viscosities of the displacing and displaced fluids, it is suspected that the viscosities of the emulsions vary with time and that this variation may actually be the result of changes in their microstructure. In order to investigate this, the rheology and microstructure of the emulsions were measured for both the 40% and 60% emulsions. The results of these measurements are presented in the following section.

3.3. Characterization of the emulsions

3.3.1. Viscosity Measurements

The steady shear viscosities of the emulsions were measured using a stress/rate controlled coaxial-cylindrical viscometer THERMO HAAKE RotoVisco-1 with a temperature controller. Each one of the figures below shows results for two samples that were prepared under the same conditions and with the same concentration.

Figure 4 depicts the viscosity of the 40% emulsion for both *fresh* and *aged* solutions. For the fresh emulsions, there is a wide variation particularly at very low shear rates. This variation may be due to the limited sensitivity of the rheometer for small shear rates and low viscosities. It may be also inferred that the fresh 40% emulsion shows a shear-thinning behaviour whereby the viscosity decreases with increasing shear rate (Figure 4–a). This trend was noticeable for one sample while the other one did not exhibit the shear-thinning character. The viscosity curves are drastically different when one considers the aged 40% emulsion (Figure 4–b). In this case the low shear rate viscosity is almost one order of magnitude larger than its fresh counterpart; however it decreases rapidly with increasing shear rates and reaches similar values to the fresh emulsions for large shear rates. It is worth noting that the aged solution is clearly shear-thinning and that the results obtained for both samples are similar.

Viscosity variations of the fresh and aged 60% emulsions are shown in Figure 5. The fresh solutions exhibit a clear shear thinning behaviour with similar trends to those observed in the case of the aged 40% emulsions, though the viscosity of 40% emulsion is systematically smaller. Similar to what has been observed in the case of the 40% emulsion, aging results in low shear rate viscosities that are one order of magnitude larger than those of the fresh emulsion and a stronger shear thinning behaviour.

Table 2. Quantitative analysis results for 40% emulsion at different aging times.

Aging time hr:min:sec	t_{bt} hr:min:sec	E_{Vbt} %	E_{Vavg} %
03:15:00	00:39:00	29.77	29.52
07:30:00	00:11:00	8.40	10.44
25:30:00	00:08:43	5.89	05.90
30:10:00	00:07:25	5.03	04.91

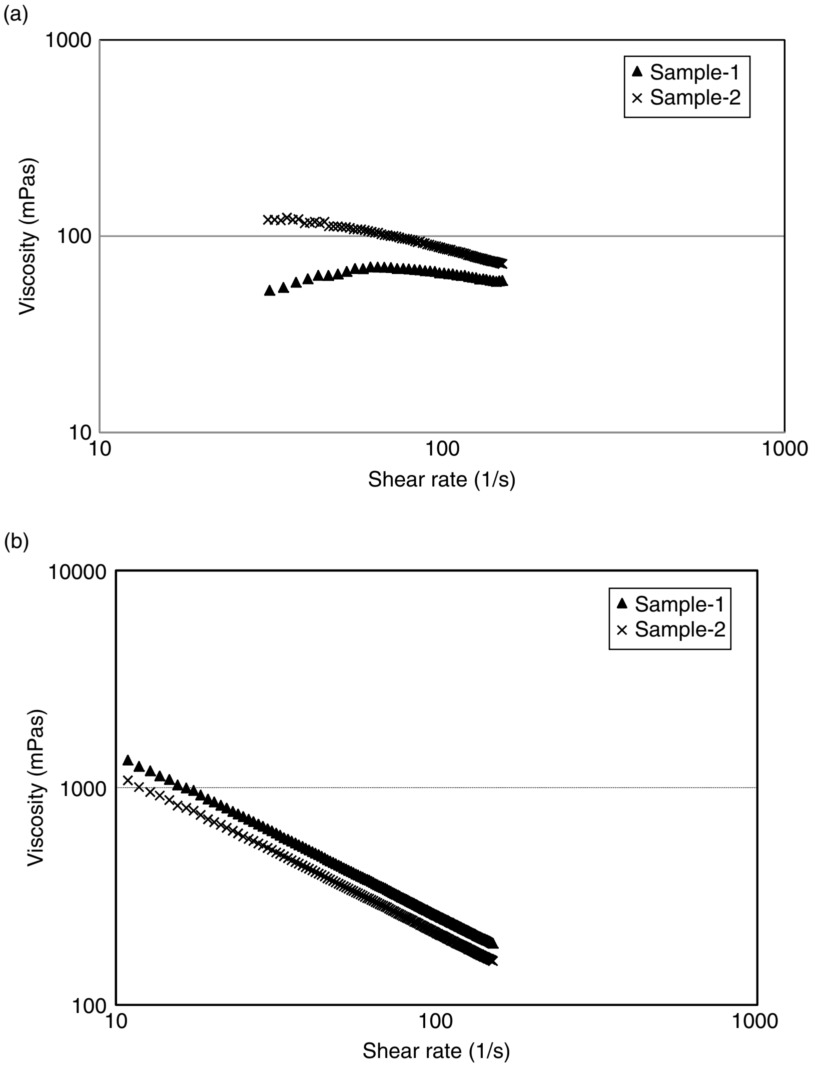


Figure 4. Viscosity vs. shear rate for 40% emulsion: (a) *fresh*, (b) *aged*.

3.3.2. Drop Size Distribution of the Emulsions

The emulsions consist of droplets of oil dispersed in water. The size and distribution of the droplets affect the physical properties of the emulsions and in particular their rheological behaviour. this section, results of droplet size analyses are presented for both *fresh* and *aged* solutions in attempt to explain the important changes in the viscosity of the emulsions with aging. The analyses were carried using a laser diffractometer Mastersizer 2000 (Malvern Instruments Co, UK) with Hydrosizer 2000S module. The results were obtained based on the intensity of the light scat tered by the droplets in the range 0.02 to 2000μm. All measurements were carried in triplicate the average values are reported with their respective error margin. The droplet size distribution was characterized using different mean diameters; the equivalent surface area mean diameter D[3, 2] and the equivalent volume mean diameter D[4, 3].

$$D[p,q] = \frac{\sum D_i^p n_i}{\sum D_i^q n_i} \tag{1}$$

Tables 3 shows mean diameters for the fresh and aged 40% and 60% emulsions. It is clear the diameters are larger for the lower concentration emulsions; however there is no clear indication of changes in the mean diameter with aging. Hence it can be concluded that the changes in viscosity of the emulsions with aging cannot be explained by changes in the droplets size. However, other factors

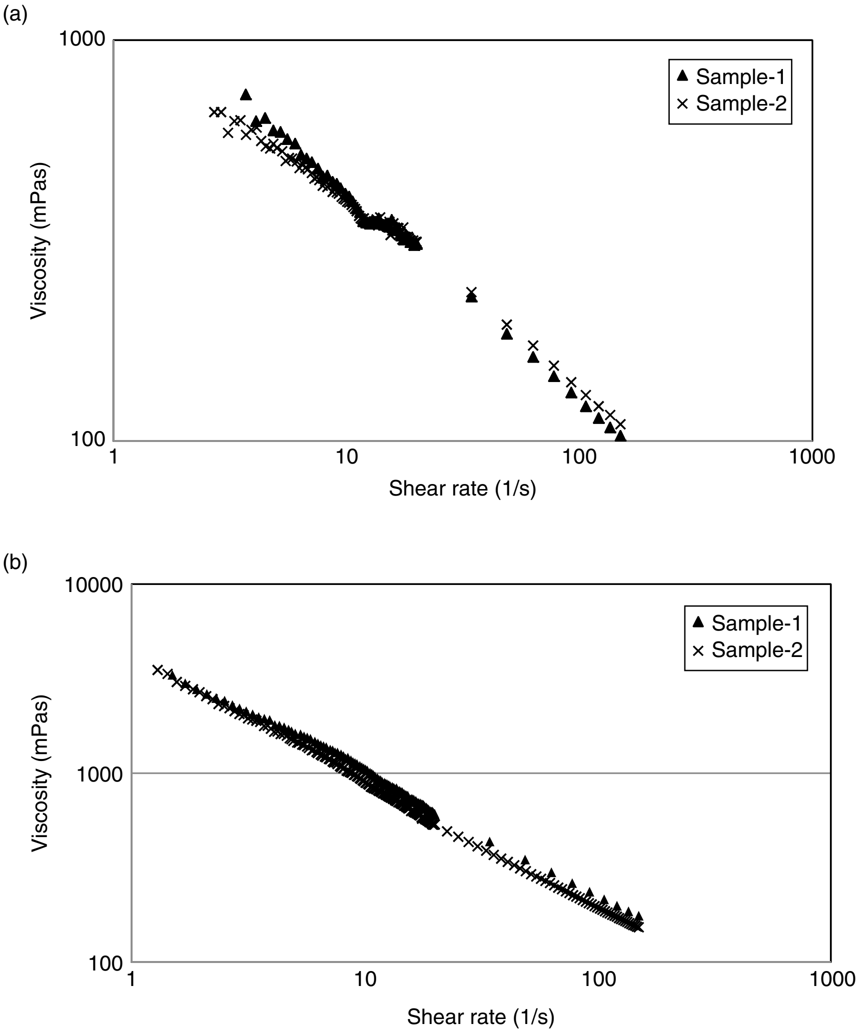


Figure 5. Viscosity vs. shear rate for 60% emulsion: (a) *fresh*, (b) *aged*.

Table 3. Mean diameters for the 40% and 60% emulsions.

% emulsion	***Fresh* emulsions Mean diameters**		***Aged* emulsions Mean diameters**	
	D [4,3] $^{\mu m}$	D [3,2] $^{\mu m}$	D [4,3] $^{\mu m}$	D [3,2] $^{\mu m}$
40	1.114 ± 0.060	0.294 ± 0.016	1.128 ± 0.053	0.304 ± 0.018
60	0.358 ± 0.055	0.159 ± 0.019	0.462 ± 0.079	0.168 ± 0.031

that include flocculation or the presence of thick adsorbed layer of surfactant continuous phase liquid may contribute to the changes [33–36]. In what follows, results of analyses of the emulsions microstructure are presented.

3.3.3. Microstructure of the Emulsions

In the process of determining the size distribution of the droplets, the samples were diluted and as a result any flocculation that may have taken place would have been broken. In an attempt to understand the mechanisms responsible for the viscosity increase with the aging time and to find if there are any flocculation or structural changes, image analyses of the microstructure were carried out. Emulsion microstructure was visualized using a Carl Zeiss Axiovert S100 inverted microscope. This microscope is equipped with a video camera and an Axio Vision Release 4.6.3 analysis software. The main purpose

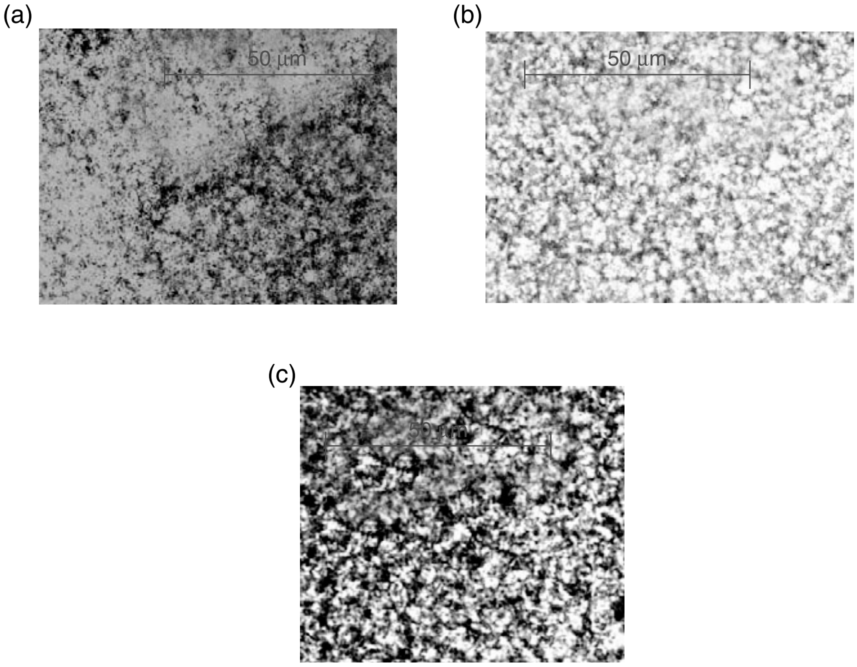


Figure 6. Microstructure of the 40% emulsion at aging time (a) 4 hrs (b) 30 hrs (c) 4 days.

of these measurements is to observe possible changes in the droplets microstructure including droplet flocculation which is an instability phenomenon that raises the viscosity of emulsion. In the process of flocculation, the droplets are attracted to each other but are separated by a thin film of the continuous phase.

Figure 6 shows the microstructure of the 40% emulsion at different aging times. At an aging time of 4 hrs the emulsion sample contains very fine droplets. As the aging time increases to 30 hrs, there seems to be some flocculation of oil droplets. Further increase in aging time actually leads to even more flocculation. In order to have clear image of what is happening with time, image analysis was also carried after 4 days. This shows even larger drop sizes compared to the aging time of 30 hrs.

Similar results were also obtained in the case of the 60% emulsion where the observed size of the droplets tends to increase with aging.

5. CONCLUSION

The viscous fingering instability that results from the displacement of oil-in-water emulsions by water in a rectilinear Hele-Shaw cell has been examined experimentally. The experimental measurements were conducted for two concentrations of the emulsions, 40% and 60% (v/v %) that were displaced at a constant injection rate of 0.21 ml/min. It was found that the flow patterns differ substantially based on the age of the emulsion. Fresh emulsions that have been used to run the flow displacements 3 hours or less from the time of their preparation resulted in smoother and less complex finger structures than the aged ones (30 hours old) which tend to exhibit strong ramified branching and needle-like fingers. Furthermore, it was found that the aged emulsions show a shorter breakthrough time and a smaller sweep efficiency which implies that it is more difficult to displace efficiently O/W emulsions as they age and that the earlier the displacement is run, the more efficient it is.

The qualitative and quantitative differences between the fresh and aged emulsions should be attributed to differences in their rheological properties. Even though the oil concentration remains the same, it is suspected that changes at the microscopic level may have resulted in new viscous behaviour. Rheological measurements revealed that indeed the steady shear viscosity of the emulsions change enormously with age. In particular, the fresh 40% emulsions exhibit a Newtonian behaviour while the aged ones are shear-thinning with low shear-viscosity one order of magnitude larger than its fresh counterpart. Similar, though less spectacular, changes were also observed in the case of 60% emulsion. One may surmise that these changes in the rheological behaviour with aging are the result of rearrangements in the microstructure of the emulsions. Hence, analyses of the microstructure and measurements of the droplets size distribution were conducted for the two emulsions' concentrations at different ages. It was found that there are no significant changes in mean drop size as the emulsions aged. However, the visualization of the emulsions microstructure under the microscope revealed that as the emulsions aged, there is a tendency for the droplets to agglomerate together and form larger cells. The droplets are getting closer and attached to each other probably either by bridging or depletion flocculation. This process was found to continue with time and the microstructure continues to change even after larger time spans. It is suspected that the flocculation explain both the increase in the viscosity as well as the shear-thinning non-Newtonian behaviour. The flocculated oil droplets tend to result in larger shear viscosity and furthermore, such structures will resist more deformation at very low shear rates, hence the large small shear viscosities. However, as the shear rate increases, this may allow it to destroy the flocculated structures leading to finer oil droplets and hence a rapid decrease of the shear viscosity.

The present study illustrates the importance of the micro structural changes of oil-in-water emulsions with time and the consequences on the flow displacements of such emulsions. It is clear that the tendency for the droplets to flocculate affects the rheological behaviour of the emulsions and hence the finger structures that develop in the flow. Even though this study showed conclusively that the changes in the microstructure with age affect the flows, it is still not clear if other.

mechanisms in addition to flocculation, may have played a role in the observed changes of the rheological behaviour. In particular, possible adsorption of the surfactant on the surface or its migration may have also contributed to changes in the response of the droplets to deformation and hence to the viscous behaviour of the emulsions. Even though this and other aspects have not been explored in the present study, it is believed that they should be analyzed in future studies.

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