Flow characteristics of concentrated emulsions
of very viscous oil in water

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Abstract

This paper advances ideas and presents experiments on the flow characteristics of concentrated emulsions of Venezuelan bitumen in water plus surfactant. These emulsions are studied under a variety of flow conditions, namely between rotating cylinders, in a colloid mill and in pipes. The ideas advanced here concern the modeling of the highly viscous bitumen drops as solid spheres and their fracture under contact forces between neighboring drops, as in comminution, rather than break up by hydrodynamic forces. Further, we observe and discuss the local inversion of an emulsion due to local increases of the bitumen fraction induced by flow and the conditions that lead to slip flow, in which the drag is reduced by the formation of a lubricating layer of water at the wall.

The identification of flow characteristics of highly concentrated dispersions of bitumen droplets in water plus surfactant is complicated beyond the complexities inherent to composite media because of lubrication effects at bounding walls, drop refinement by contact forces, as well as by hydrodynamic forces, and by local inversion instabilities. In this paper we shall identify flow characteristics of concentrated emulsions of heavy Venezuelan bitumen in water, emphasizing novel features which are associated with the high viscosity of the disperse phase. Such emulsions are currently being marketed worldwide as coal-substitute fuels, under the name of Orimulsion.

The paper is divided in two parts: Part I deals with the conceptual framework of this research and part II outlines the experiments carried out to support our conclusions. The review of relevant literature is carried out in Part I.

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Part I: Concepts

1. Highly concentrated emulsions

Our attention is focused on highly concentrated emulsions of a very viscous oil or bitumen in water. In the case of an ideal monodisperse emulsion, the maximum volume fraction that the disperse phase can reach is smaller than the value $\phi = 0.7405$, corresponding to hexagonal close packing of spheres. In the present study, we consider polydisperse distributions centered at one (unimodal) or two (bimodal) values. Because our emulsions are polydisperse, they allow for strictly spherical emulsions even in rather highly concentrated situations, say $\phi = 0.70$ for the unimodal emulsion and $\phi = 0.80$ for the bimodal counterpart. It follows that our emulsions are not as concentrated as the ones considered in the landmark studies of Princen [1979] and Princen, Aronson and Moser [1980].

The aforementioned studies of Princen et al. focused on the equilibrium of mechanisms, thermodynamic and geometric, which could explain the existence of static bidispersed emulsions with volume fractions greater than the maximum packing fraction for monodispersed dispersions. These mechanisms require that spherical drops deform and flatten at touching points and the development of such flat places can be generated, for example, by non-zero contact angles between the thin film, the flat portions of contacting drops and the adjacent plateau borders. Princen's [1979] two-dimensional theory is capable of predicting the maximum attainable volume fraction as a function of drop size, equilibrium film thickness, contact angle and the maximum disjoining pressure that can be developed between films.

We have already noted that the packing fractions of our polydisperse systems are below the maximum allowed for monodispersed spheres in a static emulsion. In a dynamic emulsion, gradients of ϕ will be generated by motion and the maximum packing fraction can be achieved locally leading to the local inversion of the emulsion. Some of the considerations in Princen's work are possibly relevant to the phenomena observed in this work. Another very important feature which distinguishes our work from Princen's is the highly viscous nature of the disperse phase. The heavy crudes and bitumens are perhaps 10,000 times more viscous than the paraffin oil used in the experiments of Princen et al. [1980,1983,1989]. Of course, viscosity is a property that is relevant to motion and not to equilibrium. However our emulsions are concentrated, but not

maximally packed, so that the effects of the local concentration of an emulsion in a flow could be greatly influenced by the surpassingly large viscosity of a disperse phase of heavy crude.

2. The viscosity of an emulsion

Very high concentrations of heavy crude oil and bitumen (over 70%) can be emulsified in water by using surfactants and appropriate mixing strategies. The viscosity of the emulsions is claimed to be as much as three orders of magnitude smaller than the viscosity of crude oil. This has motivated the consideration of pumping heavy crudes through pipelines as concentrated oil-in-water emulsions. Lamb and Simpson [1973] report a commercial line in Indonesia which carries 40,000 barrels/day of 70% oil-in-water emulsion in a 50,8 cm (20 inches) diameter line, 283 km long; Zakin et al. [1979] also describe another commercial line. Laboratory studies of this type of transport have been reported by the two authors just mentioned and by Rose and Marsden [1970], Marsden and Raghavan [1973], Mao and Marsden [1977], Briant et al. [1982], Giordano et al. [1988], Layrisse et al. [1988], Pilehavri [1988], Plegue et al. [1989], Zhang et al. [1991], and Sanchez and Zakin [1991]. Zakin et al. [1979] and Sanchez and Zakin [1991] note that, although the viscosity of the emulsion increases with oil fraction, the energy loss per unit mass of oil decreases. Downhole emulsification of heavy oils appears to be a useful technique for improving production (see Simon and Poynter [1968], Mourits [1990] and McCafferty and McClaflin [1992]).

The viscosity of a concentrated emulsion of small, highly viscous drops with volume fractions less than the maximum packing fraction, should be independent of the viscosity of the disperse phase for large viscosities. This idea is consistent with the observation that the pressure drop in turbulent flow of emulsions tends to be independent of the oil viscosity, see Pilehavri et al. [1988] and Sanchez and Zakin [1992]. An emulsion with viscous disperse phase resembles a concentrated suspension of solid spheres of the same sizes and concentrations. The effect of the high internal phase viscosity is to inhibit relative internal motions and deformations of the bitumen spheres. This feature allows us to use theoretical and empirical relations developed for the viscosity of fluidized suspensions of solid spheres to describe the viscosity of the emulsion.   The reduction of drag in pipe flow of bitumen alone and bitumen-in-water emulsions is of the order of the ratio of the viscosities. This ratio is not relevant for the calculation of the viscosity of the suspension, provided only that the viscosity of the

viscous disperse phase be large enough to render the internal motion of the drop nil. A bitumen drop can be thought to be a solid sphere with an infinitely great viscosity.

The determination of an effective viscosity of a suspension of solid spheres can be made reliable when the volume fraction is small, say less than 0.6, as the viscosity is mainly a function of ϕ. The viscosity of more concentrated suspensions is more difficult to evaluate as it depends on details of the packing and size distribution, the interparticle interactions and even on the nature of flow in the viscometer, as well as on the volume fraction of the spheres.

One of the main impediments to the determination of the viscosity of a fluidized suspension of solids or bitumen is polydispersity. One technique for modeling polydisperse suspensions is to treat them as if the continuous phase and the small colloidal particles were a single composite fluid which forms a pseudo-continuous phase, in which the larger particles are suspended. Typically, the latter are modeled as a unimodal suspension. Farris [1968] developed an effective medium model in which the small particles in suspension were viewed as a continuum with respect to large ones. He showed that when the size ratio of large to small particles is large (>10), the viscosity of a multimodal suspension may be estimated from the viscosity-concentration behavior of a unimodal suspension, considering the fine particles and the fluid as a homogeneous phase. Sengun and Probstein [1989] modeled a polydisperse coal slurry as a bimodal dispersion in which the fluid, plus colloidal particles form the pseudo-fluid, and the non-colloidal fraction is modeled as monodisperse. This theory does not require that the size ratio be large. Further developments in bimodal modeling have recently been discussed by Probstein, Sengun and Tseng [1994]. They note that:

A polydisperse suspension with a particle size distribution from submicrometer to hundreds of micrometers is treated as bimodal, wherein it is made up of a colloidal fine fraction and a non-colloidal coarse fraction. According to the bimodal model, the fine fraction imparts to the suspension its non-Newtonian characteristics and behaves independently of the coarse fraction, whereas the coarse particles only raise the apparent viscosity through hydrodynamic interactions. It is shown that when the particle microstructure is random, the contribution to the viscosity of the coarse fraction, which is generally polymodal, is characterized by lubrication concepts with the maximum packing fraction ϕ_m as the scaling parameter.

Poletto and Joseph [1994] have shown that the concept of a fluid-solid-pseudo-fluid can be used to define an effective Newtonian fluid in Stokes flow, in which the

effective density is the average density of the mixture and the viscosity of the mixture is predicted by the empirical correlation of Thomas [1965]. This theory works well for single test particles suspended in the pseudo-fluid, even when the test particles and particles of the pseudo-fluid are of the same size.

The concept of a pseudo-fluid seems not to have been tried for bitumen-in-water emulsions, though these should be identical to solids in fluids whenever the flow is such that the structural integrity of the emulsion is left intact. That is, when there is no change in the drop size distribution and no local inversions. It will be shown later that these conditions appear to hold for emulsions with bitumen fractions under 0.7 or even larger in the case of bimodal emulsions. The effect of fines in reducing the effective viscosity of a bimodal dispersion seems to be identical in bitumen-in-water and solid spheres-in-water. Chang and Powell [1994] have noted that, for a fixed volume fraction of particles, the viscosity of the systems with a bimodal distribution of solids is less than the viscosity of a suspension with a unimodal size distribution of particles. D'Haene and [Mewis] 1994 show that the viscosity of a bimodal dispersion attains a minimum when the core volume percentage of fine particles is of the order of 30%. The same results hold for the suspensions of bitumen-in-water which are considered here, as can be seen in the work of Rivas, Dalas and Núñez [1993]. Owing to the nature of their packing, bimodal systems have greater critical packing fractions, which explains the viscosity reduction relative to a unimodal dispersion at the same disperse phase fraction.

An interesting recent study by Aral and Kaylon [1994] of wall slip in a steady torsional flow of a concentrated suspension (ϕ = 0.63 %) of glass beads in a polymeric liquid, reveals dynamic analogies between the former system and bimodal 80% bitumen-in-water emulsions. In these emulsions, the large hydrocarbon drops can be thought to be suspended in a pseudo-fluid of water and small bitumen particles, the fraction of pseudo fluid not greatly different than 37%.

3. Lubricated flows of emulsions

In flow of bidisperse systems of greatly different viscosity, there is a tendency for the low viscosity constituent to migrate into regions of high shear, lubricating the flow (see Joseph and Renardy [1992], for a detailed discussion). The bounding wall of a pipe in Poiseuille flow, or the cylinder walls in flows between rotating cylinders, form the

locus of lubricated regions which lead to great reductions in drag. Core-annular flow of crude oil in water is an example of the lubrication of a more viscous by a less viscous liquid, and the lubrication of an emulsion by a slip layer of water on the wall is another example. The same kind of lubrication occurs in shear flows of concentrated suspensions of solid particles with the caveat that the migration of particles away from regions of high shear near the wall actually creates a high viscosity region away from the wall. In all these cases, it is probable that lubrication forces of the kind that keep single particles off walls, like the Segré-Silberberg effect, provide the mechanism which levitates the high viscosity constituents off the wall.

The formation of a slip layer or lubrication layer at the walls of a pipe leads to substantial reduction of the drag above or beyond the already significant reductions achieved by emulsification (Zhang et al. [1988], Pilehavri et al. [1988] and section 7 of this paper). It is apparently not known under what conditions a flowing concentrated oil-in-water emulsion will lubricate or lose lubrication. For example, Pilehavri et al. [1988], have reported that an emulsified Wyoming crude did not slip in a concentric cylinder rheometer, but that two different emulsions of Venezuelan crudes did slip. Zhang, et al. [1991] reported that the strong reductions of drag on emulsions of a very viscous heavy crude, which they observed in the laboratory, did not carry over to tests in the field.

The influence of the material of pipe wall construction on the flow and slip at the wall of concentrated fuel oil-in-water emulsions may have been first studied by R.J. Mannheimer [1972]. He noted that the apparent absence of a yield stress in Teflon tubes can be explained if it is assumed that a film of oil forms spontaneously when the emulsion contacts a strongly hydrophobic surface. H.M. Princen [1983], in his pioneering work on the rheology of foams and highly concentrated emulsions of paraffin oil-in-water, notes that:

To obtain meaningful and reproducible results, it appears essential that the viscometer walls be perfectly wetted by the continuous phase. Otherwise, the surface induces instability of the emulsion layer adjacent to the wall.

Princen and Kiss [1989] note that stainless steel rotating cylinders are not usable for studies of such emulsions because they are hydrophobic:

With highly concentrated O/W emulsions, the oil droplets have a strong tendency to "coalesce" on the walls and to form a thick oil layer when shear is applied (wall-induced instability). This leads to erratic behavior. The problem may be solved by using....glass cylinders.

In section 6 of this paper, we show that the bitumen emulsions under consideration tend to foul stainless steel walls more than carbon steel walls and that the surfactants used to promote emulsification also promote the hydrophilicity of the carbon steel wall.

4. Fracture of bitumen drops in a concentrated emulsion.

Rock breaking machines are used to reduce the size of solids particles by crushing (comminution). The fracture of solids particles may also be achieved by shearing in certain circumstances. For example, in the study of wall slip in the torsional flow of a suspension of 63% glass spheres in a polymeric liquid, Aral and Kaylon 1994 found that increased surface roughness prevented wall slip and it frequently resulted in the fracture of the glass spheres. If the analogy between solids and concentrated suspensions of bitumen drops is valid, similar results could hold for the concentrated emulsions. Indeed, the bimodal distributions of bitumen drops of emulsions with bitumen-to-water ratio of 80:20 can be transformed into a unimodal distribution with a marked reduction in the population of large drops. This anomalous behavior is consistent with the notion that the large drops are fractured by contacts with other large drops as is the case in the comminution of solid spheres (Goddar [1991]). For the same bimodal distribution of drops of much smaller viscosity a hydrodynamic, rather than direct contact, mechanism ought to apply. Indeed, the referred comminution mechanism does not operate for the same bimodal distributions of much less viscous lubricating oils, as will be shown in section 6.

5. Local inversion.

Of course, the analogy with suspensions of solids should not be carried too far. Concentrated emulsions of heavy hydrocarbons-in-water can be unstable to creaming or inversion instabilities.

A very concentrated emulsion of oil in water is prey to an inversion instability. Obviously, if the flow further concentrates an already concentrated emulsion, the oil fraction may test the maximum packing fraction locally. One example of a local flow-induced increase of the concentration of the disperse oil is the ring instability of a uniform dispersion, discussed in section 6. Inversion is apt to occur at the places in the flow where the flow induces the maximum packing fraction.

In a local inversion, portions of O/W emulsion invert to W/O with small water fractions. This could also be described as drop coalescence, but with the additional caveat

that the resulting coalesced drop contains water; it is a water-in-oil drop. The instability here follows from the fact that the coalescence that was produced in a region starved of water, actually increases the degree of starvation by absorbing water.

Our experiments are consistent with ideas corollary to local inversion. Suppose the original composition of an emulsion is 70% oil and 30% water and, after shearing in the conditions described in section 8, this emulsion partly inverts. We are left with a water continuous phase having 70% or less of small bitumen drops, free of water, and large bitumen drops that contain water. A completely inverted emulsion would have the same volume fraction but with an oil-continuous phase and small dispersed water drops. When we separate the large bitumen drops fouling the pipe wall from the emulsion, we are left with a water-continuous phase of pure bitumen in water. The large bitumen drops contain some water proving that coalesced drops have inverted. The bitumen and water missing from the water-continuous phase emulsion must be in the large drops.

The mechanism by which inversion of an emulsion occurs seems not to be well understood. In the case under consideration, we would want to understand how water enters into the lumps that arise from the coalescence of bitumen drops. One possibility is that when bitumen drops are squeezed together they join under short range disjoining pressures at many local points of close approach. This means that the water in Plateau like borders would be encapsulated at many points in the network of coalescing drops producing a local inversion. This kind of inversion mechanism should work better for softer bitumen drops, so we expect more inversion at high than at low temperatures.

The effect of large pressure gradients on a concentrated oil-in- water emulsion can be considered. Probably in the first approximation to the motion, the water and oil will move in locked step, but further considerations leads into the idea that the water and oil could not have the same response to a pressure gradient but there are many possible responses. Since water is the continuous phase, it can flow through the bitumen matrix as in the flow in a porous media. In general, one would expect water to be squeezed out of the matrix of bitumen drops or to be absorbed by them in local inversions at places where the gradients are largest. In the cases when no inversion occurs, the water squeezed from some places must migrate to the walls. In a lubricated flow, there is a pressure gradient but no further migration in water. It would be difficult to establish a lubricated flow at places where the pressure gradients are large and variable, as in contractions in pumps and valves. A consequence of this is that pumps and valves are sites which promote further concentration of an already concentrated emulsions in procesing operations leading to local inversions through encapsulation.

Part II: Experiments with highly concentrated emulsions

Experiments were carried out between rotating cylinders, in a colloid mill and in pipes, both in the laboratory and in the field.

6. Flow between rotating cylinders

6.1 Introduction

We focus here on the hydrodynamic behavior of concentrated bitumen-in-water emulsions between rotating concentric cylinders (see Fig. 6.1). To eliminate unwanted secondary motions (Taylor vortices), the outer cylinder rotates as the inner one is stationary. Torque readings as a function of rotating frequency were obtained and changes in the distribution of particle sizes and temperature were monitored. Of paramount importance is the accurate measurement of torque. Proper alignment and balancing is the key to success. To that end, all cylinders were carefully machined in a lathe, mounted and verified for alignment before each run. Torque was measured by means of a transducer made by HBM, Germany. The rotating frequency range of the apparatus was from 0 to 3000 rpm, which was achieved using the motor and a belt-pulley system described in Fig 6.1. The inner and outer cylinders, were either of stainless steel or plexiglass, and their diameters (60 and 70 mm the inner ones and 75 mm the outer cylinder) allowed for gaps of 2.5 and 7.5 mm. Three iron-constan thermocouples were flush-mounted in the inner cylinder, in order to monitor temperature changes during a given run.

6.2 Experimental procedure

Emulsions of Orinoco bitumen (8.3° API) in water, stabilized with a commercial surfactant Intan-100, which is a nonyl phenol ethoxylated compound having 17.5 EO units, were prepared with a conventional blender at 60 °C. At 30° C this bitumen has a viscosity in excess of 1×10^5 mPa.s and the density of water. The surfactant concentration was varied between 2500 and 3000 mg/l based on the fraction of dispersed phase. The formation procedure was controlled in such a way as to produce either a unimodal particle size distribution of about 20 ± 2 µm of mean droplet diameter or a bimodal distribution. The latter had a mode of 30 ± 2 µm mean droplet size and a mode with a mean of 3 ± 2 µm. For the unimodal case, the bitumen fraction was kept constant at 0.7,

while bimodal emulsions were prepared with internal fractions of 0.6, 0.70, 0.75, and 0.80.

Torque readings as a function of the rotating frequency were taken for each sample, and the temperature history was recorded in every experiment. Viscosity and particle size distribution were measured before and after each run, so that structural changes in the emulsion could be assessed. An instrument based on a light diffraction technique (Malvern, model Master Sizer) was used for the droplet size evaluation. Viscosities were evaluated by means of a concentric cylinder viscometer (HAAKE, model RV-20/M5).

6.3 <u>Results and discussion</u>

All of the emulsions we studied entered into a lubricating regime in which the torque leveled out (and sometimes, even decreased) as the rotating frequency increased, (Figs. 6.2 to 6.4). Hydrophilic wall materials promote the lubrication of the emulsion with water. Stainless steel cylinders are more hydrophobic than plexiglass cylinders, they exhibit more fouling by bitumen and require larger torques to turn at fixed speeds (Fig. 6.3). The torque 6.3a <u>Lubricating Results</u>

measurements are also more erratic for flows between steel than plexiglass cylinders (Fig. 6.2b).

6.3.b <u>Effect of Type of Drop Size Distribution</u>.

Two classes of emulsions were considered. The first class was a bimodal distribution with bitumen fraction of 0.80,0.75,0.70 and 0.60. For the unimodal case, only the 0.70 bitumen fraction was studied. The dynamic response of these fluids in the rotating cylinder apparatus is shown in Fig. 6.2a, giving the torque as a function of the rotating frequency when the gap is 2.5 mm and the wall material is plexiglass. The torque curves depend strongly on the bitumen fraction with high torques for concentrated emulsions. The most concentrated emulsion with a 0.8 bitumen fraction gives rise to an overshoot which is not seen for smaller bitumen fractions. Torque values for unimodal emulsions are higher than for bimodal emulsions of the same bitumen fraction because the effective viscosity of a uniform dispersion is higher. Fig. 6.2 shows that the unimodal emulsion with 0.7 gives rise to higher torques than the 0.75 bimodal counterpart.

The overshoot of torque for the highly concentrated 0.8 bitumen fraction is examined more closely in Figs. 6.4 and 6.5(a). The overshoot marks a drop distribution crisis in which the large diameter mode is anihilated catastrophically in an event which transforms the 0.8 bimodal distribution to a unimodal one (Fig. 6.4) (to get this data we needed two

runs, one before the catastophe and the other after). The fact that all the torque vs mean shear rate curves are the same before the anihilation of the large diameter mode points to the importance of packings of spheres; similar packings give rise to similar responses. The viscosity of the unimodal dispersion created by the anihilation of the large drop fraction is larger than the viscosity of the bimodal dispersion and it leads to greater frictional heating (Fig. 6.5a). The effects of drop refinement at the overshoot is seen in less dramatic form in the 0.75 bimodal dispersion and it is virtually absent in bimodal dispersions with 0.7 or less bitumen fractions.

The aforementioned break-up mechanism is unusual and perhaps closely related to a size reduction process found in the crushing of solids, known as comminution. Ghaddar [1991] noted that the fracture efficiency of a cylinder piston compressing bed is enhanced in bimodal particulate systems; fines (the equivalent of the small diameter mode) are accumulated in the void spaces between unbroken particles, facilitating the transmission of the applied stress and inducing rapid break-up. A similar enhancement of the efficiency in the breaking of large drops of bitumen in concentrated bimodal dispersions can be attibuted to the presence of "fines". The comminution type of breakup of bitumen drops depends on their high viscosity which makes them solid-like. To test this idea, we did the rotating cylinder experiment with a lubricating oil-in-water emulsion identical to the bimodal $\phi = 0.8$ bitumen-in-water emulsion, except that the viscosity of the lubricating oil (1 Pa.s) was 100 times smaller than that of the bitumen. We did not observe a change in the drop size distribution. The results are shown in Fig. 6.6. The overshoot is absent; apparently, the low viscosity dispersed phase deforms rather than fractures under the action of applied stresses.

7 7. Pipe flow

7.1 Introduction

Here we study the development of lubricating regimes of unimodal and bimodal bitumen-in-water emulsions in Poiseuille flow using a controlled-pressure tube viscometer of small diameter and a once-through tube viscometer which mimics commercial pipes. We evaluate emulsions with volume fractions from $\phi = 0.65$ to 0.80, with unimodal and bimodal distributions.

7.2. Experimental procedure

The controlled-pressure tube viscometer is shown in Fig. 7.1 (a). The experiments were carried out in four stainless steel capillary tubes of equal length (3 m) with internal diameters of 1.3, 3.6, 7.5 and 10.6 mm. The emulsions were driven into the test section by pressurized air or Nitrogen. To minimize end effects, the upstream pressure gauge was placed at a distance greater than 60 pipe diameters from the tank discharge. The pressure drop was calculated by subtracting the atmospheric pressure at the outflow end from the absolute pressure measured upstream. The mass flow rate was measured by using a digital precision balance (Mettler, PE 16) and a stopwatch.

A different set of data was gathered in a two-legged once-through tube viscometer, whose schematic representation is shown in Fig. 7.1(b). Two tanks are connected by a 21.7 mm diameter steel capillary tube, which made up the test section, were used in the operation. The emulsion was driven from one tank to the other by means of a positive displacement pump of gear type (Viking HL14). The mass flow rate could be varied by means of a mechanical variator attached to the pump and was determined using a Coriolis type flow meter. The pressure drop was measured in two legs of equal length (2.7 m, see Fig. 7.1(b)), with pressure transducers. The upstream and downstream ends of both devices were placed at a distance equal to or greater than 200 and 60 pipe diameters, respectively from the 'U' turn.

The samples were prepared as in section 6.4. To monitor the stability of the samples to shearing we measured the viscosity and droplet size distributions using the same instruments described in section 6.3. The controlled-pressure tube viscometer was calibrated with a blend of lubricating oil and gasoil. The Newtonian viscosity of the blend was 96.6 mPa.s and the density 895 kg/m^3 at 20ºC. The pipe viscometer was calibrated using a Newtonian crude oil with a viscosity of 123 mPa.s and a density of 918 kg/m3 at 20ºC. The temperature history was recorded in every experiment. All experiments were conducted at a room temperature of 22 ºC.

7.3. Results and discussion

7.3.1 Viscometer calibration

The main goal of the controlled-pressure viscometer calibration was the calculation of the average capillary diameters. These were determined from pressure drop-mass flow rate data. We found that average diameters could differ from the measured diameters by up to 30% for the smaller tube, and 1% for the larger tube. All data manipulations involved the use of the calculated average diameters. The calibration curve is given in Fig 7.2 and rheological behavior was Newtonian, as expected.

The once-through loop calibration was carried out in a different way, since no dimensional source of errors was expected for such a large system. In this case, the pressure drop in both legs of the loop was recorded as a function of flow rate; the flow curve is shown in Fig. 7.3. As expected, the rheological behavior of the calibration fluid was Newtonian. It is worth noting that the pressure transducer produced slightly different readings in both legs of the loop. The difference did not exceed 3%.

7.3.2   Emulsion rheological evaluation

7.3.2a Controlled-pressure tube viscometer

We collected pressure drop data as a function of mass flow rate for all the samples. All four capillaries were used in every case, with the exception of the bimodal sample of 0.7 internal phase content. The flow rate in the large diameter pipe for this less viscous sample was too large for our measuring system.

In figures 7.4 (a) to 7.4 (c) we present flow curves for the $\phi = 0.7$ unimodal, 0.7 bimodal and 0.8 bimodal emulsions, respectively. The curves are similar to those   for rotating cylinders discussed in section 6. The 0.7 unimodal emulsion exhibits higher shear stresses than its bimodal counterpart. Different flow curves are obtained for each capillary and all of them indicate that the emulsions are slightly shear thining. As the wall shear stress increases, the curves diverge; surprisingly, the small diameter tubes have smaller shear stresses. This suggests a lubricating effect, although the onset of lubrication is not as abrupt as it is for the same emulsions between rotating cylinders (see Figs. 6.2 to 6.5). Results obtained in the open once-through loop discussed at the end of this section and in the next sub-section suggest that the differences between pipes and rotating cylinders are associated with the flow development of a lubricating layer.

In all the cases presented in Figs. 7.4, no variation of the drop size with shear rate was detected even at mean shear rates of 10,000 s-1 (see Fig. 7.5). The drop size distribution of the emulsion will not change in a straight capillary tube.

The mean shear rate ($8v/D$) vs pipe diameter is shown in Fig. 7.6. A low shear stress of 200 Pa was selected to generate the figure. The shear rate for the $\phi. = 0.7$ unimodal and the $\phi. = 0.8$ bimodal emulsion tends to a constant as the pipe diameter increases. The apparent reduction of lubrication as pipe-to-droplet size diameter ratio increases can be interpreted in terms of a developing length for lubrication discussed next.

In Fig. 7.7 we plotted the shear stress vs shear rates for emulsions in a 3.6 mm capillary with different length-to-diameter ratio (L/D). At low shear rates, the shear stress is largest in the largest L/D pipe capillary. At higher shear rates, this ordering is reversed and significantly lower values of shear stress are obtained in the large L/D capillary. Bagley plots for the data in Fig. 7.7 reveal the absence of significant excess pressure (produced by end effects). This apparently anomalous behavior of the flow curves can be explained by a development length for the formation of a lubricating layer. The data shown in Fig. 7.7 indicates complete flow development of the lubrication layer for L/D = 1200. Arguably, this could be related to the distance required to reach an equilibrated phase distribution in which the pressure gradient is balance by the drag without any further lateral migration of bitumen drops. This distribution is apparently reached by pumping water from the emulsion in a squeezing action induced by the pressure gradient in the development region. This pumping of water from the emulsion is like the flow of water through sands; obviously the water and bitumen will respond differently to a developing gradient. The same kind of flow development causes the water in the flow of concentrated slurries to migrate to the wall. If water is forced to the wall, then solid particles or bitumen drops must migrate away from the wall. The exact force balance required to maintain the compressed dispersion in equilibrium with a lubricating layer is not yet well understood.

7.3.2b Once-through open loop tube viscometer

Two type of emulsions were evaluated in this facility, unimodal and bimodal with $\phi = 0.65$ and 0.8, respectively. Figure 7.8 depicts the pressure variation as a function of mass flow rate for $\phi = 0.65$. Each data point represents the mean of at least ten pressure drop readouts, the histograms of which yielded a standard deviation below 5%. The pressure drop for the 0.65 emulsion is not linear with distance as it is for the crude oil used to calibrate the viscometer; the pressure drop of the second leg of the loop is much smaller. A similar behavior is observed for the $\phi = 0.8$ bimodal emulsion shown in Fig. 7.9. It is also evident that the pressure difference between the two legs tends to increase with the mass flow rate. It should be mentioned that no change in drop size distribution was observed at any time for either emulsion. The argument given at the end of section 7.3.2a applies here. A development distance is required for the formation of a lubricating water layer which for the controlled-pressure case exhibited in Fig. 7.7 is about 1200. By accident, the distance to the second leg of the open loop is about 1200, so we might expect lubrication to occur there. Of course, the effect of lubrication layers can be expected to be, and is more pronounced in the more highly concentrated emulsion.

This effect of concentration is made evident by comparing the $\phi = 0.65$ with $\phi = 0.8$ in Figs. 7.8 and 7.9. The pressure drop in both cases is smaller in the second leg of the loop, but clearly the fluids respond differently, with a more drastic drag reduction for the higher value of ϕ.

The results shown in these figures clearly suggest that lubrication layers evolve downstream of the entrance so that a development length is required. Moreover, we do not see effects of lubrication at low speeds; a minimum speed appears to be required to lift the bitumen drops off the wall. The pressure drop vs mass flux curves for the two legs of the open loop coincide for small values of mass flux but are increasingly different for larger values, with a markedly reduced drag in the second leg. It is possible that if the flow rate were increased, the point of onset of lubrication would move closer to the pipe entrance and could be detected in the first leg of the loop.

7.3.4 Discussion

All of the concentrated emulsions in the controlled-pressure and open-loop capillary viscometers developed lubrication layers. The data suggests the presence of a developing length which depends on the value of the disperse phase fraction and the velocity. In a fully developed lubricated flow, the lateral migrations of water and bitumen would cease and the pressure would become rogorously linear in the mass flux. Our data suggests this, but the pipes are not long enough. The drop size distribution in the pipe loops never changed, even when the mean shear rates were very large. In contrast, size distributions do change when the same emulsions are sheared between rotating cylinders. Evidently, shearing between cylinders is more severe because it extends into the interior of the emulsion. Other experiments we did suggest that size distribution instabilities occur at flow contractions in pumps and orifices. Flow contractions and pumping in the controlled viscometer capillary and the open loop are not severe. However, reports of instability of drop size distributions in pipelining under commercial conditions have been published (Zlatner, 1992). Field-scale facilities can induce severe gradients in certain types of pumps and recirculating valves (with low drop-to-clearance ratios), but never in straight pipes. In order to clarify this matter, we decoupled the pump-pipeline combination and focused attention on the behavior of an emulsion flowing through a variable-gap colloid mill. The results of this evaluation are presented next.

8. Colloid mill

8.1. *Introduction*

Here we study the flow of emulsions through narrow passages. This study allows us to assess the effect of the local variation of the bitumen fraction at its critical packing value. Such variations are induced in real systems by the low clearances which are present in the flow paths of certain pumps and process equipment such as control valves. We reproduce this situation in a controlled manner in a variable-gap colloid mill.

8.2 *Experimental procedure*

We used a colloid mill to test the notion that we can produce coalescence, with water in the resulting large drops, by forcing the emulsion through a narrow gap. The rotor, shown in Fig. 8.1, rotates at 1600 rpm during the tests. The basic unit at which the emulsion is introduced at the inlet and collected at the outlet is called a cycle. The collected emulsion can be recycled any number of times, say n times. At the end of a recycle sequence, the weight fraction of large drops with diameters greater than 150 μm is determined by sieving through a mesh of 150 μm. This gives the fraction of large drops after n cycles. The water content of these large drops is determined by distillation, only for the last cycle, due to reasons that will become apparent shortly.

The mean droplet size in the emulsion which remains after sieving was measured by a light diffraction device (Malvern, model Master Sizer) giving rise to the graph shown in Fig. 8.3 of mean droplet size vs. the number of cycles.

In practice, the measurements of the fraction of large drops which could not pass through the sieve and the fraction of water and drop size distribution in the emulsion that did go through were done at the end of each cycle. We did this by removing small samples from the copious amounts of emulsion which are expelled at the outlet of the mill. The small samples are carefully weighed before sieving. We took care to make sure that all the bitumen with sizes less than 150 μm had passed through the mesh and we repeated this procedure with many small samples after each cycle, taking averages with a standard deviation of less than 3%. All of these measurements were carried out for different clearances between the rotor and stator ranging from 0.51 to 4.92 mm.

8.3 *Results and discussion*

We milled unimodal emulsions with bitumen fractions from

$\phi = 0.65$ to 0.70, and bimodal emulsions with $\phi = 0.80$. All but one of the samples used in our tests were emulsified in an aqueous surfactant solution with a surfactant concentration of 2.5 g/L. In the exceptional case the surfactant concentration was 4.5 g/L.

Figure 8.2 shows the variation of the weight percentage of drops greater than 150 μm in a unimodal emulsion with $\phi = 0.7$ as a function of the number n of the cycles through the colloid mill for different values of the rotor-stator spacing. In the 2.5 g/L case, the weight fraction of large drops is nearly constant for the first few cycles and then increases suddenly. The number n of cycles required to produce a sudden increase is larger the larger the rotor-stator spacing; coalescence is achieved more readily with small gaps. This is consistent with the idea that coalescence can be described as a local inversion induced by concentrating an already concentrated emulsion in the small gaps of the colloid mills. Total inversion was obtained for the 0.80 bimodal emulsion in only one cycle.

Figure 8.2 also shows that the 4.5 g/L sample could withstand more cycles without undergoing structural changes. We believe that this increased stability is due to an increase in the strength of the protecting surfactant film which could allow drops to flatten, deform, and even to refine for more cycles before coalescence.

The rate of change of the relative mean droplet size seems to be the same for all the rotor-stator gap sizes, as we can see in Figure 8.3, but this change is delayed as the gap and the surfactant concentration are increased. We get more more coalescence and, in consequence, larger mean drop sizes for the smaller gaps. Two phenomena control phase inversion after a given number of cycles. Coalescence is induced by the flow through a contraction (which increases the mean drop size) and water is encapsulated in the process. At first, the size of coalesced drops is small because only a small number of drops coalesce locally and only a small amount of water is encapsulated. This encapsulation gets the emulsion closer to its critical packing every cycle. This process appears to be sensitive to the mean inter-particle distance prevailing in emulsion, like when our 0.7 emulsion is diluted to 0.65 without changing the drop size distribution. We repeated the experiment described in the two previous figures with such a diluted emulsion and the results are presented in Fig. 8.4. They show that after 20 cycles there is virtually no change in the drop size distribution with no drops greater than 150 μm. At such bitumen fraction, the gaps tested in the colloid mill do not concentrate the emulsion so as to induced coalescence. The 0.7 case presented also in this figure follows the same behavior as before.

Fig 8.5 shows the evolution of the drop size distribution of a 0.70 unimodal emulsion tested with a gap size of 0.76 mm. There we can see an increase not only in the mean diameter but also in the fraction of drops greater than 100 μm. We can see also how the distribution becomes broader after each cycle as a result of the induced

coalescence. We inquired into the mechanism by which water is encapsulated within the large bitumen drops as a result of coalescence. We measured the water content of the already sieved emulsion sample after a given cycle. We also measured the water content in the large bitumen drops that remained in the sieve at the last cycle, in order to have enough resolution in the distillation (in the initial cycles the encapsulated water cannot be resolved by this method). The former results are presented in Fig.8.6. There, we can see that the filtered emulsion increases its water content as a function of gap size and number of cycles. Even though this trend is logical because bitumen is being retained by the sieve's mesh, it is no proof that the emulsion has inverted locally. However, the distillation of the large bitumen drops revealed a water content of 0.22 for the last cycle in virtually all cases. This means that when coalescence is taking place only a fraction of the continuous phase (water) is encapsulated into the new greater bitumen drops.

The foregoing evidence appear to support the idea that the local inversion of an emulsion occurs when many neighboring bitumen drops coalesce and encapsulate the water in the Plateau like borders. If only two drops were to coalesce, no water would be trapped. When three closely packed drops coalesce some water in between could be encapsulated; in two dimensions the water between would be completely trapped by three touching drops. Oviously, the chance of trapping water in an array of coalescing drops increases with the size of the array, and all the water in the intertices would be trapped if the whole emulsion were suddenly to invert. Generally, in a local inversion some water will be driven off so that the water content of coalesced drops can be expected to be less than the water fraction of the emulsion, with equality only in the ideal cases of total inversion. In the colloid mill, the water content in the coalesced drops is at first rather small. The recycled emulsion has larger drops and the water content in the large drops is an increasing function of the number of cycles but is never greater than 22%. In the end, after the local inversion is complete, there is no more than 22% of water and there is always some free water after coalescence.

9. Concluding remarks

We have considered three topics related to the flow characteristics of concentrated emulsions with a highly viscous dispersed phase and very small (5 - 50 μm) drops in water. The first topic is about drop refinement of the most highly concentrated emulsion when flowing between rotating cylinders. This was observed in unimodal emulsions

with bitumen fractions equal or greater than 0.7 and in the 0.8 bimodal emulsion. As the angular velocity is increased, we observed a rise followed by a sharp fall in the torque. The drop sizes are reduced near the point of maximum torque and the reduction is specially severe in the bimodal distribution where the large drop fraction is completely anihilated. We think that shearing the concentrated and viscous dispersion effectively breaks the larger drops by direct contact with other drops as in the comminution of solids rocks; in both cases the largest particles are broken and the mean effective diameter is reduced. We could not change identical size distributions in concentrated emulsions of much less viscous oil under the same dynamic conditions; these drops deform rather than fracture.

We could not change the drop distributions in pressurized flow in straight capillary tubes even in the more concentrated and highly viscous bitumen-in-water emulsions. Such dispersions could shear strongly only in the boundary layer, but boundary layers are effectively eliminated by the development of lubrication layers of clear water at the wall. The lubricated flows are esentially shear free.

The second topic considered here deals with the development of lubrication layers in the flow of concentrated emulsions. Lubricated flows are well known; they are enhanced by hydrophillic pipe walls and suppressed by oleophillic counterparts. The lubrication layers require a development region with lateral migration of water to the wall and bitumen drops away from the wall. A minimum speed appears to be required suggesting that inertia is important.

The third topic is the local inversion of a concentrated emulsion testing the maximum packing fraction locally. Encapsulation of water in the interstices between closely packed drops is an inevitably consequence of the simultaneous coalescence of many drops. In fact, water was always found in coalesced bitumen drops. The water fraction in a totally inverted emulsion does not change, but the fraction of water encapsulated in a local inversion can be less, with some water driven off into reservoirs of free water. Local inversions are industrially important in pumps and contractions where squeezing motions further concentrate an already concentrated emulsion testing the maximum packing fraction locally.

11. References

Aral, B.K., and Kaylon, D.M.: *Effects of temperature and surface rougness on time-dependent development of wall slip in steady torsional flow of concentrated suspensions.* J. Rheol. 38(4), Jul-Aug. 1994. pp 957-972.

Briant, J; Fruman, D.H.; Quemada, D. and Makria , A.: *Transport de Petroles Bruts Lourds sous forme d´émulsions huile dans eau.* Revue de L´Institut du Petrole. Vol.37, No.6, Nov.1982. pp 809-821.

Chang, Ch., and Powell, R.L.*: Effect of particle size distributions on the rheology of concentrated bimodal suspensions*, J. Rheol. 38(1), Jan-Feb 1994. pp 85-98.

D´Haene, P., and Mewis, J.: *Rheological Characterization of bimodal colloidal suspensions*. Rheol. Acta 33:165-174 (1994).

Farris, K.J., *Prediction of the Viscosity of Multimodal Suspensions from Unimodal Viscosity Data.* Trans. Soc. Rheol. **12**, 281-301(1968).

Gaddar, C.: *Fracture Efficiency within Particle Beds.* M.S. Thesis, MIT (1991).

Giordano, J.P; Plegue, T.H.; Frank, S.G.; Fruman, D.H.; and Zakin, J.L.: *A Study of Concentrated Viscous Crude Oil-in-Water Emulsions*. Progress and Trend in Rheology II. Rheology Acta, 26,302 (1988).

Joseph, D.D. and Renardy, Y.Y.,: *Fundamentals of Two Fluids Dynamics.. Part II: Lubrication Transport, Drops and Miscible Fluds* (Springer, Berlin, 1992).

Lamb, M.S. and Simpson, W.C.: *Pipeline Transportation of WaxLaden Crude Oil as Water Suspensions*. Proc. Sixth World Petroleum Congress, Section VII, p.23 (1973).

Layrisse, I., Rivas, H., Quintero, L. Rivero M. and Martinez, M.: *Imulsion Technology for Heavy , The Answer for production, Handling and Transportation of Extra Heavy Crudes and Bitumens*. 4th UNITAR/UNDP International Conf. of Heavy Crude and Tar Sands, Edmonton, Alberta, Canada, Aug 7-12, 1988.

Mannheimer, R.J.: *Anomalous Rheological Characteristics of a High-Internal-Phase-Ratio Emulsion*. Journal of Colloid and Interface Science, Vol. 40. No.3, pp 370-382. (1972).

Mao, M.L. and Marsden, S.S.: *Stability of Concentrated Crude Oil-in-Water Emulsions as a function of shear rate, temperature and oil concentration*, J. Can. Pet. Tech, Apr-June 54 (1977).

Marsden, S.S. and Raghvan, R.: A *System for Producing and Transporting Crude Oil as an Oil/Water Emulsion*. Journal of the Institute of Petroleum, Vol.59, No.570, 1973. pp 273-278.

Mc.Cafferty,J.F., and Mc.Clafin, G.G.: *The Field Application of a Surfactant for the Production of Heavy Viscous Crude Oil*. SPE Paper 24850 (1992) 857-868.

Mourits, F.M.: *Heavy Oil Production Improvemeent Through Down Hole Emulsification*. 2nd China-Canada Heavy Oil Technology Symposium, Zouszou, China, october 29- November 1, 1990.

Pileharvi, A., Saadevandi, B., Halvaci, M., and Clark, P:E*: Oil/Water emulsions for pipeline transport of viscous crude oils*. SPE, Paper No. 18218 (1988).

Plegue, T.H.; Frank, S.G. Fruman, D.H. and Zakin J.L.: *Concentrated Viscous Crude Oil-in-Water Emulsions for Pipeline Transport*, Chem. Eng. Comm. 1989, Vol 82, pp. 11-122.

Poletto, M. and Joseph, D.D.: *Effective density and viscosity of a suspension*. J. of Rheology, 39 (2), 1994. pp 323-343.

Princen, H.M.: *Highly Concentrated Emulsions, I. Cylindrical Systems*. J. of Colloid and Interface Science, Vol .71 No.1 August 1979. pp 55-66.

Princen, H.M., Aronson, M.P., and Moser, J.C.: *Highly Concentrated Emulsions, I. Real Systems. The Effect of Film Tyhickness and Contact Angle on the Volume Fraction in Creamed Emulsions*. J. of Colloid and Interface Science, Vol .71 No.1 May 1980. pp 246-270.

Princen, H.M.: *Rheology of Foams and Highly Concentrated Emulsions. I. Elastic properties and Yield Stress af a Cylindrical Model System*. J. of Colloid and Interface Science, Vol .91 No.1 January 1983 . pp 160-175.

Princen, H.M., and Kiss, A.D.: *Rheology of Foams and Highly Concentrated Emulsions. IV. An Experimental Study of the Shear Viscosity and Yield Stress of Concentrated Emulsions*. J. of Colloid and Interface Science, Vol. 128, No.1, March 1, 1989, pp 177-187.

Probstein, R.F., Sengun, M. Z., and Tseng, T-C.*: Bimodal model of concentrated suspension viscosity for distributed particle size*. J. Rheol. 38(4), Jul-Aug. 1994. pp 811-829.

Rivas, H., Núñez, G.A., and Dalas, C.: *Emulsiones de Viscosidad Controlada*. Visión Tecnológica. Vol. 1, No.1, Venezuela 1993.

Rose, S.C., and Marsden, S.S.: *The Flow of North of North Slope Crude Oil and Its Emulsions at Low Temperatures*. 45th Anual Fall Meeting of the SPE of AIME. Houston, Texas, Oct. 1970, SPE 2996.

Sanchez,L.E. and Zakin, J.L.: *Transport of Viscous Crudes as Concentrated Oil-in-Water Emulsions*. Paper 170c prfesented at the AlChE meeting in Los Angeles (Nov. 17-21 1991).

Sengun, M.Z. and Probstein, R.F. (1989a) *Bimodal model of slurry viscosity with application to coal-slurries. Part1: Theory and Experiment*. Rheol. Acta 28:382-393.

Sengun, M.Z. and Probstein, R.F. (1989b) *Bimodal model of slurry viscosity with application to coal-slurries. Part2: High Shear limit behaviour*. Rheol. Acta 28:394-401.

Simon, R. and Poynter, W.G. 1968.*: Down Hole Emulsification for Improving Viscous Crude Production*. J. Petr. Technol, Vol. 20, 1349-1353.

Thomas, D.G.: Transport Characteristics of Suspensions: *A Note on the Viscosity of Newtonian suspensions of uniform spherical particles*. J. Coll. Sci., **20,** 267 (1965).

Zakin, J.L.; Pinaire, R. and Borgmeger, M.E.: *Transport of Oils as Oil-in-Water Emulsions*. Transactions of the ASME. Journal of Fluid Engineering. Vol 101. March 1979. pp 100-104.

Zhang, J., and Dafan Y.*: Rheological Measurement of Oil-in-Water Emulsions: Concentric Cylinder Viscometer*. 4th UNITAR/UNDP International Conf. of Heavy Crude and Tar Sands, Edmonton, Alberta, Canada, Aug 7-12, 1988.

Zhang, J.; Chen, D.; Yan, D.; Yang, X. and Shen, Ch.: *Pipelining of Heavy Crude Oil as Oil-in-Water Emulsions*. SPE 21733, 1991. pp 911-918.
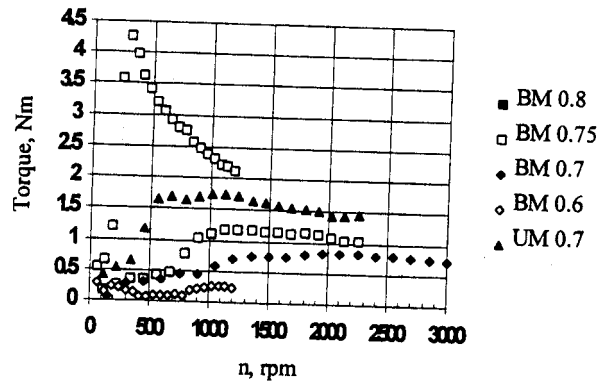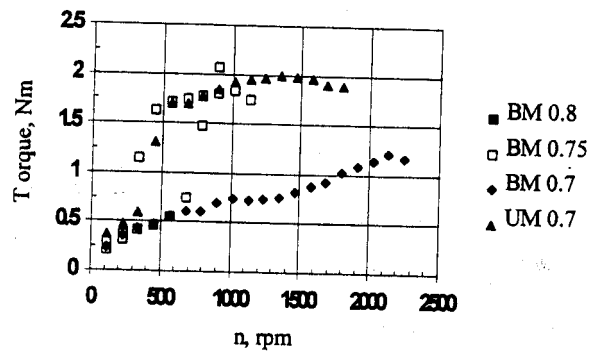
FIG. 1. Torque as a function of the rotating frequency (n), for a 2.5 mm annular gap. (a) Plexiglas cylinders. (b) Stainless steel cylinders.
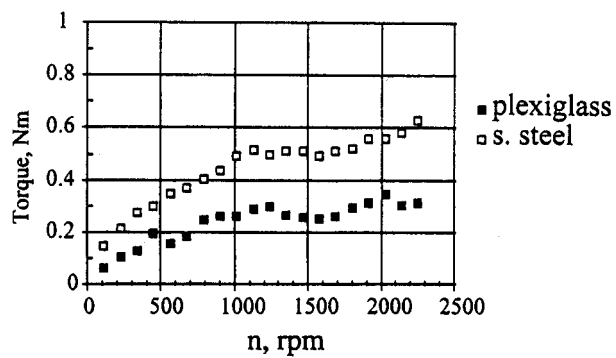
FIG. 2. Torque as a function of the rotating frequency (n) for a bimodal emulsion of 0.7 internal phase fraction and 7.5 mm annular gap.

plex. 2.5mm
plex. 2.5mm
s. s. 7.5mm
plex. 7.5mm
s. s. 2.5mm
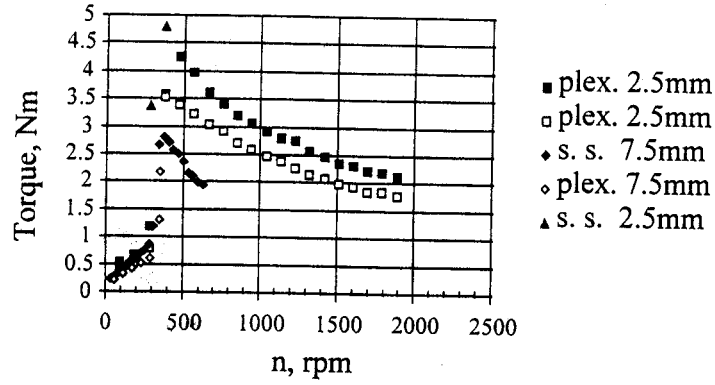
FIG. 3. Torque as a function of rotating frequency (n) for a bimodal emulsion of 0.8 internal phase fraction and variable annular gap.

spin up
spin down

before
after

(a)

spin up
spin down

before
after

(b)
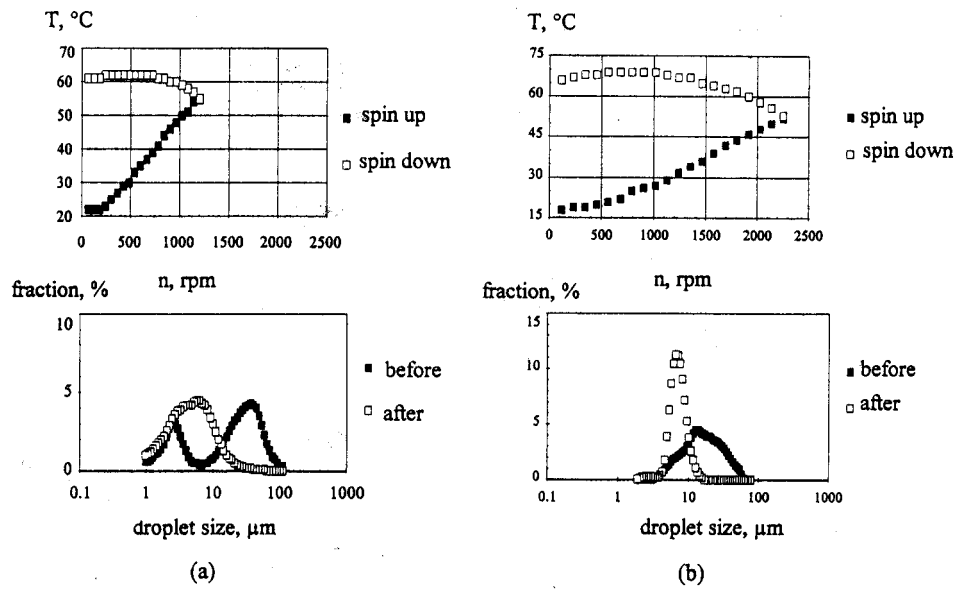
FIG. 4. Temperature and drop size distribution history. (a) bimodal 0.8; (b) unimodal 0.7.
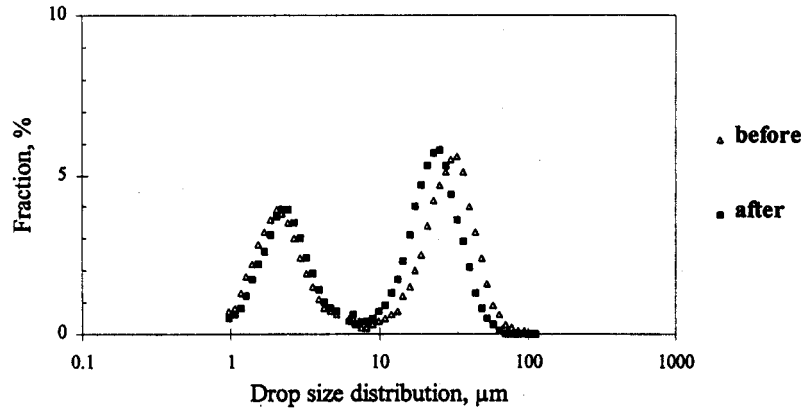
FIG. 5. Drop size distribution before and after spin up for a bimodal 0.8 internal phase emulsion of lubricating oil.
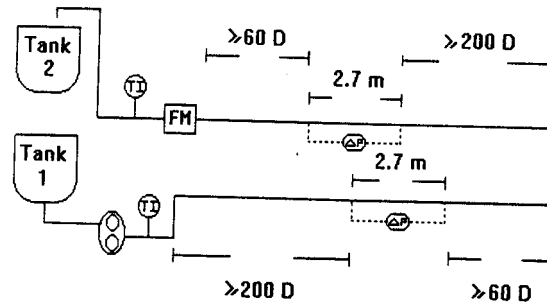
FIG. 6. Open loop pipe viscometer. PI: pressure indicator; FM: flow meter; TI: temperature indicator; D: pipe diameter.

(a)
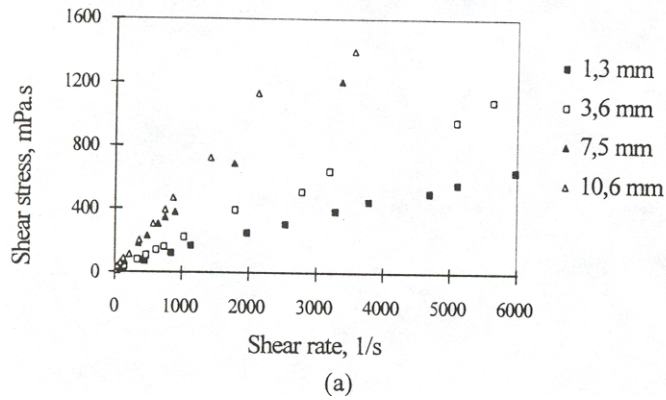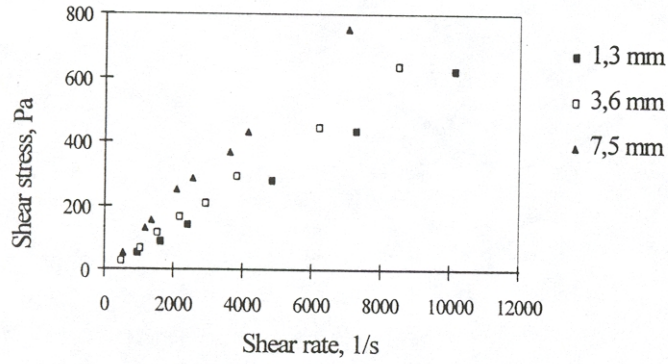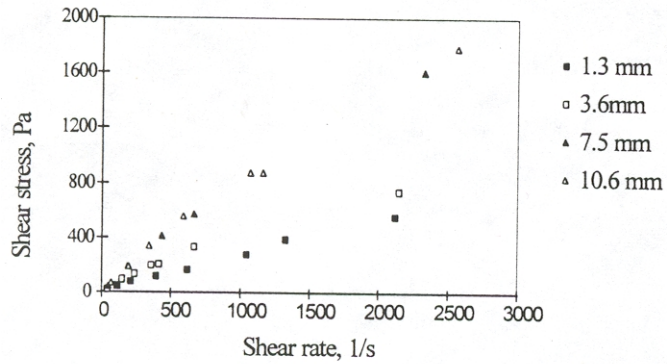
FIG. 7. Shear stress as a function of shear rate of various emulsions for several tube diameters of equal lengths. (a) 0.7 unimodal; (b) 0.7 bimodal; (c) 0.8 bimodal.

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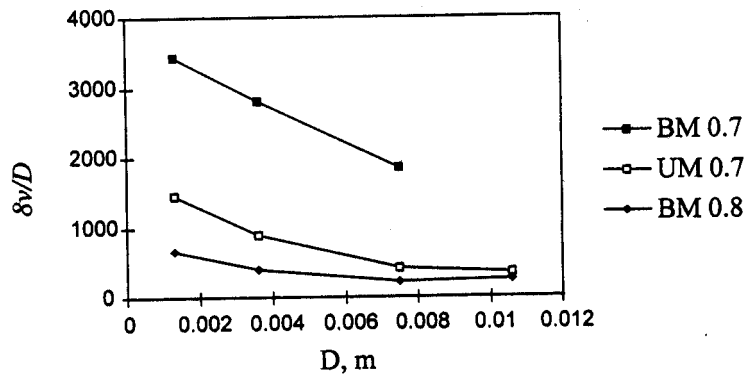
FIG. 8. Mean shear rate as a function of pipe diameter for a fixed shear stress of 200 Pa.
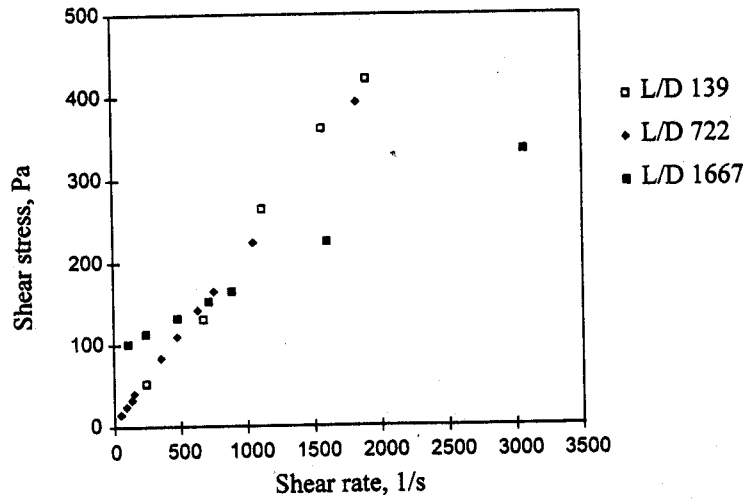
FIG. 9. Shear stress as a function of shear rate of a 0.7 unimodal emulsion for a 3.6 mm diameter pipe of different lengths.
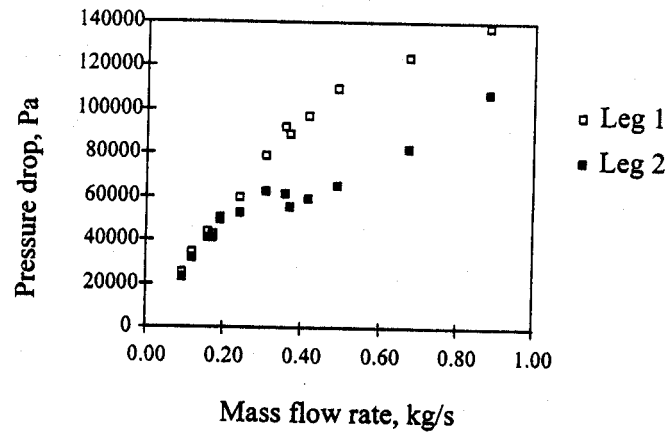
FIG. 10. Pressure drop as a function of mass flow rate for a 0.8 bimodal emulsion in a 21.7-mm diam pipe.
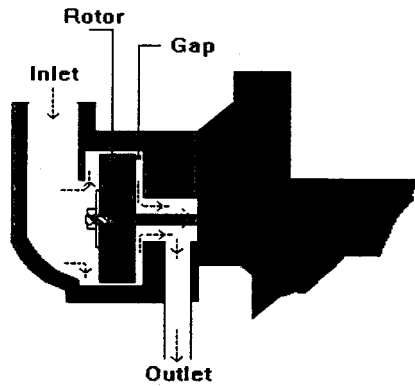
FIG. 11. Schematics of flow inside the colloid mill.
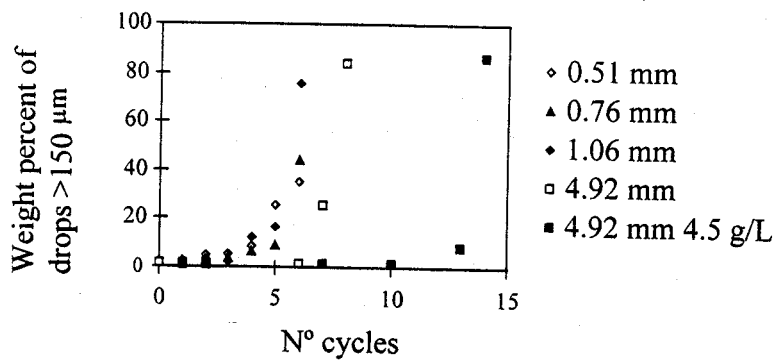
FIG. 12. Weight percent of droplets greater than 150 μm as a function of the number of cycles for a 0.7 internal phase unimodal emulsion tested in a colloid mill with various gap sizes. Most cases prepared with 2.5 g/ℓ of surfactant.
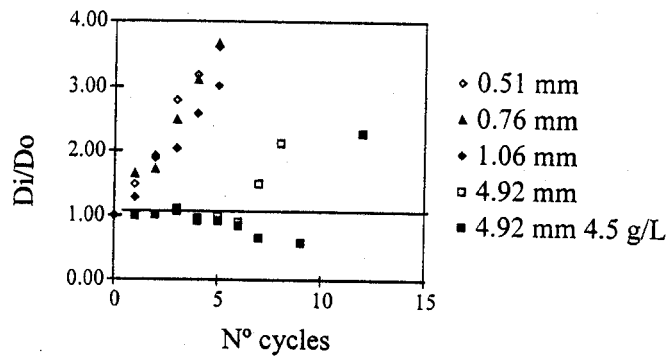
FIG. 13. Relative change of mean droplet size as a function of the number of cycles for a 0.7 internal phase unimodal emulsion tested in a colloid mill with various gap sizes. Most cases prepared with 2.5 g/ℓ of surfactant.
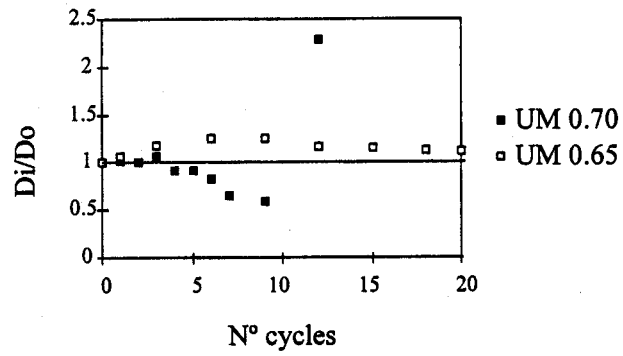
FIG. 14. Relative change of mean droplet size as a function of the number of cycles for a 0.7 and a 0.65 internal phase unimodal emulsion tested in a colloid mill with a gap size of 4.92 mm. Both cases prepared with 2.5 g/ℓ of surfactant.
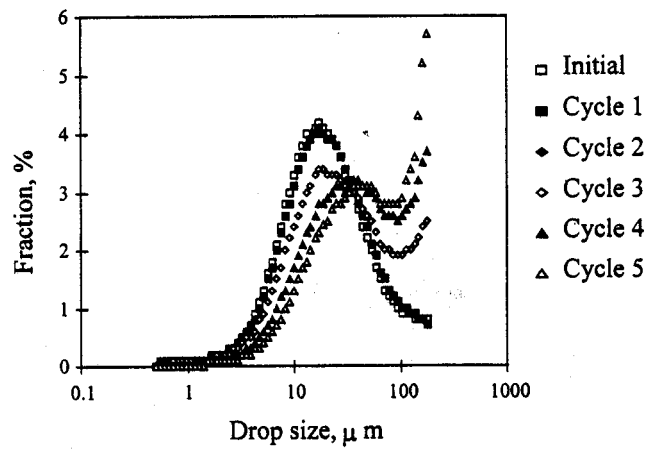

FIG. 15. Drop size distribution as a function of the number of cycles for a 0.7 unimodal emulsion tested in a colloid mill with gap size of 0.76 mm.
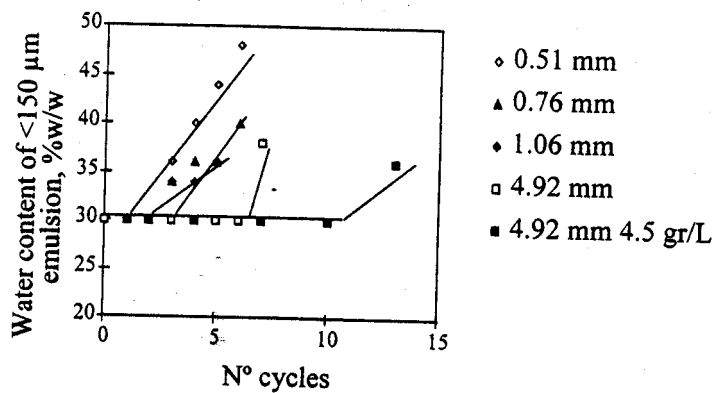
FIG. 16. Relative change of water content of the sieved emulsion as a function of the number of cycles for a 0.7 internal phase unimodal emulsion tested in a colloid mill with gap size of 4.92 mm. Most cases prepared with 2.5 g/ℓ of surfactant.