

Experimental investigation of phase inversion in an oil–water flow through a horizontal pipe loop

K. Piela^{a,*}, R. Delfos^a, G. Ooms^a, J. Westerweel^a,
R.V.A. Oliemans^b, R.F. Mudde^b

^a *J.M. Burgerscentrum for Fluid Mechanics, Delft University of Technology, Laboratory for Aero- and Hydrodynamics, Leeghwaterstraat 21, 2628 CA Delft, The Netherlands*

^b *Delft University of Technology, Kramers Laboratorium, Prins Bernhardlaan 6, 2628 BW Delft, The Netherlands*

Received 21 January 2006; received in revised form 5 May 2006

Abstract

An experimental study was made of phase inversion in an oil–water flow through a horizontal pipe loop. The experiments started with the flow of a single liquid through the pipe loop; thereafter the second liquid was gradually added (using different injectors and different injection flow rates) until inversion took place. It was found that in this way the point of inversion could be postponed to high values of the dispersed phase volume fraction (>0.8). Samples were taken from the flowing mixture and inspected with the aid of a microscope. Multiple drops consisting of oil droplets in water drops were observed, but multiple drops consisting of water droplets in oil drops were never found. The significance of these observations for the phase inversion mechanism is discussed.

© 2006 Elsevier Ltd. All rights reserved.

1. Introduction

The flow of two immiscible liquids often occurs as a dispersed flow, where one liquid is present in the other liquid in the form of drops. Dispersions are widely applied in the petrochemical-, food-, chemical- and pharmaceutical industries. For our investigation we used oil and water without a surfactant. Both water-in-oil and oil-in-water dispersions were possible. Conversion between these two types of dispersion is called phase inversion. Which type is present, depends on several parameters such as the volume fraction of the two phases, the viscosities of the liquids, the inter-facial tension and the turbulence in the flowing mixture.

An often used model for phase inversion takes into account two opposite processes: break-up and coalescence of drops (Arashmid and Jeffreys, 1980; Nienow, 2004). Usually the break-up of drops is described by comparing the drop deformation due to turbulent pressure fluctuations with the deformation-restoring effect due to the inter-facial tension. The coalescence of drops is calculated by modelling the collision between drops.

* Corresponding author. Tel.: +31 15 278 2861; fax: +31 15 278 2947.
E-mail address: k.piela@wbmt.tudelft.nl (K. Piela).

Phase inversion is then assumed to take place, when a certain criterion is satisfied. For instance, it is assumed that phase inversion occurs when the rate of breakup is not equal to the rate of coalescence, or that the viscosity of the mixture grows larger than a certain limiting value (Vaessen et al., 1996; Ioannou et al., 2004). In another model phase inversion is assumed to take place, when the inter-facial energy of the water-in-oil dispersion and the inter-facial energy of the oil-in-water dispersion are equal (Yeo et al., 2002; Brauner and Ullmann, 2002). For the calculation of the inter-facial energy it is necessary to know the drop size distribution and, therefore, again to model the break-up and coalescence processes.

Another way of explaining phase inversion has been presented by Groeneweg et al. (1998) and Bouchama et al. (2003). They observed that for certain conditions the drop size increases considerably, before phase inversion takes place. This is explained by assuming the inclusion of small droplets of the continuous phase into the dispersed phase drops. They describe this as the formation of a multiple dispersion; for instance oil droplets in water drops in a continuous phase of oil are formed. As the drops grow in size, their collisions and coalescence become more probable which leads to phase inversion. In the publication by Bouchama et al. (2003) an interesting experiment is reported, which seems to confirm (in an indirect manner) the formation of a multiple dispersion as a mechanism for phase inversion. They carried out experiments in a stirred vessel where inversion was detected by a jump in dispersion conductivity. Their system contained paraffin oil, water and a surfactant. The dispersed phase fraction at phase inversion was compared for two emulsification routes for phase inversion, namely the direct route and the wash-out route. In the direct route emulsions were prepared at a certain composition by the direct mixing of the proper oil and water volumes. In the wash-out route the vessel was filled with oil, the impeller switched on and water was added in small volumes to the vessel at a constant flow rate until the same composition was reached as for the direct route. The occurrence of phase inversion was found to be strongly different for both cases. In particular for the wash-out route the way of dilution appeared to be an important parameter. According to the authors this can only be explained on the basis of the role of multiple-dispersion formation during phase inversion. In the wash-out experiments the generated drops were very small, therefore their Laplace pressure was high and multiple drop formation due to entrainment of droplets from the continuous phase was not possible. During the direct experiments the drops formed during the mixing process were much larger and entrainment of small droplets was possible, leading to a multiple dispersion.

The aim of our research, presented in this publication, is to study phase inversion in an oil–water flow through a horizontal pipe. For that purpose we carried out wash-out type experiments in a pipe loop by starting with the flow of a single liquid through the loop and then gradually adding a second liquid using different types of injectors and applying different injection flow rates. We performed experiments starting with water and then adding oil (water-to-oil experiments) and the other way around (oil-to-water experiments). Additional experiments were done in which the injection was stopped before the point of inversion was reached, after which the mixture continued to flow through the pipe loop at the same mixture velocity until inversion had occurred. Much attention was given to the possibility of postponing phase inversion to higher values of the dispersed phase volume fraction (like in the experiments of Bouchama et al., 2003) and to the possibility of multiple drop formation. Particular attention was paid to the influence of these phenomena on the inversion mechanism. In publications by Ioannou et al. (2004, 2005) also attention is given to phase inversion in dispersed liquid–liquid pipe flows. Contrary to our experiments they performed direct-route type of experiments, as in their case the two liquids are mixed in the pipe at certain composition from the start of the experiment.

2. Experimental set-up

The experiments were carried out in the Laboratory for Aero- and Hydrodynamics of the Delft University of Technology. The set-up is shown in Fig. 1. It is an (acrylic) pipe loop with an inner pipe diameter of 16 mm, consisting of two straight parts of 6 m connected via two bends. The two immiscible liquids used in our experiments were tap water and Shell Macron EDM 110 oil. The oil properties are given in Table 1.

During an experiment first one of the liquids (water or oil) was taken from tank 2 in Fig. 1 by means of pump 3 and recirculated through the pipe loop by means of pump 1. Pump 1 is a positive displacement pump, chosen to minimize the pumping effect on the dispersion morphology. Pump 3 pressurized the system, so that no entrapment of air into the pipe loop could occur. After recirculating the liquid for a few minutes to ensure

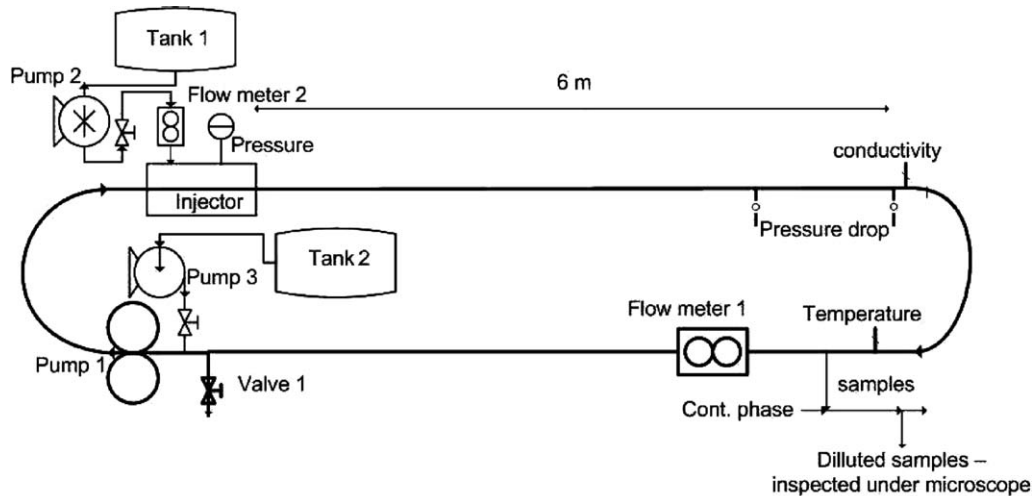


Fig. 1. Sketch of the experimental set-up. For an explanation see the text.

Table 1
Properties of Shell Macron EDM 110 oil

Density	794 kg/m ³
Kinematic viscosity	
at 20°	3.9 mm ² /s
at 40°	2.4 mm ² /s
Oil–water inter-facial tension	0.045 N/m

that the pipe walls are wetted by the liquid, injection started by pumping (using pump 2) the other liquid from tank 1 via an injector into the pipe loop. Flow meter 1 (a Krohne Optimass 7000 mass flow meter with a measuring error lower than 0.26% of the measured value) measured the density and the flow rate of the mixture in the pipe loop. The flow meter is validated for oil–water mixtures. All experiments were performed at such high Reynolds numbers and Froude numbers (see Table 2), that a fully dispersed flow was present according to Brauner (2001). The dispersed phase fraction could therefore be calculated from the measured density. The flow rate and density measurements made with flow meter 1 were used to control pump 1 via a feedback system. The mixture velocity in the pipe loop was kept constant during the experiment. (This was achieved in the following way: Flow meter 2 measured the injection flow rate through pump 3.

Table 2
Dimensionless numbers for water and oil at different superficial velocities

Water	Re	Fr	We	
1 m/s	15,936	6.4	354	
2 m/s	31,872	25.5	1418	
3 m/s	47,808	57.3	3190	
1.34 m/s	21,354	11.4	637	Dispersed phase boundary – Brauner (2001)
Shell Macron EDM 110	Re	Fr	We	
2 m/s	6515	25.5	1129	
3.5 m/s	11,401	78	3458	
1.35 m/s	4398	11.6	515	Dispersed phase boundary – Brauner (2001)

For the calculation of the Froude number the density difference between water and oil is used and for the calculation of the Weber number the inter-facial tension between the two liquids.

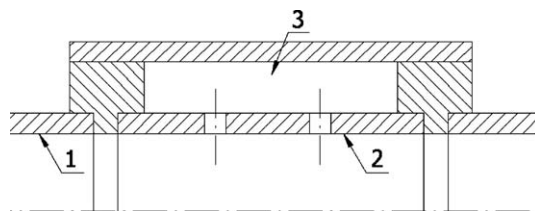


Fig. 2. Injector: 1 – wall of pipe loop, 2 – removable injector and 3 – injector chamber.

Pump 3 is a gear pump, which ensured that the injection flow rate was constant during the experiment. During the injection valve 1 was opened and the same volume of dispersion liquid was removed from the pipe loop as the volume that was injected.) We used as injector a cylindrical container (placed around the pipe loop) with a number of holes in it. The oil was pumped through the holes into the pipe loop. Three different types of injector were used, viz. a container with 2 holes of 2 mm diameter, a container with 8 holes of 2 mm diameter, and one with 100 holes of 3 mm diameter (see Fig. 2). The pressure was measured at 5 m downstream of the injector (see Fig. 1) over a distance of 1 m. Differential pressure transducers (with a measuring error lower than 1% of the measured value) were used, that sampled signals with a frequency of 50 Hz. Averaging over 50 samples was applied. Conductivity measurements (at a frequency of 50 Hz) were done with a cell consisting of four electrodes (4 mm diameter) mounted in the pipe wall: two in the vertical direction (at the top and bottom of the pipe) and two in horizontal direction (in the side walls). When the flowing mixture was fully dispersed the measured conductivity in vertical and horizontal direction were equal. Conductivity measurements are not presented in this paper. They were used as a confirmation of phase inversion found from pressure measurements. Also temperature measurements were carried out during the experiments. To inspect the dispersion morphology samples were taken from the mixture and photographed. The samples were taken via a small hole in the pipe loop, injected in a pipe with a flowing liquid (the same liquid as the continuous phase in the pipe-loop) and then inspected under a microscope (of a magnification of 50) where also pictures were taken. A sketch of the sampling technique is given in Fig. 1. Perhaps this procedure has some influence on the drop size distribution. However, the most important reason for taking samples was to study the composition of the drops, in particular to see whether or not multiple drops were formed. To study the influence of the length of the pipe loop (and of pump 1) also experiments were performed for which, each of the two straight parts of the loop was reduced to 4.5 m. Of course during these experiments the mixture flows more often through the pump. However, we observed no differences with the experiments carried out in the longer pipe loop. Each result presented in this paper is from a single experiment, but all experiments were conducted at least two times and the reproducibility of the experiments was good.

3. Experimental results

3.1. Water-to-oil experiments

A water continuous to oil continuous (water-to-oil) experiment starts with pumping water through the pipe loop. Thereafter, injection of oil begins and an oil-in-water dispersion develops. With continuing oil injection the volume concentration of drops increases and at a certain moment in time inversion to a water-in-oil dispersion takes place. Pictures have been taken at different stages of the inversion process (see Fig. 3). The left picture shows the dispersion before inversion. Only “clean” oil drops are present; so multiple drops (consisting of small water droplets in larger oil drops) are not formed before inversion. The middle picture shows the dispersion just after inversion. As can be seen during the inversion process multiple drops (consisting of small oil droplets in larger water drops) are generated. The right picture shows the dispersion a few minutes after inversion. Multiple drops are still present, but they contain less oil droplets compared to the situation just after inversion. It appears that oil droplets have escaped from the water drops.

We studied the influence of different injector types on the phase inversion process. As mentioned, we used three different types of injector, viz. a container with 2 holes of 2 mm diameter, a container with 8 holes of

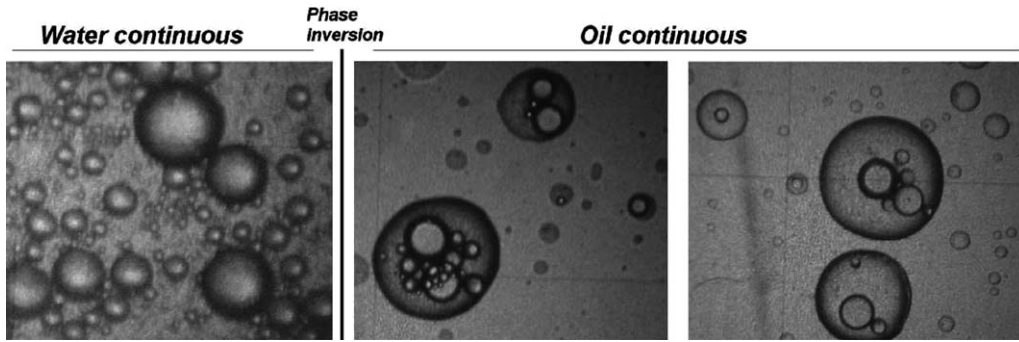


Fig. 3. Dispersion morphology during water-to-oil experiments. From left to right: before inversion, just after inversion and a few minutes after inversion.

2 mm diameter, and one with 100 holes of 3 mm diameter. These three types of injector were chosen to investigate the influence of injection velocity (and therefore different drop sizes) on the phase inversion mechanism. We kept the oil injection flow rate the same during the experiments. It is evident, that for the same oil flow rate the jet exit velocity of the oil decreases with increasing number of holes. During the experiments we kept the mixture velocity in the pipe loop at 2 m/s (so a highly turbulent flow – see Table 2) by using the information from flow meter 1. The injection flow rate was 12.5% of the mixture flow rate in the pipe loop. The exit velocities of the oil jets issuing from the injector holes (for the three types of injectors) are given in Table 3. We made an estimate of the size of the oil drops as generated by the jet break-up and as generated by the turbulence in the flowing mixture at the start of the experiments. For all conditions the drop size was determined by the turbulence. The results of the experiments are given in Fig. 4, which shows the scaled pressure drop (ratio

Table 3
Jet exit velocities at a mixture velocity of 2 m/s and an injection flow rate of 12.5%

Injector type	Jet exit velocity
100 × 3 mm	0.07 m/s
8 × 2 mm	2 m/s
2 × 2 mm	8 m/s

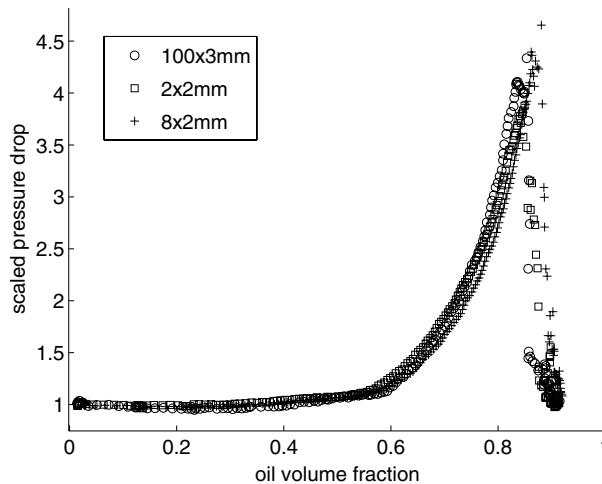


Fig. 4. Scaled pressure drop as function of oil volume fraction for water-to-oil experiments at a mixture velocity of 2 m/s and an injection rate of 12.5% for three different injector types. Inversion occurs at an oil volume fraction between 0.8 and 0.85.

of the actual pressure drop and the pressure drop at the start of the experiment with the single-phase flow of only water) over a part of the pipe loop as function of the oil volume fraction for the three different injectors. Inversion occurs at an oil volume fraction between 0.8 and 0.85.

The figure shows that the inversion process is independent of the injector type. Of course the drop size distribution depends on the break-up of the jets issuing from the injector holes (which depends on the jet exit velocity). However, as can be seen on Fig. 4, this effect has hardly any influence on the inversion process for the injectors that we used.

Next we investigated the influence of the injection rate. For that purpose we carried out experiments with injection rates of 5%, 15% and 26.5% of the mixture flow rate in the pipe loop using one injector type (100×3 mm). The mixture velocities for these experiments was 1 m/s and the flow was still fully dispersed. The results are given in Fig. 5. As can be seen in this figure (giving the scaled pressure drop as function of the oil volume fraction for the three different injection rates) the development towards inversion appears to be independent of the injection rate. However, the oil volume fraction at the point of inversion decreases with increasing injection rate.

Inversion does not take place at all locations in the pipe loop at the same time. It starts at a certain location with the formation of an inverted pocket which grows as it flows downstream. This explains, that during inversion the pressure drop over the pipe loop fluctuates considerably. This can be seen in Fig. 6, in which the scaled pressure drop (not an averaged signal) is shown as function of time (for a mixture velocity of 2 m/s and the injection rate of 12.5%). The inversion process starts at about $t = 270$ s, when the pressure drop strongly decreases. Thereafter the pressure drop increases due to the arrival of a non-inverted pocket of fluid and when this has passed the pressure taps, the pressure drop again decreases. After one circulation through the pipe loop the non-inverted pocket still exists, causing another peak in the pressure drop. When the inverted pocket passes the injector, it is stimulated to grow due to newly injected oil. Usually after two (sometimes more) passes through the pipe loop the complete mixture has inverted. So inversion is a kind of statistical process. At higher injection rates the probability of fast growing inverted pockets increases and so (on average) the oil volume fraction at the point of inversion decreases.

We also investigated the influence of the mixture velocity on the inversion process. For that purpose we carried out water-to-oil experiments at mixture velocities of 1 m/s, 2 m/s and 3 m/s (with Reynolds numbers ranging from 15,000 to 50,000 – see Table 2) for various injection rates. As can be seen in Fig. 7 the influence of the mixture velocities on the inversion process is negligible in the range of mixture velocities that we studied. The influence of the injection flow rate on the oil volume fraction at the point of inversion for mixture velocities of 2 m/s and 3 m/s is similar to the case of 1 m/s.

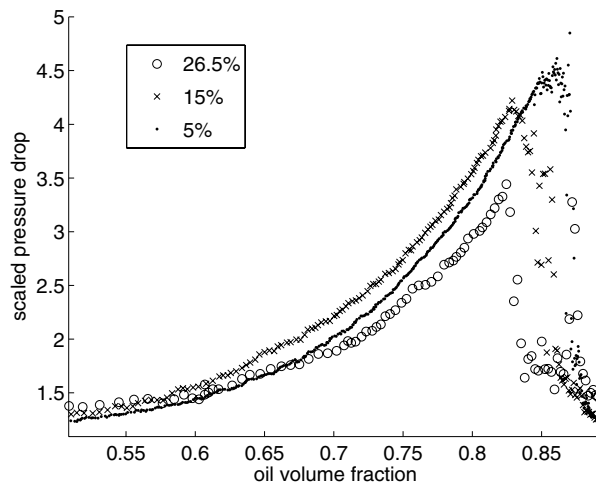


Fig. 5. Scaled pressure drop as function of oil volume fraction for water-to-oil experiments at a mixture velocity of 1 m/s and for three different injection rates.

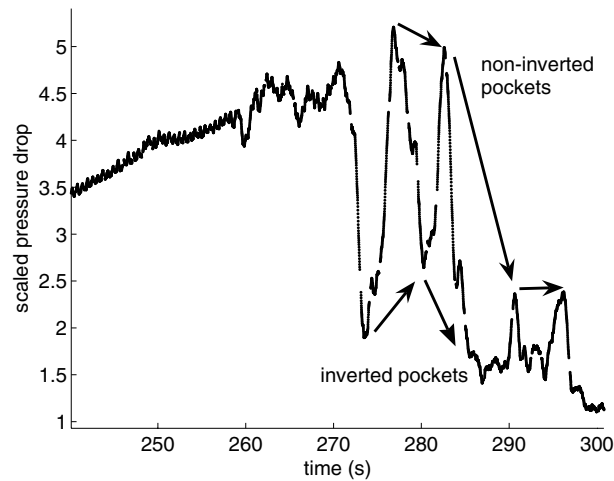


Fig. 6. Scaled pressure drop as function of time for a water-to-oil experiment at a mixture velocity of 2 m/s and injection rate of 12.5%.

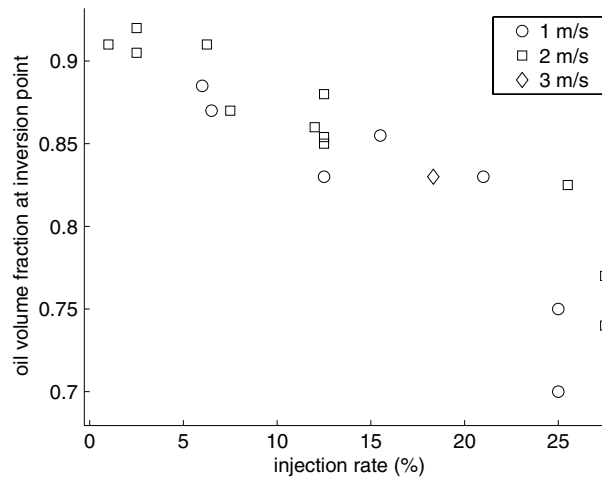


Fig. 7. Oil volume fraction at point of inversion as function of injection rate and several values of the mixture velocity.

3.2. Oil-to-water experiments

An oil continuous to water continuous (oil-to-water) experiment starts with pumping oil through the pipe loop. Then injection of water begins and a water-in-oil dispersion develops. With continuing water injection the volume concentration of drops increases and at a certain moment in time inversion to an oil-in-water dispersion takes place. Also in this case pictures have been taken at different stages of the inversion process (see Fig. 8). These pictures have been taken during an experiment as indicated in Fig. 9. In this experiment the mixture flow rate was 3.5 m/s and the injection rate was 5%. The injection of water was stopped before inversion occurred at $t = 220$ s, when the water volume fraction reached 0.64. The mixture continued to flow through the pipe loop without further water injection, although the pressure drop continued to increase (see Fig. 9) and finally at $t = 450$ s inversion took place. The left-top picture of Fig. 8 shows a sample of the mixture taken at $t = 160$ s, the right-top picture at $t = 280$ s, the left-bottom picture at $t = 400$ s and the right-bottom picture at $t = 465$ s. It is evident, that in this case multiple drops (oil droplets in water drops) were formed before inversion. The volume fraction of oil inside the water drops increased with time. After inversion only “clean” oil drops were present; so no multiple drops were formed after the inversion process.

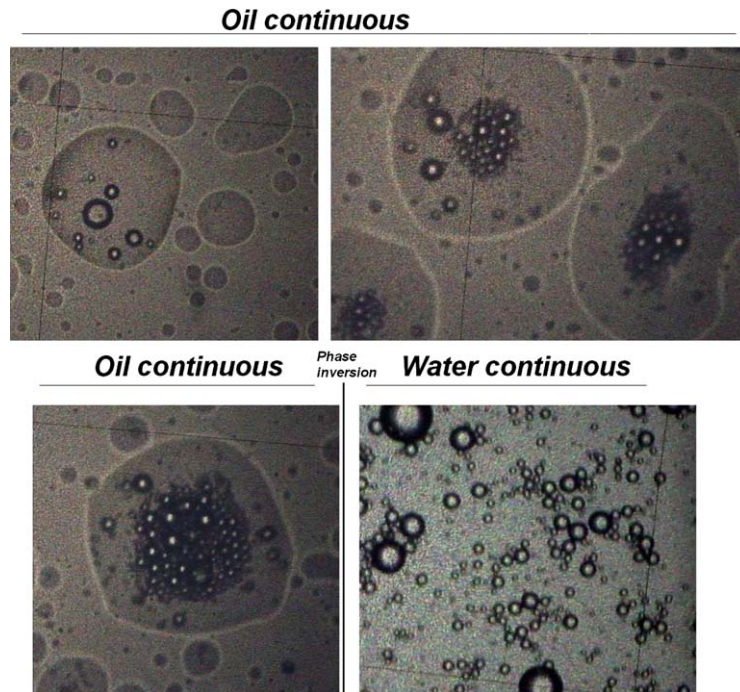


Fig. 8. Dispersion morphology for oil-to-water experiment as described in Fig. 9; left-top picture is taken at point 1 in Fig. 9, right-top picture at point 2, left-bottom picture at point 3 and right-bottom picture at point 4.

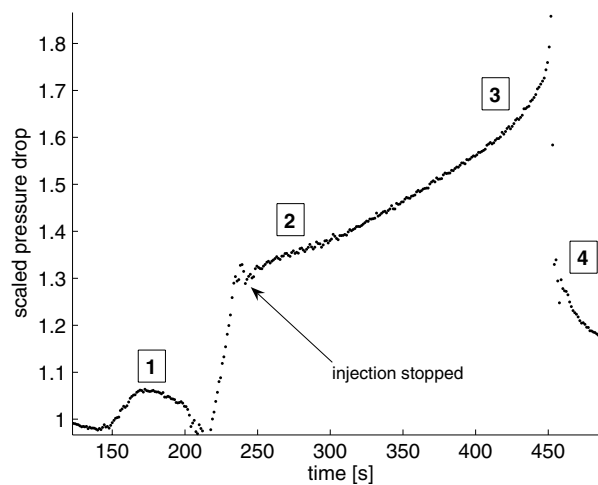


Fig. 9. Scaled pressure drop as a function of time for an oil-to-water experiment at a mixture velocity of 3.5 m/s and an injection flow rate of 5%. Injection stopped before inversion at a water volume fraction of 0.64.

For the oil-to-water experiments we also investigated the influence of the injection rate. For that purpose experiments were carried out for a mixture velocity of 3.5 m/s and injector flow rates of 7%, 10% and 15%. The Reynolds number at the start of the experiments was 11,000. For safety reason (too high pressure inside the loop) we could not go to larger values of the Reynolds number in this case. The results are given in Fig. 10, showing the scaled pressure drop as function of the water volume fraction for the three values of the injection flow rate. As is the case for the water-to-oil experiments, also for the oil-to-water experiments the development towards inversion is rather independent on the injection flow rate. However, the (critical) water volume frac-

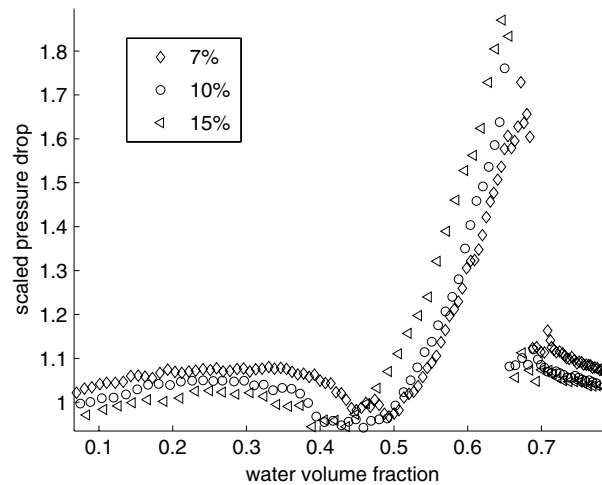


Fig. 10. Scaled pressure drop as a function of the water volume fraction for the oil-to-water experiments at a mixture velocity of 3.5 m/s and three different injection flow rates.

tion at the point of inversion depends on the injection rate (see Fig. 11 in which also the results for other injection rates are shown). The explanation for the decrease of the critical water volume fraction with increasing injection rate at large values of the injection rate is the same as for the water-to-oil experiments. However, the small value of the critical volume fraction at low values of the injection rate is new. The explanation is the fact (based on our observations), that at low injection rates the water drops have more time to entrain oil droplets and grow to a larger size than at high injection rates. So, a smaller amount of injected water is necessary for the inversion process to occur. As for the case of the water-to-oil experiments we did not find for the oil-to-water experiments any significant influence of the injector type on the inversion process at a mixture velocity of 3.5 m/s.

We also performed oil-to-water experiments at a mixture velocity of 2 m/s. As the oil viscosity is significantly larger than the water viscosity the Reynolds number of the flow is for this case significantly smaller than for the water-to-oil experiments carried out at 1 m/s, 2 m/s and 3 m/s. It is only 6500 at the start of the experiment. So for this case we could expect an influence of the injector type, as the turbulence of the mixture is not so dominant. The results are given in Fig. 12, showing the scaled pressure drop as function of water volume

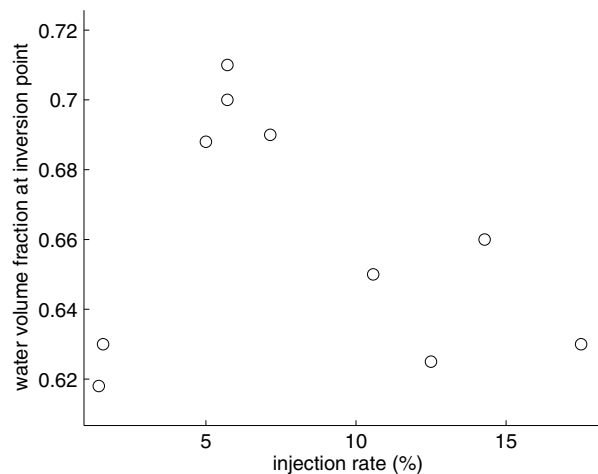


Fig. 11. Water volume fraction at point of inversion as function of injection rate and a mixture velocity of 3.5 m/s.

fraction for three types of injector. Indeed we find for this case an influence of the injector type on the inversion process.

We carried out more experiments similar to the one shown in Fig. 9, where the injection of water was stopped before inversion took place. After stopping the water injection the mixture continued to flow through the pipe loop at the same mixture velocity. All the conditions were the same as for the experiment of Fig. 9, only the water volume fraction at which the water injection was stopped was varied. The results are given in Fig. 13, which shows the scaled pressure drop as function of the water volume fraction for the different experiments. As can be seen the maximum pressure drop at the point of inversion is always nearly the same. Similar behavior was observed by Tyrode et al. (2005) in a rheomixer. In Fig. 14 we give the time that is needed after stopping the water injection at a certain water volume fraction to reach the inversion point. This time decreases with increasing value of the water volume fraction at which the water injection was stopped. The reason is that, at a high water volume fraction (at which the water injection was stopped) less time is needed for the multiple drops to grow and achieve a volume fraction large enough for inversion than at a low water volume fraction (at which the water injection was stopped). In all these experiments we observed the same

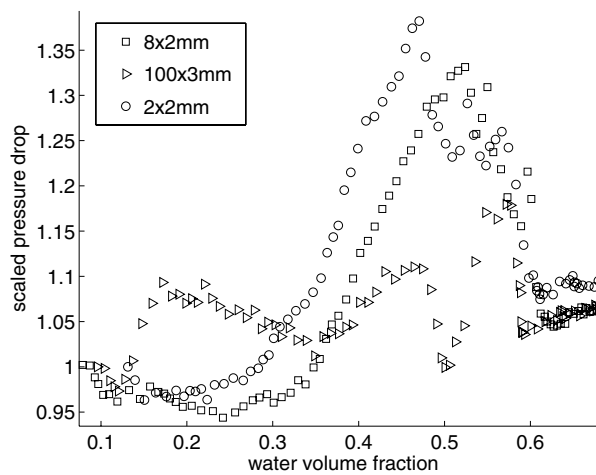


Fig. 12. Scaled pressure drop as function of water volume fraction for oil-to-water experiments at a mixture velocity 2 m/s, an injection rate of 12.5% and three different injectors.

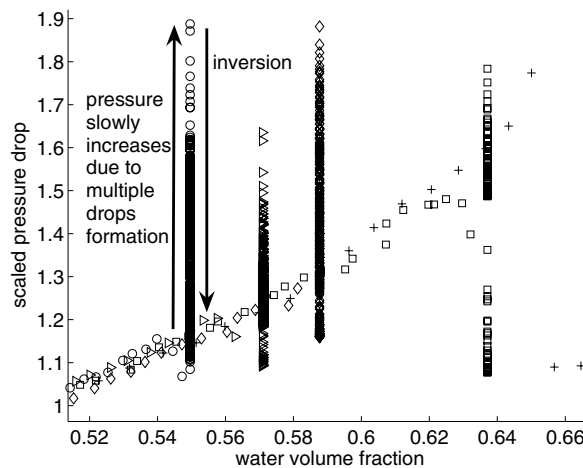


Fig. 13. Scaled pressure drop as function of water volume fraction for oil-to-water experiments at a mixture velocity of 3.5 m/s and for several values of the water volume fraction at which injection was stopped.

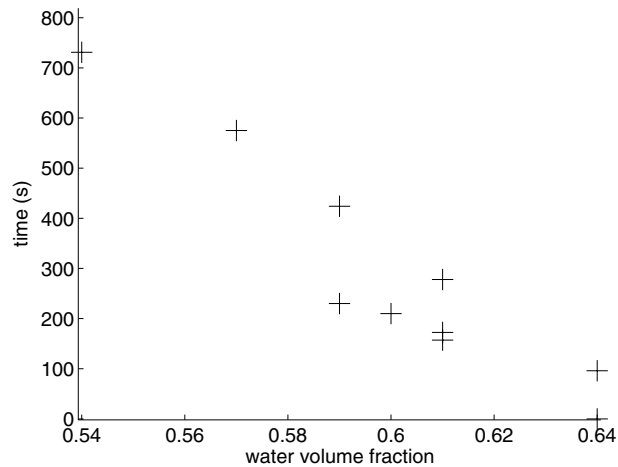


Fig. 14. Time to inversion after injection stop for oil-to-water experiments for a mixture velocity of 3.5 m/s as function of the water volume fraction at which injection was stopped.

phenomenon as shown in Fig. 8. Multiple drops (oil droplets in water drops) were formed before inversion. The volume fraction of oil inside the water drops increased with time. After inversion only “clean” oil drops were present; so no multiple drops were formed after the inversion process.

At this point we want to discuss the possible influence of the length of the pipe loop on our oil-to-water experiments. Inversion is for a significant part determined by the volume fraction of the dispersed phase. During our experiments this volume fraction increases with time because of two mechanisms: injection of the dispersed phase liquid and growth of the multiple droplets by taking-up more oil droplets. Apart from the experiments with a very low injection flow rate, for all other experiments the first mechanism is dominant. For these experiments the equilibrium drop size distribution and volume fraction of the dispersed phase establish themselves very quickly and there is no influence of the length of the pipe loop. We checked that by reducing the length of the loop and finding the same results. Also pressure drop measurements taken at various distances from the injector show the same results.

Of course, there is also no influence of the length of the pipe loop when only the second mechanism (growth of multiple drops) is present, as in that case there is no injection of liquid (and we know that the pump has no influence on the morphology of the dispersion). The growth of the multiple droplets is a slow process. In the experiment when injection was stopped at a water volume fraction of 0.54, it took 730 s for inversion to occur. This means that the dispersion travelled for 2.5 km in the pipe loop before inversion.

So only at very low injection flow rates, when the two mechanisms become of the same importance, we can expect an influence of the length of the pipe loop.

4. Discussion

For the case of water-to-oil experiments multiple drops (water droplets in oil drops) were not observed before inversion. Only after inversion multiple drops (oil droplets in water drops) were present. For the oil-to-water experiments the behavior was the other way around. Multiple drops (oil droplets in water drops) were observed before inversion; no multiple drops (water droplets in oil drops) were present after inversion. Similar behavior was observed by Pacek and Nienow (1995). They observed this non-symmetrical behavior even when the viscosities and densities of water and oil were nearly the same. A possible explanation for this phenomenon is, that the chemical properties of the two-phase system are important for multiple drop formation. Tyrode et al. (2005) introduces the so-called HLD-factor (hydrophilic–lipophilic deviation from an optimum formulation) to indicate the chemical formulation. When $HLD < 0$ the surfactant exhibits a stronger affinity to oil than to water, for $HLD > 0$ it is the other way around. Tyrode et al. (2003) and Tyrode et al. (2005) shows that for $HLD < 0$ two types of dispersion are possible: a dispersion with multiple drops

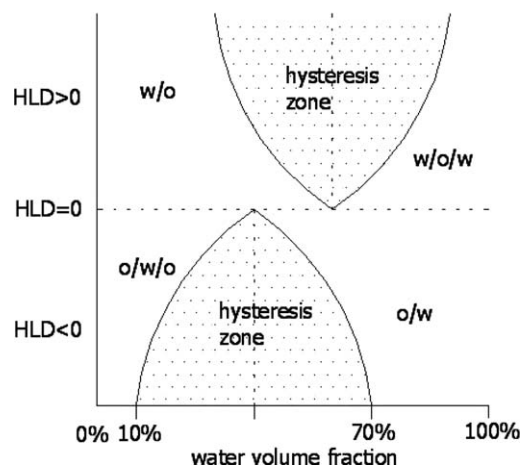


Fig. 15. Composition map for different hydrophilic-lipophilic deviation values.

consisting of oil droplets in water drops in oil, and a dispersion with clean oil drops in water. When $HLD > 0$ there are again two dispersion types: a dispersion with multiple drops consisting of water droplets in oil drops in water, and a dispersion with clean water drops in oil (see also Fig. 15 of Tyrode et al., 2005). In our experiments we observed the following two types of dispersion: a dispersion with multiple drops consisting of oil droplets in water drops in oil, and a dispersion with clean oil drops in water. So we assume that for our case $HLD < 0$. We plan to carry out experiments at $HLD > 0$ to check this explanation.

These observations let us conclude, that for our experiments the inversion mechanism for water-to-oil experiments is different from the mechanism for oil-to-water experiments. For water-to-oil experiments multiple drops are not formed before inversion; so the effective dispersed phase fraction is equal to the actual oil volume fraction. The volume fraction of the oil drops increases until concentration becomes so high, that phase inversion occurs. The value of the dispersed volume fraction at the point of inversion depends on the injection rate. At higher injection rates the inversion takes place at lower values of the dispersed volume fraction than at lower injection rates. As explained earlier the reason is likely, that at higher injection rates the probability of fast growing inverted pockets increases. In this case the dispersion is very stable, even pumping it through the pipe loop for a long time did not change the pressure drop or dispersion morphology. Even at a very high dispersed phase volume fraction multiple collisions did not lead to encapsulation of water as reported by Núñez et al. (1996). The explanation is in our opinion, that encapsulation is determined by chemical properties of the system and that in their case $HLD > 0$.

For oil-to-water experiments water drops are present, that continuously entrain small amounts of oil. So the effective dispersed phase volume fraction is larger than the water volume fraction. The multiple droplets grow continuously in size, until their volume fraction is so large that inversion takes place. Entrainment of oil into the water drops and the possible escape of oil droplets from the water drops are the two mechanisms, that determine the growth rate of the drops (Groeneweg et al., 1998; Liu et al., 2005; Sajjadi et al., 2000, 2002). Based on this reasoning it is to be expected, that the dispersed phase volume concentration at inversion is lower for the oil-to-water experiments than for the water-to-oil experiments. This is in agreement with our results, although the effect for our experiments is not very strong because also other influences are important (for instance the injection flow rate). It is important to emphasize at this point, that the dispersed phase volume fraction at inversion for (all) our wash-out route type experiments is much higher than the dispersed phase volume fraction at inversion for the direct route type of experiments performed by Ioannou et al. (2004, 2005), where inversion occurs typically for an oil volume fraction around 0.7. In our experiments inversion was delayed up to 0.75 for the water volume fraction for oil-to-water inversion and up to 0.90 for the oil volume fraction for the water-to-oil inversion.

The pressure drop increase at inversion compared to the pressure drop at the start of the experiment is for oil-to-water experiments equal to about 2, whereas for the water-to-oil experiment it is about 4 (so consider-

ably larger). This is likely caused by the difference in drop size for the two experiments: for the oil-to-water experiments the drops are significantly larger (because of multiple drop formation) than for the water-to-oil experiments. According to Pal (2000) the effective viscosity of a dispersion with drops of a certain size is larger than the effective viscosity of a dispersion with larger drops, when the dispersed volume fraction is the same for both cases. Moreover, at inversion the dispersed volume fraction for water-to-oil experiments is larger than for oil-to water experiments.

Acknowledgements

The author are grateful to Shell and FOM (the foundation of fundamental physics in The Netherlands) for the financial support to this project.

References

- Arashmid, M., Jeffreys, G., 1980. Analysis of the phase inversion characteristics of liquid–liquid dispersions. *AIChE J.* 26 (1).
- Bouchama, F., van Aken, G., Autin, A., Koper, G., 2003. On the mechanism of catastrophic phase inversion in emulsions. *Colloids Surf. A: Physicochem. Eng. Aspects* 231, 11–17.
- Brauner, N., 2001. The prediction of dispersed flows boundaries in liquid–liquid and gas–liquid systems. *Int. J. Multiphase Flow* 27, 885–910.
- Brauner, N., Ullmann, A., 2002. Modelling of phase inversion phenomenon in two-phase pipe flows. *Int. J. Multiphase Flow* 28, 1177–1204.
- Groeneweg, F., Agterof, W., Jaeger, P., Janssen, J., Wieringa, J., Klahn, J., 1998. On the mechanism of the inversion of emulsions. *Trans IChemE A* 76.
- Ioannou, K., Hu, B., Matar, O., Hewitt, G., Angeli, P., 2004. Phase inversion in dispersed liquid–liquid pipe flows. In: 5th International Conference on Multiphase Flow, ICMF'04. Yokohama, Japan.
- Ioannou, K., Nydal, O., Angeli, P., 2005. Phase inversion in dispersed liquid–liquid flows. *Exp. Thermal Fluid Sci.* 29, 331–339.
- Liu, L., Matar, O., Perez de Ortiz, E., Hewitt, G., 2005. Experimental investigation of phase inversion in a stirred vessel using lif. *Chem. Eng. Sci.* 60, 85–94.
- Nienow, A., 2004. Break-up, coalescence and catastrophic phase inversion in turbulent reactors. *Adv. Colloid Interface Sci.* 108, 95–103.
- Núñez, G., Brieceno, M., Mata, C., Rivas, H., Joseph, D., 1996. Flow characteristics of concentrated emulsions of very viscous oil in water. *J. Rheol.* 40 (3), 405–423.
- Pacek, A., Nienow, A., 1995. A problem for the description of turbulent dispersed liquid–liquid systems. *Int. J. Multiphase Flows* 21, 323–328.
- Pal, R., 2000. Shear viscosity behavior of emulsions of two immiscible liquids. *J. Colloid Interface Sci.* 225, 359–366.
- Sajjadi, S., Zerfa, M., Brooks, B., 2000. Morphological change in drop structure with time for abnormal polymer/water/surfactant dispersions. *Langmuir* 16, 10015–10019.
- Sajjadi, S., Zerfa, M., Brooks, B., 2002. Dynamic behaviour of drops in oil/water/oil dispersions. *Chem. Eng. Sci.* 57, 663–675.
- Tyrode, E., Mira, L., Zambrano, N., Márquez, L., Rondón-Gonzalez, M., Salager, J., 2003. Emulsion catastrophic inversion from abnormal to normal morphology. 3. Conditions for triggering the dynamic inversion and application to industrial processes. *Ind. Eng. Chem. Res.* 42, 67–74.
- Tyrode, E., Allouche, J., Choplin, L., Salager, J., 2005. Emulsion catastrophic inversion from abnormal to normal morphology. 4. Following the emulsion viscosity during three inversion protocols and extending the critical dispersed-phase concept. *Ind. Eng. Chem. Res.* 44, 67–74.
- Vaessen, G., Visschers, M., Stein, H., 1996. Predicting catastrophic phase inversion on the basis of droplet coalescence kinetics. *Langmuir* 12, 875–882.
- Yeo, L., Matar, O., Perez de Ortiz, E., 2002. A simple predictive tool for modelling phase inversion in liquid–liquid dispersions. *Chem. Eng. Sci.* 57, 1069–1072.