Liquid–Liquid Phase Separation in Gravity Settler with Inclined Plates

Lars Schlieper, Michael Chatterjee, Martin Henschke, and Andreas Pfennig
Lehrstuhl für Thermische Verfahrenstechnik, RWTH Aachen, D-52062 Aachen, Germany

Experimental investigations were made into the separation behavior of three different liquid–liquid dispersions in horizontal phase separators including internals. The test setup used, consisting of two separators of 200 mm diameter, enables direct comparative measurement of different internal configurations. The investigation focused on how inflow to the plates, the plate material, and the distance between the plates influenced separation length in the separator. Measurements performed for a range of total volume flows and phase ratios reveal that internals can achieve a reduction in the separation length required of up to 75% compared to that of a separator with no internals. It was also shown that discontinuous settling tests in a glass cylinder including internals can be used in achieving an initial engineering design for separators including internals. © 2004 American Institute of Chemical Engineers AIChE J, 50: 802–811, 2004
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Introduction

The goal in separating liquid–liquid dispersions is to separate the dispersed droplets from the continuous phase, to obtain two uniform phases. One of the main applications for dispersion separation is liquid–liquid extraction (Bohnet, 1976; Deibele et al., 2000; Eckert and Gormely, 1989). In the field of environmental technology, undesired dispersions, for example, oil–water mixtures in offshore technology (Hansen et al., 1995), frequently require separation. The main steps of separation are the sedimentation of the dispersed droplets and coalescence, as shown in Figure 1 and described in detail by Henschke (1995). Dispersion separation is frequently achieved using continuously running, horizontal gravity settlers. In terms of practical application, a distinction is made between gravity settlers with no internals and those including internals. Internals are used specifically for enhancing coalescence, thus increasing separation efficiency. Additional coalescent agents

enable the separator volume to be reduced (Mizrahi and Barnea, 1973), which as well as saving space also reduces the potential hazard with toxic and inflammable materials. Figure 2 shows a separator of this kind with parallel plates longitudinally impacted by the flow. The plates are inclined perpendicular (see Figure 3) to the direction of flow. The inclination ensures that the large droplets or trickling films formed by the plate internals can reach the main interface (Figure 2) for the final separation. An exchange of the phases between the plates is realized by a gap between the wall of the vessel and holes at the top of each plate. The installed sealing rings avoid a bypass flow. The uniform influence of the plates with dispersion is achieved by a perforated plate in the inflow zone, which simultaneously reduces the momentum of the inflowing dispersion.

For engineering liquid–liquid phase separators without internals, a great number of experimental investigations (Amasheh, 1989; Drown and Thomson, 1977; Henschke, 1995; Jeffreys et al., 1971; Murray, 1979; Siemons, 1985; Vijayan and Ponter, 1976) and also efficient engineering design models (Golob and Modic, 1977; Hartland and Jeelani, 1989; Henschke, 1995; Jeelani and Hartland, 1988; Jeffreys and Davies, 1971; Müller, 1969; Müller and Kreichelt, 1979; Reissinger et
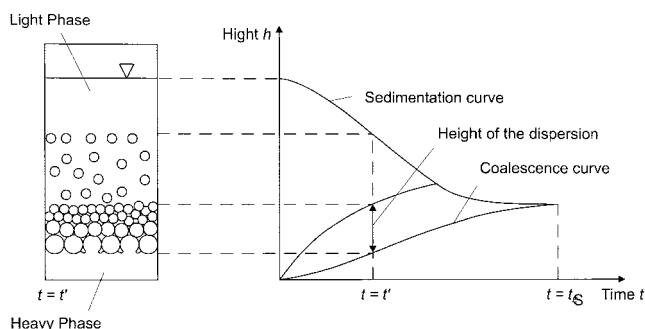
Figure 1. Description of the settling test.

al., 1981; Sklokin et al., 1988; Stönner, 1981; Stönner and Wöhler, 1975) are available. In contrast, only few publications have focused on the properties of separators including internals.

The experimental investigations into separators including internals can be subdivided into experiments with a high and a small amount of dispersed phase. In cases of a high amount, a dense-packed dispersion layer occurs in the separator (see Figure 2) when the sedimentation of the droplets is faster than the coalescence. In the second case, mostly the sedimentation of small single droplets can be observed. Regarding the latter, Morrison (1970) carried out experiments separating oil-contaminated wastewater with the aim of investigating the separation performance of separators with corrugated plates. Weiß (1987) also investigated a particular application for the separation of small quantities of dispersed phase using internals in the field of producing basic ingredients of washing powder. The publications by Franken and Heikamp (1991, 1992) relate both to the separation of small dispersion phase fractions and experiments in which a dense-packed dispersion layer was formed. In their experiments, both authors compare the separation performance of separators fitted with inclined profiles and separators with no internals. Investigation into the separation of dense-packed dispersion layers using internals were completed both by Lewis (1977) and Barnea and Mizahi (Barnea and Mizrahi, 1977; Mizrahi, 1973). These experiments centered largely on the investigation of dispersion bands. In practice, however, the dense-packed layer mostly occurs in the form of a wedge [see, for example, Figure 2 in Henschke (1995)]. Regarding the latter, only a few investigations are available in the literature, so that achievement of a design model requires the completion of additional experiments.

Alongside these practical investigations, the literature includes the engineering approach from Hooper and Jacobs (1979), consisting of a design diagram for a plate separator.
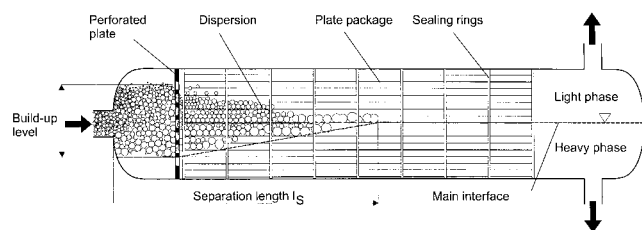

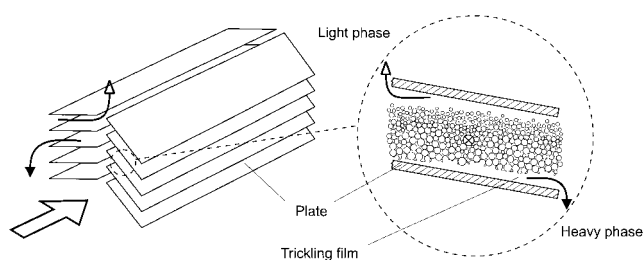**Figure 2. Separator including internals with a heavy dispersed phase.**

Figure 3. Plates of the separator with trickling film.

This enables determination of the diameter of the smallest droplet to reach the interface during its residence time in the separator because of sedimentation. Rowley and Davies (1988) propose a further sedimentation-oriented model, describing droplet sedimentation between parallel plates. In this model, droplets having reached the surface of the plate are regarded as separated. Blaß et al. (Meon et al., 1993; Rommel et al., 1992a,b, 1993) developed a model describing the coalescence of a single droplet in the trickling film of dispersed phase, which wets the inclined plate. Derivation of the model is based on extensive investigation into the coalescence of single droplets on inclined plates.

Models describing separation in the dense-packed layer that take account of the coalescence at the plate are not presently available. Unlike for separators without internals, there are also no comparable detailed investigations into the impact of system properties on the separation process. Thus, the aim of this investigation was to build an experimental database for the separator with internals, shown in Figure 2, for later development of a design method based on sound physics (Chatterjee, 1998; Schlieper, 2001).

Test Setup and Experimental Procedure

Experimental separator setup

The investigations into continuous separation of dispersions were performed with an experimental plant of pilot scale (the settler is shown in Figures 2 and 3; the flowchart in Figure 4). The pilot plant essentially consisted of two geometrically identical separators of 200 mm diameter. The separators operate both with and without internals, thus allowing the increase in separation rate attributed to the internals to be quantitatively
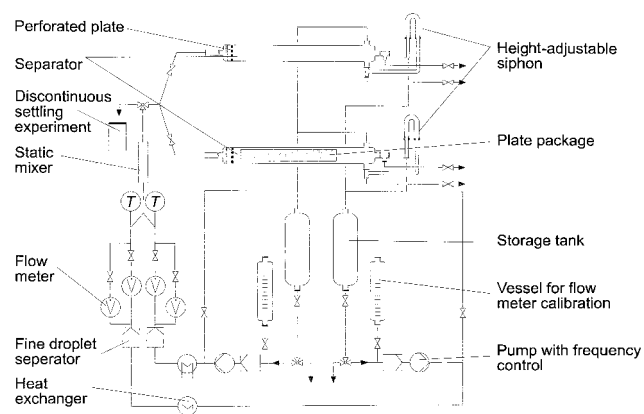

Figure 4. Flowchart of the separator used.

evaluated by direct comparison. The piping shown in Figure 4 allows the separator to be operated consecutively with different internal configurations without the need for any structural modifications. This ensures that during the series of measurements no contamination enters the separator. Contamination can greatly affect the process of coalescence, as investigation of single droplets has shown (Hartland, 1968,; Krassimir et al., 1999). To ensure that the system was not affected, the equipment was built from borosilicate glass standard components (Schott Engineering, Mainz, Germany), PTFE couplings, and stainless steel components.

The inflow zone of each separator includes an extension adapter (nominal diameter 50 to 200 mm) and a perforated plate, to reduce inflow momentum and ensure distribution of the dispersion. During operation with internals, the plate package is located just behind the perforated plate and is housed in a 2-m-long glass tube. At the end of this tube are the phase discharge exits (nominal diameter 50 mm). The two phases flow through these exits into separate collecting tanks. The phases, now separated, flow into PTFE-capped peripheral magnet centrifugal pumps (type: GEKO Block 25-100 RM-30-SIC-gek._d, 1.85 kW, 380 V, 50 Hz, 3000 min^{-1} Eex e II T3, supplied by GEKO, Eltville am Rhein, Germany) by separating sieves. From here the fluid is transported to heat exchangers (type NAK 6/25; Schott Engineering), from where it enters the fiber bed separators (glass-fiber mats; Schroer Caspar, Aachen, Germany). The fiber-bed separators are to prevent enrichment of the nonseparated fine droplets in each phase. Next, each phase is fed through a suspended solid volume flowmeter (nominal diameter 40 mm, type G2.2500, accuracy of measurement ±2%; Schott Engineering). Both phases flow through a hose (nominal diameter 25 mm), in which they are contacted. The dispersion is generated with the aid of a static mixer including six elements (type: KMS-KMR 25; Kenics, Hamburg, Germany). The dispersion reaches one of the separators through the pipe configuration (nominal diameter 15 mm) located downstream. A dispersion pickoff point permits completion of settling tests in a glass cylinder. Various other components permit calibration of the flowmeters.

The control part of the system essentially consists of two separate frequency converters (type: ACP 6004-0, 4 kW; Berges Electronic, Marienheide-Rodt, Germany) used to control the pump flow rate. The system also includes, upstream of the static mixer, temperature measurement points (resistance thermometer PT 100, type WID 25/150/D to DIN 43750; Schott Engineering). The temperature measurement points ensure that the external closed-circuit cooler (type: WK5000, 6 kW, 380 V, control precision ± 0.5°C, constancy of temperature < ±1°C; Lauda, Lauda-Königshofen, Germany) maintains the fluid at a constant temperature of 20°C.

Two perforated plates, designated type A and type B, respectively, are used. In type A, with a perforation ratio of $m = 0.15$, the perforations of 14 mm are uniformly distributed over the surface, whereas the central section of type B (same perforation ratio) contains no perforations (see Figure 5). The geometry of perforated plate B is used to reduce the dispersion inflow speed, particularly at the interface at the midline of the separator. Investigations were made with smooth, parallel plates inclined at an angle of $\beta = 10°$, supplied by Franken Filtertechnik (Hürth, Germany) and shown in Figures 2, 3, and 5. Preliminary investigations proved this type of plate as par-
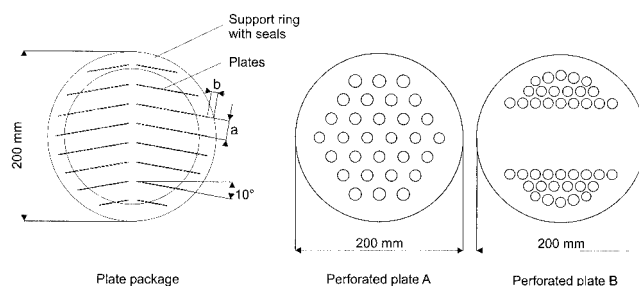

Figure 5. Plate assembly and perforated plates viewed in the direction of flow.

ticularly well adapted for dispersion separation (Chatterjee, 1998). Distances between the plates of $a = 5, 10, 22$, and 46 mm were realized for the plate materials polypropylene and stainless steel. The plates had a thickness of 2 mm and a length of 640 mm ($a = 5$ mm) or 960 mm ($a = 10, 22, 46$ mm). The individual plates were joined by sealing rings, located longitudinally along the plates at intervals of 80 mm, and thus forming a plate package.

Preparation of the setup

To exchange internals after each series of tests, the adapter in the inflow zone of the separator was removed and the perforated plate (or plate package) replaced. Before assembly in the separator the internals were first cleaned with ethanol and then deionized water in an ultrasonic bath. When the dispersion was changed, the plant was rinsed several times using deionized water after removal of the dispersion. The plant was then filled with about 70 L of organic phase and about 85 L of aqueous phase of the new dispersion.

Experimental procedure and quantities measured

In each test series, the separation performance of the internals in the separator for total volume flows of $\dot{V}_T = 0.3$ to 1.2 m^3/h was investigated. Additionally, at a constant total volume flow, the *w*-phase ratio

$$\alpha = \frac{\dot{V}_{ac}}{\dot{V}_{ac} + \dot{V}_{org}} \tag{1}$$

was varied. Before recording any measurements, for each test the volume flows are adjusted by the setup control device. Then the level of the interface near the outlets of the settler is adjusted by varying the height of the siphons to lie in the middle of the separator. After steady state in the separator is reached (normally after about 15 min), the measurements describing separation behavior in the separator are recorded.

When the dispersion enters the separator (including or without internals) a dense-packed layer of droplets forms in the area in front of the perforated plate as a result of sedimentation, as shown in Figure 2. This layer is defined in terms of total congestion level. A wedge-shaped dense-packed dispersion geometry then becomes visible in the separator including internals, which, starting from the initial wedge height, becomes reduced because of coalescent and flow processes. The length along the glass wall of the separator at which separation ter-

minates is described as the separation length ($L_{s\ with\ Internals}$; see Figure 2). While measuring separation length it is observed that single droplets or clusters of droplets remain longer at the interface and are carried away from the plate package. If separation takes place in a separator with no internals, a congestion layer also forms in front of the perforated plate. Downstream of the perforated plate a zone is formed in which the droplets show very little coalescence, but continue to merge because of sedimentation. The corresponding length is designated inflow length, after which the sedimentation is almost completed. A wedge of dense-packed droplets is also subsequently formed, but in this case the wedge is much flatter than that with internals. At the end of the wedge the dispersion becomes separated from the wall. However, a proportion of droplets are moving further forward with the flow, so that the end of the wedge becomes parabolic. In the separator with no internals, separation length is thus defined as the mean value of two lengths: the point where the dispersion separated from the wall and the end of the wedge.

$$L_{s\ without\ Internals} = \tfrac{1}{2}\left(L_{s\ wall\ separations} + L_{s\ end\ of\ wedge}\right) \qquad (2)$$

In the separator with no internals, inflow and separation length can be measured to an accuracy of ± 50 mm. In contrast, the accuracy of separation length measurement in the separator including internals is ± 80 mm. Measurement of the initial congestion level in front of the perforated plate can be measured to an accuracy of ± 10 mm.

It can be observed that, in contrast to the experiments with internals, in those experiments without baffles and internals carried out by Henschke (1995) the inflow length can take more than 50% of the total separation length. Because of the density difference between the light phase, dense-packed droplet layer, and the heavy phase an additional pressure in the wedge drives the dense-packed droplets in the flow direction to the end of the vessel. Depending on the dimensions of the vessel and the flow rate a dense-packed dispersion band can also be formed. This geometry can be calculated by the model of Henschke (1995) for separators without internals as well.

Discontinuous settling test setup and experimental procedure

Alongside the continuous dispersion separation in the separator, investigation is also made into the discontinuous separation behavior of the dispersions used. In the engineering design of separators with no internals discontinuous settling tests are performed to take the specific coalescence properties of the dispersions into account (Henschke, 1995). Furthermore, settling tests provide the basis of engineering design of separators with no internals (Golob and Modic, 1977; Henschke, 1995; Müller, 1969; Müller and Kreichelt, 1979; Reissinger et al., 1981; Sklokin et al., 1988) and can thus also be regarded as an appropriate basis for the design of separators including internals (Schlieper, 2001).

The settling tests are completed in a glass cylinder of 300 mm height and 80 mm inner diameter. In the following, a distinction is made between two types of settling test. First, settling tests completed in the agitator vessel and, second,
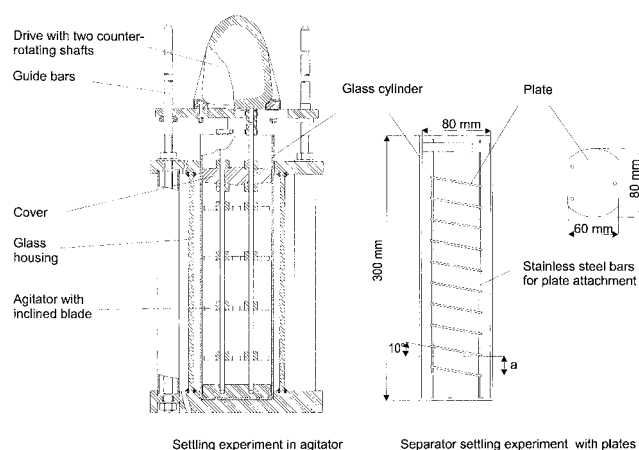

Figure 6. Test assembly used for settling tests.

settling tests of dispersions taken directly from the continuous separator test setup (see Figure 6).

The settling tests according to Henschke (1995) are carried out in an agitator vessel (see Figures 1 and 6). The agitator consists of two counterrotating shafts, each bearing four inclined agitator blades. The top of the glass cylinder is closed by a cover that prevents air being mixed in. Before performing the tests, the glass cylinder is first cleaned with ethanol, followed by deionized water in an ultrasonic bath. A 1.2 L sample of the dispersion with the appropriate phase ratio was placed in the glass cylinder. The dispersion process takes place at 800 min^{-1} for 60 s. Time recording starts at the moment when the agitator is stopped. The settling experiment ends when 50% of the interface between the formally dispersed and the continuous phase remains covered by a single layer of droplets. It is appropriate to define the settling time t_s in this way, given that because of stochastic processes, single droplets remain for a very long period at the interface, thus leading to strongly varying experimental results. Defining the end of the coalescence as the time when 50% of the liquid–liquid interface remains covered with a single layer of droplets leads to a reproducibility of ± 1 s also in the case with internals (see also Figure 1). In addition to measuring settling time, the separation process is recorded by a video camera. Subsequent video analysis records the measurement values for the sedimentation and coalescence curve (Jeelani and Hartland, 1998) of the settling test.

When performing the discontinuous settling tests in the separator, a distinction is made between settling tests with no internals and those including internals. The plates used are made of polypropylene, as used in the separator. The plates are fixed in relation to each other by three stainless steel bars. Distances between plates of $a = 10$, 22, and 46 mm at an angle of inclination of $\beta = 10°$ were achieved (see Figure 6). When completing the tests, the glass cylinder is positioned below the stopcock of the continuous separator test setup. When the stopcock of the installation is opened the dispersion flows into the glass cylinder. The phase ratio is the same in the continuously operation settler as adjusted by the control device of the pumps. When a fill-up volume of 1.2 L is reached, the stopcock is closed and measurement of settling time and video recording starts.

Table 1. Property Data of the Dispersions Investigated

| Property | Dispersion water + | | |
	n-Butanol	Tridecanol	Cyclohexanone
$w_{org,org}$ in kg/kg	0.80	0.98	0.92
$w_{aq,aq}$ in kg/kg	0.92	1.00	0.98
ρ_{org} in kg/m^2	846	848	953
ρ_{aq} in kg/m^3	987	1000	999
η_{org} in MPa $\cdot$ s	3.324	41.9	2.306
η_{aq} in MPa $\cdot$ s	1.463	1.00	1.323
σ in mN/m	1.75	14.0	3.8
Sources	(Franken and Heikamp, 1992; Stönner, 1981)	(Stönner and Wöhler, 1975)	(Drown and Thomson, 1997)

Stopwatch recording of settling time can be completed to an accuracy of approximately ± 3 s. Video recording of the discontinuous settling tests allow the values of the coalescence curve and sedimentation curve to be measured to a tolerance of ± 3 mm. Before each test with the continuous separator test setup, a discontinuous settling test is performed using the dispersion from the test setup. This ensures that the settling time for each dispersion used does not change by more than $\pm 10\%$ of the average value in the test period.

Dispersions used

Three dispersions, water + n-butanol, water + cyclohexanone, and water + tridecanol, were selected for investigation. All dispersions had already been used as test systems in extraction (Hirschmann, 1984; Misek et al., 1985; Wagner, 1999) because they yield a broad range of property data. Table 1 compares the property data of the systems.

As Table 1 shows, the essential differences between the systems are those of density and viscosity of the homogenous phases, as well as surface tension. The dispersion water + cyclohexanone shows a density difference of 46 kg/m^3, whereas this difference in the other two dispersions (141 and 152 kg/m^3, respectively) is much higher. The water + tridecanol system is notable for the high difference in viscosity of the two phases (40.9 mPa $\cdot$ s), which is ten times smaller for the other two dispersions. Also, the water + tridecanol system has a much greater surface tension than that of the other two systems.

Experimental Results

Settling experiments

Alongside definition of the dispersions in terms of system properties, dispersion behavior is described by the discontinuous settling experiments in the glass cylinder. Figure 7 shows the sedimentation and coalescence curves resulting from analysis of the mixing experiments tests for the three dispersions with w-phase fractions $\alpha = 0.167$ and 0.833. For the water + tridecanol system the organic phase was always dispersed over the entire range of phase ratio investigated. The change of dispersion direction from dispersed aqueous phase to a dispersed organic phase for the water + n-butanol system occurs in the transition range of $\alpha = 0.36$ to 0.383; for water + cyclohexanone the transition range lies between $\alpha = 0.35$ and $\alpha = 0.45$.

For a dispersed organic phase ($\alpha = 0.833$ in Figure 7) upward sedimentation of the droplets occurs. Here the lower curve corresponds to the sedimentation curve. The sedimenta-

tion curve can be used to calculate the Sauter diameter from the sedimentation speed of a single droplet in the swarm (Jeelani and Hartland, 1998). The swarm model according to Pilhofer and Mewes (1979) combined with the single-droplet sedimentation model according to Ishii and Zuber (1979) was applied here. As the calculation results in Table 2 show, the dispersion water + cyclohexanone yields the largest droplets, whereas water + tridecanol results in the smallest droplets.

At the interface, the droplets become deposited in a dense-packed layer of droplets and coalesce with their homogeneous phase. After droplet sedimentation is complete, the dense-packed layer of droplets coalesces completely. With the water + tridecanol dispersion, the good adhesion of the tridecanol droplets to the glass cylinder, which was particularly evident, made recording of measurements more difficult.

As Figure 7 shows, the water + tridecanol system is notable for settling times of 176 and 186 s. Both the water + n-butanol (54 and 62 s) and the water + cyclohexanone systems (45 and 55 s) show markedly shorter separation times. This fact alone, under otherwise identical conditions, would lead us to expect markedly longer separation lengths for water + tridecanol in the separator compared to those of the other two systems.

Separation behavior in the separator with no internals

Before describing the tests including internals, first a summary of the facts revealed about separation in the separator with no plates but including a perforated plate is given. Figure 8 shows the separation lengths measured for the dispersions investigated at a constant w-phase fraction $\alpha = 0.5$. As can be seen in Figure 8, there is a much greater increase in separation
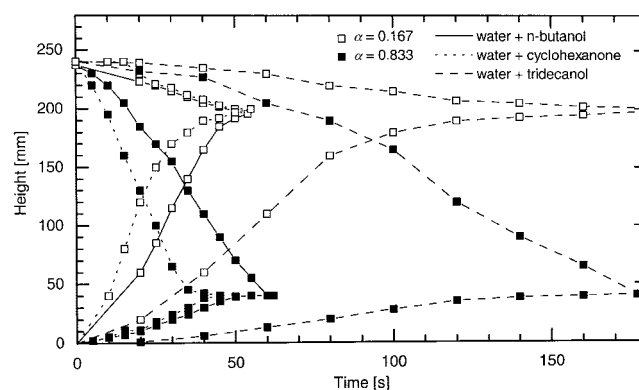

Figure 7. Sedimentation and coalescence curve of the systems investigated.

Table 2. Sauter Diameter Calculated from Discontinuous Settling Experiments

	Water + n-butanol		Water + tridecanol		Water + cyclohexanone	
α	Dispersed phase	ϕ_{32} (mm)	Dispersed phase	ϕ_{32} (mm)	Dispersed phase	ϕ_{32} (mm)
0.167	w-phase	0.59	o-phase	1.23	w-phase	1.1
0.333	w-phase	1.17	o-phase	0.77	w-phase	1.6
0.500	o-phase	1.01	o-phase	0.57	o-phase	2.1
0.667	o-phase	0.65	o-phase	0.41	o-phase	1.4
0.833	o-phase	0.49	o-phase	0.26	o-phase	0.9

length with volume flow in the water + tridecanol system than occurs with water + n-butanol or water + cyclohexanone, thus confirming the tendency suggested by the settling times. Especially significant is the volume flow of $\dot{V}_T = 0.2$ m³/h, at which the separation length and the inflow length become identical to the water + tridecanol system. In this case, dispersion separation is completed upstream of the perforated plate. This behavior occurs likewise with the two other dispersions at volume flows below $\dot{V}_T = 0.4$ m³/h. This can be regarded as the main indicator that dispersion separation occurs in front of the perforated plate. Additionally it can be observed from the continuous experiments that, similar to the settling experiments in front of the perforated plates, the droplet diameter rises in the dense-packed layer toward the main interface (see Figures 1 and 2), which indicates coalescence. The amount of coalescence in front of the perforated plate depends on the phase ratio, velocity, and the incoming droplet distribution.

Figure 8 gives the general impression that the responses of the water + n-butanol and water + cyclohexanone dispersions are very similar. Examination of the settling times of these two systems reveals that, although this result is to be expected, it is simultaneously very surprising in view of the very large difference in density between the homogeneous phases of the two systems (see Table 1). However, as in the settling test, the water + cyclohexanone system exhibits larger droplets than those of the water + n-butanol system in the separator. In relation to separation in the separator, the effect of density difference and droplet size would appear to be compensatory.

Effect of perforated plates on separation behavior in the separator including internals

The distribution of the dispersion over the plates is a key factor in dispersion separation in the separator including inter-

nals. High congestion levels in front of the perforated plate enable the dispersion to enter between plates over the whole cross section of the separator. A quantitative comparison of the perforated plates A and B investigated (see Figure 5) is given in Figure 9, which shows initial congestion level vs. w-phase fraction. Measurements were performed for a separator with a distance between plates of $a = 22$ mm at a volume flow of $\dot{V}_T = 0.6$ m³/h.

As expected, perforated plate B, without perforations at the level of the separator midline, shows greater initial congestion levels for the investigated dispersions than perforated plate A. The water + tridecanol system always shows a decrease in congestion level as w-phase fraction increases with a dispersed phase, which is always organic. Related to the volume fraction of the dispersed phase (designated holdup) the level of congestion decreases as holdup decreases. This phenomenon is also evident for water + n-butanol when perforated plate B is used. In contrast, the level of congestion for perforated plate A is almost completely independent of the w-phase fraction.

Effect of perforated plates on separation lengths in the separator including internals

The differences in level of congestion in front of the perforated plate affect the separation process in the internals zone. Figure 10, which shows separation lengths for the investigated dispersions vs. total volume flow at constant w-phase fraction $\alpha = 0.5$, shows this clearly. For the dispersion water + n-butanol, Figure 10 shows slightly shorter separation lengths for perforated plate B than for perforated plate A. This tendency is attributed to the previously mentioned improvement in the way in which the dispersion is distributed over the plate package, and is also reflected in w-phase fractions other than those shown.
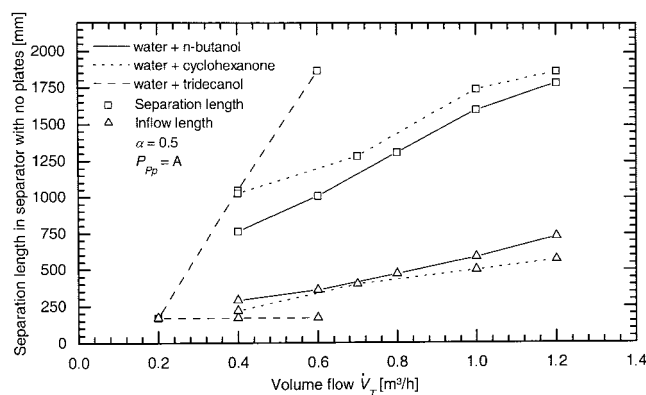

Figure 8. Separation length in the separator with no internals using perforated plate A.
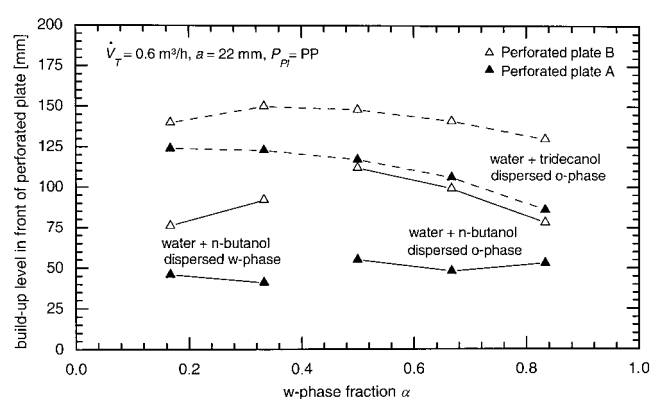

Figure 9. Impact of perforated plate geometry on build-up level of dispersion.
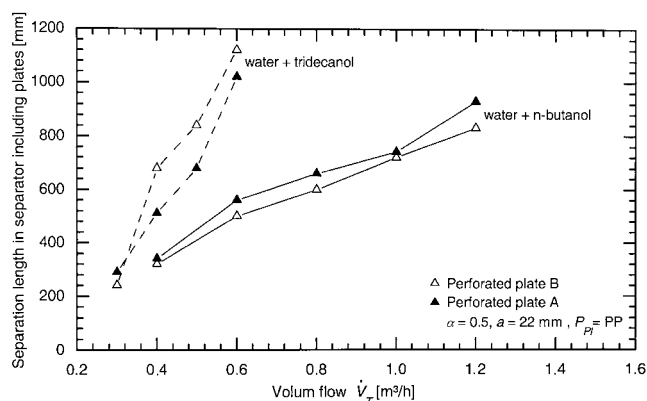
Figure 10. Separation length in the separator with internals for both perforated plate geometries.

Figure 10 also spotlights a particularity of the experimental results for the water + tridecanol system: using plates with a plate distance of $a = 22$ mm, the separation length is greater for perforated plate B than for perforated plate A. In contrast, measurements taken in the separator with no internals for this dispersion reveal that perforated plate B achieves faster dispersion separation than perforated plate A. This fact can be explained with reference to the position of the dispersion layer in front of perforated plate B in relation to the horizontal separator midplane. With identical parameters, this level is normally higher for the water + tridecanol dispersion than for the water + n-butanol system. Because of this, the dispersed tridecanol phase especially influences the upper plates, whereas almost all plates in and below the central zone of the separator remain unused when perforated plate B is in use. The circular cross section of the separator means that both the plate width and flow cross section are greater at the separator midline than in the upper section. This produces a flow that is faster in the upper section of the separator. In contrast, with perforated plate A in use, the flow channels at the separator midline are used, thus resulting in better separation than that with perforated plate B.

Effect of plate material on separation length in the separator

Beside the effect of the perforated plates on the way the dispersion enters the plates, the separation behavior of the separator including internals is also affected by the plate material. The interaction between phase and plate can be discussed in terms of plate wetting. Evaluation of plate wetting by the dispersed phase makes use of the wetting angle φ, measured between one droplet of the dispersed phase and the plate (Chatterjee, 1998; Meon, 1993; Schlieper, 2001). If wetting of the plate material by the droplets of the dispersed phase is good, the droplets spread over the plate ($0° < \varphi < 90°$). In contrast, wetting is poor between $90° < \varphi < 180°$. Observations of the three dispersions show that wetting of the polypropylene plates by the organic phase is always good, whereas wetting by the aqueous phase droplets is poor. Use of stainless steel plates shows a reverse of wetting properties in the separator. However, special wetting tests conducted in the labora-

tory show that the wetting behavior of stainless steel plates is always middling (Chatterjee,1998; Schlieper, 2001).

The various reciprocal effects between the plates and the various dispersions result in a flow behavior of the droplets at the plates, which is markedly different. When the dispersed phase wets the plate material sufficiently, a thin trickling film of the previously dispersed phase forms on the plates. Droplets move along the trickling film, finally coalescing with the film. If, in contrast, the wetting of the plates by the dispersed phase is only poor, no continuous trickling film is formed. In this case, the droplets on the plate are driven forward by the flow of following droplets. Because of this, there is increasing coalescence between the droplets in the main direction of flow. This phenomenon thus results in larger droplets than would occur with good wetting of the plates.

A particularly impressive comparison of the separation results with differing wetting properties of the plate material is given by consideration of the dispersions water + n-butanol and water + cyclohexanone. As shown in Figure 11, both dispersion directions (water in organic and organic in water) were measured for these systems. Figure 11 shows the separation lengths at a distance between plates of $a = 5$ mm and $\dot{V}_T = 0.6$ m³/h in a comparison of stainless steel and polypropylene plates. As Figure 11 shows, both dispersions exhibit only marginal differences in separation length for an aqueous dispersed phase ($\alpha < 0.35$). Better plate wetting by the dispersed phase does not directly result in improved separation performance. The difference for different plate materials with an organic dispersed phase is even more notable ($\alpha > 0.45$). Stainless steel plates poorly wet by the organic phase separated the dispersion over an even shorter length.

One possible reason for this is the previously mentioned coalescence and flow behavior. Poor wetting results in larger droplets, which become wedged between the plates, thus increasing flow resistance attributed to the plate package. This makes the dispersion wedge steeper and shorter. However, if a trickling film is formed, the droplets glide over the trickling film through the plate package.

Effects of the distance between plates on separation length in the separator

The next step was to compare how separation length can be reduced by varying the distance between the plates. The bench-
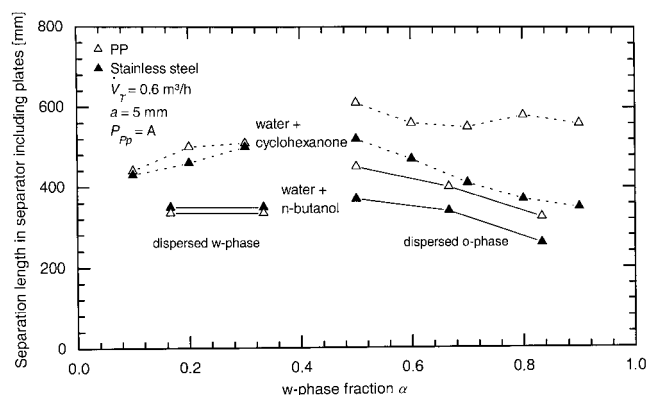

Figure 11. Separation length in the separator with internals for varied plate material.

mark reference is the separation length in the separator with no internals. To provide direct information about the reduction in separation length achieved by using internals, relative separation length is introduced as a ratio of the separation length in the separator with to that without internals. Figure 12 shows the relative separation length of the three dispersions vs. various distances between plates at constant w-phase fraction $\alpha = 0.5$. As Figure 12 shows, the use of plates at a distance $a = 22$ mm already achieves a considerable reduction in separation length compared to a separator with no plates. For the dispersions water + tridecanol and water + cyclohexanone, Figure 12 suggests that a further reduction in distance between plates will produce no direct advantage. However, it must be underlined that the plates not only have a positive effect in separating the dense-packed layer of droplets, but also promote separation of the finest droplets generally present in homogeneous phases. Small distances between plates of $a = 5$ and 10 mm result in a shortening of the sedimentation distances, enabling very small droplets to reach the plates by sedimentation and thus be separated. This effect was especially observed in the dispersion water + cyclohexanone with a low difference in density.

For the water + n-butanol system shown in Figure 12, a reduction in distance between plates always results in a reduction of the corresponding separation length. In the flow rate and w-phase–fraction range investigated, this effect can also be observed in some cases for the two remaining dispersions. However, halving the distance between plates generally achieves only a less than proportional reduction in the corresponding separation length. There are a number of explanations for this. As the distance between plates decreases, the cross-sectional surface available for flow becomes smaller. This reduces the residence time of the dispersion in the plate package. Furthermore, the dispersed phase coalescing at an individual plate cannot be regarded as separated. The dispersed phase always has to reach and coalesce finally (regardless of the distance between plates) at the main interface (see Figure 2). This fact always reduces the positive separation effect resulting from any additional plates installed in the separator.
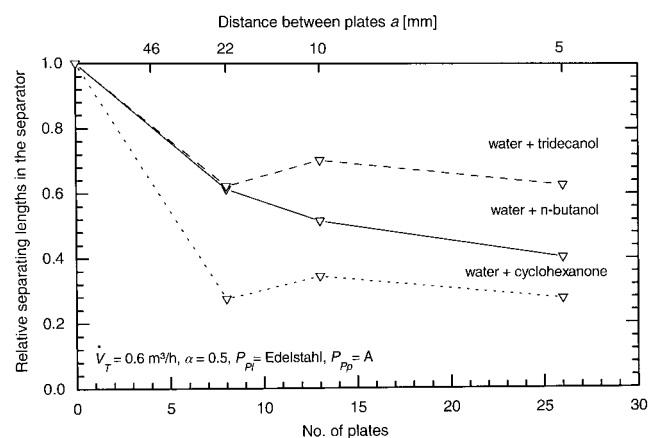

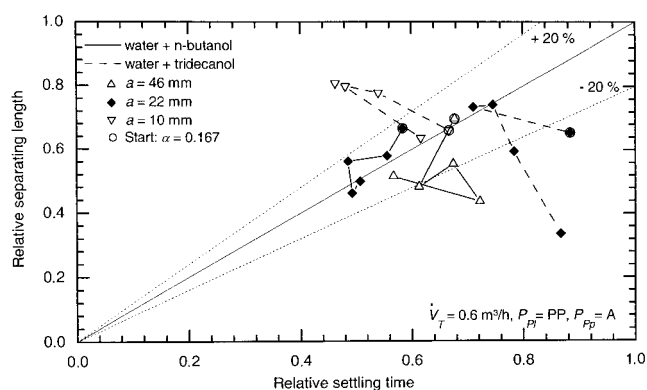**Figure 12. Reduction in separation length by using plates.**

Figure 13. Relationship between relative separation length and relative settling time for various distances between plates and systems.

Pragmatic approach for design of separators including internals

For design of separators with no internals, the settling experiment is successfully applied in calculating the separation length required (Chatterjee, 1998; Schlieper, 2001). In the following we thus investigate the suitability of settling experiments in the design of separators including internals. The starting point for this is constituted by the settling tests carried out in the separator both with and without plates. Just as in definition of relative separation length, the relative settling time is introduced as a ratio of the settling time in the settling experiment with to that without plates. Figure 13 shows the relative settling time vs. the relative separation length for the dispersions water + cyclohexanone and water + tridecanol at various distances between plates. The w-phase fractions α for the various experimental points of a measurement series have the values $\alpha = 0.167, 0.333, 0.5, 0.667,$ and 0.833.

If it is found that the two relative variables are directly proportional (shown by the central diagonal line in Figure 13) in a first approximation. In particular, the water + n-butanol system agrees well with this approximation. The values measured for this dispersion are almost all within $\pm 20\%$ from this simple relationship.

This fact is of significance in relation to a new method for separator design. A settling experiment, with or without internals, can be completed fast and cost-effectively in the laboratory, enabling the corresponding settling time to be determined without using a continuous separator. For calculation of separation length in the separator with no internals, appropriate design models are also available (Golob and Modic, 1977; Henschke, 1995; Müller and Kreichelt, 1979; Sklokin et al., 1988; Stönner, 1981; Stönner and Wöhler, 1975). The direct proportionality between relative settling time and relative separation length allows calculation of separation length in separators including internals

$$L_{s,\, with\, Internals} = \frac{t_{s,\, with\, Internals} L_{s,\, without\, Internals}}{t_{s,\, without\, Internals}} \tag{3}$$

In contrast, the water + tridecanol system, as shown in Figure 13, shows a much greater deviation from proportionality. A possible explanation is the higher viscosity of the organic

phase, which affects flow behavior especially in the separator. However, the assumption of direct proportionality between the relative quantities does not take sufficient account of fluid dynamic effects. This results in deviations for the dispersion of high viscosity.

Summary and Conclusions

This investigation focused on the separation behavior of three liquid–liquid dispersions. The separation experiments were carried out discontinuously and also continuously in the horizontal separator. The results reveal that dispersions with a long settling time also have longer separation length in the separator than that of dispersions with short settling time. This applies for separators both with and without internals.

During investigation of inflow to the plates, buildup of the dispersion before entry into the plate package was achieved by means of perforated plates. Part of the dispersion can be reduced in the dense-packed layer formed in front of the perforated plate. Varying the geometry of the perforated plate can be used to modify the level of dispersion buildup and thus the way the dispersion enters the plates, whereby the distribution of the dispersion and its system properties also affect the situation.

Observation of the properties of the two plate materials when wet by the dispersed phase revealed basic differences, resulting in differences in coalescence at the plates. Poor wetting of the plate material, compared to good wetting, may result in shorter separation lengths. In addition, investigation into the effect of the distance between plates showed that halving the distance produced only a less than proportional reduction in separation length. However, even with a distance between plates of $a = 22$ mm, it was possible to achieve a reduction in separation length of up to 75% compared to that of a separator with no internals. An important factor in the separation process is the size of the dispersed droplets. Droplet size can be specifically affected by the choice of plate material.

In addition to the continuous separator experiments, investigation into discontinuous dispersion separation revealed that settling experiments including internals can be an appropriate engineering aid for separator design.

Acknowledgments

Notation

a = distance between plates (m)
b = distance between settler tube and plates (m)
m = opening ratio
L = length (m)
P = parameter
t = time (s)
$\dot{V}$ = volume flow rate (m³/s)
w = mass fraction

Greek letters

α = phase fraction of the aqueous phase
β = angle of inclination for plates (°)
ε = holdup
η = viscosity (Pa · s)

ρ = density (kg/m³)
σ = surface tension (N/m)
φ = wetting angle between aqueous phase and plate (°)
ϕ = droplet diameter (m)

Subscripts

s = separation
T = total
Pp = perforated plate
org = organic phase
Pl = plates
aq = aqueous phase
32 = mean value according to Sauter

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