Dynamic Response of Gravity Settlers to Changes in Dispersion Throughput

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Recent publications (Dalingaros et al., 1987; Hartland and Jeelani, 1985, 1987; Jeelani, 1985; Jeelani and Hartland, 1985, 1986a, b) have shown how it is possible to design steady state liquid-liquid gravity settlers from experimental data obtained from small-scale unsteady state batch settlers, thus eliminating the need for pilot plant experiments. Unsteady state conditions also occur in continuous settlers due to throughput variations. These include the growth in dispersion height with time during start-up as well as increases and decreases in the steady state height due to changes in dispersion throughput. In extreme cases entrainment and flooding can result. It is important to know the effect of changes in throughput on the dispersion height for the efficient operation and design of steady state settlers.

Hartland and Vohra (1978) derived an equation for the increase in dispersion height with time during the growth of a dense-packed dispersion, assuming constant binary and interfacial coalescence times and basing the drop residence time within the dispersion on the feed-specific dispersed phase throughput. Their equation applies to both plug flow of droplets through the dispersion and well-mixed dispersions. However, for plug flow of droplets in a decaying dispersion their model predicted a linear decrease in dispersion height with time. For well-mixed dispersions the rate of interfacial coalescence increased with time as the drops grew in size, in contrast to experimental observations.

In order to predict the experimentally observed exponential decrease in dispersion height with time it is necessary to allow correctly for the growth of drop size and the effect of gravitational forces acting within the dispersion. This implies a dependence of interfacial coalescence time on the drop size and dispersion height, which is used in this paper to derive improved forms of the equations describing the growth and decay of liquid-liquid dispersions.

Equations are presented for the variation in dispersion height with time during settler start-up and due to changes in dispersion throughput. These have been used to obtain the flow rates of the continuous and dispersed phases leaving the dispersion. Theoretical expressions are also given for the decrease in dispersion height with time during the decay of the steady state dispersions.

Experiments have been carried out on a gravity settler to study the behavior of growing liquid-liquid dispersions in which the variation in dispersion height and the cumulative volumes of continuous and dispersed phases leaving the sedimenting and coalescing interfaces, respectively, are measured as functions of time. The effect of step changes in throughput of the phases is also investigated, the experimental data being used to verify the proposed theoretical models.

Theory

When a liquid-liquid dispersion formed in a mixer is continuously fed into a gravity settler, Figure 1, the height of the dispersion h grows with time t until the steady state dispersion height H is attained. During this period the sedimenting drops grow in size due to binary coalescence before entering the dense-packed zone and finally coalescing with their homophase at the coalescing interface. A dense-packed zone is formed when the volume rate of sedimentation is greater than the volume rate of interfacial coalescence. Initially the height of this zone is practically zero, since the drops take finite time to grow in size through binary coalescence in the sedimentation zone. Thus at any time t the total dispersion height is the sum of the heights of these zones.

Let q_c and q_d be the volumetric flow rates of the continuous and dispersed phases per unit settler cross-sectional area in the dispersion feed. The volume rate per unit area of the continuous phase leaving the sedimentation zone at the sedimenting interface is λ_s, the volume rate of coalescence of drops per unit area at the coalescing interface being ψ_i. The quantities λ_s and ψ_i thus increase with time until the steady state is reached, when $\lambda_s = q_c$ and $\psi_i = q_d$.

If $\bar{\epsilon}$ is the spatial average holdup fraction of the dispersed

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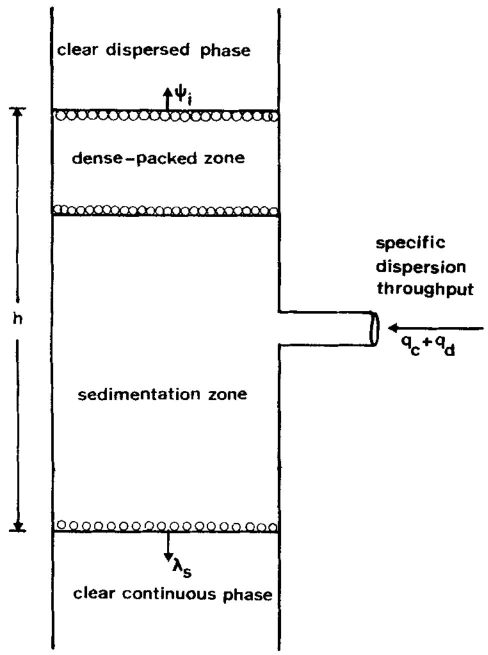

Figure 1. Schematic representation of a growing dispersion.

phase in the dispersion, the rate of increase in volume of the dispersed and continuous phase is given by:

$$\frac{d(\bar{\epsilon}h)}{dt} = q_d - \psi_i \tag{1a}$$

$$\frac{d[(1-\bar{\epsilon})h]}{dt} = q_c - \lambda_s \tag{1b}$$

The variation in total height is thus:

$$\frac{dh}{dt} = q_d + q_c - \psi_i - \lambda_s \tag{1c}$$

which is independent of the dispersed phase holdup.

As explained, the drops grow in size through binary coalescence in the sedimentation and dense-packed zones, finally coalescing with their bulk homophase at the coalescing interface in a growing dispersion. The specific rate of coalescence ψ_i is given by (Jeelani and Hartland, 1985):

$$\psi_i = 2\epsilon_i\phi_i/3\tau_i \tag{2a}$$

where ϵ_i and ϕ_i are the holdup and drop diameter at the coalescing interface and the coalescence time τ_i can be written in terms of the drop size as

$$\tau_i = k_i\phi_i^i \tag{2b}$$

where k_i is a constant depending only on the physical properties of the liquid-liquid system.

Equations 1a and 2a thus become:

$$\frac{d(\bar{\epsilon}h)}{dt} = q_d - \frac{2\epsilon_i}{3k_i}\phi_i^{1-i} \tag{3}$$

in which ϕ_i for a constant binary coalescence time τ_b is given (Hartland and Vohra, 1978) by

$$\phi_i = \phi_o e^{t/6\tau_b} \tag{4a}$$

where ϕ_o is the diameter of drops in the feed dispersion. In a decaying batch dispersion $q_d = 0$ and t is the elapsed time. On the other hand, in a steady state dispersion, $d(\bar{\epsilon}h)/dt = 0$ and $t = \bar{\epsilon}H/q_d$ is the average residence time of drops within the dispersion. In a growing dispersion the residence time is $\bar{\epsilon}h/u$, in which u is the velocity with which the drops move through the dispersion. This may be the sedimentation velocity during the initial growth period when the dense-packed zone is of negligible height and the dispersion is fully turbulent. As the steady state is approached and the thickness of the dense-packed zone becomes appreciable, the velocity u is modified accordingly. However, the magnitude of u always lies between ψ_i and q_d; the value of ψ_i itself approaches q_d as the steady state is reached, and its initial value is finite and greater than zero. The space-time average value of u can thus be written as γq_d, where γ is a fraction close to unity. The drop diameter ϕ_i at the coalescing interface is thus:

$$\phi_i = \phi_o e^{\bar{\epsilon}h/6\gamma\tau_b q_d} \tag{4b}$$

Substituting this in Eq. 3 and integrating with the initial condition $h = H_o$ at $t = 0$ thus gives:

$$h = H - \frac{Tq_d}{\bar{\epsilon}} \ln\{1 + [\exp(\bar{\epsilon}(H - H_o)/Tqd) - 1]e^{-t/T}\} \tag{5a}$$

in which

$$H = \frac{Tq_d}{\bar{\epsilon}} \ln(q_d/\psi_o) \tag{5b}$$

is the final steady state height corresponding to the specific dispersed phase throughput q_d, where

$$T = 6\gamma\tau_b/(1-i) \tag{5c}$$

and

$$\psi_o = 2\epsilon_i\phi_o^{1-i}/3k_i \tag{5d}$$

For positive step changes in q_d, $H_o < H$, and for negative step changes $H_o > H$. When $H_o = 0$, this equation describes the start-up of the settler. When $H_o = 0$, $\gamma = 1$, and $i = 0$, so $k_i = \tau_i$ and Eqs. 5a and 5b reduce to those derived by Hartland and Vohra (1978).

Assuming that the modulus of $\bar{\epsilon}(H - H_o)/Tq_d$ is less than unity, and expanding the corresponding exponential term followed by an expansion of the logarithmic term, Eq. 5a reduces to

$$h = H_o e^{-t/T} + H(1 - e^{-t/T}) \tag{6}$$

which represents the response of a first-order system to a step change in the dispersed phase throughput with time constant T.

Specific flow rates of dispersed and continuous phases leaving the coalescing and sedimenting interfaces

The flow rates ψ_i and λ_s of the dispersed and continuous phases leaving a unit area of the dispersion are defined by Eqs. 1a and 1b, where $\bar{\epsilon}$ is constant and dh/dt is determined by differentiating Eq. 5a and can be independently obtained from the measured cumulative volumes v_d and v_c, since

$$\psi_i = dv_d/dt \tag{7a}$$

$$\lambda_s = dv_c/dt \tag{7b}$$

the total flow rate being given by the sum of these two quantities.

Decay of steady state dispersions

Although Eq. 5a can be used to predict the decrease in dispersion height h with time t for negative step changes in throughput (where $H_o > H$ and q_d is finite), it cannot be used to predict the absolute decay when both q_d and H are zero. An alternative form of equation must therefore be derived to describe the decay of steady state dispersions.

The residence time $t = \bar{\epsilon}h/u$ in Eq. 4a, in which u is the actual dispersed phase velocity and can be written in terms of the actual specific interfacial coalescence rate $\psi_i = \gamma u$, rather than the dispersed phase throughput $q_d = u/\gamma$, since $\psi_i \leqq u \leqq q_d$ and $\gamma \lesssim 1$ [with these assumptions $\gamma = (\psi_i/q_d)^{1/2}$].

Combining Eqs. 2a, 2b, and 4b and expanding with $\bar{\epsilon}\gamma h/6\tau_b\psi_i < 1$, leads to a quadratic equation in ψ_i. Further expansion with $k_i\bar{\epsilon}\gamma(1 - i)h/\epsilon_i\phi_o^{1-i}\tau_b < 1$ yields the linear relationship between ψ_i and dispersion height h:

$$\psi_i = \frac{2\epsilon_i\phi_o^{1-i}}{3k_i} + \frac{\bar{\epsilon}\gamma(1 - i)h}{6\tau_b} \tag{8}$$

Substituting in Eq. 1a and integrating with $h = H_o$ at $t = 0$ gives Eq. 6 with $H = (q_d - \psi_o)T/\bar{\epsilon}$ when $\gamma = 1$. When $q_d = 0$ this reduces to the decay equation

$$h = \left(H_o + \frac{\psi_o T}{\bar{\epsilon}}\right)e^{-t/T} - \frac{\psi_o T}{\bar{\epsilon}} \tag{9}$$

which predicts a finite decay time t_f when $h = 0$.

Experimental Method

Equipment and liquid-liquid system

The equipment involved a mechanically agitated single-stage mixer-settler unit, the experimental arrangement and liquid-liquid system being the same as described elsewhere (Jeelani and Hartland, 1986a), except that the settler was a 200 mm dia. and 500 mm tall QVF glass column with a dispersion inlet port of 27 mm dia. located 150 mm from the base. The continuous and dispersed phases were demineralized water and 50 vol. % *n*-heptane in paraffin oil, respectively.

Dispersion growth experiments

After thoroughly cleaning the experimental equipment (the procedure is explained by Jeelani and Hartland, 1986a), the aqueous and organic storage tanks were respectively charged with demineralized water and 50 vol. % of *n*-heptane in paraffin oil. The speed of the mixer impeller was set at a value greater than the minimum speed required for uniform dispersion ($11\ s^{-1}$). Before performing the actual growth experiments both phases were circulated through the mixer-settler unit until a constant steady state dispersion height at a fixed total throughput was obtained, thereby ensuring mutual saturation of the phases.

At the commencement of each growth experiment the thermostats and pumps were activated and the temperature set at 20 ± 0.2°C. The organic and aqueous phases were then circulated at the desired flow rates, bypassing the mixer-settler unit until the temperatures had attained the set values. The mixer was then charged with volumes of aqueous and organic phases corresponding to the desired phase flow ratio. The circulation loop was then closed, the aqueous and organic feed inlet valves to the mixer simultaneously opened, and the impeller started. The height of the dispersion in the settler was recorded as a function of time until steady state was reached. For this type of run the initial mixing time is zero. A second type of run was also performed under the same conditions except that both phases in the mixer were stirred for 60 min before the aqueous and organic phases were continuously fed to the mixer.

The difference between the two types of experiment can be explained as follows: In the first type the drop size distribution in the mixer, and hence in the feed to the settler, changes with time. In the second type the equilibrium drop size distribution is achieved, so the initial mixing time is considered infinite. Both types of experiment were performed at different throughputs for fixed values of phase flow ratio, impeller speed, and temperature. Additional experiments were also carried out involving a step change in throughput, in which the settler was initially at the steady state. The variation in dispersion height with time was recorded until the new steady state had been reached.

Cumulative volumes of continuous and dispersed phases leaving the sedimenting and coalescing interfaces

In order to measure the cumulative volume of continuous phase v_c leaving the sedimenting interface the settler was first partially filled with the aqueous phase and the initial level recorded. The same procedure used in the growth experiments was repeated, marking the level of the rising sedimenting interface instead of the total dispersion height as a function of time. This was continued until the coalescing interface reached the top of the settler, the inlet feed valves then being closed. The height of the rising sedimenting interface relative to the initial aqueous phase level then represented the cumulative volume of the continuous phase v_c leaving the sedimenting interface per unit area. Similarly, the measurement of the cumulative volume of the dispersed phase v_d leaving the coalescing interface per unit area involved filling the settler with the aqueous phase up to dispersion feed inlet. The feed inlet valves were then opened after the two feed liquids had attained the desired temperature. A time check was made by stopwatch, which was started as soon as the dispersion entered the settler. The levels of the coalescing interface and coalesced dispersed phase were accordingly recorded as a function of time until the latter level reached the top of the settler. The jack leg was then adjusted from time to time in order to keep the dispersion well below the feed inlet in the settler. The difference in levels of the coalescing interface and coalesced dispersed phase represented the cumulative volume of the dispersed phase v_d leaving the coalescing interface per unit area at any time. Both v_c and v_d were measured when the initial drop size distribution in the mixer had reached equilibrium.

Decay of steady state dispersions

When the dispersion height had attained its steady state value, the continuous and dispersed phase feed inlet valves and those at the top and bottom of the settler were simultaneously closed. The dispersion height was then recorded as a function of time until two clear phases had formed. Such decay experiments were performed for different initial steady state heights.

Results and Discussion

Growing dispersions: effect of mixing time

Figure 2 shows two typical increases in dispersion height h with time t measured (symbols) in growth experiments during the start-up of the settler at a specific volumetric dispersed phase throughput q_d of 0.354 mm/s at a feed dispersed phase holdup fraction $\epsilon_F = 0.5$. The circles and triangles correspond to zero and infinite initial mixing times in the mixer. It can be seen that the steady state dispersion height is the same for both cases, but the rate of growth is faster when the initial mixing time is infinite, as expected. For zero initial mixing time the drop size distribution in the mixer, and hence in the feed dispersion to the settler, changes only slowly with time. On the other hand, for an infinite initial mixing time the drop size distribution corresponds closely to its steady state value, leading to a faster rate of dispersion growth since the average drop size is smaller.

Predicted variation in dispersion height with time

It can be seen from Figure 2 that the theoretical variations (solid lines) in the dispersion height h with time t obtained from Eq. 5a with $H_o = 0$ by using the constants H and T, given in Table 1, determined from a least-squares fit of the experimental data, agree well with those measured experimentally (symbols) when the initial mixing time is both zero and infinite.

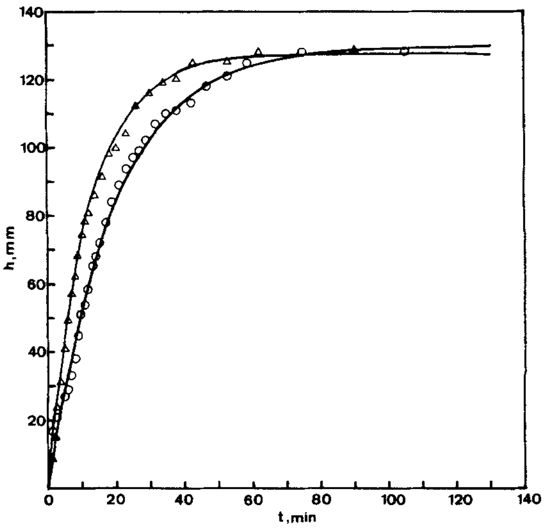

Figure 2. **Experimental growth in dispersion height compared with time for zero and infinite initial mixing times during start-up.**

Table 1 shows that the value of ψ_o calculated from Eq. 5d is almost constant and independent of the dispersed phase throughput. Table 1 also shows that almost identical agreement is obtained by using the first-order response Eq. 6 with $H_o = 0$ with modified constants H and T, determined by nonlinear regression of the experimental data. The parameters in Table 1 are calculated using an average value of $\bar{\epsilon} = 0.55$.

Effect of step changes in throughput

Figure 3 shows the variation in dispersion height h with time t during start-up, followed by positive and negative step changes

Table 1. Experimental Variation in Steady State Dispersion Height H_{exp} with Specific Dispersed Phase Throughput q_d

							First-order Response		
q_d mm/s	Initial Mixing Time s	H_{exp} mm	H mm	T s	Avg. Dev. in h %	ψ_o mm/s	H mm	T s	Avg. δ Dev. in h %
Start-up									
0.283	∞	74	84	1,030	10.2	0.242	85	1,114	10.6
0.310	0	91	94	933	6.1	0.260	94	1,013	6.6
δ	∞	86	89	475	6.4	0.222	91	543	6.9
0.331	0	92	86	678	7.2	0.268	87	732	7.6
δ	∞	96	104	1,069	30.6	0.281	105	1,150	31.3
0.354	0	128	130	1,084	6.7	0.294	131	1,169	7.1
δ	∞	128	127	668	3.7	0.264	128	745	4.0
0.376	0	148	140	692	8.5	0.300	141	982	6.2
δ	∞	144	141	898	6.4	0.299	142	981	8.2
Mean Value	0	—	—	847	7.1	0.281	—	974	6.9
	∞	—	—	828	11.5	0.262	—	907	12.2
Step Change									
0.310 to 0.354	∞	123	126	632	0.9	0.245	126	930	1.0
0.354 to 0.310	∞	85	84	901	0.9	0.285	84	608	0.9

Model parameters H and T during dispersion growth and positive and negative step changes in q_d are obtained from Eq. 5a with $\bar{\epsilon} = 0.55$, ψ_o being calculated from steady state Eq. 5b. Values of H and T corresponding to first-order Eq. 6 are also given.

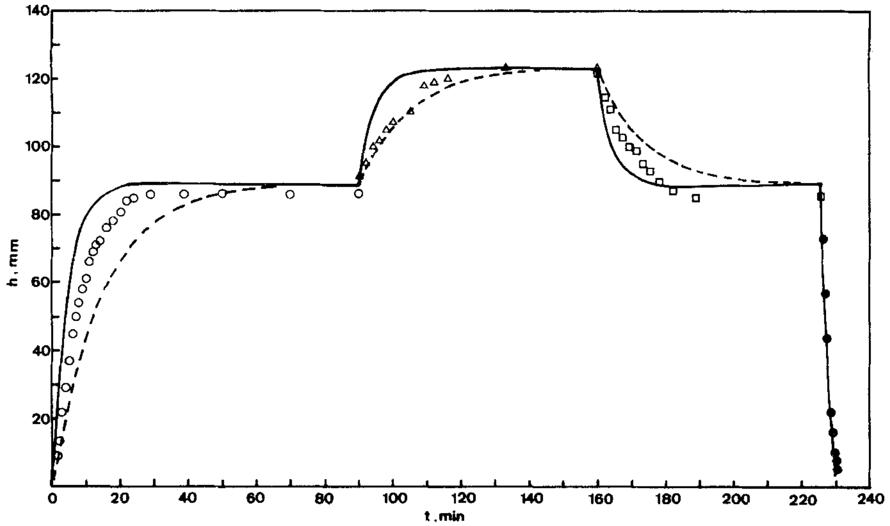

Figure 3. Variation in dispersion height with time during start-up followed by positive and negative step changes compared with theoretical prediction.

in the specific dispersed phase throughput when ϵ_F is constant and equal to 0.5. The initial mixing time was infinite, and during start-up the throughput was 0.31 mm/s (open circles); the positive step change involved an increase in throughput from 0.31 to 0.354 mm/s (open triangles) and the negative step change from 0.354 to 0.31 mm/s (open squares). No hysteresis was observed in the steady state dispersion height corresponding to the specific dispersed phase throughput of 0.31 mm/s before and after the positive and negative step changes. However, the initial rate of decrease in dispersion height in the negative step change is greater than the initial rate of growth in the positive step change, as reflected by the values of T in Table 1. This is due to the fact that the drop size at the coalescing interface (and hence the interfacial coalescence rate) preceding the negative step change is initially larger than that before the positive step change, thus leading to a faster decay of the dispersion.

The experimental variation in the steady state dispersion height H with specific dispersed phase throughput q_d is represented by Eq. 5b, in which $T = 252$ s and $\psi_o = 0.166$ mm/s with an average absolute percentage deviation in H of 5.2 for the runs shown in Table 1 with infinite initial mixing time. The theoretical curves generated from these values of the constants in Eq. 5a are indicated by the solid lines. The broken lines represent the variation predicted from the steady state height H and the average value of $T = 828$ s corresponding to these five runs. Almost identical curves are obtained if Eq. 5a is replaced by the first-order response Eq. 6. It can be seen that for the start-up and the positive and negative step changes in q_d, the steady state dispersion height H is reached sooner with the small time constant $T = 252$ s than with the larger time constant $T = 828$ s. During start-up and following the negative step change the experimental values lie between the two extremes. For the positive step change the experimental values are predicted better by the larger time constant.

Comparison between predicted and experimental phase flow rates leaving the dispersion

Figure 4 shows typical experimental variations in the specific flow rates of the dispersed phase (open squares) and continuous phase (open circles) leaving the coalescing and sedimenting interfaces ψ_i and λ_s with time t obtained during settler start-up for $q_d = 0.31$ mm/s ($\epsilon_F = 0.5$) with infinite initial mixing time. The solid line represents the variation in ψ_i and λ_s predicted by Eqs. 1a and 1b with $\bar{\epsilon} = 0.5$ and dh/dt determined from Eq. 5a with $H_o = 0$, using the values of $T = 475$ s and $H = 89$ mm obtained by the dispersion growth data given in Table 1. The variation in ψ_i and λ_s with $\bar{\epsilon} = 0.55$ lie slightly below and above the line for $\bar{\epsilon} = 0.5$ (the maximum deviation being about 4% when $t = 0$ falls to 0 as t increases). The filled circles show the experimental variation in $\psi_i + \lambda_s$ and the solid line the variation predicted by Eq. 1c. The broken line shows the sum of the predicted variations in dh/dt and ψ_i or λ_s. This represents the maximum flow of dispersed or continuous phase leaving the settler when one interface of the dispersion remains stationary. The movement of the dispersion is controlled by the hydraulics of the system. Should both interfaces move in the same direction, the flow of one phase will exceed the value shown and the flow of the

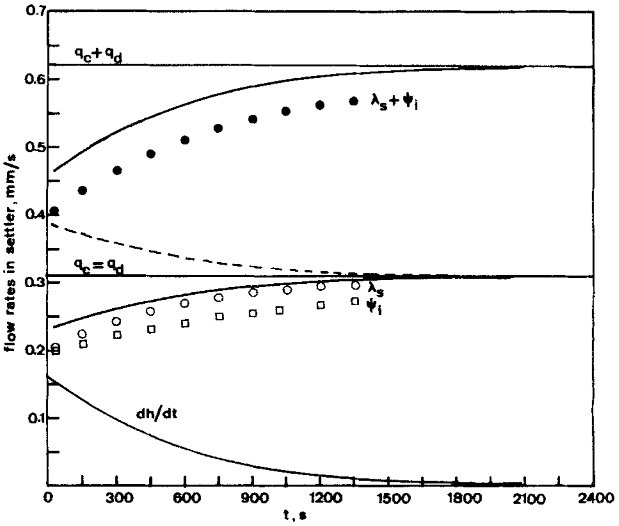

Figure 4. Experimental and predicted settler flow rates.

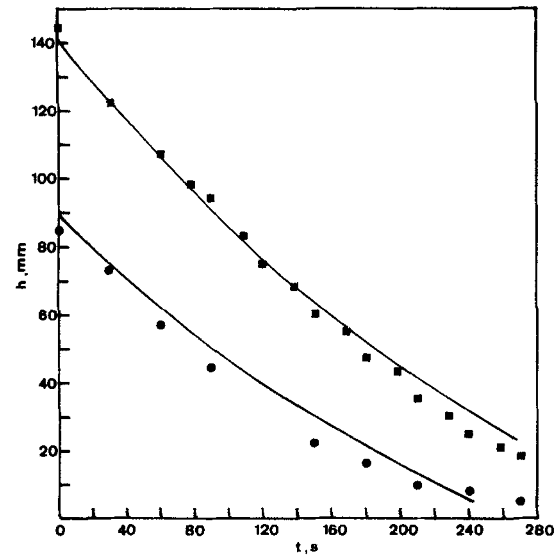

Figure 5. Experimental and predicted decay in dispersion height.

other will be correspondingly diminished. However, in general, the interfaces will move in opposite directions and the flows of the individual phases will be less than indicated. Such variations in phase flow rates during step changes in dispersion throughput could lead to hydraulic instability and phase inversion in the adjacent mixers of a mixer-settler battery.

Decay of steady state dispersions

Figure 3 also shows the experimental decay of a steady state dispersion (filled circles) with initial specific dispersed phase throughput q_d of 0.31 mm/s. The same experimental data (filled circles) are also shown on a larger time scale in Figure 5, together with that for an initial throughput q_d of 0.354 mm/s (filled squares). In both cases the experimental data are well represented by Eq. 9 (solid lines) with the values of $T/\bar{\epsilon} = 458$ s and $\psi_o = 0.166$ mm/s obtained from Eq. 5b using the experimental steady state data. For a decaying dispersion the holdup $\bar{\epsilon}$ increases from the initial value of 0.55 to a final value approaching unity. After rapid sedimentation the entire dispersion is dense-packed, so the space-time average holdup $\bar{\epsilon}$ is assumed to be 0.75, corresponding to $T = 344$ s.

Acknowledgment

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Notation

e = exponential
h = dispersion height in growing and decaying dispersions, mm
H = steady state dispersion height, mm
H_o = initial steady state dispersion height or initial dispersion height in decaying dispersion, mm
i = exponent
k_i = constant, s/mm^i
ln = natural logarithm
q_c, q_d = volume rates of flow of continuous and dispersed phases per unit area of settler in feed dispersion, mm/s
t = elapsed or residence time, s
T = time constant, s
u = velocity of drops in dispersion, mm/s
v_c, v_d = cumulative volumes of continuous and dispersed phases leaving dispersion per unit area in time t, mm

Greek letters

γ = factor accounting for velocity of drops in dispersion
ϵ_F = dispersed phase holdup fraction in feed dispersion
ϵ_i = dispersed phase holdup fraction at coalescing interface
$\bar{\epsilon}$ = average dispersed phase holdup fraction in dispersion
λ_s = volume rate per unit area of continuous phase leaving sedimenting interface, mm/s
τ_b = binary coalescence time, s
τ_i = interfacial coalescence time, s
ϕ_i = drop diameter at coalescing interface, mm
ϕ_o = initial drop diameter, mm
ψ_i = volume rate of interfacial coalescence per unit area at any time, mm/s
ψ_o = volume rate of interfacial coalescence per unit area corresponding to drop diameter ϕ_o, mm/s

Subscripts

b = binary
c = continuous
d = dispersed
i = interfacial
o = initial
s = sedimenting

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