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Experimental Study on the Effect of Parameters on Sedimentation and Coalescing Profiles in Liquid-Liquid Batch Settler

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Abstract

Efficient separation of liquid-liquid mixture based on dispersion is often required in many industrial processes. Influence of mixing time, initial height of dispersion, mixing intensity and oil phase ratio on the separation time in liquid-liquid batch settler/separator were investigated in terms of coalescence and sedimentation profile. Experiments were carried out with oil in water type dispersion. Results showed that in most of cases coalescence controls the time of separation. The experiment results of liquid-liquid separation profiles were compared with the predefined model. MATLAB was used for the fitting of coalescence profile and calculating fitting constants. The data obtained from the batch experiments along with the mathematical model can be used as a good monitoring tool for studying the liquid-liquid dispersion behaviour in industrial units.

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Keywords: Liquid Liquid separation; Oil-water separation; Sedimentation; Coalescing; Dispersion; Batch separation

1. Introduction

Efficient separation of liquid-liquid mixture based on dispersion is often required in many industrial processes such as separation of water from crude oil, separation of dispersion in different settlers for extraction of solvent, separation of dispersion after mass transfer columns[1]. Dispersion of liquid-liquid such as oil in water or vice versa is resulted from the shear and turbulence produced while passing through pressure relief valves, control valves and chokes. Many parameters affects the separation such as mixing intensity[1], physical properties[2], oil phase ratio[3] etc. The influence of parameters on the coalescence and sedimentation of liquid-liquid separation is investigated to

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gain better understanding of the continuous separation device. Gravity separators are based on the difference in densities between two liquid in contact so the best approach is to investigate batch settler because of its low cost and simplicity.

The industrial requirement is to separate both liquids efficiently with minimum entrainment and at small settler size. Generally information obtained from extensive pilot plant experiments is used as the basis for the design of continuous gravity separators. This can also decrease the cost if the information obtained from small and cheap batch experiments can be used for scale up purpose. In order to reduce scale-up expenses, many authors have suggested to relate batch experiments to continuous operation of phase separation [4-12]. Many experimental work related to the liquid-liquid separation based on dispersion is given in the literature [3, 11, 13-17]. Most of the experimental work in previous studies is performed in a very small settler/separator. Separator's diameter also plays an important role in separation but most of the researcher have ignored this fact. Slenderness ratio, which is an important factor in designing of gravity separator was ignored in the previous studies except in the work of Yu and Mao [17]. Very high slenderness ratio is used for studying the separation profile in most of the previous work. In the work of Yu and Mao [17] the slenderness ratio was in considerably acceptable range, but they studied the effect of only two parameters on separation time. In this work, experiments were performed on a liquid-liquid batch separator/settler keeping the slenderness ratio in acceptable range. Effect of four parameters such as mixing time, height of dispersion, mixing intensity and oil phase ratio on separation time is carefully studied. Diesel-water system is used in order to determine the variation of the coalescing and sedimenting interface. The mathematical model given elsewhere[17] for the separation of liquid-liquid dispersion systems was used to verify the experimental data.

Nomenclature

a_{ll}	(m^2/m^3)	interfacial area of liquid-liquid dispersion
H_o	(mm)	initial height of dispersion
h_c	(mm)	height of coalescence interface
h_p	(mm)	height of lower boundary of dense-packed zone
h_s	(mm)	height of sedimentation interface
k_1	(s^{-k_2})	constant
k_2	(-)	constant
k_3	(s^{-k_4})	constant
k_4	(-)	constant
n	(RPM)	mixing Speed
t_i	(s)	time of complete sedimentation
v_o	(mm/sec)	initial sedimentation velocity of drops
v_i	(mm/sec)	sedimentation velocity of drops at t_i
ε_o	(-)	initial oil phase holdup fraction
ϕ	(mm)	average droplet diameter of dispersed

2. Theory

2.1. Phase Separation Characteristics

Schematic diagram of oil/water dispersion in a batch settler is shown in Fig. 1. H_o is the total dispersion height; ε_o is the oil fraction/holdup. When oil density is less than the water, the oil droplets form a dense pack layer of holdup ε_p and the thickness Δh of dense pack layer increases with time as shown in Fig. 1. Coalescing interface height h_c decreases with time t while the height of sedimentation interface increases with time as the drops continue to sediment/rise. When the sedimentation ends at time t_i, the thickness of the dense packed layer reaches the maximum height Δh_i. After this time the drops at the top of the dense packed zone keep coalescing with the oil phase until coalesce interface meets the bottom of the dense packed layer. The separation of oil/water dispersion is considered as complete at time t_f when the dense packed zone disappears.

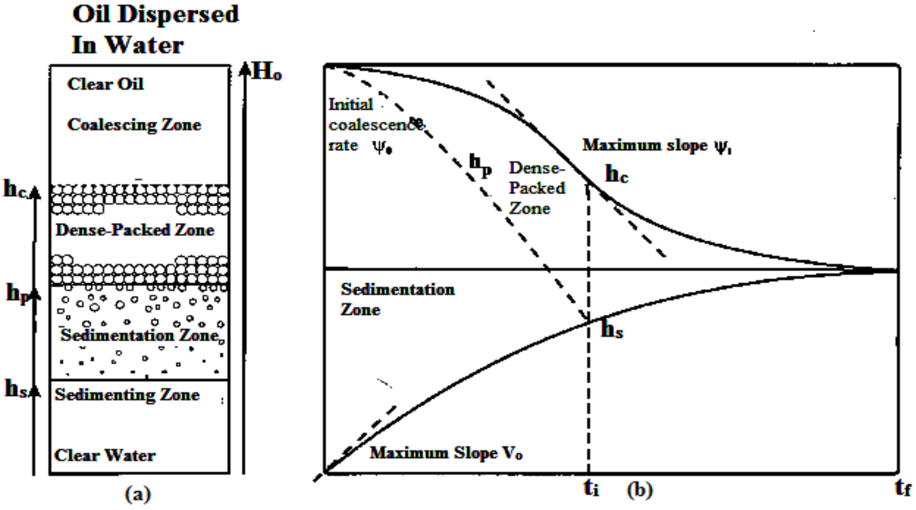

Fig. 1. (a) Heights of the sedimenting and coalescing interfaces (b) Schematic variation in the heights of sedimenting and coalescing interfaces hs and hc with time t for the oil in water dispersion shown in Fig. 1. (a). [15]

2.2. Model for Predicting Profiles

The model developed by Yu and Mao is used for predicting the sedimentation and coalescence profiles. The detailed derivation of the model is given elsewhere[17]. The main equations used for the prediction of profiles are given in following sections.

2.2.1. Coalescence Profile

The Coalescence height with respect to time can be calculated by following equation[17].

$$h_c = H_o(1-\varepsilon_o) + H_o\varepsilon_o(1+k_1 t^{k_2})e^{-k_3 t^{k_4}} \tag{1}$$

Where k_1, k_2, k_3 and k_4 are fitting constants with no physical meanings and can be obtained by fitting the experimental coalescence profile to equation (1)

2.2.2. Sedimentation Profile

According to Jeelani and Hartland (1998) [3] height of sedimentation interface should be evaluated into two intervals: $0 < t < t_i$ and $t > t_i$. The height of sedimentation interface before t_i can be evaluated by using the following equation [3]

$$h_s = v_o t - (v_o - v_i)\frac{t^2}{2t_i} \tag{2}$$

Where v_o and v_i is the initial sedimentation velocity of drops and sedimentation velocity of drops at t_i respectively. The detail for calculating v_o and v_i is given elsewhere [3].The sedimentation profile after t_i can be evaluated by [14].

$$h_p = H_o(1-\varepsilon_o) + \left(1-\frac{1}{\varepsilon_p}\right)H_o\varepsilon_o\left(1+k_1 t^{k_2}\right)e^{-k_3 t^{k_4}} \tag{3}$$

3. Determination of Coalescence and Sedimentation Profile's Parameters

For the oil-water coalescence and sedimentation profile, equation (1) can be used to fit the experimental coalescence profile data to obtain the fitting constants (k_1, k_2, k_3 and k_4) using MATLAB. Then t_i is obtained by substituting the values of fitting constants. When value for t_i and fitting constants are known, the values for h_{ci}, ψi, h_{pi}, v_i and v_o can be calculated by equations given elsewhere[17]. After getting values for all these parameters sedimentation profile can be predicted using equation (2) and (3). The average drop diameter can be calculated by using the following equation [3]. Parameters evaluated from the model are presented in Table 1-4.

$$v_o = \frac{12\mu_w}{0.53\rho_w\phi_o}\left[-1+\sqrt{1+\frac{0.53\rho_w(\rho_w-\rho_o)g\phi^3(1-\varepsilon_o)}{108\mu_w^2\left(1+4.56\varepsilon_o^{0.73}\right)}}\right] \quad (4)$$

The liquid-liquid interface area can be calculated by [17]

$$a_{11} = \frac{6\varepsilon_o}{\phi_o} \quad (5)$$

4. Material and Methods

4.1. Materials and Apparatus

The continuous phase was distilled water and the dispersed phase was diesel. The density of water and diesel was 998 kg/m^3 and 848 kg/m^3 respectively. The viscosity for the dispersed phase was 1mPa-s. The interfacial tension was 52.4 mN/m. A 240 mm internal diameter and 300 mm high flat bottomed cylindrical settler was used to carry out the experiment. The settler was equipped with four wall baffles. Rushton impeller was used for mixing. The baffles and impeller blades were made of stainless steel.

4.2. Experimental Procedure

The vessel was filled with the known volume of diesel and water. The impeller position was set at the required height. The impeller speed was set and mixer was switched on. Both phases were agitated for the selected time. After mixing both phases for the specified time, mixing was stopped and the position for coalescing and sedimentation interfaces with respect to time was recorded with the help of digital camera. The separation is considered complete when a clear immobile interface appears while some drops cling to the wall. The final separation time was recorded. The experiments were performed by varying mixing time, Initial height of dispersion, dispersed phase holdup fractions, and mixing intensity in the mixer.

5. Result and Discussion

The experiments were performed by studying the effect of mixing time, initial height of dispersion, mixing intensity and oil phase ratio. The coalescence and sedimentation height were recorded with respect to time.

5.1. Effect of Mixing Time

The effect of mixing time on the separation of oil-water dispersion is shown in Fig. 2(a). Experiments were performed by changing the time of mixing between 1 and 5 minutes. Other parameters such as Initial height of dispersion, oil phase ratio and speed of mixing were kept constant. Normal mixing speed 450 RPM was selected in order to get the visible influence of the mixing time that might decrease at higher mixing speed. The initial height of dispersion was kept constant at 240 mm. Oil phase fraction was kept at 0.5. Fig. 2(a) demonstrate that with increasing mixing time the separation time increases, this is because increase in mixing time increases the shear and because of high shear the size of oil droplet decreases (see Table 1.). Small droplets take more time to coalescence hence taking longer time to separate. Droplet size decreases with the increase in time of mixing, as predicted by the model (see Table 1). In Fig. 2(a), the agreement between experimental data (symbols) and model prediction (solid line) for sedimentation zone is not so good; this may be due to short mixing time. Model parameters are presented in Table 1.

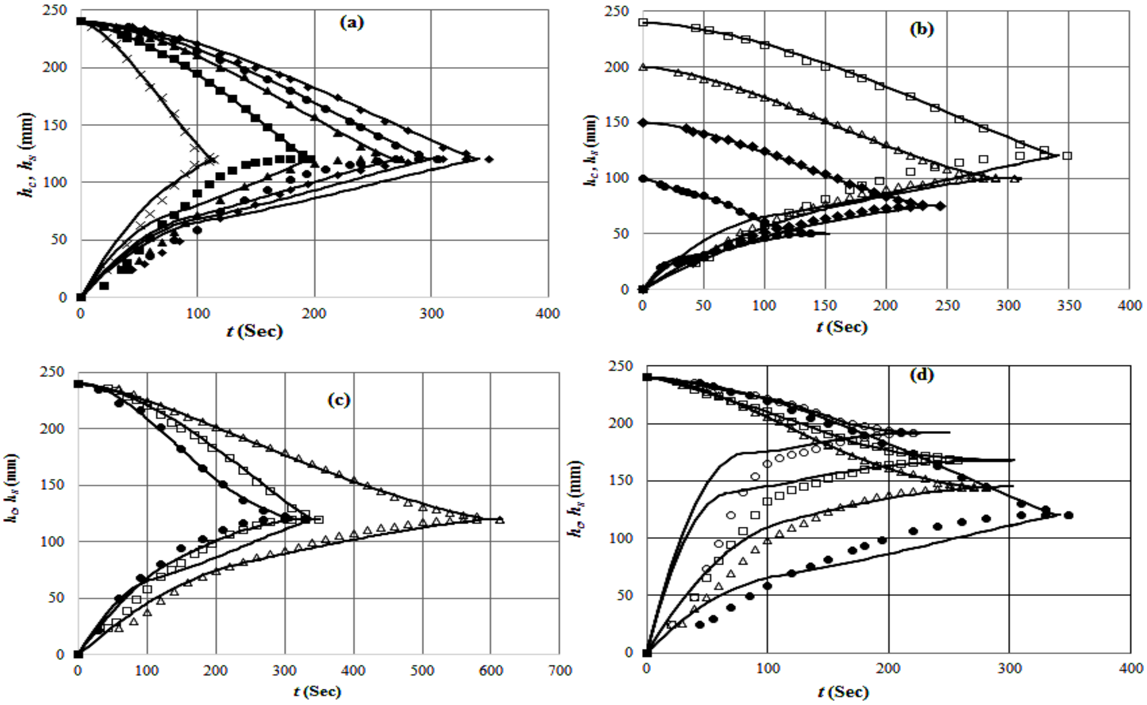

Figure 2 Effect of parameters on Separation profiles, symbols indicates the experimental data points while solid line indicates the model prediction. (a) Effect of mixing time on separation time. Symbol ×, ■, ▲, ● and ♦ indicates 1, 2, 3, 4 and 5 minute mixing time respectively (b) Effect of initial height of dispersion on separation time. Symbols ●, ♦, Δ and ☐ indicates Ho = 100, 150, 200 and 240 mm respectively (c) Effect of mixing intensity on separation time. Symbols •, □ and Δ correspond to n = 300, 450 and 600 mm respectively (d) Effect of oil phase ratio on separation time. Symbols ○, □, Δ and • indicates εo = 0.2, 0.3, 0.4 and 0.5 respectively.

5.2. Initial Height of Dispersion

The effect of an initial height of dispersion on the time required for separation is shown in Fig. 2(b). Mixing time was kept constant at 5 minutes and mixing speed was kept at 450 RPM for all the experiments. Oil phase fraction was kept at 0.5. Impeller was located at the interface for all the experiments. From Fig. 2(b) it can be observed that overall time required for separation strongly depends on the initial height of dispersion. The higher the initial height of dispersion, the longer the time it require for separation. The coalescence profiles are almost parallel; this shows that initial height of dispersion has no effect on coalescing velocity. The sedimentation rate is approximately same for the first 50 second. As the height increases oil droplets have to cover more height to coalescence hence takes more time. Nadiv and Semiat[1] also observed the same trends for different height of dispersion. In Fig. 2(b) the prediction by the model for sedimentation zone is reasonable good for different initial height of sample except for

H_o = 240 mm. Model parameters evaluated for different height of dispersion are given in Table 2 .The average droplet size for 100, 150, 200, and 240mm height of dispersion is 0.336, 0.239, 0.255 and 0.316mm respectively.

Table 1 Model parameter evaluated for different time of mixing

Mixing Time Parameters	1 min	2 min	3 min	4 min	5 min
h_{ci} (mm)	169.3	208.9	213.2	216.6	219.0
h_{pi} (mm)	93.4	72.1	69.8	67.9	66.6
k_1 (sec^{-k2})	-2.6E-08	-1E-07	-2.1E-09	-9.4E-11	-2.3E-10
k_2 (-)	3.702	3.056	3.573	4.053	3.812
k_3 (sec^{-k4})	1.021E-03	9.27E-04	1.79E-04	1.16E-04	8.78E-05
k_4 (-)	1.525	1.284	1.578	1.631	1.635
t_i (sec)	71.1	76.0	93.1	98.0	106.0
Ψ_i (mm/sec)	1.480	0.598	0.454	0.378	0.320
v_i (mm/sec)	0.797	0.322	0.244	0.203	0.172
v_o (mm/sec)	1.83	1.57	1.25	1.18	1.08
ϕ (mm)	0.41	0.382	0.34	0.331	0.31
a_{11}(m^2/m^3)	7263	7853	8823	9063	9493

Table 2. Model parameters evaluated for different initial height of dispersion

Height parameters	100 mm	150 mm	200 mm	240 mm
h_{ci} (mm)	69.9	111.7	150.6	219.0
h_{pi} (mm)	39.2	55.2	72.7	66.6
k_1 (s^{-k2})	4.87E-06	-1.1E-09	-7.8E-10	-2.3E-10
k_2 (-)	3.168	3.788	3.706	3.812
k_3 (s^{-k4})	1.05 E-03	1.99 E-04	1.72 E-04	8.78E-05
k_4 (-)	1.792	1.651	1.621	1.635
t_i (s)	81.0	128.4	150.9	106.0
Ψ_i (mm/sec)	-0.468	0.443	0.476	0.320
v_i (mm/sec)	-0.252	0.238	0.256	0.172
v_o (mm/sec)	1.22	0.62	0.70	1.08
ϕ (mm)	0.336	0.239	0.255	0.316
a_{11}(m^2/m^3)	8928	12552	11764	9493

5.3. Mixing Intensity

Fig. 2(c) shows the influence of mixing intensity on the separation time. The initial height of dispersion was kept constant at 240mm. 300, 450 and 600 RPM were selected to check the influence. 5 minutes time of mixing was selected for all three experimental runs. Oil phase ratio was kept same for all three runs. Fig. 2(c) exhibits that with the increase in mixing intensity the time of overall separation also increases. As mixing intensity increases, high shear reduces the size of the droplet which also increases the time required for separation. It is confirmed from the experiment that coalescence is controlling the separation time because the sedimentation at the beginning moves faster and then slows down, waiting for the coalescence to take place. Yu and Mao[17] also observed the same trend for different mixing intensity, high shear produces small droplets which take more time to coalescence resulting in large time of separation. The sedimentation profile predicted by model is in good agreement for n = 300 and 600 r/min. Model parameters evaluated for different mixing intensities are given in Table 3. The droplet size predicted by this model for n = 300, 450 and 600 r/min is 0.284, 0.316 and 0.222 mm respectively. Table 3. Shows the parameters evaluated from the model.

Table 3. Model parameters evaluated for different mixing intensity

n (r/min) Parameters	300 r/min	450 r/min	600 r/min
ε_o	0.5	0.5	0.5
h_{ci} (mm)	186.6	219.0	191.3
h_{pi} (mm)	84.1	66.6	81.5
k_1 (s^{-k2})	-9.2E-10	-2.3E-10	-6E-11
k_2 (-)	3.613	3.812	3.684
k_3 (s^{-k4})	5.85E-05	8.78E-05	1.67E-04
k_4 (-)	1.841	1.635	1.454
t_i (s)	141.3	106.0	240.7
Ψ_i (mm/sec)	0.586	0.320	0.256
v_i (mm/sec)	0.315	0.172	0.138
v_o (mm/sec)	0.874	1.08	0.53
ϕ (mm)	0.284	0.316	0.222
a_{11}(m^2/m^3)	10563	9493	13513

5.4. Oil Phase Ratio

The influence of oil phase ratio on separation time is shown in Fig. 2(d). 20%, 30%, 40% and 50% oil phase ratios were selected. Other parameters were kept constant. The initial height was kept at 240 mm, mixing speed was kept at 450 rpm. Mixing time of 5 minutes was given to each experimental run. With the increase in oil phase ratio, time of separation also increases. From Fig. 2(d), it can be seen that coalescence control the time of separation, sedimentation is faster in the initial stage up to first 100 sec then it decreases, awaiting for the coalescence to occur. Same kind of trend for different oil phase ratio was observed by Jeelani and Hartland[3]. A Different behaviour was observed by Henschke [18] that coalescence rate varies while sedimentation rate was independent of the phase ratio. This difference could be because of different solvent used as an organic phase. The change in oil phase ratio generally cause change in droplet size because at generally low oil phase ratio small droplets are formed.

The sedimentation profile predicted by the model (solid line) is not in good agreement with the experimental data as shown in Fig. 2(d). The model used in this work is not able to predict accurately the sedimentation profiles for different oil phase ratio as also shown in [17] for the experimental data of Nadiv and Semiat[1]. The model parameters evaluated for different oil ratio is presented in tabulated form in Table 4.

Table 4. Model parameters evaluated for different oil phase ratios

ε_o Parameters	0.2	0.3	0.4	0.5
h_{ci} (mm)	225.7	221.3	199.7	219.0
h_{pi} (mm)	173.8	139.3	113.9	66.6
k_1 (s^{-k2})	1.73E-08	3.85E-06	-8.8E-09	-2.3E-10
k_2 (-)	3.907	2.656	3.306	3.812
k_3 (s^{-k4})	2.99E-05	2.76E-04	1.79E-04	8.78E-05
k_4 (-)	2.305	1.804	1.679	1.635
t_i (s)	81.4	66.90	111.5	106.0
Ψ_i (mm/sec)	-0.06	0.160	0.52	0.32
v_i (mm/sec)	-0.03	0.086	0.28	0.172
v_o (mm/sec)	4.29	4.07	1.76	1.084
ϕ (mm)	0.405	0.463	0.384	0.316
a_{11}(m^2/m^3)	2962	3887	6250	9493

6. Conclusion

The final time of separation increases with the increase in mixing time, initial height of dispersion, mixing intensity and oil phase ratio. In most of the experimental runs coalescence controls the time of separation. Experimental Batch data and mathematical model can be used for studying the separation behaviour of the industrial units. The overall time of separation predicted by the model is not accurate. A new model is required which can accurately predict the final time of separation. Modification is required in the model for accurate prediction of sedimentation and coalescence profiles, as the prediction by the model in this work is not that accurate.

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