Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

Analysis of a Single-Stage Mixer-Settler Performance

Abu Bakr S. H. Salem[a]
[a] CHEMICAL ENGINEERING DEPARTMENT, KING FAHD UNIVERSITY OF PETROLEUM & MINERALS, DHAHRAN, SAUDIA ARABIA

To cite this Article Salem, Abu Bakr S. H.(1993) 'Analysis of a Single-Stage Mixer-Settler Performance', Separation Science and Technology, 28: 7, 1479 — 1500

To link to this Article: DOI: 10.1080/01496399308018052

URL: http://dx.doi.org/10.1080/01496399308018052

SEPARATION SCIENCE AND TECHNOLOGY, **28**(7), pp. 1479–1500, 1993

Analysis of a Single-Stage Mixer–Settler Performance

ABU BAKR S. H. SALEM
CHEMICAL ENGINEERING DEPARTMENT
KING FAHD UNIVERSITY OF PETROLEUM & MINERALS
DHAHRAN 31261, SAUDIA ARABIA

ABSTRACT

A new design of a single-stage mixer–settler is presented. The mixer, a vertical cylindrical vessel 5.5 cm in diameter and 18.0 cm long, is used for mixing the phases in its lower part. Complete solute transfer occurs in the mixer since coalescence of the dispersed phase droplets takes place on their way up via the mixer outlet to the settler. The settler, a vertical tower 3.4 cm in diameter and 40.0 cm long, serves only as a flash separator. The contactor has been tested by the toluene–acetic acid–water system in both directions of mass transfer. The contactor operates as an ideal stage when tested with the acid system. The hydrodynamics and the dynamic behavior of the contactor have been studied. A model approach has been adopted for the mixer. Agreement between the model output data and the experimental results was excellent.

INTRODUCTION

Contactors of different types have been developed for liquid–liquid extraction, and the selection among them can involve many parameters. These include the stages required, flow rates, floor space available, residence times, type of solvent used, scale-up reliability, capital, and economic factors. Logsdail and Lowes (1) presented an excellent review on this subject. Robbins (2) proposed a decision network based on the criterion that the least complicated contactor which will perform the extraction with low maintenance is preferred for industrial use.

Mixer–settlers are usually preferred over tower extractors, with or without mechanical agitation in cases of long residence times and limited headroom space. Shaw and Long (3) reported that mixer–settlers exert a high agitation intensity which results in an increase of interfacial area and hence in mass transfer rates.

However, mixer–settlers are usually arranged in a horizontal pattern, and they therefore require a large floor space, a large inventory of liquids, independent agitation for each stage, and interstage pumping (4). In addition, multiple pumps and mixer shafts increase the likelihood of fugitive emissions. Nevertheless, they are still considered one of the most efficient contactors for industrial applications.

In the past decade, considerable effort has been expended on improving the design of mixer–settlers, and many new units that attempt to reduce the space requirement and to increase the throughput per unit volume without introducing interstage pumping have been reported.

Horvath and Hartland (4) introduced a mixer–settler extraction column which has the advantage of having both mixer–settlers and column contactors. The column can contain a number of stages placed vertically one above the other. A typical stage contains one mixer and a settler. The stirrers of the mixers are attached to one common shaft along the axis of the column. Stage efficiencies up to 170% have been obtained at 375 rpm agitation speed and 2.1 minutes residence time per stage. They reported that this was due to repeated complete coalescence and redispersion in each stage and also to the large interfacial area produced by the intensive mixing combined with low backmixing in the column.

The object of the present work was to develop a contactor that combines the advantages of columns with those of mixer–settlers. The proposed contactor is simply a single-stage mixer–settler in which the mixer has to perform essentially two functions, mixing the phases and then coalescing the dispersed-phase droplets. The settler in this case may serve as a separating vessel, and it can be a vertical tower with a relatively small size compared to the mixer. Conventionally, the settler is usually larger than the mixer (5). In a previous investigation, Salem (6) introduced a single-stage unit in which the mixer consisted of two sections, a horizontal section which receives the feed and solvent phases and an integral vertical section of similar size which provides a suitable space for the dispersed-phase drops to coalesce during their passage upward via the outlet tube to the settler. However, it was observed that the fractional heavy phase holdup in the mixer was comparatively larger than that of the light phase. This was attributed to the inability of the agitator to help the heavy phase move up to the outlet tube of the mixer. Such phenomena may lead to instability in the contactor and may delay the attainment of steady-state conditions.

Therefore, a vertical single section mixer has been built for the present investigation. The hydrodynamics of this mixer have been studied by using two different systems. Extraction tests have been performed using the water–acetic acid–toluene system in both directions of mass transfer. The dynamic behavior of the single-stage contactor has been also studied.

The mixer was modeled by the number of transfer units approach. Agreement between the data obtained from the model and the experimental results was excellent.

EXPERIMENTAL

Contactor Used

A simplified flow diagram of the system used is shown in Fig. 1. Technical data of this system are given in Table 1.

Saturated light and heavy liquid phases are fed from two ground level tanks by centrifugal pumps through flowmeters to the mixer where they are agitated. The dispersion is transferred to the vertical settler where it is allowed to separate into two phases. The outlet phases are then collected in glass drums before being transferred to receivers through two outlet flowmeters. The valve leading to the heavy-phase drum serves as a means for controlling the interface. Samples are taken from the outlet tube of the mixer and from the sample points of the drums. This mixer consists of a single vertical compartment in which the light phase is introduced at the bottom and the heavy phase is fed through a valve in the middle of the compartment (7.5 cm from the top). The agitator is a stainless steel shaft provided at its end with two turbine blades. The agitator blades are located at the bottom of the mixer so agitation will take place only around the inlet of the light phase. This may assist in achieving a high mixing intensity between this phase and the heavy phase which may be forced downward by the fast movement. It was also expected that after the mixing process in the bottom section is performed, the incoming light phase may help in pushing the heavy phase up to the outlet tube at the top of the mixer. In other words, this single-section mixer may be expected to achieve the two functions of a double section mixer, i.e., mixing at its bottom and coalescence of the dispersed-phase droplets on their way up to the outlet tube of the settler. A Teflon ring and an oil seal were fitted to the shaft through the top cover of the mixer to prevent liquid leakage. The dimensions of this mixer are included in Table 1.

The settler in most mixer–settler designs is a horizontal cylinder. The main functions of such settlers are the coalescence of the dispersion coming from the mixer and then separation of the phases. Rocha et al. (7) reported that almost 30% mass transfer can take place in the settler. Therefore, in designing horizontal settlers, the dimensions and location of the coalescence wedge depend on which phase is to be dispersed (8). For that reason, control of the interface in horizontal settlers is usually not easy. Such settlers are characterized by a relatively larger capacity than the mixer, which means a large holding inventory of liquids. However, in this work

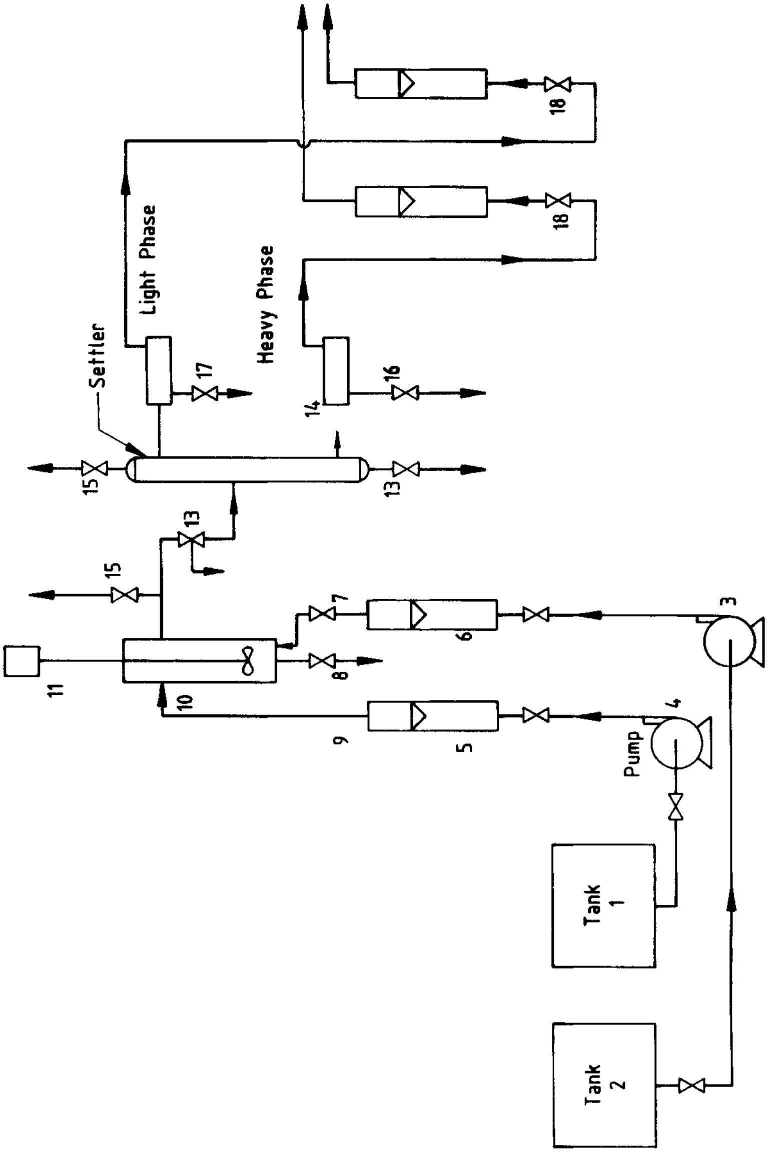


FIG. 1 Flow diagram for the extraction system.

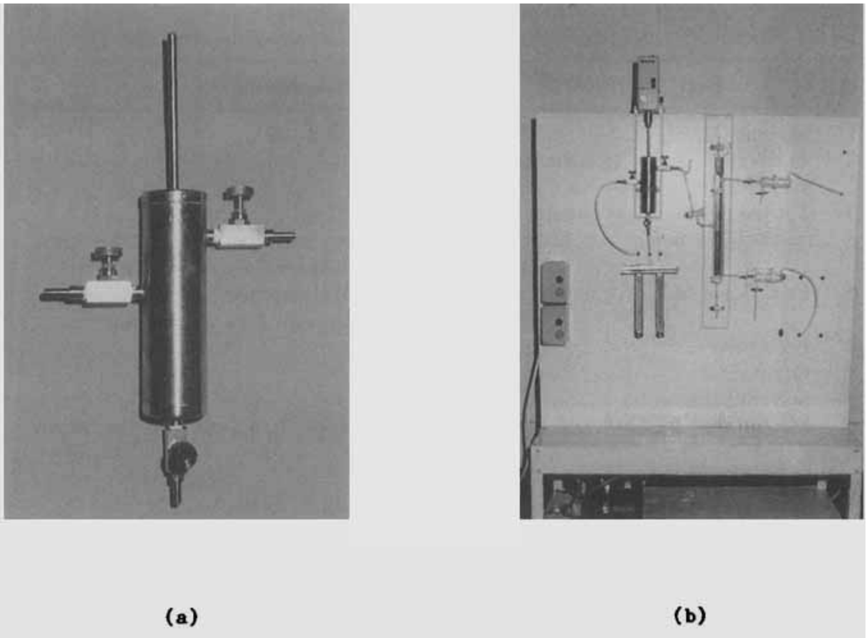
TABLE 1
Technical Data of the Developed Single-Stage Mixer–Settler (see Fig. 1)

No.	Item	Technical data
1	Solvent tank	Stainless steel drum, capacity 20 L
2	Feed tank	Same as 1
3	Feed pump	Centrifugal, ⅙ hp
4	Solvent pump	Same as 3
5	Solvent flowmeter	Scale reading: 0–150. Float material: 316 SS. Maximum flow: 250 mL/min. Minimum flow: 8.5 mL/min
6	Feed flowmeter	Scale reading: 0–150. Float material: 316 SS. Maximum flow: 304 mL/min. Minimum flow: 13 mL/min
7	Feed valve	316 SS.
8	Drain valve	Same as 7
9	Solvent valve	Same as 7
10	Mixer	Single vertical section of 316 SS, 5.5 cm diameter, 18 cm high, 310 mL capacity
11	Agitator	Speed range: Speed 1: 45–300 rpm Speed 2: 300–1200 rpm Regulation of speed by a control knob with a scale. Shaft: Hollow shaft, inside diameter 10.2 mm. With 2 turbine stirrer blades, 1 cm × 1.5 cm each
12	Settler	Cylindrical glass tube of 3.4 cm diameter, 40 cm high, 363 mL capacity
13	Discharge valve	Glass, lined with Teflon, also used for sampling
14	Interface control valve	Same as 13
15	Vent	Same as 13
16	Heavy-phase drum	Glass cylinder of 3.4 cm diameter, 10 cm length, 100 mL capacity
17	Light-phase drum	Same as 16.
18	Outlet flowmeters	Same as 5 and 6.

the settler was designed for phase separation only because coalescence and solute transfer were expected to be completed in the mixer compartment. Therefore, the settler size was greatly reduced in order to minimize the holdup of liquids in this equipment. The settler was merely a vertical glass tube of 3.4 cm diameter and 40 cm in length. Control of the interface in such settlers is simple and practical. This was done through the valve leading to the heavy-phase receiving drum as shown in Fig. 1. Figure 2 shows the mixer and settler used.

Materials Used

The chemicals used in this study were acetic acid, distilled water, kerosene, and toluene. Some physical properties of these chemicals are given in Table 2, including some measured data.



(a) (b)

FIG. 2 (a) The single section mixer. (b) The vertical settler.

The equilibrium data of the water–acetic acid–toluene system were obtained from Rahma (9) and are shown in Table 3.

Typical Run

The saturated feed solutions were made up before. All adjustments to the operating conditions were made during the early stages of the run.

Feed flowmeter calibrations were checked by a measuring cylinder and a stopwatch. The phases were fed to the mixer at specified flow rates until

TABLE 2
Physical Data of the Materials Used

Component	Formula	Molecular weight	Boiling point (°C)	Density (g/mL)	Viscosity (cP)	Refractive Index
Acetic acid	CH_3COOH	60	118.1	1.0440	1.1631	1.3759
Distilled water	H_2O	18	100	0.997	0.9054	1.3350
Kerosene	—	—	—	0.779	1.0633	1.4410
Toluene	$C_6H_5CH_3$	92	110.8	0.8670	0.5555	1.4960

TABLE 3
Equilibrium Data of the Water–Acetic Acid–Toluene System at 25°C [Rahma (9)]

Water phase		Toluene phase	
R.I.	Solute (wt%)	R.I.	Solute (wt%)
1.3450	16.10	1.4948	1.00
1.3500	23.58	1.4937	1.80
1.3524	27.00	1.4932	2.30
1.3570	34.00	1.4912	3.80
1.3615	41.38	1.4890	5.60
1.3644	46.00	1.4872	7.50
1.3670	50.40	1.4855	8.50
1.3692	54.60	1.4838	10.20
1.3780	57.80	1.4817	11.50

it was approximately half full. At that point the agitator was started. The speed of agitation was kept at 1200 rpm throughout runs. This was the maximum speed of the agitator motor. This speed was checked from time to time by a speedometer. When the dispersion was transferred to the settler, the interface was controlled by the valve leading to the heavy-phase drum. Initially the contactor was run for holdup measurements without mass transfer. Extraction runs were then followed. The holdup of the heavy phase was estimated in the mixer, settler, and stage, and compared to that in the feed liquids in each run. Different runs were performed at different phase ratios. These parameters have been estimated by the following relations:

$$R = (V\rho)_H/(V\rho)_L \tag{1}$$

$$\phi_{HF} = \frac{(V\rho)_{HF}}{(V\rho)_{HF} + (V\rho)_{LF}} \tag{2}$$

$$\phi_{HM} = \frac{(v\rho)_{HM}}{(v\rho)_{HM} + (v\rho)_{LM}} \tag{3}$$

where R is the phase ratio (heavy phase flow rate divided by light phase flow rate, g/g); V is the volumetric flow rate (mL/min); v is the volume (mL); ρ is the density (g/mL); ϕ_{HF} and ϕ_{HM} are the heavy-phase holdup in the feed and mixer, respectively (mass fraction); and L and H stand for the light and heavy phases, respectively. The fractional holdup of the heavy phase in the settler, ϕ_{HS}, was estimated by an equation similar to Eq. (3)

used for the mixer. The fractional holdup of the heavy phase in the whole stage, ϕ_{HTS}, was estimated by the following equation:

$$\phi_{HTS} = \frac{(v\rho)_{HM} + (v\rho)_{HS}}{(v\rho)_{HM} + (v\rho)_{LM} + (v\rho)_{HS} + (v\rho)_{LS}} \tag{4}$$

The residence time, τ, of the liquids in the stage was estimated by the following equation:

$$\tau = \frac{v_M + v_S}{Q_T} \tag{5}$$

where Q_T is the total input flow rate (mL/min).

For holdup measurements the contactor was tested with water–kerosene and water–toluene. During the subsequent mass transfer runs, the holdup was also measured. Measurement of the holdup was achieved by discharging the contents of the mixer and the settler into separate vessels at the end of the run, and the phases were then separated and their quantities measured. In mass transfer runs, samples from these phases were taken instantaneously for concentration, density, and viscosity measurements. During these runs the solute concentrations in both streams leaving the settler were monitored from time to time to check for steady-state conditions. Samples from the outlet of the mixer were also taken and separated quickly in order to measure the solute concentrations in both phases. Calibration curves giving the relationship between certain solute concentrations in both phases and their respective refractive indexes were determined in a previous stage at 25°C. A refractometer, calibrated beforehand for the system phases, was used for measuring the refractive indexes of the phases. The densities of the pure and liquid phases were measured by a digital density meter DMA35 (Anton Par K.G.). A Cannon–Ubbelohde viscometer (Cannon Instrument Co.) was used to measure the viscosities of pure liquids and mixtures of liquids.

RESULTS AND DISCUSSION

Hydrodynamics without Mass Transfer

The results of the hydrodynamic measurements are given in Table 4. Table 4(i) contains the data obtained when the water–kerosene system was used for testing the contactor. The residence time of the liquids in the system was kept around 4 minutes throughout. Different phase ratios were used, and the corresponding fractional holdup of the heavy phase in the feed, mixer, settler, and stage were estimated by using Eqs. (2), (3), and (4). Table 4(ii) shows the results obtained when the contactor was tested with the water–toluene system. This table also includes the values of the

TABLE 4
Holdup in the Contactor at 1200 rpm

	Flow rate (mL/min)			Mass fraction			
No.	V_L	V_H	$R = \frac{(V\rho)_H}{(V\rho)_L}$	ϕ_{HF}	ϕ_{HM}	ϕ_{HS}	ϕ_{HTS}
			(i) Water–Kerosene System				
1	145.0	15.0	0.133	0.117	0.047	0.328	0.219
2	130.0	30.0	0.267	0.228	0.160	0.603	0.439
3	100.0	60.0	0.770	0.435	0.678	0.576	0.617
4	90.0	70.0	0.998	0.500	0.660	0.597	0.623
5	70.0	90.0	1.650	0.623	0.701	0.571	0.624
6	62.0	98.0	2.029	0.670	0.691	0.543	0.604
7	38.0	122.0	4.121	0.805	0.698	0.603	0.644
8	22.0	138.0	8.052	0.890	0.736	0.551	0.627
			(ii) Water–Toluene System				
1	145.0	15.0	0.119	0.106	0.022	0.198	0.168
2	130.0	30.0	0.266	0.210	0.082	0.483	0.357
3	100.0	60.0	0.692	0.409	0.309	0.519	0.759
4	90.0	70.0	0.897	0.473	0.438	0.497	0.716
5	70.0	90.0	1.483	0.597	0.676	0.554	0.788
6	38.0	122.0	3.703	0.787	0.692	0.478	0.574
7	22.0	138.0	7.235	0.879		Flooding	

parameters discussed in Table 4(i). Flooding was observed for water–toluene system runs at phase ratios larger than 7. In this range the light liquid was entrained in the heavy liquid coming out of the settler. Therefore, the phase ratios in the subsequent extraction runs were kept below 4. This allowed for smooth operation without wasting chemicals.

Figure 3 shows a comparison between the trend of ϕ_{HF} and ϕ_{HM} over the values of the phase ratios in the water–toluene test runs. This figure indicates that at lower values of heavy phase inputs, the values of ϕ_{HF} are greater than those of ϕ_{HM}. At values of R in the range from 1 to 3, the values of ϕ_{HM} and ϕ_{HF} are approximately the same. At higher values of R, the values of ϕ_{HM} are relatively lower than those of ϕ_{HF} which are in the range of acceptable operation.

The water–kerosene system was found to behave similarly in this mixer.

Figure 4 shows a comparison between the ratio of (ϕ_{HM}/ϕ_{HF}) for the two systems studied at different phase ratios. The figure shows that at any value of R for the water–toluene system, the ratio of the heavy phase holdup in the mixer to that in the feed was generally less than 1. This means that a single sectioned mixer is suitable for this system and for systems of a similar

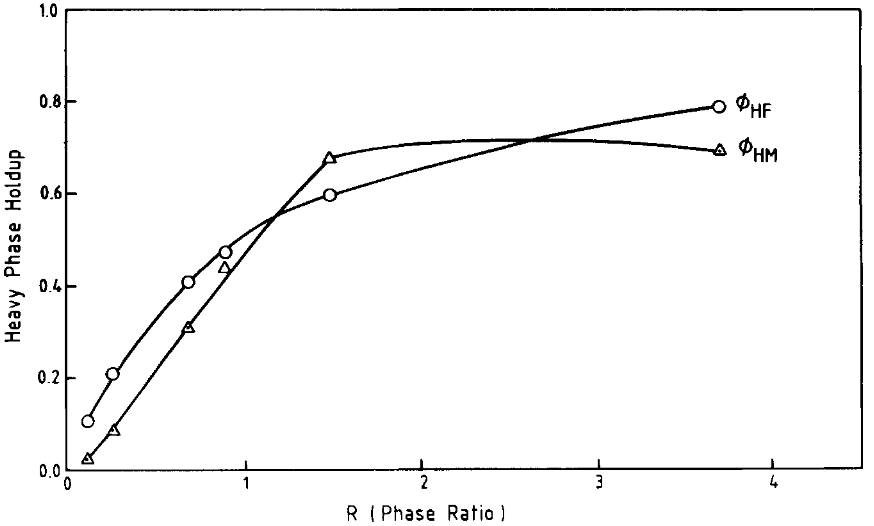


FIG. 3 Effect of the phase ratio on the heavy phase holdup in the mixer using the water–toluene system at 1200 rpm.

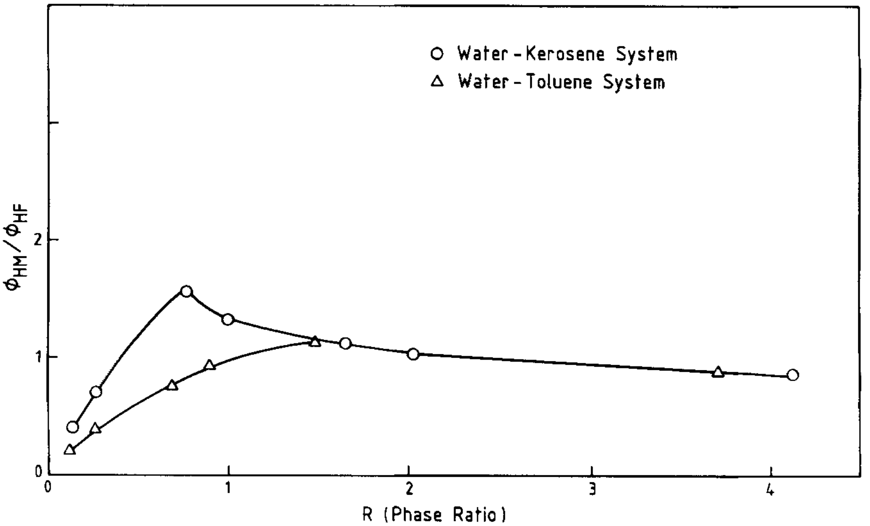


FIG. 4 Effect of the phase ratio on the heavy phase holdup ratio in the mixer for two systems at 1200 rpm.

nature. For systems similar to water–kerosene, no accumulation of the heavy phase in the mixer will be encountered at R values greater than 2.

Extraction Runs

Extraction runs at different phase ratios were carried out using the toluene–acetic acid–water system. Both directions of mass transfer were investigated. Typical results are given in Table 5. This table includes the operating conditions data: flow rates, solute concentrations, densities and

TABLE 5
Extraction of Acetic Acid from Toluene by Water (toluene dispersed)

(a) Operating Conditions

Item	Flow rate (mL/min)	R.I.	Solute (wt%)	ρ (g/mL)	Flow rate (g/min)	μ (cP)	Mass phase ratio	Mass fraction	Speed (rpm)
Feed	39	1.4788	14.50	0.8816	34.38	0.5693	1.00	0.23	1200
Solute	112	1.3350	0.00	1.000	112.00	0.9050	3.26	0.77	

Item	Mixer	Settler	Stage
Operating capacity (mL)	310	395	705
Residence time (min)	2.05	2.62	4.67

(b) Results: Basis: Mass Percent

	Mixer output				Settler output			
	Raffinate		Extract		Raffinate		Extract	
Time (min)	R.I.	x (%)	R.I.	y (%)	R.I.	x (%)	R.I.	y (%)
8	1.4953	0.60	1.3375	4.50	1.4955	0.45	1.3378	4.75
10	1.4955	0.45	1.3377	4.70	1.4955	0.45	1.3377	4.70
12	1.4955	0.45	1.3375	4.50	1.4955	0.45	1.3375	4.50
15	1.4955	0.45	1.3378	4.75	1.4955	0.45	1.3380	4.80

Mixer holdup

Light phase					Heavy phase					
V	ρ	μ	R.I.	x (%)	V	ρ	μ	R.I.	y (%)	ϕ_{HM}
85	0.8622	0.5549	1.4955	0.45	225	1.0029	0.9746	1.3380	4.80	0.75

viscosities of the feed solutions. Operating capacities and residence times in the mixer, settler, and stage are also included. For the system studied, it can be seen that steady-state conditions have been attained in the contactor after a time equivalent to four times the residence time of the liquid in the mixer has elapsed. Table 5 shows that the output streams from the settler were identical to those leaving the mixer. This indicates that almost complete mass transfer has occurred in the mixer.

The results of other runs are summarized in Tables 6(i) and 6(ii). These tables include the input mass flow rates, the phase ratio (S/F), the input and output concentrations x and y, the equilibrium concentration of the raffinate phase x^* corresponding to the output extract phase y, and the distribution coefficient m. The value of the extraction factor, α, for each run is also included, where

$$\alpha = \frac{Sm'}{F(1 - x_0)} \tag{6}$$

where m' is the distribution coefficient on a solute-free basis. The Murphree efficiency of the raffinate phase, E, was estimated and is included in the table, where

$$E = \frac{x_0 - x}{x_0 - x^*} \cdot 100 \tag{7}$$

TABLE 6

Results of Extraction Runs: (i) Water–Acetic Acid–Toluene System

Input flow			Mass percent							
F (toluene) (g/min)	S (water) (g/min)	S/F (g/g)	x_0	x	y	x^*	m	α	E (%)	PR
(*a*) *Acid Transfer from Toluene to Water Phase* (*toluene dispersed in the first two runs—water dispersed in the other runs*)										
34.4	112.0	3.260	14.5	0.25	4.4	0.31	14.2	56.42	100.4	98.27
46.7	98.0	2.100	14.5	0.40	6.5	0.46	14.2	37.08	100.2	97.24
79.1	70.0	0.885	14.5	0.85	12.8	0.95	13.5	15.87	100.7	94.14
97.9	48.9	0.500	14.5	2.25	26.5	2.70	9.8	7.57	100.2	84.5
(*b*) *Acid Transfer from Water to Toluene Phase* (*toluene dispersed*)										
10.3	125.1	12.10	26.5	12.50	0.85	14.0	0.0607	0.0871	112	52.83
16.5	120.8	7.30	26.5	16.50	1.20	18.3	0.0660	0.0546	117.6	37.74
32.9	110.5	3.36	26.5	20.55	1.55	21.3	0.0730	0.0267	114.2	22.45

The percentage recovery, PR, was also estimated and is included where

$$PR = \frac{x_0 - x}{x_0} \cdot 100 \tag{8}$$

However, the values obtained may be compared with the values at $\alpha = 1$ at which a minimum solvent-to-feed ratio is required to remove all of a specific component from the feed solution.

Contactor Efficiency

The Murphree efficiencies of the extraction runs in both mass transfer directions are plotted against the phase ratio R in Fig. 5. The figure shows that the contactor is equivalent to an ideal stage in the range of phase ratios used, i.e., from 0.5 to 3.5 for acid transfer from the organic to the water phase as indicated by Curve 1. For the other direction of mass transfer, represented by Curve 2, the range of operation was limited to phase ratios less than 0.5 since the distribution coefficient in this direction is very small. The heavy phase (feed) in this case was kept very low in order to be able to detect the concentration of the solute in the extracting light phase, which should be more than seven times the feed phase for a sizable extraction. The efficiency of the unit was found to be more than 100%.

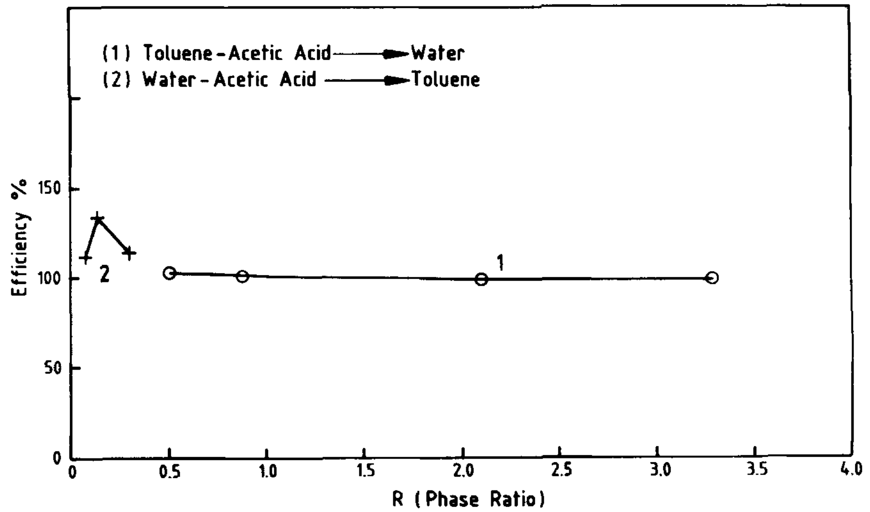


FIG. 5 Effect of the phase ratio on the Murphree efficiency of the contactor.

It can be seen that values of Murphree efficiencies of more than 100% were obtained in the case of acid transfer from the water to the toluene phase as shown in Table 6. Horvath and Hartland (4) reported a similar trend where 170% efficiency was obtained for the transfer of acetone from the water to the toluene phase compared with 100% for the solute transfer in the reverse direction. They also reported that other workers have observed similar trends. They explained that this may be due to changes in physical properties of the liquid systems. However, it can be said in general that this trend may be observed when a solute transfers from a certain phase to another of a lower density and viscosity. Diffusion in the receiving phase may be faster due to a possible reduction in resistance to solute transfer. This may take place due to the different drop size distribution obtained in this direction of transfer. Possible changes in the interfacial area and interfacial tension may also lead to an increase in the rate of mass transfer in that direction.

Trends similar to those shown in Fig. 5 can be observed if the percentage solute recovery obtained for the systems studied are plotted against the phase ratio.

Hydrodynamics with Mass Transfer

Figure 6 shows the effect of increasing the heavy phase input ratio, *R*, on the heavy phase holdup ratio (ϕ_{HM}/ϕ_{HF}) for the system investigated

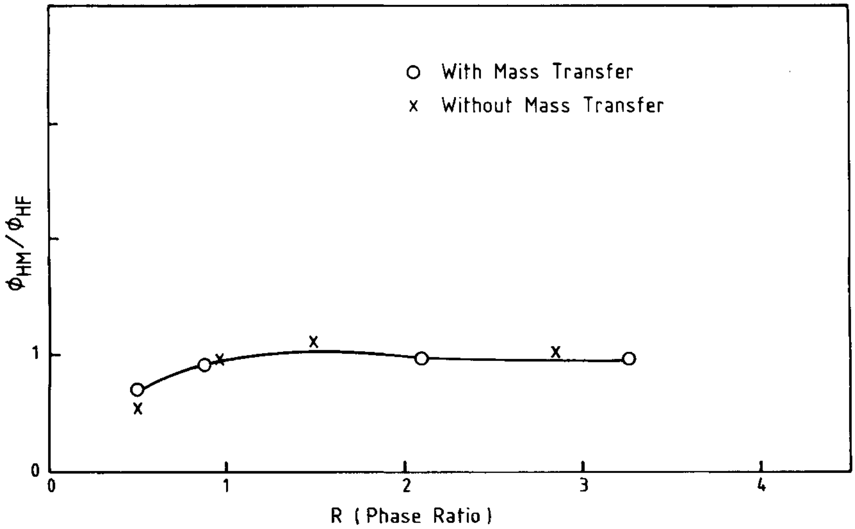


FIG. 6 Effect of the phase ratio on the heavy phase holdup ratio for the system used with and without mass transfer at 1200 rpm.

with and without mass transfer between the phases. The figure indicates that mass transfer has very little effect on the trend. Therefore, it is possible to use the system without performing mass transfer tests to predict the hydrodynamic behavior of the contactor.

Dynamic Behavior of the Mixer

Steiner and Hartland (10) indicated that most evidence suggests that the mixers which must be well stirred in order to achieve efficient mass transfer can be modeled as perfectly mixed tanks.

For the mixer, the dynamic component balance equation can be represented by

$$H\frac{dy}{dt} + h\frac{dx}{dt} = F(x_0 - x) + S(y_0 - y) \tag{9}$$

where H and h are the mass holdup of the phases in the mixer. When the equilibrium condition is satisfied, $y = mx$, Eq. (9) can be rearranged and rewritten as

$$\frac{dx}{dt} = \frac{(\phi_F) + (\phi_S m)}{(\tau_F + \tau_S m)}\left[\frac{\phi_F x_0}{\phi_F + \Phi_S m} - x\right] \tag{10}$$

where ϕ_F and ϕ_S are the feed and solvent fractions in the input feeds,

$$\phi_F = \frac{F}{F + S}; \qquad \phi_S = \frac{S}{F + S} \tag{11}$$

and τ_F and τ_S are the residence times of the feed and solvent phases in the mixer (minutes),

$$\tau_F = \frac{h}{F + S}; \qquad \tau_S = \frac{H}{F + S} \tag{12}$$

When Eq. (10) is solved using the boundary conditions at $t = 0$ and $x = 0$:

$$t = \left(\frac{\phi_F + m\tau_S}{\phi_F + m\phi_S}\right) \ln \frac{1}{1 - \dfrac{x}{x_0}\left(1 + \dfrac{m\phi_S}{\phi_F}\right)} \tag{13}$$

Equation (13) includes the parameters involved in the behavior of the mixer.

This equation has been used to find the time required for the mixer to reach steady-state, assuming that the phases in the mixer are perfectly mixed.

TABLE 7
Dynamic Behavior of the Mixer

Input flow					
F (toluene) (g/min)	S (water) (g/min)	$R = S/F$ (g/g)	S_M/S_F (g/g)	τ_M (min)	τ_{ss}/τ_m
34.4	112.0	3.260	0.946	2.05	1.65
46.7	98.0	2.100	0.930	1.94	1.97
79.1	70.0	0.885	0.850	1.95	1.32
97.9	48.9	0.500	0.700	1.91	1.98

Table 7 shows the results obtained when some runs were used to test this model. The table includes values of the input flow rates, phase ratio, ratio of the solvent fractional holdup in the mixer to that in the feed (a parameter which is not included in the model, on the assumption that for perfectly mixed phases this parameter should be less or equal to 1), and the liquid residence time in the mixer. The last column in the table shows the ratio of the time required for the mixer to attain steady state, τ_{SS}, and the actual residence time of the liquids in the mixer, τ_m.

For the acetic acid system, the ratio of (τ_{ss}/τ_m) is of the order of 2, which seems to be reasonable according to previous findings (11).

Mathematical Model

Modeling of the mixer in the present work has followed the approach adopted by Treybal (5). This approach depends upon the following steps. The Murphree dispersed phase efficiency, E, can be estimated by

$$E = \frac{N_0}{N_0 + 1} \tag{14}$$

where N_0 is the overall number of transfer units in the dispersed phase, which can be estimated by

$$N_0 = Z/H_0 \tag{15}$$

where Z is the height of the mixer (m). H_0 is the overall height of the transfer unit of the dispersed phase (m). H_0 can be determined by

$$H_0 = V_D/K_Da \tag{16}$$

where V_D is the dispersed phase flow rate (m/s), K_D is the overall mass transfer coefficient based on the dispersed phase resistance (m/s), and a is the interfacial area (m^2/m^3).

The value of K_D can be estimated from the two film theory equation:

$$\frac{1}{K_D} = \frac{1}{k_D} + \frac{1}{mk_C} \tag{17}$$

where k_D and k_C are the mass transfer coefficients of the dispersed and continuous phases, respectively, and m is the distribution coefficient of the solute transferred from the dispersed to the continuous phases.

k_C can be determined from the Sherwood number of the continuous phase, $(\mathrm{Sh})_C$, as follows:

$$k_C = (\mathrm{Sh})_C \cdot \frac{D_C}{d_p} \tag{18}$$

where D_C is the diffusivity of the solute in the continuous phase (m^2/s), and d_p is the drop size (m).

The Sherwood number can be estimated by the following correlation:

$$(\mathrm{Sh})_C = 2 + 0.47 f_1^{0.17} \cdot f_2^{0.36} \cdot f_3^{0.62} \tag{19}$$

$$f_1 = \frac{d_i}{T}; \qquad f_2 = \left(\frac{\mu}{\rho D}\right)_C \tag{20}$$

$$f_3 = d_p^{4/3} \cdot \left(\frac{P}{V_{LM}}\right)^{1/3} \cdot \left(\frac{\rho}{\mu}\right)_C^{2/3} \tag{21}$$

where d_i is the impeller diameter (m), T is the vessel diameter (m), V_{LM} is the liquid contents in the mixer (m^3), and P is the agitator power output (W).

The power of the agitator is estimated by

$$P = P_0 \rho_M N^3 d_i^5 \tag{22}$$

where ρ_M is the density of the mixture of liquids in the mixer (kg/m^3):

$$\rho_M = (\phi\rho)_C + (\phi\rho)_D \tag{23}$$

N is the speed of agitation (rps), and P_0 is the power number of the agitator. The value of P_0 can be obtained from certain correlations that depend upon the Reynolds number of the impeller, which is estimated by

$$\mathrm{Re} = \frac{\rho_M d_i^2 N}{\mu_M} \tag{24}$$

where μ_M is the viscosity of the liquids in the mixer which depends upon which of the phases is dispersed (5).

Once the power output is estimated, the drop size of the dispersed phase can be determined by

$$d_p = 10^{a_0} b_1^{0.0473} b_2^{-0.204} b_3^{0.14} \tag{25}$$

where

$$a_0 = -2.066 + 0.732\phi_D \tag{26}$$

$$b_1 = \left(\frac{\mu}{\rho}\right)_C; \qquad b_2 = \frac{P}{(V_{LM}\rho_M)} \tag{27}$$

$$b_3 = \left(\frac{\sigma}{\rho}\right)_C \tag{28}$$

where σ is the interfacial tension between the phases (N/m). The interfacial area, a, can then be determined from

$$a = 6\phi_D/d_p \tag{29}$$

The previous steps have to be followed to determine the Schmidt number and the continuous phase transfer coefficient.

The mass transfer coefficient for the dispersed phase, k_D, can be estimated from

$$k_D = -\frac{d_p}{6\tau} \ln\left[\frac{3}{8}\sum_{1}^{\infty} B_n^2 \exp\left(-\frac{64\lambda_n D_D \tau}{d_p^2}\right)\right] \tag{30}$$

where τ is the residence time in the mixing vessel (seconds), B_n and λ_n are eigenvalues in circulating drops obtained from Table 10.1 of Treybal (5) according to the value of the Sherwood number for the continuous phase determined before, and D_D is the solute diffusivity in the dispersed phase (m^2/s).

This approach was used to estimate the efficiency of the contactor when it was tested with the acid system. Only one experimental run data were used. The input data used are given in Table 8. The physical properties of the phases are also included in the table. The diffusivities of the acid in the water and toluene phases were taken from Liley et al. (12). The interfacial tension, σ, for the water–toluene phase was taken from Salem (6). The calculation results are given in Table 8. It was found that the efficiency obtained for the contactor was in excellent agreement with the experimental result for the toluene–acetic acid–water system.

TABLE 8
Results of the Mathematical Model of the Mixer

(i) Operating Conditions

System		V_C (mL/min)	V_D (mL/min)	V_L (mL)	ϕ_D (fraction)	N (rps)	T (cm)	D (cm)	Z (cm)	τ (min)	x_0 (%)	x (%)
D phase	C phase											
Toluene	Acetic acid–water	112	39	310	0.23	20	5.5	2.2	18.0	2.05	14.5	0.25

(ii) Physical Properties

System		ρ_C (kg/L)	ρ_D (kg/L)	$\mu_C \cdot 10^{-3}$ (kg/ms)	$\mu_D \cdot 10^{-3}$ (kg/ms)	$D_C \cdot 10^{-9}$ (m^2/s)	$D_D \cdot 10^{-9}$ (m^2/s)	m_{C-D}	σ (N/m)
D phase	C phase								
Toluene	Acetic acid–water	1002.9	862.2	0.9746	0.5549	2.26	1.19	14.200	0.018

(iii) Calculation Results

System		Re	P (W)	$d_p \cdot 10^{-3}$	$(\mathrm{Sh})_C$	$K_C \times 10^{-5}$	$K_D \times 10^{-5}$	$K_D \times 10^{-5}$	H_0 (m)	N_0	E (%)
D phase	C phase										
Toluene	Acetic acid–water	4946	0.08	0.85	25.2	6.7	1.62	1.59	2.5×10^{-5}	7143	99.98

CONCLUSION

A single-stage mixer–settler of high efficiency has been developed. The mixer in this contactor is designed to achieve the two main requirements of any extraction process: intensive mixing of the phases in the lower part of the mixer and subsequent coalescence of the dispersed phase through its top section. In this way, complete mass transfer may occur in the mixer. The settler in such designs can be of very small size since it is used only for separating the phases. Such a contactor allows for larger throughputs to be handled with a minimum inventory of liquids.

Data analysis using the perfect mixing model resulted in excellent agreement between the experimental results and the model data. The hydrodynamics and the dynamic response analysis of the experimental results gave evidence of the stability of the contactor operation and its flexibility for similar extraction applications.

Heating and cooling facilities can be easily fitted to this extractor, which allows for the investigation of the effects of temperature profiles during extraction. Horizontal or vertical arrangements of multistage units of this type can be easily constructed. A laboratory-size contactor of this type may be useful for evaluating the solvents used in aromatics recovery from raffinate. The solvents used for this purpose are usually heavier than water.

NOMENCLATURE

a	interfacial area (m^2/m^3)
a_0	parameter defined by Eq. (26)
b_1, b_2, b_3	parameters in Eq. (25) defined by Eqs. (27) and (28)
B_n	parameters in Eq. (30) defined as eigenvalues
C	continuous phase
d_i	impeller diameter (m)
d_p	drop size (m)
D	diffusivity (m^2/s); dispersed phase
E	Murphree efficiency of the raffinate phase
f_1, f_2, f_3	parameters in Eq. (19), defined by Eqs. (20) and (21)
F	feed flow rate (mL/min, g/min)
h	holdup of the feed phase in the mixer (g)
H	holdup of the solvent phase in the mixer (g); heavy phase
H_0	overall height of transfer unit (m)
k_C, k_D	mass transfer coefficients of the continuous and dispersed phases, respectively (m/s)
K_D	overall mass transfer coefficient of the dispersed phase (m/s)
L	light phase; liquid

m	distribution ratio; mixer
m'	distribution ratio, solute-free basis
M	mixer
N	speed of agitation (rpm; rps)
N_0	overall number of transfer units
P	agitator power output (W)
P_0	power number (dimensionless)
PR	percentage recovery
Q_T	total input flow rate (mL/min)
rpm	revolutions per minute
rps	revolutions per second
R	phase ratio (g/g); raffinate flow rate (g/min)
Re	Reynolds number
R.I.	refractive index
S	settler; solvent flow rate (g/min); solvent; separation factor
Sh	Sherwood number
SS	stainless steel
T	tank diameter (m)
V	volume flow rate (m^3/s); velocity (m/s)
v	volume (mL)
x, y	raffinate and extract phase concentration (mass fraction)
x_0, y_0	initial concentration in the feed and solvent phases (mass fraction)
x^*	raffinate concentration in equilibrium with a certain extract phase concentration (mass fraction)
Z	height of the mixer (m)
α	extraction factor
λ_n	parameters in Eq. (30) defined as eigenvalues
σ	interfacial tension (N/m)
μ	viscosity (cP; $N{\cdot}s/m^2$; kg/ms)
ρ	density (g/mL; kg/m^3)
ϕ	fractional holdup (mass fraction)
τ	residence time (min; s)

ACKNOWLEDGMENT

The experimental part of this work was performed by Hassan M. Ahmed in his final year project.

REFERENCES

1. D. H. Logsdail and L. Lowes, "Industrial Contacting Equipment," in *Recent Advances in Liquid–Liquid Extraction* (C. Hanson, Ed.), McGraw-Hill, New York, 1971, p. 142.

2. I. A. Robbins, 'Liquid–Liquid Extraction," in *Handbook of Separation Techniques for Chemical Engineers* (P. A. Schweitzer, Ed.), McGraw-Hill, New York, 1979, pp. 1–268.
3. K. G. Shaw and R. S. Long, "Solvent Extraction of Uranium," *Chem. Eng.*, *64*, 251–256 (1957).
4. M. Horvath and S. Hartland, "Mixer–Settler Extraction Column: Mass Transfer Efficiency and Entrainment," *Ind. Eng. Chem., Process Des. Dev.*, *24*, 1220–1225 (1985).
5. R. E. Treybal, "Liquid Extraction," in *Mass Transfer Operations*, McGraw-Hill, Auckland, 1986, pp. 521–526.
6. A. S. Salem, "Extraction in a Single-Stage Mixer–Settler," *Ind. Eng. Chem. Res.*, *30*, 1582–1588 (1991).
7. J. A. Rocha, J. L. Humphrey, and J. R. Fair, "Mass Transfer Efficiency of Sieve Tray Extractors," *Ind. Eng. Chem., Process Des. Dev.*, *25*, 862–871 (1986).
8. G. V. Jeffreys and G. A. Davies, "Coalescence of Liquid Droplets and Liquid Dispersion," in *Recent Advances in Liquid–Liquid Extraction* (C. Hanson, Ed.), Pergamon Press, Oxford, 1975, pp. 544–555.
9. I. A. Rahma, *Developing a Single-Stage Mixer–Settler*, Chemical Engineering Department, Qatar University, Doha, Qatar, 1990.
10. L. Steiner and S. Hartland, "Dynamic Characteristics of Solvent Extraction Contactors," in *Handbook of Solvent Extraction* (T. C. Lo, M. H. I. Baird, and C. Hanson, Eds.), Wiley-Interscience, New York, 1983, p. 864.
11. A. B. Salem and M. A. Sheirah, "Dynamic Behavior of Mixer–Settlers," *Can. J. Chem. Eng.*, *66*, 867–875 (1990).
12. P. E. Liley, R. C. Reid, and E. Buck, "Physical and Chemical Data," in *Perry's Chemical Engineers Handbook*, 6th ed. (R. H. Perry and D. Green, Eds.), McGraw-Hill, New York, 1984.

Received by editor June 8, 1992