Mass Transfer Studies in Perforated-Plate Extraction Towers

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EARLIER WORK (*1, 4, 5, 6, 9, 15, 16*) on perforated-plate extraction columns has been concerned primarily with the evaluation of the column operating variables such as flow rates, nature of the continuous phase, plate spacing and plate geometry. Some information is also available in the literature on the wetting characteristics in these columns (*5, 6, 9, 16*). Experimental work has not, however, been reported where the effects of the geometry factors such as hole diameter, % hole area, plate spacing, and column diameter; and of the operating factors such as flow rates, flow rate ratio, physical properties of the system, choice of the continuous phase, and direction of mass transfer, have been considered together. The present investigation is a part of work on these design factors that affect extraction efficiency in perforated-plate columns; the variables studied are flow rates, flow rate ratio, inlet acid concentration in the dispersed solvent, and direction of extraction, while carrying out the extraction of acetic acid from dispersed methyl isobutyl carbinol-(4-methyl,-2-pentanol) to a continuous phase of water. To standardize the designed extraction apparatus, the system toluene–benzoic acid–water was used, and a few runs were obtained, while extracting benzoic acid from dispersed toluene to continuous water.

APPARATUS

The geometric factors of hole size, per cent hole area, and plate spacing were fixed in the present study at what seemed to be an optimum level based on the literature information (*10–12, 15–17*) by selecting ⅛-inch thick brass plates of 3 inches in diameter with 95 of ⅛-inch holes, giving a free area of 16.7%. The plate spacing was held constant at 6 inches. Each of the perforated plates, designed in accordance with the recommendations of the previous workers (*7, 8, 17*) carries a downcomer, which is a ½-inch I.D. copper tubing of 4½ inches in length. The column itself was 3-1/16 inches I.D., borosilicate glass tubing, with 6-inch diameter disengaging sections at top and bottom, and the assembly of the 8 plates was supported at 6-inch plate spacing inside the glass column by means of a central rod of brass of ¼-inch diameter and ¼-inch I.D. brass spacer sleeves. The experimental setup of the extraction apparatus was similar to that of Rao and Rao (*14*) except for the change of column from packed to perforated plates.

MATERIAL

Toluene. Nitration grade toluene, supplied by Amritlal Bhurabhai & Co., Bombay, was used. B.P. = 110.5° C., sp. gr. (D^{30}_{30}) = 0.859, and refractive index at 30° C. = 1.4860.

Methyl Isobutyl Carbinol. The technical grade (Burmah-Shell Co.) was used. B.P. = 131.0 to 131.5° C., sp. gr. = 0.8017, and refractive index at 30° C. = 1.4080.

Benzoic Acid. Reagent grade (May & Baker) benzoic acid was used and its purity by chemical analysis was 99.8%.

Acetic Acid. Reagent grade (E. Merck), 99.5% pure, was used.

The equilibrium distribution data were determined at 30° C. for both toluene–benzoic acid–water and methyl isobutyl carbinol–acetic acid–water (*13*) systems (Table I).

Table I. Equilibrium Distribution Data at 30° C.

No.	c_W	c_S[a]
(Distribution of Benzoic Acid between Toluene and Water)		
1	0.000209	0.00072
2	0.000407	0.00204
3	0.000568	0.00430
4	0.000736	0.00745
5	0.000855	0.00905
6	0.001028	0.01282
(Distribution of Acetic Acid between M.I.B.C, and Water)		
1	0.003063	0.003063
2	0.005714	0.005405
3	0.009307	0.008776
4	0.011480	0.010660
5	0.017380	0.015430
6	0.025390	0.021380

[a] In lb. moles per cu. ft. of acid.

EXPERIMENTAL PROCEDURE

Before operation was started, the liquids in the feed drums were mutually saturated. This is particularly necessary when the solvent and water have any appreciable mutual solubilities, as in water and methyl isobutyl carbinol. The continuous phase (water) was admitted at a predetermined rate until the column was about three-fourths full; and dispersed phase (the solvent solution having the desired solute concentration) was then slowly introduced. The position of the two-phase interface was maintained at 2 inches above the top edge of the glass column in the top section by regulating the control valve in the exit water line, and the flows were set and held at the desired rates. The effective extraction height was 59 inches in the present work. As preliminary experiments have shown that the steady state conditions were established when the column contents were displaced four times, the column was operated for 10 minutes more, before samples (250 cc. each) of exit water, exit solvent, and inlet solvent were taken out for analysis. The acid content of an aliquot portion of these samples were determined by titration with standard NaOH using phenolphthalein as an indicator. In all runs, water formed the continuous phase, and solvent, the dispersed phase.

All runs were taken at ambient room temperature, which varied from 29° to 32° C. in the course of the experimental work. This slight variation of temperature was found to have a negligible effect on the equilibrium curves of the two systems studied. The range of flow rates investigated was from about 8 to 70 ft. per hr. Table II, giving the summary of over-all mass transfer data, includes the ranges of the different variables studied.

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Table II. Summary of Over-all Mass Transfer Data

($Z = 4.92$ ft.)

Toluene–Benzoic Acid–Water System
Dispersed phase toluene, direction of extraction toluene → water

System	Direction of Extn.	Inlet Acid Concn., Lb. Moles./Cu. Ft. Soln.	Range of Phase Flow Rates Cu. Ft./Hr. Sq. Ft.		No. of Runs	
			Solvent	Water	Taken	Rejected
Toluene-Benzoic Acid-Water Water: Continuous Phase Toluene: Dispersed phase	Toluene → Water	0.010	8.1–69.6	23.3–67.6	15	. . .
M.I.B.C.-Acetic Acid-Water Water: Continuous phase M.I.B.C: Dispersed phase	M.I.B.C. → Water	0.005	10.1–30.1	11.0–43.6	13	1
	M.I.B.C. → Water	0.010	11.1–37.4	11.9–40.2	12	. . .
	M.I.B.C. → Water	0.015	10.2–43.6	10.3–45.2	14	2
	Water → M.I.B.C.	0.010	11.4–44.3	24.6 (av.)	6	. . .

CALCULATION OF MASS TRANSFER

The mass transfer results were expressed in terms of over-all transfer coefficients (K_Wa), over-all transfer units (H.T.U.$_{OW}$), H.E.T.S. and stage efficiency E_A. The following relationships were used:

$$K_Wa = (N/\theta)_{av}/V\Delta C_{W1m} = (N/S\theta)_{av}/Z\Delta C_{W1m} = (N'_{av})/Z\Delta C_{W1m} \qquad (1)$$

$$\text{H.T.U.}_{OW} = V_W/K_Wa \qquad (2)$$

$$\text{H.T.U.}_{OW} = \text{H.T.U.}_W + \text{H.T.U.}_S\,(mV_W/V_S) \qquad (3)$$

$$N_t = \log\left[C^*_{W_1}/(C^*_{W_2} - C_{W_2})\right]/\log\left[C_{W_2}/(C^*_{W_2} - C^*_{W_1})\right] \qquad (4)$$

$$\text{H.E.T.S.} = Z/N_t \qquad (5)$$

$$E_A = 100.\,Nt/Na \qquad (6)$$

Equations 1 to 4 hold good where the system does not depart widely from the simple distribution law, and where volume changes, amount extracted, and concentrations involved are not large (*3*). Equation 3 provides an interesting method of correlating extraction data (2) under widely varying conditions of flow. Such runs were discarded where the material balance error exceeded 10%.

COLUMN BEHAVIOR IN OPERATION

At a constant water flow rate with increasing solvent rate, the solvent droplets were generally more nearly spherical, moved faster, and coalesced rapidly beneath the plates. Methyl isobutyl carbinol droplets were observed to be relatively smaller and more uniform in size than the toluene drops. The tendency for the solvent drops to become slightly elliptical was observed at higher solvent rates; this was more marked with toluene drops. All the holes did not operate at low solvent rates, and this did not seem to interfere with the performance of the column. At higher solvent rates, however, the drops issued from the perforations at the end of a stream above the plate surface, before breaking up into globules. The thickness of the solvent layer beneath each plate was measured for all the runs and seemed to be practically independent of the solvent rate for any given water rate. Similarly, the holdup of the dispersed solvent phase, which was not measured, was observed to be more a function of the continuous water phase than of the dispersed solvent phase.

At a constant solvent rate with increasing water rate, the solvent drops were observed slightly elliptical in shape; this was more pronounced, especially with toluene. At higher water rates, the drops generally showed greater tendency to coalescence; the solvent layer thickness beneath each plate markedly increased, resulting in increased holdup, and ranged from about 1 to 6 cm. for dispersed toluene and 0.5 to 2 cm. for dispersed methyl isobutyl carbinol.

DISCUSSION OF RESULTS

A summary of over-all mass transfer data was presented in Table II and the experimental data and calculated results reported in Table III.

Toluene-Benzoic Acid-Water System. As this system has been chosen with a view to standardize the apparatus, the mass transfer data obtained were compared in Table IV and Figure 1 with similar data reported in the literature under nearly similar conditions of operation. Reasonably good agreement was observed between present and literature values of the individual film transfer units—namely, H.T.U.$_W$ and H.T.U.$_S$. The slight variation in the values might be attributed to the slightly different sizes of the columns used by the other investigators.

The graphical treatment of the data showed that the over-all mass transfer coefficients increased with increase in toluene rate at a constant flow rate of water, and the stage efficiencies also showed an increase from about 2% to 7%. On the other hand, over-all coefficients were not practically affected by the water rate at a given solvent rate, while stage efficiencies decreased from about 4% to 2% with increasing water rate.

Inspection of Table III shows that values of H.E.T.S. like H.T.U.$_{OW}$ decreased with increasing toluene flow rate at a constant water rate of about 26.5 ft./hr., and further they increased with increasing water rate at a toluene rate of about 20.5 ft./hr. Over the series of flow rates studied, H.E.T.S. ranged from about 10 to 38 ft.

Methyl Isobutyl Carbinol–Acetic Acid–Water. FLOW RATES. To establish the effect of flow rates on the efficiency of mass transfer, both dispersed and continuous phase flow

Table III. Experimental Results and Mass Transfer Data

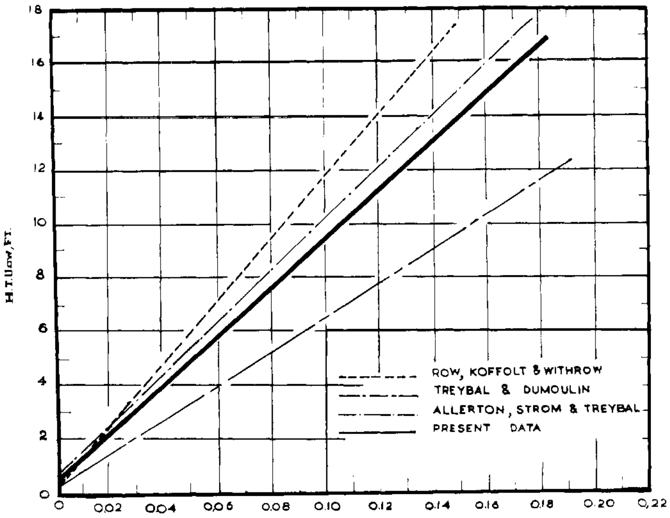
Run No.	Phase Flow Rates[a]		Phase Solute Concn.[b]										
	V_T	V_W	Toluene in[e]	Toluene out[e]	H_2O in[f]	H_2O out[f]	N_{av}[c]	Solute M.B.E., %	K_Wa[d]	H.T.U.$_{OW}$, Ft.	$\frac{mV_W}{V_T}$	H.E.T.S., Ft.	E_A, %
1	8.1	26.2	10.250	9.409	...	2.669	0.690	−2.5	1.90	13.8	0.156	26.99	2.28
2	9.6	26.3	10.030	8.920	...	3.818	1.023	5.2	2.97	8.85	0.127	30.86	1.99
3	20.4	26.2	10.030	9.296	...	5.465	1.467	4.7	5.23	5.02	0.062	15.63	3.93
4	25.4	27.0	10.250	9.588	...	5.940	1.647	4.9	6.10	4.43	0.051	16.02	3.83
5	27.0	26.3	9.961	9.409	...	5.430	1.457	4.4	5.17	5.06	0.047	17.72	3.47
6	33.2	26.8	10.120	9.545	...	6.790	1.867	4.6	7.72	3.48	0.037	13.41	4.58
7	39.5	26.2	10.150	9.623	...	7.422	1.992	4.6	9.47	2.77	0.032	9.94	6.18
8	54.5	26.8	10.120	9.740	...	7.450	2.059	5.8	9.88	2.72	0.024	10.96	5.61
9	69.6	26.3	10.150	9.835	...	7.980	2.142	4.9	12.20	2.17	0.018	9.30	6.69
10	19.5	23.3	10.200	9.520	...	5.482	1.301	4.0	4.62	5.05	0.057	17.05	3.61
11	19.9	36.5	9.859	9.098	...	4.320	1.547	−4.1	4.78	7.64	0.088	19.27	3.18
12	21.2	46.8	10.040	9.280	...	3.319	1.580	3.0	4.48	10.21	0.106	26.68	2.31
13	19.9	56.0	10.020	9.200	...	2.845	1.616	2.5	4.45	12.59	0.135	29.76	2.07
14	20.8	67.6	10.040	9.202	...	2.462	1.704	4.5	4.57	14.80	0.142	33.04	1.86
15	20.8	84.4	10.040	9.200	...	2.010	1.710	1.68	4.46	18.90	0.195	37.83	1.63
M.I.B.C.[g]–Acetic Acid–Water System Dispersed phase M.I.B.C., direction of extraction M.I.B.C. → water Inlet acid concentration 0.005 lb. moles/cu. ft.													
1	10.1	24.3	5.03	1.06	...	1.72	0.0409	−4.0	3.90	6.24	2.56	3.78	16.25
2	14.8	25.0	5.01	1.20	...	2.34	0.0574	−4.1	5.80	4.31	1.81	3.18	19.33
3	19.8	24.8	5.01	1.74	...	2.73	0.0662	−4.6	6.51	3.82	1.33	3.08	20.30
4	24.5	24.5	5.03	1.93	...	3.22	0.0776	−3.6	7.56	3.24	1.06	3.14	19.61
5	30.1	24.9	4.97	2.16	...	3.50	0.0859	−3.1	8.53	2.92	0.88	3.10	19.85
6	37.4	24.9	4.97	2.74	...	3.97	0.0973	−3.2	9.80	2.54	0.72	3.20	19.21
7	20.4	11.0	5.01	2.55	...	4.75	0.0512	−4.2	7.80	1.41	0.57	1.91	32.25
8	20.4	15.8	5.01	1.92	...	4.06	0.0635	−2.2	7.76	2.04	0.83	2.18	28.18
9	20.9	20.7	5.05	1.73	...	3.40	0.0697	−1.3	7.43	2.78	1.06	2.70	22.79
10	20.5	26.2	5.01	1.01	...	2.83		−10.8					
11	20.7	32.5	5.05	1.31	...	2.46	0.0781	−4.5	7.74	4.20	1.68	3.20	19.20
12	20.4	38.4	4.99	1.16	...	2.08	0.0790	−2.3	7.72	4.98	2.01	3.45	17.84
13	20.6	43.6	4.99	1.07	...	1.85	0.0805	−0.9	7.70	5.66	2.27	3.60	17.09
Inlet acid concentration 0.010 lb. moles/cu. ft.													
1	11.1	24.3	10.02	3.27	...	3.28	0.0773	−5.5	3.06	7.95	2.33	5.15	11.94
2	15.8	24.7	10.02	3.79	...	4.20	0.1011	−5.3	3.92	6.18	1.72	4.75	12.94
3	19.5	24.5	10.01	4.08	...	4.93	0.1177	−3.6	4.78	5.13	1.34	4.38	14.00
4	24.1	24.1	9.96	4.89	...	5.36	0.1261	−5.7	4.92	4.92	1.06	4.70	13.10
5	31.6	24.3	9.96	5.80	...	5.84	0.1368	−7.2	5.10	4.76	0.82	4.81	12.75
6	37.4	24.3	10.01	5.92	...	6.48	0.1555	−3.1	6.10	3.98	0.68	4.75	12.94
7	20.2	11.9	9.96	4.74	...	8.60	0.1034	−3.1	6.40	1.85	0.63	2.33	26.85
8	20.8	19.5	9.96	3.80	...	6.31	0.1257	−3.8	6.10	3.20	1.00	3.26	18.85
9	20.6	24.3	10.01	3.39	...	5.44	0.1344	−3.3	6.25	3.88	1.26	3.49	17.75
10	19.9	29.2	10.01	2.70	...	4.78	0.1428	−4.1	6.78	4.30	1.57	3.35	18.36
11	20.2	34.2	10.02	3.01	...	4.13	0.1413	−1.1	6.24	5.47	1.81	4.08	15.08
12	20.4	40.2	10.02	2.99	...	3.42	0.1387	−2.4	5.75	6.97	2.10	4.68	13.13
Inlet acid concentration 0.015 lb. moles/cu. ft.													
1	10.1	24.8	14.99	5.04	...	4.12	0.1017	−1.0	2.52	9.84	2.62	5.86	10.48
2	17.5	24.3	14.99	6.42	...	6.42	0.1532	−3.9	3.48	6.97	1.48	5.18	11.86
3	23.8	25.0	15.02	8.04	...	6.75	0.1675	−1.4	3.84	6.50	1.12	6.18	9.95
4	29.1	24.2	15.02	8.87	...	7.43	0.1795	−0.7	4.28	5.65	0.89	6.36	9.67
5[h]	33.4	24.3	15.05	8.81	...	7.62		−11.2					
6	37.4	24.5	15.05	9.54	...	8.55	0.2074	−1.4	4.80	5.10	0.70	6.04	10.18
7	43.6	25.2	15.05	9.75	...	9.32	0.2330	−1.4	5.57	4.52	0.62	5.62	10.93
8	20.6	10.3	15.05	8.87	...	12.50	0.1278	−0.9	4.47	2.31	0.53	3.20	19.21
9	20.8	16.0	14.99	7.73	...	9.60	0.1523	−2.0	4.25	3.26	0.82	4.14	14.88
10	21.1	20.7	15.05	7.23	...	8.10	0.1665	−1.6	4.34	4.77	1.05	4.70	13.09
11[h]	20.9	27.1	15.05	5.27	...	6.58		−12.2					
12	20.6	31.1	15.02	5.87	...	6.07	0.1888	−1.1	4.75	6.55	1.61	5.03	12.20
13	20.4	37.8	15.02	5.60	...	5.21	0.1946	−2.3	4.72	8.03	1.98	5.43	11.32
14	20.6	45.2	14.99	5.49	...	4.32	0.1977	−2.3	4.60	9.83	2.34	6.08	10.11
Direction of extraction water → M.I.B.C.													
1	11.4	24.5	0.24	4.94	10.02	7.89	0.0530	2.9	1.74	14.10	2.99	9.75	6.31
2	17.4	25.0	0.24	3.99	10.02	7.30	0.0667	4.5	2.13	11.73	1.53	9.48	6.49
3	25.0	24.9	0.21	3.86	9.96	6.22	0.0920	1.7	3.18	7.82	1.06	7.58	8.10
4	33.3	25.1	0.21	3.31	9.96	5.78	0.1039	1.4	3.54	7.17	0.80	7.91	7.77
5	39.0	24.9	0.27	3.19	10.01	5.39	0.1145	1.2	3.97	6.27	0.68	7.52	8.18
6	44.3	24.6	0.27	3.09	10.01	4.81	0.1263	2.1	4.57	5.38	0.59	6.83	9.01

[a] In cu. ft./hr. sq. ft. [b] In lb. moles/cu. ft. [c] In lb. moles/(hr.) (sq. ft.) × 10^{-2} [d] In lb. moles/(hr. cu. ft.) (Δ_c). [e] Times 10^{-3}. [f] Times 10^{-4}. [g] Methyl isobutyl carbinol. [h] Runs rejected due to poor material balance.

Table IV. Toluene–Benzoic Acid–Water System

(Comparison of film H.T.U. values with those reported in the literature)

Ref. No.	Size of Column I.D., In.	Hole Size, In.	Plate Spacing, In.	Range of Flow Rates, cu. ft. (hr.) (sq. ft.)		H.T.U.$_{w}$, Ft.	H.T.U.$_{s}$, Ft.
				Solvent	Water		
(*15*)	8.75	⅛	6	11.8–47.7	11.8–37.1	0.25	115
(*16*)	3.56	$^3/_{16}$	6	30.2–76.8	22.9–41.5	0.25	63
(*1*)	3.63	$^3/_{16}$	4.75	38.6–14.4	36.4–77.6	0.70	95
This work.	3.06	⅛	6	8.1–69.6	23.3–67.6	0.50	90



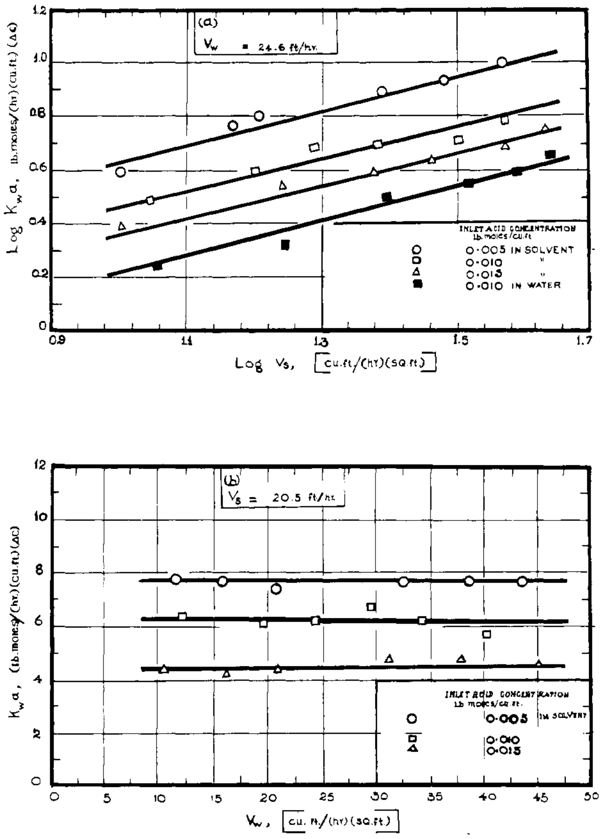


Figure 2. Extraction of acetic acid between methyl isobutyl carbinol and water

Effect of phase flow rates on over-all extraction coefficients

rates were varied from about 10 to 50 ft./hr., and the upper limits of these were well within the flooding rates. Examination of Figure 2 shows that K_Wa values increased with increased rate of the dispersed solvent phase, owing to increased holdup of this phase. For a constant inlet acid concentration, the extraction coefficients were almost constant, being very little influenced by the continuous phase rate. This is in agreement with earlier observations on the effect of flow rates on mass transfer in perforated-plate columns.

Flow Rate Ratio. To obtain economic throughputs in the column, flow rate ratio rather than the individual values was considered important. Figure 3 shows that the mass transfer efficiency decreased as the ratio V_W/V_S was increased. This decrease was believed to be largely due to the greater effect of the water film resistance at higher values of V_W/V_S.

The slope and intercept values, obtained from the plot of H.T.U.$_{OW}$ *vs.* mV_W/V_S (Figure 4) according to Colburn (*2*) were listed in Table V, as indicative of the individual film values but not true values.

The H.E.T.S. values were found to vary from about 2 to 10 ft. over the range of flow rate ratios from 0.5 to 2.5, and they were observed to decrease with increasing solvent rate or decreasing water rate for a constant flow rate of the other phase.

Inlet Acid Concentration. Figure 2 shows that at a constant V_W of 24.6 ft./hr., the mass transfer efficiency, expressed as K_Wa decreased by about 15 to 35% on increasing the solute concentration from 0.005 to 0.015 lb. moles per cu. ft. Further, the intercept values in the H.T.U.$_{OW}$ plots, indicative of H.T.U.$_{W}$, increased from 1 to 3.5 ft., while the slope values, representing H.T.U.$_{S}$, remained substantially constant at 2.2 ft.

At a constant solvent rate of 20.5 ft./hr., with increasing water rate, the effect of increase in inlet acid concentration was found to be such that K_Wa values decreased by about

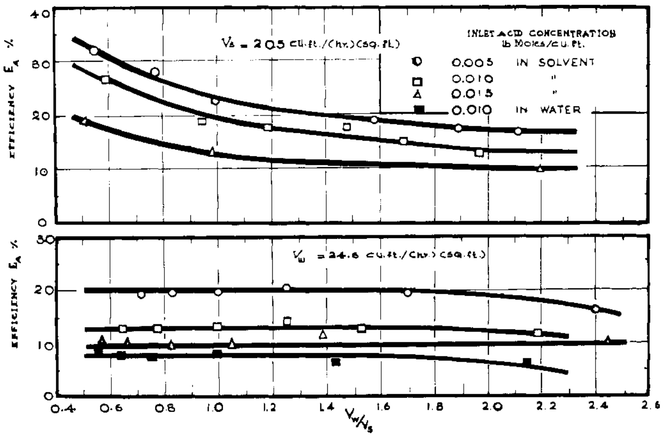


Figure 3. Extraction of acetic acid between methyl isobutyl carbinol and water

Effect of flow rote ratio on stage efficiencies, with inlet acid concentration and direction of extraction as parameters

20 to 30% (Figure 2). Further H.T.U.$_S$ values increased from 2.5 to 4.3 ft., while the H.T.U.$_W$ values were almost zero, showing thereby negligible resistance in the water film on increasing the inlet acid concentration.

H.E.T.S. values increased by about 50% with increase in solute concentration from 0.005 to 0.010 lb. mole/cu. ft., and by about 90% with increase to 0.015 lb. mole/cu. ft.

Direction of Extraction. Mass transfer efficiency (Figure 2) was reduced about 40% by changing the direction of solute transfer from methyl isobutyl carbinol to water to that in the reverse direction. This might be explained as probably due to the greater forces of adhesion caused by hydrogen bonding in the reverse direction of extraction, which led to poor extraction efficiency. H.T.U.$_S$ values increased from 2.2 to 5.0 ft., whereas the H.T.U.$_W$ values were substantially constant at 2.7 ft. (Figure 4 and Table V). H.E.T.S. values increased by 60 to 70% in the reverse direction of extraction.

The curves of Figure 3 approximated to rectangular hyperbolas of the form, $K' = E_A\ (V_W/V_S)^{\gamma'}$ where K' and γ' are constants which are obtained as intercept and slope values of a log-log plot of E_A against V_W/V_S (Figure 5). Efficiencies of the toluene-benzoic acid-water system were also brought on the same plot for a comparative study. For maximum contacting efficiency, it is obvious that both constants, K' and γ', should be as high as possible. These values are specially necessary for computing economic throughputs in the column. The values of these constants were presented in Table VI.

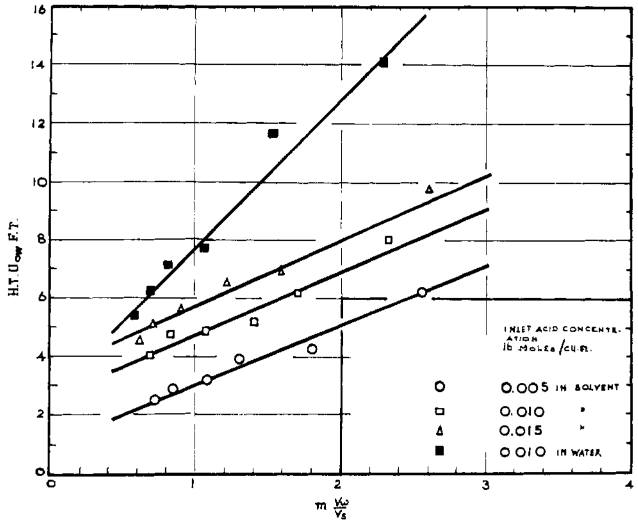


Figure 4. Extraction of acetic acid between methyl isobutyl carbinol and water

Variation of H.T.U.$_{OW}$ with extraction factor, mV_W/V_S, at constant V_W 24.6 cu. ft./(hr.) (sq. ft.)

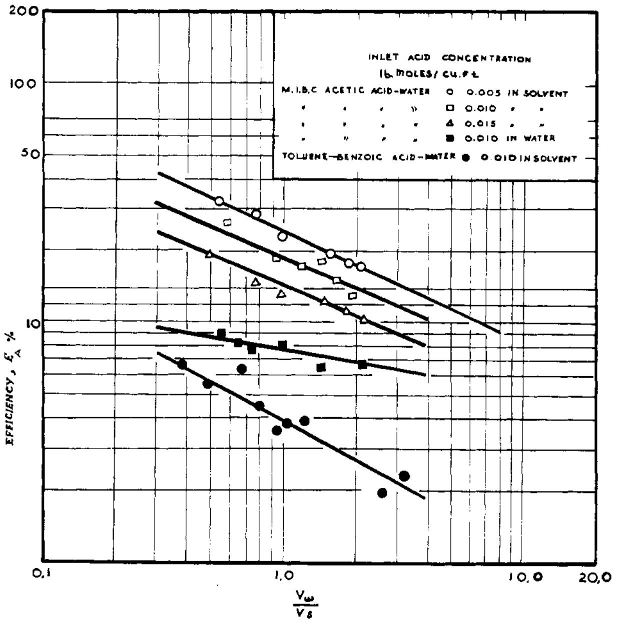


Figure 5. Effect of flow ratio on stage efficiencies for toluene–benzoic acid–water, and methyl isobutyl carbinol–acetic acid–water systems

Table V. Values of H.T.U.$_W$ and H.T.U.$_S$ for the System M.I.B.C.–Acetic Acid–Water in a 3$\frac{1}{16}$-Inch Perforated Plate Extraction Column

Inlet Acid Concn.[a]	Direction of Transfer	Phase Flow Rates[b] Water	Solvent	H.T.U.$_W$, Ft.	H.T.U.$_S$, Ft.
0.005	Solvent → Water	(A) 24.6 (av.)	10.1-37.4	1.0	2.2
		(B) 11.0-43.6	20.5 (av.)	. . .	2.5
0.010	Solvent → Water	(A) 24.6 (av.)	11.1-37.4	2.7	2.2
		(B) 11.9-40.2	20.5 (av.)	. . .	3.2
	Water → Solvent	(A) 24.6 (av.)	11.4-44.3	2.7	5.0
0.015	Solvent → Water	(A) 24.6 (av.)	10.1-43.6	3.5	2.2
		(B) 10.3-45.2	20.5 (av.)	. . .	4.3

[a] In lb. moles/cu. ft. [b] In cu. ft./(hr.)(sq. ft.)[b]

Table VI. Effect of Flow Rate Ratio on Stage Efficiencies for the Toluene–Benzoic Acid–Water System and M.I.B.C.–Acetic Acid–Water System: Values of K' and γ'

System	Inlet Acid Concn., Lb. Moles/Cu. Ft. Solution	Direction of Extraction	K'	γ'
Toluene-Benzoic Acid-Water	0.010	Toluene → Water	14	-0.55
M.I.B.C.-Acetic Acid-Water	(A) 0.005	M.I.B.C. → Water	76	-0.45
	(B) 0.010	M.I.B.C. → Water	52	-0.45
	(C) 0.015	M.I.B.C. → Water	38	-0.45
	(D) 0.010	Water → M.I.B.C.	12	-0.21

NOMENCLATURE

a = interfacial surface area, sq. ft./cu. ft. of volume
c = concentration of solute, lb. moles/cu. ft. of solution in the phase indicated by the subscript
E_A = stage efficiency, dimensionless
H.E.T.S. = height equivalent to a theoretical stage, ft.
H.T.U. = individual film height of a transfer unit based on the phase indicated by the subscript, ft.
H.T.U.$_O$ = over-all height of a transfer unit based on the phase indicated by the subscript, ft.
Ka = over-all mass transfer coefficient, based on the phase indicated by the subscripts, lb. moles/hr., cu. ft., ΔC
K' = constant
M.I.B.C. = methyl isobutyl carbinol
m = slope of the equilibrium curve = dC_W^*/dC_S
M.B.E. = material balance error for the solute, %.
N' = rate of mass transfer, lb. moles/sq. ft., hr.
N_a = number of actual stages
N_t = number of theoretical stages
V = superficial velocity, cu. ft./hr., sq. ft., of the phase indicated by the subscript
Z = effective extraction height, ft.
Δ = difference
γ' = constant
θ = time

Subscripts

C or W = continuous phase (water)
D or S = dispersed phase (solvent)
T = toluene phase
1 = conditions at the top of the column
2 = conditions at the bottom of the column

LITERATURE CITED

(1) Allerton, J., Strom, B.O., and Treybal, R.E., *Trans. Am. Inst. Chem. Engrs.* **39**, 361 (1943).
(2) Colburn, A.P., *Ibid.*, **35**, 211 (1939).
(3) Elgin, J.C., Browning, F.M., *Ibid.*, **31**, 639 (1935).
(4) Garner, F.H., Ellis, S.R.M., Fosbury, D.W., *Trans. Inst. Chem. Engrs. (London)* **31**, 348 (1953).
(5) Garner, F.H., Ellis, S.R.M., Hill, J.W., *Ibid.*, **34**, 223 (1956).
(6) Garner, F.H., Ellis, S.R.M., Hill, J.W., *A.I.Ch.E. Journal* **1**, 185 (1955).
(7) Hayworth, C.B., Treybal, R.E., *Ind. Eng. Chem.* **42**, 1174 (1950).
(8) Logsdail, D.H., Thornton, J.D., *Trans. Inst. Chem. Engrs. (London)* **35**, 331 (1957).
(9) Mayfield, F.D., Church, W.L., *Ind. Eng. Chem.* **44**, 2253 (1952).
(10) Moulton, R.W., Walkey, J.E., *Trans. Am. Inst. Chem. Engrs.* **40**, 695 (1944).
(11) Perry, J.H., Chemical Engineers' Handbook, McGraw-Hill, 3rd ed. p. 752, 1950.
(12) Pyle, C., Colburn, A.P., Duffey, H.R., *Ind. Eng. Chem.* **42**, 1042 (1950).
(13) Rao, M.R., Rama Murty, M. Rao., C.V., *Chem. Eng. Sci.* **8**, 267 (1958).
(14) Rao, M. Raja., Rao, C. Venkata., J. CHEM. ENG. DATA. **6**, 200 (1961).
(15) Row, S.B., Koffolt, J.H., Withrow, J.R., *Trans. Am. Inst. Chem. Engrs.* **37**, 559 (1941).
(16) Treybal, R.E., Dumoulin, F.H., *Ind. Eng. Chem.* **34**, 709 (1942).
(17) Treybal, R.E., "Mass Transfer Operations," McGraw-Hill, 1st ed., 1. 378, 1955.

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Ternary Liquid Equilibria: 1-Hexanol–Water–Fatty Acids

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LIQUID EXTRACTION has lately received considerable attention as an important separation process, especially in view of its applicability in such processes where vaporization methods are not practicable. For the successful development of liquid extraction processes and the design of extraction equipment, it is necessary to have a knowledge of the phase equilibrium relations of ternary liquid systems.

The present work, part of a study of fatty acid extraction and investigations of liquid phase equilibria in search of potential solvents, was undertaken to find out the applicability of higher alcohols as solvents for the extraction of fatty acids from dilute aqueous solutions.

MATERIALS

Propionic Acid. Propionic acid of reagent grade from E. Merck Co. was used. Chemical analysis, as determined by titration with standard alkali, shows it to be 99.0% pure. Density at 30° C. = 0.9829 gram per cc.

Butyric Acid. Butyric acid of reagent grade from Naarden Chemical Co. (Holland) was used. The purity as determined by titration with standard alkali is 99.3%. Density at 30° C. = 0.9508 gram per cc.

***n*-Hexanol.** *n*-Hexanol of Jean A. du Crocq, Jr., Ltd., (Holland) was subjected to fractionation in an all-glass fractionating column and the fraction, boiling between 156° and 156.5° C. was collected and used.

Distilled water, free from carbon dioxide, was used in all the cases.

EXPERIMENTAL PROCEDURE

Solubility and tie-line data were determined by the method of Othmer and others (*2*). The binodal curve was determined by the appearance and disappearance of turbidity, the former being used for building up the end wings; the latter, for establishing the middle portion of the curve.

For the determination of the tie-line data, ternary liquid mixtures of known composition within the two-phase region were prepared in separating funnels, shaken vigorously for

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