PERFORATED-PLATE EXTRACTION-COLUMN PERFORMANCE AND WETTING CHARACTERISTICS

F. H. Garner, S. R. M. Ellis, and J. W. Hill, The University, Edgbaston, Birmingham, England

The type of surface of the perforated plate influences the performance of a perforated-plate column. Pilot plant data on a 4-in column for the system toluene-diethylamine-water at 20°C. show that when the water phase is dispersed and the direction of mass transfer is from the continuous toluene to the dispersed water phase polyethylene plates give a continuous stream of droplets and a higher efficiency, but that when the direction of mass transfer is from the dispersed water to the continuous toluene phase the metal plates give the higher efficiency.

The design of perforated plates for liquid-liquid extraction columns has been studied with reference to the number of plates in the column and their spacing and percentage of free area, but little information has appeared in the literature concerning the effects of the nature of the surface of the perforated tray and of its drop-releasing properties.

Usually the two phases in liquid-liquid extraction possess different characteristics; water is generally one of the phases, and some organic liquid the other phase, and it is usual to disperse the organic phase in the aqueous one. The surface tension of the two phases thus vary considerably, and in consequence each phase will have different wetting properties. The continuous water phase preferentially wets steel plates; the layer of organic liquid below the plate gives rise to droplets of regular size and shape which are formed very close to the plate surface, and the extraction column operates satisfactorily. If however the water phase is dispersed, it still preferentially wets the plate, and irregular droplet formation results unless some alteration is made to the plate to improve droplet formation. This is probably the reason why little use has been made in perforated-plate columns of systems with the water phase dispersed.

An application of the wetting theory has been made to the operation of a pulsed extraction column by use of a perforated plate divided into two layers of different material, each layer possessing different wetting properties(*3*). The selection of packings with correct wetting characteristics giving higher contacting efficiency in extraction columns has also received attention(*12*). Recently a successful attempt has been made to disperse the water phase efficiently by fitting sharp-edged jets to the plates and operating the column above the streaming velocity; the efficiency of operation was greatly increased(*13*). Another method of ensuring good droplet release is changing or altering the surface of the plate so that the organic phase instead of the water phase will wet the trays. This can be done in two ways: by selecting a material with suitable surface characteristics for all the plates or by coating the surface of the existing plates with a suitable compound that will be unaffected by the solvent action of the organic phase. The former method is by far the simpler.

In order to provide a method of selecting an appropriate metallic or nonmetallic surface for use in a perforated-tray column under certain conditions of phase dispersion, the contact angles made by the organic droplet in a water phase and by the water droplets in an organic phase have been measured for a number of different surfaces by use of the two liquid phases of the system under investigation. From an inspection of the results of these measurements a suitable material was chosen and a set of perforated plates made.

EXPERIMENTAL WORK

The experimental program investigated the wetting properties of different surfaces, leading to the selection of polyethylene for use as the perforated tray when the water phase is dispersed. Mild-steel plates have been found to be suitable *for toluene* dispersion in a continuous water phase(*5*).

The results obtained with a 4-in. pilot plant plate column provide a comparison of operation for polyethylene and metal plates with the water phase dispersed. The factors which have been investigated are given below in order of importance:

1. Surface of plate—affecting droplet release.
2. Direction of solute transfer (in connection with 3).
3. Phase dispersed—whether that extracting or giving up the solute.
4. Upcomer height—affecting column operation.

Wetting Characteristics of Surfaces. The contact angles made by the droplets on a number of different materials in a surrounding continuous phase were measured by observing the sessile droplet through a traveling microscope, which contained cross wires that could be rotated and placed along the tangent to the droplet at the point of contact. The angle through which the cross wires had been turned was read from a scale fixed to the barrel of the microscope. Values of the vertical height of the drop were also recorded together with its base length. The latter measurements enabled a check to be made on the angle of contact by application of the equations of Talmud and Lubman(*19*). Samples of the surfaces investigated were made 1 5/8-in. long and 1/2 in. wide and by means of a special brass clipped holder were immersed in the continuous phase, contained in a 6 × 6 × 6 cm. glass cell with plain plate sides.

It was considered necessary to measure both the contact angle made by the solvent droplet on the solid in water and also that made by the water droplet in the solvent. These typify instances when the water phase was receding and advancing, respectively. It can be shown(*7*) that the difference in the cosine value of the two contact angles when the water phase is receding and advancing is an empirical measure of the adhesive work between the fluids and the surface in question.

TABLE 1.—TOLUENE-WATER SYSTEM AT 20°C.

	Water receding θ_1, degrees	Water advancing θ_2, degrees	$\cos \theta_1 - \cos \theta_2$
Glass	134	17	−1.66
Porcelain glazed	144	88	−0.88
Mild steel (rusted)	160	95	−0.77
Brass	135	130	−0.07
Porcelain unglazed	88	120	+0.85
Mild steel (clean)	58	141	+1.31
Mild steel cadmium plated	43	144	+1.54
Mild steel chromium plated	35	138	+1.56
Hostaflon	35	≅180	+1.82
Polyethylene	32	≅180	+1.85
Kel-F	32	≅180	+1.85
P.T.F.E. (Teflon)	23	≅180	+1.92

The results of these measurements are given in Table 1. The negative sign for $\cos \theta_1 - \cos \theta_2$ indicates that the surface is hydrophilic in nature, being preferentially wet by the water phase, whereas a positive value of $\cos \theta_1 - \cos \theta_2$ indicates that the surface is preferentially wet by the solvent phase (hydrophobic). When the water phase is dispersed, the more hydrophobic the nature of the surface, the more suitable is the material for a perforated tray. When the solvent phase is dispersed, a hydrophilic surface is more efficient with regard to droplet release.

From Table 1 polyethylene was chosen as the perforated-plate material in a 4-in. extraction unit using the system toluene-diethylamine-water, in order to observe the effect of water phase dispersion and at the same time to compare operation with the more conventional mild-steel plates. Polytetrafluoro ethylene (P.T.F.E.) would be a better material to employ, but its cost is at present restrictive. Polyethylene is relatively cheap and easy to machine, but it has a tendency to swell slightly in the presence of toluene. This was overcome to a large extent by machining the plates undersize and fitting gaskets to avoid buckling.

Polyethylene was used in a pulsed column as a perforated-plate material by Werning and Higbie(*24*), because of the need for resistance against attack in acid solutions. Teller(*20*) has also used polyethylene in the form of rosette rings in a packed ammonia absorption tower; this material was chosen in order to minimize any wetting contribution to transfer by the liquid phase and so to obtain higher interstitial holdup.

Apparatus. A flow diagram of the pilot plant used in this study is given in a previous paper(*5*). The column was constructed from two 2-ft. lengths of 4-in. nominal-bore glass pipe with flat ground joints connected to a 1-ft.-long 6-in. bore-head section by means of a steel-throat section. The base of the glass column was attached to a 21-in.-high conical sectional enlarged from 4 to 9 in. in diam. in a distance of 12 in.

TABLE 2.—DETAILS OF RUN TYPES OF THE 4-IN.-TRAY COLUMN

Series	No. of runs performed	Plate type	Upcomer height, in.	Phase dispersed	Transfer direction	Average continuous phase rate, cu. ft./(sq. ft.)(hr.)	Comments
E	3	Metal	4½	Water	*W*→*T*	56.5	Eff. 2-3% poor dispersion
L	4	Metal	4½	Water	*W*→*T*	21.0	Eff. 7-8% poor dispersion Low solute transfer, but extreme turbulence
K	5	Metal	4½	Water	*T*→*W*	21.0	Eff. 8-11% poor dispersion Extreme turbulence of dispersed phase
F	4	Polyethylene	4½	Water	*W*→*T*	2 @56.5 2 @21.0	Eff. 2-3% low solute transfer
I	4	Polyethylene	4½	Water	*T*→*W*	21.0	Eff. 7-15% good droplet release
J	1	Polyethylene	4½	Water	*T*→*W*	75.4	Good droplet release
G	15	Polyethylene	2	Water	*W*→*T*	21.0	Eff. 3-7% low solute transfer
H	7	Polyethylene	2	Water	*T*→*W*	21.0	Eff. 7-14% good droplet release

In series *A*, *B*, *C*, and *D*, metal plates were used with 4½-in.-high downcomers, and the toluene phase was dispersed. Transfer direction was mainly toluene to water (*5*).

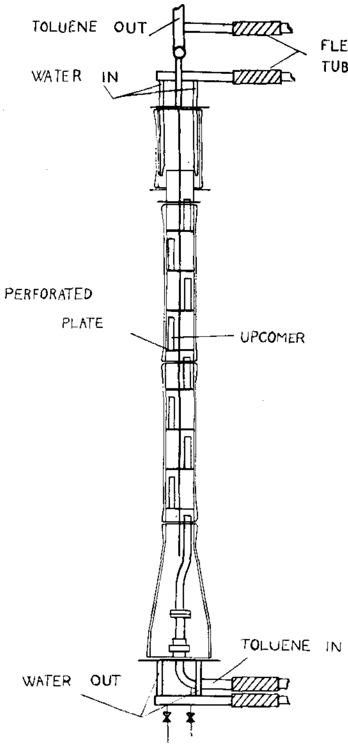

FIG. 1.

The interface was maintained at a constant height in the bottom conical section by adjustment of the back pressure on the column by means of control valves in the exit water line. These valves were a 3/4-in. taper plug valve in parallel with a 1/4-in. needle valve for fine control.

The equilibrium distribution data used in this investigation were obtained by Garner, Ellis and Fosbury (*5*) and Wehn and Franke(*23*). As these limiting-solubility data had been determined at 20°C., it was necessary to adjust the incoming feed liquids to this temperature by circulating water at a suitably controlled temperature through jackets surrounding the constant-head standpipe between the feed pumps and overflows. The temperatures of the incoming phases were read at phase-separating pots and those of the outgoing phases at the head and base cover plates.

The extraction column is shown in more detail in Figure 1. The toluene inlet came to within 1 1/2 in. of the underside of the lower plate. The entering continuous lighter phase was thus admitted to the column as though it came from another plate below the bottom one. The heavier water phase flowed over the 3-in. weir down the wall of the column until the top plate was reached, where it formed a layer on the uppermost side. The water phase was thus acting in a similar manner to that of a wetted-wall heavier phase for the first 7 in. of its passage down the column.

Details of the Perforated Plate. The perforated plate, a plan view of which is given in Figure 2, was of similar design to plates used in the previous investigation(*5*). The polyethylene plates and the metal ones were of the same dimensions, but the plastic plates were 1/8 in. thick and the metal plates were made from 16-gauge tinned-steel plates; each contained fifty-nine 1/8-in. holes representing 6.02% of column cross-sectional area. The upcomer diameter in both cases was 1 in. and represented 5.35% of the column cross-sectional area. The upcomers in the polyethylene plates were made "push fits" instead of being soldered in position. Metal plates were made with 4 1/2-in.-high upcomers but the polyethylene plates could be fitted with either 4 1/2- or 2-in.-high upcomers.

Eight plates were suspended at 6-in. spacings on a 1/4-in. central rod. Because of a constriction in the column diameter at each butt weld, the plates were cut to 3 15/16 in. and fitted with overlap gaskets in order to seal the gaps between the plates and the column wall.

The height of the upcomer assigned to each plate, together with the details of each group of runs, can be seen from Table 2.

RUN PROCEDURE

Most of the runs were carried out at the same low continuous toluene rate of approximately 21.0 cu.ft./(sq.ft.)(hr.), or 12.3 lb.moles/(sq.ft.)(hr.), with the exception of the *E*, *F*, and *J* runs, which were at higher toluene rates (*see* Table 2). This low rate of continuous phase was chosen so that a usual study of droplet release with variations in dispersed-phase flow rate might be made, and at the same time a variation in H.T.U.$_{OC}$ and E_A as a result of changing L_C was avoided, as observed by Garner, Ellis and Fosbury(*5*).

The following liquids were used: toluene of nitration grade, diethylamine of 97% purity, and distilled water. This system has previously been used in packed columns by Morello and Beckmann(*15*), Leibson and Beckmann(*10*), and Wehn and Franke(*23*), and in earlier work by Garner, Ellis, and Fosbury(*5*), using the same 4-in. column. The equilibrium data of the latter two groups of workers have been used.

Before each run the phase containing the solute was made up to approximately 1.0% diethylamine by weight, and after the run about 10 to 15 gal. of extracting phase were replaced by an equal quantity of solute free phase.

With toluene as the continuous phase, water was admitted until a sufficient layer had collected at the base of the column to provide an interface reading, and then the column was filled with the continuous phase. After the interface valve was closed, the desired water rate was set, while the interface steadily rose. On opening of the interface valve, water was released until no further toluene was pushed out of the column. The desired toluene rate was then set and the interface accurately adjusted.

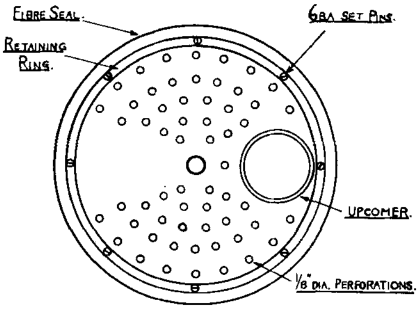

FIG. 2. PLAN VIEW OF THE PERFORATED PLATE.

After the elapse of sufficient time for two to three changes of continuous phase to take place, samples of each phase entering and leaving the column were taken, and again at intervals of 15 min. for the next 1/2 hr. Outlet flow rates were measured at points immediately after the weir vessels twice during this period. The time taken for each run was between 1 1/2 and 2 hr.

The three sets of samples of the inlet and outlet phases were bulked and analyzed for solute content using N/15 HCl. The density of the phases at 20°C. was determined by use of a Westphal balance. The average of the flow checks was used to determine the flow rates during the runs.

CONTROLLING RESISTANCE AND EXTRACTION FACTOR

When the equilibrium distribution for this system is plotted on a mole fraction basis at 20°C., the solute favors the toluene phase with the partition coefficient, $\frac{dC_T}{dC_W}$, having a value of 3.18. The equilibrium line is straight at low solute concentrations.

If *y* refers to the dispersed phase and *x* to the continuous phase, then

$$\frac{dy^*}{dx} = m$$

When toluene is dispersed, $m = \frac{dC_D}{dC_C} = 3.18$. When, however, the water phase is dispersed, $m = \frac{dC_D}{dC_C} = 0.314$, with the solute still favoring the toluene phase.

Thus if the water phase is dispersed, the factor $\frac{1}{mk_D}$ in Equation (1), relating the over-all continuous mass transfer coefficient to the film values, is relatively high and the dispersed-water-phase resistance is controlling.

$$\frac{1}{K_{OC}} = \frac{1}{k_C} + \frac{1}{mk_D} \tag{1}$$

The extraction factor, which is an indication of economic operation, has been defined by Treybal (*21*) and other workers in the following manner:

Extraction factor $= \frac{dC_E}{dC_R}\cdot\frac{E}{R}$ where

$E =$ the extracting phase — the phase receiving the solute

$R =$ the raffinate phase

C_E and $C_R =$ the concentrations of solute in the extract and raffinate phases

As indicated by Pike(*16*), the extraction factor will change according to the conditions existing in the column:

1. *Toluene dispersed.* a. Transfer $T \rightarrow W$ (water the extracting phase)

   Extraction factor $= \frac{dC_W}{dC_T}\cdot\frac{V_W}{V_T} = \frac{dC_C}{dC_D}\cdot\frac{V_C}{V_D} = \frac{L_C}{mL_D}$

   b. Transfer $W \rightarrow T$ (toluene the extracting phase)

   Extraction factor $= \frac{dC_T}{dC_W}\cdot\frac{V_T}{V_W} = \frac{dC_D}{dC_C}\cdot\frac{V_D}{V_C} = m\frac{L_D}{L_C}$

2. *Water dispersed.* a. Transfer $T \rightarrow W$

   Extraction factor $= \frac{dC_W}{dC_T}\cdot\frac{V_W}{V_T} = \frac{dC_D}{dC_C}\cdot\frac{V_D}{V_C} = m\frac{L_D}{L_C}$

   b. Transfer $W \rightarrow T$

   Extraction factor $= \frac{dC_T}{dC_W}\cdot\frac{V_T}{V_W} = \frac{dC_C}{dC_D}\cdot\frac{V_C}{V_D} = \frac{L_C}{mL_D}$

Hence the magnitude of the extraction factor will depend not only on the individual flow rates but also on the direction of transfer and on which phase is dispersed. The range of extraction factor used in this work was 0.1–2.0 with transfer $D \rightarrow C$, and 2.0–12.0 with transfer $C \rightarrow D$, the water phase being dispersed.

CALCULATION OF MASS TRANSFER DATA

For low solute concentrations and immiscibility of the two phases, the operating line can be expressed in the form of a linear equation:

$$y = \frac{L_C}{L_D}x + \left(y - \frac{L_C}{L_D}x\right)_{avg} \qquad (2)$$

where $\left(y - \frac{L_C}{L_D}x\right)_{avg}$ is the arithmetic average of $y_1 - \frac{L_C}{L_D}x_1$ and $y_2 - \frac{L_C}{L_D}x_2$.

With zero mass balance error $y_1 - \frac{L_C}{L_D}x_1$ and $y_2 - \frac{L_C}{L_D}x_2$ are equal and the operating line passes through the terminal composition points. When a mass balance error exists, the composition points lie equidistant on either side of the operating line. As the flow-rate data are believed to be more accurate than the volumetrically determined concentration data, corrected values of the terminal compositions were used for all performance calculations. The terminal compositions were obtained graphically by taking the points on the operating line nearest to the composition points and were not greatly different from the original values.

The evaluation of H.T.U. data was made by dividing the column height used by the number of transfer units, which were determined by Equations (3) and (4).

$$N_{toD} = \frac{1}{1 - \frac{mL_D}{L_C}}\cdot\ln\left[\frac{y_1 - mx_1}{y_2 - mx_2}\right] \pm \frac{1}{2}\ln\left[\frac{1 - y_2}{1 - y_1}\right] \qquad (3)$$

Interterm sign positive when $y_1 > y_2$

$$N_{toC} = \frac{1}{\frac{L_C}{mL_D} - 1}\cdot\ln\left[\frac{y_1 - mx_1}{y_2 - mx_2}\right] \pm \frac{1}{2}\ln\left[\frac{1 - x_2}{1 - x_1}\right] \qquad (4)$$

Interterm sign positive when $x_1 > x_2$

These equations apply where $L_C/L_D \neq m$. For the case where $L_C/L_D = m$, then

$$N_{toD} = N_{toC} = N_A = \frac{y_1 - y_2}{\left(y - \frac{L_C}{L_D}x\right)_{avg}}$$

Equations (1) and (2) are Colburn's equations simplified by Wiegand to apply to dilute solutions. In most runs the correction terms to Equations (3) and (4) were less than 0.008. From the foregoing equations it can be seen that

$$H_{toC} = \left(\frac{L_C}{mL_D}\right)H_{toD} \qquad (5)$$

The efficiency of column performance has been expressed in a number of ways. Mayfield and Church(*13*) used the McCabe-Thiele type of plot(*14*) to obtain a continuous- or dispersed-phase efficiency, whereas Pyle, Colburn, and Duffey(*17*) used the Baker-Stockhardt modification to give an average efficiency based on either phase. The efficiency E_A used in this work was evaluated from the number of theoretical stages, determined by Equation (6), and the actual number of plates in the column. The H.E.T.S. was evaluated from the effective height and N_A. The number of theoretical stages is determined by

$$N_A = \frac{\ln\left[\frac{y_1 - mx_1}{y_2 - mx_2}\right]}{\ln\frac{L_C}{mL_D}} \qquad (6)$$

This equation was obtained from a stepwise analysis(*5*) of the theoretical concentration changes in a column containing N_A theoretical stages and is not dependent on either of the phases.

The over-all volume mass transfer coefficient K_Da and K_Ca were calculated from the H.T.U. data by use of the simplified relations for dilute solutions:

$$K_Ca = \frac{V_C}{H_{toC}} \text{ and } K_Da = \frac{V_D}{H_{toD}} \qquad (7)$$

For the purposes of calculation the phase concentrations were converted to mole fraction units by use of the average phase molecular weights, which were not significantly different from 18.1 for the water phase and 91.8 for the toluene phase. The average phase temperatures were all within the range 20 ± 1°C.

RESULTS

The results obtained experimentally from the operation of the column are presented in Table 3 together with the calculated performance data. The phase flow rates are given in pound moles per square feet per hour and also cubic feet per square feet per hour and the phase solute concentrations for the entering and leaving liquids in pound moles per square feet. The column height, as shown in Table 3, is the distance between the top of the inlet weir and the two-phase interface. The percentage mass balance error, also in Table 3, is defined by (solute out—solute in)/solute in. The over-all H.T.U. and over-all volume mass transfer coefficient based on the dispersed and continuous phases are also given. The efficiency, as calculated from Equation (6) and the number of plates in the column, is included in Table 3.

TABLE 3.—EXPERIMENTAL RESULTS AND PERFORMANCE DATA

Run	Phase flow rates, Cu. ft./(sq. ft.) (hr.), Toluene V_T	Phase flow rates, Cu. ft./(sq. ft.) (hr.), Water V_W	Phase flow rates, Lb. moles/(sq. ft.) (hr.), Toluene L_T	Phase flow rates, Lb. moles/(sq. ft.) (hr.), Water L_W	Column height used, ft.	Phase solute concentrations, lb. moles/cu. ft., Toluene In	Phase solute concentrations, lb. moles/cu. ft., Toluene Out	Phase solute concentrations, lb. moles/cu. ft., Water In	Phase solute concentrations, lb. moles/cu. ft., Water Out	Solute mass balance error, %	$\frac{L_C}{mL_D}$	H.T.U.$_{OD}$ ft.	K_Ca, hr.$^{-1}$	E_A, %
*E*1	56.01	30.59	32.88	104.1	5.73	0.00197	0.00243	0.00885	0.00795	+0.9	1.004	30.3	1.85	2.4
2	56.89	16.37	33.48	55.2	5.83	0.00236	0.00269	0.00956	0.00849	−0.4	1.924	20.5	1.44	2.6
3	56.23	24.01	33.01	82.4	5.83	0.00267	0.00301	0.00936	0.00860	−0.1	1.275	25.5	1.74	2.5
*L*1	21.20	50.15	12.39	171.4	5.80	0.00240	0.00387	0.00880	0.00817	+0.2	0.2300	23.8	3.88	7.0
2	21.28	105.60	12.43	360.1	5.73	0.00234	0.00452	0.00943	0.00900	+0.9	0.1098	33.0	5.88	8.0
3	21.29	128.50	12.45	439.8	5.79	0.00232	0.00392	0.00789	0.00764	−0.1	0.0900	36.9	6.39	8.2
4	21.00	72.10	12.27	246.0	5.47	0.00360	0.00474	0.00991	0.00955	+0.3	0.1573	27.0	4.90	7.3
*K*1	21.04	84.91	12.30	292.2	5.36	0.00867	0.00256	0.00127	0.00283	−1.5	0.1338	25.2	6.21	8.6
2	20.83	44.86	12.17	154.3	5.37	0.00856	0.00417	0.00161	0.00362	+0.5	0.2509	23.1	3.60	6.3
3	21.32	29.01	12.46	99.8	5.40	0.00851	0.00514	0.00139	0.00386	0.0	0.3969	21.6	2.49	5.2
4	21.42	150.02	12.52	517.1	5.62	0.00913	0.00112	0.00057	0.00181	−5.0	0.0770	29.1	9.54	11.3
5	21.51	108.90	12.57	374.9	5.55	0.00907	0.00225	0.00162	0.00302	−1.7	0.1066	26.8	7.52	9.7
*F*1	56.23	15.44	32.88	52.8	5.94	0.00284	0.00306	0.00939	0.00875	−0.7	1.980	29.0	0.98	1.9
2	57.29	21.80	33.52	74.5	5.95	0.00306	0.00328	0.00909	0.00858	−0.3	1.435	25.3	1.58	2.5
3	21.28	47.40	12.42	161.9	5.88	0.00313	0.00364	0.00881	0.00856	0.0	0.244	61.4	1.42	2.6
4	21.26	76.79	12.43	262.9	5.71	0.00331	0.00376	0.00866	0.00851	+1.6	0.1504	94.4	1.50	2.3
*I*1	20.82	40.72	12.17	140.3	5.51	0.00906	0.00361	0.00010	0.00307	−4.0	0.2758	18.1	4.16	7.8
2	21.26	92.90	12.41	319.6	5.55	0.00898	0.00177	0.00115	0.00284	−1.3	0.1235	20.6	8.40	11.5
3	20.81	66.44	12.16	228.5	5.38	0.00870	0.00275	0.00168	0.00360	−1.6	0.1692	19.5	6.28	9.5
4	21.00	143.60	12.27	494.0	5.58	0.01031	0.00105	0.00101	0.00243	−2.6	0.0790	21.6	12.30	14.9
*J*1	75.36	139.60	44.05	480.0	5.68	0.01018	0.00480	0.00128	0.00429	−1.7	0.2925	20.3	12.70	6.9
*G*1	20.78	49.73	12.15	170.1	5.51	0.000761	0.00291	0.00858	0.00768	+0.1	0.2271	28.2	3.25	5.6
2	20.78	97.47	12.14	332.9	5.67	0.00152	0.00398	0.00956	0.00902	+0.2	0.1159	41.4	4.33	6.1
3	20.84	53.89	12.18	184.1	5.60	0.00220	0.00367	0.00927	0.00872	−0.1	0.2104	35.2	2.81	4.8
4	20.43	65.40	11.96	223.6	5.65	0.00259	0.00378	0.00901	0.00862	+4.0	0.1700	39.4	3.05	4.9
5	21.10	68.49	12.34	234.2	5.63	0.00296	0.00400	0.00930	0.00901	−0.3	0.1676	48.6	2.59	4.0
6	20.80	89.91	12.15	306.8	5.67	0.00326	0.00450	0.01031	0.00996	+0.4	0.1259	53.8	3.07	4.4
7	21.21	88.66	12.38	303.0	5.58	0.00295	0.00428	0.00997	0.00965	+0.7	0.1299	53.5	3.05	4.3
8	17.63	85.91	10.30	293.1	5.57	0.00326	0.00421	0.00952	0.00935	−0.2	0.1065	76.0	2.06	3.3
9	21.48	107.90	12.54	368.0	5.48	0.00289	0.00414	0.00935	0.00909	0.0	0.1083	57.6	3.44	4.4
10	20.93	82.12	12.22	280.3	5.51	0.00327	0.00441	0.01008	0.00978	+0.1	0.1387	53.3	2.84	4.1
11	21.88	99.24	12.78	338.9	5.53	0.00304	0.00392	0.00875	0.00857	−0.8	0.1199	55.7	3.28	4.3
12	21.49	31.48	12.55	107.6	5.14	0.00328	0.00389	0.00941	0.00897	+0.3	0.3711	29.6	1.95	3.7
13	21.08	57.55	12.32	196.2	5.77	0.00354	0.00433	0.00989	0.00964	−0.3	0.1996	47.9	2.21	3.9
14	21.49	29.52	12.32	100.7	5.58	0.00344	0.00437	0.00970	0.00906	−0.2	0.3889	17.3	3.19	6.7
15	20.47	55.54	11.96	189.6	5.65	0.00326	0.00446	0.00925	0.00885	−0.3	0.2006	32.4	3.14	5.4
*H*1	20.93	59.11	12.24	203.1	5.57	0.00894	0.00226	0.00374	0.00256	+5.4	0.1915	19.6	5.55	9.1
2	21.52	29.19	12.57	100.3	5.30	0.00855	0.00474	0.00167	0.00457	−1.3	0.3985	16.5	3.28	6.6
3	21.10	43.84	12.34	150.7	5.33	0.00904	0.00390	0.00190	0.00438	−0.5	0.2604	18.4	4.47	7.8
4	21.12	68.88	12.35	236.6	5.33	0.00940	0.00300	0.00218	0.00419	−1.1	0.1660	18.9	6.68	9.8
5	20.83	96.12	12.18	330.2	5.63	0 00802	0.00247	0.00336	0.00457	−0.4	0.1172	19.4	9.15	12.8
6	21.83	77.17	12.76	265.0	5.65	0.00958	0.00330	0.00352	0.00535	−1.1	0.1531	19.2	7.18	10.8
7	21.18	123.70	12.38	424.9	5.62	0.00946	0.00252	0.00372	0.00493	−0.6	0.0927	20.7	10.97	14.0

The results for the *G* runs in Figures 9 and 11, with solute transfer from the dispersed water phase to the continuous phase, show a lack of reproducibility in contrast to all the other curves, although the mass balance errors for all the runs are quite low.

Effect of the Plate Surface. If the *I* and *K* runs are compared (both have 4 1/2-in.-high upcomers and transfer direction continuous to dispersed, but the *I* runs were with the polyethylene plates and the *K* runs with mild-steel plates), it can be seen from Figure 10, showing efficiency E_A vs. V_D, that the efficiency at the same dispersed flow rate is about 25 to 30% higher for the runs with the polyethylene plates than with the metal plates. This increase in efficiency would probably be higher if a system had been used in which the droplets circulated; it has been shown (*6*) that the system toluene-diethylamine-water does not circulate under any conditions.

Typical photographs showing the performance of the polyethylene plates in the *G* and *I* runs and the mild-steel plates in the *L* and *K* runs are presented in Figures 3 to 8. The photographs are enlargements taken from 16-mm. cine film shots of the conditions existing during a particular run on the sixth plate from the base of the column.

It was not possible to determine accurately the number and size of the droplets in the column at any one time because the surface through which they were photographed was the curved column wall, which undoubtedly gave rise to distortion.

In Figures 3 to 8, the polyethylene plates give a continuous stream of relatively small droplets of fairly uniform size, but the metal plates produce "blobs of dispersed phase at regular intervals"; between these intervals a few droplets were formed, but they were quite irregular in size (*see* Figure 6, run *K* 4). Figures 3, 4, and 5 are with polyethylene plates and Figures 6, 7, and 8 with metal plates. The blobs of water phase that occurred with the metal plate underwent considerable turbulence as they fell from plate to plate. Some of the dispersed phase adhered to the central supporting rod and some fell down the column wall as a falling film. This unfortunately cannot be observed in the photographs as the continuous toluene phase at times became quite cloudy owing to the presence of water.

FIG. 3.

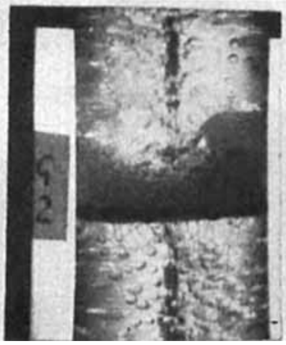

FIG. 4.

FIG. 5.

Effect of Transfer Direction. The direction of solute transfer will affect the resistance of the water film under certain conditions. Figure 11 shows a plot of the over-all volume mass transfer coefficient for the continuous toluene phase vs. the dispersed phase flow rate; the continuous rate was constant throughout except for run *J* 1. A similar plot can be drawn for K_Da as K_Da/K_Ca equals the distribution coefficient in volume units. The higher the value of K_Ca, the less the resistance of the controlling film. Both the polyethylene and metal plates show higher values of K_Ca for transfer from continuous to dispersed than in the opposite direction for the same upcomer size, the polyethylene plates having a higher K_Ca than the metal plates. The metal plates, however, appear more efficient than the polyethylene plates when the solute transfer is from the dispersed water phase to the continuous toluene phase as more turbulence and breakup of the dispersed phase is present with the metal plates, and this assists transfer although the area of contact is reduced. The plot of E_A vs. mL_D/L_C in Figure 9 also shows that the polyethylene plates are more suitable for water dispersion when the solute transfer is to the water phase.

Licht and Conway (*11*) have suggested that considerable hydrogen bonding exists between the solute molecule and the water molecules and that hence when the solute is put in the water phase (extraction *W* to *T*) orientation-and-hydrogen bonding takes place with the *NH* end of the diethylamine molecule in the water phase. When the solute is extracted from the water phase, the final break comes between the *NH* linkage and the *OH* groups of the water. If however the diethylamine is placed in the toluene phase (extraction *T* to *W*), no orientation occurs and the final separation involves overcoming of the Van der Waals forces between the organic part of the solute molecule and the toluene. As the forces of adhesion due to hydrogen bonding are more intense than those holding the organic end of the solute molecule and the organic phase together, transfer will be easier when water is the extracting phase. This is in agreement with the foregoing observations.

The Dispersed Phase. The work of Johnson and Blias (*8*) with a spray tower indicates that the phase receiving the solute should be dispersed for efficient operation. It will therefore be advantageous to operate the column at a high extraction factor by dispersing the water phase efficiently, with the use of polyethylene plates, and transferring the solute to the water phase. This results in higher solute transfer, as seen in the previous

FIG. 6.

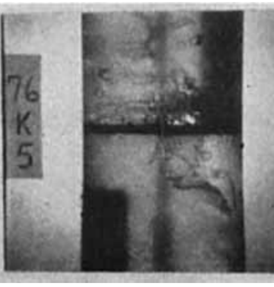

FIG. 7.

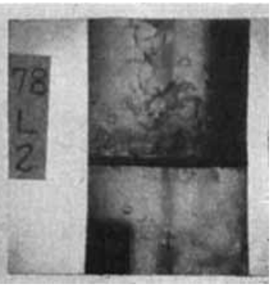

FIG. 8.

section on the direction of transfer.

Although droplet release was extremely good with the polyethylene plates in the *G* runs (*see* Figures 3 and 4), with transfer from the dispersed to the continuous phase, the metal-plate runs with the same transfer direction (*L* runs) exhibited higher efficiencies. The extraction factor was unfavorable for high solute transfer, and consequently the greater turbulence and shear of the water phase (in the *L* runs) due to the breakup of the blobs was more effective than the higher interfacial area obtained with good droplet release in the *G* runs.

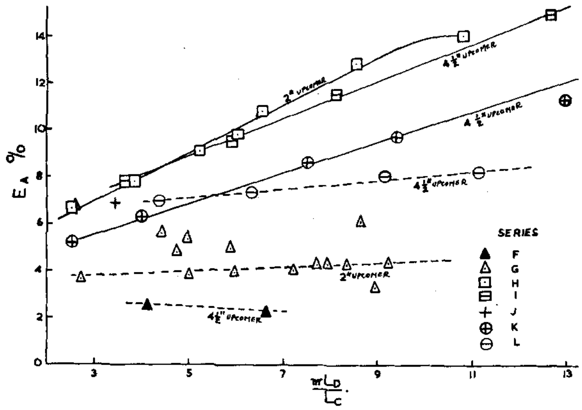

FIG. 9. EFFECT OF UPCOMER SIZE AND TRANSFER DIRECTION ON E_A ($L_C = 21$ FT./HR. EXCEPT *J*).

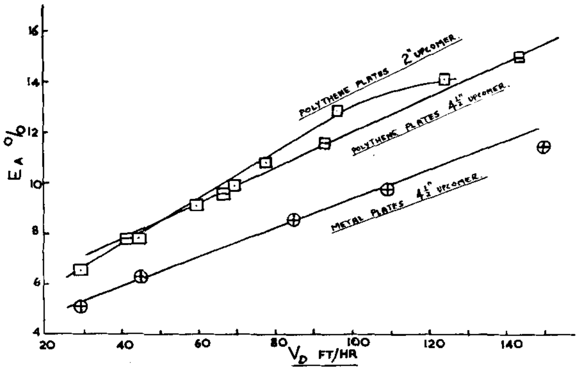

FIG. 10. EFFECT OF UPCOMER SIZE ON E_A ($L_C =$ 21 FT./HR.; WATER EXTRACTING).

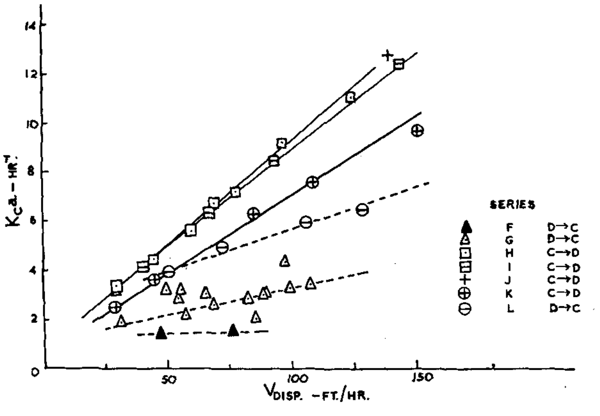

FIG. 11. EFFECT OF TRANSFER DIRECTION ON K_Ca AS A FUNCTION OF WATER RATE ($L_C = 21$ FT./HR. EXCEPT *J*).

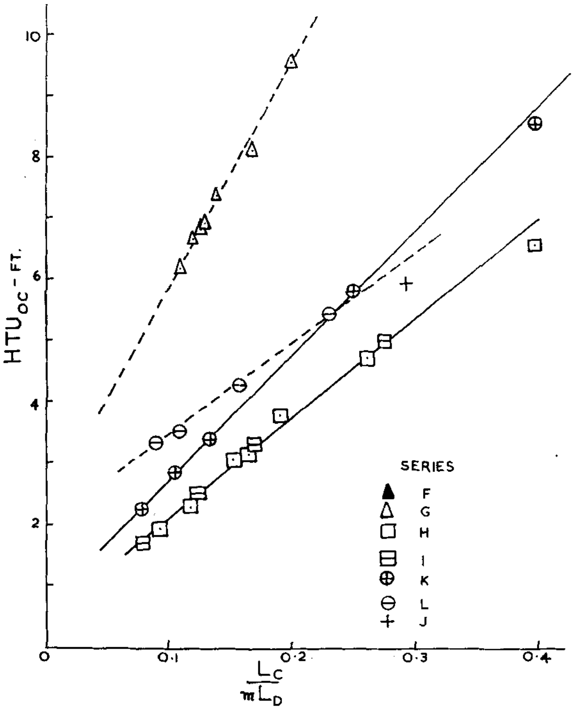

FIG. 12. H.T.U.$_{OC}$ VS. EXTRACTION FACTOR L_C/m_{LD} ($L_C = 21$ FT./HR. EXCEPT *J*).

Effect of Upcomer Height. Figure 10 indicates that the 2-in.-high upcomer is more efficient than the 4 1/2-in.-high one at high dispersed phase flow rates, but the effect is not so noticeable at the lower rates. This increase in efficiency is irrespective of transfer direction (*see* Figure 9 for transfer in the opposite direction with polyethylene plates). No comparisons were made for metal plates, as only one upcomer size was available. In Figures 9 and 10 run *H* 7 was on the verge of flooding, and hence the fall off in efficiency at high dispersed rates for the 2-in. upcomer with polyethylene plates.

The height of the upcomer has a direct bearing on the amount of turbulence and contact in the zone of free fall of dispersed phase between each plate. A short upcomer results in better counter current contact than does a large upcomer where the movement of continuous phase is nearly at right angles to the flow of dispersed phase; that is, the continuous phase moves across from plate to plate rather than vertically up the column. On these grounds it would be expected that a short upcomer would give more efficient operation than a large one.

H.T.U. DATA

As a matter of further interest the H.T.U.$_{OC}$ data for the runs are plotted in Figure 12 in the manner adopted by most workers(*1, 9, 15, 18, 22*).

A set of straight lines is obtained. For the *G* runs the line was selected from the line shown in Figure 11 in order to avoid scatter.

It has been the usual practice in the past to identify the slope and intercept of this line with the film values in accordance with the equation

$$\text{H.T.U.}_{OC} = \text{H.T.U.}_C + \text{H.T.U.}_D\left(\frac{L_C}{L_D}\cdot\frac{1}{m}\right) \quad (8)$$

In some instances however negative intercepts have been obtained (*9, 15, 18*), and the identification of the slope and intercept with the film values has been discussed(*5*).

It is interesting to note from Figure 12 that similar intercepts are obtained for different plate surfaces. For both polyethylene and metal plates the direction of mass transfer does influence the intercept and slope of the H.T.U.$_{OC}$ plot. Table 4 includes the slope and intercept data extrapolated from Figure 12.

TABLE 4

Plate type, water dispersed	Intercept	Slope	Transfer direction
Polyethylene..	0.55	15.6	$T \to W$
Metal........	0.70	20.1	$T \to W$
Polyethylene..	2.15	36.2	$W \to T$
Metal........	2.00	14.8	$W \to T$

Because the continuous phase flow rate was constant, both the slope and intercept appear to be independent of the dispersed phase flow rate. Garner, Ellis and Fosbury(*5*) found that the effects of the dispersed and continuous phase flow rates on H.T.U.$_{OC}$ were equal but operated in opposite directions.

CONCLUSIONS*

Pilot plant data for the system toluene-diethylamine-water at 20°C. are given for a 4-in. perforated-plate column using metal and polyethylene plates with the water phase dispersed and solute transfer in both directions. The column was operated under varying conditions of dispersed flow and a constant continuous phase throughput of 21 cu.ft./(sq.ft.) (hr.). The range of dispersed phase flow rates covered was 15 to 150 ft./(hr.). Stage-efficiency data vary from 2 to 15% and the H.T.U.$_{OC}$ data range from 2 to 50 ft.

The contact angles made by toluene and water droplets on a number of different materials in a surrounding continuous phase have been measured by the sessile droplet method. The surface of the perforated plate has been found to have a distinct influence on the operation of the plate column. The surface used for the plate must be hydrophobic when the water phase is dispersed, to ensure production of droplets from the plate surface at regular intervals. Polyethylene is suitable for this purpose.

The system toluene-water-diethylamine is noncirculating, and therefore the increase in efficiency obtained by the use of polyethylene plates instead of metal plates, which produce poor dispersions of water phase, is not so high as that expected from a system in which the droplets circulate. It is hoped in the future to investigate such a system.

With the diethylamine system the controlling resistance lies in the water phase when mole fraction units are employed, and it has been found that the efficiency of operation is higher when solute transfer is to the dispersed water phase rather than from the water phase, because of hydrogen bonding. The direction of mass transfer has also been shown to affect the value of the extraction factor significantly.

Single-line correlations have been obtained for the H.T.U.$_{OC}$ vs. L_C/mL_D plots, the slopes and intercepts of which depend on the direction of mass transfer and the plate surface used. Although the transfer direction affects both the slope and intercepts of these correlations, the type of plate surface affects only the slopes.

NOTATION

C_T, C_W = concentration of solute in the toluene and water phases, g.moles/ liter
E_A = average over-all stage efficiency, from N_A
H.T.U.$_{OD}$, H.T.U.$_{OC}$ = height of
H_{tOD}, H_{tOC} over-all transfer unit, based on dispersed and continuous phases, ft.
H.T.U.$_D$, H.T.U.$_C$ = height of film transfer unit, based on dispersed and continuous phases, ft.
K_{OC},K_{OD},$k_C k_D$ = over-all and individual extraction coefficients,lb.moles /(cu.ft.) (hr.) (lb. moles/cu.ft.)
$K_D a$,$K_C a$,$k_D a$,$k_C a$ = over-all and individual volume coefficients, lb. moles/(sq.ft.) (hr.) (lb.moles/ cu.ft.)
L_D, L_C = dispersed and continuous phase flow rate, lb.moles/ (sq.ft.) (hr.)
m = slope of the equilibrium distribution curve dy^*/dx
N_A = number of theoretical stages
N.T.U.$_{OD}$,N.T.U.$_{OC}$ = number of
N_{tOD}, N_{tOC} over-all transfer units
V_D, V_C = dispersed and continuous phase flow rate, cu.ft./ sq.ft.hr.
x = concentration of solute in the continuous phase, mole fraction
y = concentration of solute in dispersed phase, mole fraction
y^* = concentration of solute in dispersed phase, at equilibrium mole fraction

Subscripts

1 and 2 = column base and top concentration terminals

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*Since this paper was written, another paper on the subject has come to the authors' notice. R. H. Buchanan [*Australian J. Appl. Sci.*, 3, 233 (1952)] in his qualitative study of drop formation from wetted orifices has considered the effect of velocity and varying orifice materials. He concludes that strongly hydrophobic materials cannot be used to disperse a hydrophobic liquid or strongly hydrophilic materials a hydrophilic liquid.