

The Effect of Plate Wetting Characteristics on Pulse Column Extraction Efficiency

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Liquid-liquid extraction of acetic acid from the methyl isobutyl ketone-water system was studied as a function of plate wetting characteristics and other variables in a pulsed perforated-plate extraction column.

Various combinations of stainless steel plates and polyethylene plates were used with different directions of solute transfer at a constant throughput of 82.6 lb. total flow/(min.) (sq. ft. column area) and with other operating variables in the following range:

frequency	— 16 to 117.1 c./min.
amplitude	— 0.492 and 0.973 in.
W/K flow ratio	— 0.46 to 2.8 lb. water/lb. ketone

The plate wetting characteristics were found to affect the column extraction efficiency when the solute transfer was from the continuous ketone phase to water. An all polyethylene plate arrangement provided the best efficiency ($H. T. U_{oc} = 4.1$ in.) while an all stainless steel plate arrangement was less efficient ($H. T. U_{oc} = 6.1$ in.) under the most favorable operating conditions. A combination of these two arrangements in the column produced efficiencies midway between the all-plastic plate arrangement and the all-stainless steel plate arrangement.

Within the column flooding limits the extraction efficiency did not seem to be affected by the plate wetting characteristics when the solute transfer was from water to the continuous ketone phase.

Usage of liquid-liquid extraction pulse columns by industry has been on the upswing in recent years, especially in those connections related to the processing of nuclear fuels. Heretofore most of the information that has appeared in the literature on the pulsed extraction column has been concerned primarily with the evaluation of the column operating variables, such as flow rates, nature of continuous phase, pulse frequency, pulse amplitude, plate spacing, and plate geometry. It has been observed (1, 10, 11, 21) that the mutual wetting characteristics of the plates and column liquids have an effect on the extraction efficiency. Two articles have appeared (5, 8) in which the magnitude of this result was determined for the pulsed extraction column. Somewhat more information is available in the literature concerning wetting characteristics in packed or perforated plate nonpulsed extraction columns (5, 10, 11, 14, 15).

Since the two phases in liquid-liquid extraction possess different physical properties, it is feasible that the wetting characteristics of the plate would affect droplet formation. The frequent reformation of droplets of the dispersed phase in a pulse column emphasizes the significance of the relationship between drop formation and extraction efficiency. The importance of good droplet formation is significant because of the work of Sherwood, Evans, and Lorgeor (19);

Licht and Conway (13); and West (23). Sherwood and co-workers showed that up to 40% of the extraction by water of acetic acid from benzene drops occurred during the formation of the drop, although this amount of extraction has been shown to depend on many factors. Licht and Conway reported that the amount of acetic acid extracted during drop formation by use of hexone as solvent was 17%, and the values reported by West ranged from 14 to 20%. Even with the lower range of values the importance of drop size and formation is apparent.

Several attempts have been made to avoid large blobs and promote uniform droplet formation by some alteration of the plate construction or by coating the plates. The more apparent method is presented in a patent (5) by Burns and Johnson in which the plate consists of two layers of different materials, each material possessing different wetting characteristics. This principle along with the use of metal and plastic plates was included by Sege and Wedgfield (18) in their work on pulse-column variables. Their results indicated the effect of the wetting characteristics of a plate on extraction efficiency is dependent upon the direction of mass transfer, the choice of the continuous phase, and/or the ratio of the organic to the aqueous flow rates.

Garner, Ellis, and Hill (9, 10, 11) studied the wetting characteristics and performance of a nonpulsed perforated plate extraction column and found that

the character of the surface of the plate influences the performance of the column. They concluded that the best efficiency was obtained when the continuous phase wetted the plate and the dispersed phase did not wet the plate. Thus when the water phase is dispersed, it was concluded that the plate should be hydrophobic in nature.

Buchanan (3) in his qualitative study of drop formation from wetted orifices concluded that strongly hydrophobic material cannot be used to disperse a hydrophobic liquid nor strongly hydrophilic materials a hydrophilic liquid.

Based upon the results of these investigators concerning the effect of plate wetting characteristics it seemed desirable to investigate more thoroughly the effect of the use of plates with different wetting characteristics in a pulse column, and especially the effect of combinations of these two types of plates in the column. If the results of Buchanan could be applied to the perforated plate pulsed extraction column, then the possibility existed of interchanging the discontinuous and continuous phases within the column at various heights without local flooding by using various combinations of plates having hydrophobic or hydrophilic surfaces.

The possibility also existed that if the plate with the best wetting characteristics could be located at a particular place within the column, such as at a pinch zone, it might permit the use of other plates in the rest of the column. This could be very advantageous if the cost of the plates were different or if an entire column of a particular type of plate could not be used because of structural strength, radiation damage, or similar design problems.

Based on this background information the objectives of this investigation were to determine the following: What is the magnitude of the effect of stainless steel vs. plastic plates under different operating conditions on extraction efficiency? What is the effect of com-

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binning these two plate materials in various arrangements within the column?

EXPERIMENTAL APPARATUS

To accomplish the two objectives it was necessary to reduce the number of variables to a reasonable number. The factors which have been noted by various workers to affect the extraction efficiency of a pulsed column are geometry factors: plate hole diameter, percent free plate area, column diameter, plate spacing; pulse factors: amplitude, frequency, pulse shape or form; other factors: flow rates, ratio of flow rates, physical properties of the system, choice of continuous phase, direction of mass transfer.

The first three geometry factors were fixed at what seemed to be an optimum level based on information found in the literature by selecting plates 1.98 in. in diameter with forty-eight $\frac{1}{8}$ -in. holes giving a free area of 18.7%. The column itself was a standard 2-in. Pyrex pipe combined with 3-in. disengaging sections at top and bottom. A very limited investigation indicated that small changes in plate spacing of about $\frac{1}{2}$ -in. in range did not seem to affect the best height of a transfer unit value appreciably, and the plate spacing was subsequently held constant at $\frac{1}{2}$ -in. The pulse form used was approximately sinusoidal and consistent. To compensate somewhat for holding the plate spacing constant two levels of amplitude were used, $\frac{1}{2}$ in. and 1 in. The frequency was varied to give a $a \times f$ ratios from 10 to 70 and yet stay within the flooding limits.

The total flow rate has been shown to have little effect on extraction efficiency and was held constant at values ranging between 1.73 to 1.81 lb./min. Only one system was studied, water-methyl isobutyl ketone-acetic acid. The other factors were varied, the ratio of flow rates being changed over sixfold.

A schematic diagram of the extraction unit composed of a perforated-plate column, rotameters, pumps, storage tanks, and other auxiliary equipment is shown in Figure 1. The position of the interface was maintained by adjusting the outlet water flow or inlet ketone rate.

EXPERIMENTAL PROCEDURE

Materials used were acetic acid, distilled water, and bulk methyl isobutyl ketone.

The column was operated both as an extraction column to remove acetic acid from water with ketone as the solvent and also as a stripping column to remove the acetic acid from the ketone by using water as a stripping medium. Feed to the extraction column was about 12 wt. % acetic acid in water, while the ketone stream leaving the top of the column contained about 7% acetic acid. Some of the aqueous raffinate from the extraction operation (containing about 2% acetic acid) was diluted with distilled water and used to strip acetic acid from the ketone. The aqueous extract, about 9% acetic acid, was collected and brought back up to strength to be used as feed for the next extraction run. This type of operation was selected to conserve acetic acid and also permit the evaluation of the effect of direction of mass transfer on extraction efficiency.

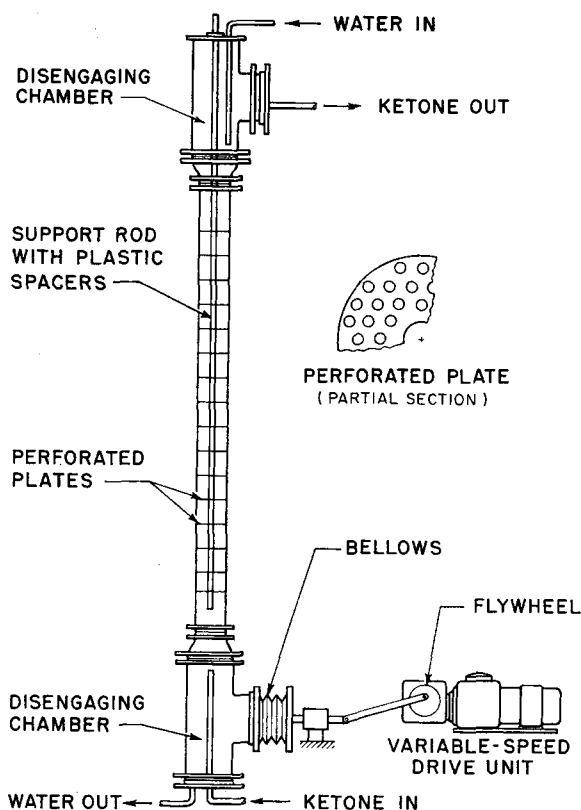


Fig. 1. Pulse-column diagram.

During each run the interface was maintained at the selected position in the column (top of the column or bottom of the column, depending upon which phase was continuous) by adjusting the outlet water flow rate or the inlet ketone rate. Steady state operation was determined by taking repeated samples of the concentration of the outlet streams and noting when they reached their asymptotic values. The time to achieve steady state operation was about 25 min., but runs of 1 hr. were made to insure that the steady state really had been achieved and was being maintained. Operating variables in the column were changed when one series of runs with one plate arrangement had been completed.

Acid concentrations were determined by titration with standard aqueous sodium hydroxide and hydrochloric acid to a phenolphthalein pink end point. The error in the analysis was approximately 1% or less. Flow rates were determined by measuring the time required to collect approximately 250 g. of the stream flowing. Although the pulsing effect on the flow rates may have introduced an error up to 5%, the flow rates were not used in determining the slope of the operating line, but only to check the material balance in the column.

Equilibrium data for this system (Table 1) were determined at the average temperature of the experiments (90°F.) by mixing together measured amounts of acetic acid, water, and ketone. The equilibrium solubility data determined in this investigation were in agreement within 2% of that of references 2, 17a, and 19 but did not agree as well with the data recorded at higher concentrations in reference 16 or agree well at all with the data of reference 16a.

CALCULATION OF RESULTS

The measure of extraction effectiveness for this investigation could have been expressed in terms of the height equivalent to a theoretical stage, the height of a transfer unit, or as an overall mass transfer coefficient. The $H.T.U.$ and K_a are derived to represent the actual extraction operation which is a more or less a true countercurrent operation. The $H.E.T.S.$ is derived to represent a step-by-step operation in which most of the mass transfer occurs at equilibrium stages or plates, but if the number of theoretical plates is large and the concentration change per plate is small, then the $H.E.T.S.$ is without too great an error applicable to a countercurrent extraction operation.

The pulsed extraction column tends to operate somewhere between the two conditions of continuous contact and step-by-step mass transfer. At the low pulse velocities the liquids coalesce between pulses, and most of the solute transfer probably occurs in the region near the plate during each pulse. However at the higher pulse velocities the droplets do not coalesce between pulses and are in contact with the solvent between plates, thus tending to approach countercurrent contact.

Because for this experiment only the relative values of the extraction efficiencies were required, and because most of the previous articles in the liter-

TABLE 1. EQUILIBRIUM DATA FOR THE METHYL-ISOBUTYL-KETONE, WATER, ACETIC ACID SYSTEM AT 90°F.

X	Weight ratio acid	Y
(in water)		(in ketone)
0.04209		0.02641
0.06267		0.04062
0.06417		0.04171
0.07239		0.04757
0.1093		0.07542
0.1105		0.07660
0.1356		0.09649
0.1732		0.1276

TABLE 2. PLATE ARRANGEMENT AND LOWEST VALUES OF $H.T.U._{oc}$

Plate arrangement	Direction of solute transfer	$H.T.U._{oc}$, in.	
		Ketone continuous	Water continuous
All-stainless steel	K → W	6.1	6.6
	W → K	5.6	—
All-plastic	K → W	4.1	5.8
	W → K	5.7	—
Alternating sections	K → W	5.0	6.3
Half-plastic on top	K → W	5.0	
Half-stainless steel on top	K → W	5.1	

ature had employed the $H. T. U.$, this same choice was adopted as the means of reporting the results of this investigation, although some comparisons are made below among all three methods. The continuous phase was arbitrarily chosen as the basis for the $H. T. U.$ and the Ka .

Since the experiment was performed with relatively dilute concentrations, and the ketone and water were mutually saturated before each run, the flow rates of both phases were assumed constant throughout the column, and consequently the operating line could be considered to be straight. From a material balance on the column the equation for the operating line is

$$Y = \frac{W}{K} (X - X_1) + Y_1 \quad (1)$$

The theoretical background of the extraction efficiency calculations are well known (20, 22). The $H. E. T. S.$ and $H. T. U.$ are calculated by dividing the effective column height by the number of theoretical plates or the number of transfer units respectively.

Equations (2) and (3) are most commonly used for calculating the number of transfer units:

$$N_{t,ok} = \int_{Y_1}^{Y_2} \left[\frac{dY}{Y - Y^*} \right] + \frac{1}{2} \ln \frac{1 + rY_1}{1 + rY_2} \quad (2)$$

$$N_{t,ok} = \frac{1}{\left(1 - \frac{mK}{W}\right)} \ln \left[\left(1 - \frac{mK}{W}\right) \left(\frac{Y_1 - mX_2}{Y_2 - mX_2} \right) + \frac{mK}{W} \right] \quad (3)$$

Equation (2) is considered to be the rigorous method for determining N_t , whereas Equation (3) is derived with the assumptions of a straight operating line, a straight equilibrium line, and dilute solutions.

For the system studied it was found that the equilibrium line was somewhat curved (its slope varied from 0.62 to 0.78) and that values of the $H. T. U.$ calculated by the use of Equation (3) were in error by as much as 30% from the value found by graphically evaluating the integral in Equation (2). To

avoid the rather time-consuming calculations involved in a graphical integration for each run a graphical procedure was used to step off the number of theoretical stages, and this was used together with the column height to calculate the $H. T. U._{oc}$ by using Equation 4:

$$H.T.U._{oc} = \frac{(H.E.T.S.) \left(1 - \frac{mK}{W}\right)}{\ln \left(\frac{W}{mK}\right)} \quad (4)$$

Although Equation (4) also includes the assumptions of a straight operating line, a straight equilibrium line, and dilute solutions, it was found by comparing $H. T. U.$'s calculated by Equation (2) with those calculated with Equation (4), that if the average slope of the equilibrium line was determined by the method of Pike (17), the $H. T. U.$ values calculated in this way were within 3% of the values obtained by using Equation (2) as long as the W/K ratio was less than 1. For the W/K ratios larger than 1 the error was found by a similar procedure to be within 7%, which was accurate enough to define the effect of W/K on extraction efficiency.

The over-all mass transfer coefficient can be calculated by the use of Equation (5):

$$K_0 a = \frac{L'_i}{\rho_i H. T. U'_{oi}} \quad (5)$$

The height of the column used in the above calculations was measured as the distance between the top plate and the interface at the bottom for runs when the ketone was continuous, or from the bottom plate to the top interface when the water phase was continuous.

RESULTS AND DISCUSSION

The experimental results* obtained from the operation of the column are presented graphically in Figures 2, 3, 4, and 5. A summary of the lowest values of the $H. T. U._{oc}$ experienced for each of the various plate arrangements and directions of solute transfer is shown in Table 2.

* The original experimental data are available in reference 20a.

The accuracy of the results was evaluated by comparing the calculated $H. T. U.$'s for several runs that were repeated. The deviation between the repeated runs at identical conditions (including the net effect of errors in analysis, in equilibrium line slopes, and in measured column heights) varied from 0.14 to 0.31 in., the average deviation being 0.24 in. The estimated average over-all error in the $H. T. U.$ values, including the errors just mentioned plus the calculational errors discussed in the previous section, was 0.38 in. (about 10%). The experimental results will now be discussed in terms of the effect of the different variables on the pulse column extraction efficiency.

Pulse Velocity

The variation of the extraction efficiency as a function of the pulse velocity (amplitude-frequency product) is shown in Figure 2 for the five various plate arrangements investigated. The water to ketone flow rates were maintained at 0.75 lb./lb. for the extraction of acetic acid from ketone to water.

The curves are consistent in shape and in general agreement with the curves obtained by most of the previous investigators in that the $H. T. U.$ decreases (increasing extraction efficiency) as the pulse velocity is increased until the point is reached where a further increase in pulse velocity does not increase the efficiency, and in most cases actually decreases the efficiency.

The most vigorous pulsing has been shown (8) to increase the amount of back mixing, approaching the operation of a baffled mixer with countercurrent flow superimposed on this concurrent mixing. The curves showing the variation of $H. T. U.$ as a function of the pulse velocity probably represent the sum of two opposing effects. The increased pulsation causes an increase in dispersion and turbulence, resulting in better extraction efficiency. This higher pulsation also increases the amount of back mixing which tends to decrease the extraction, thus resulting in the leveling off or slight increase of $H. T. U.$ at the higher pulse velocity.

Observation of the column operation indicated that at the lower pulse velocities the column was operating in the

relatively inefficient mixer settler type of operation in which the phases tended to coalesce between pulses. As the frequency was increased, the dispersion of the liquids was gradually increased until a point was reached where the liquids were very finely dispersed. This point corresponds to the best efficiency. As the pulse velocity was increased further, the droplets seemed to be passing through the plates without being able to coalesce or be redispersed. Finally an emulsion would be formed that would take as long as 30 sec. to break after the apparatus was stopped.

The method of measuring the column height probably contributed somewhat to the apparent increase in $H. T. U.$ at the higher pulse velocities. For most of the runs when the ketone was continuous, the interface was about 3 in. below the bottom plate, and the effective heights of the column were similar for each run. However for a few runs at the higher pulse velocities, an emulsion or near emulsion would build up below the bottom plate; to prevent the emulsion volume from rising into the perforated-plate section the interface was lowered, sometimes to as much as 15 in. below the bottom plate, and hence a longer effective column height would be reported. Although this extra height should certainly have contributed to mass transfer, it was probably not as effective as an equal length of column filled with plates. Thus in a few cases the column height reported was too large by an undetermined amount and caused the calculated extraction efficiencies to be lower than might be determined for the same type of extraction from a long column in which the end effects were minimized. This minor uncertainty in no way alters the conclusions drawn in the discussion below.

Plate Arrangement

The plate arrangement with the all-plastic plates was the most efficient arrangement used. The arrangement with half-plastic plates and half-stainless steel plates (including the alternating sections) each had about the same efficiency and were intermediate between the all-plastic arrangement and the all-stainless steel arrangement, which was the most inefficient. The best $H. T. U.$'s obtained with the various arrangements are summarized in Table 2. The runs with the water phase continuous were found to be more inefficient at the same ($\alpha x f$) values than with the ketone phase continuous. Therefore most of the runs were made with the ketone phase continuous.

One possible reason the combination of plate materials did not give better $H. T. U.$ values than for the all-plastic plates is that the stainless steel plates did not appear to be as easily wetted

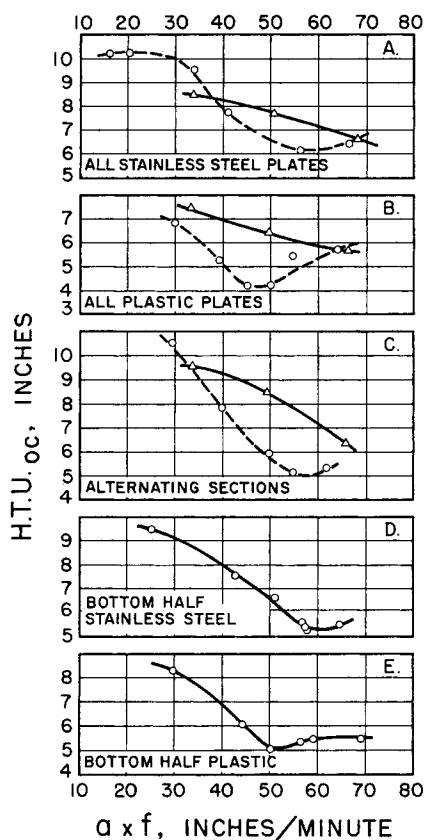


Fig. 2. Effect of plate arrangement on extraction efficiency, direction of solute transfer—ketone to water: W/K ratio: 0.75 \circ -ketone continuous, Δ -water continuous.

by water as the plastic plates were wetted by the ketone. The plastic plates were wetted by the ketone so well that a layer of ketone adhered to them even under conditions of high pulsation and with the water phase continuous. Thus the phase not wetting the

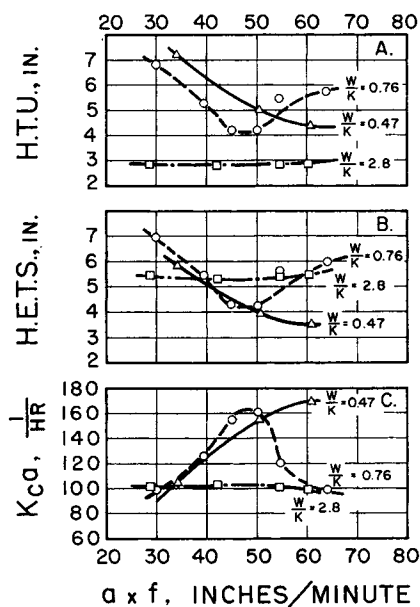


Fig. 3. Effect of W/K ratio on extraction efficiency for polyethylene plates, direction of solute transfer: ketone to water—continuous phase: ketone Δ - $W/K = 0.47$, \circ - $W/K = 0.76$, \square - $W/K = 2.80$.

plate only contacted the wetted phase and not the plate itself.

Flow-Rate Ratio

The flow ratio was varied to find out how it might affect the extraction efficiency for the different plate arrangements. The total throughput was held constant, and the water and ketone flow rates were changed. The trend as shown in Figures 3 and 4 was the same for the two arrangements on which the ratio was varied, and it was concluded that the extraction efficiency could be expected to vary in the same way with changes in flow ratio for the other plate arrangements.

To illustrate that the method of presentation of results does not affect the conclusions reached so far, the results presented in Figures 3 and 4 were calculated in terms of $H. E. T. S.$, $H. T. U.$ based on the continuous phase, and the over-all mass transfer coefficient. Each of these methods (for different plate arrangements) indicated the same trends of extraction efficiency as the pulse velocity was varied. Of course the results presented here concerning plate arrangement depend upon the system used and are not necessarily applicable to a different system.

It should be noted that the effect of the W/K ratio on the $H. T. U.$ is dependent upon whether the $H. T. U.$ is based on the continuous or discontinuous phase. For the runs shown in Figures 3 and 4 (ketone-phase continuous) the $H. T. U._{\infty}$ and $H. T. U._{\infty}$ are related by Equation (6):

$$H. T. U._{\infty} = \frac{W}{mK} H. T. U._{\infty} \quad (6)$$

If the results had been reported as $H. T. U._{\infty}$, the curve for W/K of 2.8 would have had the highest $H. T. U.$, and the inverse of the reported trend of W/K on extraction efficiency would have been described.

The $H. E. T. S.$, which is not based upon a single phase, tends to indicate an over-all change for both phases together, and the magnitude of the $H. E. T. S.$ roughly corresponds to the average of the $H. T. U._{\infty}$ and $H. T. U._{\infty}$ values for a given run. The K_a is based on an individual phase, and it exhibits the same trends as the $H. E. T. S.$ since the K_a value is adjusted somewhat for changes in flow rates, as shown by Equation (5).

Phase Reversal

Phase reversal within the column was not obtained for the plate arrangements and operating conditions investigated. The flow ratio was varied in an attempt to cause phase reversal of the continuous phase, but even at very high or low water to ketone flow ratios there was little, if any, tendency for the con-

tinuous phase to reverse. If a plate with a more hydrophilic surface had been used, it might have been possible to reverse the phases, but it was not possible with the stainless steel-plastic plate combinations. It is doubtful if any advantage could accrue even if the phase reversal had been accomplished, since the runs with the water phase continuous gave high values of $H. T. U.$ as compared with the runs where the ketone was continuous (at the same levels of pulsation).

Direction of Mass Transfer

As may be seen from Figure 5 the direction of mass transfer had a very pronounced effect on the $H. T. U.$, especially for the same pulse velocity. The best $H. T. U.$ for the stainless steel plates did not change appreciably with direction of solute transfer, although much more energy was required to obtain the same $H. T. U.$ for the water to ketone runs.

This is in accordance with the explanation (13) that hydrogen bonding between the carboxyl groups of the solute molecules and the hydroxyl group of the water molecule may cause a difference in extraction efficiency, depending upon the direction of solute transfer. When the acetic acid is dissolved in the ketone, the surface molecules have no special orientation, and as the acid is extracted from the ketone into water, the water exerts a dissolving action on the acid molecules because of the formation of hydrogen bonds with the carboxyl group of the acid molecules.

In the case of extraction of acetic acid from water the opposite effect exists in which these forces of adhesion caused by hydrogen bonding must be overcome. This was indicated by the higher pulse velocity required to decrease the $H. T. U.$ to the range obtained for the extraction of acid from ketone to water. For each type of plate arrangement when the transfer was from water to ketone the column flooded before enough energy could be supplied to reach the minimum $H. T. U.$ value.

There was also an obvious difference in droplet formation depending upon the nature of the continuous phase. The ketone drops coalesced very quickly in the water phase when the water was continuous, while the water in the ketone did not coalesce as rapidly and, in fact, the drops would often not coalesce at all until they reached the interface. In the case of the water the drops were very small and tended to remain dispersed until they had settled to the interface where they then coalesced. A difference in the tendency to coalesce was also noted between the dilute and concentrated ends of the column.

The total acid content in the column was not appreciably different for the

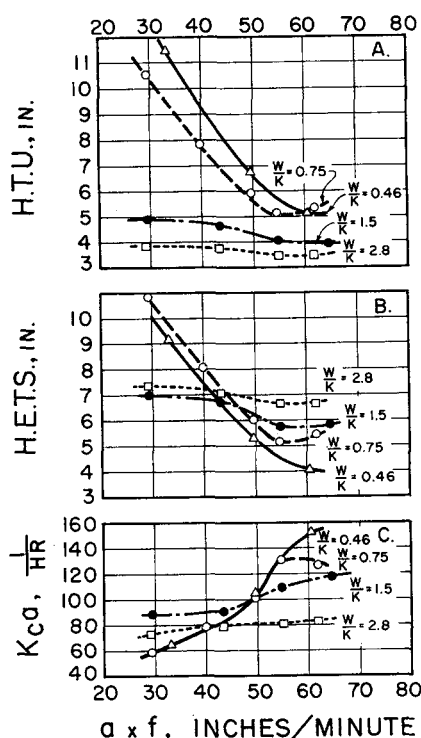


Fig. 4. Effect of W/K ratio on extraction efficiency for alternating sections, direction of solute transfer: ketone to water continuous phase: ketone \triangle - $W/K = 0.46$, \circ - $W/K = 0.75$, \bullet - $W/K = 1.5$, \square - $W/K = 2.8$.

water to ketone or ketone to water runs, although the concentration was naturally different between the ends of the column. It has been noted that the efficiency increases with an increase of acid concentration (12, 13), most likely caused by the fact that the addition of acid lowers the interfacial tension between the phases (6), thus permitting better dispersion of the liquids.

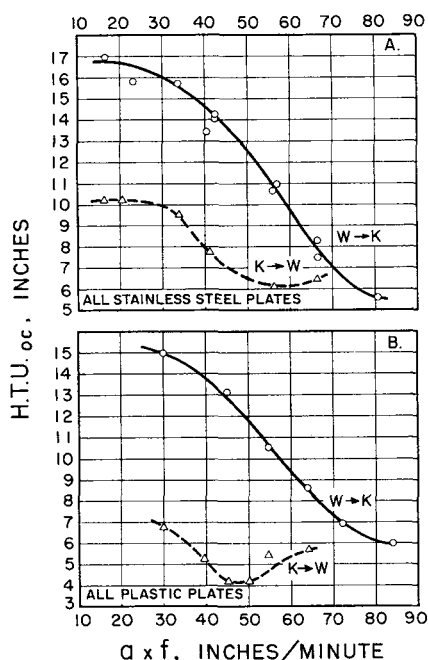


Fig. 5. Effect of direction of solute transfer on extraction efficiency, continuous phase: ketone — W/K ratio: 0.75.

Comparison of Results

Only two articles were found which used the system employed in this work in a pulsed column. Even then the operating conditions and design variables were different and did not afford a common basis for comparing results. Belaga and Bigelow (2) obtained $H. T. U.$ values based on the continuous ketone phase with solute transfer from the ketone to water but with a different plate geometry, plate spacing, and column size. Their range of $H. T. U.$ was 2.2 to 6.3 in. for a W/K ratio of 1.0. Burkhart and Fahien (4), in their study of five pulse column variables, presented two combination of variables which were similar to the design variables and operating conditions used in this work. For a W/K ratio of 1.0 and the water phase continuous they obtained $H. T. U.$ values of 6.0 to 8.0 in., which is in the range of the $H. T. U.$ obtained this work with water as the continuous phase.

ACKNOWLEDGMENT

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NOTATION

- a = pulse amplitude, in.
- f = pulse frequency, min^{-1}
- $H.E.T.S.$ = height equivalent to a theoretical state, in.
- $H.T.U.$ = over-all height of a transfer unit based on the phase indicated by the subscript, in.
- $H.T.U.'_o$ = over-all height of a transfer unit based on the phase indicated by the subscript, ft.
- k = total ketone flow rate, lb./min.
- K = solute-free ketone flow rate, lb./min.
- Ka = over-all mass transfer coefficient based on the phase indicated by the subscript, hr^{-1}
- L' = phase flow, lb./hr.-sq. ft.
- L'_i = solute-free flow rate of phase i , lb./hr.-sq. ft.
- \ln = natural logarithm
- m = slope of the equilibrium-distribution curve = dY^*/dX
- N_p = number of theoretical plates, dimensionless
- N_t = over-all number of transfer units based on the phase indicated by the subscripts, dimensionless
- r = molecular weight nonsolute/molecular weight solute
- W = solute-free water flow rate, lb./min.
- X = concentration of solute in water phase, lb. solute/lb. nonsolute
- Y = concentration of solute in ke-

tone phase, lb. solute/lb. nonsolute
 Z = effective column height, in.
 ρ_i = density of liquid i , lb./cu. ft.

Subscripts

c = continuous phase
 d = dispersed phase
 i = indicates either phase can be used throughout the equation where i appears
 k = ketone phase
 w = water phase
 1 = bottom of the column
 2 = top of the column

Superscript

* = equilibrium concentration

LITERATURE CITED

- Ballard, John H., and Edgar L. Piret, *Ind. Eng. Chem.*, **42**, 1088 (1950).
- Belaga, M. W., and J. E. Bigelow, *U.S. Atomic Energy Commission Report KT-133* (1952).
- Buchanan, R. H., *Aust. J. Appl. Sci.*, **3**, 233 (1952).
- Burkhart, L. E., and R. W. Fahien, *U.S. Atomic Energy Commission Report ISC-860* (1956).
- Burns, W. A., and W. F. Johnson, *U.S. Patent 2 662 001/1953*.
- Cervi, A. F., Thesis, Columbia University, New York (1943).
- Choffe, B., and Y. L. Gladel, *J. Appl. Chem.*, **8**, 580 (1958).
- Cohen, R. M., and G. H. Beyer, *Chem. Eng. Progr.*, **49**, 279 (1953).
- Garner, F. H., S. R. M. Ellis, and D. W. Fosbury, *Trans. Inst. Chem. Engrs. (London)*, **31**, 348 (1953).
- Garner, F. H., S. R. M. Ellis, and J. W. Hill, *A.I.Ch.E. Journal*, **1**, 185 (1955).
- , *Trans. Inst. Chem. Engrs. (London)*, **34**, 223 (1956).
- Johnson, H. F., and Harding Bliss, *Trans. Am. Inst. Chem. Engrs.*, **42**, 331 (1946).
- Licht, William, and J. B. Conway, *Ind. Eng. Chem.*, **42**, 1151 (1950).
- Livingston, M. J., *U.S. Patent 2 215 359/1940*.
- Mayfield, F. D., and W. L. Church, *Ind. Eng. Chem.*, **44**, 2253 (1952).
- Oldshue, J. Y., and J. H. Rushton, *Chem. Eng. Progr.*, **48**, 297 (1952).
- Othmer, D. F., R. E. White, and E. Treuger, *Ind. Eng. Chem.*, **33**, 1240 (1941).
- Pike, F. P., *U.S. Atomic Energy Commission Report ORNL-929*, (1951).
- Scheibel, E. G., and A. E. Karr, *Ind. Eng. Chem.*, **42**, 1048 (1950).
- Sege, G., and F. W. Woodfield, *Chem. Eng. Progr.*, **50**, 396 (1954).
- Sherwood, T. K., J. E. Evans, and J. A. Longcor, *Ind. Eng. Chem.*, **31**, 1144 (1939).
- Sherwood, T. K., and R. L. Pigford, "Absorption and Extraction," McGraw-Hill, New York (1952).
- Sobotik, R. H., M. S. thesis, Univ. Tex., Austin, Texas (1959).
- Treybal, R. E., *Ind. Eng. Chem.*, **34**, 709 (1942).
- , "Mass-Transfer Operations," McGraw-Hill, New York (1955).
- West, F. B., P. A. Robinson, A. C. Morgenthaler, Jr., T. R. Beck, and D. K. McGregor, *Ind. Eng. Chem.*, **43**, 234 (1950).

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Effects of Solids on Turbulence in a Fluid

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The effect of solid particles on fluid turbulence was studied for fully developed flows of slurries in a vertical 3-in. pipe for solids concentrations ranging from 0.13 to 2.5 volume %. Point source turbulent diffusion data in the slurry flows were compared with data for flows without solids present. The solids do not appear to have a large effect on the diffusion rate unless there is an appreciable average slip velocity between the solids and the fluid and unless the solids concentration is high enough.

A theoretical treatment of turbulent flow involving the suspension of solids in a fluid would use information on turbulent flow of single-phase systems as a starting point. The question arises whether the presence of the solids affects the fluid turbulence. Very little information is available in the literature on the interaction of solid particles with a turbulent field; therefore this study was undertaken to gain some insight regarding the conditions under which the presence of solid particles would change the fluid turbulence. In particular the effect of solids on the rate of diffusion in a fluid was studied by comparing data on point source turbulent diffusion with and without solids in the field. The technique is similar to that used in a previous study of turbulent diffusion in fluidized beds (4).

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Water and solid-water suspensions were circulated through a 3-in. vertical pipe. A 1N solution of potassium chloride was injected through a small tube in the center of the pipe at a sufficient distance from the pipe inlet such that the flow was fully developed. Samples were withdrawn from the flow over a period of time, long compared with the time scale of the turbulence. The samples were taken across diameters at several distances from the plane of the injector. The spread of the material at a plane downstream from the injector could be measured by the mean square of the x -component of the displacement of the diffusing particles, $\overline{x^2}$. The variation of $\overline{x^2}$ with distance from the injector is a measure of the rate of diffusion. The variation of $\overline{x^2}$ at large times measures the gross properties of the turbulent mixing process and is influ-

enced by the largest eddies. The effect of the presence of solid particles on such measurements reflects the interaction of the particles with the large scale fluid eddies.

During the course of this investigation Soo (11) reported results on a similar study. He carried out measurement in a horizontal pipe on the diffusion of helium gas in air and in suspensions which contained 0.0005 to 0.0030 volume % glass spheres of diameter less than 0.01 in. He found that the presence of the solids did not affect the diffusion in the fluid phase. The volume percent and the type of solids which Soo could investigate were limited by fall out of the particles from the flow stream. By carrying out experiments in a vertical pipe larger solids concentrations and larger particle densities can be investigated. For flow in a vertical pipe a region far from the entry