NOTATION

C_r = fraction of carbon on regenerated catalyst
C_s = fraction of carbon on spent catalyst
D_p = average particle diameter representing specific surface of catalyst, μ
f = fraction of inlet oxygen unconverted in regenerator (N/N_0)
F = rate of gas feed to regenerator, lb./hr.
G = gas mass velocity in regenerator, lb./(hr.)(sq. ft.)
k_d = coefficient of mass transfer, lb. mole/(hr.)(atm.)(lb.)
k_r = specific reaction velocity constant, lb. mole/(hr.)(atm.)(lb.)
m = exponent on G
n = exponent on D_p
N = oxygen concentration in gas, lb. mole/lb. feed
N_0 = inlet oxygen concentration in gas, lb. mole/lb. feed
N_T = total moles gas/lb. feed to regenerator
p = partial pressure of oxygen in bubbles, atm.
p_i = partial pressure of oxygen in interstices of catalyst, atm.
P = total pressure, atm.
r = over-all rate of reaction of oxygen, lb. mole/(hr.)(lb.)
r_d = rate of diffusion of oxygen from bubbles to catalyst, lb. mole/(hr.)(lb.)
r_r = rate of reaction of oxygen at surface of catalyst, lb. mole/(hr.)(lb.)
R = carbon burning rate, lb./(hr.)(100 lb. catalyst)
S = space velocity, moles/(hr.)(lb.)
T = catalyst temperature,°F.
U = catalyst circulation rate, lb./hr.
W = catalyst inventory, lb.

Greek Letters

α = a proportionality constant, slope of Equation (8)
β = a proportionality constant in Equation (10)

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Performance of an Internally Baffled Multistage Extraction Column

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Internally agitated extraction columns generally require increasing heights for a theoretical stage for the larger column diameters. The present work describes a design developed to minimize this objection and presents the performance data on a system considered easy to extract and on systems considered difficult to extract. H.E.T.S. values as low as 3 in. on the first system and 4 in. on the second type of system were obtained in an 11½ in. I.D. glass column. The stage efficiencies were correlated as a function of power input per unit volume of solvent throughput and the ratio of the flow rate of the dispersed phase to that of the continuous phases. The supplementary effect of packing was studied and found to be most beneficial in the system which has a low interfacial tension and is considered easy to extract. By the use of this particular arrangement of internal baffles, it is believed possible, in the larger diameter columns, to reduce the height to a ratio of H.E.T.S. to diameter below the value of ¼ obtained in the present column.

The performance studies on mechanically agitated liquid-liquid extraction columns generally indicate that the height required for a theoretical stage increases as the diameter of the column becomes greater although not necessarily in a direct proportionality. In the design of extraction columns consisting of alternate mixing sections and packed calming sections, it was found that the optimum packing height which gives the minimum H.E.T.S. values was approximately proportional to the square root of the column diameter for the 1- and 12-in. sizes. Early studies on the use of vertical baffles in the mixing sections of such a column indicated that although these baffles eliminated the rotational motion of the fluids and probably improved the mixing, they transformed this motion into vertical motion and required an even greater

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amount of packed height to isolate the flow patterns of the individual mixing sections. It then became obvious that horizontal baffles were essential for the best performance of an internally agitated extractor. Oldshue and Rushton (*4*), using a flat-bladed turbine to promote the mixing, studied the performance of a column with annular horizontal baffles.

Reman (*5* and *6*) presented performance data on a similar column with a rotating disk used in place of the turbine for the agitating means. On an easy extraction system such as methyl isobutyl ketone–water–acetic acid, an 8-in.-diam. column gave a theoretical stage in as little as 5 in. of height. This was in substantial agreement with the performance of the Oldshue and Rushton column on the same system. On a difficult extraction system such as water–kerosene–butylamine, a 16-in.-diam. extractor required more than 2 ft. for a theoretical stage. A 4-in.-diam. column of the same design gave substantially the same stage height and on this basis Reman and Olney (*6*) postulated that there is no loss of efficiency in scaling up this type of column.

In general, however, in internally agitated extractors the height required for a theoretical stage increases with diameter because of the tendency of the flow pattern in the mixing section to follow a geometrically similar shape when scale-up is made on the basis of dimensional similitude. The present work describes an arrangement of internal baffling in a mixing section whereby the relationships of dimensional similitude need not be followed in a scale-up design. Figure 1*a* illustrates a normal vertical-flow pattern in a fully baffled tank. Figure 1*b* shows the geometric scale-up of this flow in a tank four times the diameter. In order to retain the same mixing height at the larger diameter, it will be necessary to direct the flow across the entire diameter by means of the stationary horizontal baffles shown in Figure 1*c*. These baffles can be so spaced as to give any desired flow pattern across the diameter of the column without regard to dimensional similarity. Scale-up based on dimensional similitude and a constant linear velocity through the column will increase the residence time in the mixing section proportional to the scale-up factor. This will tend toward better mass transfer and will allow some decrease in the spacing of the baffles for the same efficiency.

On the other hand, if the spacing is decreased so that residence time in the mixing section is maintained constant, the corresponding linear velocities between the baffles, which will maintain the same number of recycles of the liquid over the flow pattern in passing through the mixing section, will be increased in proportion to the scale factor. This will increase the contacting and resulting mass transfer between the phases but will also increase the difficulty of phase separation between the mixing stages. Obviously a compromise is necessary and desirable. Increasing the height of the mixing section based on the square root of the scale-up factor represents a compromise halfway between the two previous extremes. In the present work only one column diameter was tested and so this conclusion could not be tested. However in the previous design with unbaffled mixing sections separated by packing, the data on the easy extraction system, acetic acid–methyl isobutyl ketone–water, gave a minimum observed H.E.T.S. of 2¼ in. in a 1-in. column (*7*) and 9.2 in. in a 12-in.-diam. column (*8*). In the latter column no runs were made at the optimum packing height, which was intermediate between the two heights investigated, and it was estimated that for the best design the H.E.T.S. would be reduced by about 10%. On a difficult extraction system such as *o*-xylene-water the 1-in. column gave a minimum observed H.E.T.S. of 4.2 in. (*7*) and the 12-in. column gave a minimum value of 15 in. (*8*). In the latter case one of the packing heights investigated was close to the optimum, and so the value represents very nearly the lowest value obtainable. From these data it is readily established that the H.E.T.S. on this type of column varies as the square root of the diameter, and this has been successfully extrapolated to the design of larger columns. In the new design described in this work it is expected that the control of the mixing height will give an even smaller variation of H.E.T.S. with diameter, and square root factor can be conservatively used until more data are available.

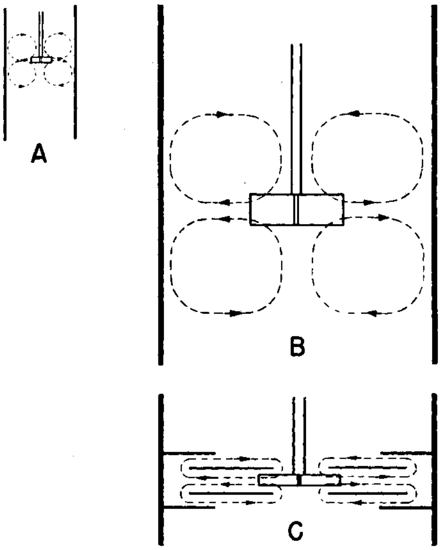

Fig. 1. Schematic design of vertical flow components in agitated column. A, original pattern; B, dimensional-similitude scale-up by factor of 4; C, modified pattern for scale-up factor of 4 with original height.

When packing was used to separate the mixing stages without baffles, the normal flow pattern carried the fluids up into the packing, where all the rotational motion was removed before the fluids returned to the agitator. Thus there was no accumulation of rotational energy, as in an unbaffled tank. In this new design with no appreciable flow expected through the packing, the rotational energy might accumulate excessively and interfere with the desired net flow through the column. To eliminate this possibility, vertical baffles can be installed around the column wall or between the two inner annular baffles at the discharge of the impeller. In the present study two layers of knitted-wire mesh were inserted between these inner annular plates and held in place against the vertical support rods by threading them on a circular ring of ⅛-in. stainless steel rod. This served to promote the contacting of the two phases by pumping them through the fine openings in the mesh, and it also served to remove most of the rotational energy. Preliminary tests on a single stage at low agitator speeds indicated that a double thickness of mesh was adequate, but in the subsequent tests on the column one of the liquid systems required such large agitator speeds that this thickness of mesh removed only a fraction of rotational motion and more layers would be necessary to achieve a fully baffled appearance. In spite of the appearance, however, the measured power consumption of the agitator at these conditions was in perfect agreement with the correlation for a fully baffled tank.

EQUIPMENT AND OPERATIONS

Figure 2 shows the details of the column assembly. Three different arrangements were investigated. The first consisted of nine mixing sections 2 in. high with 2 in. of packing between the mixing sections and at the ends. In the second arrangement the packing bundles were removed and the mixing sections were separated by the void chambers. In the third arrangement baffles and agitators were installed in all the intermediate void sections to give a total of seventeen mixers and the two end sections were left void to ensure complete phase separation at the top and bottom of the column.

The flow sheet for the tests was identical with that previously described for the study of the performance of a multistage semicommercial extraction column (*8*). Much of the same equipment was used, including the original column shell, consisting of two 12 in. O.D. glass cylinders 24 in. high.

The solvent systems used in this work were methyl isobutyl ketone–water–acetic acid, *o*-xylene–water–acetic acid, and *o*-xylene–water–phenol. The first system is considered easy to extract and employs approximately equal solvent ratios. The other systems are considered difficult to extract, the second one requiring solvent ratios of about 20 to 1 and the third requiring about

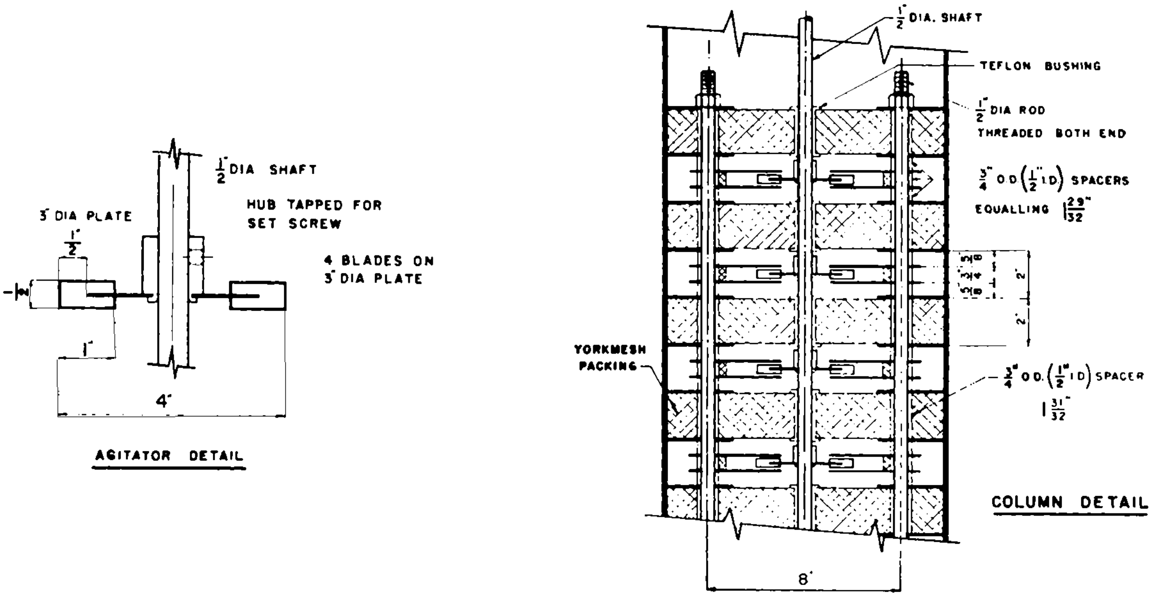

Fig. 2. Construction details of experimental extraction column.

equal solvent ratios for comparison with the first system.

All solvents were the commercially available materials and were used as received. In the test runs the large number of stages gave a close approach to equilibrium at one of the ends of the column even though an effort was made to maintain the operating line parallel to the equilibrium curve. This was difficult to achieve because of the curvature of the equilibrium curve. In all runs where the approach to the equilibrium curve was so close that an error of 1 or 2% would appreciably change the number of theoretical stages, the samples of two liquid streams at this end of the column were mixed and brought to equilibrium. This equilibrium was used in the graphical calculations, and it soon became evident that a slight difference in equilibrium data was present over the previously used data.

In the acetic acid systems, concentrations up to 20% in the water were used, corresponding to concentrations up to 20% in the methyl isobutyl ketone and 1.5% in *o*-xylene. Phenol solute concentrations ranged up to 0.25% in each of the phases.

The same operating technique used in the previous work (*8*) was employed in which the solute was transferred alternately from one solvent to the other so that errors in the equilibrium data would produce a twofold discrepancy among the observed stage efficiencies. The agreement between consecutive runs made at the same conditions was taken as a check of the equilibrium data, and toward the end of the series successive runs were made at different conditions to accelerate the investigation of the different variables. Direction of diffusion has been found to affect the performance of the mixing section and small differences could be attributed to this effect. Because of the large number of stages involved in this work a relatively small error in the equilibrium curve would produce a significant discrepancy in the number of theoretical stages. Thus the present data could not be used to evaluate the small effect for the direction of diffusion, but rather the close agreement verified the equilibrium data.

Previous work described on this same unit indicated that changing the total holdup in the column two to three times was adequate for attaining steady state conditions (*8*), and in all these runs the holdup was turned over three to five times before the products were collected. The collecting period was also sufficient to change the total holdup two to four times, and in the first few runs spot samples were taken before and after the collecting period and confirmed that steady state had been reached. Material balances checked generally within 5%, and in the methyl isobutyl ketone-water-acetic acid system the material-balance point on the ternary diagram determined from the acid concentrations of all four streams coincided with the point located according to the measured product-stream quantities.

Stage efficiencies were calculated by stepping off the theoretical stages on a graph of the solute ratio in the light phase against the solute ratio in the heavy phase as previously described (*8*), because this method of plotting gives a straight operating line when changes in the mutual solubility are small over the range of solute concentrations. The application of the method to the *o*-xylene-water system is obvious, and the rigorous method of Varteressian and Fenske (*9*) in the other solvent system gave a straight operating line within the limits of accuracy of the scale of construction used.

MEASUREMENT OF POWER INPUT

The first runs on the column using methyl isobutyl ketone-water-acetic acid showed that at low agitator speeds the stage efficiency was greater at the low throughput and at high agitator speeds the efficiency was greater at the high throughput. This indicated that there was an optimum power input per unit volume of solvents flowing through the column, and for the second series of runs using the *o*-xylene-water-acetic acid system a torque indicator was constructed and installed on the agitator shaft as an integral part of the drive pulley.

The power was transmitted through a coil spring to the column shaft from the drive pulley fixed to the outer housing, which rotated along a spiral path on small bearings. This rotation converted the rotational distortion of the spring into a vertical displacement, which was measured by a depth gauge resting on the top of the outer housing at its center of rotation. The torque indicator was calibrated in position by measuring the vertical displacement produced by suspending different weights from a cord wrapped around the outer housing and passing downward over a pulley rotating in a plane tangent to the outer housing. After the initial small preloading of the spring was relieved, the calibration curve of torque against depth reading was linear.

The power input to the agitator shaft was calculated from the speed and torque, and it was found to vary as the cube of the speed, as has been observed for fully baffled tanks at high Reynolds numbers.

In the columns without packing, both for the nine- and the seventeen-stage arrangements the observed power consumption per agitator was in exact agreement with that calculated from the correlation of Mack (*3*) for fully baffled tanks at turbulent flow. The packing bundles used in this work contained Teflon bushings at the center, which had been bored $\frac{1}{16}$ in. greater than the shaft diameter, but there was apparently some friction in these bushings because the observed power input per stage was 15% greater when they were installed than was calculated from the previously mentioned correlation. Thus for all the runs on the column, including those with the packing, the calculated power input per agitator was used for the correlation of the data.

For the type of agitator used in this

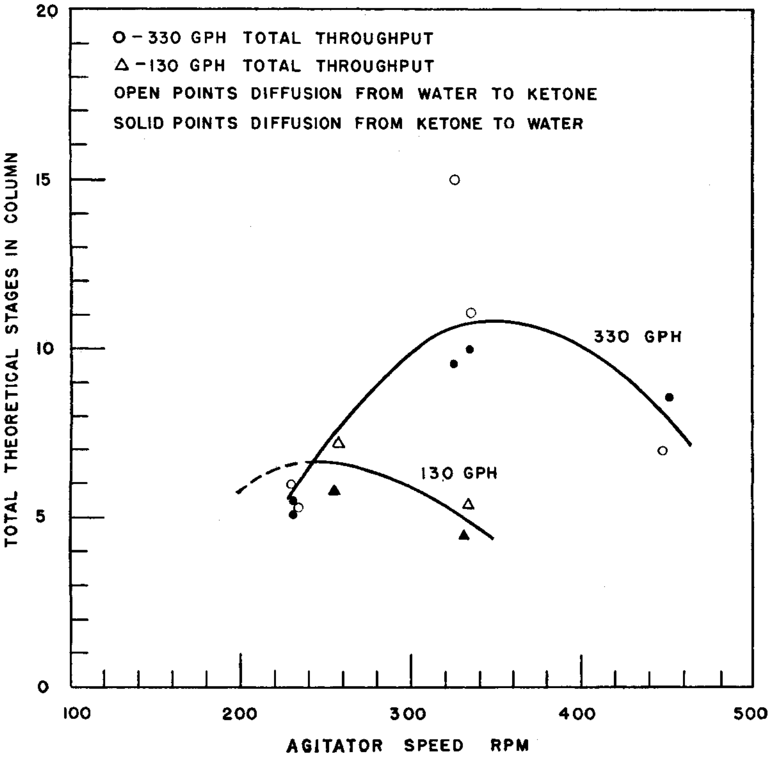

Fig. 3. Effect of agitation speed at different throughputs on acetic acid–methyl isobutyl ketone–water system in nine-stage column.

work, the general correlation for the power consumption gives

$$P = 1.85\,\frac{D^5 \rho N^3}{g}$$

and, with an average density in the mixing section based on about one-third holdup of dispersed phase, the following equations were used to calculate the power consumed per stage:

$P = 0.0132N^3$ for water dispersed in *o*-xylene
$P = 0.0140N^3$ for *o*-xylene dispersed in water
$P = 0.0128N^3$ for water dispersed in MIBK
$P = 0.0136N^3$ for MIBK dispersed in water

INTERPRETATION OF DATA*

The first series of runs on the acetic acid–methyl isobutyl ketone–water system indicated a maximum efficiency with agitator speed at a given throughput. As shown in Figure 3, the maximum occurs at lower agitator speeds at the lower throughput. Excessive agitator speeds produce finer dispersions which do not coalesce in the calming section and also cause some overlapping of the flow patterns in successive mixing section. Both effects contribute to lowering of the stage efficiency. At speeds below 230 rev./min. the appearance of the column did not indicate good efficiency and so no runs were made. However, the transfer of material between the phases in this system is so easy that fairly good performances have been obtained on unagitated columns either packed or void. It is therefore not the best system with which to study the performance of an agitated column. The additive effects provide abnormally high efficiencies of more than one theoretical stage per actual stage. In this case the high efficiencies are very sensitive to the operating and equilibrium data. Also the curves were drawn between the points determined for the different directions of diffusion although, as acknowledged previously, the performance curves probably differ.

Systems with hydrocarbon and water as solvents are generally classified as difficult extraction systems because they exhibit very poor efficiencies in the unagitated columns. The *o*-xylene–water system gave the same general shape of curves in the agitated column, but the maxima occurred at lower values because of the lack of extraction in the packed separating section. Also the absolute value of the function in this system was much different when the acetic acid and the phenol were used as solutes. This was attributed to the difference in the solvent ratio in the column, and it was observed that considerably more power was required to give the best efficiency with the acetic acid solute when the small amount of water had to be uniformly dispersed in the *o*-xylene phase. It was assumed that the power necessary to produce a satisfactory dispersion of one volume of solvent in ten volumes of another was ten times as great as when five and one half volumes of one solvent was dispersed in an equal volume of the other solvent.

The additional extraction obtained by the countercurrent flow through the packing masked the effect in the mixing section in the first arrangement. In the second and third arrangements, in which the packing was omitted, the stage efficiencies of the nine- and seventeen-stage columns are shown in Figure 4 plotted against the function $[P/(L_C + L_D)]\,L_D/L_C$. No consistent difference can be noted between the efficiency of the nine- and seventeen-stage columns, and it was concluded that the void intermediate calming section could be eliminated to save half the height of the column without affecting the individual stage efficiency. This will then double the number of theoretical stages per foot of height.

The data show appreciable scattering; however, all the points significantly below the curve were obtained at the highest power input of the given series of runs and represent a close approach to flooding conditions. Flooding is not adequately covered in this correlation. The amount of power that can be put into a particular system without producing an emulsion or a flooding condition is a function of fluid properties. However, the maximum power input also differs, depending upon which phase is dispersed. For example, the series of points for the phenol–*o*-xylene–water system at the opposite ends of the curve represent the same solvent and solute system; the points at the left correspond to a dispersion of the light phase, and at the right the points correspond to a dispersion of the heavy phase. These data could obviously not be correlated on the basis of static fluid properties. Qualitatively it is recognized that in a mixing section the tendency of the mass of liquid to rotate will force the lighter liquid to the middle. When the problem is one of dispersing the light phase, this effect facilitates the mixing, and when it is desired to disperse the heavy phase, this effect opposes the mixing. This had been noted in a previous work (*2*). Nevertheless, it does not seem reasonable to attribute the three-hundredfold increase in the permissible power input in the latter situation to this rotational effect alone, and differences in the dynamic interfacial conditions for the two types of dispersion may also contribute to the discrepancy. At any rate, it appears to be much more difficult to disperse water in the *o*-xylene than in the inverse arrangement and the physical reasons for this are not fully understood.

The data on the column with packing between the mixing sections (shown in

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*Performance data have been filed as document 4784 with the American Documentation Institute, Photoduplication Service, Library of Congress, Washington 25, D. C., and may be obtained for $1.25 for photoprints or 35-mm. microfilm.

Figure 5) are consistently above the curve for the column without packing. Also in this case the different throughputs and properties of the system affect the deviation. The curve for a total throughput of 330 gal./hr. on the methyl isobutyl ketone-water-acetic acid system is much higher than the curve for 130 gal./hr. on the same system and also much higher than the curve for 270 gal./hr. of total throughput on the *o*-xylene-water-acetic acid system. The usefulness of the packing in providing additional transfer of material is thus an inverse function of the interfacial tension, or, more specifically, it is probably a function of the Weber number.

It is significant that the maximum efficiencies in all cases are obtained in the region of values of 100 to 150 lb./sq. ft. for the function $[P/(L_C + L_D)]\ L_D/L_C$. It was also noted that greater power inputs per unit volume of solvents were possible with the packing than without, but this was not the most economical method of operation.

CONCLUSIONS

A new type of baffling arrangement has been devised for an internally agitated extraction column which provides uniform mixing of the phases in a smaller height than previously possible, and this will allow the design of large-diameter columns without requiring a proportional increase in height.

A correlation of the efficiency of the mixing section is proposed based on the power input per unit volume of fluids flowing and the ratio of the dispersed to continuous phase. Packing between the stages was found to increase the over-all efficiency and to decrease somewhat the solvent-flow capacity of the column. The minimum H.E.T.S. in an 11½-in. I.D. column was 5 in. when no packing was used between the stages. When packing was used, the minimum H.E.T.S. was 6 in. on the *o*-xylene-water system and 3 in. on the methyl isobutyl ketone-water system at the higher throughput. Thus it appears that in a difficult extraction system where the interfacial tension is high insufficient benefit is derived from the packing to justify its use. On the other hand, in a system which has a low interfacial tension and is easy to extract, the greater amount of mass transfer obtained in the packing gives it an economic advantage over the use of additional mixing sections.

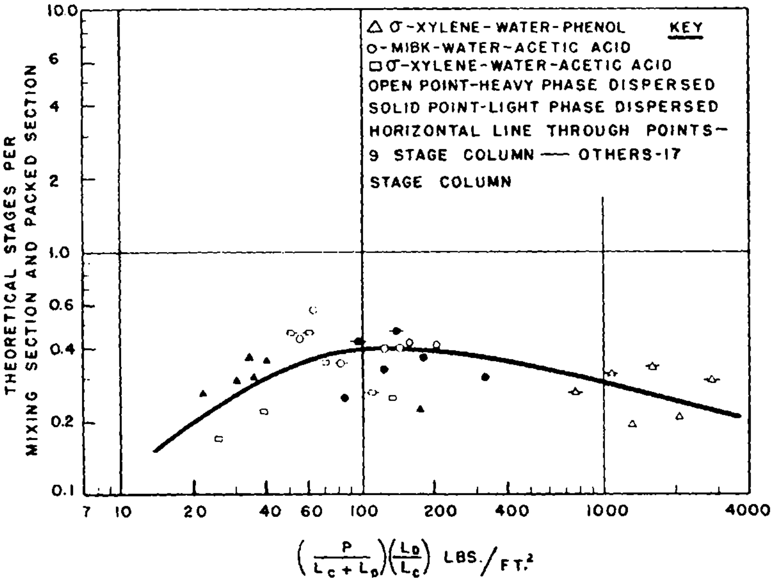

Fig. 4. Correlation of mixing efficiency with power throughput and solvent ratios.

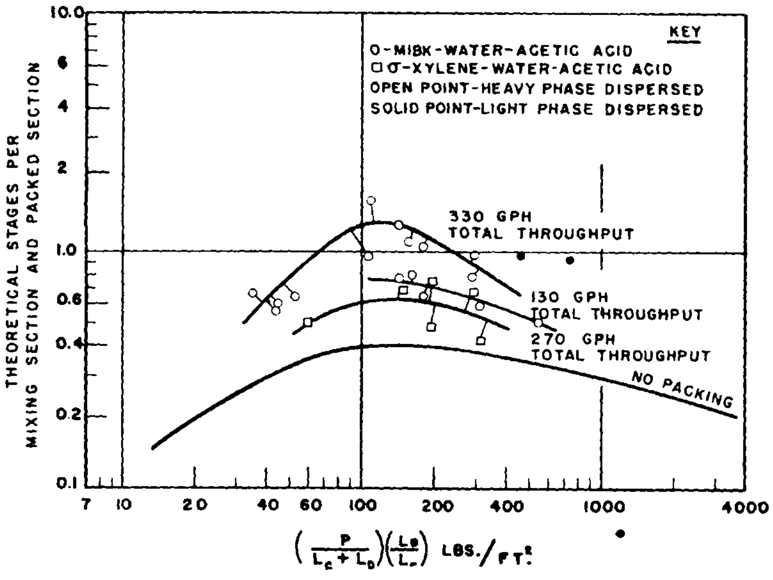

Fig. 5. Effect of packing on stage efficiency for different throughputs and different extraction systems.

By use of this baffling arrangement in larger diameter columns, it should be possible to decrease the ratio of H.E.T.S. to column diameter considerably below the value of one quarter reported in the present work.

Patent application has been filed on the extraction-columns described, and an exclusive license to fabricate and furnish them has been granted to the York Process Equipment Corporation.

ACKNOWLEDGMENT

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NOTATION

D = outside diameter of the agitator, ft.
g = gravitational constant, 32.2 ft./sec.^{2}
L = solvent flow rates, cu. ft./sec.
N = agitator speed, rev./sec.
P = power input, ft.-lb./sec.
ρ = fluid density, lb./cu. ft.

Subscripts

D = dispersed phase
C = continuous phase

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