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**EXTRACTION EFFICIENCY OF A PULSE
COLUMN OF VARIED GEOMETRY**

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EXTRACTION EFFICIENCY OF A PULSE COLUMN OF VARIED GEOMETRY*

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Lawrence E. Burkhart and R. W. Fahien

ABSTRACT

An investigation was made of the effects of plate spacing, hole diameter, percent free plate area, pulse amplitude, and pulse frequency on the extraction efficiency of a 1-in. diameter pulse column. The hexone-acetic acid-water system was used. Two levels of each factor were incorporated in an experimental design based on statistical methods. The data were analyzed by the analysis of variance technique.

The diameter of the holes in the perforated plates of a pulse column produced the greatest effect on extraction efficiency of the factors studied. Percent free area produced only a small effect which was dependent on the degree of pulsation applied to the column. Extraction efficiency varied inversely with pulse amplitude and pulse frequency. The recycle or back mixing, which exists in the pulse column was postulated to have a profound effect upon extraction efficiency.

*This report is based on an M.S. thesis by Lawrence E. Burkhart submitted June, 1956, to Iowa State College, Ames, Iowa. This work was done under contract with the Atomic Energy Commission.

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INTRODUCTION

In the search for better liquid-liquid contactors, continuous counter-current extractors which utilize mechanical means of agitation from an external source have been shown to give higher extraction efficiencies than contactors with no external means of producing the turbulence necessary for high extraction rates. One such extractor of current interest is the pulse column.

A diagram of a pulse column is shown in Figure 1. Heavy liquid enters from the top of the column and light liquid enters from the bottom of the column. Ordinarily, the size of the holes in the sieve plates in the column is small enough that no counter-current flow due to gravity will occur through the plates. An up and down pulse is applied to the fluid contents of the column by an externally powered bellows or diaphragm-type pulse generator. This pulse generator serves to disperse or mix the two liquid phases and also to pump them through the column.

The degree of dispersion in the pulse column depends on the amplitude and the frequency of the pulse. Figure 1 shows the beginning of an upstroke of the pulse generator for a column operating at low amplitude and frequency. As the bellows contracts, light liquid which has collected under each plate is forced through the holes and dispersed in droplets into the layer of heavy liquid above the plate. These droplets rise through the heavy-liquid layer and coalesce under the next plate. On the following downstroke, the heavy liquid above the plates is pulled down through the plates and dispersed into the light liquid below. As the degree of agitation applied to the pulse column is increased, the dispersed droplets become smaller and the amount

Figure 1. Diagram of a pulse column

of coalescence between pulses decreases until the fluid in the column appears to be a rather homogeneous emulsion of fine droplets.

The limiting conditions of flow in the extractor are called flooding points. Flooding is defined as the entrainment of light-liquid phase with the heavy-liquid effluent or vice versa. Two types of flooding have been observed in pulse columns. One flooding point, called flooding due to insufficient pulsation, occurs when the pumping action of the pulse generator is not sufficient to maintain the required rate of flow through the column and accumulation results in the end sections. The second flooding point, called flooding due to excessive pulsation, occurs when the drops of liquid in the column become so small that the net rate of flow through the column is less than the rate determined by the superficial flow rates of the two phases. Once again accumulation begins and the column floods.

In general, the factors which would appear to affect the extraction efficiency of a pulse column would be:

- I. Pulse factors
 1. amplitude
 2. frequency
 3. pulse shape or form
- II. Geometry factors
 1. plate spacing
 2. plate hole diameter
 3. percent free plate area
 4. column diameter
- III. Other factors
 1. flow rates
 2. physical properties of the system
 3. choice of continuous phase

The present investigation concerned the effects of plate spacing, hole diameter, and percent free area on the extraction efficiency. Because of the wide ranges

of operation which can be obtained by changing the pulsing conditions, the factors pulse amplitude and pulse frequency were also studied. The system methyl isobutyl ketone(hexone)-acetic acid-water was used.

DESIGN OF EXPERIMENT

The purpose of the experiment was to obtain a broad picture of the effects of plate spacing, hole diameter, percent free area, pulse amplitude, and pulse frequency on the extraction efficiency of a pulse column. The criterion of extraction efficiency was taken as the $(HTU)_{OG}$, the over-all height of a transfer unit based on the extract phase. The HTU decreases with increasing extraction efficiency. Before proceeding farther it will be convenient to define certain symbols which shall represent the factors studied. Let the small letters s, d, p represent respectively the geometry factors plate spacing, hole diameter, and percent free area. Let the small letters a and f represent the factors pulse amplitude and pulse frequency.

Two levels of each factor were chosen. Each factor was tested at each level of every other factor, so that 32 different experimental combinations were required. Each run was repeated three times to provide a measure of the experimental error. A compatible system of constant flow rates and levels of each factor under investigation had to be found so that the column operated without flooding for all experimental combinations of factors tested. Now, for a fixed total throughput, the pulse column has two flooding points. The lower flooding point, flooding due to insufficient pulsation, is a function of only the pulse factors (2). The upper flooding point, flooding due to excessive pulsation, is a function of both the pulse factors and the geometry

factors for a given fluid system at a constant total throughput (9). Hence the operating range for a pulse column is sandwiched between these two flooding points and the permissible values for the pulse and geometry factors are limited accordingly. On the basis of these considerations, the levels of factors shown in Table 1 were used. The flow rates were held constant at 36 grams per minute for each phase.

Table 1. Levels of factors studied

Factor	Level 1	Level 2
plate spacing	1 in.	2 in.
hole diameter	1/32 in.	1/16 in.
percent free area	13%	25%
pulse amplitude	0.25 in.	1.0 in.
pulse frequency	25 cpm.	50 cpm.

A split-plot experimental design was employed (4). On the main plots a completely randomized factorial design containing all possible combinations of levels of the geometry factors s , d , and p was used. Superimposed upon these main plots were a series of sub-plots, each of which was completely randomized and consisted of all possible combinations of levels of the pulse factors a and f . The main plots and the sub-plots can be seen in Figure 4 where the data from the investigation have been arranged on these plots.

The data were analyzed using the analysis of variance technique (4) by which the effects of the factors were divided into main effects, interactions, and experimental error.

INVESTIGATION

Description of Equipment

Two pulse columns were constructed. The first was used as an extracting column to perform the investigation and the second served as a stripping column to recover solvent and acid. The columns were of similar design.

The contacting section of the extraction column was composed of short sections of 1-in. diameter heavy walled glass tubing between which polyethylene gaskets and stainless steel perforated plates were inserted. Calming sections of 3-in. diameter glass tubing were located at each end of the contacting section. The entire assembly was held together in compression by four aluminum tie rods which extended through metal plates at the top and the bottom of the column.

Two sets of glass sections provided the two plate spacings used. In each case the compression on the column was adjusted until the height of the contacting section was 18 $\frac{3}{8}$ in. The four sets of perforated plates used were made of 26 gauge type 316 stainless steel and represented the four combinations of 1/16-in. and 1/32-in. diameter holes with 13% and 25% free plate area. The plates of 25% free area were perforated by the Harrington and King Perforating Company. Plates which had 13% free area were made by drilling 2-in. square pieces of stainless stock. All holes were on staggered centers. Because of reports that pulse column operation is sensitive to the wetting characteristics of the plates (2) (9), all of the plates were electropolished to a brilliant luster at the beginning of the work to provide a clean, uniform plate surface.

A photograph of the equipment is shown in Figure 2. Figure 3 is a flow diagram of the pulse columns and their associated equipment. The fluid contents of the extraction column were pulsed by a pulse generator made from a Lapp Pulsafeeder. The pump head was replaced with a brass bellows and the unit was powered with a U.S. Varidrive variable speed drive. A continuously variable frequency of 10 to 125 cycle per minute could be obtained.

Pulse amplitude was measured by a method described by Beyer and Edwards (2), in which a sharp pointed probe connected to a micrometer stem was inserted from the top of the column down to the air-liquid interface.

Procedure

At the beginning of a run, the column was filled with aqueous phase to a point about three inches above the top plate. The pulse amplitude and the pulse frequency were set at the desired values. The pulse generator was started and flow rates were set at 36 grams per minute for each phase. All runs were made with the water phase continuous and with extraction from the water into the solvent. The jackleg in the aqueous effluent line was adjusted to maintain the interface at a point 2 ± 0.5 in. above the top plate.

After apparent steady-state had been reached, as indicated by a constant position of the interface, the pulse column was permitted to run for at least a half hour. When the interface had remained constant for a half-hour period, flow rate measurements of 10 minutes each were made until two consecutive measurements checked within 2%. Samples from each run were analyzed by titration with sodium hydroxide for the aqueous samples and sodium methoxide for the organic samples. Acid concentration in the feed was kept at 15% acid by weight. The concentration of the incoming solvent was kept at 1% acid.

Figure 2. Photograph of pulse columns and associated equipment

A, H AQUEOUS FEED TANKS
 B, G ORGANIC FEED TANK
 C EXTRACTION COLUMN
 D, K PULSE GENERATOR
 E, F GRADUATED CYLINDERS
 J STRIPPING COLUMN

Figure 3. Flow diagram

After every four runs, the column was disassembled and rebuilt using different plates and/or different lengths of glass sections to conform with the geometry conditions dictated by the design of the experiment.

The organic liquid was distilled at intervals to remove impurities which had accumulated with time. However, this operation produced no noticeable effects on the performance of the column.

The $(HTU)_{OE}$ for each run was calculated by graphical integration, using the approximation of Wiegand (11).

Results

The experimental data are tabulated in Table 5 (see Appendix). The data are summarized in Figure 4. Each number in Figure 4 represents the arithmetic mean value of three runs made at the given conditions.

The general trends of the data can be seen by examining Figure 4. In general, increasing amplitude, frequency, hole diameter, and plate spacing resulted in a higher HTU and hence a lower extraction efficiency. The effect of increasing the percent free area of the perforated plates was not so well defined. In seven out of sixteen cases, increasing the percent free area resulted in a decrease in extraction efficiency. Six of the seven cases in which efficiency decreased with increased percent free area occurred at the low pulse amplitude. Hence, for increased percent free plate area, the extraction efficiency appeared to increase at high pulse amplitude and to decrease at low pulse amplitude.

An analysis of variance was made on the data collected. The results are given in Table 2. Because of the split-plot design employed, the experiment

		s_1				s_2			
		d_1		d_2		d_1		d_2	
		a_1	a_2	a_1	a_2	a_1	a_2	a_1	a_2
p_1	f_1	3.65	6.49	6.55	6.77	4.83	6.81	6.26	6.98
	f_2	4.16	7.33	6.43	7.48	6.33	7.56	7.09	8.28
p_2	f_1	3.91	5.47	5.96	6.29	4.77	6.12	6.81	7.05
	f_2	4.71	5.61	6.26	7.32	6.40	6.33	7.68	7.29

a = PULSE AMPLITUDE **d** = HOLE DIAMETER
f = PULSE FREQUENCY **p** = PERCENT FREE AREA
s = PLATE SPACING

Figure 4. Summary of data arranged on main plots of s , d , p , with a and f as sub-plots. Each value is the mean of three repetitions.

Table 2. Analysis of variance

Source of variation	Degrees of freedom	Mean square	F-ratio
S	1	13.98	49.93*
D	1	37.62	134.36*
P	1	2.36	8.43
SD	1	1.11	3.96
SP	1	0.24	0.86
DP	1	0.67	2.39
SDP	1	0.25	0.89
Repetitions within geometries	16	0.28	
A	1	28.34	157.44*
F	1	12.51	69.50*
AF	1	0.11	0.61
AS	1	2.27	12.61*
AD	1	6.87	38.17*
AP	1	5.19	38.83*
FS	1	0.94	5.22
FD	1	0.15	0.83
FP	1	0.12	0.67
AFS	1	1.15	6.39
AFD	1	1.44	8.00*
AFP	1	0.76	4.22
FSD	1	0.00	0.00
FSP	1	0.27	1.50
SDA	1	0.87	4.83
SPA	1	0.05	0.28
DPF	1	0.02	0.11
DPA	1	1.17	6.50
AFSD	1	0.02	0.11
FSDP	1	0.32	1.78
SDPA	1	1.31	7.28*
DPAF	1	0.01	0.06
PAFS	1	0.06	0.33
AFSDP	1	0.15	0.83
Residual	48	0.18	

* Significant at the 0.01 level.

was less sensitive to the effects of the geometry factors, s , d , and p , than to the effects of the pulse factors, a and f . Thus the error term used in testing for statistical significance of main plot effects was greater than the error term used for testing sub-plot effects.

Geometry Factors

Previous investigators (1) (3) (6) have used the product of pulse amplitude times pulse frequency, called the (af) product, as an index of the amount of agitation supplied to the pulse column by the pulse generator. Pulse amplitude was measured in inches per cycle and pulse frequency was measured in cycles per minute. The (af) product then had the dimensions, inches per minute. In this work, as in previous pulse column work, the pulse amplitude was considered to be the linear displacement of the fluid contents of the column from one extreme position to the other. Thus the (af) product represented a measure of the linear displacement per minute of the liquid in the column by the pulse generator.

The main plots of the geometry factors of Figure 4 were rearranged into four groups, using the (af) product as a parameter. These four groups are shown in Figure 5. The effects of the geometry factors were tested for significance at each value of the parameter by means of a t -test (4) for the difference between the means of the two levels of a geometry factor. The results are given in Table 3. Hole diameter affected extraction efficiency at all four conditions of pulsation. No effect of percent free area was demonstrated at the two low (af) products and no effect of plate spacing was demonstrated at the highest (af) product.

Figure 6. Effect of the (af) product on $(HTU)_{OE}$ at $s1d1P1$

Figure 7. Effect of the (af) product on $(HTU)_{OE}$ at $s_1 d_1 p_2$

Figure 8. Effect of the (af) product on $(HTU)_{OE}$
at $s_2d_1p_1$

Figure 9. Effect of the (af) product on $(HTU)_{OE}$ at $s2d1p2$

Figure 10. Effect of the (af) product on $(HTU)_{OE}$
at $s_1d_2p_1$

Figure 11. Effect of the (af) product on $(HTU)_{OE}$ at $s_1d_2p_2$

Figure 12. Effect of the (af) product on (HTU)_{OE}
at $s_2d_2p_1$

Figure 13. Effect of the (af) product on $(HTU)_{OE}$ at $s_2d_2p_2$

degree of pulsation at high (af) products. HTU varied directly with both pulse amplitude and pulse frequency. Pulse amplitude produced a greater effect than pulse frequency on extraction efficiency. It should be recalled, however, that amplitude was varied fourfold while frequency varied only two-fold.

Error

With two exceptions, the error in the acid material balance for a run was less than 5 percent. For runs 21 and 22 the error in the material balance was 5.05 percent and 5.75 percent, respectively. The standard error for a single run in terms of HTU was ± 0.42 in. The standard error of the mean for three runs made at the same conditions was ± 0.24 in. A pictorial representation of the precision of the data can be seen in Figures 6 through 13. The error term for "repetition within geometries" in Table 2 included errors introduced by disassembling and rebuilding the pulse column to change the levels of the geometry factors for the experiment. An F-ratio (4) of this term with the "residual" error term gave a value which was not statistically significant at the 0.01 level. This result means that no detectable error was introduced by disassembling and rebuilding the column. Hence a satisfactory method of changing the levels of the geometry factors was used.

DISCUSSION

A. Effects of Pulse Amplitude and Pulse Frequency

Photographs of the column were taken to show the drop size and the dispersion of the fluids with the column operating at low pulse amplitude

and frequency (a_1f_1) and at high pulse amplitude and frequency (a_2f_2). The levels of the geometry factors used were: 2-in. plate spacing, 1/32-in. hole diameter, and 25 percent free area. However, the pictures are representative of column operation at these pulsing conditions for all combinations of levels of the geometry factors. Figure 14 was taken at a_1f_1 shortly after the beginning of an upstroke of the pulse generator and shows droplets of the organic solvent rising up through the plate perforations and into the aqueous phase above the plates. Figure 15 was taken at the end of an upstroke of the pulse generator but before the beginning of a downstroke. This picture shows the coalescence which occurred between pulses at low amplitude and frequency. A picture of the pulse column during the downstroke can be inferred from Figure 15. During the downstroke, aqueous phase from above the plates was pulled down through the plate perforations and through the thin layer of organic phase below the plates.

Figure 16 was taken at a_2f_2 during an upstroke of the pulse generator. This photograph could well represent a picture taken at any time during column operation at this pulsing condition since no coalescence and redispersion could be detected. Rather, the two phases gave the appearance of a uniform emulsion of fine droplets at all times.

These photographs show a marked decrease in drop size at the two extreme levels of pulsation. The increased velocity of the fluids through the plate perforations and the increased turbulence within the column produced smaller drops and thus created a greater interfacial area between the two phases. The increased velocity of the fluids through the plate perforations should increase the mass transfer coefficient by decreasing the film thicknesses.

Figure 14. Photograph of the pulse column operating at an amplitude of 0.25 in. and a frequency of 25 cpm. ($a_1 f_1$); taken just after the beginning of an upstroke of the pulse generator.

Figure 15. Photograph of the pulse column operating at an amplitude of 0.25 in. and a frequency of 25 cpm. ($a_1 f_1$); taken at the end of an upstroke of the pulse generator.

Figure 16. Photograph of the pulse column operating at an amplitude of 1.0 in. and a frequency of 50 cpm. (a_2f_2).

Now the rate of mass transfer, dN , in a differential height of column, dH , is written:

$$\frac{dN}{dH} = K_E a_i S (C^* - C)$$

where, K_E = over-all mass transfer coefficient based on the extract phase

a_i = interfacial area per unit volume

$(C^* - C)$ = solute concentration gradient (driving force for mass transfer).

Throughout the investigation the mass transfer decreased with increasing values of the (af) product. If K_E and a_i both increased then $(C^* - C)$ must have decreased considerably in order to cause the rate of mass transfer to decrease.

The decrease in the concentration gradient, $(C^* - C)$, with increasing values of the (af) product was caused by forced recycle, or back mixing, within the pulse column. Edwards (2) derived an equation for predicting flooding due to insufficient pulsation in a pulse column to which a sinusoidal pulse was applied. In deriving this equation he found an expression for the downward rate of flow, q_d , of liquid through the column. The column flooded when q_d was less than L , where L represented the flow rate of the heavy phase entering from the top of the column. When q_d was greater than L the column operated without flooding but a condition existed in which liquid in the column was recycled. This forced recycle, or back mixing, represented the return of an amount of liquid back from whence it came. Hence during normal column operation, where q_d must be greater than L , some liquid was recycled and passed through a single plate more than once. The result was that some cocurrent flow was superimposed upon the countercurrent flow of both phases. Edwards made a similar analysis of the rate of flow of fluid through the column on the upstroke of the pulse generator.

The recycle rate was found to be equal for both upstroke and the downstroke.

The equation presented for recycle rate was:

$$V_r = afA \cosh \Theta - \frac{G}{2} - L$$

where, V_r = recycle rate, ml./min.

A = cross-sectional area of the column

a = pulse amplitude, cm./cycle

f = pulse frequency, cycles/min.

G = flow rate of organic phase, ml./min.

L = flow rate of aqueous phase, ml./min.

$$\Theta = \frac{\bar{G}}{\pi Aaf}$$

The recycle or back mixing which occurs in a pulse column represents a deterioration of true countercurrent flow and decreases the driving force or concentration gradient which is available for mass transfer. Constant flow rates of 35 ml./min. for the aqueous phase and 44.5 ml./min. for the organic phase were used in this investigation. For these flow rates, the recycle rates calculated from Edwards' equation for each value of the (af) product used is presented in Table 4. The recycle rate increased rapidly with increased pulsation. At low (af) products, where coalescence of the phases occurred as shown in Figure 15, the recycled liquid consisted of aqueous phase during the downstroke and a mixture of both aqueous and organic phases on the upstroke. When pulsation was more vigorous, as shown in Figure 16, the recycled liquid was composed of aqueous and organic phases during both the upstroke and the downstroke of the pulse generator.

As the (af) product is increased the increased interfacial area and the increased turbulence tend to increase the extraction efficiency of the pulse

column. The increase in the recycle rate tends to decrease the extraction efficiency. Another factor which tends to decrease the extraction efficiency at high (af) products is the lack of coalescence and redispersion of the phases between pulses. At low levels of pulsation, the two phases coalesce between pulses and are then redispersed. In studying extraction from single drops, Licht and Conway (8) found that, for the same liquid system used in the present

Table 4. Recycle rates at different values of (amplitude x frequency)

a x f	Recycle rate ml./cycle	Recycle rate ml./min.
6.25	1.34	33.53
12.50	2.30	114.79
25.00	11.29	282.21
50.00	12.38	619.28

investigation, about 13 percent of the extraction occurred during drop formation and another 8 per cent of the extraction occurred during drop coalescence. Sherwood, Evans and Longcor (10) studied extraction from single drops in columns of different heights. The results were plotted and the curve obtained was extrapolated to zero column height. For the hexoneacetic acid-water system, about 40 percent of the extraction appeared to occur during drop formation.

If the two phases coalesce between pulses, then fresh surface for mass transfer is created when the phases are redispersed. However, as the (af) product increases, there is less coalescence of the phases in the column.

Finally a point is reached where the size of the drops is of the same order of magnitude as the holes in the perforated plates. At this point many of the drops would merely be "massaged" or deformed in passing through the plates, while a few larger drops may be broken into smaller drops. Although this massaging action helps mass transfer by creating eddies within the drops, it would not be as effective as the creation of fresh interfacial surface which occurs at low (af) products where the phases coalesce and are then redispersed.

For the range of pulse column operation covered in this investigation, the recycle rate controlled extraction efficiency for a given column geometry. Apparently the recycle rate was sufficient to overcome the effects which tended to improve extraction efficiency as the value of the (af) product was increased. However, this may not be the case for different fluid systems. The ease of dispersion of the liquids due to their physical properties and to the plate-wetting properties which influence drop formation in a pulse column may cause the effects which tend to increase extraction efficiency to control extraction for other fluid systems. For example, liquids with a low interfacial tension are easily dispersed. If such a system were used in the pulse column, then good dispersion could be obtained at relatively low values of the (af) product, where the recycle rate is low. In this case the extraction efficiency would probably increase with increased pulsation before the recycle rate became large enough to cause extraction efficiency to decrease. This would cause a plot of HTU versus (af) product to be concave upward.

The flow rate ratio is an important factor which was held constant in this investigation. The relative magnitudes of the effects which tend to increase or decrease extraction efficiency as the (af) product increases may be different

for different flow rate ratios. Belaga and Bigelow (1) studied the effects of pulse amplitude and pulse frequency on HTU using the same fluid system as was used in this investigation. The flow rates were also the same. The curves presented of HTU versus (af) product were comparable to those shown in Figures 6 through 13. Cohen and Beyer (5) extracted boric acid from isoamyl alcohol with water. At (af) products from 5 to 30 in./min. the H.E.T.S. (height equivalent to a theoretical stage) decreased from 23 in. to 12 in. The dispersed to continuous phase flow rate ratio was 5:1. Sege and Woodfield (9) reported that, for a complex uranium extraction system, a plot of HTU versus (af) product was concave upward for (af) products in the range 20 to 70 in./min. In view of these results the relationship between flow rate ratio and the (af) product should be investigated. Extraction efficiencies for different fluid systems should also be compared to see if the curve of HTU versus (af) product has a characteristic shape or if the shape of this curve varies widely with the extraction system being employed. Such information would be useful in predicting the proper operating conditions for pulse columns.

Many of the curves of HTU plotted against the (af) product shown in Figures 6 through 13 have a hump at an (af) product of 12.5. Hayworth and Treybal (7) studied drop size as a function of velocity through a nozzle. They concluded that drop size was uniform and increased with velocity up to a velocity of 10 cm./sec. At higher velocities the drop size decreased and became less uniform. This phenomenon could explain the fact the HTU decreased slightly at an (af) product of 12.5 until the effect of recycle rate became large enough to mask the increase in interfacial area due to smaller drop size.

B. Effects of the Geometry Factors

Plate Spacing

Plate spacing affects extraction efficiency through the property of the plates of redispersing the liquids as they flow through the extraction column. This creates fresh interfacial surface for mass transfer between the two phases. The first perforated plate or sieve plate columns were developed because of reports that the rate of extraction in a spray column was greatest in the immediate vicinity of the inlet distributor for the dispersed phase. This led to the idea of providing many distributors by placing perforated plates at regular intervals up the column, thus taking advantage of the higher extraction rate which occurred near the distributor. Sieve plate columns were built and tested. They are used today but their use has probably been limited because of the lack of performance data.

Close plate spacing gives better extraction efficiency in the pulse column by redispersing the two fluids more frequently as they pass through the column. Only two levels of plate spacing were used in this experiment. Future work involving this factor should include levels of plate spacing from approximately 1/2 in. up to large spacings where spray column operation is approached. For very small plate spacings the diameter of the holes in the perforated plates may have to be large in order to obtain satisfactory column throughput capacity.

Hole Diameter

Hole diameter affects extraction efficiency in the pulse column by changing interfacial area for mass transfer between the two phases. Hayworth and Treybal

(7) showed experimentally that drop size decreases with a decrease in the diameter of the nozzle from which a stream of liquid is issuing. Figure 4 shows that the HTU decreased with a decrease in the diameter of the holes in the perforated plates. The smaller diameter holes produced smaller droplets, thus increasing the interfacial area between the two phases.

Hole diameters which have been used in pulse columns lie in the range $1/32$ to $1/8$ in. The hole diameter must be chosen with respect to both extraction efficiency and throughput rate. Another factor to be considered is clogging of small diameter holes. This investigation showed that the hole diameter should be as small as is consistent with the above requirements. Holes smaller than $1/32$ -in. diameter would probably limit the capacity of a pulse column considerably and the value of $1/32$ in. would be an approximate lower limit for hole diameter in future investigations. A hole diameter of $1/8$ in. is a popular size for larger diameter pulse columns and additional work should include this value for the hole diameter.

Percent Free Area

Percent free area of the plates produced only small changes in the extraction efficiency of the pulse column over the range of conditions studied. For small holes, the percentage of perforated area that can be produced in a plate is limited to about 30-35 percent for perforating presses. Approximately the same limit would apply from consideration of the strength of the resulting perforated plate. Thus the range of percent free area covered in this investigation represents the probable range of the factor from a design standpoint. Values much below 13 percent would seriously limit the flooding capacity of the column.

The factor percent free area could probably be set at some value in the range of 25 percent in future investigations.

Interactions

The results of the analysis of variance showed two-factor interactions between amplitude-plate spacing, amplitude-percent free area, and amplitude-hole diameter. The first two interactions can be seen from the results of the t-test applied to the geometry factors. No effect of plate spacing on extraction efficiency could be detected at the highest level of the (af) product, a_2f_2 , while an effect on extraction efficiency was demonstrated at the three lower levels of the (af) product. An opposite case occurred for the effect of percent free area. No effect of percent free area could be detected at the two low levels of the (af) product, a_1f_1 and a_1f_2 while an effect was demonstrated at the two high levels of the (af) product, a_2f_1 and a_2f_2 . Although hole diameter produced an effect on HTU which could be detected at all levels of amplitude and frequency, the interaction effect which appeared in the analysis of variance can be seen by comparing the results presented in Figure 4.

Any interpretation which might be given for these interactions would be clouded by the unknown effect of liquid recycle in the column. Consider the figures for recycle rate presented in Table 4. From Table 4 it is seen that both amplitude-frequency combinations which contained a_1 were at the two low recycle rates while the two amplitude-frequency combinations which contained a_2 were at the high recycle rates. It has been postulated that this recycled liquid produces a profound effect upon the operation of a pulse column; therefore any valid interpretation of the interactions of amplitude with the

geometry factors should be made from data taken at the same recycle rate. This discussion points out another phase of investigation on the pulse column which should be studied -- the relationship between pulse amplitude, pulse frequency, the (af) product, and their effect on extraction efficiency. The question arises whether the (af) product, regardless of the combination of amplitude and frequency which produces it, is the main factor to be considered or whether amplitude and frequency should be considered separately. In Table 4, the recycle rate in cm.³ per minute increases almost linearly with the (af) product, whereas the recycle rate in cm.³ per cycle shows a sharp break between the two low (af) products and the two high (af) products. An experiment in which the extraction efficiency of a pulse column is studied at various combinations of amplitude and frequency which produce a constant (af) product would provide information on this subject.

CONCLUSIONS AND RECOMMENDATIONS

1. The geometry factors plate spacing, hole diameter, and percent free area all affected the extraction efficiency of the pulse column within the range studied. Hole diameter produced an effect at all levels of pulsation investigated. The effect of plate spacing was significant at (af) products of 6.25, 12.5, and 25 in./min. The effect of percent free area was significant at (af) products of 25 in./min. and 50 in./min.

2. Pulse amplitude and pulse frequency both affected extraction efficiency although the effects of amplitude, frequency, and the (af) product were not clearly defined. Extraction efficiency was highest at the lowest value (6.25 in./min.) of the (af) product regardless of the levels of the geometry factors. Extraction efficiency generally decreased with increasing pulsation.

3. For the range of pulse column operation covered, the extraction efficiency of the pulse column at different levels of pulsation was controlled by the recycle rate within the column.

4. The results of the investigation were reported by statistical analysis, at the 0.01 significance level. This method of approach was successful in separating the effects of the factors investigated in the presence of experimental error. The method also provided an objective view of the reproducibility of the data and of the reliability of the results. Standard error for the individual runs in terms of $(HTU)_{OE}$ was 0.42 in.

As a result of this investigation, the following recommendations are made for further work.

1. The effect of hole diameter on the extraction efficiency of a pulse column should be investigated at several levels to determine the nature of the effect. Additional work with this factor should include a hole diameter of 1/8 in. since this is a common value which is used in commercial pulse columns. A lower limit of hole diameter could be taken as 1/32 in.

2. The effect of plate spacing on the extraction efficiency of a pulse column should be investigated further. Multiple levels of plate spacing should be studied to determine the nature of its effect on extraction efficiency.

3. Percent free area should be fixed at some value near 25 percent in future investigations and further work on this factor should be delayed until the effects of more important factors have been considered.

4. Pulse amplitude and pulse frequency should be included in future investigations of the effects of pulse column geometry factors and the experiments should be designed to evaluate or to nullify interactions which might occur between pulse and geometry factors.

5. The effects of flow rate ratio on extraction efficiency should be studied at various pulse amplitudes and pulse frequencies.

6. The individual effects of pulse amplitude and pulse frequency at fixed values of the (af) product should be investigated.

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Table 5. Calculated data for individual runs

Run No.	Pulse amplitude (inches)	Pulse frequency (cycles/min)	Plate spacing (inches)	Hole diameter (inches)	Free area (%)	Acid in feed (gm/gm H)	Acid in solvent (gm/gm L)	Acid in extract (gm/gm L)	Acid in raffinate (gm/gm H)	(HTU) _{OE} (inches)	Material balance (% error)
8	0.25	25	1.06	0.0312	13	0.1754	0.0105	0.1084	0.0553	3.86	-1.08
15						0.1754	0.0106	0.1169	0.0530	3.55	+1.56
26						0.1754	0.0104	0.1141	0.0527	3.53	-1.04
6	0.25	50	1.06	0.0312	13	0.1754	0.0105	0.1053	0.0590	4.51	-1.62
16						0.1754	0.0106	0.1138	0.0544	3.70	-1.39
27						0.1754	0.0104	0.1137	0.0565	4.28	-0.35
7	1.00	25	1.06	0.0312	13	0.1754	0.0105	0.0945	0.0688	7.12	-2.17
14						0.1754	0.0106	0.1046	0.0670	5.89	-0.70
25						0.1754	0.0104	0.1011	0.0666	6.47	-1.57
5	1.00	50	1.06	0.0312	13	0.1754	0.0105	0.0943	0.0710	7.32	-1.99
13						0.1754	0.0106	0.0978	0.0727	7.44	-0.86
28						0.1754	0.0104	0.0981	0.0711	7.23	-2.44
47	0.25	25	1.06	0.0312	25	0.1767	0.0100	0.1137	0.0578	4.02	+0.50
59						0.1754	0.0100	0.1119	0.0522	3.81	-2.41
92						0.1771	0.0102	0.1135	0.0543	3.89	+0.35
48	0.25	50	1.06	0.0312	25	0.1767	0.0100	0.1094	0.0586	4.64	-1.21
57						0.1754	0.0100	0.1102	0.0608	4.60	+0.34
89						0.1771	0.0102	0.1110	0.0590	4.88	+0.87
46	1.00	25	1.06	0.0312	25	0.1767	0.0100	0.1062	0.0261	5.37	-2.77
58						0.1754	0.0100	0.1045	0.0633	5.57	-1.20
90						0.1771	0.0102	0.1058	0.0624	5.48	0.00
45	1.00	50	1.06	0.0312	25	0.1743	0.0100	0.1050	0.0637	5.43	-0.70
60						0.1754	0.0104	0.1045	0.0647	5.70	-0.35
91						0.1771	0.0102	0.1051	0.0639	5.69	+0.35

APPENDIX

Table 5. (continued)

Run No.	Pulse amplitude (inches)	Pulse frequency (cycles/min)	Plate spacing (inches)	Hole diameter (inches)	Free area (%)	Acid in feed (gm/gm H)	Acid in solvent (gm/gm L)	Acid in extract (gm/gm L)	Acid in raffinate (gm/gm H)	(HTU) _{OE} (inches)	Material balance (% error)
4 22 80	0.25	25	1.06	0.0265	13	0.1754 0.1754 0.1754	0.0105 0.0104 0.0100	0.0916 0.1009 0.1068	0.0783 0.0616 0.0613	8.43 6.02 5.19	-0.18 -5.75 -0.87
3 23 77	0.25	50	1.06	0.0625	13	0.1754 0.1754 0.1754	0.0105 0.0104 0.0100	0.0946 0.1041 0.1034	0.0759 0.0648 0.0647	7.59 5.76 5.93	+1.62 -1.96 -0.30
1 21 78	1.00	25	1.06	0.0625	13	0.1754 0.1754 0.1754	0.0105 0.0104 0.0100	0.0955 0.0996 0.1004	0.0712 0.0659 0.0688	7.04 6.58 6.70	-0.72 -5.05 -0.17
2 24 79	1.00	50	1.06	0.0625	13	0.1754 0.1754 0.1754	0.0105 0.0104 0.0100	0.0966 0.0976 0.0957	0.0712 0.0723 0.0762	6.88 7.41 8.16	-0.54 -1.75 -0.52
42 52 61	0.25	25	1.06	0.0625	25	0.1743 0.1754 0.1754	0.0106 0.0100 0.0101	0.1018 0.1057 0.1029	0.0641 0.0635 0.0646	6.06 5.85 5.98	-2.44 +0.86 -1.21
41 51 64	0.25	50	1.06	0.0625	25	0.1743 0.1754 0.1754	0.0106 0.0100 0.0102	0.1025 0.1041 0.1020	0.0693 0.0671 0.680	6.36 5.92 6.49	+0.20 +1.04 -0.52
44 50 62	1.00	25	1.06	0.0625	25	0.1743 0.1754 0.1754	0.0106 0.0100 0.0100	0.1014 0.1038 0.1014	0.0685 0.0694 0.0668	6.36 6.14 6.36	-1.22 +1.92 -1.04
43 49 63	1.00	50	1.06	0.0625	25	0.1743 0.1767 0.1754	0.0106 0.0100 0.0102	0.0981 0.0990 0.0990	0.0741 0.0718 0.0731	7.47 7.23 7.26	-0.50 -0.70 +0.87

Table 5. (continued)

Run No.	Pulse amplitude (inches)	Pulse frequency (cycles/min)	Plate spacing (inches)	Hole diameter (inches)	Free area (%)	Acid in feed (gm/gm H)	Acid in solvent (gm/gm L)	Acid in extract (gm/gm L)	Acid in raffinate (gm/gm H)	(HTU) _{OE} (inches)	Material balance (% error)
11 38 56	0.25	25	2.00	0.0312	13	0.1754 0.1743 0.1754	0.0106 0.0106 0.0100	0.1030 0.1095 0.1074	0.0612 0.0591 0.0592	5.10 4.47 4.91	-1.08 -0.30 -0.17
9 39 53	0.25	50	2.00	0.0312	13	0.1754 0.1743 0.1754	0.0105 0.0106 0.0100	0.0978 0.1039 0.1012	0.0689 0.0663 0.0696	6.51 5.89 6.58	-0.90 0.00 -0.17
12 40 55	1.00	25	2.00	0.0312	13	0.1754 0.1743 0.1754	0.0106 0.0106 0.0100	0.0977 0.1010 0.1074	0.0705 0.0716 0.0699	6.58 6.83 7.01	-1.08 -0.87 -1.55
10 37 54	1.00	50	2.00	0.0312	13	0.1754 0.1743 0.1754	0.0105 0.0106 0.0100	0.0954 0.0970 0.0968	0.0717 0.0748 0.0753	7.15 7.69 7.85	-0.90 +0.30 +0.52
35 72 82	0.25	25	2.00	0.0312	25	0.1743 0.1754 0.1754	0.0104 0.0100 0.0100	0.1075 0.1089 0.1093	0.0613 0.0591 0.0590	4.95 4.75 4.62	-1.56 -0.86 -0.52
33 71 84	0.25	50	2.00	0.0312	25	0.1743 0.1754 0.1771	0.0111 0.0100 0.0100	0.1035 0.1019 0.1019	0.0694 0.0676 0.0699	6.14 6.40 6.66	-0.30 -0.52 -0.86
36 69 81	1.00	25	2.00	0.0312	25	0.1743 0.1754 0.1754	0.0108 0.0100 0.0100	0.1045 0.1017 0.1046	0.0667 0.0659 0.0667	6.12 6.40 5.83	+1.20 -3.41 0.00
34 70 83	1.00	50	2.00	0.0312	25	0.1743 0.1754 0.1771	0.0110 0.0100 0.0100	0.1030 0.1028 0.1038	0.0683 0.0677 0.0695	6.16 6.61 6.23	-0.30 -0.70 0.00

Table 5. (continued)

Run No.	Pulse amplitude (inches)	Pulse frequency (cycles/min)	Plate spacing (inches)	Hole diameter (inches)	Free area (%)	Acid in feed (gm/gm H)	Acid in solvent (gm/gm L)	Acid in extract (gm/gm L)	Acid in raffinate (gm/gm H)	(HTU) _{OE} (inches)	Material balance (% error)
30 87 96	0.25	25	2.00	0.0625	13	0.1712 0.1771 0.1771	0.0111 0.0102 0.0102	0.1019 0.1039 0.1036	0.0742 0.0663 0.0663	6.51 6.10 6.18	+3.16 +1.04 -0.18
32 86 94	0.25	50	2.00	0.0625	13	0.1712 0.1771 0.1771	0.0111 0.0102 0.0102	0.0987 0.1004 0.1000	0.0740 0.0717 0.0724	7.06 7.04 7.18	+0.52 +0.69 +0.18
31 88 93	1.00	25	2.00	0.0625	13	0.1712 0.1771 0.1771	0.0111 0.0102 0.0102	0.0982 0.0987 0.1025	0.0752 0.0728 0.0651	7.23 7.47 6.23	+0.70 +0.53 -0.89
29 85 95	1.00	50	2.00	0.0625	13	0.1712 0.1771 0.1771	0.0111 0.0102 0.0102	0.0942 0.0951 0.0963	0.0743 0.0785 0.0786	7.99 8.50 8.35	-1.76 +0.70 +0.35
18 68 75	0.25	25	2.00	0.0625	25	0.1754 0.1754 0.1754	0.0104 0.0100 0.0100	0.0996 0.1005 0.1011	0.0748 0.0675 0.0705	7.20 6.54 6.70	-0.70 -1.60 +0.52
17 67 76	0.25	50	2.00	0.0625	25	0.1754 0.1754 0.1754	0.0104 0.0100 0.0100	0.0961 0.0966 0.0970	0.0750 0.0749 0.0734	7.75 7.65 7.65	-2.44 -0.17 -0.87
19 65 74	1.00	25	2.00	0.0625	25	0.1754 0.1754 0.1754	0.0104 0.0101 0.0100	0.0981 0.0999 0.0997	0.0726 0.0705 0.0696	7.35 6.88 6.91	-2.62 -0.52 -1.34
20 66 73	1.00	50	2.00	0.0625	25	0.1754 0.1754 0.1754	0.0104 0.0100 0.0100	0.1000 0.0988 0.0984	0.0739 0.0712 0.0720	7.44 7.12 7.32	-1.05 -0.17 -0.35

geometry factors should be made from data taken at the same recycle rate. This discussion points out another phase of investigation on the pulse column which should be studied -- the relationship between pulse amplitude, pulse frequency, the (af) product, and their effect on extraction efficiency. The question arises whether the (af) product, regardless of the combination of amplitude and frequency which produces it, is the main factor to be considered or whether amplitude and frequency should be considered separately. In Table 4, the recycle rate in cm^3 per minute increases almost linearly with the (af) product, whereas the recycle rate in cm^3 per cycle shows a sharp break between the two low (af) products and the two high (af) products. An experiment in which the extraction efficiency of a pulse column is studied at various combinations of amplitude and frequency which produce a constant (af) product would provide information on this subject.

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2. Pulse amplitude and pulse frequency both affected extraction efficiency although the effects of amplitude, frequency, and the (af) product were not clearly defined. Extraction efficiency was highest at the lowest value (6.25 in./min.) of the (af) product regardless of the levels of the geometry factors. Extraction efficiency generally decreased with increasing pulsation.

3. For the range of pulse column operation covered, the extraction efficiency of the pulse column at different levels of pulsation was controlled by the recycle rate within the column.

4. The results of the investigation were reported by statistical analysis, at the 0.01 significance level. This method of approach was successful in separating the effects of the factors investigated in the presence of experimental error. The method also provided an objective view of the reproducibility of the data and of the reliability of the results. Standard error for the individual runs in terms of $(\eta)_{OE}$ was 0.12 in.

As a result of this investigation, the following recommendations are made for further work.

1. The effect of hole diameter on the extraction efficiency of a pulse column should be investigated at several levels to determine the nature of the effect. Additional work with this factor should include a hole diameter of 1/8 in. since this is a common value which is used in commercial pulse columns. A lower limit of hole diameter could be taken as 1/32 in.

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