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FLOODING CHARACTERISTICS OF A PULSE EXTRACTION COLUMN

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FLOODING CHARACTERISTICS OF A PULSE EXTRACTION COLUMN

by

G. H. Beyer and R. B. Edwards

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ABSTRACT

An investigation was made of flooding in a 1-in. diam. 10-plate pulse column, using the hexone-water system. Flooding was demonstrated to be caused either by inadequate pulsation or by excessive pulsation. An analysis of column operation led to the derivation of an equation for predicting conditions of inadequate pulsation, and for establishing the amount of liquid recycled under any operating conditions.

A simple, rapid procedure for the determination of flooding caused by excessive pulsation was developed. This type of flooding was found to be extremely sensitive to plate-wetting characteristics.

INTRODUCTION

In recent years liquid-liquid extraction has become increasingly important and considerable attention has been given to the development of more efficient types of extractors. One important way of improving extraction efficiency is to pulse the fluid contents of sieve-plate and packed extraction columns.

Pulsed sieve-plate extraction columns are called pulse columns. The plate perforations in a pulse column are usually so small that counterflow of the two liquid phases due to density difference does not occur. However, application of a cyclical pulsation to the column provides the additional energy necessary to force the liquids through the plates. The small plate perforations of a pulse column provide high fluid velocities and small drops, creating the turbulence and transfer area necessary for high extraction rates.

The height of column and the maximum allowable column throughput are the quantities required to design a pulse column for a specific extraction operation. Some studies have dealt with the extraction efficiency, which controls the height of a column,

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but little attention has been given to the limiting conditions of flow in a pulse column. The limiting conditions of flow are identified by flooding--defined as the entrainment of light-liquid phase with the heavy-liquid effluent, or vice versa. It is this latter aspect of column design which was the principal subject of this investigation.

The general performance characteristics of pulse columns were presented by Sege and Woodfield (4). The relationship illustrated in Fig. 1 was observed between certain types of phase dispersion, pulse frequency (at constant amplitude), and total column throughput. The various regions of pulse column operation were designated as follows:

- (A) Flooding region due to insufficient pulsing.
- (B) Mixer-settler region, characterized by large drop size, high stability of operation, a clear heavy-phase layer above each plate during a portion of the upward pulse, and a clear light-phase layer below each plate during a portion of the downward pulse.
- (C) Emulsion-type region, characterized by small drop size and uniform dispersion of phases.
- (D) Unstable region, characterized by operation near the flooding point, local flooding, and irregular dispersion.
- (E) Flooding region due to excessive pulsation.

Sege and Woodfield stated that at low frequencies the capacity of the column is equal to the pulsed volume velocity (the pulse amplitude-frequency product). Thus, the flooding curve at low frequencies was shown as a straight line through the origin. At higher frequencies the flooding curve began to deviate from the pulsed volume velocity line, first passing through a maximum and then decreasing. Finally a limiting frequency (or amplitude-frequency product) was reached, above which no countercurrent flow through the column was possible. Other investigators (3,5) also reached the conclusions illustrated in Fig. 1.

At a given throughput, flooding of a pulse column can be caused either by inadequate pulsation or by excessive pulsation. Decreasing the pulse frequency causes flooding when the pulsation becomes insufficient to pump the feed streams through the column. Increasing the pulse frequency causes flooding when the pulsation causes emulsification, or the rate of countercurrent flow decreases because of the small drop size.

These two types of flooding were also described in a study by Cohen and Beyer (1). They presented a simplified analysis of the operating mechanism of a pulse column for conditions of

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FIG. 1 PULSE COLUMN OPERATING CHARACTERISTICS

inadequate pulsation, in which the effect of the flow of light-liquid phase to the column was considered. In the same analysis, conditions of vigorous pulsing were shown to be accompanied by a definite amount of recycle, or back-mixing, within the column. Their work indicated that the simple relationship between total throughput and pulsed volume velocity indicated in Fig. 1 is an over-simplification.

The major purpose of this investigation was to initiate a systematic study of the pulse column operating variables which influence flooding. The operating variables considered were pulse frequency, pulse amplitude, light-liquid flow rate, and heavy-liquid flow rate. An equation is developed for predicting conditions of inadequate pulsation. Experimental data are presented in support of this equation.

APPARATUS AND PROCEDURE

A 1-in. diam., 10-plate pulse column with a 2-in. plate spacing was used. The column consisted of stacked glass sections, with gaskets and plates inserted between the glass sections as shown in Fig. 2. Four tie rods surrounding the glass sections placed the entire assembly in compression. The contacting section of the column was 18 in. high and consisted of nine sections of heavy-walled glass tubing, each 1-in. i.d. and 1-7/8 in. high. End sections of the column were made from 2-in. long sections of 3-in. i.d. glass tubing fitted to 2-in. long conical glass sections which reduced the diameter of the end sections to the diameter of the column. A 1-in. length of 1-in. i.d. tubing was fitted to the conical section, giving a total over-all length of 5 in. for the end section. The plates were made from 2-in. squares of 26 ga. stainless steel sheets perforated with 1/32-in. diam. holes punched on staggered centers 0.55 in. apart, providing 25 per cent free area. The 1/16-in. teflon gaskets located on each side of each plate were punched with a 1-in. diam. hole to match the inside diameter of the glass sections.

The schematic flow diagram presented in Fig. 3 illustrates pulse column assembly and operation. The hexone-water system was selected for this investigation; no solute was used since flooding, rather than extraction, was studied. Tank (A), containing the aqueous feed (heavy-liquid phase) saturated with hexone, and tank (B), containing the organic feed (light-liquid phase) saturated with water, were elevated so that flow was maintained by gravity. Quick-closing valves (C) were installed in each feed line. Each feed stream was metered through a needle valve and rotameter (D).

The aqueous feed stream entered the top of the column. The aqueous effluent stream passed from the bottom of the column through an adjustable leg (F) and was collected in a glass flask (H). A glass check valve (G) was located in the aqueous effluent line

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Fig. 2 - Close-up photograph showing stacked column construction

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FIG. 3 SCHEMATIC FLOW DIAGRAM OF PULSE COLUMN AND AUXILIARY EQUIPMENT

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to eliminate backflow in the flexible leg during the downstroke of the pulse. The adjustable leg was used to control the position of the interface within the column.

The organic feed stream entered the bottom of the column. The organic effluent stream left the settling section at the top of the column through an overflow tube and was collected in a glass flask (I).

Pulsation of the fluid contents of the column was introduced through a chamber located at the base of the column. Since the top of the column was open to the atmosphere, only liquid in the column and liquid in the pulse line between the pulse generator (J) and the bottom of the column was pulsed. Pulsation was produced either by a duplex diaphragm proportioning pump with the check valves removed or by a reciprocating brass bellows. Both pulsators possessed sinusoidal variation of displacement with time.

Amplitude, frequency and wave form are necessary to identify the characteristics of a pulse. A sinusoidal pulse wave form was used in this investigation, as well as in the majority of pulse column studies reported by other workers. The pulse amplitude was defined in this investigation as the total linear displacement of the fluid contents in the column cross-section, from one extreme position to the other. The product of the pulse amplitude and the column cross-sectional area was equal to the pulse volume.

Two methods of measuring pulse amplitude (or volume) were used. At low pulse frequencies and amplitudes, the pulse volume was measured by the variation in height of a column of fluid in a buret. Installation of such a pulse volume measuring buret (K) is illustrated in Fig. 3.

The second method of measuring pulse amplitude consisted of measuring the total linear displacement of the fluid directly in the column. A sharply pointed probe attached to a micrometer was mounted at the top of the pulse column, as shown in Fig. 4. By raising the point of the probe until it just broke free from the liquid surface at the bottom of the downsurge and by lowering it until it just made contact with the liquid surface at the top of the upsurge, a measurement of the fluid displacement was possible. The effect of surface tension tended to prevent the liquid from breaking free from the point of the probe so that a static correction was necessary.

The pulse frequency was determined by a visual count of the number of pulses occurring during a known interval of time. A sufficient number of pulses were observed so that the frequency could be calculated with an error of less than 0.1 cycle/min.

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Fig. 4 - Close-up photograph showing installation of micrometer probe used to measure pulse volume

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Before any column runs were made, the aqueous and organic phases were mutually saturated by pumping both phases through the column several times. The pulse column was usually placed in operation at a given pulse frequency, with flow rates adjusted to the desired values by the needle valves and rotameters. The principal interface was maintained at the top of the column by the flexible leg.

After the flow rates and the interface level were constant for a period of at least a half hour, the column was assumed to be at steady state in the normal operating region (region composed of areas B and C in Figure 1). At a given organic flow rate, pulse frequency, and aqueous flow rate, the amplitude was gradually decreased until flooding was observed. After each decrease of the pulse amplitude, the column was allowed to return to steady state operation unless flooding occurred. The transition from normal operation to flooding was usually well defined by an uncontrollable rising of the interface at the top of the column. Simultaneously, a buildup of an organic phase layer in the end section beneath the bottom plate was observed. At incipient flooding, the flow rates were measured by collecting the effluent streams during three consecutive 5-min. increments of time and measuring these volumes. Column operation was then interrupted to measure the pulse volume.

INVESTIGATION

Flooding Caused by Inadequate Pulsation

For mixer-settler type operation, a definite minimum rate of pulsation exists which is just sufficient to push upwards the amount of light liquid fed, and to pull downwards the amount of heavy liquid fed. Flooding caused by inadequate pulsation is not governed by hydrodynamic considerations; it is caused by the inability of the pulse to pump the desired amount of fluid through the plates.

An equation will now be derived from an analysis of column operation at inadequate pulsation. In the following derivation, operation will be considered at the bottom section of the column immediately below the lowest plate, as indicated by the level P in Fig. 5. It will be assumed that the organic and aqueous phases are both fed continuously to the column at constant rates, but that organic and aqueous effluent streams both leave the column only during the pulse upsurge. Also, it will be assumed that the pulse wave form is sinusoidal.

Consider the upward flow of fluid at P as positive. Then,

$$Q = \frac{v}{2} \sin(2\pi ft) + Gt, \quad (1)$$

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where

Q = net quantity of fluid in cc. which has flowed upward past level P (See Figure 5) at any time t in min. from the time $t = 0$.

G = light phase (organic) flow rate, cc./min.

f = pulse frequency, cycles/min.

v = pulse volume, the volume displaced during the pulse movement of the fluid content of the column from one extreme position to the other, cc./cycle

and

$$V_p = vf \quad (2)$$

where

V_p = pulsed volume velocity, cc./min.

In equation (1), the quantity, Gt , represents the amount of fluid which has passed P as a result of the continuous feed of organic phase into the bottom of the column. Likewise, the quantity, $\frac{v}{2} \sin(2\pi ft)$, represents the amount contributed by the sinusoidal pulse. Equation (1) is illustrated in Fig. 6.

Differentiation of equation (1) with respect to time gives the velocity past P at any time.

$$dQ/dt = \pi v f \cos(2\pi ft) + G. \quad (3)$$

Equation (3) is illustrated in Fig. 7.

The fluid volume pulled down the column on each down-surge, q_d , is obtained by integrating equation (3) between the limits of t_1 and t_2 , where t_1 and t_2 are the first and second roots, respectively, of equation (3) when the velocity is zero as shown in Figure 7. Thus,

$$q_d = - \int_{t_1}^{t_2} [\pi v f \cos(2\pi ft) + G] dt \quad (4)$$

or

$$q_d = v \cos\phi + (2\phi - \pi)G/2\pi f. \quad (5)$$

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FIG. 5 SCHEMATIC ILLUSTRATION OF BOTTOM OF PULSE COLUMN

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FIG. 6. Q VERSUS t AT LEVEL P FOR A SINE-WAVE PULSE FORM.

FIG. 7. dQ/dt VERSUS t AT LEVEL P FOR A SINE-WAVE PULSE FORM.

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where

$$\phi = \arcsin (G/\pi v f). \quad (6)$$

Because of the check valve located in the aqueous effluent line, no backflow is assumed to exist in the flexible leg so that the downward movement of fluid occurs only in the column.

The rate at which fluid flows down the column is then obtained from the product of the pulse frequency and q_d as given by equation (5). The rate of downward flow must be at least equal to the aqueous flow rate, L , since the aqueous phase will be drawn down through the column only when the fluid velocity is negative at P. If the rate of downward flow is greater than the aqueous flow rate, a condition will exist in which liquid in the column is recycled. Recycle, or back-mixing, represents the return of fluid back from whence it came. When recycle is present, therefore, some fluid can be expected to pass through a single plate more than once, and some cocurrent flow will be superimposed upon the net countercurrent flow of both phases.

For the general case, letting V_r represent the recycle rate in cc./min.,

$$\frac{V_r + L}{f} = v \cos \phi + (2\phi - \pi)G/2\pi f, \quad (7)$$

or

$$V_r = V_p \cos \phi + G\phi/\pi - G/2 - L. \quad (8)$$

When $V_r > 0$, conditions of back-mixing exist in the column. When $V_r = 0$, no recycle is present and the pulsation is just adequate to pump L cc./min. of aqueous phase down the column. When $V_r < 0$, the pulsation is not adequate to pump the required amount of aqueous phase down the column and flooding occurs.

A similar analysis of column operation considering the upsurge also results in equation (8), indicating the recycle on the upsurge is equal to the recycle on the downsurge. On the upsurge, fluid flows not only upward through the column, but also upwards through the aqueous effluent line. Thus, aqueous phase leaves through the flexible leg, while organic phase plus recycled fluid flows up the column.

Equation (8) represents an exact expression of the flow behavior of a pulse column, but it is cumbersome to use in the form given. Through the use of a series expansion of equation (8), it may be shown (2) that an excellent approximation to equation (8) is given by

$$V_r = V_p (\cosh \theta) - G/2 - L, \quad (9)$$

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where

$$\theta = G/\pi V_p. \quad (10)$$

At incipient flooding where $V_r = 0$, rearrangement of equation (9) gives

$$\pi L/G + \pi/2 = (\cosh \theta)/\theta. \quad (11)$$

Equation (11) is a convenient expression for predicting conditions of flooding due to inadequate pulsation. The left side of the equation is a function only of the flow rate ratio, a quantity usually established by the stage requirements of a given extraction problem. With the flow rate ratio known, the value of θ may be determined, establishing the minimum pulsed volume velocity necessary for any value of G . It should be noted that when the quantity, $\pi L/G + \pi/2$, is greater than the quantity, $(\cosh \theta)/\theta$, a condition of negative recycle, or flooding, exists. When the reverse is true, normal operation accompanied by recycle occurs.

Equation (11) indicates that total throughput is inadequate for describing the conditions of flooding caused by inadequate pulsation. Instead, each individual flow rate must be considered. To compare equation (11) with the flooding relation presented by earlier workers in Fig. 1, equation (11) can also be written as

$$G + L = V_p (\cosh \theta) + G/2. \quad (12)$$

It may be recalled that the equation of the inadequate pulsation line illustrated in Fig. 1 is

$$G + L = V_p. \quad (13)$$

Equations (12) and (13) may be compared by plotting $G + L$ against V_p at constant G , as shown in Fig. 8.

Figure 8 predicts that throughputs greater than the pulsed volume velocity should be possible. Equation (12) is represented in Fig. 8 by a slightly curved line which nearly parallels the straight line of equation (13). Because $\theta \leq 1$, the line representing equation (12) terminates at an abscissa of $V_p = G/\pi$; the ordinate for this point is G . When the total throughput is G , the minimum ordinate for both flooding lines in Fig. 8, equation (13) predicts a pulsed volume velocity equal to G will be necessary to avoid flooding. However, the flooding line given by the theoretically derived equation (12) predicts that a pulsed volume velocity of only G/π will be required. Such a low pulsed volume velocity is possible

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FIG.8 COMPARISON OF DERIVED FLOODING EQUATION
PROPOSED BY EARLIER INVESTIGATORS

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because the organic feed entering the bottom of the column enhances the pulse upsurge. Thus, the volume pushed upwards through a plate is larger than if it were due to the pulsation alone.

A number of flooding runs were made to test equation (11) experimentally. A series of flooding runs were made at an aqueous flow rate of 100 cc./min. in which pulse frequencies of 17.5, 35.7, 59.9 and 72.6 cycles/min. were studied. The results of runs made at different frequencies with aqueous flow rate held constant are given in Table 1. The data are compared with equation (11) in Fig. 9. The data substantiate the conclusion that flooding due to inadequate pulsation is, indeed, a function of the pulsed volume velocity, rather than a function of pulse volume and pulse frequency acting independently of one another.

The data shown in Fig. 9 indicated a tendency for flooding to occur with a slight amount of recycle present, so that the column appeared to flood at higher pulsed volume velocities than predicted. One possible explanation of why a slight recycle would be present was the existence of a condition which may be designated as "forced recycle" - the inability of a droplet forming on the surface of a plate to break free before being sucked back through the perforation on the reverse pulse surge. Also, the check valve failed to close immediately as the pulse began its downsurge, thereby allowing some fluid to be pulled back into the bottom of the column from the flexible leg. The agreement of the data with the equation, however, was sufficiently good so that additional modification of the equation was considered unnecessary.

In addition to the flooding studies made at an aqueous flow rate of 100 cc./min., runs were made at nominal aqueous flow rates of 50, 200 and 300 cc./min. The experimental results of the study of different aqueous flow rates are given in Table 2, and the data are compared in Fig. 10 with the theoretical relation. Fair agreement of the data with the equation existed, although most of the points fell slightly below the line as described above. However, several flooding points for runs at high aqueous flow rates were above the line in the region where flooding should occur, indicating that operation with negative recycle was possible without flooding. In these runs at high aqueous flow rates, the check valve was observed not to close on the pulse downsurge so that the aqueous effluent flowed from the column continuously, instead of leaving only on the upsurge as assumed in the derivation of the flooding equation. Apparently in these runs, such a low flexible leg position was used that fluid could flow from the column simply due to the effect of gravity, without the aid of pulsation.

In addition to the assumptions made in the derivation of the equation, the equation is further limited by the alternate type of flooding caused by excessive pulsation. The transition region between flooding caused by inadequate pulsation to flooding caused by excessive pulsation is illustrated in Fig. 1 as that portion of the flooding curve deviating from the pulsed volume

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Table 1. Results of Inadequate Pulsation Studies at Different Pulse Frequencies with a Constant Aqueous Flow Rate

Nominal Aqueous Flow Rate = 100 cc./min.

Run	v	V _p	G	L	θ	cosh θ	$\frac{\cosh \theta}{\theta}$	$\frac{\pi L}{G} + \frac{\pi}{2}$	V _r
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Pulse frequency = 72.6 cycles/min.

NG-15	2.25	163.4	114.0	98.5	0.222	1.0248	4.62	4.28	11.9
HG-1-R	1.6	116.2	17.0	103.6	0.046	1.0011	21.76	20.71	3.2
HG-2-R	1.7	123.4	41.3	99.9	0.106	1.0055	9.48	9.17	3.6
HG-3-R	3.4	246.8	257.7	99.1	0.332	1.0556	3.18	2.78	32.6
HG-4-R	2.6	188.8	141.7	103.4	0.239	1.0287	4.30	3.86	20.0
HE-5-R	2.3	167.0	137.7	98.8	0.262	1.0345	3.95	3.83	5.2
HE-6-R	5.6	406.6	654.7	100.9	0.512	1.1340	2.21	2.06	32.8
HE-7-R	2.0	145.2	103.3	98.9	0.226	1.0257	4.54	4.58	- 1.6
HE-8-R	1.9	137.9	78.8	100.8	0.182	1.0166	5.58	5.56	0.9
HE-9-R	2.9	210.5	204.0	98.9	0.308	1.0478	3.40	3.10	19.7
HE-10-R	4.9	355.7	504.3	102.4	0.451	1.1035	2.45	2.21	37.9
HE-11-R	3.9	283.1	369.5	102.1	0.415	1.0874	2.63	2.44	20.9

Pulse frequency = 59.9 cycles/min.

WG-31-R	3.64	217.7	234.2	103.6	0.342	1.0591	3.10	2.96	9.9
WG-32-R	4.30	257.1	333.3	103.0	0.413	1.0865	2.63	2.54	9.7
WG-33-R	2.63	157.3	122.5	99.9	0.248	1.0309	4.16	4.14	1.1
WG-34-R	5.11	305.6	443.3	101.3	0.462	1.1087	2.40	2.29	15.9
WG-35-R	5.70	340.9	524.4	101.6	0.490	1.1225	2.29	2.18	18.9
WG-36-R	5.77	345.0	527.8	99.7	0.487	1.1210	2.30	2.16	23.1
EG-53-R	2.26	135.3	90.0	98.7	0.212	1.0225	4.82	5.02	- 5.4

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Table 1 (Continued)

Run	v	V _p	G	L	θ	cosh θ	$\frac{\cosh \theta}{\theta}$	$\frac{\pi L}{G} + \frac{\pi}{2}$	V _r
Pulse frequency = 35.7 cycles/min.									
EG-5	8.9	317.7	498.0	103.0	0.499	1.1271	2.26	2.22	6.1
HE-16-R	3.3	117.8	66.5	98.4	0.180	1.0162	5.64	6.22	-12.9
HE-17-R	9.3	332.0	432.9	99.5	0.415	1.0874	2.64	2.29	45.1
WE-46-R	7.81	278.8	382.4	98.8	0.437	1.0971	2.51	2.38	15.9
WE-47-R	7.80	278.5	370.3	98.9	0.423	1.0908	2.58	2.41	19.7
EE-52-R	3.78	134.9	91.8	103.0	0.217	1.0236	4.72	5.10	-10.8
Pulse frequency = 17.5 cycles/min.									
HE-12-R	7.0	122.5	43.7	101.7	0.114	1.0065	8.83	8.88	-0.2
HE-13-R	8.2	143.5	77.3	103.3	0.171	1.0147	5.93	4.77	3.7
HE-14-R	9.1	159.2	117.0	98.5	0.234	1.0275	4.39	4.22	6.6
HE-15-R	13.8	241.5	266.6	100.1	0.351	1.0623	3.03	2.75	23.1

Table 2: Results of Inadequate Pulsation Studies at Different Aqueous Flow Rates

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Run	f	v	V _p	G	L	θ	cosh θ	$\frac{\cosh \theta}{\theta}$	$\frac{\pi L}{G} + \frac{\pi}{2}$	V _r	22
Nominal aqueous flow rate = 50 cc./min.											
HE-18-R	72.6	1.3	94.4	81.7	51.3	0.275	1.0381	3.77	3.54	5.9	
HE-19-R	72.6	2.7	196.0	256.0	49.3	0.416	1.0878	2.61	2.17	35.9	
HE-20-R	72.6	2.2	159.7	165.3	48.2	0.329	1.0450	3.18	2.49	36.1	
HE-21-R	72.6	4.6	334.0	575.1	46.9	0.548	1.1540	2.10	1.83	50.9	
HE-22-R	72.6	1.15	83.5	20.5	47.4	0.078	1.0031	12.86	8.83	26.2	
HE-30-R	72.6	3.9	283.1	440.3	50.8	0.495	1.1251	2.27	1.93	47.5	
WG-37-R	59.9	2.05	122.8	120.5	49.9	0.312	1.0490	3.36	2.87	18.7	
WG-38-R	59.9	2.51	150.3	205.5	51.5	0.435	1.0961	2.52	2.36	10.4	
WG-39-R	59.9	3.16	189.3	302.0	51.0	0.508	1.1318	2.23	2.10	12.2	
WG-40-R	59.9	4.04	242.0	447.5	50.7	0.588	1.1780	2.00	1.93	10.6	
WG-41-R	59.9	4.35	260.6	495.8	49.9	0.606	1.1893	1.96	1.89	12.1	
EE-54-R	17.5	5.42	94.8	90.7	50.3	0.304	1.0465	3.44	3.31	3.5	
Nominal aqueous flow rate = 200 cc./min.											
EE-55-R	72.6	2.67	193.8	86.8	193.7	0.142	1.0101	7.11	8.58	-41.3 ^a	
WG-50-R	59.9	3.43	205.4	101.6	200.1	0.157	1.0123	6.45	7.76	-43.0 ^a	
WG-51-R	59.9	5.79	346.6	302.9	205.2	0.278	1.0389	3.74	3.70	3.5 ^a	
Nominal aqueous flow rate = 300 cc./min.											
HE-25-R	72.6	3.0	217.8	52.5	288.7	0.077	1.0030	13.02	18.85	-96.5 ^a	
HE-26-R	72.6	5.2	377.8	104.6	299.1	0.088	1.0039	11.41	10.55	27.6	
HE-28-R	72.6	2.95	214.2	34.2	291.0	0.051	1.0014	19.64	28.30	-93.6 ^a	

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^aCheck valve not seated, so that continuous flow of aqueous effluent existed.

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FIG. 9 COMPARISON OF THEORETICAL FLOODING EQUATION WITH EXPERIMENTAL RESULTS OBTAINED AT FOUR FREQUENCIES AND AN AQUEOUS FLOW RATE OF 100 cc./MIN.

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FIG.10 CONDITIONS AT INCIPIENT FLOODING DUE TO INADEQUATE PULSATION OBSERVED FOR THREE DIFFERENT AQUEOUS FLOW RATES.

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velocity line and extending to the maximum point of the flooding curve.

The theoretical analysis presented here may also be used to calculate the amount of recycle for various conditions of operation. Recycle represents a deterioration of the true countercurrent operation required for most efficient extraction. The recycle rate, V_r , might prove useful as a correlating device for pulse column extraction studies. It is suggested that most efficient operation might be realized in the transition region, where high throughputs and high pulsed volume velocities are possible with little or no recycle.

Flooding Caused by Excessive Pulsation

Flooding caused by excessive pulsation is a complex hydrodynamic phenomenon much more complicated than flooding due to inadequate pulsation. Flooding caused by excessive pulsation may be the result of either the low effective velocity of the small dispersed phase drops or the formation of a stable emulsion. Such flooding is a function of the physical properties of each phase, the plate wetting characteristics, and column geometry, as well as the primary operating variables.

Because of the high pulsed volume velocities necessary to produce flooding due to excessive pulsation, the experimental procedure used to observe flooding caused by inadequate pulsation was modified somewhat for runs in which excessive pulsation was studied. At a fixed pulse frequency, aqueous flow rate and organic flow rate, and with the primary interface controlled at the top of the column, the pulse volume was gradually increased until flooding occurred. As the second interface appeared at the bottom of the column, however, the violent pulsation of the second interface up and down through the bottom plate produced a very fine dispersion of organic drops in the bottom end section. This dispersion made it difficult to observe the appearance of the second interface and created a tendency to predict flooding prematurely. The drops became entrained with the aqueous effluent because the end section did not provide sufficient time for phase separation.

However, by allowing the formation of a secondary interface in the bottom end section and positioning this interface low enough so as not to come in contact with the bottom plate, the tendency toward emulsification was inhibited. The flooding capacity was obtained when the column was operated with an interface at both the top and the bottom of the column, as shown in Fig. 11. By keeping the position of these interfaces constant, the flow rates to the end sections of the column could be balanced against the flow rates through the contacting section. Under these conditions the maximum flow through the column without flooding was established. This method was rapid and simple and inhibited emulsification at the bottom plate of the column.

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FIG.II SCHEMATIC ILLUSTRATION OF PULSE COLUMN APPEARANCE
AT INCIPIENT FLOODING DUE TO EXCESSIVE PULSATION

That flooding caused by excessive pulsation is extremely sensitive to plate wetting characteristics is illustrated in Fig. 12, which presents the results of three different series of experiments. Series I represents experiments made about a month after a nitric acid wash was given to the column. The column was thoroughly flushed with distilled water to remove all traces of the nitric acid wash before the experiments of Series I were made. Series II represents an attempt to duplicate Series I, but Series II was made approximately a month later, during which time the column was not washed with nitric acid. Series III represents flooding runs made shortly after the column was washed with nitric acid, to test the possibility that plate wetting characteristics might be varying. Before the experiments in Series III were made, the column was thoroughly flushed with distilled water to remove all traces of the nitric acid wash.

Each series of experiments falls on a different straight line in Fig. 12, although the three lines are nearly parallel. The nitric acid wash effected better than a twofold increase in the capacity of the pulse column, as indicated by the results of Series III and II. The plate wetting characteristics clearly exert a large effect on flooding of a pulse column due to excessive pulsation and emphasize the need for careful control of plate wetting characteristics.

NOMENCLATURE

Q = net quantity of fluid in cc. which has flowed upwards past level P (see Figure 5) at any time t in min. from the time $t = 0$.

t = time, min.

G = light phase (organic) flow rate, cc./min.

L = heavy phase (aqueous) flow rate, cc./min.

f = pulse frequency, cycles/min.

v = pulse volume, the volume displaced during the pulse movement of the fluid contents of the column from one extreme position to the other, cc./cycle.

V_p = pulsed volume velocity, cc./min.

q_d = fluid volume pulled down the column on each downsurge, cc./cycle.

V_r = recycle rate, cc./min.

$V_p = vf$

$\phi = \arcsin (G/\pi vf)$

$\theta = G/\pi V_p$

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FIG. 12. EXCESSIVE PULSATION FLOODING LINES FOR DIFFERENT PLATE WETTING CHARACTERISTICS

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