Flooding Characteristics of a Pulse Extraction Column

R. B. EDWARDS and G. H. BEYER

Iowa State College, Ames, Iowa

An investigation using the hexone-water system was made of flooding in a 1-in.-diam. ten-plate pulse column. 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.

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 and thus create the turbulence and transfer area necessary for high extraction rates.

The height of column and the maximum allowable column throughput are quantities required in designing a pulse column for a specific extraction operation. Some studies have dealt with the extraction efficiency, which controls the height of a column, 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 heavy-liquid effluent, or vice versa. This latter aspect of column design was the principal subject of this investigation.

The general performance characteristics of pulse columns were presented by Sege and Woodfield (*4*), who observed the relationship, illustrated in Figure 1, 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 separation of phases into clear layers between plates during the quiescent portions of the pulse cycle.

C. Emulsion-type region, characterized by fairly uniform dispersion of the discontinuous phase and little change in phase dispersion throughout the pulse cycle.

D. Unstable region, characterized by local flooding and irregular dispersion at operation near the flooding point.

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 Figure 1.

At a given throughput, flooding of a pulse column can be caused by either inadequate or excessive pulsation. Decreasing the pulse frequency causes flooding when the pulsation becomes insufficient to pass 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 small drop size.

These two types of flooding were also described in a study by Cohen and Beyer (*1*), who presented a simplified analysis of the operating mechanism of a pulse column for conditions of inadequate pulsation, in which the effect of the flow of light-liquid phase to the column was considered. 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 Figure 1 is an oversimplification.

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. ten-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 Figure 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 1⅞ 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-gauge stainless steel sheets perforated with 1/32-in.-diam. holes punched on staggered centers 0.055 in. apart, providing 25% 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 Figure 3 illustrates pulse-column assembly and operation. The hexone-water system was selected for this investigation; no solute was used, as flooding, rather than extraction, was studied.

Pulsation of the fluid contents of the column was introduced through a chamber located at the base of the column. A glass check valve was installed in the aqueous effluent line to eliminate backflow in the flexible leg during the downsurge of the pulse. As the top of the column was open to the atmosphere, only liquid in the column and in the pulse line between the pulse generator 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

R. B. Edwards is at present with Eastman Kodak Company, Rochester, New York, and G. H. Beyer with the University of Missouri, Columbia, Missouri.

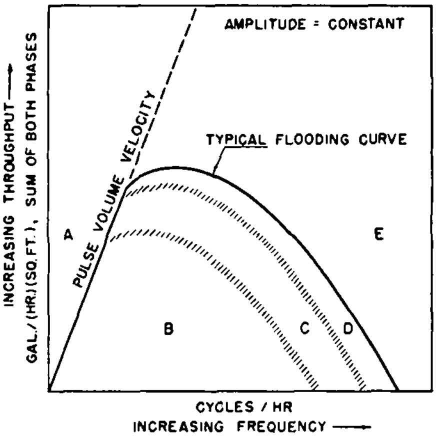

Fig. 1. Pulse-column operating characteristics.

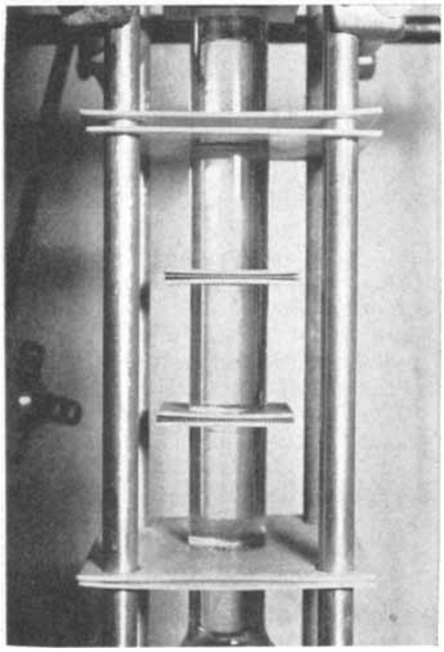

Fig. 2. Close-up photograph showing stacked-column construction.

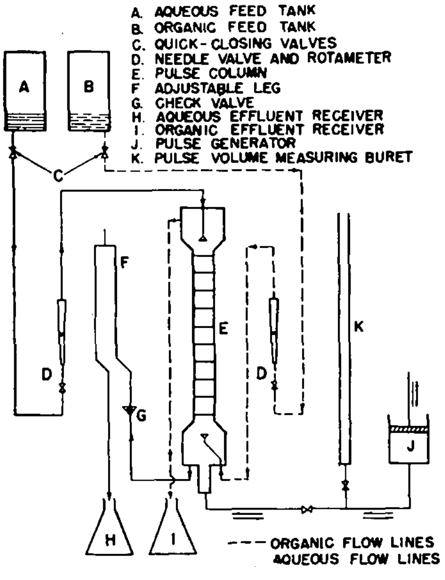

Fig. 3. Schematic flow diagram of pulse column and auxiliary equipment.

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 burette. 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 Figure 4. Raising the point of the probe until it just broke free from the liquid surface at the bottom of the downsurge and lowering it until it just made contact with the liquid surface at the top of the upsurge made possible a measurement of the fluid displacement. The effect of surface tension tended to prevent the liquid from breaking free from the point of the probe and so necessitated a static correction.

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 was observed so that the frequency might be calculated with an error of less than 0.1 cycle/min.

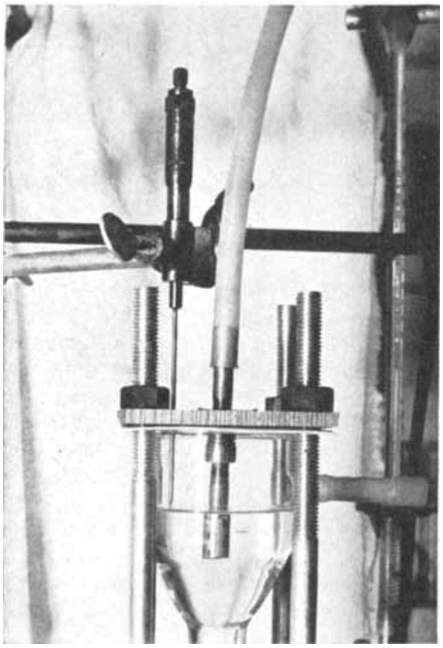

Fig. 4. Micrometer probe used to measure pulse volume.

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 (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 of operation, a definite minimum rate of pulsation exists which is just sufficient to push upward the amount of light liquid fed and to pull downward 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 pass 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 Figure 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. It will also be assumed that the pulse wave form is sinusoidal.

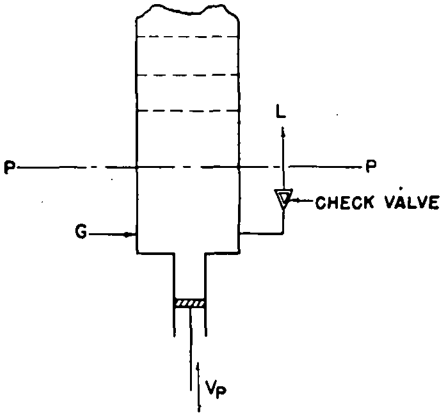

Fig. 5. Schematic illustration of bottom of pulse column.

If the upward flow of fluid at *P* is considered as positive, then

$$Q = \frac{v}{2}\sin(2\pi f t) + Gt \qquad (1)$$

and

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

where

V_p = pulsed volume velocity, cc./min.

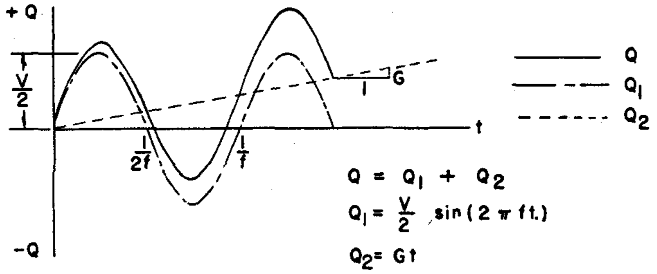

Fig. 6. Q vs. t at level P for a sine-wave pulse form.

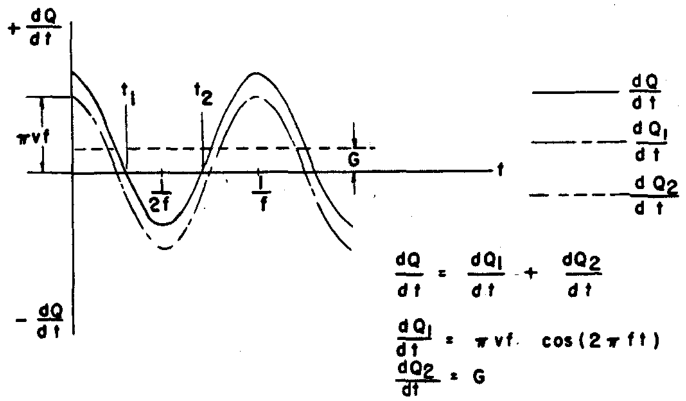

Fig. 7. dQ/dt vs. t at level P for a sine-wave pulse form.

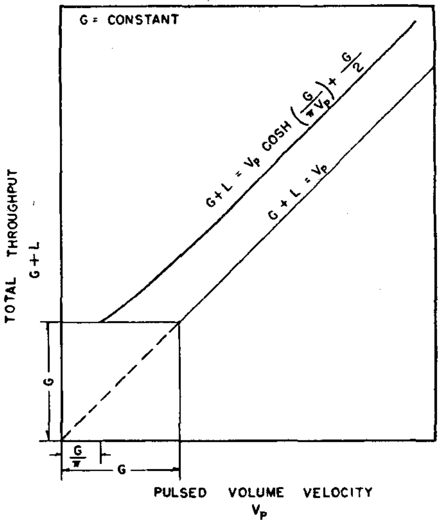

Fig. 8. Comparison of flooding equations.

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 $v/2 \sin(2\pi ft)$ represents the amount contributed by the sinusoidal pulse. Equation (1) is illustrated in Figure 6.

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

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

Equation (3) is illustrated in Figure 7.

The fluid volume that is pulled down the column on each downsurge 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

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

or

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

where

$$\phi = \arcsin(G/\pi vf) \quad (6)$$

Because of the check valve located in the aqueous effluent line, no backflow is assumed to exist in the flexible leg, and so 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, as 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 to its source. 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, with V_r representing 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 $V_r > 0$, back mixing exists in the column. When $V_r = 0$, no recycle is present and the pulsation is just adequate to pass L cc./min. of aqueous phase down the column. When $V_r < 0$, the pulsation is not adequate to pass 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 that 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 upward through the aqueous effluent line. Thus aqueous phase leaves through the flexible leg, and 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 of it is given by

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

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 of only 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 and the minimum pulsed volume velocity necessary for any value of G thus established. It should be noted that when the quantity $\pi L/G + \pi/2$ is greater than the quantity $(\cosh\theta)/\theta$, 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 indi-

TABLE 1. INADEQUATE PULSATION STUDIES AT DIFFERENT PULSE FREQUENCIES

Nominal Aqueous Flow Rate = 100 cc./min.

Run	v	V_p	G	L	θ	cosh θ	(cosh θ)/θ	$\pi L/G + \pi/2$	V_r
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
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	5.77	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

vidual flow rate must be considered. To compare Equation (11) with the flooding relation presented in Figure 1 by earlier workers, 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 Figure 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 Figure 8.

Figure 8 predicts that throughputs greater than the pulsed volume velocity should be possible. Equation (12) is represented in Figure 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 Figure 8, Equation (13) predicts that 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 because the organic feed entering the bottom of the column enhances the pulse upsurge. Thus the volume pushed upward through a plate is larger than that due to pulsation alone.

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

The data shown in Figure 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 are given in Table 2, and the data are compared in Figure 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, and so 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 owing to the effect of gravity, without the aid of pulsation.

The theoretical analysis presented here may also be used to calculate the amount

Table 2. Inadequate Pulsation Studies at Different Aqueous Flow Rates

Run	f	v	V_p	G	L	θ	cosh θ	(cosh θ)/θ	$\pi L/G + \pi/2$	V_r
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*
WG-50-R	59.9	3.43	205.4	101.6	200.1	0.157	1.0123	6.45	7.76	−43.0*
WG-51-R	59.9	5.79	346.6	302.9	205.2	0.278	1.0389	3.74	3.70	3.5*
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*
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*

*Check valve not seated; so continuous flow of aqueous effluent existed.

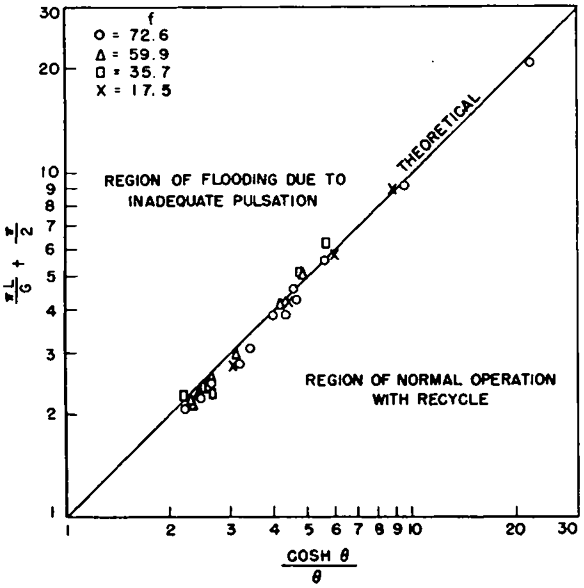

Fig. 9. Comparison of theoretical flooding equation with experimental results obtained at four frequencies and an aqueous flow rate of 100 cc./min.

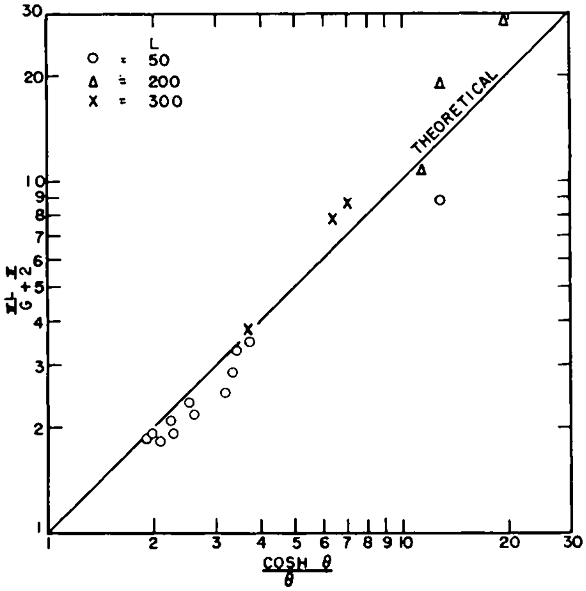

Fig. 10. Conditions at incipient flooding due to inadequate pulsation observed for three different aqueous flow rates.

of recycle for various conditions of operation. Recycle represents a deterioration of true countercurrent operation. The recycle rate V_r might prove useful as a correlating device for pulse-column-extraction studies.

NOTATION

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 $t = 0$.
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, $v \times f$
q_d = fluid volume pulled down the column on each downsurge, cc./cycle
V_r = recycle rate, cc./min.
ϕ = arcsin $(G/\pi v f)$
θ = $G/\pi V_p$

ACKNOWLEDGMENT

The authors wish to express their appreciation to the Ames Laboratory of the Atomic Energy Commission and to the Institute for Atomic Research, Iowa State College, for support of this project.

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Presented at Nuclear Science and Engineering Congress, Cleveland.