1954

Flooding characteristics of a pulse column

Robert B. Edwards
Iowa State College

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UMI®
FLOODING CHARACTERISTICS
OF A PULSE COLUMN

by

Robert B. Edwards

A Dissertation Submitted to the
Graduate Faculty in Partial Fulfillment of
The Requirements for the Degree of
DOCTOR OF PHILOSOPHY

Major Subject: Chemical Engineering

Approved:

Signature was redacted for privacy.

In Charge of Major Work

Signature was redacted for privacy.

Head of Major Department

Signature was redacted for privacy.

Dean of Graduate College

Iowa State College

1954
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SUMMARY

An investigation was made of flooding in a 1-in. diam. pulse column, using the hexone-water system. A 10-plate column was constructed of stacked glass sections held in compression by four tie rods. The plates, spaced 2-in. apart, were perforated with 1/32-in. diam. holes to give a plate free area of 25 per cent.

Flooding was demonstrated to be caused either by inadequate pulsation or by excessive pulsation. Earlier investigators concluded that the pulsed volume velocity equals the total throughput at incipient flooding due to inadequate pulsation, but such a conclusion was shown to be an oversimplification. A theoretical flooding equation,

\[ V_r = V_p \cosh(G/w_p) - G/2 - L, \]

was derived, where, \( V_r \) is the rate of recycle (or vertical back mixing), \( V_p \) is the pulsed volume velocity (the product of pulse volume and frequency), \( G \) is the organic flow rate, and \( L \) is the aqueous flow rate. When \( V_r > 0 \), recycle was present; when \( V_r = 0 \), incipient flooding existed; and when \( V_r < 0 \), flooding due to inadequate pulsation occurred. Flooding data for conditions of inadequate pulsation were well correlated by use of this equation.

Incipient flooding predicted by the flooding equation was a function only of the primary operating variables, but the range of
applicability of the equation was limited by the alternate type of flooding caused by excessive pulsation. The equation permitted the calculation of the amount of recycle existing in a pulse column at various conditions of operation. Since recycle represents a deterioration of true countercurrent operation, recycle rate possibly may prove useful as a correlating device in pulse column extraction studies.

Pulse column performance was observed to be extremely sensitive to plate wetting. Variation in the plate wetting characteristics as produced by a nitric acid wash was shown to result in more than a twofold increase in the capacity of a pulse column. Hold-up was apparently less sensitive than flooding to changes in plate wetting. Variation in the plate wetting characteristics during the course of this investigation restricted the development of any correlation of flooding caused by excessive pulsation or of pulse column hold-up.

A simple procedure was demonstrated for measuring the hold-up of a pulse column. Success of the method required that flow of both phases to the column and the pulsation be stopped instantly and simultaneously and that no counterflow occur in the column when not pulsed. When such requirements were met, it was possible to effectively "freeze" the contents of the column while at steady state operation and make hold-up measurements with a ruler.

A satisfactory method was developed for the determination of
flooding caused by excessive pulsation. The flooding capacity was obtained when the column was operated with an interface at both the top and the bottom of the column. By keeping the position of these interfaces constant, the flow rates to the end sections of the column could be balanced against the flow through the contacting section. Under these conditions the maximum flow through the column without flooding was established. This method was rapid, simple and inhibited emulsification in the bottom end section of the column.

The transition from flooding caused by inadequate pulsation to flooding caused by excessive pulsation must be investigated further before the limitations of the theoretical flooding equation can be defined. Studies which will provide a correlation of flooding conditions due to excessive pulsation are recommended. However, until a basis for establishing the effect, as well as a characteristic measurement, of plate wetting is developed, accurate prediction of pulse column performance will not be possible.
INTRODUCTION

Liquid-liquid extraction as a separations process has become increasingly important in recent years and considerable attention has been given to the development of more efficient types of extractors. Most liquid-liquid extraction equipment recently devised depends on the addition of energy from a source external to the extraction system to obtain high extraction efficiencies. Specifically, 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 and are the subject of considerable investigation because of their high extraction efficiencies. The plate perforations in a pulse column are usually so small that countercflow due to density difference of the two liquid phases is not possible, but the application of a cyclical pulsation to the fluid contents of the column provides the additional energy necessary to overcome resistance to flow. The small plate perforations of a pulse column provide high fluid velocities and small drops which create the turbulence and the large transfer area necessary for high extraction rates.

In the design of a pulse column, it is necessary to calculate the height of column necessary for a specified extraction, and the maximum allowable column throughput, in order to establish the column diameter.
Considerable study has dealt with the extraction efficiency of pulse columns, but little attention has been given to the limiting conditions of flow in a pulse column. The limiting conditions of flow are identified by the condition of flooding, defined as the entrainment of the light-liquid phase with the heavy-liquid effluent, or vice versa. It is this latter aspect of column design which is the principal subject of this investigation.

The general performance characteristics of pulse columns are presented in an article by Sege and Woodfield (3). A relationship was observed between certain types of phase dispersion, the range of pulse frequency at any given amplitude, and the total column throughput. The various types of pulse column operation, illustrated in Figure 1, 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 upwards pulse, and a clear light-phase layer below each plate during a portion of the downwards 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.
AMPLITUDE = CONSTANT

TYPICAL FLOODING CURVE

INCREASING THROUGHPUT

INCREASING FREQUENCY

CYCLES / HR

INCREASING VOLUME VELOCITY

PULSE VOLUME VELOCITY

GAL./HR(SQ.FT.), SUM OF BOTH PHASES

A

B

C

D

E

FIG. 1 PULSE COLUMN OPERATING CHARACTERISTICS
Sege and Woodfield stated that at low frequencies the capacity of the column is equal to the pulsed volume velocity. The pulse volume velocity, which is a measure of the intensity of pulsation, was defined in units such as gallons pulsed/(hour)(square foot of column cross section) and was calculated by multiplying the pulse frequency in cycles/hour by the pulse displacement in gallons and then divided by the column cross section in square feet.

In Figure 1, then, the flooding curve at low frequencies was shown as a straight line, with the maximum possible total throughput at a given frequency equal to the pulsed volume velocity. At higher frequencies, the limiting total throughput became increasingly less than the pulsed volume velocity, and the flooding curve began to deviate from the pulsed volume velocity line. As the frequency was further increased, the flooding curve became increasingly lower than the pulsed volume velocity, first passing through a maximum and then decreasing as indicated in Figure 1. Finally a limiting frequency (or amplitude-frequency product) was reached, above which no countercurrent flow through the column was possible.

Two mechanisms of flooding are suggested by Figure 1. Selecting a point in region (B) in Figure 1 and following along a horizontal line of constant total throughput through this point, flooding is observed to occur either when the frequency becomes too low, or when the frequency becomes too high. Flooding of a pulse column is indicated, therefore, to be caused either by inadequate pulsation or by
excessive pulsation. By decreasing the pulse frequency along the line of constant total throughput, flooding occurs when the pulsation is insufficient to pump the necessary amount of each phase through the column. Flooding is also possible by increasing the pulse frequency at the same total throughput until the rate of countercurrent flow becomes too small because of the small drops formed or perhaps emulsification.

Sege and Woodfield presented several excessive-pulsation flooding curves for several pulse amplitudes similar to Figure 1. No general correlation of the flooding data was given, although it was suggested that the pulse frequency raised to some power times the pulse amplitude might yield a single correlation of the data.

The two causes of flooding in a pulse column have also been described in an investigation by Griffith, Jasny, and Tupper (2). For the conditions of inadequate pulsation, they stated that at a given organic-to-aqueous flow rate ratio the volumetric flow rate is equal to the maximum quantity of light-liquid (organic) phase which can be pushed through a plate per pulse times the stroke frequency. Based on this analysis of operation, an equation was developed which predicted a straight line passing through the origin would be obtained by plotting the maximum total flow rate against the pulse amplitude-pulse frequency product. This equation was similar to the conclusion reached by Sege and Woodfield, although the equation required a plot such as Figure 1 to be restricted to a constant flow rate ratio.
Griffith, Jasny and Tupper indicated that the pulse column affords higher throughputs than packed columns but lower throughputs than spray columns. Operation in the region where flooding was caused by excessive pulsation was characterized by emulsification and the product of pulse amplitude and frequency was essentially constant.

Both causes 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 the conditions of inadequate pulsation, in which the effect on the pulse conditions 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 as indicated in Figure 1 was an oversimplification.

Wiegandt and Von Berg (4) have recently reviewed the history and progress of studies made on pulse columns and pulsed packed columns. They discussed the unclassified work of several investigators and presented a bibliography of classified as well as unclassified articles. The discussion of flooding in pulse columns by Wiegandt and Von Berg led to the same conclusions illustrated in Figure 1.

Because no extraction studies were planned, the hexone (methyl-isobutyl-ketone)-water system with no solute distributed between either phase was selected for this investigation. The use of such
a mutually saturated binary system eliminated effects accompanying any extraction within the column, such as changes in density, viscosity and interfacial tension. This system has been used by others in capacity studies on other types of extraction equipment and, therefore, provides a basis for comparison of the pulse column with other extraction columns.

In addition to the physical properties of the liquid-liquid system, a large number of design variables and operating variables affect the flooding capacity of a pulse column. Design variables of a pulse column include the plate spacing, size of plate perforations, plate free area, column diameter, column height and plate material. Operating variables are the pulse frequency, pulse amplitude, light-liquid flow rate, heavy-liquid flow rate, and choice of continuous phase. Because of the extremely large number of runs required to investigate completely the effect of each variable on the flooding capacity of a pulse column, the following investigation of pulse column flooding was confined to a study of the operating variables only.

In addition to the paucity of flooding data for pulse columns, very little information has appeared in the literature on the determination of hold-up in pulse columns, where hold-up is defined as the volume fraction of dispersed phase in the column. The only report of hold-up measurements on pulse columns noted was that of Cohen and Beyer (1). They measured the phase volumes by draining the fluid contents of the column into a graduated cylinder at the conclusion of a run. The
liquid contained in the column end sections was included in the hold-up inventory, and so the values obtained were not true hold-up ratios. A portion of this investigation was concerned with the measurement of hold-up.

As stated earlier, the height of column required and the maximum permissible flow rates are essential facts which must be known for the design of pulse columns. A review of the literature has revealed that a number of workers have investigated the variation of column efficiency with respect to column design and operating variables, but relatively little attention has been given to column capacity studies, and no satisfactory flooding correlations have been made. Therefore, the major purpose of this investigation was to initiate a systematic study of pulse column variables as related to the flooding capacity.

This investigation was concerned with flooding caused by inadequate pulsation, as well as flooding caused by excessive pulsation. Although several earlier investigators have reached the conclusion, illustrated in Figure 1, that at incipient flooding due to inadequate pulsation the total throughput equals the pulsed volume velocity, this relationship is actually not correct. Consequently, a more exact expression was developed for establishing the operating conditions at flooding caused by inadequate pulsation. In addition, an experimental procedure was developed for the determination of flooding conditions caused by excessive pulsation, and a better understanding was obtained regarding the various factors which influenceflooding caused by excessive pulsation.
APPARATUS AND PROCEDURE

Description of Apparatus

Two types of construction were considered for the pulse column used in this investigation. The design most often used in previous investigations consisted of a single glass section, inside of which perforated plates were supported at intervals on a central rod. Such a single column is expensive since it requires uniform bore glass tubing and accurate machining of the plates.

An alternate design, shown in Figure 2, used stacked glass sections with gaskets and plates inserted between the glass sections. Four tie rods surrounding the glass sections placed the entire assembly in compression. The stacked column possesses considerable flexibility with regard to changes in column height, plate spacing and plate geometry. Also, the stacked column eliminates leakage caused by clearance between column walls and plates. Because of the unavailability of uniform bore glass tubing, elimination of leakage between plates and column walls, less expense and greater flexibility, the stacked column construction was used in this study.

The pulse column studied experimentally was a 1-in. diam., 10-plate column with a 2-in. plate spacing. The contacting section of the column was 18 in. in height 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
Fig. 2. Close-up photograph showing stacked column construction
were made from 2-in. long sections of 3-in. i.d. glass tubing fitted to conical glass sections also 2-in. in length. The conical section accomplished the reduction of the diameter of the 3-in. i.d. of the end section to the 1-in. i.d. of the column. A 1-in. length of 1-in. i.d. tubing was fitted to the conical section, giving a total overall length of 5-in. for the end section. The plates were made from 2-in. squares of number 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 gaskets located on each side of each plate were made from 1/16-in. teflon sheets and were punched with a 1-in. diam. hole to match the inside diameter of the glass sections.

Two such columns were constructed. The columns were identical so that experimental results obtained with one could be compared with those of the other. Both pulse columns with their auxiliary equipment are shown in Figure 3. The schematic flow diagram presented in Figure 4 illustrates pulse column assembly and operation.

The tank (A), containing the aqueous feed (heavy liquid phase) saturated with hexone, and the tank (B), containing the organic feed (light liquid phase) saturated with water, were 30-gal. stainless steel barrels. Both feed tanks were located above the columns, and flow was maintained by a hydrostatic head of about 10 ft. Quick-closing valves (C) were installed in the primary feed line leaving each tank. Each primary feed line was split by means of a tee into two secondary feed lines to provide the necessary feed streams to each column. Each feed
Fig. 3. Photograph showing installation of both pulse columns and auxiliary equipment.
FIG. 4 SCHEMATIC FLOW DIAGRAM OF PULSE COLUMN AND AUXILIARY EQUIPMENT
stream was metered through a needle valve and rotameter (D).

The aqueous feed stream entered the top of the column (E), and the aqueous effluent stream passed from the bottom of the column through an adjustable leg (F) and was collected in a glass flask (H). The adjustable leg of pure gum rubber tubing was used to control the position of the interface within the column. A glass check valve (G) was located in the aqueous effluent line to eliminate the backflow in the flexible leg produced by the cyclical pulsation.

The organic feed stream entered the bottom of the pulse column through a perforated inlet tube. Experimentation indicated a tendency to produce emulsification when the organic stream itself was "pulsed" into the column. To avoid this difficulty, the column was pulsed through a chamber located at the base of the column, and the organic phase was introduced directly into the column through the perforated inlet tube. The hydrostatic head furnished by the elevated organic feed tank maintained sufficient pressure in the organic feed line so that pulsation of the rotameter float was slight. Since the top of the column was open to the atmosphere, only the liquid in the column and in the pulse line between the pulse generator (J) and the bottom of the column was pulsed. 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 was accomplished by the use of
either of two different pulse generators, each of which possessed sinusoidal variation of displacement with time. The first of the two pulse generators installed was a duplex diaphragm proportioning pump with the check valves removed. Pulse volumes were varied between 0.0 and 26.0 cc./cycle by turning a stroke-length adjusting knob. The pulse volume represents the volume displaced during the pulse movement of the fluid contents of the column from one extreme position to the other. By means of a 3-step cone pulley, pulse frequencies of 17.5, 35.7 and 72.6 cycles/min. were possible with the diaphragm pulsator.

Another pulse generator, built to provide a greater range of pulse volumes, consisted of a 4.7-in. diam. brass bellows 4-in. long with one end held stationary. The other end of the bellows was closed with a blind flange and connected to the end of a reciprocating piston, adapted from the drive mechanism of a Lapp Pulsafeeder. The stationary end of the bellows was connected by a pulse transmission line with the chamber located at the base of the pulse column. The bellows pulsator operated at a frequency of 59.9 cycles/min. and was capable of generating pulse volumes up to 200 cc./cycle. The equipment was installed such that either of the two pulse generators could be used to pulse the fluid contents of either of the two columns.

Measurement of Pulse Characteristics

Amplitude, frequency and the wave form are the factors necessary to identify the pulse characteristics of a pulse column. A sinusoidal
pulse waveform was used in this investigation, as well as in the majority of pulse column studies reported by other workers. Two methods of measuring pulse amplitude were used. The pulse amplitude was defined in this investigation to represent the magnitude of the pulse movement of the fluid contents of the column from one extreme position to the other. Thus, the product of the pulse amplitude and the column cross-sectional area was equal to the pulse volume, which was defined previously.

At low pulse frequencies and pulse volumes, direct measurement of the pulse volume was possible by measuring the variation in height of a column of fluid pulsed in a buret. Installation of such a pulse volume measuring buret (X) in relation to the other equipment is illustrated in Figure 4.

Another procedure established later for the measurement of the pulse volume measured the pulse amplitude directly in the column. A sharply pointed rod attached to a micrometer was mounted at the top of the pulse column in such a fashion that the liquid level in the top calming section of the column could be measured. Installation of the micrometer with the attached probe is shown in Figure 5.

By lowering the point of the probe until it just made contact with the liquid surface at the top of the upsurge and by raising the point until it just broke free from the liquid surface at the bottom of the downsurge, a measurement of the magnitude of the fluid dis-
Fig. 5. Close-up photograph showing installation of micrometer probe used to measure pulse volume
placement in the column was possible. However, 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 needed. The procedure
used in measuring the pulse volume was as follows:

(I) With the pulse generator running and the point of the
probe completely immersed throughout the entire pulse
cycle, raise the point until liquid barely breaks free
at the bottom of each pulse and record the micrometer
reading.

(II) With the pulse generator running and the point of the
probe completely free of the fluid throughout the entire
pulse cycle, lower the point until liquid barely contacts
the point at the top of each pulse and record the micro-
meter reading.

(III) Stop the pulse generator and allow the liquid level to
come to rest so that the static correction may be made.
Repeat as in step (II).

(IV) With the pulse generator still at rest and with the point
submerged in the liquid which is maintained at the same
level as in step (III), repeat as in step (I).

The magnitude of the pulse movement of the column contents was
calculated from the following relation:

\[ \text{pulse amplitude} = [\text{(II)} - (I)] + [\text{(IV)} - (III)]. \]
Steps I and II gave the dynamic measurement, while steps III and IV provided the static correction. Multiplication of the pulse amplitude by the cross-sectional area of the fluid column at the point of measurement gave the pulse volume. Care was taken to insure that the pulse movement which was measured occurred only in a section of constant cross-sectional area. Good reproducibility was possible with this method of pulse volume measurement which measured directly the pulse displacement actually delivered to the column. However, the turbulence produced by very large pulse volumes created ripples on the surface of the liquid so that the precision of the measurement decreased with an increase in pulse volume.

The pulse frequency was determined by a visual count of the number of pulses occurring during a known interval of time. Periodic checks of pulse frequency were made during the course of the investigation and no variation with time was observed. A sufficient number of pulses were observed in each count so that the frequency could be calculated with an error of less than 0.1 cycle/min.

Experimental Procedure

The procedure followed in making column runs varied somewhat depending on the type of flooding being studied, since, as stated earlier, flooding of a pulse column may be caused either by inadequate pulsation or excessive pulsation. Since the frequency of the pulse generators could be varied only by discrete increments while the pulse volume could
be varied continuously, the magnitude of pulsation was most easily
controlled by either increasing or decreasing the pulse volume.

Column runs in which flooding caused by inadequate pulsation
was studied were made by decreasing the pulse volume with all other
operating variables held constant until flooding occurred. In a
similar fashion, flooding caused by excessive pulsation was observed
by increasing the pulse volume. When the pulse column was flooding,
organic phase flowed out the aqueous effluent line and aqueous phase
usually, though not always, flowed out the organic effluent line.
Specific exceptions to the procedure presented here will be described
later in the discussion of the results of this investigation.

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 and with the flow rates adjusted to the desired
values by means of needle valves and rotameters. The principal inter­
face was maintained at the top of the column by means of the flexible
leg. The interface was controlled at the point where the expansion to
the calming section ended since it was desirable to have the position
at a point where a large cross-sectional area existed to minimize the
travel of the interface with column pulsation.

After the flow rates and the interface level were constant for a
period of at least a half hours, the column was assumed to be at steady
state in the normal operating region (region composed of regions B and C in Figure 1). An adjustment of the pulse volume to cause the type of flooding under study was then made, and the column was allowed to return to steady state operation provided the operating conditions permitted the column to operate in the normal operating region. In this fashion, the pulse volume was gradually decreased (or increased) until flooding was observed, at which point the pulse volume was re-adjusted to the conditions of incipient flooding and the column was returned to steady state operation in the normal operating region. Often a variation in the pulse volume of only 0.05 cc./cycle was sufficient to achieve the transition from flooding to the normal operating region, although in some runs the variation was as much as 0.5 cc./cycle. The flow rates were then determined by collecting the effluent streams during three consecutive 5-min. increments of time and measuring the volumes of each so collected. Column operation was then stopped in order that the column hold-up and pulse volume could be measured.

The method used in measuring hold-up required that all flow and pulsation to the column be stopped simultaneously and that no counter-flow due to density difference occur in the column. Hence, after the column was at steady state operation, the flow rates had been measured, and the hold-up measurement was ready to be made, the pulse generator was stopped and in essentially the same instant both quick-closing valves in the primary feed lines were closed. The flexible leg was then elevated slightly to prevent any liquid flowing down the column.
and altering the column hold-up. It was possible, therefore, to effectively "freeze" the contents of the column while at steady state operation and make hold-up measurements. Little, or no, counterflow did occur, and the two phases separated into two layers between each set of plates.

In this investigation, the hold-up measurement was made only over the contacting section of the pulse column, where the contacting section was considered to be that portion of the pulse column between the top and bottom perforated plates. The amounts of each phase contained in the end sections of the column were not included in the hold-up measurement. By measuring the depth of each organic phase layer between each set of plates and summing the individual measurements over the column height, it was possible to determine the hold-up of the column.

In the initial stages of this investigation, an alternate method of measuring hold-up was tested in which a manometer was connected across the ends of the column to measure the effective density of the liquid mixture contained in the column. Knowing the density of each phase, the hold-up of the column could be calculated from the effective density. However, measurements with the hold-up manometer gave results which were inconsistent and not reproducible so the method was considered unreliable. An interfacial tension effect apparently prevented transmission through the plate perforations of the pressure due to the hydrostatic head of fluid.

As mentioned earlier, hold-up has usually been defined as the
volume fraction of dispersed phase in the column by workers studying packed and spray columns. However, identification of a single dispersed phase in a pulse column is very difficult, if not impossible, under many conditions of pulse column operation. Therefore, hold-up was defined throughout this investigation as the volume of organic phase contained in the contacting section of the column divided by the total volume of the contacting section.
INVESTIGATION

Plate Wetting Effects

Soon after both pulse columns were placed in operation, a marked difference was observed in the appearances of the two columns supposedly operating at the same conditions. The difference was found to be the result of different plate wetting characteristics possessed by the two columns. Consequently, a cursory investigation was made in which both columns were operated simultaneously under identical operating conditions to establish what effect, if any, different plate wetting characteristics would have on hold-up. The purpose of the investigation was twofold. First, a basis for establishing a specific plate wetting condition and interface location for all hold-up and flooding studies was desired. Second, information concerning the effects produced by different plate washes, as well as the duration of these effects, was of interest.

In the following discussion, the column constructed first and installed on the right (see Figure 3) will be designated as column E, while the other column will be known as column G. Column E was cleaned with nitric acid, and after column E was placed in operation, the aqueous phase was observed to preferentially wet the plates. The plates of column G were washed briefly with hydrochloric acid, and when column G was placed in operation, the organic phase preferentially wet the plates.
Four different column conditions were selected and ten runs at each condition were made in the study of plate wetting. The four column conditions which were studied represented the four possible combinations of two interface positions and two plate washes. Variables held constant in all of the plate wetting experiments were the following:

- Pulse frequency = 35.5 cycles/min.
- Pulse volume = 11.0 cc./cycle.
- Nominal aqueous flow rate, L = 200 cc./min.
- Nominal organic flow rate, G = 800 cc./min.

The results of the tests are summarized in Tables 1 and 2.

Table 1. Characteristics of column operation observed for each condition studied

<table>
<thead>
<tr>
<th>Column condition</th>
<th>Column</th>
<th>Acid wash</th>
<th>Interface position</th>
<th>Principally dispersed phase</th>
<th>Degree of dispersion</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>E</td>
<td>HNO$_3$</td>
<td>Top</td>
<td>Organic</td>
<td>Good</td>
</tr>
<tr>
<td>2</td>
<td>G</td>
<td>HCl$_2$</td>
<td>Top</td>
<td>Organic</td>
<td>Poor</td>
</tr>
<tr>
<td>3</td>
<td>E</td>
<td>HNO$_3$</td>
<td>Bottom</td>
<td>Aqueous</td>
<td>Poor</td>
</tr>
<tr>
<td>4</td>
<td>G</td>
<td>HCl$_2$</td>
<td>Bottom</td>
<td>Aqueous</td>
<td>Good</td>
</tr>
</tbody>
</table>

The values indicated for the flow rates and hold-up at each condition represent the averages of the results of ten runs at each condition. For all runs, and regardless of the primary interface location, holdup was defined as the volume of organic phase contained...
in the contacting section divided by the total volume of the contacting section. The effect of plate wash was confounded with the possible effect produced by physical differences in the two columns, making it impossible to distinguish effects caused by plate washes from effects caused by physical differences in the columns. Preliminary experimentation with the columns had indicated that the columns behaved identically when operated under identical conditions. Therefore, the columns were concluded to be physically identical and to operate alike, so that any effect which might be attributed to either the plate washes or physical differences in the two columns was taken to be produced solely by the plate washes.

For column conditions 1 and 2, where the principal interface was controlled at the top of the column, a fairly uniform dispersion of hexone in water was obtained in column B, where the aqueous phase preferentially wet the plates. During all points of the pulse cycle, the hexone drops appeared to fill all of each section of the column.
between a pair of plates. For the same conditions of operation in column G, where the organic phase preferentially wet the plates, practically no dispersion of the organic phase was obtained. Instead the hexone drooled upwards from plate to plate during the pulse upsurge, coalescing rapidly to form a layer of hexone under each plate. The hold-up of column G was noticeably lower than the hold-up of column E.

For column conditions 3 and 4, where the principal interface was located at the bottom of the columns, essentially the reverse occurred with the exception that the hold-ups observed for both columns were nearly the same. A good dispersion of the aqueous phase was obtained in column G, where the organic phase preferentially wet the plates, while a relatively poor dispersion of the aqueous phase was observed under the same conditions of operation in column E, where the aqueous phase preferentially wet the plates.

The results of the plate wetting study obviously did not provide a clearly defined basis for selecting a standard column condition to be fixed for all hold-up and flooding studies, although several conclusions were reached. In liquid-liquid extraction operations with a pulse column, the choice of which phase should be the principally dispersed phase for most efficient performance has been primarily a matter of trial and error. For the hold-up and flooding studies of this investigation, extraction efficiency was of no concern so that the choice of dispersed phase was largely arbitrary.
In selecting which phase was to be dispersed, however, it was desirable to establish conditions such that the principally dispersed phase did not wet the plates, in order that a more uniform and complete dispersion be obtained. Thus, if the interface were to be located at the top of the column, in which case the organic phase would be dispersed, the nitric acid wash was desirable. Alternately, if the interface were located at the bottom of the column, in which case the aqueous phase would be the principally dispersed phase, the hydrochloric acid wash was desirable. Factors considered in making the selection of a plate wash were the effect of the wash on experimental precision, corrosion of equipment by the wash, and regression of the plate wetting effect produced by the wash with time.

During the course of the plate wetting experiments, approach to steady state operation, ease of operation, and the ease and reliability of the hold-up measurement for each of the four conditions studied appeared to be approximately the same. Hold-up measurements of column G for column condition 2 were perhaps questionable because the hexone wet the plates and tended to drool upwards continuously throughout the hold-up measurement. However, a detailed statistical analysis indicated no significant difference existed among the four conditions with regard to reliability of results and ease of operation. Corrosion of the equipment by the hydrochloric acid was visibly greater than by the nitric acid. Repeated washings with hydrochloric acid probably would have significantly altered the size of the plate perforation, whereas nitric acid would have had essentially no effect on the stainless steel
plates.

The forty runs of the plate wetting study were made over a period of a month so that the influence of time on the effectiveness of the plate washes was tested. Assuming that the hold-up would vary linearly with the time which had elapsed since the plates were given the respective acid washes, an analysis was made of the variation of hold-up with time for each of the four conditions under study. A significant variation was found for column C (hydrochloric acid wash) operated with the interface at the top of the column. No significant variation of hold-up over the period of a month was found for the three other conditions studied.

Results of other experiments which will be presented later indicated, however, that the plate wetting characteristics produced by a nitric acid wash did change with time, provided a sufficient amount of time had elapsed since the plates were washed. Apparently the plate wetting characteristics produced by the nitric acid wash deteriorated only slightly during the first month after the wash, but later the plate wetting effects changed with time at a greater rate.

The use of a nitric acid wash appeared to be more desirable than the hydrochloric acid wash for the hold-up and flooding studies, although all variables which were considered sufficient to establish a fixed column performance were essentially the same for both columns, considerable differences in their operation existed because of the effects
produced by the plate washes. The characteristic measurement which would be required to predict the wash effect was not known so that both columns were treated with nitric acid in order that hold-up and flooding data from the two columns could be compared.

Investigation of the relation of pulse column performance to the plate wetting characteristics will be necessary before pulse column performance can be completely predicted. Studies of plate wetting effects are recommended.

However, flooding caused by inadequate pulsation is dependent only on the pumping capacity of the column pulsation and would not be expected to be a function of the plate wetting characteristics or the physical properties of the liquid system. Studies of flooding caused by inadequate pulsation made in the initial stage of this investigation substantiated this conclusion, that such flooding is independent of the physical properties of the liquids and the perforated plates.

Because flooding caused by excessive pulsation is a hydrodynamic phenomenon, however, plate wetting would be expected to affect the flooding characteristics. Results of the excessive pulsation studies which will be presented later showed that flooding due to excessive pulsation is indeed extremely sensitive to the plate wetting characteristics.
Flooding Caused by Inadequate Pulsation

Mechanism of flooding

Earlier workers (1, 2) with pulse columns have indicated that two distinct flooding mechanisms exist which depend upon the pulse frequency and pulse volume. Between pulsations at low pulse frequencies the two phases separate into two layers in each section between adjacent plates. On the upsurge of the pulse cycle the lighter phase beneath each plate is dispersed into the heavier phase above each plate. The droplets rise through this layer and coalesce beneath the plate above. On the downsurge the heavy phase is dispersed downwards through the layer of light phase in a similar fashion. Neither phase may be considered truly continuous in the column as both move counter-currently from plate to plate by alternate dispersion and recoalescence. Such operation for a pulse column has been designated as mixer-settler operation.

For mixer-settler operation of a pulse column at a fixed set of flow rates, a definite minimum rate of pulsation exists. The minimum rate of pulsation is just sufficient to push the amount of light phase upwards on each upsurge and to pull the amount of heavy liquid downwards on each downsurge necessary to maintain the required flow rates of each phase. When the pulsed volume velocity, which is an index of the rate of pulsation, is lowered beyond the minimum rate, flooding caused by inadequate pulsation occurs. Such flooding is not determined by
hydrodynamic considerations as in packed and spray columns, but it is caused by the inability of the pulsation to pump the desired amount of fluid through the column. Consequently the physical properties of the liquid phases, the plate wetting characteristics, and column design would be expected to be independent of flooding due to insufficient pulsation insofar as the idealized condition of mixer-settler operation is possible.

However, the range of operating conditions for which mixer-settler operation is possible of course depends upon the factors which control droplet velocities and droplet coalescence. Drop velocity is related to drop size which is a function of the fluid velocity through the plate perforations, the perforation diameter, physical properties of the system and the plate wetting characteristics. For low pulsed volume velocities where mixer-settler operation is obtained, drops of either phase are produced which travel to the next plate and coalesce before being redispersed. As the pulsed volume velocity is increased, a point is reached where coalescence becomes incomplete between successive pulses. At still higher pulsed volume velocities, the droplets become permanently dispersed, no longer coalescing in the pause between pulses. Flooding is then no longer a function only of the simple pumping action of the pulsation, but becomes a hydrodynamic phenomenon similar to flooding in packed and spray columns. Such flooding caused by conditions other than inadequate pulsation will be discussed later.
Derivation of flooding equation

A number of earlier workers have concluded that the total throughput of a pulse column is equal to the pulsed volume velocity at conditions of incipient flooding due to inadequate pulsation. An equation derived from an analysis of column operation at inadequate pulsation indicates, however, that such a conclusion is an oversimplification. The analysis used in the following derivation will consider operation at the bottom section of the column immediately below the lowest plate as indicated by the level P in Figure 6. It is assumed in the derivation of this equation 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 only during the pulse upsurge. It is further assumed that the pulse wave form is sinusoidal.

Let

\[ Q = \text{net total quantity of fluid in cc. which has flowed upwards past level P (see Figure 6) at any time } t \text{ in min. from the time } t = 0. \]

\[ G = \text{light phase (organic) flow rate, cc./min.} \]

\[ L = \text{heavy phase (aqueous) flow rate, cc./min.} \]

\[ f = \text{pulse frequency, cycles/min.} \]

\[ a = \text{pulse amplitude, which represents the magnitude of the pulse movement of the fluid content of the column from one extreme position to the other, cm./cycle.} \]

\[ A = \text{cross-sectional area of the contacting section of the} \]
FIG. 6 SCHEMATIC ILLUSTRATION OF BOTTOM OF PULSE COLUMN
Thus,

\[ v = nA \]  \hspace{1cm} (1)

where

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

and

\[ V_p = \frac{\pi}{2} f \]  \hspace{1cm} (2)

where

\( V_p = \) pulsed volume velocity, cc./min.

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

\[ Q = \frac{\pi}{2} \sin(2\pi ft) + Gt. \]  \hspace{1cm} (3)

The quantity, \( Gt \), represents the amount of fluid which has passed P as a result of the continuous flow of organic phase into the bottom of the column. Likewise, the quantity, \( \frac{\pi}{2} \sin(2\pi ft) \), represents the amount contributed by the pulse. Equation (3) is illustrated in Figure 7.

Differentiation of equation (3) with respect to time gives the velocity past P at any time,

\[ \frac{dQ}{dt} = \pi vf \cos(2\pi ft) + G. \]  \hspace{1cm} (4)
\[ Q = Q_1 + Q_2 \]
\[ Q_1 = \frac{V}{2} \sin(2 \pi ft) \]
\[ Q = Gt \]

**FIG. 7.** \( Q \) VERSUS \( t \) AT LEVEL \( P \) FOR A SINE-WAVE PULSE FORM.

\[ \frac{dQ}{dt} = \pi vf \cos(2 \pi ft) \]
\[ \frac{dQ_2}{dt} = G \]

**FIG. 8.** \( \frac{dQ}{dt} \) VERSUS \( t \) AT LEVEL \( P \) FOR A SINE-WAVE PULSE FORM.
Equation (4) is illustrated in Figure 5.

Since the aqueous phase will be drawn down through the column only when the fluid velocity is negative at P, it is desirable to determine that portion of each pulse cycle during which the fluid velocity past P is negative. Solving equation (4) for the time when the fluid velocity is zero gives

$$\cos(2\pi ft_1) = -\frac{2}{\pi vt}$$

or

$$t_1 = \frac{1}{4f} + \phi/2\pi f$$

where

- $t_1$ is the first root of equation (4) when the velocity is zero as shown in Figure 5.
- $\phi = \arcsin \frac{2}{\pi vt}$

Making use of the symmetry gives

$$t_2 = \frac{3}{4f} - \phi/2\pi f$$

where

- $t_2$ is the second root of equation (4) when the velocity is zero as shown in Figure 5.

The fluid volume pulled down the column on each downsurge, $q_d$, is obtained by integrating equation (4) between the limits of $t_1$ and $t_2$. Because of the check valve located in the aqueous effluent line, no back-flow is assumed to exist in the flexible leg so that the downward movement of fluid occurs only in the column. Thus,
The rate at which fluid flows down the column is then obtained from the product of the pulse frequency and the volume withdrawn on each downsurge as given by equation (10). This rate of downward flow must be at least equal to the aqueous flow rate, \( L \). 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 an amount of fluid back from whence it came. When recycle is present, therefore, some fluid would be expected to pass through a single plate more than once, and some cocurrent flow would be superimposed upon the countercurrent flow of both phases.

For the general case, then,

\[
\frac{V_r + L}{\frac{q_d}{2}} = v \cos \phi + (2\phi - \eta)g/2\pi f
\]  

(11)

where

\[
V_r = \text{recycle rate, cc./min.}
\]

Equation (11) may be rearranged to give

\[
V_r = V_p \cos \phi + \eta g/\pi - 0/2 - L
\]  

(12)
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. If conditions exist
such that \( V_r < 0 \), the pulsation is inadequate to pump the required
amount of aqueous phase down the column and flooding occurs.

In a similar fashion, the volume of fluid pushed upwards on each
upsurge, \( q_u \), may be shown to be

\[
q_u = v \cos \theta + (2\phi + \eta)G/2\pi. \tag{13}
\]

On the upsurge, fluid flows not only upwards 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, so that

\[
\frac{L + G + V_r}{P} = v \cos \theta + (2\phi + \eta)G/2\pi. \tag{14}
\]

Rearranging equation (14) gives

\[
V_r = V_p \cos \phi + G\phi/\pi - G/2 - L \tag{15}
\]

which is the same as equation (12). Therefore, recycle rate on the
upsurge is seen to be equal to the recycle rate on the downsurge.

As before, when

\[
V_r > 0, \text{ recycle is present;}
\]
\[
V_r = 0, \text{ incipient flooding exists;}
\]
\( v_r < 0 \), flooding due to inadequate pulsation occurs.

Equation (15) represents an exact expression without any approximations of the flow behavior of a pulse column, but it is cumbersome to use in the form given. However, equation (15) can be simplified by using equation (7) and knowing that

\[
\cos x = (1 - \sin^2 x)^{\frac{1}{2}}.
\]  

(16)

Therefore,

\[
v_r = \sqrt{p} \left[ 1 - \frac{1}{2} \left( \frac{g/\nu_p}{1 - \xi^2} \right)^2 \frac{1}{2} + \frac{g}{w} \arcsin\left( \frac{g/\nu_p}{1 - \xi^2} \right) - \frac{g}{2} - L \right].
\]  

(17)

Knowing that

\[
(1 - \xi^2)^{\frac{1}{2}} = 1 - \frac{\xi^2}{2} - \frac{\xi^4}{8} - \frac{\xi^6}{16} - \ldots
\]  

(18)

and

\[
\arcsin x = x + \frac{x^3}{6} + \frac{3x^5}{120} + \ldots
\]  

(19)

equation (17) may be written as

\[
v_r = \sqrt{p} \left( 1 + \frac{g^2}{2} + \frac{g^4}{24} + \frac{g^6}{80} + \ldots \right) - \frac{g}{2} - L
\]  

(20)

where

\[
g = \frac{g}{\nu_p}.
\]  

(21)

When \( g < 1 \), and using the expression,

\[
\cosh x = 1 + \frac{x^2}{2} + \frac{x^4}{24} + \frac{x^6}{720} + \ldots
\]  

(22)
equation (20) may be approximated closely by

$$v_r = v_p \cosh \theta - G/2 - L.$$  \hspace{1cm} (23)

From equation (7), it is seen that $\theta \leq 1$ so that equation (23) represents an excellent approximation for the general case. At incipient flooding when $v_r = 0$,

$$v_p \cosh \theta = G/2 + L$$  \hspace{1cm} (24)

which can also be written as

$$\frac{v_p}{G} + \frac{\pi}{2} = \frac{(\cosh \theta)}{\theta}.$$  \hspace{1cm} (25)

Equation (25) is a convenient expression which predicts conditions of flooding due to inadequate pulsation. The left side of equation (25) is a function only of the ratio of the column feed rates, which is a quantity usually established by the stage requirements of a given extraction problem. Once the flow rate ratio is known, the value of $\theta$ is determined by equation (25). Knowing $\theta$ permits the establishment of the minimum pulsed volume velocity which must be used for a desired column flow capacity. A plot of equation (25) on log-log paper with the left side of the equation taken as the ordinate and the right side as the abscissa is a straight line with a slope of unity passing through the point $(1, 1)$. Such a log-log plot permits the presentation of a wide range of operating conditions on a graph having a convenient scale.
Equation (23) may be rearranged to give

$$V_p = \frac{G}{2\pi} \left( \frac{\cosh \theta}{\theta} \right) + \frac{\pi/2}{\theta} - \left( \frac{\pi L}{G} + \frac{\pi}{2} \right). \tag{26}$$

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. Equality of the two quantities as represented by equation (25) represents incipient flooding conditions.

Equation (25) indicates that the total throughput is inadequate for describing the conditions of flooding caused by inadequate pulsation. Instead, each individual flow rate must be considered. In order that the theoretically derived equation might be compared with the flooding relation presented by earlier workers in Figure 1, however, equation (25) can also be written as

$$G + L = V_p \left( \frac{\cosh \theta}{\theta} \right) + \frac{\theta}{2}. \tag{27}$$

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

$$G + L = V_p. \tag{28}$$

Equations (27) and (28) may be compared by plotting $G + L$ against $V_p$ at constant $G$ as shown in Figure 9.

Figure 9 predicts that throughputs greater than the pulsed volume velocity should be possible. The theoretically derived equation (27)
G = CONSTANT

\[ G + L = V_P \cosh \left( \frac{G}{\pi V_P} \right) + \frac{G}{2} \]

Fig. 9 Comparison of derived flooding equation proposed by earlier investigators.
is represented in Figure 9 by an almost straight line which nearly parallels the straight line given by equation (26). Since it was shown earlier that \( \theta \leq 1 \), a statement is possible regarding the range of variation of the hyperbolic cosine term in equation (27). Thus, when \( \theta = 0 \), \( \cosh \theta = 1.00 \), and for \( \theta = 1 \), in which case \( L \) can be shown to be zero, \( \cosh \theta = 1.543 \). Because \( \theta \leq 1 \), the line representing equation (27) terminates at an abscissa of \( V_p = G/\pi \), and the ordinate for this point is \( 1.543(G/\pi) + G/2 \), or 0.991 \( G \). Actually the true flooding line may be shown through the use of equation (15), which contains no approximations, to terminate at a point having an ordinate of \( G \). However, the approximate form of the flooding equation is a more convenient expression of conditions at flooding caused by inadequate pulsation and should be sufficiently accurate for the prediction of such flooding. When the total throughput is \( G \), which represents the minimum ordinate for both flooding lines in Figure 9, equation (28) predicts a pulsed volume velocity equal to \( G \) is necessary. However, the flooding line given by the theoretically derived equation (27) states that a pulsed volume velocity of only \( G/\pi \) is required.

Experimental verification of flooding equation

A number of flooding runs were made to experimentally verify equation (25). Normally, the procedure followed in making a flooding run was to decrease the pulse volume in very small increments at a given organic flow rate, pulse frequency, and aqueous flow rate until flooding was observed. Occasionally the pulse volume was fixed and
the organic flow rate was increased until flooding existed, but either method was found to give the same result so the more convenient method of varying the pulse volume was usually used. The transition from normal operation to flooding was usually well defined. An uncontrollable rising of the interface at the top of the column was the first noticeable sign of the flooding condition. A build-up of an organic phase layer in the end section beneath the bottom plate was next observed. At this time the organic hold-up in each section between a pair of adjacent plates started to increase, and eventually the entire column was nearly filled with the organic phase.

A series of flooding runs was 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 objective of studying the effect of frequency was to establish that only the product of the pulse volume and frequency as proposed by the equation would be necessary for the prediction of flooding. The results of the runs made at different frequencies with the aqueous flow rate held constant are given in Table 3. The data are plotted in Figure 10 with the quantity, \(\bar{V}/\theta + \pi/2\), representing the ordinate and the quantity, \((\cosh \theta)/\theta\), representing the abscissa as suggested by equation (25). The close fit of the data taken at different frequencies to the same straight line substantiates 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, independently of one another. The close fit
Table 3. Results of inadequate pulsation studies at different pulse frequencies with a constant aqueous flow rate

Nominal aqueous flow rate = 100 cc./min.

<table>
<thead>
<tr>
<th>Run</th>
<th>f</th>
<th>V</th>
<th>Vp</th>
<th>G</th>
<th>L</th>
<th>$\theta$</th>
<th>$\cosh \theta$</th>
<th>$\cosh \theta / \theta$</th>
<th>$nL / \theta + 2$</th>
<th>$V_r$</th>
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<tbody>
<tr>
<td>WG-15</td>
<td>72.6</td>
<td>2.25</td>
<td>163.4</td>
<td>114.0</td>
<td>98.5</td>
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<td>103.6</td>
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<td>1.0011</td>
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<td>20.71</td>
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<tr>
<td>HG-3-R</td>
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<td>HG-6-R</td>
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<td>5.64</td>
<td>6.22</td>
<td>-12.9</td>
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<td>Run</td>
<td>$f$</td>
<td>$v$</td>
<td>$V_p$</td>
<td>$G$</td>
<td>$L$</td>
<td>$\Theta$</td>
<td>$\cosh\Theta$</td>
<td>$\frac{\eta L}{G + \frac{\eta}{2}}$</td>
<td>$V_r$</td>
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<tr>
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<td>HE-12-R</td>
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<td>101.7</td>
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<td>5.77</td>
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<td>0.234</td>
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<td>4.22</td>
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<td>3.03</td>
<td>2.75</td>
<td>23.1</td>
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</table>
FIG. 10 COMPARISON OF THEORETICAL FLOODING EQUATION WITH EXPERIMENTAL RESULTS OBTAINED AT FOUR FREQUENCIES AND AN AQUEOUS FLOW RATE OF 100 cc./MIN.
of the data which represents a wide range of pulse conditions and a
wide range of organic flow rates strongly supports the theoretical
equation.

Although the experimental runs made at incipient flooding would
have ideally represented conditions in which recycle was absent, the
data shown in Figure 10 exhibit a tendency for flooding to occur with
a slight amount of recycle present. Specifically, the column appears
to flood at higher pulsed volume velocity than would be expected from
the equation. This deviation of the experimental from expected results
has, however, several possible explanations.

Perhaps the most apparent reason for the observed discrepancy is
the manner in which incipient flooding conditions are established. Thus,
the pulse volume is decreased by small increments until flooding is
observed at which time the pulse volume is increased slightly until the
column returns to normal operation. This condition is assumed to re­
present incipient flooding, but a slight bias for such points to fall
in the recycle region is understandable.

Another possible explanation why a slight recycle would be present
at flooding is 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. Such an explanation might appear more
reasonable when the amount of forced recycle per pulse is calculated.
For the purpose of illustration, Run WG-35-R may be considered in which the recycle rate, $V_r$, was 15.9 cc./min. at a pulse frequency of 59.9 cycles/min. Thus, the amount recycled per pulse was only 0.32 cc. Since approximately 150 droplets of $\frac{1}{16}$ in. diam. would be contained in such a volume, rationalization of the experimental deviation from the theoretical equation entirely on the basis of forced recycle seems unlikely because each plate contained approximately 250 holes and forced recycle was present at probably less than one-fourth of the holes. However, the concept of forced recycle probably does account for the presence of some recycle at flooding.

Consideration of the amount recycled per pulse introduces still another reason for the bias in which flooding points fall in the recycle region. A third reason for this bias is the failure of the check valve to close immediately as the pulse begins its downsurge, thereby allowing some fluid to be pulled back into the bottom of the column from the flexible leg. Rewriting equation (11) for this case gives

\[ \frac{V_r + L + L'}{x} = v \cos \phi + (2\phi - \pi)G/2\pi \]  

(29)

which can be simplified to the form

\[ V_r + L' = V_p \cosh \theta - G/2 - L \]  

(30)

where
$L' = \text{rate of back flow in the flexible leg, cc./min.}$

Actually $V_r$ and $L'$ both represent the same manner of fluid flowing and differ only in location. Referring to Run WG-35-R discussed above, and considering zero recycle to exist within the column proper, the rate of back flow in the flexible leg as calculated from equation (30) is 18.9 cc./min., or 0.32 cc. per pulse. Thus, loss of 5.6 per cent of the pulse ($v = 5.70$ cc./cycle in Run WG-35-R) is seen to account for the experimental deviation from the equation.

No attempt was made to correlate the experimental discrepancy at incipient flooding with any of the causes given. Deviations accounted for by forced recycle or check valve leakage would be expected to increase with the pulse frequency and perhaps with the pulse volume. 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., experimental runs were made at nominal aqueous flow rates of 50, 200 and 300 cc./min. Variation of the aqueous flow rates was studied in order to test the validity of the flooding relation for different aqueous flow rates. Although the frequency parameter was not held constant in this series of experiments, the pulsed volume velocity has been shown to be the characteristic quantity necessary to establish the conditions of flooding caused by inadequate pulsation.
The experimental results of the study of different aqueous flow rates are tabulated in Table 4. The data are compared in Figure 11 with the theoretical relation for flooding caused by insufficient pulsation. A fairly good fit of the data to the required straight line exists. Similar to Figure 10, most of the points fall slightly below the theoretical line in Figure 11, but may be explained in the same fashion. However, several flooding points for runs at high aqueous flow rates lie above the line in the region where flooding should occur, indicating that operation with negative recycle is possible without flooding.

Operation with conditions which correspond to negative recycle may be explained because in these runs the check valve was observed not to close on the pulse downsurge. At high aqueous flow rates, a low flexible leg position was required to maintain the proper interface position in the column. Apparently the flexible leg was positioned low enough for the aqueous phase to flow down through the column by gravity instead of being pumped down through by the pulsation. The small flow capacity of the aqueous effluent line probably made necessary the low flexible leg position, because the required amount of aqueous effluent at high aqueous flow rates was not able to flow through the flexible leg entirely on the upsurge. To prevent flooding, therefore, it became necessary for the aqueous effluent to leave the column continuously. The continuous flow of aqueous effluent was a result of the low flexible leg position, although what caused the aqueous phase to leave rather than cause flooding
Table 1. Results of inadequate pulsation studies at different aqueous flow rates

<table>
<thead>
<tr>
<th>Run</th>
<th>f</th>
<th>v</th>
<th>Vp</th>
<th>G</th>
<th>L</th>
<th>q</th>
<th>cosh q</th>
<th>cosh q</th>
<th>(\frac{vL}{G^2 + \pi^2})</th>
<th>(V_r)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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<td></td>
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</table>

Nominal aqueous flow rate = 50 cc./min:

<table>
<thead>
<tr>
<th>Run</th>
<th>f</th>
<th>v</th>
<th>Vp</th>
<th>G</th>
<th>L</th>
<th>q</th>
<th>cosh q</th>
<th>cosh q</th>
<th>(\frac{vL}{G^2 + \pi^2})</th>
<th>(V_r)</th>
</tr>
</thead>
<tbody>
<tr>
<td>EE-18-R</td>
<td>72.6</td>
<td>1.3</td>
<td>94.4</td>
<td>81.7</td>
<td>51.3</td>
<td>0.275</td>
<td>1.0381</td>
<td>3.77</td>
<td>3.54</td>
<td>5.9</td>
</tr>
<tr>
<td>EE-19-R</td>
<td>72.6</td>
<td>2.7</td>
<td>196.0</td>
<td>256.0</td>
<td>49.3</td>
<td>0.416</td>
<td>1.0878</td>
<td>2.61</td>
<td>2.17</td>
<td>35.9</td>
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<tr>
<td>EE-20-R</td>
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<td>36.3</td>
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<td>1.0450</td>
<td>3.15</td>
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<td>EE-21-R</td>
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<td>1.83</td>
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<tr>
<td>EE-22-R</td>
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<td>1.0031</td>
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<td>EE-30-R</td>
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<td>3.9</td>
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<td>1.1251</td>
<td>2.27</td>
<td>1.93</td>
<td>47.5</td>
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<tr>
<td>WS-37-R</td>
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<td>2.05</td>
<td>122.8</td>
<td>120.5</td>
<td>49.9</td>
<td>0.312</td>
<td>1.0090</td>
<td>3.36</td>
<td>2.87</td>
<td>18.7</td>
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<td>150.3</td>
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<td>51.5</td>
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<td>1.0961</td>
<td>2.52</td>
<td>2.36</td>
<td>10.4</td>
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<tr>
<td>WS-39-R</td>
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<td>189.3</td>
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<td>51.0</td>
<td>0.508</td>
<td>1.1318</td>
<td>2.23</td>
<td>2.10</td>
<td>12.2</td>
</tr>
<tr>
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<td>4.04</td>
<td>242.0</td>
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<td>2.00</td>
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<tr>
<td>WS-41-R</td>
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<td>4.50</td>
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<td>49.9</td>
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<td>1.1893</td>
<td>1.96</td>
<td>1.89</td>
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Nominal aqueous flow rate = 200 cc./min:

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<th>Vp</th>
<th>G</th>
<th>L</th>
<th>q</th>
<th>cosh q</th>
<th>cosh q</th>
<th>(\frac{vL}{G^2 + \pi^2})</th>
<th>(V_r)</th>
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<td>EE-43-R</td>
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<td>2.67</td>
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<td>193.7</td>
<td>0.142</td>
<td>1.0101</td>
<td>7.11</td>
<td>8.56</td>
<td>41.3</td>
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<td>3.43</td>
<td>205.4</td>
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<td>200.1</td>
<td>0.157</td>
<td>1.0123</td>
<td>6.45</td>
<td>7.76</td>
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<td>5.79</td>
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<td>302.9</td>
<td>205.2</td>
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<td>1.0389</td>
<td>3.74</td>
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\(^a\)Check valve not seated so that continuous flow of aqueous effluent existed.
<table>
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<th>v</th>
<th>$V_p$</th>
<th>G</th>
<th>L</th>
<th>$\theta$</th>
<th>$\cosh \theta$</th>
<th>$\frac{mL}{G^2 + \pi}$</th>
<th>$\gamma_r$</th>
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<td>1.0030</td>
<td>15.02</td>
<td>15.85$^a$</td>
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<td>377.8</td>
<td>104.6</td>
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<td>0.088</td>
<td>1.0039</td>
<td>11.41</td>
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<td>291.0</td>
<td>0.051</td>
<td>1.0014</td>
<td>19.64</td>
<td>28.30$^a$</td>
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</tbody>
</table>

Nominal aqueous flow rate = 300 cc./min.

Note: $^a$Check valve not seated so that continuous flow of aqueous effluent existed.
FIG. II CONDITIONS AT INCIPient FLOODING DUE TO INADEQUATE PULSATION OBSERVED FOR THREE DIFFERENT AQUEOUS FLOW RATES.
is not clearly understood.

Since a portion of the aqueous flow down through the column was accomplished without the benefit of pulsation, greater throughputs were achieved than expected from the equation, which requires that flow be caused only by the pumping action of the pulsation. Because the aqueous effluent lines were too small, verification of the flooding equation for conditions of inadequate pulsation is indefinite at high aqueous flow rates. A wide range of conditions are predicted closely by the equation, however, so that agreement at high flow rates seems probable if the capacity of the effluent lines were sufficient to allow the check valve to close on the pulse downsurge.

In addition to the inability of the equipment to conform exactly to the operation assumed in the derivation of the equation, the equation is, of course, further limited by the alternate type of flooding caused by excessive pulsation. The transition from flooding caused by inadequate pulsation to flooding caused by excessive pulsation needs to be understood before the limitations of the theoretical equation can be defined. This transition region is illustrated in Figure 1 as that portion of the flooding curve where the curve deviates from the pulsed volume velocity line and extending to the maximum point of the flooding curve. More specifically, the transition region is that portion of the total flooding curve in which the column capacity begins to be less than that predicted as possible by the equation for flooding caused by inadequate pulsation. Studies made in the transition region and at excessive pulsation conditions will be presented later.
Before leaving the discussion of the equation which predicts flooding caused by inadequate pulsation, however, some further observations regarding the equation are of interest. In addition to its application for predicting flooding conditions, the equation can also be used to determine a quantitative measure of the amount of recycle for various conditions of operation. Recycle represents a deterioration of the true countercurrent operation which is required for the most efficient extraction. The recycle rate, \( V_r \), might prove useful as a correlating device for pulse column extraction studies. Although operation at conditions where recycle is present might seem undesirable, some evidence exists which indicates that high pulsed volume velocities increase turbulence and dispersion sufficiently so that higher extraction efficiencies are obtained as the recycle rate is increased up to a certain limit. It is suggested, however, that the most efficient operation might be realized in the transition region where the high throughputs and high pulsed volume velocities are possible with little or no recycle.

**Hold-up measurements**

A knowledge of how hold-up varies is important to the understanding of pulse column behavior because of the relation of interfacial area to hold-up. For that reason, a part of this investigation was devoted to the study of a method for the determination of hold-up in a pulse column. A procedure was described earlier whereby the liquid contents of the
column operating at steady state conditions were immobilized and the respective volumes of each liquid phase were measured with a ruler. Such a procedure was tested and considered acceptable for the measurement of the pulse column hold-up.

Although no systematic study of hold-up variation was made, the hold-up was measured in the experimental runs made with the pulse column while investigating the conditions of flooding caused by inadequate pulsation. The results of these hold-up measurements are given in Table 5. Attempts at correlating these hold-up data with the different operating variables were unsuccessful. However, it may be recalled that usually a sequence of flooding runs was made at a fixed pulse frequency and aqueous flow rate, while the pulse volume was adjusted to cause flooding for several values of the organic flow rate. Since all hold-up measurements were made in flooding experiments in which essentially two variables were varied instead of only one, difficulty in obtaining a correlation of the hold-up data was not unexpected.

Griffith, Jasny and Tupper (2) state that for conditions such as exist at incipient flooding caused by inadequate pulsation, the amount of organic phase pulsed upward through a plate per stroke must be sufficient to maintain the required net flow through the column. Making the assumption that all of the organic phase in a section is pulsed upward into the next section during the upstroke and is not pulsed downward again, they conclude that the volume of organic phase appearing in each section must equal the volumetric flow rate of the organic
Table 5. Results of hold-up measurements at the condition of incipient flooding due to inadequate pulsation

<table>
<thead>
<tr>
<th>Run</th>
<th>$f$</th>
<th>$v$</th>
<th>$G$</th>
<th>$L$</th>
<th>$h$</th>
<th>$G/f$</th>
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</thead>
<tbody>
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<td>HG-1-R</td>
<td>72.6</td>
<td>1.6</td>
<td>17.0</td>
<td>103.6</td>
<td>0.0152</td>
<td>0.234</td>
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<td>HG-2-R</td>
<td>1.7</td>
<td>41.3</td>
<td>99.9</td>
<td>0.0304</td>
<td>0.569</td>
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<td>HG-3-R</td>
<td>3.4</td>
<td>257.7</td>
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<td>0.226</td>
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<tr>
<td>HG-4-R</td>
<td>2.6</td>
<td>141.7</td>
<td>103.4</td>
<td>0.161</td>
<td>1.952</td>
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<tr>
<td>HG-5-R</td>
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<td>137.7</td>
<td>98.8</td>
<td>0.134</td>
<td>1.897</td>
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<tr>
<td>HG-6-R</td>
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<td>69.4</td>
<td>100.9</td>
<td>0.568</td>
<td>9.018</td>
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<td>HG-7-R</td>
<td>2.0</td>
<td>103.3</td>
<td>98.9</td>
<td>0.191</td>
<td>1.423</td>
<td></td>
</tr>
<tr>
<td>HG-8-R</td>
<td>1.9</td>
<td>78.8</td>
<td>99.9</td>
<td>0.0661</td>
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<tr>
<td>HG-9-R</td>
<td>2.9</td>
<td>204.0</td>
<td>98.9</td>
<td>0.187</td>
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<tr>
<td>HG-10-R</td>
<td>4.9</td>
<td>904.3</td>
<td>102.4</td>
<td>0.474</td>
<td>6.946</td>
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<td>HG-11-R</td>
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<td>102.1</td>
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<td>81.7</td>
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<td>0.427</td>
<td>3.526</td>
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<td>HG-15-R</td>
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<td>0.282</td>
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<td>HG-16-R</td>
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<td>50.8</td>
<td>0.562</td>
<td>6.665</td>
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<td>HG-17-R</td>
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<td>0.723</td>
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<td>291.0</td>
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<td>0.471</td>
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<td>HG-20-R</td>
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<td>86.8</td>
<td>193.7</td>
<td>0.163</td>
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<tr>
<td>HG-21-R</td>
<td>59.9</td>
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<td>234.2</td>
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<td>0.313</td>
<td>3.210</td>
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<td>HG-22-R</td>
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<td>333.3</td>
<td>103.0</td>
<td>0.528</td>
<td>5.664</td>
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<tr>
<td>HG-23-R</td>
<td>2.63</td>
<td>122.5</td>
<td>99.9</td>
<td>0.126</td>
<td>2.045</td>
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<tr>
<td>HG-24-R</td>
<td>5.11</td>
<td>443.3</td>
<td>101.3</td>
<td>0.648</td>
<td>7.401</td>
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<tr>
<td>HG-25-R</td>
<td>5.70</td>
<td>524.4</td>
<td>101.6</td>
<td>0.600</td>
<td>6.755</td>
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<tr>
<td>HG-26-R</td>
<td>5.77</td>
<td>527.8</td>
<td>99.7</td>
<td>0.650</td>
<td>9.563</td>
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<tr>
<td>HG-27-R</td>
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<td>90.0</td>
<td>98.7</td>
<td>0.102</td>
<td>1.502</td>
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<tr>
<td>HG-28-R</td>
<td>2.05</td>
<td>120.5</td>
<td>49.9</td>
<td>0.111</td>
<td>2.012</td>
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<tr>
<td>HG-29-R</td>
<td>2.51</td>
<td>205.5</td>
<td>51.5</td>
<td>0.461</td>
<td>3.131</td>
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<tr>
<td>HG-30-R</td>
<td>3.16</td>
<td>302.0</td>
<td>51.0</td>
<td>0.554</td>
<td>5.042</td>
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</tr>
<tr>
<td>HG-31-R</td>
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<td>447.5</td>
<td>50.7</td>
<td>0.696</td>
<td>7.471</td>
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<tr>
<td>HG-32-R</td>
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<td>495.8</td>
<td>49.9</td>
<td>0.685</td>
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<tr>
<td>HG-33-R</td>
<td>3.43</td>
<td>101.6</td>
<td>200.1</td>
<td>0.100</td>
<td>1.696</td>
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</tr>
<tr>
<td>HG-34-R</td>
<td>5.79</td>
<td>302.9</td>
<td>205.2</td>
<td>0.476</td>
<td>5.057</td>
<td></td>
</tr>
<tr>
<td>HG-35-R</td>
<td>35.7</td>
<td>3.3</td>
<td>66.5</td>
<td>98.4</td>
<td>0.0925</td>
<td>1.863</td>
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<tr>
<td>HG-36-R</td>
<td>9.3</td>
<td>432.9</td>
<td>99.5</td>
<td>0.586</td>
<td>12.126</td>
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<tr>
<td>HG-37-R</td>
<td>7.81</td>
<td>382.4</td>
<td>98.8</td>
<td>0.604</td>
<td>10.711</td>
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<tr>
<td>HG-38-R</td>
<td>7.80</td>
<td>370.3</td>
<td>98.9</td>
<td>0.449</td>
<td>10.372</td>
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</tr>
<tr>
<td>HG-39-R</td>
<td>3.78</td>
<td>91.8</td>
<td>103.0</td>
<td>0.132</td>
<td>2.571</td>
<td></td>
</tr>
</tbody>
</table>
The hold-up data tabulated in Table 5 are plotted in Figure 12 with the hold-up as the ordinate and the ratio, $G/f$, as the abscissa as suggested by equation (31). The points are quite widely scattered, especially at the higher hold-ups, indicating that the above analysis is an oversimplification. Most of the points fall above the line predicted by equation (31), but such behavior could be expected due
FIG. 12. COMPARISON OF HOLD-UP DATA WITH RELATION SUGGESTED BY GRIFFITH, JASNY, & TUPPER (2)
to the presence of forced recycle. The points which fall below the line are the results of runs made at very low pulse frequencies. It is possible that for low frequencies that organic phase drops rise from one plate to the next before the pulse changes direction. By allowing some of the organic phase to pass upward through more than one plate per pulse, the hold-up might be expected to be less than predicted by equation (31).

The lack of success in correlating the hold-up data may be due to the inability to measure the plate wetting effect. As has been shown, hold-up is influenced by the wetting characteristics of the plates of a pulse column. Although tests indicated that wetting characteristics of the plates given a nitric acid wash did not change with time, some variation of the plate wetting characteristics over the duration of the hold-up studies was possible since the hold-up tests extended over a much longer period of time than the plate wetting studies.

Contrary to the conclusions reached as a result of the plate wetting studies discussed earlier, any effect produced by the nitric acid wash might change with time, and the rate of change conceivably would not be constant. In the case of the nitric acid wash, each plate should be covered with an oxide film. As long as the oxide film remains intact, the surface properties of the plates probably are constant so that the plate wetting characteristics would not be expected to change. If, however, the oxide film were penetrated, the oxide film would very likely be washed away by the agitated fluid in the column and the plate
wetting characteristics could change markedly in a short time.

Another factor which would help explain the difficulty in correlating the hold-up data was the inertia of the pulse generators. The method used to determine the hold-up required that the flow of both phases to the column and the pulsation be stopped instantly and simultaneously. Unfortunately, no braking mechanism was used so that it is probable that the column contents were pulsed slightly after the pulse generator was turned off.

Flooding Caused by Excessive Pulsation

Mechanism of flooding

Flooding of a pulse column may be caused by excessive pulsation, as well as by inadequate pulsation. Incipient flooding due to inadequate pulsation has been shown to be characterized by mixer-settler type operation and by the absence of recycle. If the pulsed volume velocity is gradually increased with the flow rates held constant for a pulse column initially operated at incipient flooding due to inadequate pulsation, the recycle rate increases correspondingly and coalescence between pulses decreases until the mixer-settler type operation disappears entirely. Eventually one phase appears to be dispersed in the other phase at all times throughout the entire column in a fashion similar to spray and packed columns. As the pulsed volume velocity is increased, the velocity of the dispersed phase through the
plate perforations increases and the average drop size decreases. As the drop size decreases, the rising (or settling) drop velocity which is the result of the density difference of the two phases also decreases, and the average rising (or settling) velocity with which the drops move through the column decreases. Finally a pulsed volume velocity is reached where any higher pulsed volume velocities cause flooding. The average effective velocity of the dispersed drops in their passage through the column becomes so low that the dispersed phase does not flow through the column as fast as the dispersed phase is fed to the column.

Flooding caused by excessive pulsation is a complex hydrodynamic phenomenon and is much more complicated than flooding due to inadequate pulsation, which is simply a function of the pumping capacity of the column pulsation. Flooding caused by excessive pulsation may be the result of either the low effective velocity of the small dispersed phase drops or of the formation of a stable emulsion. Consequently, 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.

Excessive pulsation studies

Investigation of the flooding behavior caused by excessive pulsation with respect to each of the variables which affect such flooding would necessitate a very large number of experimental runs, but it was
the purpose of this study to consider only the primary operating variables. The primary operating variables were the organic flow rate, aqueous flow rate, pulse frequency and pulse volume. Unfortunately, only the bellows pulsator possessed sufficient pulsing capacity to produce flooding at conditions of excessive pulsation. Since the bellows pulsator operated at the single frequency of 59.9 cycles/min., a study of the effect of frequency was not made in the investigation of flooding caused by excessive pulsation.

The experimental procedure which was used to observe flooding caused by inadequate pulsation was modified somewhat for the 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. However, just prior to the appearance of the second interface at the bottom of the column, the high pulsed volume velocities which were required to cause flooding produced a very fine dispersion of organic drops in the bottom end section of the column. The dispersion of fine organic droplets made it difficult to observe the appearance of the second interface and created a tendency to predict flooding prematurely because the drops became entrained with the aqueous effluent due to the inadequacy of the end section to provide sufficient time for phase separation.

The fine dispersion of organic drops was believed to be produced by the violent pulsation of the second interface up and down through
the bottom plate. By allowing the formation of a secondary interface in the bottom end section and positioning this interface low enough so as to not come in contact with the bottom plate, the tendency toward emulsification was inhibited. At incipient flooding, operation of the column was possible with the interfaces at the top and bottom of the column both maintaining constant positions. Raising or lowering the flexible leg produced a similar movement of both interfaces, although the same distance was maintained between the two interfaces.

With this procedure, the layer of organic phase beneath the bottom plate and the layer of aqueous phase above the top plate each acted as a reservoir for the respective phase. The flooding capacity of the column was obtained when both interfaces were maintained at constant positions, so that the volume of each phase contained in its respective reservoir remained constant and the flow of each phase to the contacting section from the reservoir equaled the feed rate to the column. When the column was operated in the flooding region, both interfaces moved toward the ends of the column until flooding eventually occurred. Only at incipient flooding could stable operation be achieved in which both interfaces remained at constant positions. Furthermore, when the operating conditions were in the normal operating region, it was possible to maintain only the single interface at the top of the column.

Using this procedure, flooding runs were made at aqueous flow rates of 100, 200 and 300 cc./min. over a wide range of pulse volumes and
organic flow rates. Each aqueous flow rate was usually studied in a sequence of runs made at several different pulse volumes. However, duplicate runs made later differed greatly from initial runs. Since the results of each sequence of experiments appeared to be consistent, it was concluded that some physical property of the system was changing with time. Since the equipment was inert to the solvent action of the liquids being circulated and the two phases were mutually saturated so that the properties of the two liquid phases were believed constant, the plate wetting characteristics were concluded to be changing. To test the possibility that plate wetting characteristics might be varying, the plates of the pulse column were washed with nitric acid, and then a sequence of flooding runs was made at an aqueous flow rate of 100 cc./min.

The results of three different series of experiments made at an aqueous flow rate of 100 cc./min. are tabulated in Table 6 and plotted in Figure 13. Series I represents the original sequence of experiments which were made more than a month after the original nitric acid wash was given to the columns at the initiation of this investigation. Series II represents the attempted duplication of series I, but series II was made approximately a month later, during which time no more nitric acid washes were given to the column. Series III represents flooding runs made immediately after the nitric acid wash was given to the plates to test the possibility that plate wetting characteristics might be varying. Each series of experiments falls on a different
Table 6. Effect of plate wetting characteristics on flooding caused by excessive pulsation

Pulse frequency = 59.9 cycles/min.
Nominal aqueous flow rate = 100 cc./min.

<table>
<thead>
<tr>
<th>Run</th>
<th>v</th>
<th>G</th>
<th>L</th>
<th>h</th>
</tr>
</thead>
<tbody>
<tr>
<td>Series I:</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>WG-43-F</td>
<td>31.32</td>
<td>209.3</td>
<td>98.5</td>
<td>0.665</td>
</tr>
<tr>
<td>WG-44-F</td>
<td>25.87</td>
<td>318.7</td>
<td>98.3</td>
<td>0.672</td>
</tr>
<tr>
<td>WG-45-F</td>
<td>21.22</td>
<td>416.2</td>
<td>98.1</td>
<td>0.726</td>
</tr>
<tr>
<td>WG-49-F</td>
<td>16.58</td>
<td>520.0</td>
<td>101.2</td>
<td>0.787</td>
</tr>
<tr>
<td>WG-67-F</td>
<td>9.13</td>
<td>698.9</td>
<td>98.5</td>
<td>0.822</td>
</tr>
<tr>
<td>Series II:</td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>EG-56-F</td>
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<td>301.3</td>
<td>101.9</td>
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</tr>
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<td>483.3</td>
<td>103.6</td>
<td>0.798</td>
</tr>
<tr>
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<td>102.1</td>
<td>0.746</td>
</tr>
<tr>
<td>EG-91-F</td>
<td>9.78</td>
<td>336.0</td>
<td>102.3</td>
<td>0.813</td>
</tr>
<tr>
<td>Series III:</td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>EG-93-F</td>
<td>9.90</td>
<td>872.8</td>
<td>101.3</td>
<td>0.765</td>
</tr>
<tr>
<td>MG-94-F</td>
<td>37.34</td>
<td>292.3</td>
<td>103.1</td>
<td>0.591</td>
</tr>
<tr>
<td>HJ-96-F</td>
<td>28.06</td>
<td>463.8</td>
<td>100.6</td>
<td>0.663</td>
</tr>
</tbody>
</table>

straight line in Figure 13, although the three lines are nearly parallel.

The plate wetting characteristics clearly exert a large effect on flooding of a pulse column due to excessive pulsation. For example, in run EG-91-F of series II, the organic flow rate was only 336.0 cc./min. at incipient flooding for a pulse volume of 9.78 cc./cycle, while at essentially the same pulse volume in run EG-93-F of series III, an organic flow rate of 872.8 cc./min. was possible. Thus, the nitric
FIG. 13. EXCESSIVE PULSATION FLOODING LINES FOR DIFFERENT PLATE WETTING CHARACTERISTICS
acid wash is seen to have effected better than a twofold increase in the capacity of the pulse column. The plate wetting effect on pulse column capacity emphasizes the need for controlling, and for developing a method of measuring, the plate wetting characteristics.

Hold-up measurements made in conjunction with the flooding runs for each of the three series are also presented in Table 6. Hold-up is plotted against pulse volume for all three series of experiments in Figure 14. Hold-up tends to decrease as the pulse volume increases at a constant aqueous flow rate for the condition of incipient flooding. It is interesting to note that the results of all three series appear to fall on the same line, which indicates that hold-up did not vary with the time elapsed after a plate wash. Such a result lends support to the plate wetting studies discussed earlier in which no variation of hold-up with time for a nitric acid wash was noted. Thus, hold-up is apparently much less sensitive than flooding to plate wetting.

The results of flooding runs made to show the effect of different aqueous flow rates are given in Table 7 and plotted in Figure 15. These runs were obtained with the wash conditions described previously for series I of Table 6 and were made in a single sequence of experiments so that the anomaly which occurs when plate wetting varies was diminished. As might be expected, the organic flow rate at flooding decreased as the aqueous flow rate increased.

To eliminate as completely as possible the effect caused by varying
FIG. 14. HOLD-UP BEHAVIOR AT INCIPIENT FLOODING DUE TO EXCESSIVE PULSATION.
Table 7. Flooding due to excessive pulsation at different aqueous flow rates

Pulse frequency = 59.9 cycles/min.

<table>
<thead>
<tr>
<th>Run</th>
<th>v</th>
<th>G</th>
<th>L</th>
<th>h</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nominal aqueous flow rate = 100 cc./min.</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>WG-43-F</td>
<td>31.23</td>
<td>209.3</td>
<td>98.5</td>
<td>0.665</td>
</tr>
<tr>
<td>WG-44-F</td>
<td>25.87</td>
<td>318.7</td>
<td>98.3</td>
<td>0.672</td>
</tr>
<tr>
<td>WG-45-F</td>
<td>21.22</td>
<td>416.2</td>
<td>98.1</td>
<td>0.726</td>
</tr>
<tr>
<td>WG-49-F</td>
<td>16.58</td>
<td>520.0</td>
<td>101.2</td>
<td>0.787</td>
</tr>
<tr>
<td>WG-67-F</td>
<td>9.13</td>
<td>698.9</td>
<td>98.5</td>
<td>0.822</td>
</tr>
<tr>
<td>Nominal aqueous flow rate = 200 cc./min.</td>
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<td></td>
<td></td>
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</tr>
<tr>
<td>BG-72-F</td>
<td>30.26</td>
<td>125.6</td>
<td>204.9</td>
<td>0.615</td>
</tr>
<tr>
<td>BG-74-F</td>
<td>28.53</td>
<td>197.7</td>
<td>199.0</td>
<td>0.613</td>
</tr>
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<td>BG-75-F</td>
<td>20.74</td>
<td>330.7</td>
<td>203.7</td>
<td>0.595</td>
</tr>
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<td>BG-77-F</td>
<td>18.67</td>
<td>363.3</td>
<td>194.1</td>
<td>0.593</td>
</tr>
<tr>
<td>BG-78-F</td>
<td>9.76</td>
<td>415.1</td>
<td>203.7</td>
<td>0.652</td>
</tr>
<tr>
<td>BG-79-F</td>
<td>13.89</td>
<td>398.5</td>
<td>199.3</td>
<td>0.709</td>
</tr>
<tr>
<td>BG-80-F</td>
<td>7.49</td>
<td>389.8</td>
<td>202.9</td>
<td>0.546</td>
</tr>
<tr>
<td>BG-84-F</td>
<td>13.74</td>
<td>368.0</td>
<td>204.9</td>
<td>0.680</td>
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<tr>
<td>Nominal aqueous flow rate = 300 cc./min.</td>
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<tr>
<td>MG-81-F</td>
<td>26.54</td>
<td>198.3</td>
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<tr>
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<td>15.94</td>
<td>315.0</td>
<td>300.0</td>
<td>0.498</td>
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<tr>
<td>MG-85-F</td>
<td>22.19</td>
<td>260.7</td>
<td>297.7</td>
<td>0.524</td>
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</tbody>
</table>

Plate wetting characteristics, the plates of the column were given a nitric acid wash, and a run at each of the three flow rates was made on the same day. The results of these three experiments, given in Table 6, also indicate that at incipient flooding the organic flow rate decreases as the aqueous flow rate increases.
Fig. 15: Flooding due to excessive pulsation for three different aqueous flow rates.

$f = 59.9$ cycles/min.

- $\bigcirc$ $L = 100$
- $\triangle$ $L = 200$
- $\square$ $L = 300$

**Axes:**
- Organic Flow Rate, cc./min.
- Pulse Volume, cc./cycle
Table 5. Flooding due to excessive pulsation at three aqueous flow rates with a minimum variation in plate wetting effect

<table>
<thead>
<tr>
<th>Run</th>
<th>f</th>
<th>v</th>
<th>G</th>
<th>L</th>
<th>h</th>
</tr>
</thead>
<tbody>
<tr>
<td>MG-96-F</td>
<td>59.9</td>
<td>28.06</td>
<td>463.8</td>
<td>100.6</td>
<td>0.663</td>
</tr>
<tr>
<td>MG-98-F</td>
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<td>28.10</td>
<td>451.3</td>
<td>199.6</td>
<td>0.520</td>
</tr>
<tr>
<td>MG-97-F</td>
<td>59.9</td>
<td>28.02</td>
<td>386.3</td>
<td>299.5</td>
<td>0.487</td>
</tr>
</tbody>
</table>

The most important conclusion drawn from the excessive pulsation studies is the extreme sensitivity of pulse column performance to plate wetting. Until a basis for establishing the effect of plate wetting, as well as a characteristic measurement of it, is developed, accurate prediction of pulse column performance will not be possible. Because of the inability to measure or control plate wetting characteristics and because of the uncertainty associated with the effect of variation in plate wetting, the results of the excessive pulsation studies are considered only to represent qualitative trends regarding the relation of the primary operating variables to flooding. However, the procedure in which two interfaces are maintained at constant positions is considered to have been demonstrated satisfactorily and to be acceptable for studies of flooding caused by excessive pulsation. The method is rapid and simple and inhibits emulsification in the bottom end section of the column.

Total throughputs observed in this study compare favorably with
values reported previously. Griffith, Jasny and Tupper (2) report that a total flow rate of 955 gal./(hr.)/(sq. ft.) was the highest obtained by them with a 2-in. diam. pulse column. They also report operation of the same column with the plates removed as a simple spray column in which the maximum combined flow of both phases was 1820 gal./(hr.)/(sq. ft.). Cohen and Beyer (1) report capacities as high as 660 gal./(hr.)/(sq. ft.) were achieved in their studies with a 1-in. diam. pulse column. The highest combined throughput which was observed in this investigation (Run EG-93-F) is 974.1 cc./min. or 2831 gal./(hr.)/(sq. ft.).
CONCLUSIONS AND RECOMMENDATIONS

1. Flooding of a pulse column was demonstrated to be caused either by inadequate pulsation or by excessive pulsation. Other pulse column investigators had reached the conclusion that at incipient flooding due to inadequate pulsation the total column throughput equaled the pulsed volume velocity. However, the results of this study were correlated by the equation,

\[ \frac{nL}{u} + \frac{n}{2} = \frac{(cosh \theta)}{\theta}, \]

indicating the conclusion reached by earlier workers was an oversimplification. Flooding data for conditions of inadequate pulsation were well correlated by this equation, although a tendency existed for flooding to occur at slightly higher pulsed volume velocities than predicted by the equation. Such a tendency was probably due to forced recycle and backflow in the aqueous effluent line. The pulsed volume velocity was experimentally demonstrated to be sufficient for describing the conditions of pulsation for flooding due to inadequate pulsation.

Incipient flooding predicted by the flooding equation is a function only of the primary operating variables and is independent of the physical properties of the liquid phases, but the equation is limited by the alternate type of flooding caused by excessive pulsation. The transition from flooding caused by inadequate pulsation to flooding caused by excessive pulsation must be investigated further before the
limitations of the flooding equation can be fully defined.

The analysis which was used for the derivation of the flooding equation also permitted the development of an equation,

\[ V_r = V_p \cos \theta - \frac{a}{2} - L, \]

from which the amount of recycle could be calculated for various conditions of pulse column operation. Since recycle represents a deterioration of true countercurrent operation, recycle rate possibly may prove useful as a correlating device in pulse column extraction studies.

2. The wetting characteristics of the perforated plates in the pulse column exerted considerable influence on the flooding capacity and hold-up. Hold-up was apparently less sensitive than flooding to changes in plate wetting. Variation in the plate wetting characteristics as produced by a nitric acid wash were shown to result in better than a twofold increase in the capacity.

Because of the varying plate wetting characteristics, the results of the excessive pulsation experiments in this investigation are considered to represent only qualitative trends. At constant pulse frequency and pulse amplitude for conditions of incipient flooding caused by excessive pulsation, the organic flow rate decreases as the aqueous flow rate increases. Further work is required before the limiting capacity of a pulse column can be correlated in terms of the pulse characteristics, column geometry, physical properties of the system
and plate wetting.

3. A simple procedure for measuring the hold-up of pulse columns was demonstrated. Success of the method required that flow of both phases to the column and the pulsation be stopped instantly and simultaneously and that no counterflow occur in the column when not pulsed. When such requirements were met, it was possible to effectively "freeze" the contents of the column while at steady state operation and make hold-up measurements.

4. A satisfactory method was developed for the determination of flooding caused by excessive pulsation. The flooding capacity was obtained when the column was operated with an interface at both the top and the bottom of the column. By keeping the position of these interfaces constant, the flow rates to the end sections of the column could be balanced against the flow 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 in the bottom end section of the column. Choice of a system having a high interfacial tension and use of a pulse column possessing long enlarged end sections would be recommended for future pulse column flooding studies.
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NOMENCLATURE

Q = net total quantity of fluid in cc. which has flowed upwards past level P (see Figure 6) 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.

a = pulse amplitude, which represents the magnitude of the pulse movement of the fluid contents of the column from one extreme position to the other, cm./cycle.

A = cross-sectional area of the contacting section of the column.

\( V \) = pulse volume, which represents 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.

\( q_u \) = fluid volume pushed up the column on each upsurge, cc./cycle.

\( V_r \) = recycle rate, cc./min.

\( L' \) = rate of back-flow in the flexible leg, cc./min.

\( h \) = hold-up, the volume fraction of organic phase contained in the
contacting section of the column.

\( s = \text{volume of section contained between two plates of the column,} \) cc.

\( \phi = \text{arcsin} \left( \frac{G}{\pi \nu f} \right). \)

\( \theta = \frac{G}{\pi \nu p}. \)
LITERATURE CITED


