COMPARISON OF HOLD-UP MEASUREMENTS BETWEEN CLARIFIED SOLUTION AND SOLVENT IN PULP OPERATIONS

by

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Abstract

Lowering the processing cost of liquid-liquid extraction by eliminating the clarification process, requires an understanding of the hydrodynamics and the operating regions of pulsed column when treating unclarified solutions. In order to achieve such an objective, an investigation has been carried out to measure and compare the total hold-up of clarified solutions and solvent in pulp operation for two potential commercial operations, uranium and gold tailings.

Preliminary bench scale separation tests were carried out in order to evaluate phase separation characteristics and assist in the selection of the extractant phase with a minimum of emulsion formation.

The results of these tests indicated good phase separation for the uranium aqueous tailings system. However, for the gold tailings system slight crud formation was noticed for the system chosen. Based on these bench scale tests, total hold-up measurements were made for eight systems using a pulsed sieve plate extraction column. The systems included both clarified solution (five) and solvent in pulp processing (three).

Results of the clarified solutions test showed that, the hold-up increased with an increase in the amplitude of pulsing, dispersed phase flow rate and pH of the aqueous phase; while, hold-up is unaffected by the variation in the continuous phase flow rate. An increase in the pulse frequency decreased the hold-up in the mixer-settler region till it reached a minimum at the dispersion region. Then, in the emulsion region the hold-up increased with a further increase in pulse frequency. These results were in agreement with results obtained by Koren [26] Sehmel and Babb [53]. Maintaining the operating conditions the same as in the clarified solution test; when spent uranium tailings were added to the aqueous phase, the column operated in the transition region between the mixer-settler and the emulsion region. The column remained in this region of operation till the pulse frequency or the pulse amplitude or both were increased.

Stable emulsions obtained in gold tailings system prevented reliable hold-up measurements and suggested that this system can not be studied unless the crud formation is eliminated.

A fractional factorial design was carried out during the experiments in order to minimize the number of runs and avoid erosion to the tailings pump. The experimental data of each system were fitted to a second order model as proposed by Bell and Babb [6] in order to correlate the hold-up data. No common form of these equation models for predicting hold-up was noticed, each model had its unique identity. Different equations obtained from literature were used to predict the hold-up. In the case of uranium tailings system, The models suggested by Sato et al [50] was found to be the best when uranium concentration was 10 % by weight, while, Arthayukti's [1] model gave better agreement for 25 % uranium tailings.

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Finally, I would like to dedicate this thesis to my wife whose love and support will never be forgotten.

Notation

Various symbols used frequently in this work are summarized below. All notation is fully defined where it first arises in the text.

Symbols

a	Constant-equation 2.24
A	Pulse amplitude (cm).
A_1	Constant-equation 2.25
B	A measure of drop coalescence.
B_i	Parameter in the proposed polynomial model-equation 4.2
C_i	Constants-equation 2.23
D	Constant-equation 2.26
d	Orifice diameter (cm)
D_a	Sauter mean diameter (cm)
D_e	Equivalent drop diameter (cm)
D_i	Impeller diameter (cm)
d_f	Final drop diameter (cm)
d_n	Sieve Plate hole diameter (cm)
D_v	Nozzle or hole diameter (cm)
F	Pulse frequency (1/min), (1/s)
F_B	Buoyancy force (N)
F_K	Kinetic energy force (N)

F_I	Inertia force (N)
F_T	surface tension force (N)
F_D	Drag force (N)
F_t	Transition frequency(min^{-1}), (s^{-1})
g	Gravitation constant (m/sec^2)
H	Column height (m)
h	Pulse plate spacing (cm)
l	Axial distance between plates (m)
L	Compartment height (cm)
N	Rotational speed (s^{-1})
N_{Re}	Reynold's number
r_j	Radius of the jet (cm)
T	Time (min)
t	Time at second stage (min)
t_d	Time at second stage at detachement (min)
Q	Volumetric flow rate (ml/min)
V	Velocity of drop during second stage
V_c	Continuous phase superficial velocity (cm/min)
V_d	Dispersed phase superficial velocity (cm/min)
V_D	Liquid drop volume (m^3)
V_E	Volume of the drop at the end of first stage (m^3)
V_F	Final volume of drop(m^3)
v_e	Dispersed phase drop velocity at expansion (m/s)
v_n	Dispersed phase velocity through the orifice (m/s)
V_s	Velocity of a drop relative to continuous phase (m/s)
$\overline{V_o}$	Mean characteristic velocity (m/s)

V_t	Terminal drop velocity (m/s)
	:x: Distance from nozzle tip.
X_1	Pulse frequency (min^{-1})
X_2	Aqueous flow rate (ml/min)
X_3	Organic flow rate (ml/min)
X_d	Dispersed phase hold-up %

Greek Symbols

α	Fractional free area of the pulse plate - equations 2.13, 2.18 and 2.22
β	$\alpha^2/(1-\alpha)(1-\alpha^2)$ - equations 2.13 and 2.18
γ	Surface tension (mN/m)
ψ	Parameter introduced by Miyauchi and Oya.
ψ_{mod}	Parameter introduced by Kumar and Hartland [29]
λ	Ratio of velocity difference to π times the pulse velocity.
μ_c	Viscosity of the continuous phase (Pa.s.).
μ_d	Viscosity of the dispersed phase(Pa.s.)
ρ_c	Density of the continuous phase (kg/(m^3).
ρ_d	Density of the continuous phase (kg/(m^3).
$\Delta\rho$	Density difference (kg/(m^3).
Δ_C	Average flow rate of all material past the pulsed column plates in the direction of the continuous phase flow (ml/min).
Δ_D	Average flow rate of all material past the pulsed column plates in the direction of the dispersed phase flow (ml/min).
Δh	height difference (cm).
σ	Interfacial tension (mN/m).
σ_s	Specific interfacial tension (mN/m).
σ_e	Effective interfacial tension (mN/m).

Π	3.14159.
π_M	Root mean-square of π_C andπ_D, ft/hr.
π_V	Average of π_C andπ_D, ft/hr.

Subscript

$_f$	Flooding condition

Abbreviations

Alamine 336	Tri-amine compound (R_3N)
HDTMAB	Hexadecyl Tri-Methyl Ammonium Bromide
S.D.	Standard Deviation or standard error

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Chapter 1

Introduction

Pulsed columns are used in liquid-liquid extraction processes as an alternative to mixer settlers for dispersing immiscible phases. They have proved to be reliable and safe in reprocessing of fast reactor fuels and spent nuclear fuels such as uranium and plutonium from the associated fission products. Pulsed columns are applied in treating highly corrosive solutions, in petroleum refining, petrochemical and in hydrometallurgical plants for the recovery of metals from aqueous solution.

Compared to other types of solvent extraction equipment, pulsed columns have low hold-up volume i.e., minimum contact time which reduces solvent degradation. Pulsed columns occupy less floor space thus reducing shielding requirements and are controlled by simple devices which are amenable for remote operation causing minimum failure; thus, reducing maintenance time and personnel exposure. They have operational flexibility such as flow rates, agitation intensity (frequency and amplitude), and plate characteristics which can be easily varied.

This work was undertaken to compare the hold-up characteristics when operating a solvent in pulp system to the operation of a pulsed column using clarified solutions. Solvent in pulp processing has been suggested as an alternative to the clarified solution operation, since leached pulps would be processed without sand-slime separation or filtration. This type of operation would substantially reduce space, equipment, time, and reduce solution inventories [42].

Hold-up data are required for the evaluation of the longitudinal mixing and flooding, so that, optimum throughputs can be achieved. Hold-up data are also used to evaluate residence time distributions in the determination of the mass transfer rates, and the calculation of solution inventories in extraction plants [Yih and Burkhart, [65]].

Chapter 2

Literature survey

2.1 Pulsed Column Operation

Sege and Woodfield [52] reported that there are three distinct operating regions in pulsed column processing, see Figure 2.1. These regions are functions of the throughput rate and the agitation intensity (frequency and amplitude). The operating regions are as follows:

2.1.1 Mixer-settler Operation

Mixer-settler operation takes place at low phase velocities and low agitation intensities, Figure 2.2(A). This operation is characterized by the separation of the light and heavy phases into distinct and clear layers during the quiescent portions of the pulse cycle.

For an organic light phase dispersed system and during the upward portion of the pulse movement, the organic phase initially rests under the sieve plate, it is

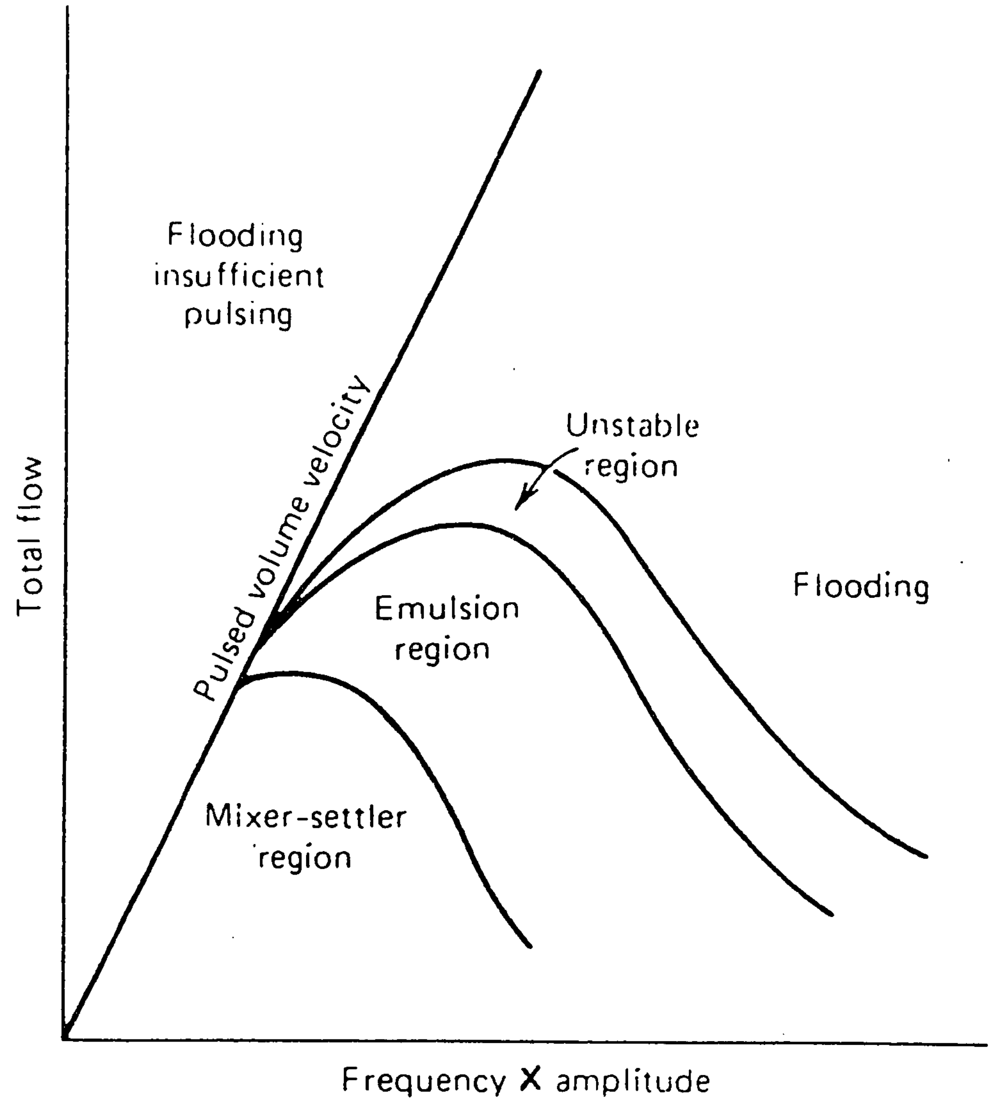


Figure 2.1: Operating characteristics of sieve plate extraction column.

forced up through the perforations and rises through the heavy continuous phase layer above the plate. At the end of the pulse cycle the drops coalesce under the next plate. When the heavy phase is dispersed the situation is reversed with the heavy phase travelling down the column. The mixer-settler type operation of the pulsed column is highly stable but extraction efficiencies are low compared to the operation of the column under the emulsion type conditions.

2.1.2 Emulsion Operation

Emulsion operation takes place at higher phase velocities and higher agitation intensities, as shown in Figure 2.2(B). This kind of operation is characterized by small drop sizes and fairly uniform dispersion of the dispersed phase. The intimate interface contact (high interfacial contact area per unit volume) renders it a more efficient of contacting region.

2.1.3 Unstable Operation

Unstable operation occurs at still higher throughput rates and agitation intensities, see Figure 2.2 (C). The operation is characterized by a mixture of fine and coarse dispersed phase drops, and the coalescence of the drops to form large irregular shaped globs of dispersed phase and periodic reversals of the continuous phase in short sections of the column (cyclic local flooding). The efficiency of the column in the unstable operation region, is generally poorer than in the emulsion type operation, and can fluctuate widely.

2.1.4 Flooding and its Characteristics

Flooding is defined as the exit of one of the entering phases through the effluent line intended for the other phase. Flooding occurs due to a further increase beyond the unstable region of the throughput rate and/or the frequency. These changes in phase dispersion behavior involve gradual blending of one type of operation into another, rather than abrupt transition. Two types of flooding conditions have been reported:

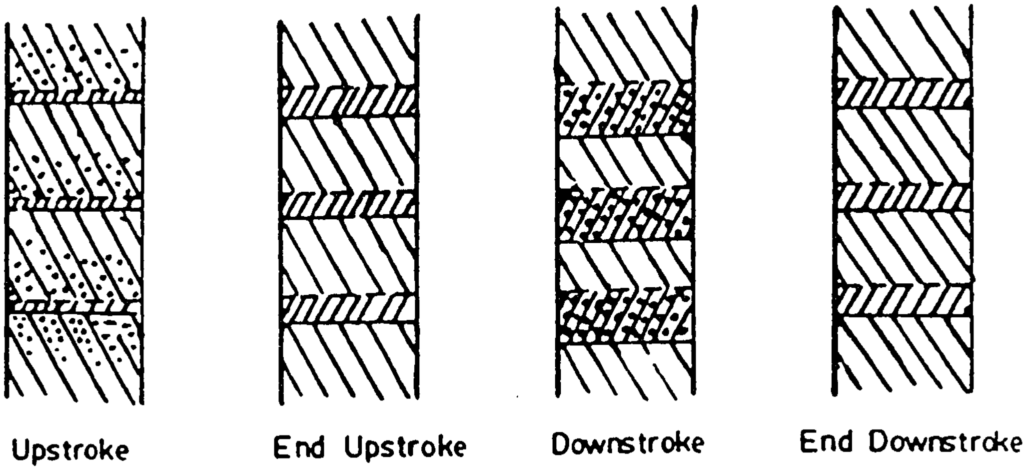
- Flooding Due to Insufficient Pulsing

The area between the "Total flow" axes and the inclined straight line in Figure 2.1 indicates flooding due to insufficient pulsing. This type of flooding is called the lower flooding point.

Sege and Woodfield [52] proposed that flooding will occur when the pulse volume is less than the total volumetric rate of flow. In terms of the aqueous and organic phases velocity, flooding takes place when:

$$V_d + V_c \geq A \times F \tag{2.1}$$

(A) The mixer-settler operation



(B) The emulsion operation

(C) The unstable operation

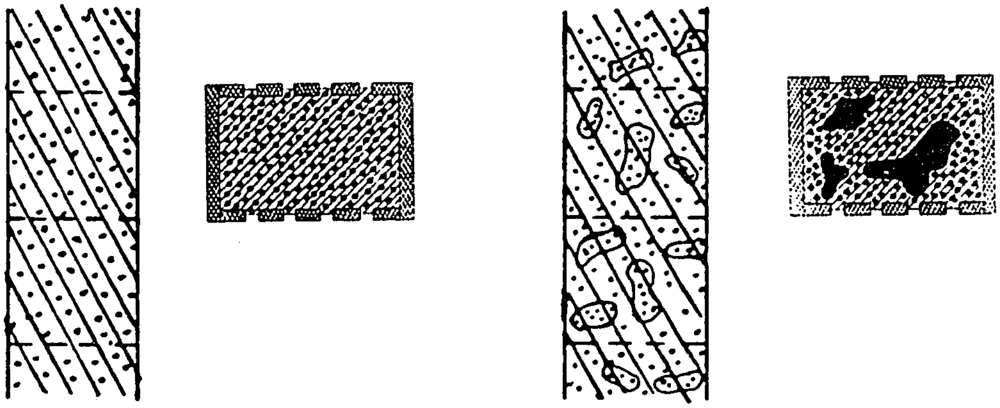


Figure 2.2: Behavior of drops in pulsed column operation, [Baird, Hanson [5]].

Mersmann [36] correlated the lower flooding point as a function of the relative velocity as follows:

$$(V_d + V_c)_f = 2 \times A \times F \tag{2.2}$$

- Emulsion Flooding

To the right of the lower flooding point area and beyond the upper curved line in Figure 2.1 shows the so called upper flooding curve. The column under this condition is overloaded and stable operation is not possible. This type of flooding occurs at higher pulse agitation and as the phase velocities increases. When the continuous phase velocity is greater than the droplet settling velocity the dispersed phase is carried out with the continuous phase and flooding occurs. This has been termed the upper flooding point or emulsion flooding, Mersmann [36]. This type of flooding is due to the emulsification of the dispersed phase, and is characterized by very dense population of drops and their coalescence into larger clusters causing an unstable mode of operation with respect to the dispersed phase holdup and drop size; as shown in Figure 2.3. The entrainment of small drops through the continuous phase outlet during stable operation is not an indicative of flooding.

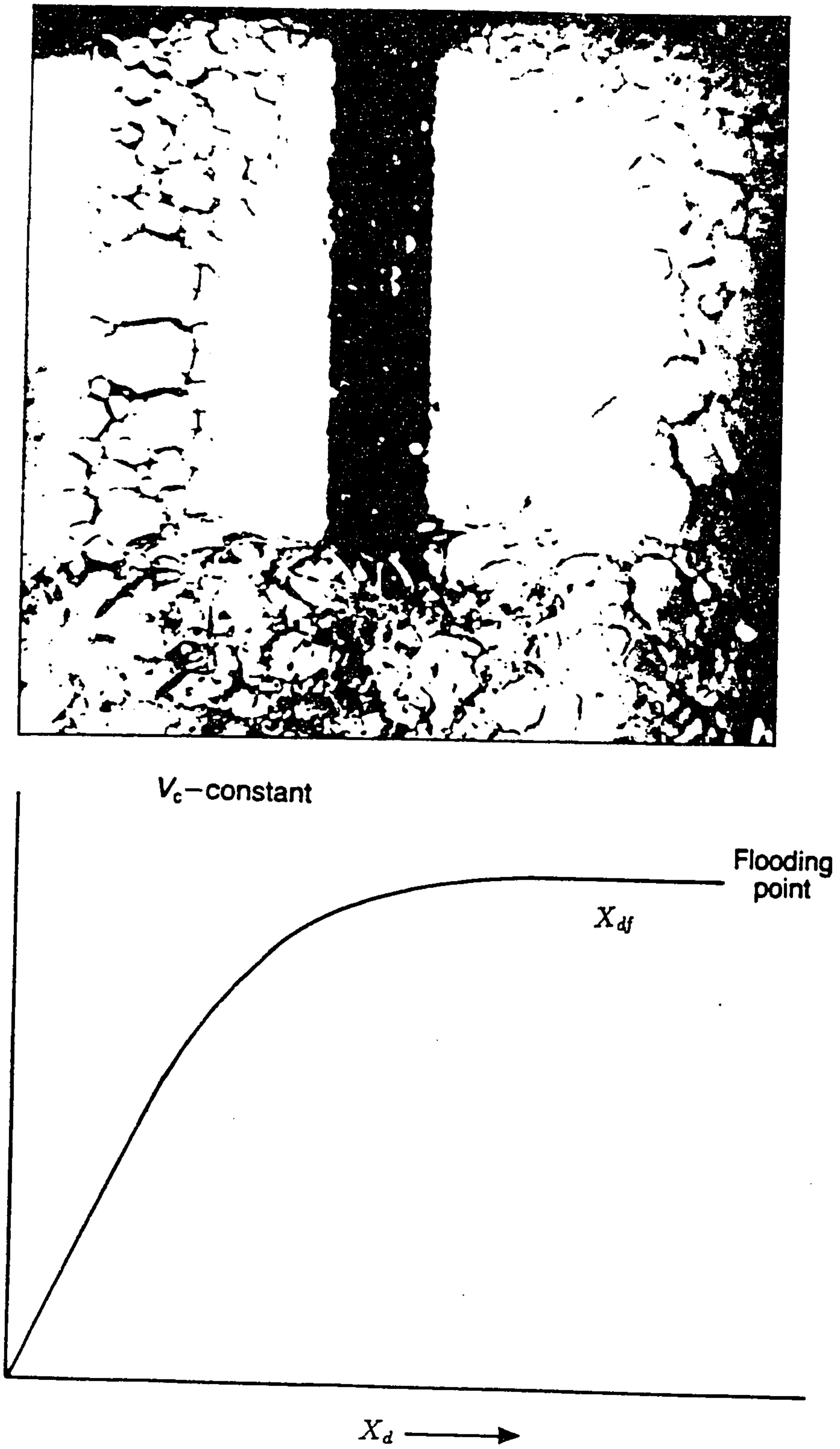


Figure 2.3: Hold-up behavior as flooding is approached.

2.2 Hold-up

The overall hold-up is the total dispersed phase volume at steady state operation travelling through the continuous phase and expressed as a fraction of the total effective column volume. Pulsed column operations using clarified solutions have been studied extensively by Niebuhr and Vogelpohl [41], Miyauchi and Oya [38], Kumar and Hartland [29] and Golding [14]. Investigations using pulsed column for solvent in pulp processing have not been widely reported. Initial studies on solvent in pulp processing in both mixer settlers and pulsed columns indicated that it was impractical due to the formation of stable emulsions. However, Joe et al [23] studied hold-up for solvent in pulp process and suggested that, when treating slurries the flow rate, amplitude and frequency should be decreased by half compared to the operation with clarified solutions. Golding and Koren [15] carried out measurements and comparison of hold-up, drop size and interfacial area when operating a solvent in pulp process using silica sand as compared to the operation using clarified phases. Above investigators found that the hold-up in solvent in pulp processes are smaller than for clarified solutions under the same conditions of flow rates, amplitude and frequency. That is where, with clarified solutions the column operates in the emulsion region while with solvent in pulp processing the column will operate in the transition region. It appears that the solvent-slurry system has a higher interfacial tension than that of the clarified solutions and more energy of pulsing would be needed in order to reach the emulsion region. However, the intensity of agitation must be limited so as to prevent emulsion formation. The above authors used the parabolic formulation as predicted by Bell and Babb [6] to predict and describe the hold-up; with the minimum of the function marking the transition frequency between the mixer-settler and the emulsion region as was found by Tutaeva and Kagan[61] and by Bell and Babb[6].

Many hold-up equations have been proposed in literature, but a generalized equation has not been found to cover all the reported data. Thornton [59] related flow rates V_d, V_c, the dispersed phase hold-up X_d and the slip velocity for the emulsion region as in this equation,

$$V_s = V_d/X_d + V_c/(1 - X_d) \tag{2.3}$$

Thornton used the concept that the slip velocity under the limiting condition of essentially zero hold-up can be considered as a characteristic velocity $\overline{V_o}$, which is the mean droplet velocity and defined as follows :

$$V_s = \overline{V_o}(1 - X_d) = V_d/X_d + V_c/(1 - X_d) \tag{2.4}$$

when $V_c = 0$ and $V_d \rightarrow 0$

Misek [37] modified the concept of Thornton using the following equation :

$$V_s = \overline{V_o}(1 - X_d)\exp(BX_d) \tag{2.5}$$

where, B is an empirical constant included as a measure of drop coalescence in the system.

The hold-up at flooding X_{df} may be obtained by differentiating Thornton's equation 2.4 with respect to X_d assuming that $\overline{V_o}$ is constant to give :

$$for\ \frac{dV_c}{d_X}=0 \quad V_{df}=2\overline{V_o}(1-X_{df})X_{df}^2 \tag{2.6}$$

$$and\ for\ \frac{dV_d}{d_X}=0 \quad V_{cf}=\overline{V_o}(1-X_{df})^2(1-2X_{df}) \tag{2.7}$$

Eliminating $\overline{V_o}$ between these equations gives :

$$V_{cf}/V_{df}=(1-X_{df})(1-2X_{df})/2X_{df}^2 \tag{2.8}$$

Knowing the flow rates at flooding V_{cf} and V_{df} enable to compute the hold-up at flooding X_{df}, which is a function of the flow ratio only. Using equation 2.6, Plotting V_{df} against $X_{df}^2(1-X_{df})$ is a straight line, whose slope is equal to the characteristic velocity $\overline{Vo}$.

Oloidi Jo and Jeffreys [44] and Prabhu et al [46] studied hold-up in a 5 cm diameter column with 42, 0.16 cm hole size plates using the system water (continuous) and kerosene (dispersed). The data were correlated as follows:

$$X_d=4.8\times10^{-2}(\Delta\rho/\rho_c)^{-0.01}(V_d/V_c)^{-0.05}(V_nd_n\rho_c/\sigma)^{0.73}(V_n/d_ng)^{-0.24} \tag{2.9}$$

Sato et al [50] proposed different correlations independent of the system physical properties in order to determine hold-up in each of the operating regions as follows:

- Mixer-settler region

$$X_d = 0.52(A \times F/V_d)^{-0.70}(1 + V_d/V_c)^{-0.70} l^{-1.2} d^{-0.26} \tag{2.10}$$

for $0 \leq A \times F \leq 1.3 V_d^{0.22} l^{-0.32} d^{-0.37}$

- Dispersion region

$$X_d = 0.42(A \times F) V_d^{0.33}(1 + V_d/V_c)^{-0.70} l^{-0.68} d^{0.37} \tag{2.11}$$

for $1.3 V_d^{0.22} l^{-0.32} d^{-0.37} \leq A \times F \leq 0.8 V_d^{0.22} l^{0.35} d^{-0.37}$

- Emulsion region

$$X_d = 0.54(A \times F)^{2.4}(1 + V_d/V_c)^{-0.70} l^{-1.2} d^{0.89} \tag{2.12}$$

for $A \times F \leq 0.8 V_d^{0.22} l^{0.35} d^{-0.37}$

Miyauchi and Oya [38] introduced a criterion by which hydrodynamic operating region can be empirically approached. This is based on value of a parameter ψ which has been observed when plotting hold-up versus ψ for the system methyl isobutyl ketone (M.I.K) 0.1 N aqueous potassium chloride system as well as for data reported by Cohen and Beyer [9], by Li and Newton [31] and by Sehmel and Babb [53] then :

$$\psi = A \times F/(\beta l)^{0.33}({\mu_d}^2/\sigma \Delta \rho)^{0.25} \tag{2.13}$$

and

$$\beta = \alpha^2/(1-\alpha)(1-\alpha^2)$$

where β is a function of the plate free area (α), for $\psi = 0.21$ where there is a marked change at the transition between the mixer-settler and the emulsion operation. Hence :

$$X_d = 0.66\psi^{0.84} V_d^{0.67} \ldots. for\ \psi < 0.21 \tag{2.14}$$

$$X_d = 6.32\psi^{2.4} V_d^{0.67} \ldots\ldots. for\ \psi > 0.21 \tag{2.15}$$

Arthuyukti [1] used the pulse intensity to identify the operating regions:

$$X_d = 0.38 V_d^{0.67} \psi^{0.75} \ldots. for\ A \times F < 4.57\ cm/s \tag{2.16}$$

$$X_d = 3.46 V_d^{0.67} \psi^{2.62} \ldots. for\ A \times F > 4.57\ cm/s \tag{2.17}$$

Kumar and Hartland [29] suggested that, the dependance of hold-up on the column geometry, operating variables and physical properties of the phases can be expressed in dimensionless form as product of dimensionless groups. Before the relevant indices can be obtained, it is necessary to determine the break-point, E, between the emulsion region and the dispersion region which is a transition region between the mixer-settler and the emulsion region.

$$E = \frac{(Af)^3}{\beta L} \frac{\rho_c}{\Delta\rho^{3/4} \sigma^{1/4} g^{5/4}} \tag{2.18}$$

- for the dispersion region (for $E < 0.05$)

$$X_d = 6.91 \left(\frac{(AF)^3 \rho_c^{1/4}}{\beta L \sigma^{0.25} g^{5/4}}\right)^{0.31} \left(\frac{V_d^4 \rho_c}{g\sigma}\right)^{0.30} \left(1 + \frac{V_c}{V_d}\right)^{0.14} \left(\frac{\Delta\rho}{\rho_c}\right)^{-0.79} \left(\frac{\mu_d^4 g}{\rho_c \sigma^3}\right)^{-0.01} \tag{2.19}$$

- for the emulsion region (for $E \geq 0.05$)

$$X_d = 3.73 \times 10^{-3} \left(\frac{(AF)^4 \rho_c}{g\sigma} \right)^{0.62} \left(\frac{V_d^4 \rho_c}{g\sigma} \right)^{0.31} \left(1 + \frac{V_c}{V_d} \right)^{0.45} \left(\frac{\Delta\rho}{\rho_c} \right)^{-2.20} \left(\frac{\mu_d^4 g}{\rho_c \sigma^3} \right)^{-0.29} \quad (2.20)$$

- for the mixer settler region the rate of energy dissipation per unit volume (E) is not a controlling factor and equation 2.21 should be used when:
  - $E < 0.05$
  - The predicted value of hold-up is greater than that predicted by equation 2.19 for the dispersion region.

$$\begin{aligned} X_d &= 3.91 \times 10^{-3} \left(\frac{A^2 \rho_c g}{\sigma} \right)^{-0.26} \left(\frac{F^4 \sigma}{\rho_c g^3} \right)^{-0.19} \left(\frac{V_d^4 \rho_c}{g\sigma} \right)^{0.28} \\ & \left(1 + \frac{V_c}{V_d} \right)^{0.19} \left(\frac{\Delta\rho}{\rho_c} \right)^{-0.81} \left(\frac{\mu_d^4 g}{\rho_c \sigma^3} \right)^{-0.13} \qquad (2.21) \end{aligned}$$

Groenier et al [17] and McAllister et al [35] recommended the following regression equation to determine flooding conditions in pulsed column as follows:

$$\begin{aligned} \ln \left(\frac{V_c + V_d}{\Delta_C + \Delta_D} \right)_f &= -3.741 + 0.2568 \ln \frac{\Delta\rho d\alpha^2}{\rho_c \pi_M^2} - 0.07194 \left(\ln \frac{\Delta\rho d\alpha^2}{\rho_c \pi_M^2} \right)^2 \\ & +0.006191 \left(\ln \frac{\Delta\rho d\alpha^2}{\rho_c \pi_M^2} \right)^3 - 1.034 \ln \frac{\mu_c \pi_v}{\alpha\sigma} + 0.09096 \left(\ln \frac{\mu_c \pi_v}{\alpha\sigma} \right)^2 \\ & +0.1424 \ln \frac{V_c}{V_d} - 0.1807 \ln \frac{A}{h} \\ & +0.07198 \left(\ln \frac{\Delta\rho d\alpha^2}{\rho_c \pi_M^2} / \ln \frac{\mu_c \pi_v}{\alpha\sigma} \right) \qquad (2.22) \end{aligned}$$

Where :

$\Delta_D = 5FA\left((1-\lambda^2)^{1/2} - \lambda(\pi/2 - \arcsin\lambda)\right)$, ft/hr

$\Delta_C = 5FA\left((1-\lambda^2)^{1/2} + \lambda(\pi/2 + \arcsin\lambda)\right)$, ft/hr

$\pi_D = 5\pi FA\left((\frac{1-\lambda^2)^{1/2}}{\pi/2-\arcsin\lambda} - \lambda\right)$, ft/hr

$\pi_C = 5\pi FA\left((\frac{1-\lambda^2)^{1/2}}{\pi/2+\arcsin\lambda} + \lambda\right)$, ft/hr

$\pi_M^2 = 1/2(\pi_D^2 + \pi_C^2)$, $(ft/hr)^2$

$\pi_V = 1/2(\pi_D + \pi_C)$, (ft/hr)

$\lambda = (V_c - V_d)/(5\pi FA)$, dimensionless.

Bell and Babb [6] proposed the following general polynomial equation for the correlation of the experimental hold-up data. This equation can be used to calculate hold-up in the three regions of the pulsed sieve-plate column operation.

$$X_d = V_d(C_1 + (C_2 + C_3V_c)(A \times F - C_4)^2) \tag{2.23}$$

Sehmel and Babb [53] determined the overall hold-up in a pulsed sieve plate extraction column. They found that the hold-up as a function of pulsation showed a minimum at the transition from the mixer-settler to the emulsion region. They suggested the following equation for the calculation of the transition frequency (F_t) between the mixer-settler and the emulsion regime,

$$F_t = 0.67(1.23 + 38.2\mu_d\sigma\Delta\rho - ln\ a) \tag{2.24}$$

Fitzpatric [12] found that on the addition of salt to water-butyl acetate system the hold-up was increased and a reduction in flood point was observed when ionizing salts are present in low concentrations. This effect was observed only with polar solvents. They suggested that it must have an electrostatic origin and the ions were adsorbed around the droplet surface by the charges on the polar groups in the dispersed molecules, which could result in mutual repulsion of the droplets and reduced coalescence.

Although the major hold-up data reported in the literature refer to an average value of hold-up for the entire length of the column, some of the point hold-up measurements have been made. Jiriciny and Prochazka [22] and Kagan et al [25] showed that the actual plate hold-up varies from plate to plate. Hans Schmidt [18] plotted hold-up versus column height expressed as the plate number; see Figure 2.4. The flow rate ratio was considered as a variable. The total volumetric flow rate and the pulsation were kept constant. An increase in dispersed phase flow rate resulted in an increase of the dispersed phase hold-up. The results also indicated a continuous increase of hold-up values in flow direction of the drop phase inside the column. The decrease of drop size resulted in higher hold-up values with an increase in column height.

2.3 Drop Formation

The drop formation mechanism at a single orifice depends on the velocity at which the dispersed phase is injected. In the mixer-settler zone i.e., at low velocities, droplets are formed at the tip of each orifice. The main variables affecting drop size

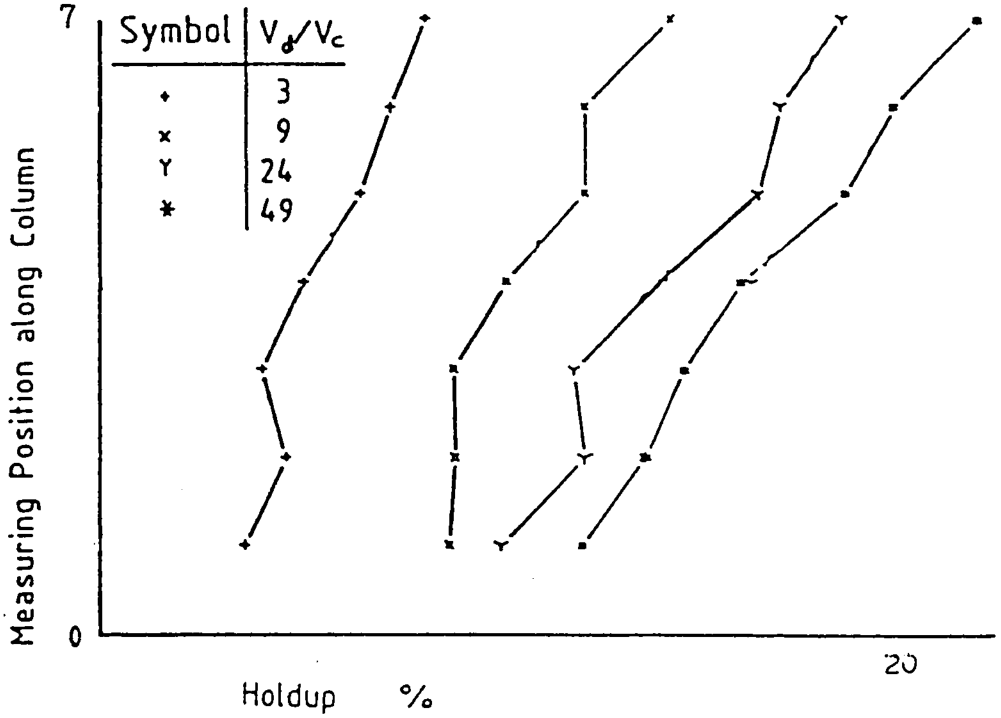


Figure 2.4: Column height versus hold-up % of the organic dispersed phase, Hans Schmidt [18].

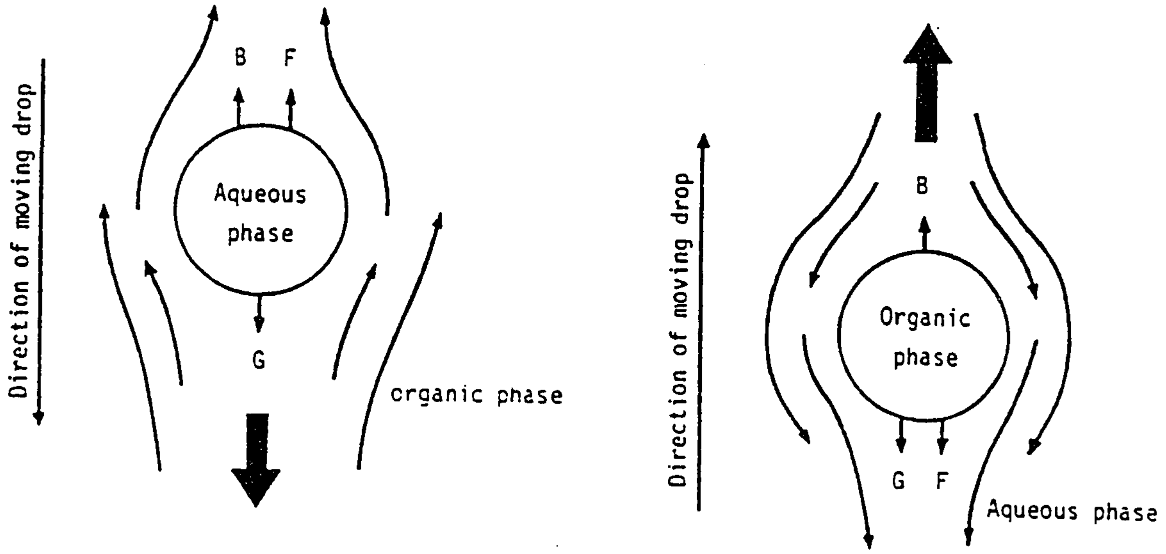
are the orifice diameter, the geometry of the orifice and the physical properties of the two phases. In general, an increase in continuous phase viscosity, nozzle diameter and interfacial tension increases the drop volume, where as the increase in density difference decreases droplet size. The increase in volumetric flow rate of the dispersed phase causes an initial increase in the drop volume in the mixer settler zone subsequent decrease in the emulsion zone and an increase in the flooding zone where cluster of drops are formed [Null and Johnson [43]].

Harkin and Brown [19] developed the following equation for drop volume based on static equilibria balance between the interfacial tension and the buoyancy at low velocity:

$$V_F(\rho_c - \rho_d)g = \Pi A_1 \sigma (d_n^2/d_f) \tag{2.25}$$

Initially, the forces keeping the drop attached to the nozzle are higher than those trying to detach it, hence the drop does not break easily. As the detaching forces exceed the attaching forces, the drop breaks away.

Rao et al [47] proposed that the drop formation takes place in two stages, expansion and detachment. The expansion stage ends when the upward and downward forces are equal. As soon as the upward force dominates the dispersed drop starts moving up as shown in Figure 2.5. The following expression was developed by Kumar [27] as follows :



B: Buoyancy of drop, F: Friction of moving drop, G: Gravity of drop
—Motion of drop in aqueous dispersed drop (a), and organic dispersed drop (b)

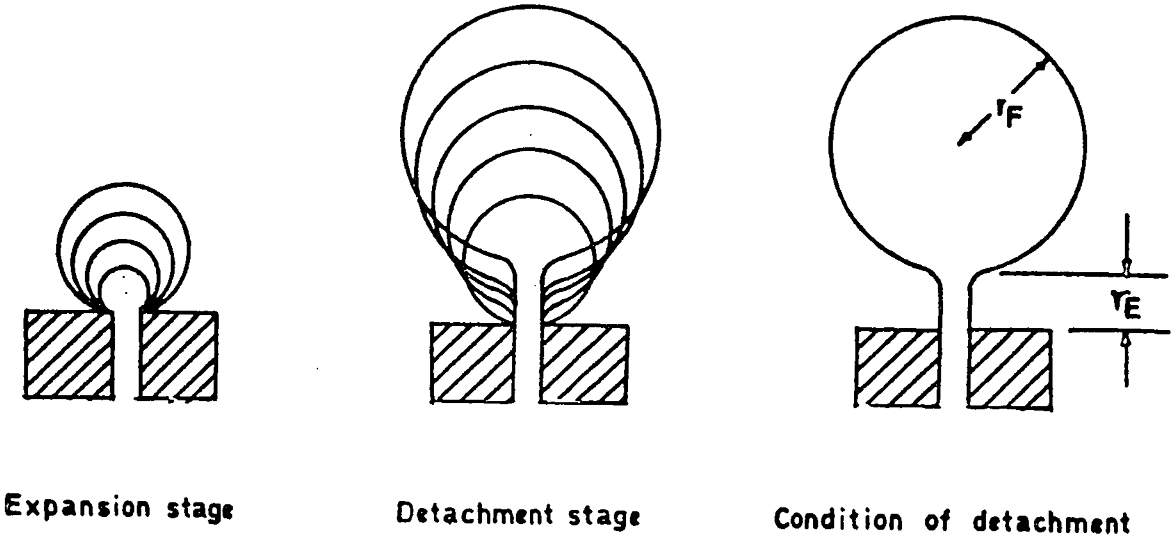


Figure 2.5: Stages of drop formation. Kumar [27] and Matsuda [34].

- Stage I, at expansion $F_B + F_K = F_T + F_I + F_D$

$$\begin{aligned} V_E(\rho_c - \rho_d)g + Q\rho_d V_n &= D\pi\sigma + \rho_s Q^2/12\pi(3/4V_E\pi)^{0.67} \\ &\quad +1.5\mu_c Q/(3/4V_E\pi)^{0.33} \end{aligned} \tag{2.26}$$

- Stage II at detachment $V_F = V_E + Q \times t_d$

$$\begin{aligned} V\rho_s d^2x/d_t^2 + Q\rho_s d_x/d_t &= V(\rho_c - \rho_d)g - 6r\pi\mu_c(d_x/d_t + v_E) - D\pi\sigma \\ &\quad -\rho_s Q^2/12\pi(3/4V\pi)^{0.67} + Q\rho_d v_n \end{aligned} \tag{2.27}$$

where :

- $\rho_s = \rho_d + 11\rho_c/16$
- $V\rho_s d^2x/d_t^2$ is the rate of momentum of the drop $(d(mv)/dt)$.
- $Q\rho_s d_x/d_t$ force due to incoming fluid to the drop.
- F_B, F_K, F_I, F_T, and F_D are forces due to the buoyancy, kinetic energy, inertia, surface tension, and drag respectively.

At higher velocities of flow that is at emulsion zone, a liquid jet is first formed which subsequently breaks down into individual droplets. the final drop size can be estimated by the following equation,

$$V_F = 12.56\Pi {r_j}^3 \tag{2.28}$$

where r_j is the radius of the jet.

Kumar [28] has shown that viscosity, density and interfacial tension influence

V_F even in the emulsion zone so that equation 2.28 can only be used as an approximation method.

Bethuel et al [7] used an alternating electric field imposed between the dispersed phase inlet and the continuous phase exit in a pulsed sieve plate column in order to coalesce entrained droplets. The authors found that it was possible to operate at increased agitation levels and at higher dispersed phase hold-up than is usual in conventional column. This process improved extraction efficiencies and stable operation was maintained close to column flooding. Also they proposed that their modification is useful in other types of column contactor.

Bailes et al [2] demonstrated that the electric field can effectively control coalescence-redispersion of droplets in vigorously rotary agitated system and determined the relative effects of field strength and impeller speed which caused enhancement in the mass transfer rates.

2.4 Crud Formation

Carino [8] described the crud formation as the most common operating problem in solvent in pulp processing. Cruds are considered to be a result of solid particles surrounding the organic phase. The drainage of the organic phase is hindered and emulsions are formed. Crud formation is normally eliminated by an efficient clarification and filtration process; and minimized by the application of low frequencies and pulse amplitudes which reduce back mixing. Addition of a controlled amount of surfactant was found to reduce crud formation. Jones and Lee [24] noted that the addition of an excess amount of surfactant decreases the dispersed phase droplet sizes and produces uniform droplets which are then entrained in the continuous phase leading to operational interruption.

Hong and Lee [21] found that the drainage time of the organic phase is one parameter that measures the difficulty of demulsification. This difficulty might be due to the excess of the surfactant quantity that leads to further delay in settling time of the solid particles. This delay was explained by above authors in terms of interface movement and a double layer repulsion.

Mizrahi and Barnea [39] proposed that the wettability of tailings can vary, that is, in the absence of organic phase, solids may have a hydrophilic nature. However, at equilibrium and when in contact with an organic phase, the tailings become slightly hydrophobic and phase coalescence is hindered.

Chapter 3

Equipment and Procedures

3.1 Experimental Work

A schematic diagram of the pulsed column apparatus is shown in Figure 3.1 with principal dimensions given in Table 3.1. The cylindrical plexi-glass column was fitted with 17 stainless steel perforated plates equally spaced along the height of the column. Pulsation was obtained by means of Denver diaphragm pump. The frequency was varied by changing the speed of rotation of the cam, while amplitude of pulsing by varying the length of the connecting rod.

The organic phase entered the column through a polypropylene 'T' fitting using Milton Roy controlled volume metering pump. The aqueous phase entered the column through a stainless steel distributor using a Robins and Myers Moyno pump. The aqueous flow was measured and regulated by monitoring a manometer while changing the speed of rotation of the Moyno pump. See Appendix A for the calibration of the aqueous and the organic phases pumps.

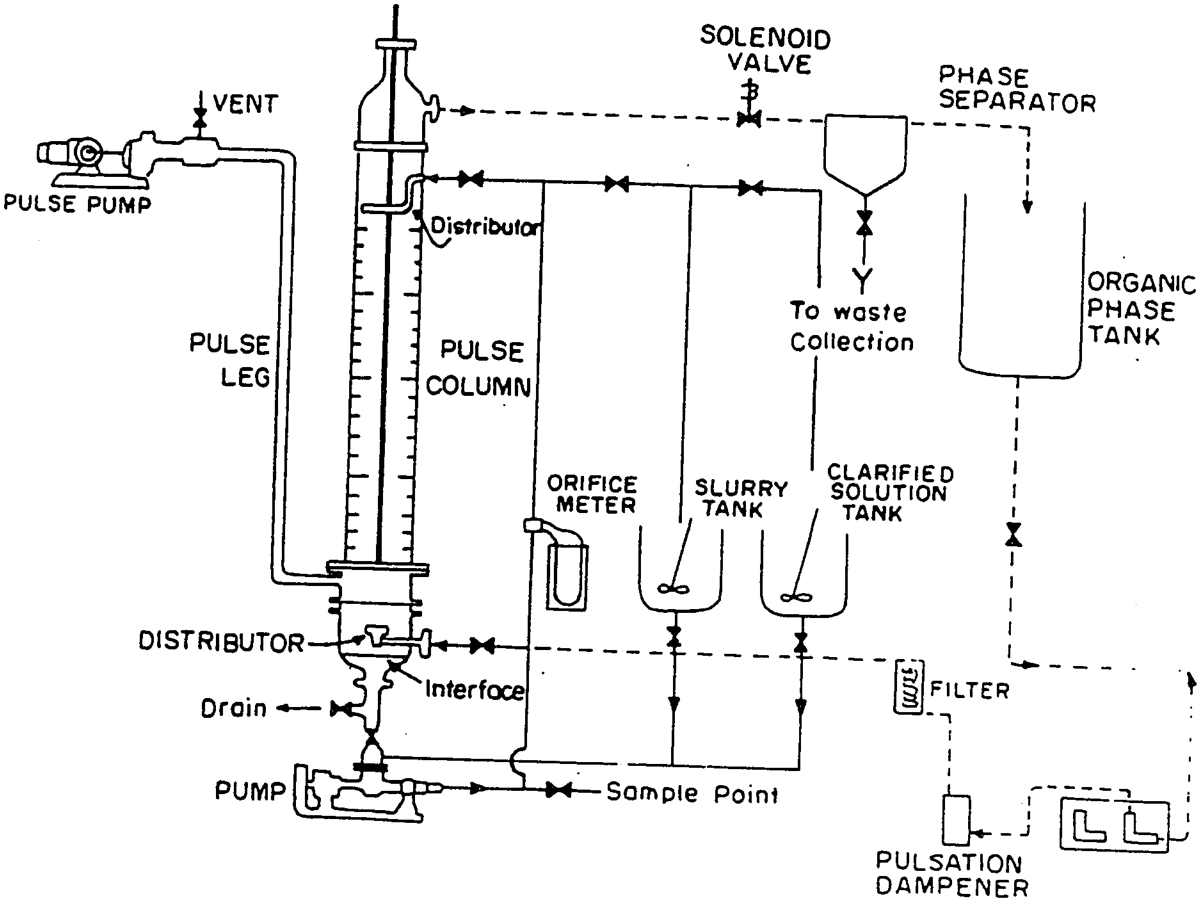


Figure 3.1: Schematic diagram of Pulsed column.

Table 3.1: Pulsed column details.

Column I.D.	10.160
Plate Diameter	10.097
Plate Thickness	0.166
Central rod O.D.	0.953
Aqueous Distributor O.D.	6.580
Plate spacing	7.620
Distance between 1st plate and organic distributor	237.8
Effective volume (Litres)	11.120
1 - Uranium tailings system:	
Number of holes	191
24.14 % free area plate.	
Sieve diameter	0.359
2 - Gold tailings system:	
Number of holes	31
12.4 % free area plate.	
Sieve diameter	0.635

Note: Dimensions are in centimeters, unless otherwise specified.

3.2 Preparation of the Chemicals

The organic and the aqueous phases used in this study were the uranium Campbell Red Lake tailings and the alkaline cyanide (3-10 ppm) gold leach tailings. Operating conditions were chosen after several bench scale tests have been made to determine the best operational condition for minimum or no stable emulsion formation; see sections 4.1 and 4.2. The uranium tailings and gold leach tailings particle size were determined by taking a dried sample of the tailings and screening, details of particle size analysis are given in Table 3.2 The physical properties of the organic and the aqueous phases used in this investigation are given in Table 3.3. Solution preparation is as given below:

3.2.1 The Uranium Tailings System

The organic phase was 5% by volume Alamine 336 (R_3N, extractant), 5% Isodecanol (Exxal) as a modifier, and Varsol DX 3641 (diluent).

The clarified phase was an aqueous phase containing 30 g/l ammonium sulphate. The solvent in pulp solution was prepared by adding the desired uranium tailings to the clarified aqueous phase. The original pH of the aqueous phase was 4.04 and was adjusted to the desired value by addition of sodium hydroxide solution.

3.2.2 The Gold Leach Tailings System

The organic phase under study was 20 % by volume xylene and 80 % by volume tributyl phosphate. The aqueous clarified phase was 91 % by volume of distilled water adjusted to an ionic strength of 0.5 M Na_2SO_4 and 9 % by volume of 0.06 M hexadecyl tri-methylammonium bromide (HDTMAB) as a surfactant. Then it was adjusted to pH 10.1 as is usual with operation of cyanide solution. The gold leach solution was prepared by adding 10 % by weight gold leach tailings to the clarified aqueous phase.

Table 3.2: Particle size analysis of tailings used in these experiments.

Screen size(mm.)	% Retained
1 - Uranium Tailings	
-1.19+0.500	39.0
-0.50+0.250	9.5
-0.25+0.177	12.0
-0.177+0.150	15.0
-0.15	24.5
Total	100
2 - Gold Tailings	
+0.149	3.41
-0.149+0.105	6.74
-0.105+0.074	10.82
-0.074+0.053	9.09
-0.53+0.044	4.03
-0.044	65.01
Total	100

N.B: The uranium tailings and gold leach tailings particle sizes were determined by screening a dried sample of the tailings.

Table 3.3: Physical properties of the clarified aqueous, organic phases and tailings at 25° C.

Liquid	pH	Density (ρ) kg/m^3	Viscosity (μ) mPa s	surface tension (γ) mN/m
1 - Uranium system				
Organic phase		796.4	1.690	25.20
Aqueous phase	4.04 10.06	1020.5	0.930	51.30
Uranium tailings		3450.0		
Interfacial tension (σ) Aq.- Org.				9.46
2 - Gold system				
Organic phase		953.0	3.389	26.78
Aqueous phase	10.10	1058.0	0.963	36.79
Gold tailings		2250.0		
Interfacial tension (σ) Aq.- Org.				0.26

Note:

- Only the physical properties of clarified aqueous solutions are listed.
- Densities were measured using an Anton Paar Digital densitometer Model 60.
- Viscosities were measured using Cannon-Fenske Routine Viscometer.
- Surface and interfacial tensions were measured using Fisher Model 215 Autotensiomat surface tension analyzer.

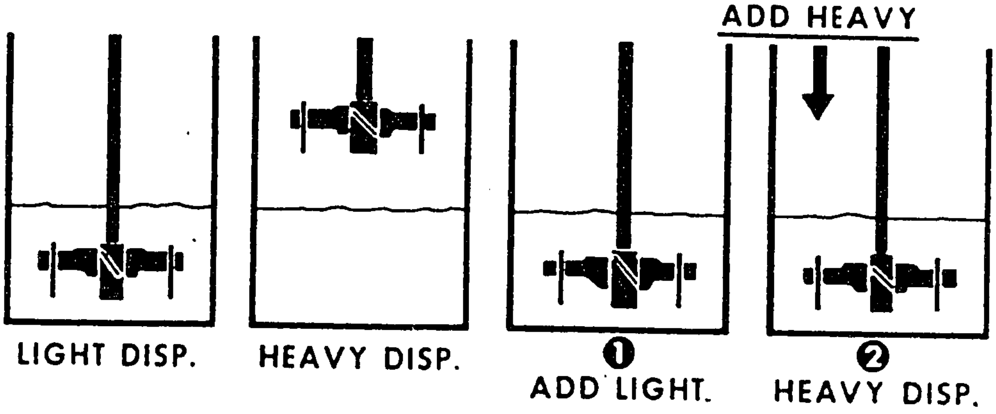


Figure 3.2: Method of selecting dispersed phase, [Baird and Hanson [5]].

3.3 Experimental Procedures

Before work was carried out on the pulsed column, a preliminary bench scale separation tests were performed to select the best operational condition for a minimum or no stable emulsion formation. These separation tests are based on procedures reported by Baird and Hanson [5] as shown in Figure 3.2.

An axial-flow turbine of 50 mm stainless steel diameter impeller with 4 curved blades was used in these bench scale tests. The organic to aqueous phase ratio was 1:2. The mixing period for each test was 10 minutes each time. The mixing speeds were as shown in Table 3.4 with their respective Reynolds' numbers based on the following formula from "Chemical Engineers' Handbook" [45].

$$N_{Re} = D_i^2 N \rho / \mu \tag{3.1}$$

Two basic modes of operation were investigated upon using the pulsed sieve plate extraction column, clarified solution operation and solvent in pulp processing.

3.3.1 Clarified Solution Operation

The aqueous and the organic phases were operated as both dispersed and continuous phases. The experimental procedure upon using the pulsed sieve plate extraction column was as follows:

a - Aqueous phase (dispersed), organic phase (continuous)

This operation was only used with the clarified solution of the uranium tailings system. The aqueous/organic interface was in the lower disengagement section of the pulsed column. This method of operation gives the best photographic data for drop size measurement. The procedure was as follows.

1. The bottom settling region of the pulsed column was filled with the aqueous phase to about 3 to 4 cm above the final interface level.

2. The rest of the column was filled with the organic phase. The pulse leg was only partially filled in order to observe and measure the pulse frequency.

Table 3.4: Axial-flow turbine speeds were expressed as Reynolds numbers based on the continuous phase and clarified solution properties.

Mixer speed (rpm)	N_{Re}	Flow range
1- Uranium system		
a- Organic phase		
450	8823	Turbulent
1000	19529	Turbulent
1500	29418	Turbulent
b- Clarified aq.		
450	20575	Turbulent
1000	45538	Turbulent
1500	68582	Turbulent
2- Gold system		
a- Organic phase		
450	5272	Turbulent
1000	11670	Turbulent
1500	17575	Turbulent
b- Clarified aq.		
450	20595	Turbulent
1000	45584	Turbulent
1500	68651	Turbulent

3. The pulsating pump was started, the pulse frequency and the amplitude were adjusted to their desired value.

4. The aqueous phase pump was started and pumped solution from the bottom settling region of the column and disperse it in the organic phase at the top of the column.

5. The organic phase pump was started and pumped the organic solution from a collecting tank and disperse it in the column through a "T" shaped fitting located at the column bottom. The flow rates was controlled by adjusting the stroke length.

6. In order to obtain consistent results it was necessary to operate by recirculating the phases for more than one hour. After which, the aqueous pump was stopped and the inlet valve closed to begin hold-up measurement procedure.

7. The organic phase and the pulse pump were operated for a further five minutes to ensure that all the aqueous phase in the column had settled to the bottom interface.

8. The hold-up measurement was then made by draining the column and measuring the volume necessary to allow the interface to reach the previously established mark.

9. The fraction hold-up volume was obtained by dividing the volume measured by the effective volume.

b - Organic phase (dispersed), Aqueous phase (continuous)

This operation was used with the clarified solution used in the gold leach tailings system (Set VII) as well as in the clarified solution (Set IV) used in the uranium system. The aqueous/organic interface was in the upper disengagement section of the pulsed column. A modification of the procedure was carried out in order to avoid draining of the column.

1. The upper column part was calibrated using a measuring tape from which the volume of the dispersed organic phase in the aqueous phase can be correlated.

2. The column was filled up with aqueous phase 3 to 4 cm above its inlet at the top of the column (initial aqueous level).

3. Steps 2, 3, 4, and 5 same for the case of aqueous phase (dispersed), organic phase (continuous).

4. When steady state was reached, i.e. no interface level change; This level was marked as final aqueous level.

5. The difference between the initial and the final level expressed as volume, is the hold-up volume.

6. Step 9 remained the same as the previous case.

3.3.2 Solvent in Pulp Operation

Spent uranium aqueous tailings were used as the dispersed phase. For the gold tailings system, runs were attempted with the organic phase and the aqueous tailings dispersed.

Maintaining the concentration of the slurry constant and uniformly distributed when using the draining process would have required continual readjusting and testing the slurry concentration after each run. This would have been a tedious and time consuming procedure. In order to avoid draining the column the following procedure was followed,

1. Both ends of the pulsed column were calibrated using a measuring tape. The divisions of the tape were correlated to volume quantities.

2. Steps 1, 2, 3, 4, 5 and 6 remained the same as before.

3. The fraction hold-up was obtained by dividing the volume measured by the effective volume.

4. Step 9 remained the same as before.

Note: During the experiments, the compartments between the 6^{th} and 7^{th} plates were photographed to allow an analysis of drops size and their distribution.

At the start up of the tailings experiments, there was very little difference in heights between the previously established mark (step 1) and the new interface (step 8). This was due to the small hold-up values found for the solvent in pulp process. The lower part of the column which used to be a bell shaped pyrex settling region of 15 cm I.D. was redesigned and replaced by a piece of plexi-glass of the same column diameter of less area than the previous one, leading to a reasonable difference in interface levels.

Chapter 4

Results

4.1 Results of Bench Scale Phase Separation Tests

Two sets of bench scale separations were investigated as shown in Table 4.1 for uranium tailings and Table 4.2 for gold tailings. The systems with the reasonable short break-time of separation (time for coalescence and settling of solids) and a minimum emulsion formation were selected as being systems suitable for operation in the pulsed sieve plate column.

From Table 4.1 it can be concluded that the best operating conditions for the uranium tailings system was to have the organic phase as the continuous phase (5 % Alamine 336 + 5 % Iosdecanol + Varsol DX 3641); and the aqueous phase (30 g/l $(NH_4)_2SO_4$ + distilled water + tailings) as the dispersed phase, as is usual when dealing with radio-active material.

Table 4.2 showed that the most suitable operating conditions for gold leach tailings system were the 80 % TBP + 20 % Xylene as the dispersed organic phase

Table 4.1: Bench scale separation tests for clarified, uranium tailings and silica sand systems.

- Organic phase : 5 % Alamine 336 + 5 % Iosdecanol + Varsol DX 3641.
- Aqueous phase : 30 g/l $(NH_4)_2SO_4$ + distilled water + tailings as below.

Cont./Disper. phases	N_{Re}	Break-time (min)	Observations
Clarified aq. as above/Org. (0 % tailings)	20575	0.16	Clear separation of phases. System is unaffected by agitation rate.
	45538	0.33	
	68582	0.33	
Org./Clar. Aq.	8823	5.00	Same behavior as above.
	19529	5.00	—
	29418	5.00	—
10 % Silica/Org.	1	5.00	Formation of a third phase at the interface, system unaffected by agitation rate.
	2	5.00	
	3	5.00	
Org./10 % Silica	8823	7.00	Same behavior as above.
	19529	to	
	29418	10.00	—
10 % U/Org.	1	5.00	Formation of a third phase at the interface which increases with the rate of mixing.
	2	5.00	
	3	5.00	
Org/10 % U	8823	5.00	Same behavior as above.
	19529	to	
	29418	10.00	—
25 % U/Org.	1	10.00	Formation of a third phase at the interface which increases with the rate of mixing.
	2	10.00	
	3	10.00	
Org./25 % U	8823	12.00	More of the third phase was formed at the recognized interface
	19529	to	
	29418	17.00	Clear visualization of interface.

Note: "1" stands for 400 rpm, "2" for 1000 and "3" for 1500.

Table 4.2: Bench scale separation tests for clarified and gold leach tailings system.
- The aqueous phase consisted of 0.5 M Na_2SO_4 + 8 % Hexadecyl trimethylammoniumbromide(HDTMAB)(surfactant) + distilled water adjusted to pH 10.1 + 10 % by weight gold tailings.
- The organic phase consisted as shown in the table below.

Aq. + Org. phases Phases	N_{Re} (min)	Break-time	Observations
Clarified aq. phase only as above+80 % TBP+20 % Xylene + 0 % HDTMAB. cont. and disper.	5272 11670 17575	2.0 2.0 2	Good separation and, and system unaffected by mixing. Visible interface.
Org. as below Aq. tailings as above		(min)	
Shell 140 + Alamine + Isodecanol (cont./disp.)	20595 *	∞	A huge stable emulsion formed at the interface.
50 % Varsol DX + 50 % Alamine (cont./disp.)	20595 *	∞	Same behavior as above.
80 % TBP + 20 % Isodecanol (disp./cont.)	20595 *	∞	Same behavior as above.
80 % TBP + 20 % Solv-Esso 150 (disp./cont.)	20595 *	∞	Same behavior as above.
100 % TBP (dispersed)	20595 *	1	Settling of solids was quick but no distinct separation of phases.
50 % TBP + 50 % Toluene (disp./cont.)	20595 *	∞	Stable emulsion formed.

Note:

Tailings solutions Reynolds numbers marked with a star (*) were based on clarified solutions physical properties.

Table 4.2 Gold Leach Tailings Separation tests continued

Organic phase Aq. tailings as above	N_{Re}	Break-time (min)	Observations
50 % TBP + 50 % Xylene (disp./cont.)	20595 *	8.0	Good separation of org./tailings, but tailings in aqueous were in suspension.
hline 80 % TBP + 20 % Xylene (continuous)	5272 11670 17575	0.33 to 5.0	Good separation. Time of settling was directly proportional to the mixing. Visible interface.
80 % TBP + 20 % Xylene (dispersed)	*20595 *45584 *86651	10.0 to 30.0	Slight crud was formed at the interface, with complete settling of tailings.
80 % TBP + 10 % HDTMAB + 20 % Xylene(dispersed)	*20595	10.0	Good solid separation. Visible interface and some crud formation.
80 % TBP +10 % Lignosulphonate + 20 % Xylene(dispersed)	*68651	∞	Gell formation.

and the 10 % HDTMAB + 0.5 M Na_2SO_4 + distilled water of pH 10.1 as the aqueous continuous phase. Since crud was formed in the column with the organic disperse the operation was reversed; i.e., aqueous phase dispersed, organic phase continuous.

4.2 Results of Pulsed Column Operation

Based on the bench scale separation tests results, eight sets of experiments were carried out using the pulsed column. Five sets were investigated using clarified solutions while, the other three sets were carried out with the addition of different concentrations of spent uranium tailings and gold leach tailings.

The variables studied were the pulse frequency, the pulse amplitude, the aqueous flow rate with and without tailings, the organic flow rate, pH, and the dispersed phase i.e., organic or aqueous phase dispersed. The conditions and results of hold-up for the clarified aqueous phase which have been used in the uranium system are summarized in Tables 4.4, 4.5, 4.6 and 4.7, with their typical graphical results shown after each table. The uranium tailings aqueous phase results are summarized in Tables 4.8 and 4.9, the corresponding graphical results are also shown after each table of results.

The conditions and results of the clarified aqueous phase used in the gold leach system are summarized in Table 4.10. A discussion on the results of the gold leach tailings system was given in this chapter, under "Results of Set VIII".

During this investigation a three level fractional factorial design was chosen in order to limit the number of runs that had to be carried out. This proved to be an advantage in avoiding a severe erosion to the stator and rotor of the Moyno pump when pumping slurry. This design required choosing three spaced values for each operating variable.

A third order model was used for the hold-up as proposed by Bell and Babb [6] and as shown in equation 4.1. Only significant parameters were retained; i.e., parameters whose 95 % confidence intervals does not include zero. The parameter estimate values and their standard deviations for different experimental sets are shown in Table 4.3.

The hold-up profiles predicted by the models for all the series of the experimental results were plotted as a function of the frequency and the aqueous flow or the organic flow at each time separately. This made it possible to study the effect of the dispersed and the continuous phases on the hold-up.

After each table of results for each set there are three plots. The first two show typical plots on the effect of either, the continuous or the dispersed phase flow rate on the hold-up. For each experimental set the operating conditions are shown in its graphical representation; additional plots are shown in Appendix B.

From the figures of the hold-up versus the pulse frequency it can be concluded that the variation of the hold-up due to the variation of the continuous phase flow rate was negligible especially in the case of the solvent in pulp process due to low

Table 4.3: Parameter estimate values of the proposed third order model for the hold-up.

$$\begin{aligned} X_d &= B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_4X_1^2 + B_5X_2^2 + B_6X_3^2 \\ &+B_7X_1X_2 + B_8X_1X_3 + B_9X_2X_3 + B_{10}X_1^2X_2 + B_{11}X_1X_2^2 \end{aligned} \tag{4.1}$$

Set #	I	II	III	IV	V	VI	VII
B_0	-11.08	-62.74	-29.23	7.62	-6.20	3.28	20.55
S.D.	1.62	26.40	2.70	8E-01	1.40	1.90	10.80
B_1		2.13		-0.05	0.05		
S.D.		1.45		5E-02	4.8E-02		
B_2	0.03	0.16	0.08		0.02	-8.1E-02	-0.02
S.D.	3.3E-03	5.9E-02	5E-03		2.7E-03	4.6E-03	1E-02
B_3	0.015	0.03	9.8E-03		-1.35E-03		
S.D.	3.1E-03	1.7E-02	3.9E-03		6.7E-04		
B_4			0.02	7.2E-03	2.7E-03	-2.6E-03	
S.D.			1.1E-03	9.E-04	2.3E-04	9.4E-04	
B_5	-1.4E-05		-3.5E-05		-7.2E-06	8.3E-06	
S.D.	2E-06		2.5E-06		1.3E-06	2.7E-06	
B_6	-1.0E-05	-2.2E-05	-6.1E-06	3.5E-06	1.22E-06		
S.D.	2E-06	1.3E-05	2.7E-06	6E-07	5.8E-07		
B_7	-2.5E-04	-6E-05	-1.9E-03	6E-06	-4E-04	1.26E-04	1.7E-03
S.D.	1.1E-04	2.4E-03	1.4E-04	3.3E-06	9.2E-05	8E-05	8.6E-04
B_8				7.9E-05			
S.D.				2.4E-05			
B_9	-5.5E-06	-7.8E-06					
S.D.	1.8E-06	1.1E-05					
B_{10}	3.4E-06					4.3E-06	
S.D.	1.4E-06					9E-07	
B_{11}	1.34E-07		8.2E-07			-2.2E-07	
S.D.	5.5E-08		7.4E-08			6.97E-08	

"S.D." stands for Standard Deviation.

continuous flow rate used. this was in agreement with the work of Sehmel and Babb [53] and Fernandes [11]. In the emulsion region, it was noticed that when studying clarified solution, the hold-up increased with an increase in the velocity of the continuous phase. The third graph after each table of Set I and Set II showed a typical comparison of the experimental data (observed hold-up % data) and the fitted model results (predicted hold-up % data). Additional plots are given in Appendix B. Results of these comparisons showed that, any discrepancy in the fitted model was not larger than the experimental error.

Tabular and graphical results of Set I

The objective of these experiments were to study the effect of amplitude on the hold-up.

Conditions of Operation of Set I

- Clarified aqueous dispersed phase flow rates 523.4, 787.5 and 1045.8 ml/min.
- Organic continuous phase flow rates 379.3, 579.1 and 778.8 ml/min.
- Pulse frequency 22, 31 and 40 min^{-1}.
- Pulse amplitude 2.55 cm.
- Plate free area 24.4 %.
- Operating pH of aqueous phase 10.06.

Table 4.4: Set I, Hold-up experimental data for clarified aqueous dispersed feed, pH 10.06, pulse amplitude 2.55 cm.

Frequency (X_1) *1/min.*	*Aqueous flow rate* (X_2) *ml/min*	*Organic flow rate* (X_3) *ml/min*	*Hold-up* (X_d) %
22	523.4	579.1	2.73
31	523.4	579.1	2.80
31	523.4	778.8	2.88
40	523.4	579.1	3.42
22	787.5	379.3	4.46
22	787.5	379.3	4.54
22	787.5	778.8	4.10
31	787.5	579.1	5.23
31	787.5	579.1	5.14
31	787.5	579.1	5.05
40	787.5	379.3	5.63
40	787.5	778.8	5.54
22	1045.8	379.3	5.04
22	1045.8	379.3	5.20
22	1045.8	579.1	5.29
31	1045.8	778.8	5.46
31	1045.8	778.8	5.04
40	1045.8	579.1	7.42

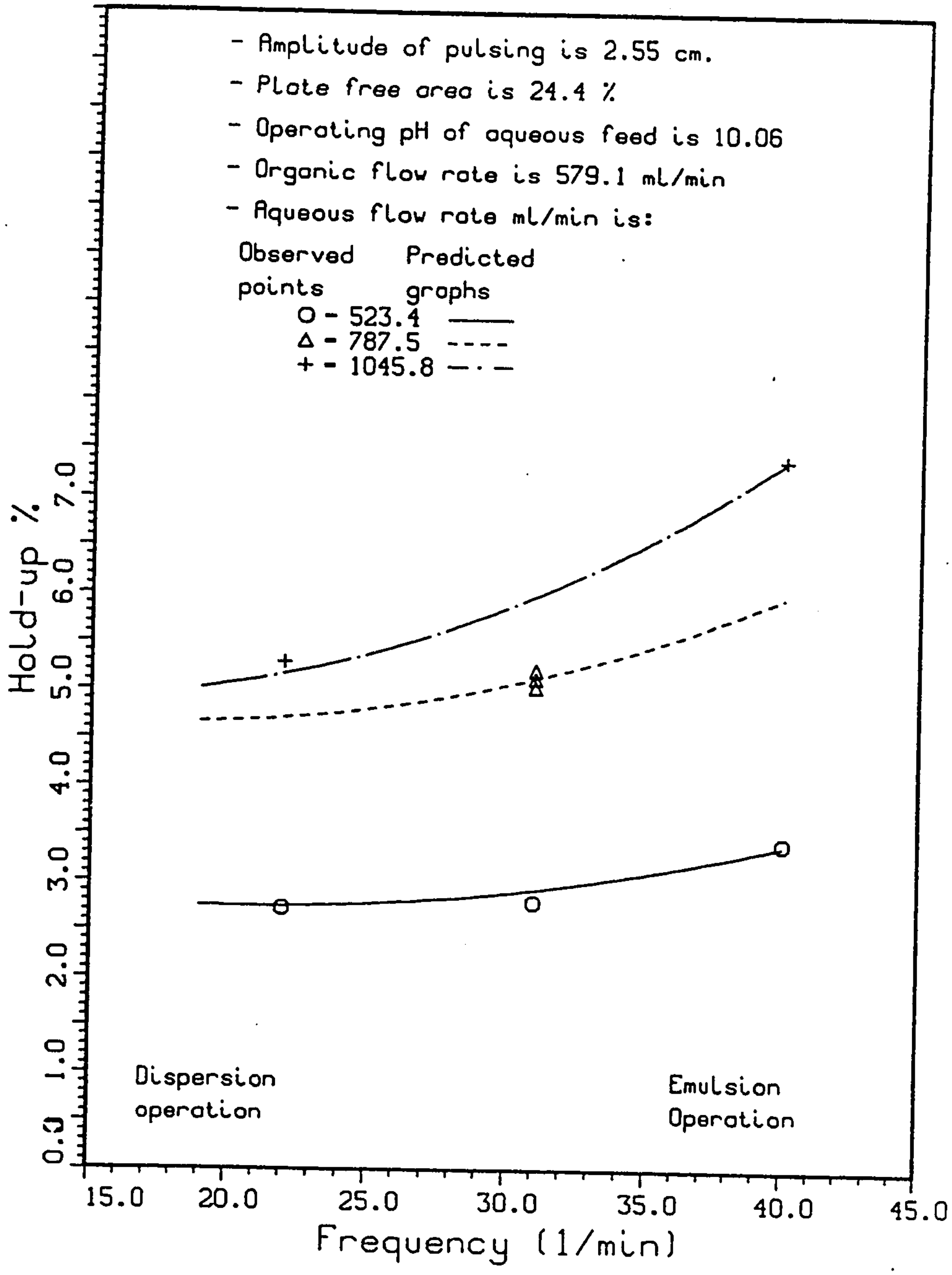


Figure 4.1: Predicted and experimental hold-up % data of Set I, clarified aqueous dispersed phase (variable), organic phase (constant).

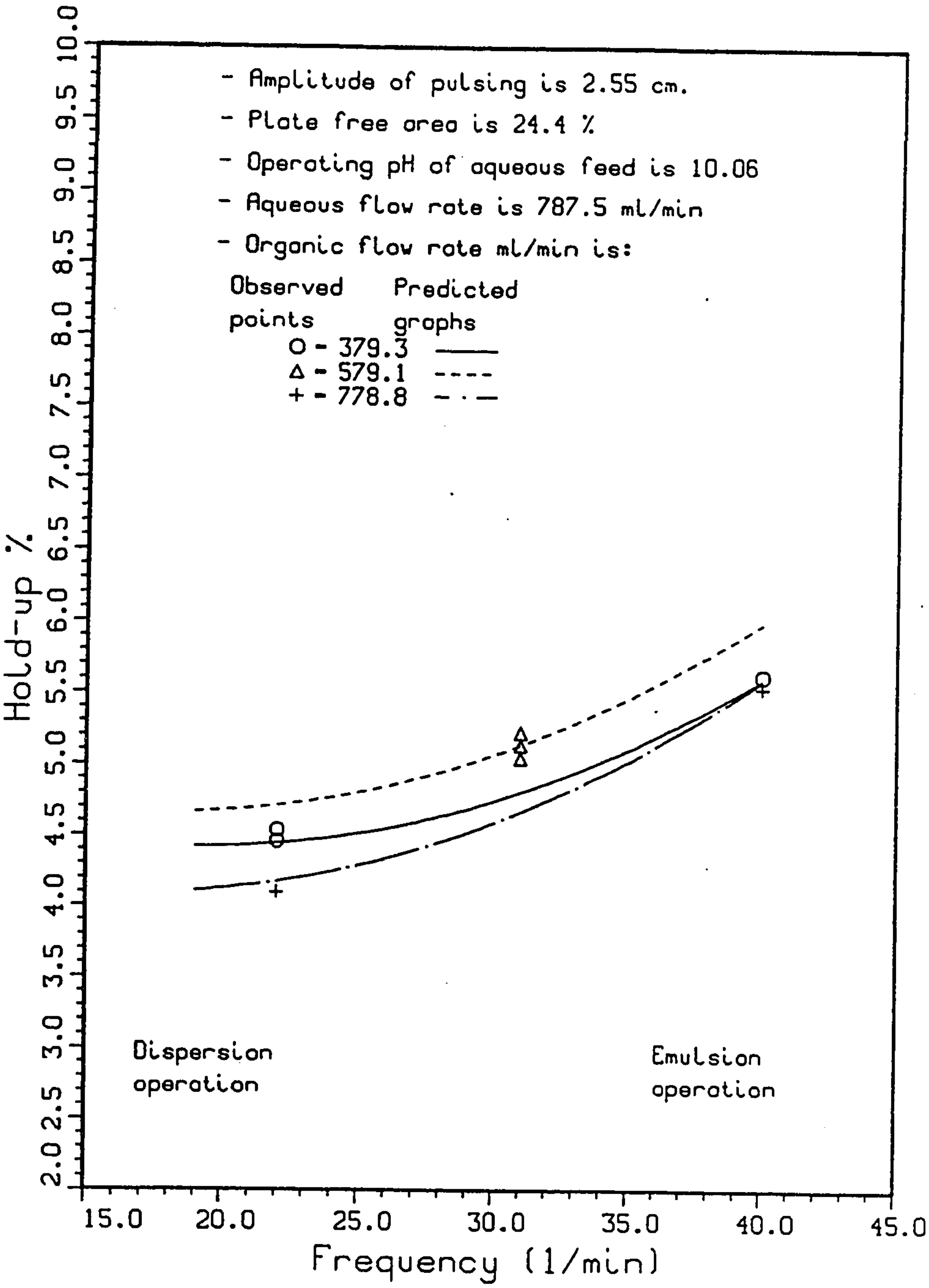


Figure 4.2: Predicted and experimental hold-up % data of Set I, clarified aqueous dispersed phase (constant), organic phase (variable).

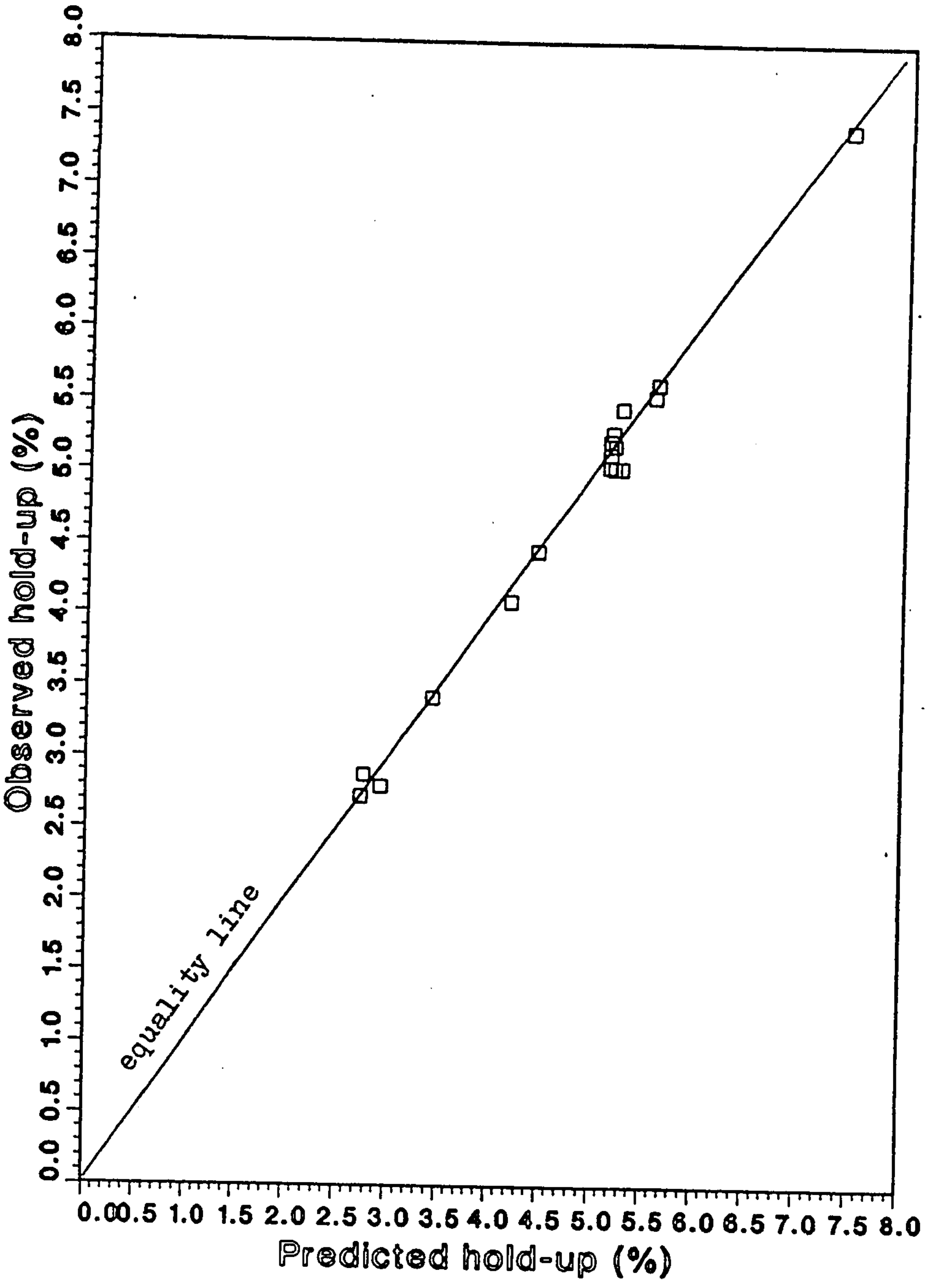


Figure 4.3: Observed versus predicted hold-up % data of Set I.

Tabular and graphical results of Set II

The objective of these experiments were to study,

- The effect of amplitude on the hold-up results, by comparing the hold-up results of Set II and Set I. The operating conditions were the same except that the pulse amplitude was 2.55 cm in Set I, compared to 0.75 cm in Set II.
- The effect of aqueous phase pH (10.06) on hold-up compared to the hold-up data of D. Koren [26] Set II (pH 4.04).

Conditions of Operation of Set II

- Clarified aqueous dispersed phase flow rates 523.4, 787.5 and 1045.8 ml/min.
- Organic continuous phase flow rates 379.3, 579.1 and 778.8 ml/min.
- Pulse frequency 22, 27, 31 and 40 min^{-1}.
- Pulse amplitude 0.75 cm.
- Plate free area 24.4 %.
- Operating pH of the aqueous phase 10.06.

Table 4.5: Set II, Hold-up experimental data for clarified aqueous dispersed feed, pH 10.06, pulse amplitude 0.75 cm.

Frequency (X_1) *1/min.*	*Aqueous flow rate* (X_2) *ml/min*	*Organic flow rate* (X_3) *ml/min*	*Hold-up* (X_d) %
19	523.4	579.1	5.73
19	523.4	579.1	5.52
22	523.4	579.1	6.38
22	523.4	579.1	6.44
31	523.4	579.1	2.80
31	523.4	379.3	2.64
31	523.4	579.1	2.80
31	523.4	778.8	2.64
40	523.4	579.1	2.80
43	523.4	579.1	2.80
22	787.5	379.3	9.38
22	787.5	778.8	9.38
27	787.5	379.3	8.27
27	787.5	579.1	8.27
27	787.5	778.8	9.38
31	787.5	379.3	5.16
31	787.5	579.1	5.16
31	787.5	579.1	5.08
31	787.5	579.1	5.08
40	787.5	379.3	2.89
40	787.5	579.1	5.08
40	787.5	778.8	2.89
27	1045.8	579.1	11.89
27	1045.8	778.8	6.76
31	1045.8	379.3	6.76
31	1045.8	579.1	6.76
31	1045.8	778.8	6.76
40	1045.8	579.1	6.76

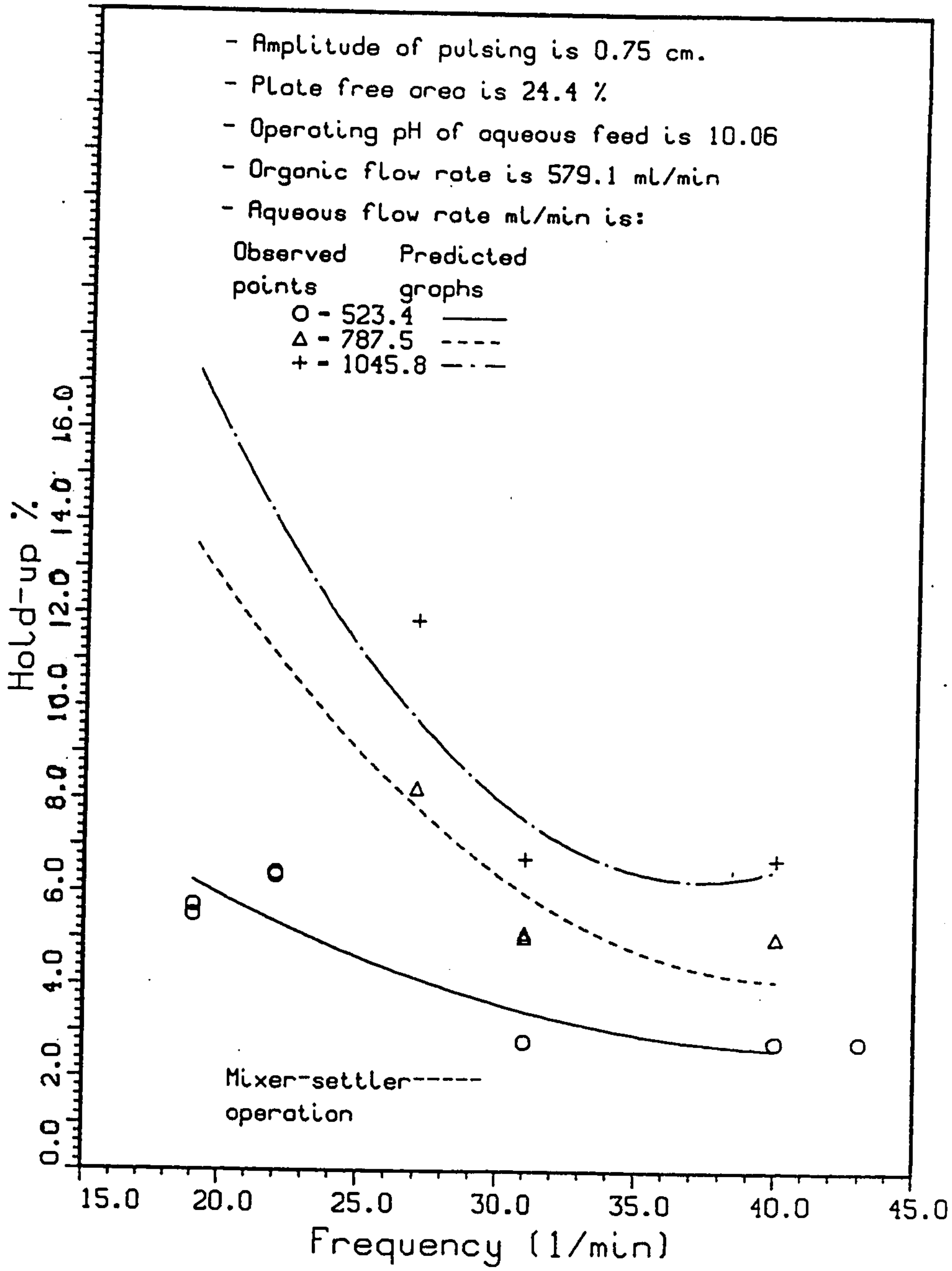


Figure 4.4: Predicted and experimental hold-up % data of Set II, clarified aqueous dispersed phase (variable), organic phase (constant).

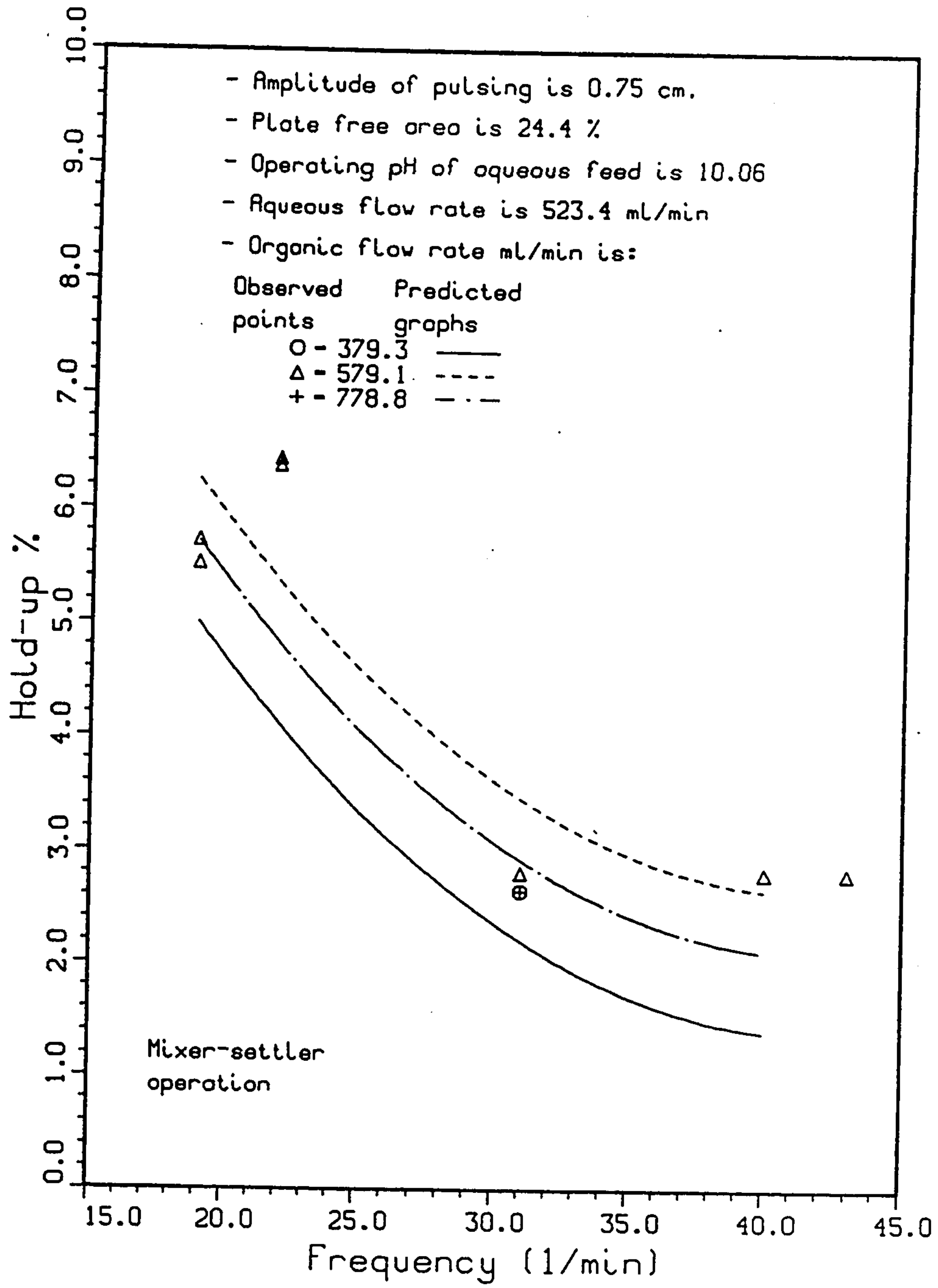


Figure 4.5: Predicted and experimental hold-up % data of Set II, clarified aqueous dispersed phase (constant), organic phase (variable).

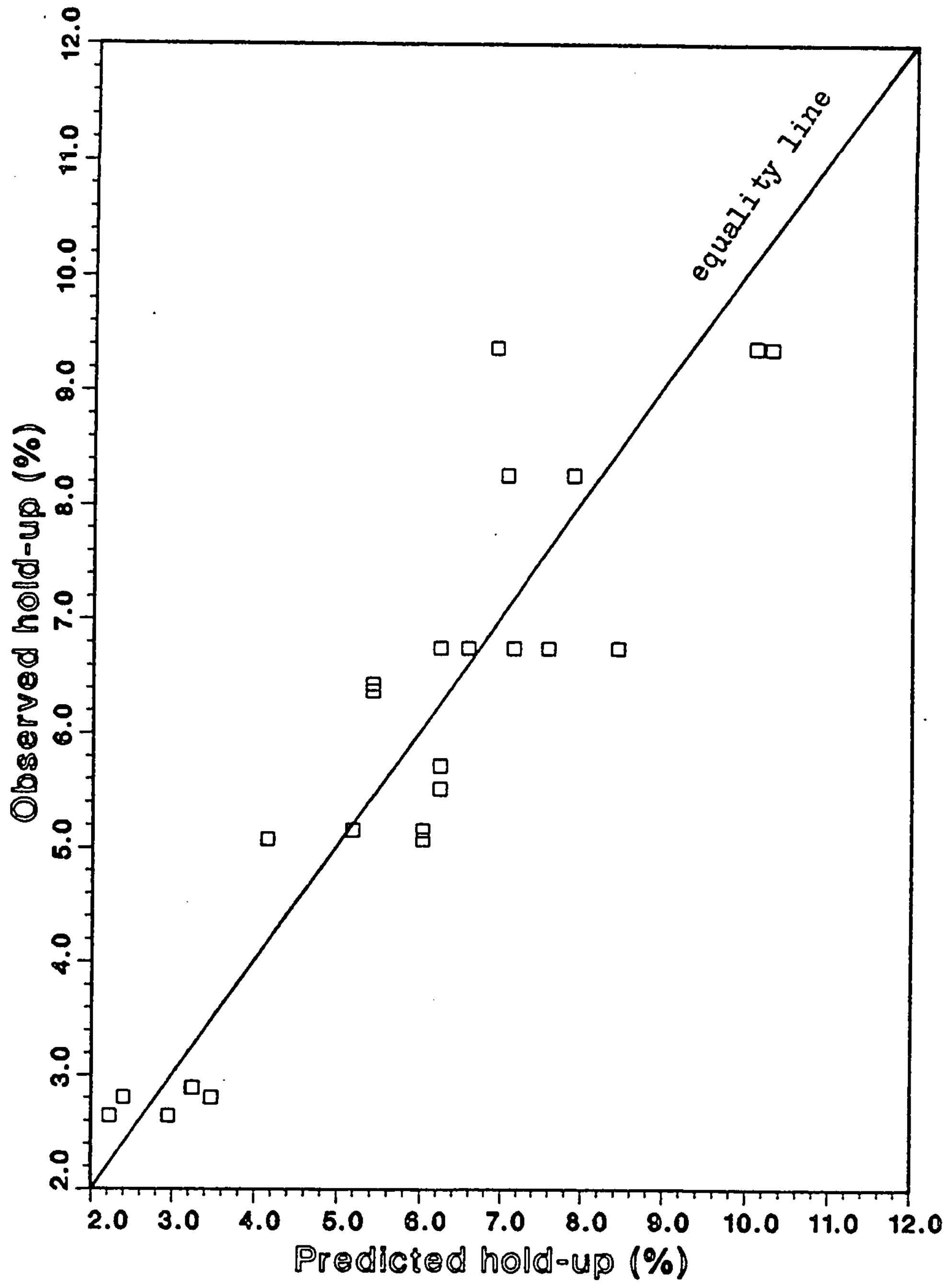


Figure 4.6: Observed versus predicted hold-up % data of Set II.

Tabular and graphical results of Set III

Two effects on the hold-up results were investigated in these experiments, they were the following :

1. The effect of an increase in the dispersed phase flow rate, by comparing the hold-up results of this set with the hold-up results of Set I, (Set I, clarified aqueous flow rates were 523.4, 787.5 and 1045.8 ml/min).

2. The effect of aqueous phase pH on hold-up by comparing results of this set of pH 10.06, with the hold-up results of D. Koren [26], Set III (pH 4.04).

Conditions of Operation of Set III

- Clarified aqueous dispersed phase flow rates 792, 1049.6 and 1303.3 ml/min.
- Organic continuous phase flow rates 379, 579.1 and 778.8 ml/min.
- Pulse frequency 19, 22, 31 and 40 min^{-1}.
- Pulse amplitude 2.55 cm.
- Plate free area 24.4 %.
- Operating pH of the aqueous phase 10.06.

Table 4.6: Set III, Hold-up experimental data for clarified aqueous dispersed feed, pH 10.06, pulse amplitude 2.55 cm.

Frequency (X_1) *1/min.*	*Aqueous flow rate* (X_2) *ml/min*	*Organic flow rate* (X_3) *ml/min*	*Hold-up* (X_d) %
19	792.0	579.1	6.10
22	792.0	579.1	5.90
31	792.0	379.0	4.67
31	792.0	379.0	4.96
31	792.0	579.1	5.10
31	792.0	579.1	5.49
31	792.0	778.8	5.10
31	792.0	778.8	5.50
40	792.0	579.1	8.31
19	1049.6	379.0	8.40
19	1049.6	778.8	8.97
22	1049.6	379.0	8.31
22	1049.6	778.8	8.52
31	1049.6	579.1	7.04
31	1049.6	579.1	7.10
31	1049.6	579.1	7.06
40	1049.6	379.0	8.64
40	1049.6	778.8	8.81
19	1303.3	579.1	9.65
22	1303.3	579.1	8.97
22	1303.3	579.1	8.48
31	1303.3	379.0	7.30
31	1303.3	379.0	7.42
31	1303.3	579.1	7.42
31	1303.3	778.8	7.46
40	1303.3	579.1	9.63

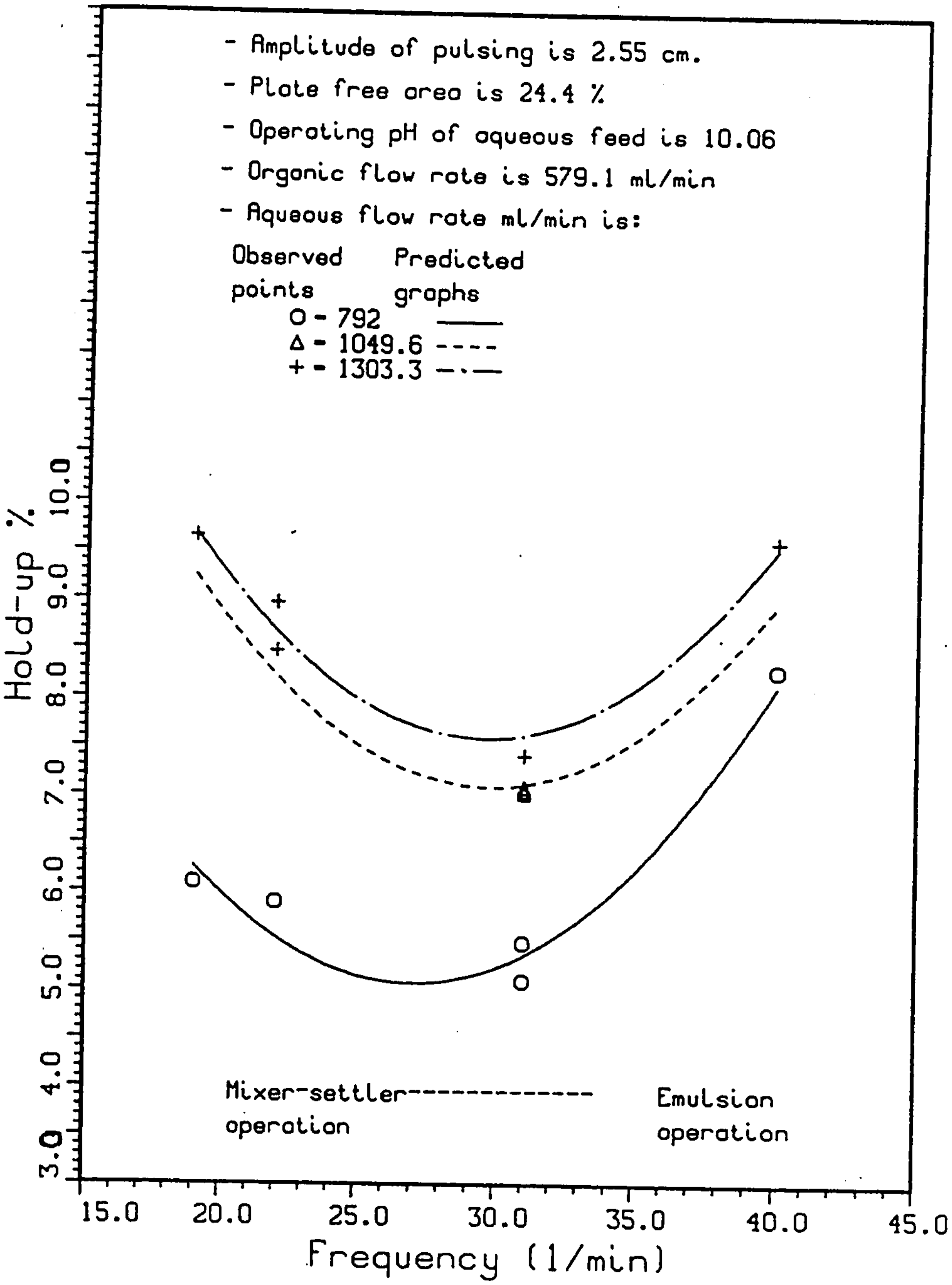


Figure 4.7: Predicted and experimental hold-up % data of Set III, clarified aqueous dispersed phase (variable), organic phase (constant).

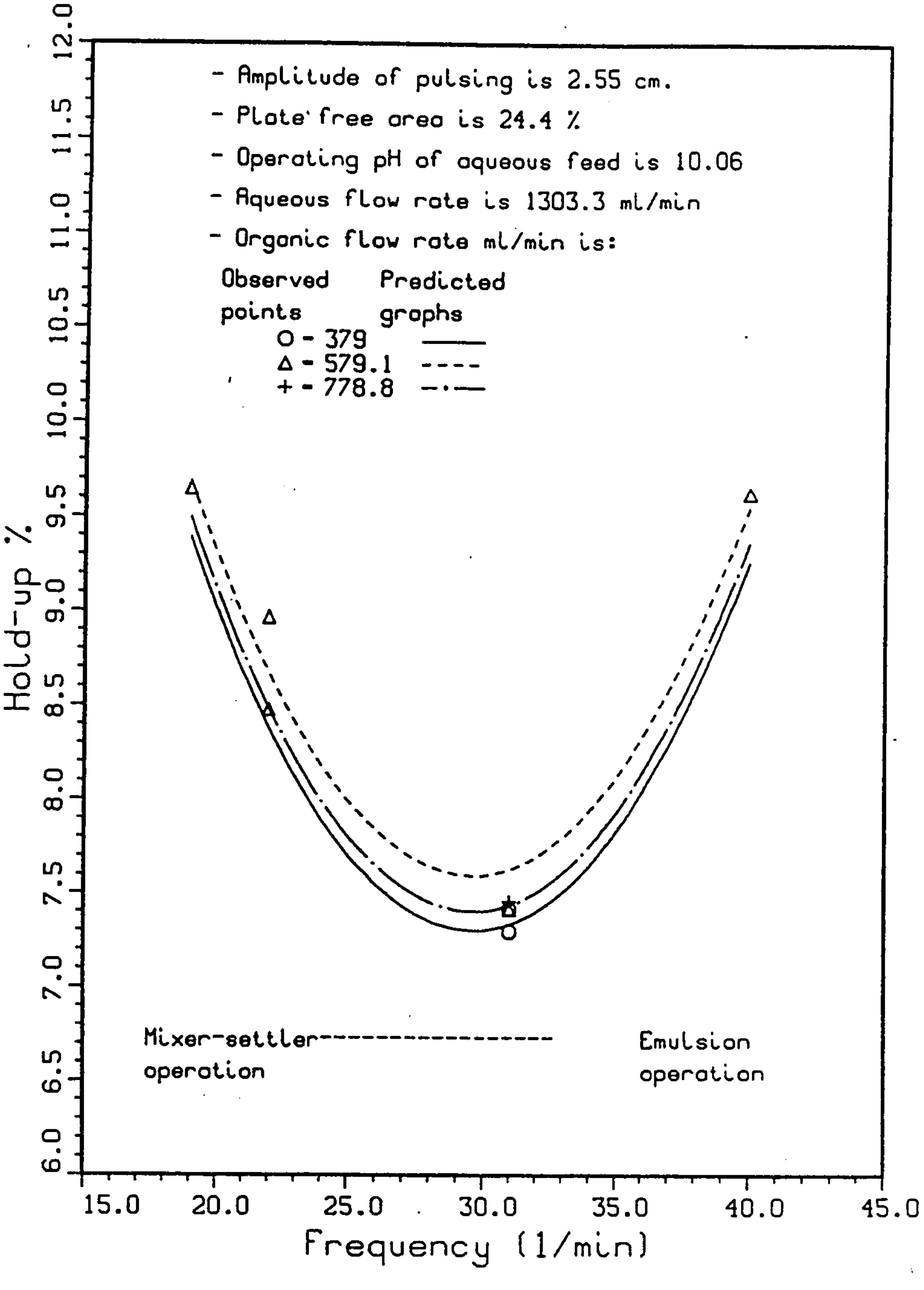


Figure 4.8: Predicted and experimental hold-up % data of Set III, clarified aqueous dispersed phase (constant), organic phase (variable).

Tabular and graphical results of Set IV

The objective of this experiment was to study the effect of the dispersed phase on the hold-up. i.e. organic dispersed compared to aqueous dispersed as in Set III.

Conditions of Operation of Set IV

- Organic dispersed phase flow rates 350, 600 and 850 ml/min.
- Clarified aqueous continuous phase flow rates 1000, 1500 and 2000 ml/min.
- Pulse frequency 19, 22, 31 and 40 min^{-1}.
- Pulse amplitude 2.55 cm.
- Plate free area 24.4 %.
- Operating pH of the aqueous phase 10.06.

Table 4.7: Set IV, Hold-up experimental data, clarified aqueous continuous phase pH 10.06, organic dispersed phase, pulse amplitude 2.55 cm.

Frequency (X_1) *1/min.*	*Aqueous flow rate* (X_2) *ml/min*	*Organic flow rate* (X_3) *ml/min*	*Hold-up* (X_d) %
19	1000	350	2.64
19	1000	600	3.70
19	1000	850	5.38
22	1000	350	2.02
22	1000	600	3.13
22	1000	850	5.25
31	1000	350	2.02
31	1000	600	3.20
31	1000	600	2.86
31	1000	850	4.55
31	1000	850	5.31
40	1000	350	2.23
40	1000	600	3.81
40	1000	850	5.80
22	1500	350	2.02
22	1500	600	3.23
22	1500	850	5.31
31	1500	350	2.25
31	1500	600	3.20
31	1500	600	3.20

Table 4.7, Set IV continued

Frequency (X_1) *1/min.*	*Aqueous flow rate* (X_2) *ml/min*	*Organic flow rate* (X_3) *ml/min*	*Hold-up* (X_d) %
31	1500	600	3.27
31	1500	850	4.55
40	1500	350	2.23
40	1500	600	4.55
40	1500	850	6.20
19	2000	350	2.64
19	2000	600	3.70
19	2000	850	5.45
22	2000	350	2.03
22	2000	600	3.34
22	2000	850	5.44
22	2000	850	5.45
31	2000	350	2.09
31	2000	600	3.54
31	2000	850	5.31
31	2000	850	4.55
40	2000	350	2.30
40	2000	600	4.08
40	2000	600	4.55
40	2000	850	6.40

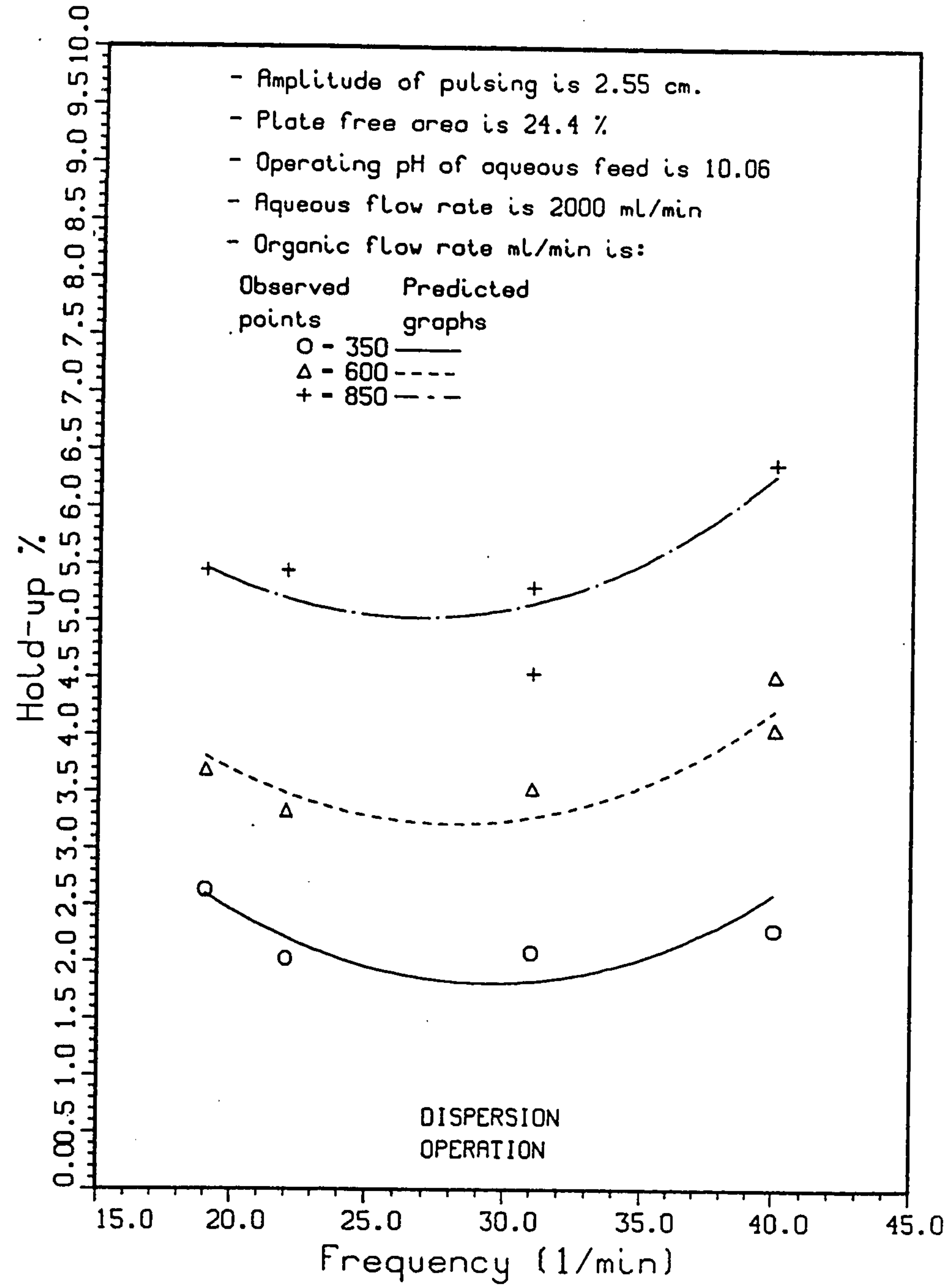


Figure 4.9: Predicted and experimental hold-up % data of Set IV, clarified aqueous continuous phase (constant), organic dispersed phase (variable).

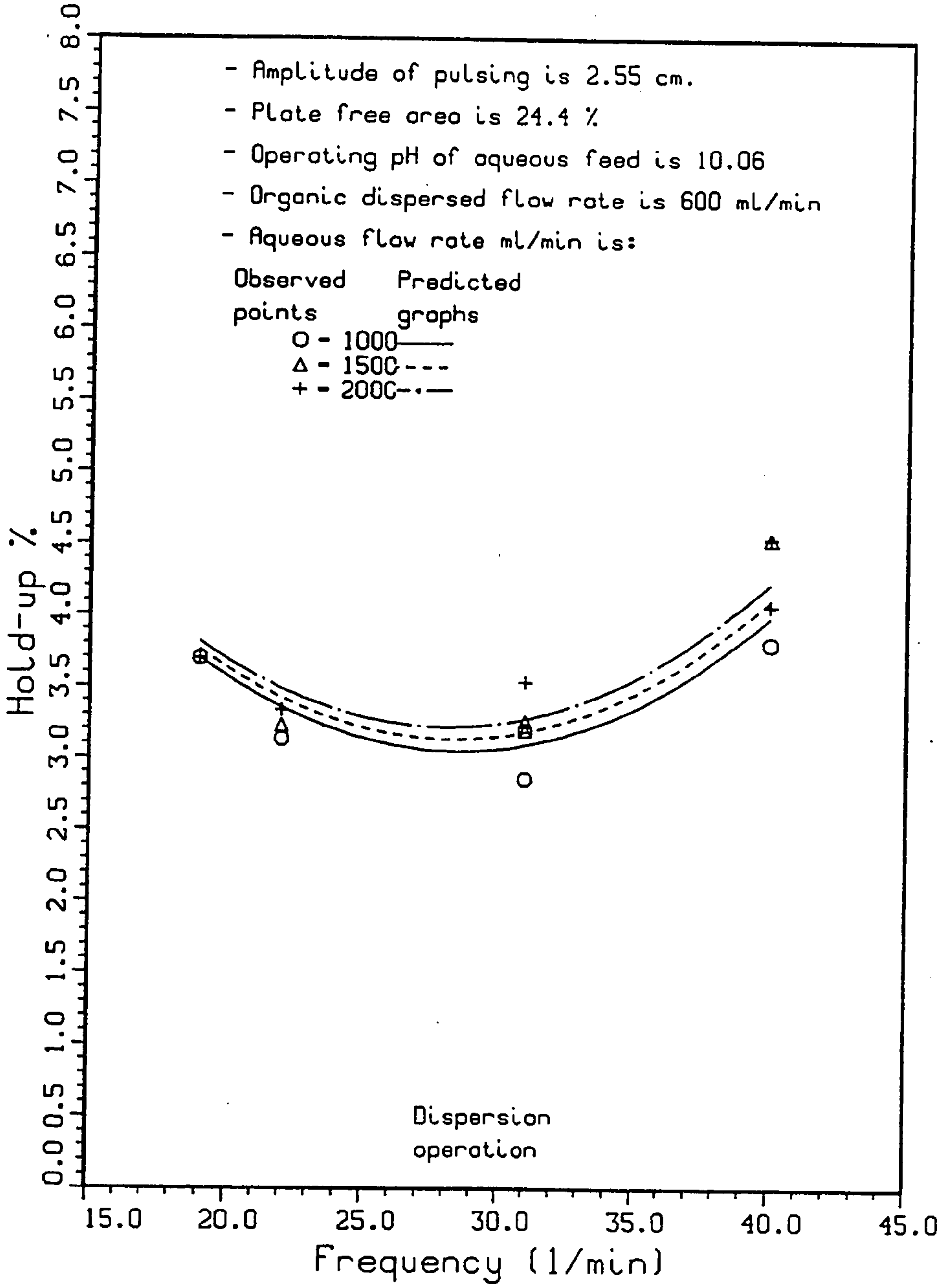


Figure 4.10: Predicted and experimental hold-up % data of Set IV, clarified aqueous continuous phase (variable), organic dispersed phase (constant).

Tabular and graphical results of Set V

The objective of these experiments were to study the following,

1. The effect of the solvent in pulp processing 10 % uranium tailings on the hold-up.
2. The effect of tailings density and percentage of solids on hold-up results. Hold-up data for uranium tailings of 3450 kg/m^3 density were compared with the hold-up results obtained by D. Koren [26] using silica sand, 2850 kg/m^3 density.

Conditions of Operation of Set V

- 10 % uranium tailings aqueous dispersed phase flow rates 792, 1049.6 and 1303.3 ml/min.
- Organic continuous phase flow rates 379, 579.1 and 778.8 ml/min.
- Pulse frequency 19, 22, 31 and 40 min^{-1}.
- Pulse amplitude 2.55 cm.
- Plate free area 24.4 %.
- Operating pH of the aqueous dispersed phase 4.04.

Table 4.8: Set V, hold-up experimental data for 10 % by weight uranium tailings dispersed phase pH 4.04, pulse amplitude 2.55 cm.

Frequency (X_1) *1/min.*	*Aqueous flow rate* (X_2) *ml/min*	*Organic flow rate* (X_3) *ml/min*	*Hold-up* (X_d) %
19	792	579.1	2.40
22	792	579.1	2.32
31	792	379.0	2.23
31	792	579.1	2.23
31	792	778.8	2.23
40	792	579.1	2.49
19	1049.6	579.1	3.74
19	1049.6	579.1	3.65
22	1049.6	379.0	3.64
22	1049.6	579.1	3.65
22	1049.6	778.8	3.65
31	1049.6	579.1	3.27
31	1049.6	579.1	3.27
31	1049.6	579.1	3.27
40	1049.6	379.0	3.50
40	1049.6	579	3.55
40	1049.6	778.8	3.58
19	1303.3	579.1	4.53
22	1303.3	579.1	4.45
31	1303.3	379.0	4.26
31	1303.3	579.1	4.27
31	1303.3	778.8	4.28
40	1303.3	579.1	4.53

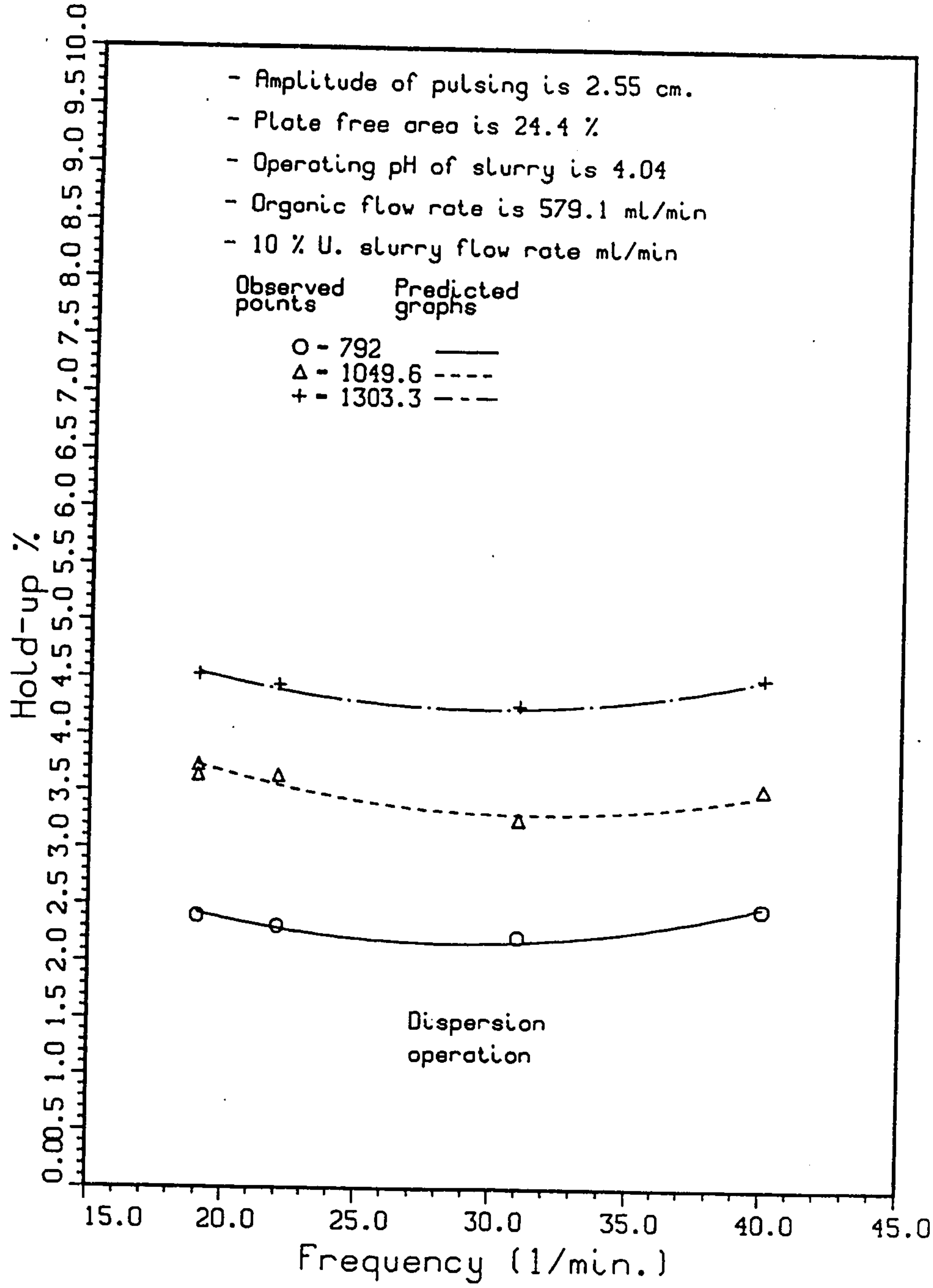


Figure 4.11: Predicted and experimental hold-up % data of Set V, 10 % uranium tailings dispersed phase (variable), organic continuous phase (constant).

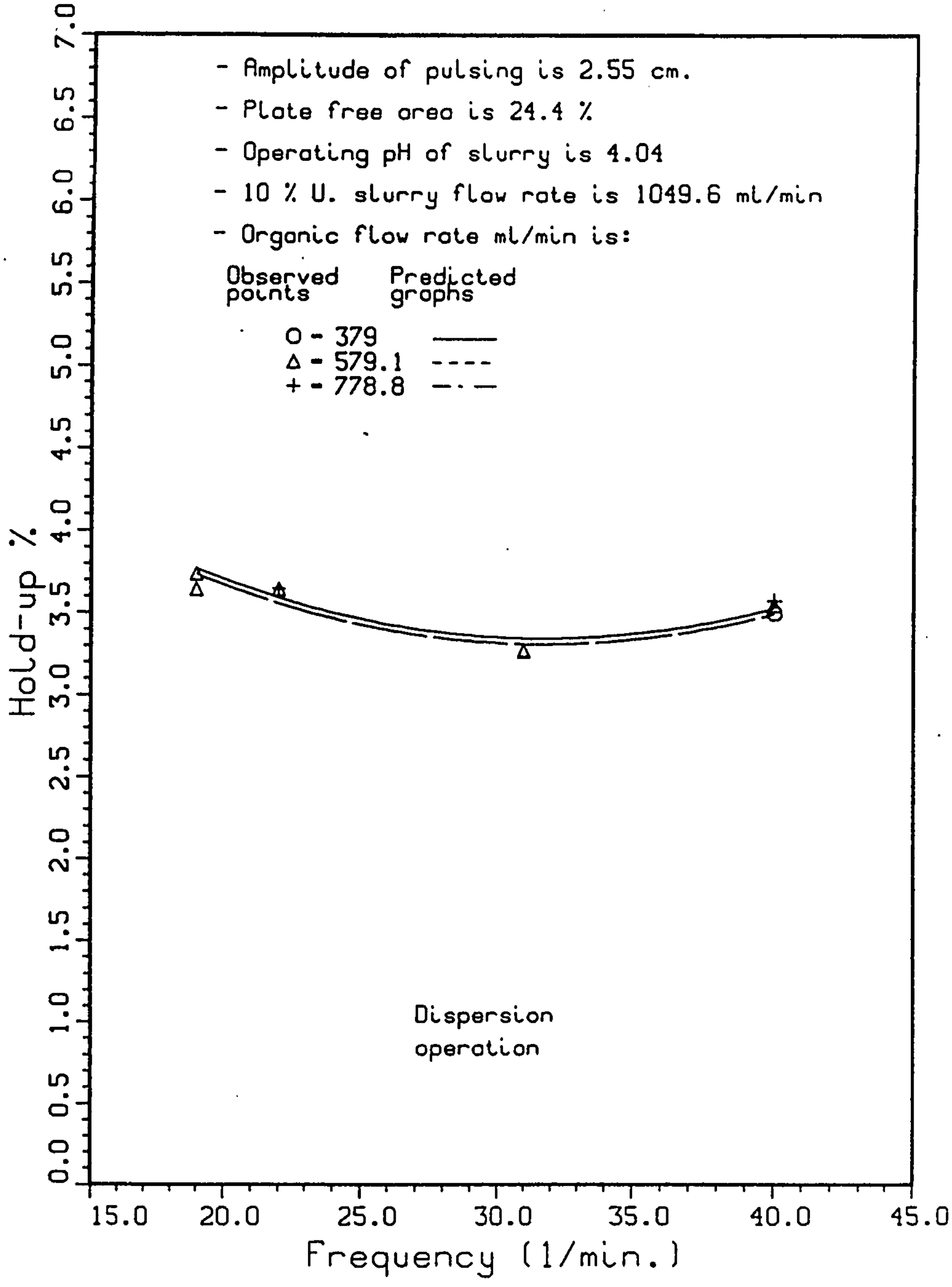


Figure 4.12: Predicted and experimental hold-up % data of Set V, 10 % uranium tailings dispersed phase (constant), organic continuous phase (variable).

Tabular and graphical results of Set VI

These runs were carried out to determine the effect of 25 % uranium tailings on hold-up in solvent in pulp processing.

Conditions of operation of set VI

- 25 % uranium tailings aqueous dispersed phase flow rates 792, 1049.6 and 1303.3 ml/min.
- Organic continuous phase flow rates 379, 579.1 and 778.8 ml/min.
- Pulse frequency 19, 22, 31, 40, 51 and 60 min^{-1}.
- Pulse amplitude 2.55 cm.
- Plate free area 24.4 %.
- Operating pH of the aqueous dispersed phase 4.04.

Table 4.9: Set VI, hold-up experimental data for 25 % by weight uranium tailings dispersed phase pH 4.04, pulse amplitude 2.55 cm

Frequency (X_1) *1/min.*	*Aqueous flow rate* (X_2) *ml/min*	*Organic flow rate* (X_3) *ml/min*	*Hold-up* (X_d) %
19	792.0	579.1	1.15
22	792.0	579.1	1.12
31	792.0	379	1.03
31	792.0	579.1	1.03
31	792.0	778.8	1.03
40	792.0	379.0	1.44
40	792.0	579.1	1.44
40	792.0	778.8	1.44
51	792.0	579.1	1.51
60	792.0	579.1	2.27
19	1049.6	579.1	2.00
22	1049.6	379	1.74
22	1049.6	579.1	1.74
22	1049.6	778.8	1.74
31	1049.6	579.1	1.49

Table 4.9, Set VI continued

Frequency (X_1) *1/min.*	*Aqueous flow rate* (X_2) *ml/min*	*Organic flow rate* (X_3) *ml/min*	*Hold-up* (X_d) %
31	1049.6	579.1	1.45
31	1049.6	579.1	1.40
40	1049.6	379.0	1.91
51	1049.6	579.1	3.04
51	1049.6	579.1	2.99
60	1049.6	579.1	3.34
19	1303.3	579.1	2.95
22	1303.3	579.1	2.72
31	1303.3	379.0	2.50
31	1303.3	579.1	2.50
31	1303.3	778.8	2.50
40	1303.3	579.1	2.65
43	1303.3	579.1	2.65
51	1303.3	579.1	2.65
60	1303.3	579.1	4.62

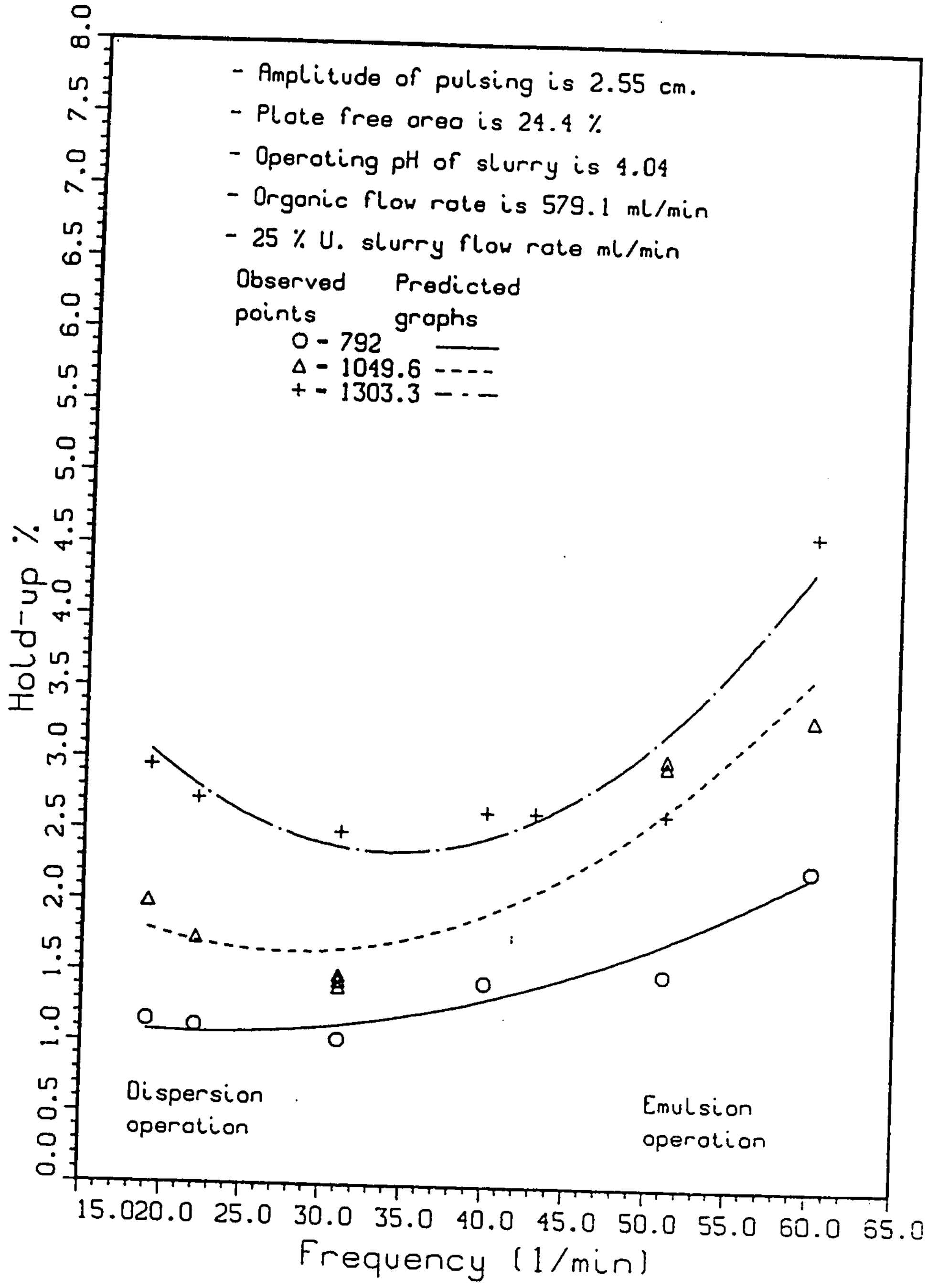


Figure 4.13: Predicted and experimental hold-up % data of Set VI, 25 % uranium tailings dispersed phase (variable), organic continuous phase (constant).

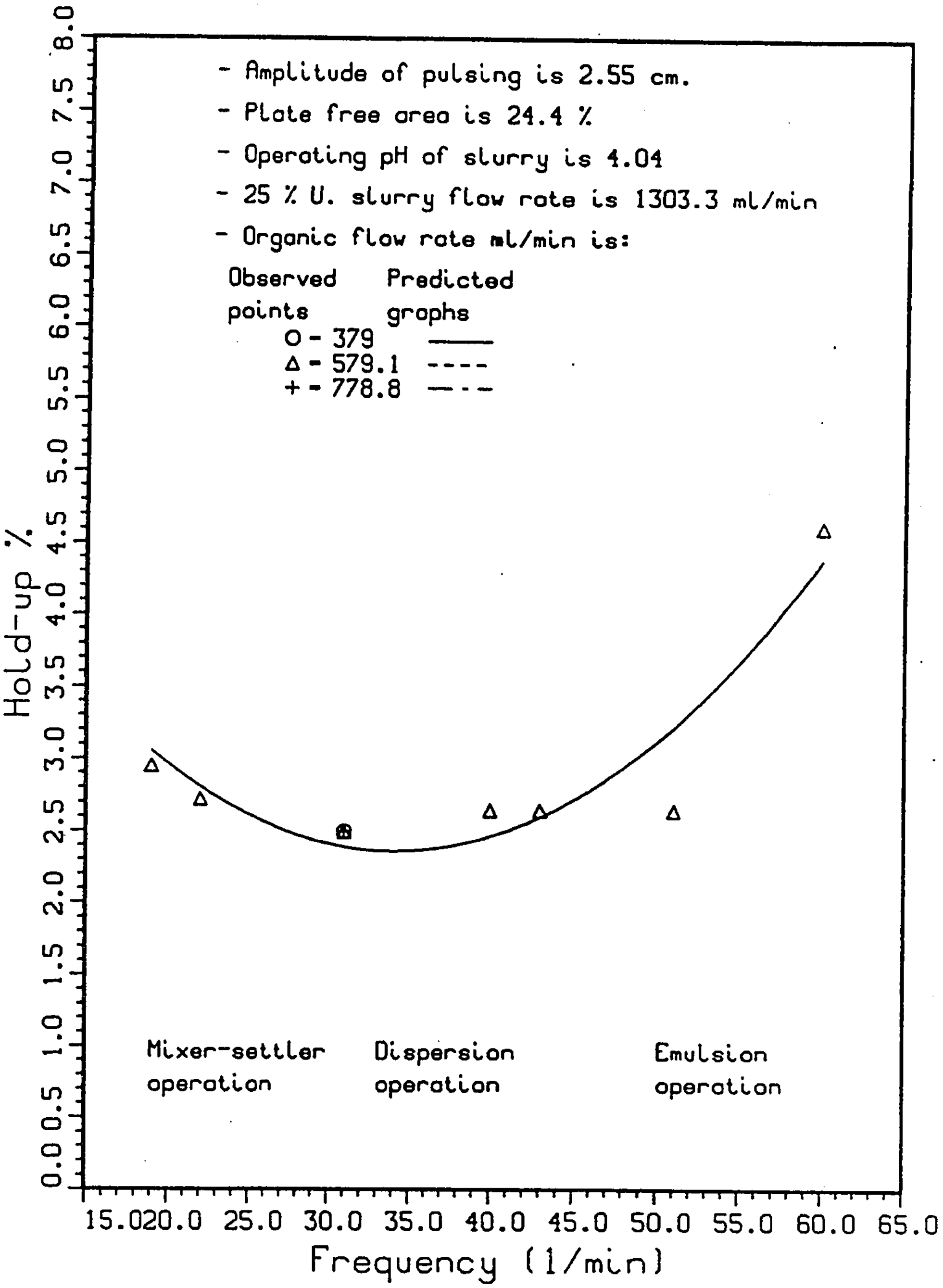


Figure 4.14: Predicted and experimental hold-up % data of Set VI, 25 % uranium tailings dispersed phase (constant), organic continuous phase (variable).

Tabular and graphical results of Set VII

These runs were carried out to obtain hold-up data for clarified solution extraction of gold. i.e, extractant phase was 20 % xylene and 80 % tributyl-phosphate; as well as to study the applicability of McAllister's equation 2.22 of flooding on the flooding conditions obtained in this set of experiments.

Conditions of Operation of Set VII

- Organic dispersed phase flow rates 379, 579.1 and 778.8 ml/min.
- Clarified aqueous continuous phase flow rates 792, 1049.6 and 1303.3 ml/min.
- Pulse frequency 19, 22, 27, 31, and 40 min^{-1}.
- Pulse amplitude 2.55 cm.
- Plate free area 12.4 %.
- Operating pH of the aqueous continuous phase 10.1.

No crud formation was noticed upon using the clarified aqueous phase with no surfactant. The continuous and the dispersed phases velocities at flooding conditions were in agreement with McAllister's equation.

Table 4.10: Set VII, hold-up experimental data for organic dispersed phase and clarified aqueous continuous phase of pH 10.1, pulse amplitude 2.55 cm

Frequency (X_1) *1/min.*	*Aqueous flow rate* (X_2) *ml/min*	*Organic flow rate* (X_3) *ml/min*	*Hold-up* (X_d) %
19	792.0	579.1	4.85
22	792.0	579.1	4.85
27	792.0	579.1	5.00
31	792.0	379.0	4.13
31	792.0	579.1	5.57
31	792.0	579.1	7.63
31	792.0	579.1	7.39
31	792.0	778.8	Flooding
40	792.0	579.1	Flooding
19	1049.6	379.3	2.16
19	1049.6	579.1	4.85
19	1049.6	579.1	4.33
19	1049.6	579.1	4.85
19	1049.6	579.1	4.70
19	1049.6	778.8	4.96
22	1049.6	379.0	2.16
22	1049.6	579.1	4.85
22	1049.6	579.1	4.33
22	1049.6	778.8	4.96
27	1049.6	778.8	5.66
27	1049.6	579.1	4.96
27	1049.6	579.1	4.75
31	1049.6	379.3	4.13
31	1049.6	579.1	5.46

Table 4.10, Set VII continued

Frequency (X_1) *1/min.*	*Aqueous flow rate* (X_2) *ml/min*	*Organic flow rate* (X_3) *ml/min*	*Hold-up* (X_d) %
31	1049.6	579.1	6.50
31	1049.6	579.1	5.54
31	1049.6	579.1	6.87
31	1049.6	579.1	6.10
31	1049.6	579.1	5.57
31	1049.6	579.1	5.98
31	1049.6	778.8	Flooding
40	1049.6	379.0	Flooding
40	1049.6	579.1	Flooding
40	1049.6	778.8	Flooding
19	1303.3	379.0	1.65
19	1303.3	379.0	1.36
19	1303.3	579.1	4.85
22	1303.3	379.0	3.11
22	1303.3	579.1	4.85
27	1303.3	379.0	3.46
31	1303.3	379.0	4.13
31	1303.3	379.0	4.50
31	1303.3	579.1	5.57
31	1303.3	579.1	6.140
31	1303.3	778.8	Flooding
40	1303.3	579.1	Flooding

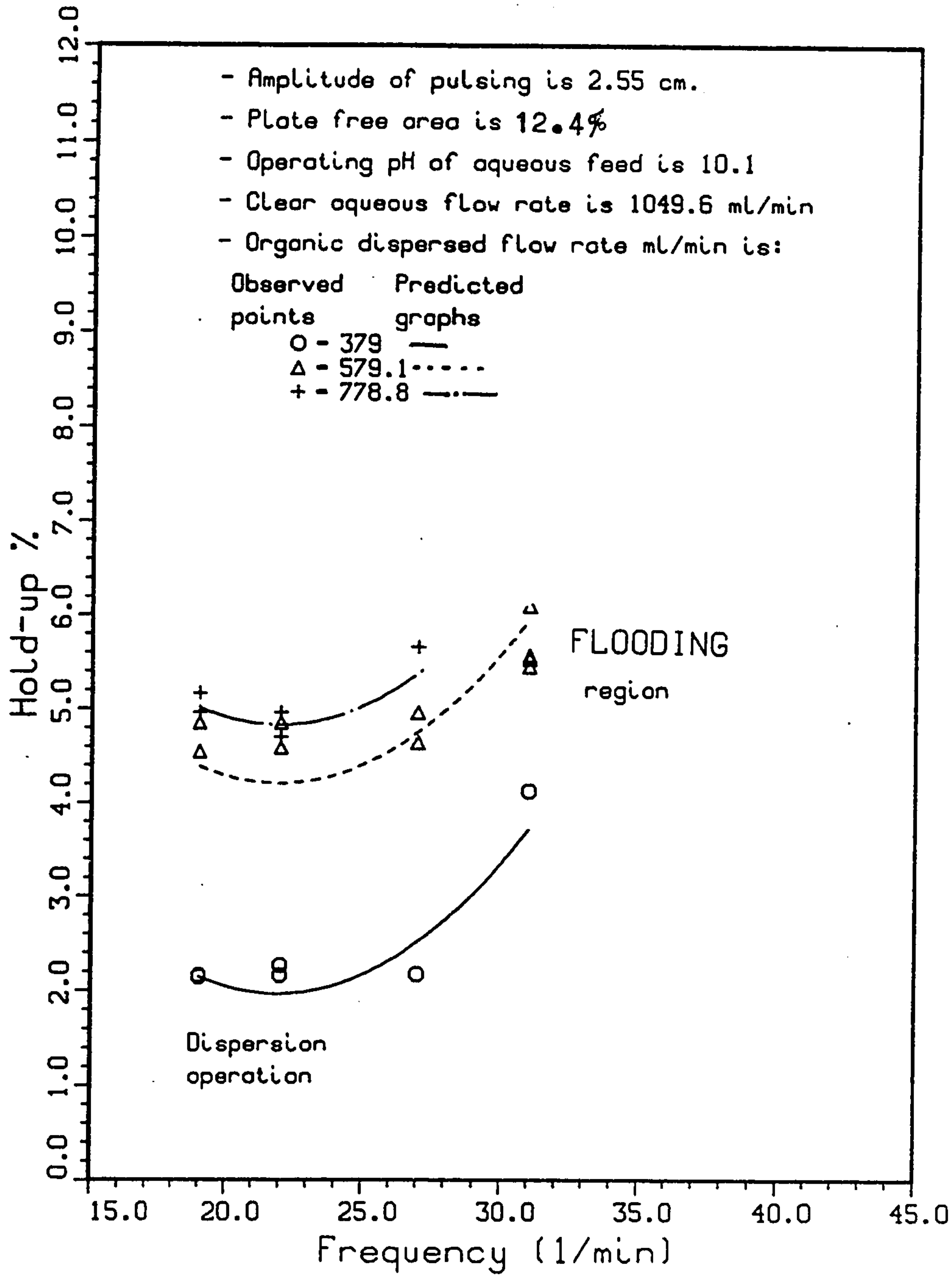


Figure 4.15: Predicted and experimental hold-up % data of Set VII, organic dispersed phase (variable), clarified aqueous continuous phase (constant).

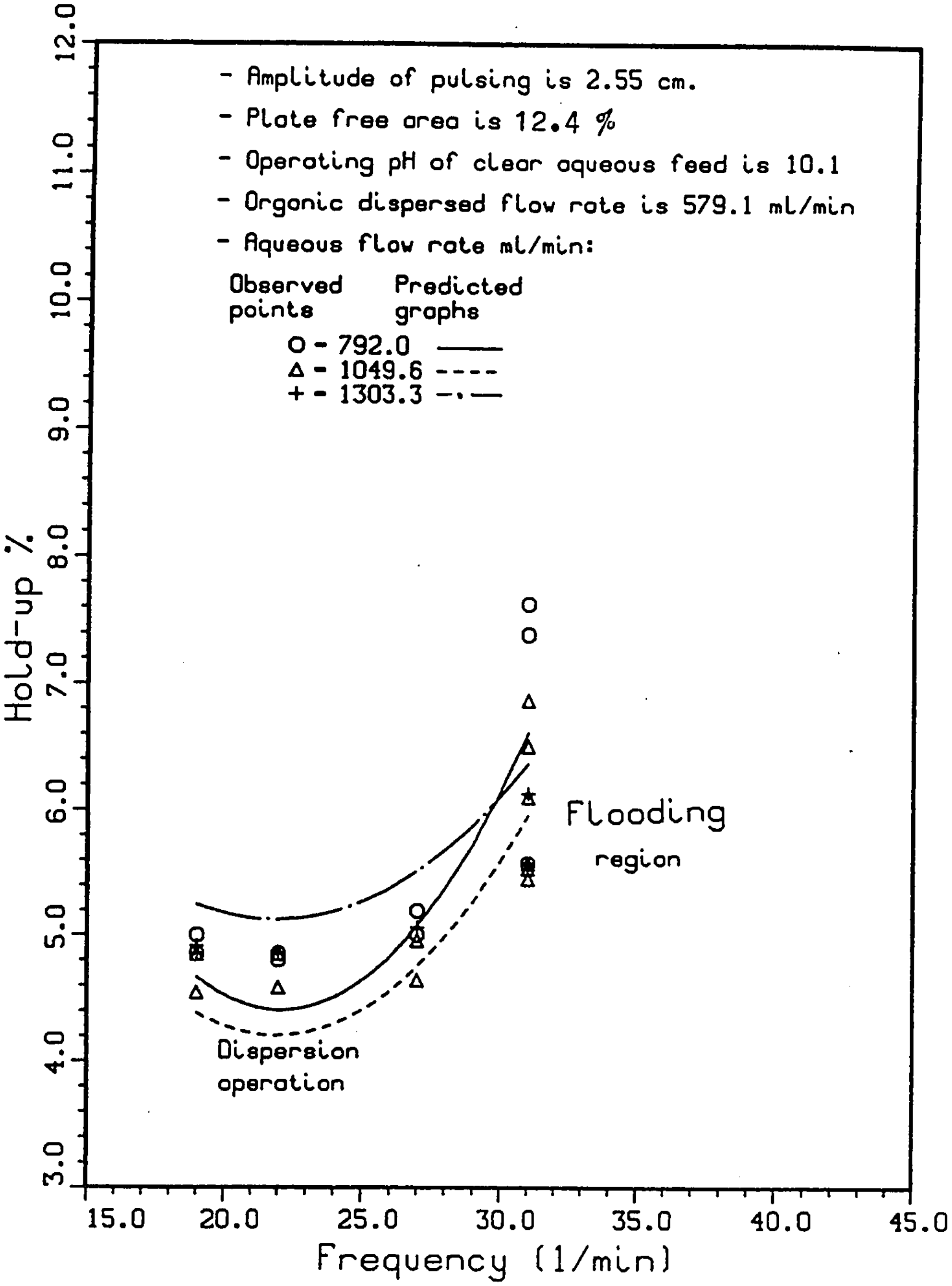


Figure 4.16: Predicted and experimental hold-up % data of Set VII, organic dispersed phase (constant), clarified aqueous continuous phase (variable).

Results of Set VIII

Experimental runs were performed using gold leach tailings (10 % by weight.). The objective was to collect data on hold-up and to investigate its effect on pulsed sieve plate column behavior.

- Conditions of Operation of Set VIII

- Organic dispersed phase flow rates 379, 579.1 and 778.8 ml/min.
- 10 % gold leach tailings aqueous continuous phase flow rates 792, 1049.6 and 1303.3 ml/min.
- Above conditions were reversed, where the aqueous phase was dispersed and the organic phase was continuous.
- Pulse frequency 19, 22, 31 and 40 min^{-1}.
- Pulse amplitude 1 cm.
- Plate free area 12.4 %.
- Operating pH of aqueous phase 10.1.

Results showed that, even with addition of surfactant (HDTMAB) .crud was formed even at the limit low pulse frequency (19 min^{-1}) and low pulse amplitude (1 cm) that the equipment used can deliver. Once stable crud formation took place, it was impossible to carry out solvent in pulp processing which lead to complete column flooding. This happened despite that the bench scale tests indicated that it was feasible as suggested by Mooiman and Miller [40]. The major problem was how to distinguish the interface when operating the pulsed column while, using the designated chemicals according to the bench scale tests and as recommended by Mooiman and Miller [40].

Chapter 5

Discussion of the Results

The pulsed column was found to operate under three different regimes as the pulse frequency and throughput rates were varied. The mixer-settler regime where the dispersed phase hold-up decreased with an increase in the pulse frequency. The transition regime where the hold-up was constant and in the emulsion regime, hold-up increased with an increase in frequency, a further increase in pulse frequency, increased rapidly the hold-up and lead to column flooding.

The hold-up profiles showed that for clarified solutions, hold-up exhibited a minimum at the same frequency value The hold-up profiles showed that on addition of uranium tailings, the transition frequency was increased compared to clarified solution operation. This was in agreement with the work of Joe et al [23].

- Effect of the Process Variables:

The experimental results were examined to determine the effect of the following variables on column behavior,

1. pulse amplitude

2. dispersed phase flow rate

3. continuous phase flow rate

4. pH

5. effect of dispersed phase kind, (aqueous or organic dispersed).

The hold-up data obtained in this investigation were compared with each other, with results of D. Koren [26] and with literature correlation results. These comparison steps were made based on comparing the following separately:

- Comparison of the clarified solution systems

- Comparison of the solvent in pulp processing systems.

5.1 Comparison of the hold-up data of this investigation

This comparison was carried out based on comparing the clarified solution system results and then, the solvent in pulp processing system results.

5.1.1 Comparison of the Clarified Solution Systems

a - Effect of the Amplitude

Hold-up profiles of data Set I, typical Figures 4.1, 4.2 (2.55 cm pulse amplitude) and data Set II, typical Figures 4.4, 4.5 (0.75 cm pulse amplitude) show the effect of changing the amplitude on the hold-up keeping all the other operating conditions the same. Hold-up profiles of Set I indicated operation in the emulsion region; while that of Set II indicated operation in the mixer-settler region. Both hold-up profiles show that an increase in the amplitude decreased the transition frequency. Then, any change in amplitude at constant aqueous and organic flow rates varied the operation of the column, the hold-up and the transition frequency. Thus, the effect on hold-up due to amplitude is similar to the changes due to the frequency as would be expected, since agitation intensity is a product of frequency times amplitude.

b - Effect of an Increase in the Aqueous Dispersed Phase Flow Rate

The aqueous dispersed phase flow rates in Set III were higher than the flow rates used in Set I. Figure 5.1 shows that the hold-up increased with the increase in the dispersed phase flow rate. Hold-up profiles of data Set I, typical Figure 4.1 indicated operation in the emulsion region while that of Set III, typical Figure 4.7 indicated operation in the mixer-settler, transition and emulsion regions. As the dispersed phase flow rate increased the transition frequency increased and the minimum hold-up at the transition region was reduced (Set I, $F_t = 17$ to 22 cycles/min. and Set III, $F_t = 30$ cycles/min).

c - Effect of Dispersed Phase

Sets III and IV were carried out under identical operating conditions. The differences were the aqueous flow rates and dispersed phase kind, (Set III, aqueous dispersed phase while Set IV, organic phase was dispersed). Hold-up % data of Set III shows higher values than in Set IV. Thus on maintaining all the operating conditions the same, the hold-up % data was slightly higher when the aqueous is th dispersed phase. Hold-up profiles of Set III indicated that the operation was in the mixer-settler, transition and emulsion regions due to high aqueous dispersed phase flow rates; while that of Set IV indicated operation in the boundary between the mixer-settler and the emulsion region, i.e., transition region where the transition frequency range is much wider and smaller in value than in Set III.

5.1.2 Comparison of the Solvent in Pulp Processing Systems

a - Effect of the Tailings Concentration in the Aqueous Phase

Sets III, V and VI were carried out at the same operating conditions. The difference was the tailings concentration present in each set. Set III, clarified aqueous

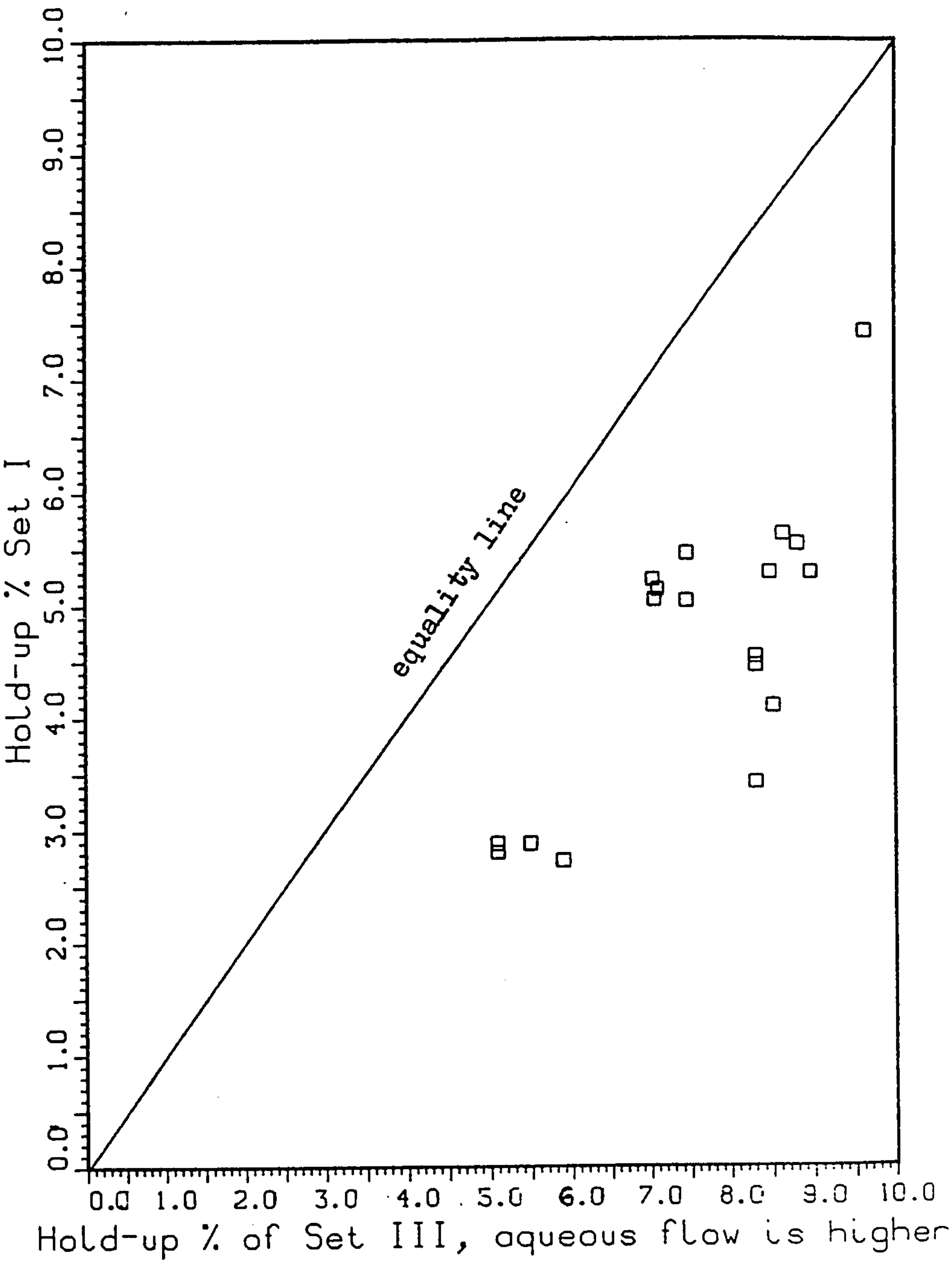


Figure 5.1: Comparison of hold-up % of Set I versus Set III of higher clarified dispersed aqueous phase flow rate.

phase. Set V, 10 % by weight spent uranium tailings and Set VI, 25 % .

Figures 5.2 and 5.3 show the effect of tailings on hold-up. As the concentration of the tailings increased in the aqueous phase the hold-up decreased and the transition frequency became higher in magnitude. With uranium it was found to be possible to operate at high frequencies and hence the column operated in the emulsion region, however, more organic entrainment was noticed. Hold-up profiles of Set III (Figures 4.7, 4.8), Set V (Figures 4.11, 4.12) and Set VI (Figures 4.13, 4.14) showed that as the aqueous flow rate increased the transition frequency for the clarified aqueous set remained relatively constant. However, when solids were present in the aqueous phase the transition frequency increased with an increase in the uranium aqueous tailings flow rate and its concentration. Since the density of a drop of an aqueous dispersed phase is a function of its percentage tailings concentration. Results indicated that, hold-up decreased with increase of tailings concentration as shown in the results of Set V (Figures 4.11, 4.12) and Set VI (Figures 4.13, 4.14).

b - Discussion of the Gold Leach Tailings Results

Runs were performed using gold leach tailings in order to collect data on hold-up and to investigate the effect of these tailings on pulsed sieve plate column behavior. Results showed that, crud was formed even at the very low pulse frequencies (19 min^{-1}) and low pulse amplitude (1 cm). that the equipment used can deliver.

This behavior may mean that a high concentration of aqueous pre-wetted solid

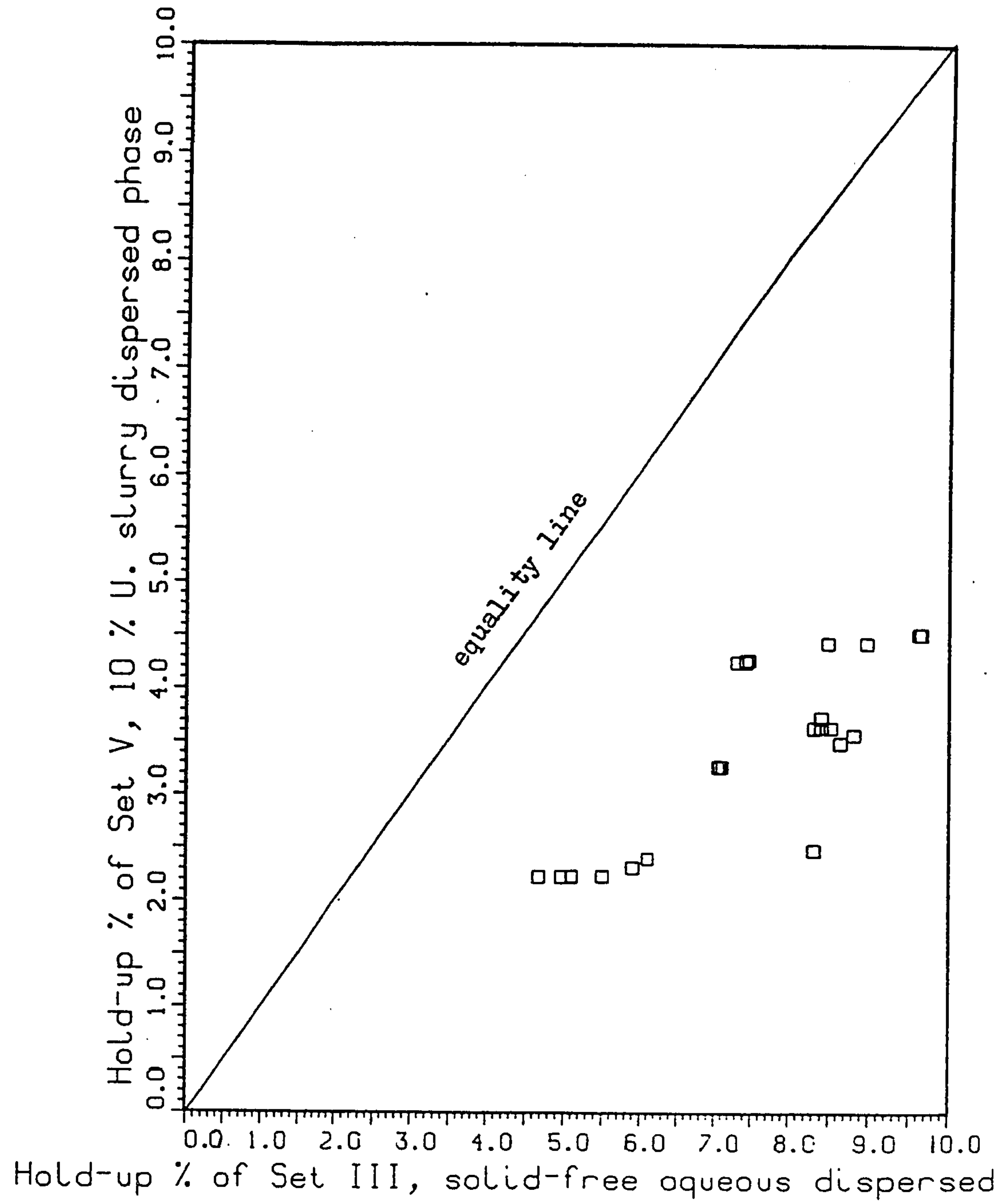


Figure 5.2: Comparison of hold-up % of Set V of 10 % uranium tailings concentration versus Set III, of clarified aqueous dispersed phase.

particles when they collide with each other, a film of the organic dispersed phase was trapped between these minute particles of solids that lifts them with its uprise in the organic phase. This is in agreement with Mizrahi and Barnea [39] who proposed that the wettability of the solid particles in aqueous phase at first may have a hydrophilic nature but at equilibrium and when in contact with an organic phase they would have a slight hydrophobic behavior which prevents them from coalescence unless the enclosed film of the organic phase was drained out. The drainage time is one of the measures of the difficulty of the coalescence due to the surfactant quantity which if it was in excess would delay coalescence and more settling time would be required.

Without surfactant addition to the gold tailings solution a stable crud was formed in the column and required several days to settle. The addition of surfactant HDTMAB reduced the settling time. However, the density difference between the aqueous and the organic phases might have made it impossible to operate the column even at the lowest agitation levels.

Runs carried out using spent uranium tailings or gold leach tailings indicated that erosion problems to the stator and rotor of the aqueous pump were minimal compared to the runs carried out by David Koren using silica sand. This is due to the fact that the tailings used were less abrasive to the internal mechanical pump parts, thus the old pump was successfully used which is in contrary to the recommendation of David Koren where the oversized pump could not at the same time deliver the low required flows and resist the friction caused by the tailings opposing the pump rotation.

5.2 Comparison of Hold-up with D. Koren [26] Results

The obtained experimental hold-up results were compared to those obtained by Koren [26].

5.2.1 Comparison of the Clarified Solution System Results

a - Effect of pH Change of the Aqueous Phase.

Comparing the results of sets II and III with Sets II and III of D. Koren. It was found that, from Figures 5.4 and 5.5 that, the hold-up increased with an increase in pH. The increase in hold-up due to aqueous phase pH increase could be due to the reduction in the droplet velocity. This reduction in the velocity is either due to the decrease in the droplets coalescence and an increase in their breakage into finer droplets; or, due to their repulsion as they pass through the ionized oxide layer around the interiors of the holes of the plates. This increase in hold-up is in agreement with data reported by North and Wells [42]. Also, it was found that the effect of pH on hold-up was greater at higher pulse amplitude.

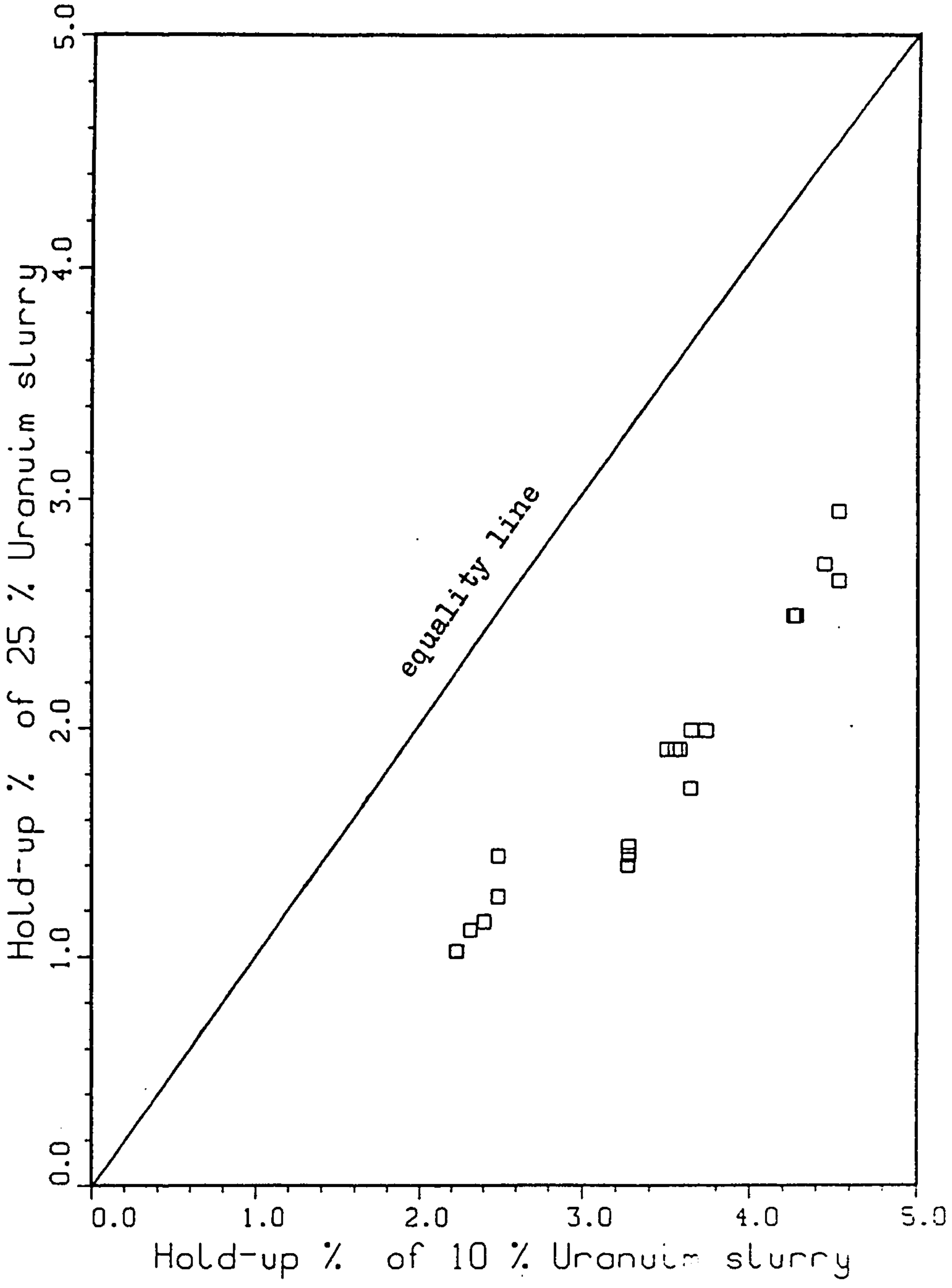


Figure 5.3: Comparison of hold-up % of Set VI versus Set V of lower uranium concentration.

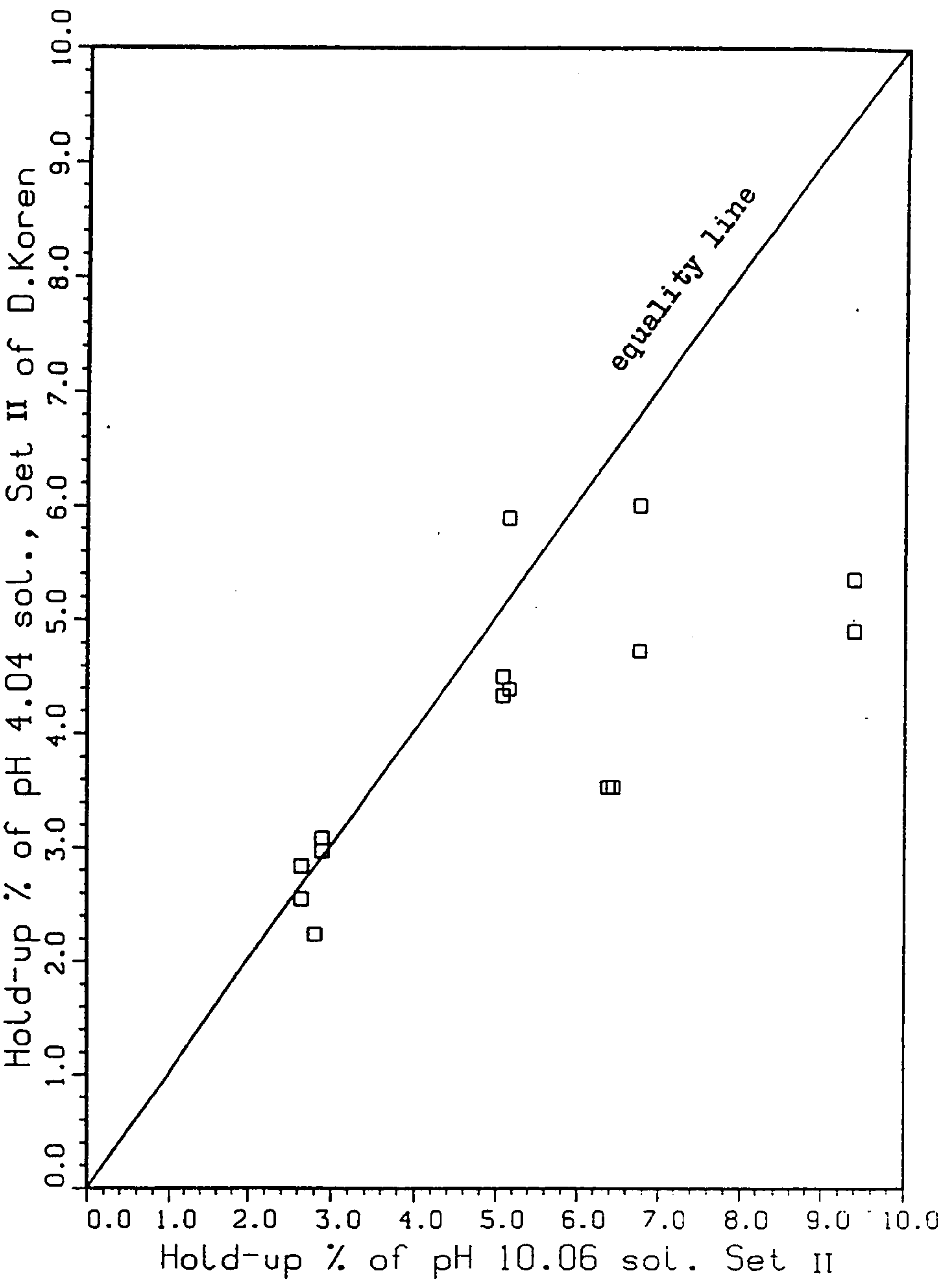


Figure 5.4: Comparison of hold-up % of Set II of D. Koren pH 4.04 versus Set II pH 10.06.

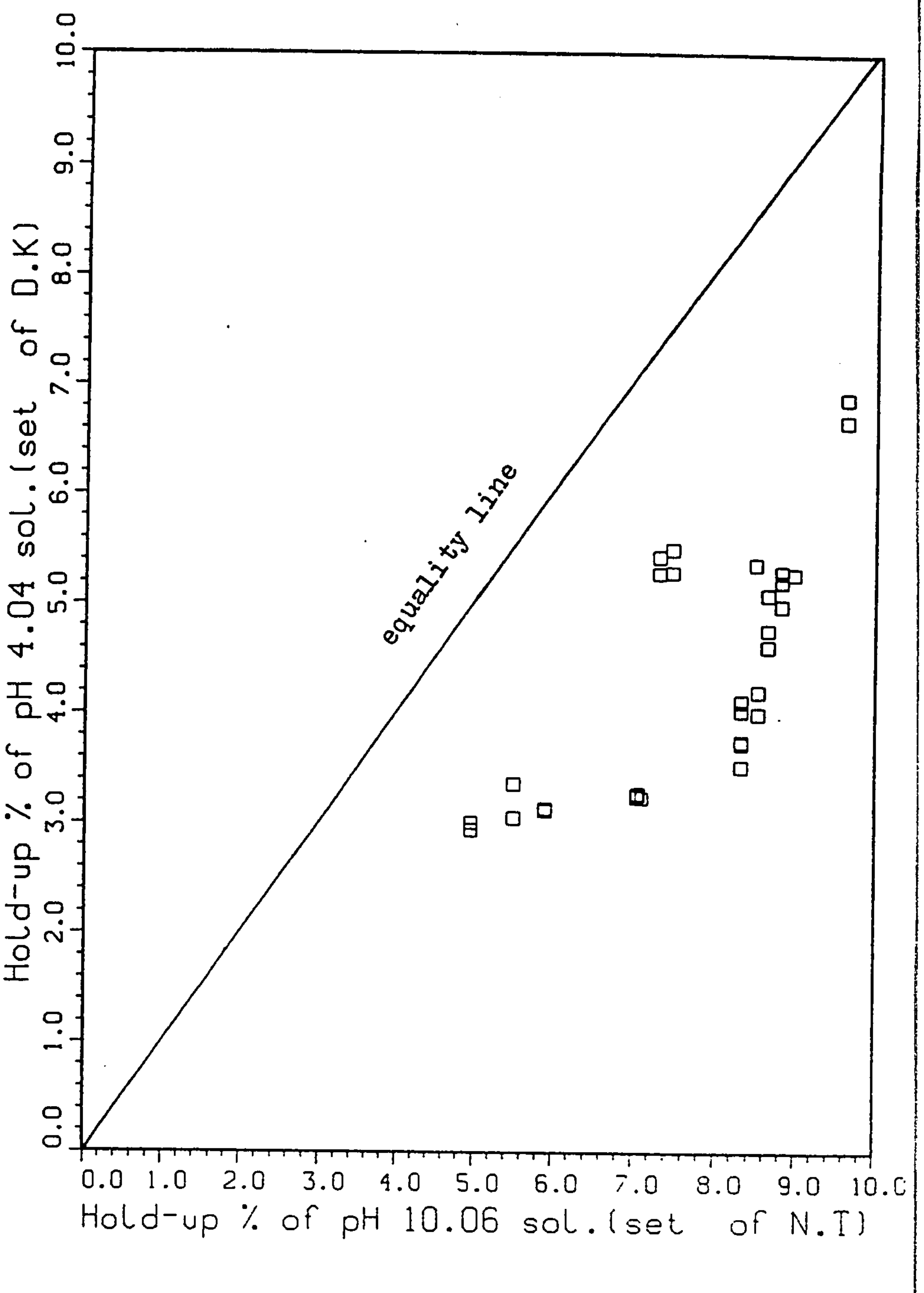


Figure 5.5: Comparison of hold-up % of Set III of D. Koren pH 4.04 versus Set III pH 10.06.

b - Effect of Dispersed Phase

Set IV (organic phase dispersed) and Set III (aqueous phase dispersed) of D. Koren were carried out at the same operating conditions. The only difference was the nature of the dispersed phase. It can be concluded that the hold-up of the aqueous dispersed phase in Set III of D. Koren showed higher values than the hold-up obtained when the organic phase was dispersed; though, its aqueous phase pH was higher (10.06 compared to 4.04 of D. Koren).

5.2.2 Comparison of Spent Uranium Tailings Hold-up with Silica Sand Hold-up

- Effect of the Tailings Density

Set V (10 % spent uranium tailings by weight) was compared with set IV (10 % silica sand by weight) of D. Koren. Figure 5.6 showed that the hold-up was slightly greater for the spent uranium tailings; though, its density is higher than the silica sand. This was not in agreement with the effect of an increase in tailings concentration. It was not possible to determine whether this was an effect of the type of tailings or of the particle size.

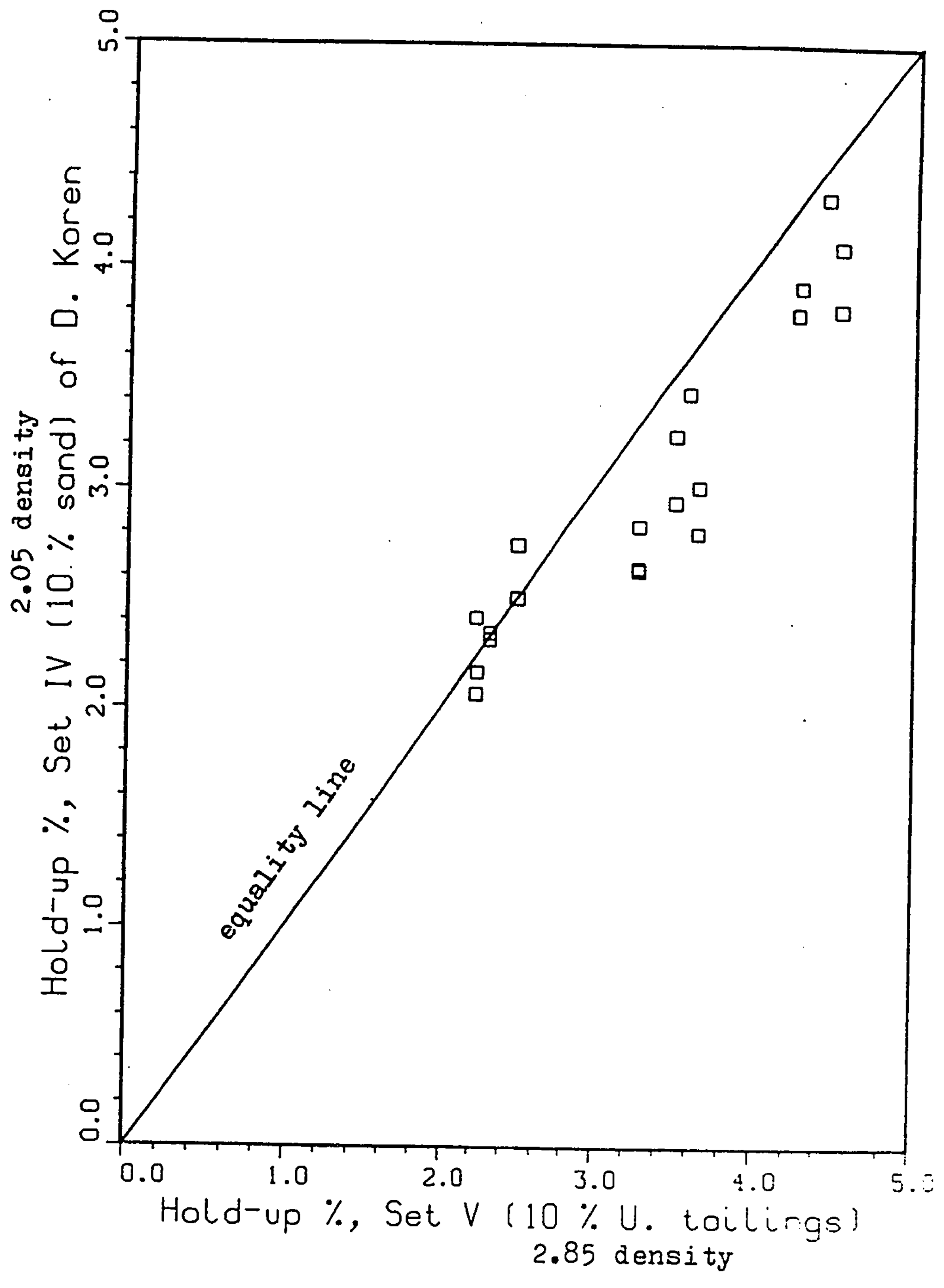


Figure 5.6: Comparison of hold-up % of Set IV of D. Koren versus Set V of different slurry density.

5.3 Comparison of Hold-up Results with Literature Correlations

A comparison was carried out between the observed hold-up of the uranium tailings and the predicted hold-up % values as computed by the equations 2.10 up to 2.21 of Sato et al [50], Arthayukti [1], Miyauchi and Oya [38] and Kumar and Hartland [29]. These equations gave different predicted values for the hold-up percent as shown in Figures 5.7 and 5.8.

a - Comparison of the Hold-up % Results of Set V (10 % Spent Uranium Tailings) with Literature Correlations

All of the above proposed equations underestimate the hold-up % with respect to the experimental results obtained for data Set V. The results of this comparison are presented in Figure 5.7. This figure showed that the model suggested by Sato et al gave the best agreement. The same result was confirmed by evaluating the Sum Squares of Residuals (SSR) for all the proposed models, Table 5.1.

b - Comparison of the Hold-up % Results of Set VI (25 % Spent Uranium Tailings) with Literature Correlations

The results are presented in Figure 5.8. It showed that the model suggested by Arthayukti [1] gave the best results.

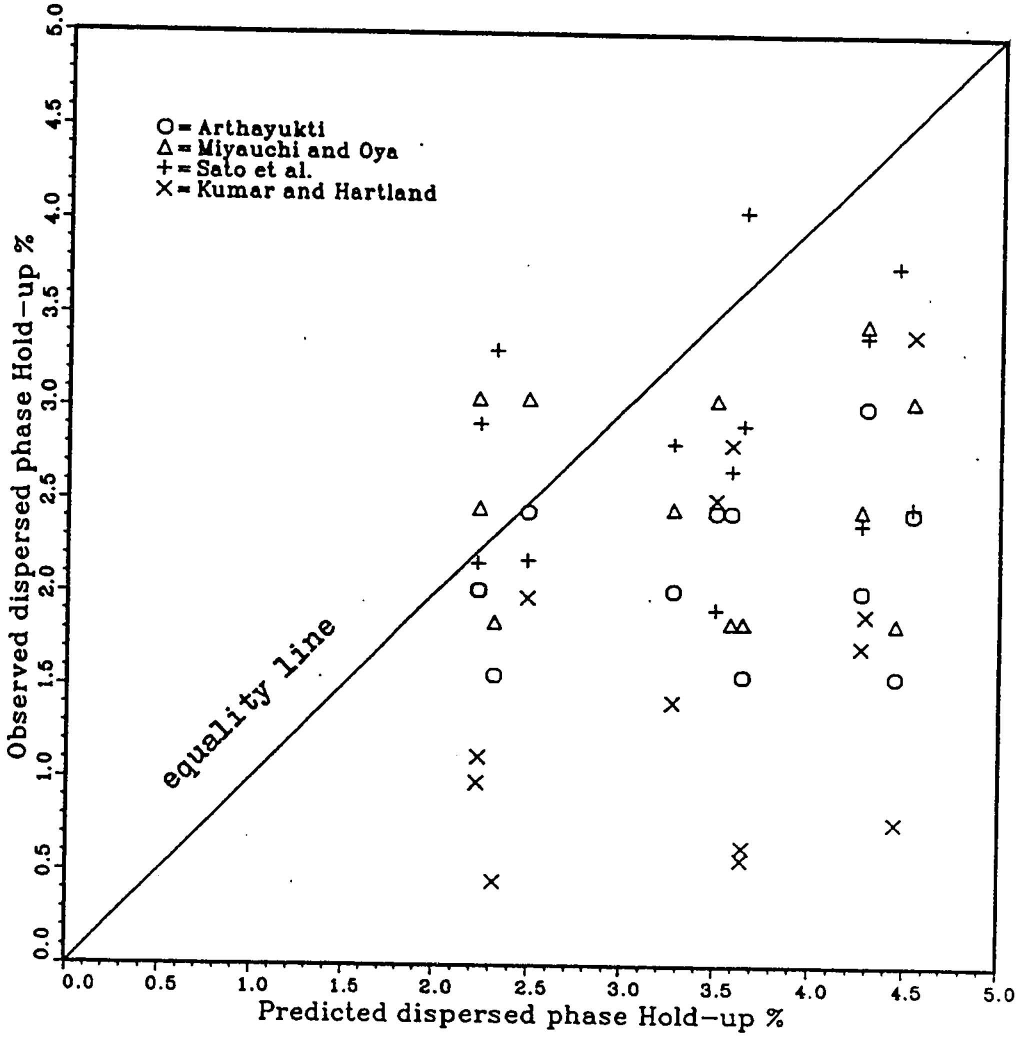


Figure 5.7: Comparison of the experimental hold-up % of 10 % spent Uranium tailings with the four literature correlations.

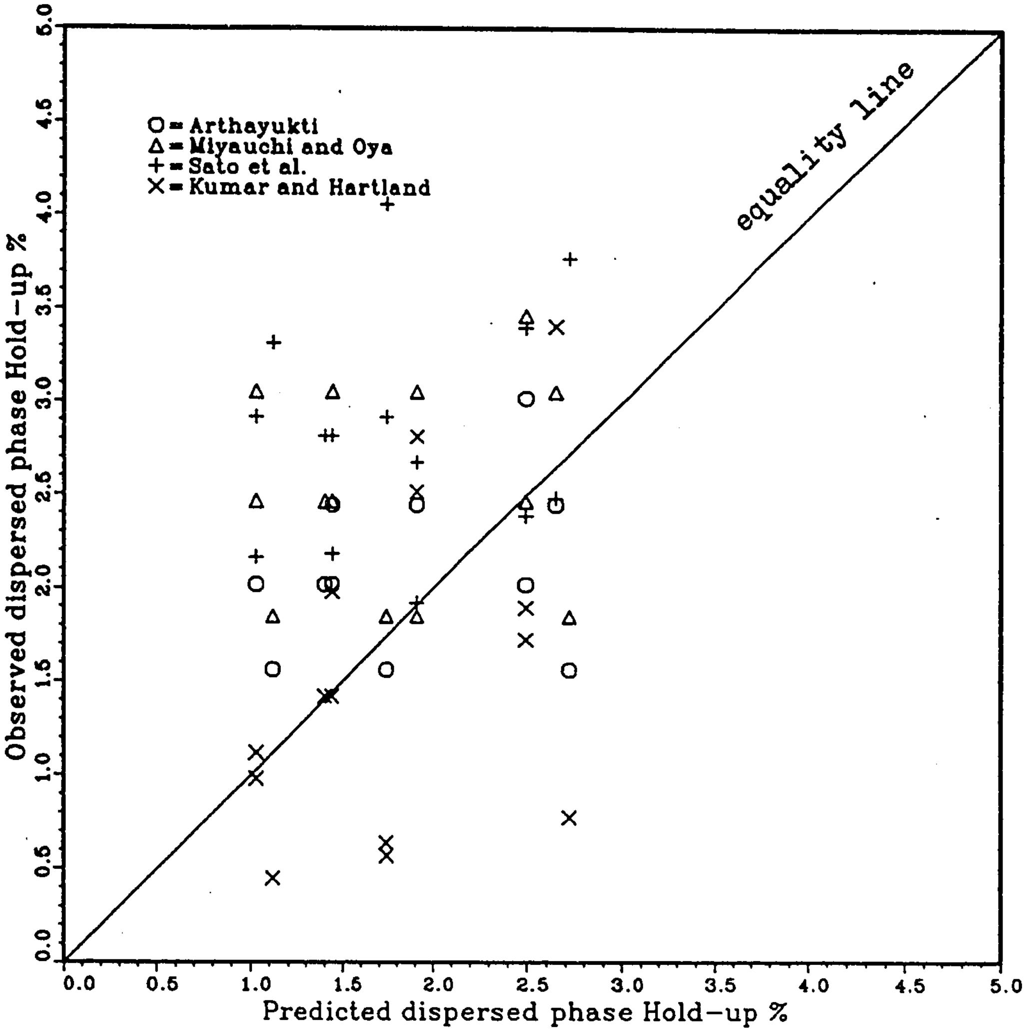


Figure 5.8: Comparison of the experimental hold-up % of Set VI (25 % spent uranium tailings) with literature correlations.

Table 5.1: The Sum Squares of the Residuals (SSR) for all the considered models .

Set#	Arthayukti	Miyauchi and Oya	Sato et al	Kumar and Hartland
V Estimation %	34.2306 - 40	25.0830 - 27	14.9824 - 16.5	60.3676 - 55
VI Estimation %	6.3910 + 17	14.6400 + 40	23.4017 + 60	9.8230 - 55

"-" stands for underestimation
"+" stands for overestimation

The same result was obtained by calculating the Sum Squares of Residuals (SSR) for all the models of Set VI. The model suggested by Arthayukti gave the lowest value of the SSR, see Table 5.1 .

From the above comparison between the observed and the predicted hold-up by the four equations, it can be concluded that none of the four equations available in the literature are suitable to predict hold-up for solvent in pulp processing. The problem is the lack of information on the physical properties of the solvent in pulp process.

5.4 Comparison of McAllister's Equation 2.22 with the Experimental Results of Set VII at Flooding Conditions.

The experimental total flow rates at flooding conditions of set VII were used in McAllister's [35] equation 2.22 to find the predicted pulse frequency at which the equation predicts flooding and compare it with the experimental pulse frequency obtained at flooding. Table 5.2 shows a comparison between the experimental and the predicted pulse frequency using McAllister's equation. McAllister's equation was mainly found to slightly over predict pulse frequency at which flooding occurred. This was in agreement with the work of W. Chiang et al [63].

5.5 Calculation of the Effective Solvent in Pulp Interfacial Tension

David Koren [26] noted that,

$$V_D = \frac{1}{6}\pi D_a^3 \tag{5.1}$$

The effective interfacial tension σ_e of the slurry can be calculated by using the following equation,

$$V_D = \frac{\pi \sigma_e D_v}{g \Delta \rho} \tag{5.2}$$

Knowing the value of the specific interfacial tension σ_s of the clarified phase

Table 5.2: Comparison of the experimental pulse frequency at flooding of set VII with McAllister's predicted pulse frequency, using equation 2.22.

		Continuous phase flow rate (ml/min.)			
		792	1049.6	1303.3	Frequency (min^{-1})
Dispersed phase flow rate (ml/min)	379	31	31	31	Experimental
		39	37	34	Predicted
	579.1	31	31	31	Experimental
		36.5	35	29	Predicted
	778.8	27	27	27	Experimental
		32.5	31	25	Predicted

system, the correcting factor K of the slurry between the interfacial tension of the clarified phase and the solvent in pulp can be calculated using this relation,

$$\sigma_s = K\sigma_e \tag{5.3}$$

Value of K in D. Koren investigation was found to be a function of the pulse frequency, the aqueous, and the organic flow rates.

5.6 Solvent Losses

It was noticed visually that the entrainment of solvent was directly proportional to the intensity of agitation and the percentage of solid contents in the aqueous phase where the solvent is adsorbed on the slurry particles. Entrainment also occurred due to the solubility of the solvent in the aqueous phase, especially due to the losses in an aqueous-solid-organic emulsion. This is again a major problem and losses have to be reduced before solvent in pulp processing can be used industrially.

Chapter 6

Conclusions and Recommendations

6.1 Conclusions

The results of this work has shown that, successful operation of solvent in pulp processing is very dependent on the system being investigated. The uranium tailings have the potential for solvent in pulp processing; where as, the gold tailings could not be processed under the conditions studied.

Bench scale phase separations were useful in identifying potential application with respect to the settling times, however, it is essential that a clear interface be established indicating absence of crud in order for the application to be successful.

Hold-up data for solvent in pulp processing indicated that the column operated in the transition region. Higher agitation intensities could be used to shift the operation to the desired emulsion region. This would lead to excessive organic phase losses. Drop sizes in solvent in pulp processing increased significantly compared to drop sizes in clarified solution operation. Experiments indicated that, with uranium tailings, rotor and stator wear may not be as great as found with silica sand.

The results of the clarified solution indicated that, although the hold-up was a function of the aqueous dispersed phase pH, there was very little change in hold-up values when either of the two phases were dispersed.

At present, there is no single equation that can be used to predict hold-up for solvent in pulp processing systems. A better understanding of the three phase system physical properties, viscosity and interfacial tension is required in order to predict hold-up using the equations proposed in the literature.

6.2 Recommendations

Further work should be directed towards the improvement of phase separation characteristics and the reduction of organic phase losses. This would best be achieved by running bench scale settling tests to determine the effect of wetting agents on settling before carrying out pulsed column runs.

Organic phase losses have to be evaluated from actual solvent in pulp processing pulsed column runs and studies undertaken to determine methods of reducing these losses.

Equipment modification should be made to improve accuracy of hold-up measurements, through the use of an Immhoff cone or direct measurements of hold-up along the column. Care should be taken to use stainless steel fittings with xylene and tri-butylphosphate as an extractant phase.

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Appendix A

Equipment Calibration

A.1 Aqueous Pump Calibration

The calibration of the aqueous pump consisted of measuring the volume delivered per unit time as a function of the difference in manometer height. The calibration equations obtained, were as follows. .

- Clarified aqueous solution.
  $Q = 623.6\Delta h^{0.51}$
- 10 % by weight uranium slurry aqueous solution.
  $Q = 695.9\Delta h^{0.57}$
- 25 % by weight uranium slurry aqueous solution.
  $Q = 709.6\Delta h^{0.601}$

A.2 Organic Pump Calibration

The organic pump calibration was done on the same bases of volume versus time. The results are presented in Table A.1

Table A.1: Calibration data for organic pump.

%Capacity	Volume collected (ml.)	Time (s)	Flow rate operated (ml/min)	Flow rate (ml/min)
2.25	334	55.70	350.0	359.78
2.25	322	55.50	350.0	348.10
2.75	392	61.50	379.3	382.46
2.75	380	60.50	379.3	376.86
4.25	610	60.00	600.0	610.00
4.25	605	60.00	600.0	605.00
5.00	760	60.00	778.8	760.00
5.25	798	60.00	778.8	798.00
7.25	870	60.00	850.0	870.40
7.25	605	60.00	850.0	856.00

Appendix B

Additional Figures

The following graphs are supplementary to the graphs already presented in chapter four. Four extra graphs for each set are presented in this section, two of which represent the aqueous phase flow rates as constant while studying the effect of varying the organic phase flow rate; The other two graphs represent, the organic phase flow rate as constant while varying the aqueous phase flow rates. Each of these graphs showed the same results as discussed previously.

SET I

Conditions of Operation of Set I

- Clarified aqueous dispersed phase flow rates 523.4, 787.5 and 1045.8 ml/min.
- Organic continuous phase flow rates 379.3, 579.1 and 778.8 ml/min.
- Pulse frequency 22, 31 and 40 min^{-1}.
- Pulse amplitude 2.55 cm.
- Plate free area 24.4 %.
- Operating pH of aqueous phase 10.06.

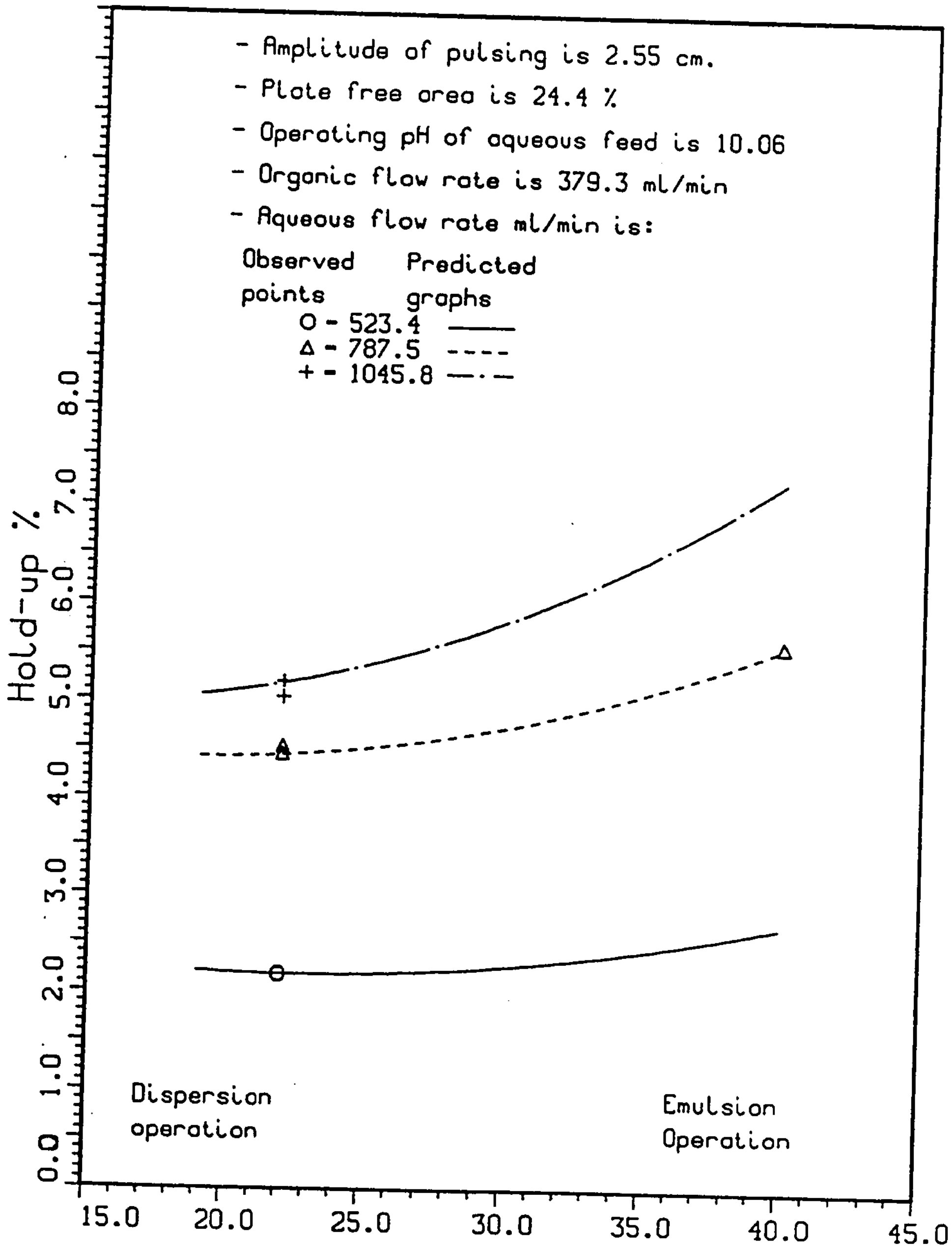

Figure B.1: Predicted and experimental hold-up % data of Set I, clarified aqueous dispersed phase (variable), organic continuous phase (constant).

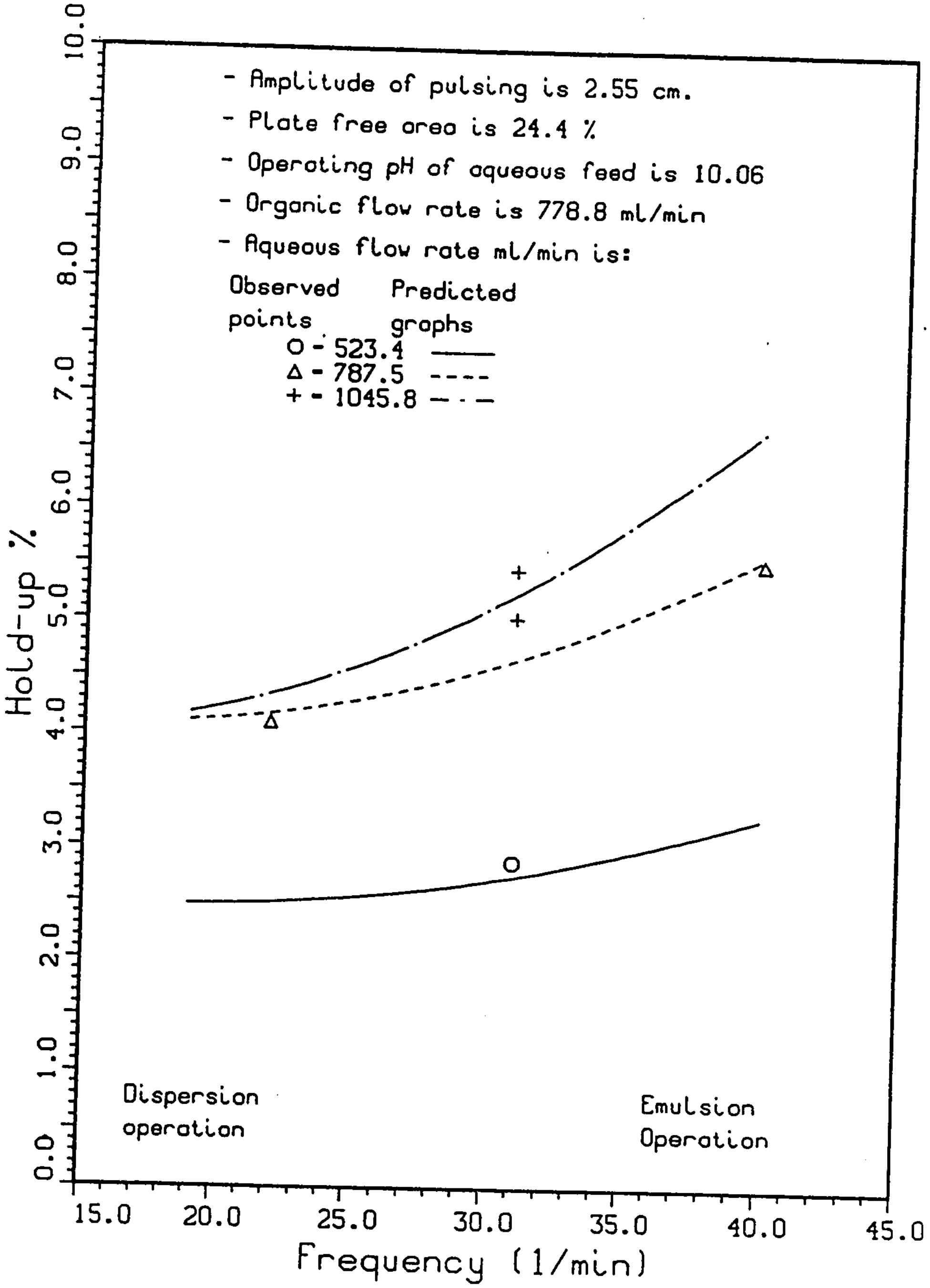

Figure B.2: Predicted and experimental hold-up % data of Set I, clarified aqueous dispersed phase (variable), organic continuous phase (constant).

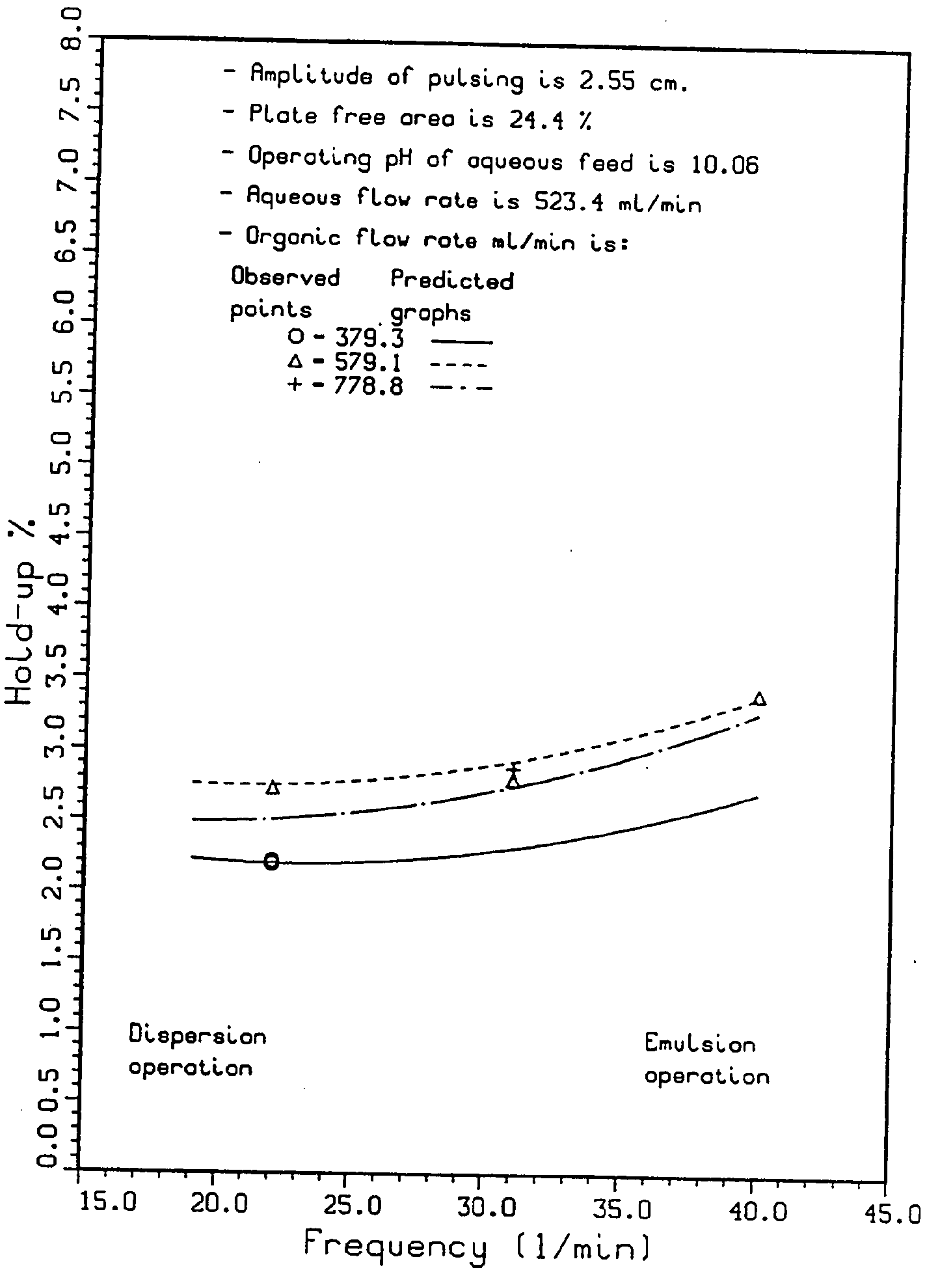

Figure B.3: Predicted and experimental hold-up % data of Set I, clarified aqueous dispersed phase (constant), organic continuous phase (variable).

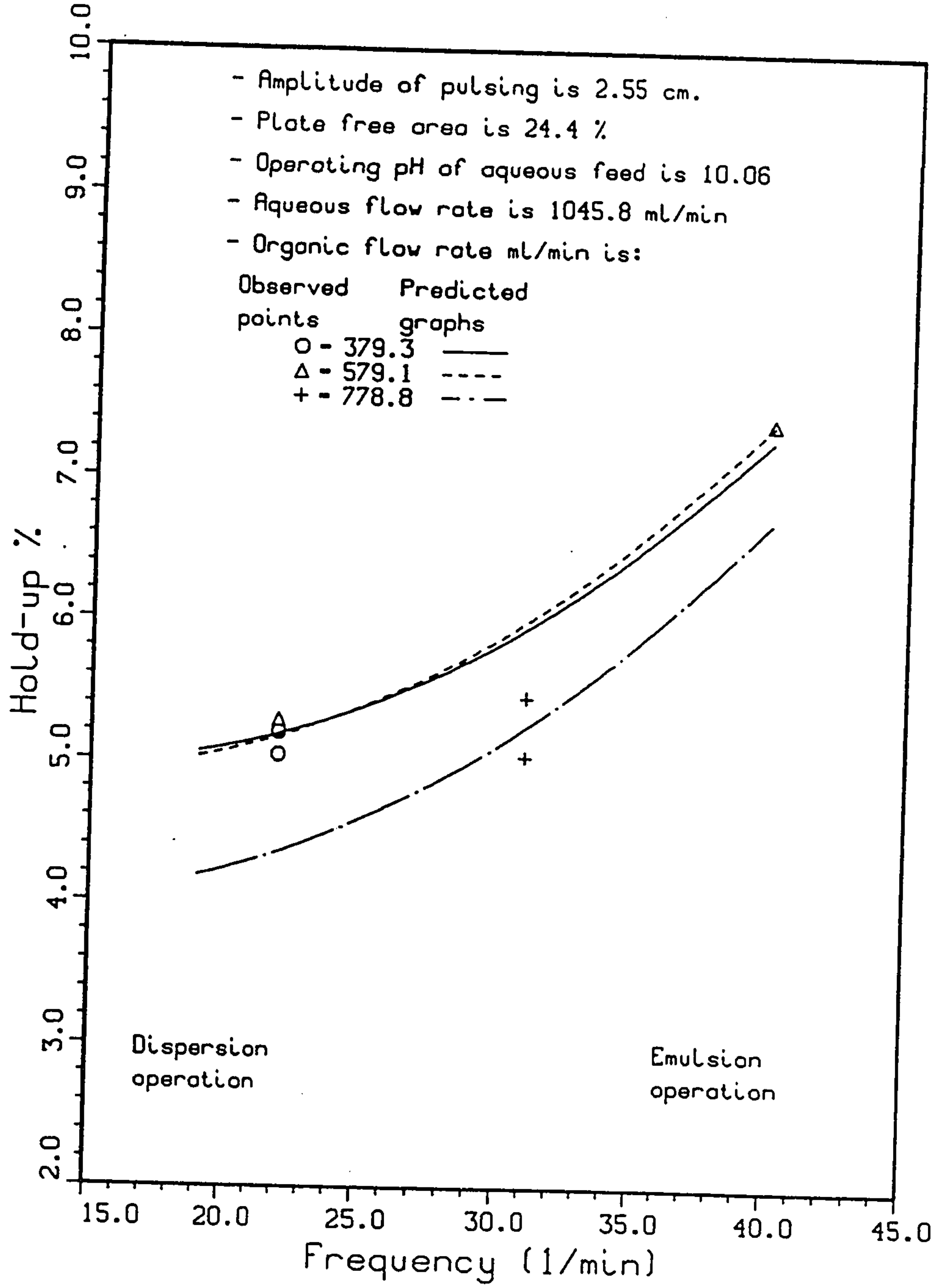

Figure B.4: Predicted and experimental hold-up % data of Set I, clarified aqueous dispersed phase (constant), organic continuous phase (variable).

SET II

Conditions of Operation of Set II

- Clarified aqueous dispersed phase flow rates 523.4, 787.5 and 1045.8 ml/min.
- Organic continuous phase flow rates 379.3, 579.1 and 778.8 ml/min.
- Pulse frequency 22, 27, 31 and 40 min^{-1}.
- Pulse amplitude 0.75 cm.
- Plate free area 24.4 %.
- Operating pH of the aqueous phase 10.06.

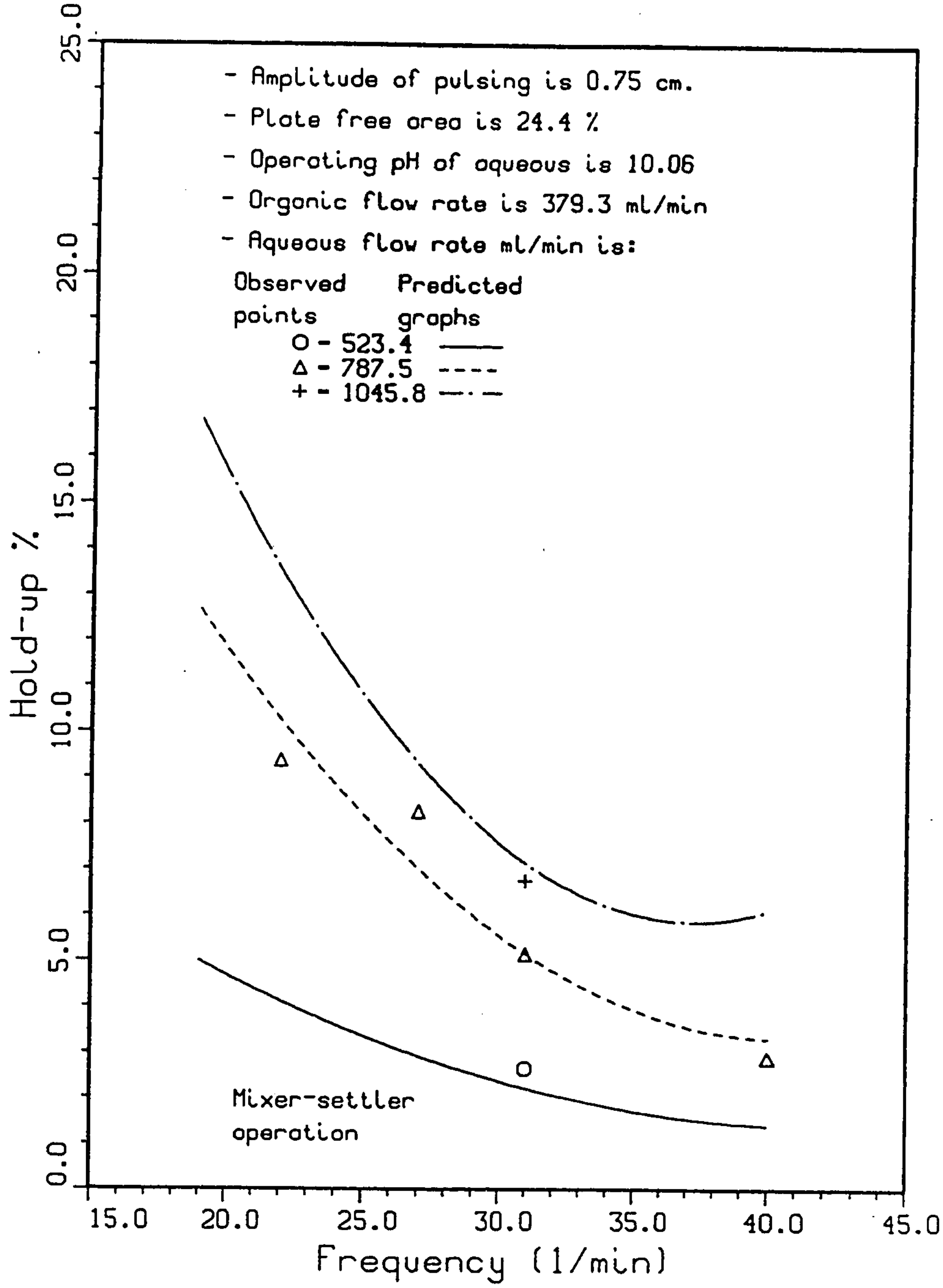

Figure B.5: Predicted and experimental hold-up % data of Set II, clarified aqueous dispersed phase (variable), organic continuous phase (constant).

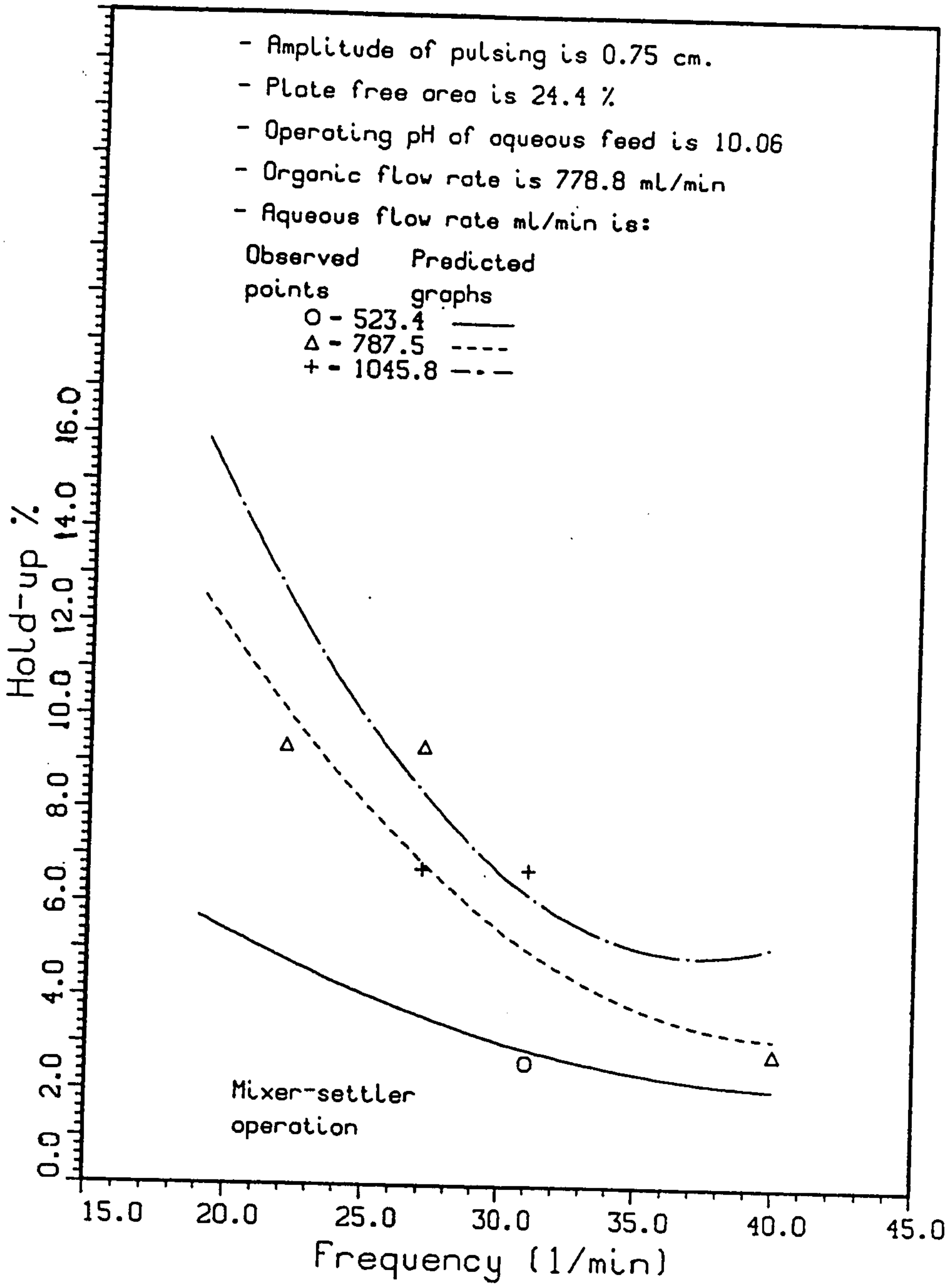

Figure B.6: Predicted and experimental hold-up % data of Set II, clarified aqueous dispersed phase (variable), organic continuous phase (constant).

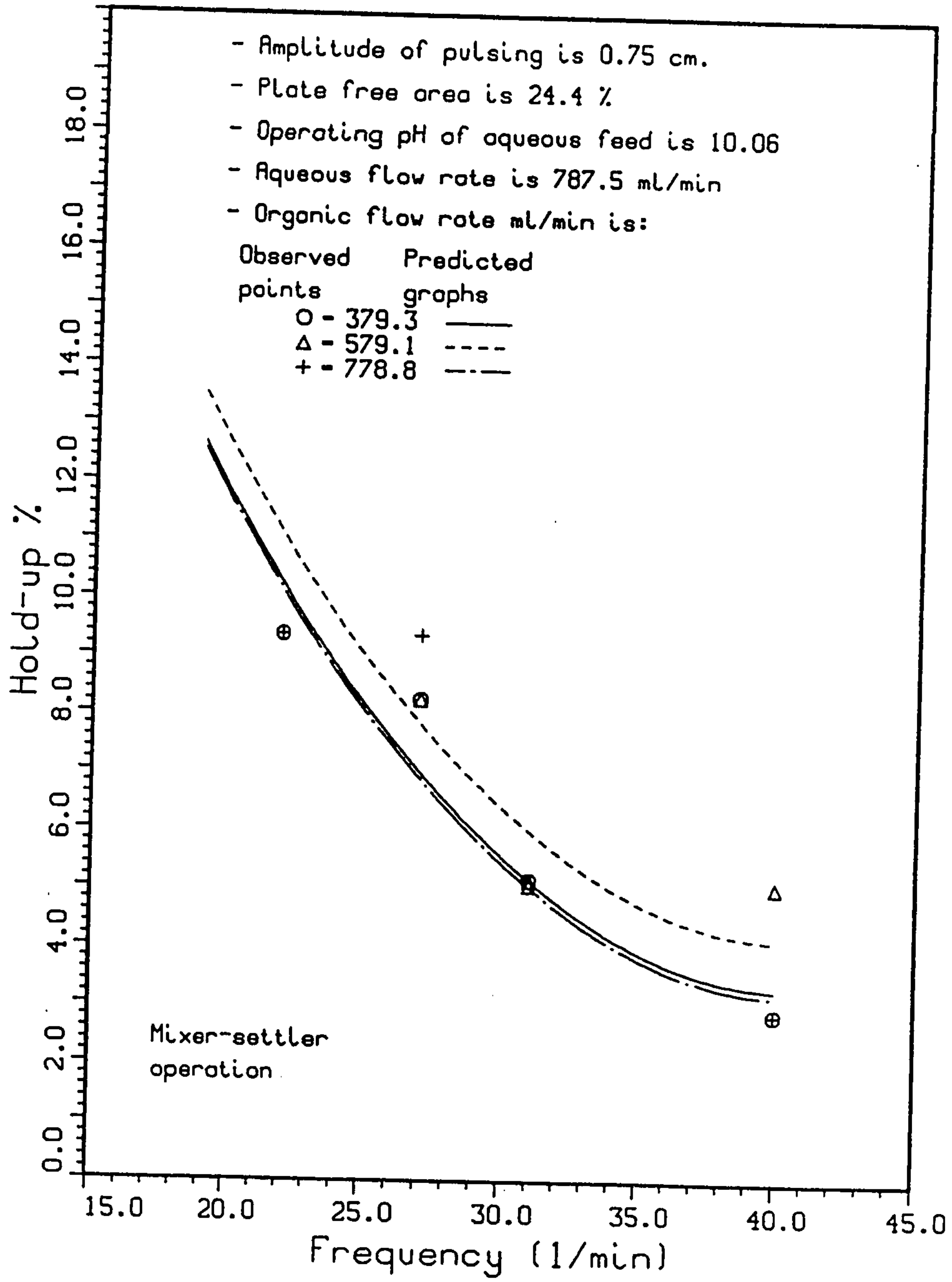

Figure B.7: Predicted and experimental hold-up % data of Set II, clarified aqueous dispersed phase (constant), organic continuous phase (variable).

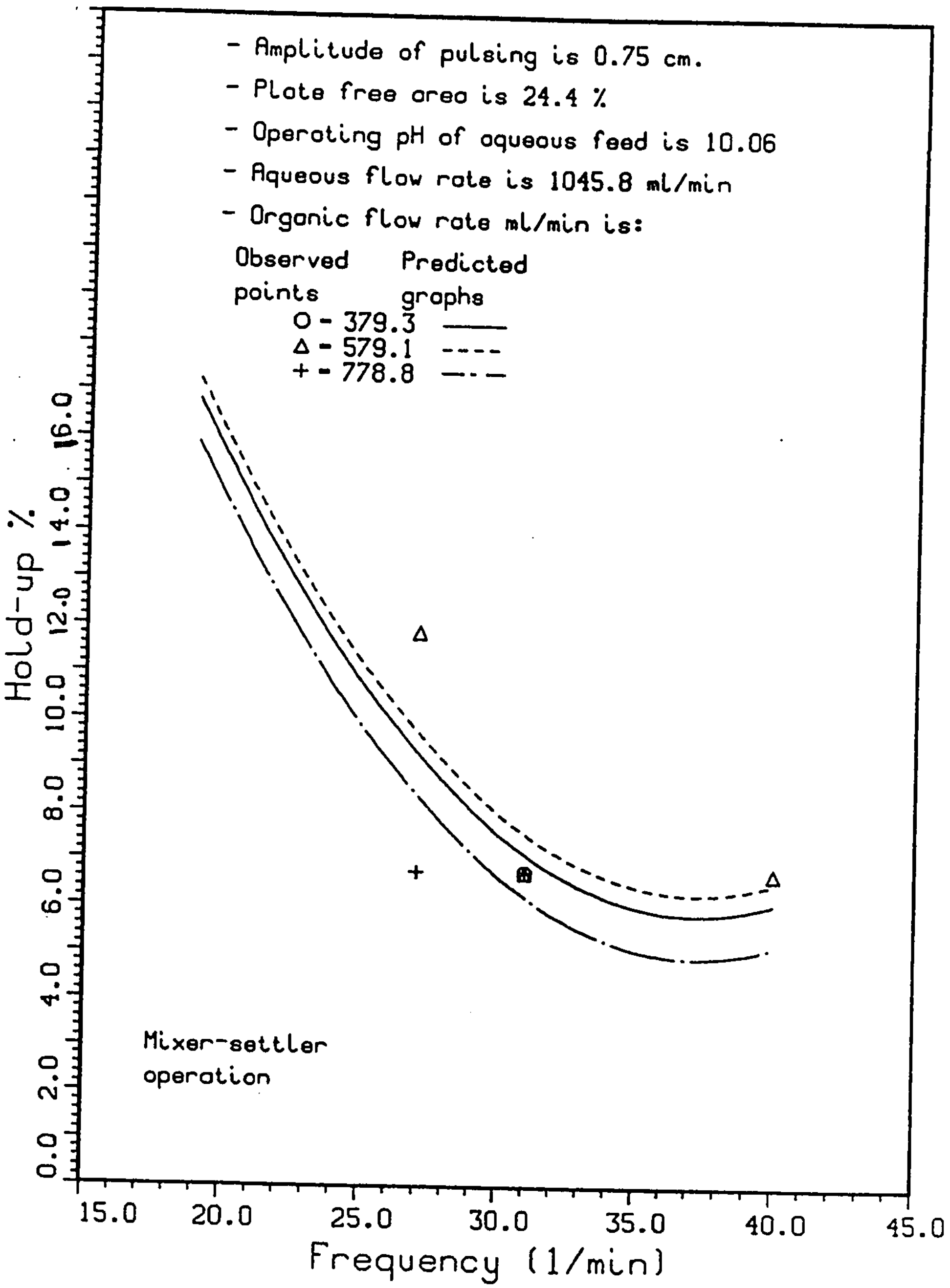

Figure B.8: Predicted and experimental hold-up % data of Set II, clarified aqueous dispersed phase (constant), organic continuous phase (variable).

SET III

Conditions of Operation of Set III

- Clarified aqueous dispersed phase flow rates 792, 1049.6 and 1303.3 ml/min.
- Organic continuous phase flow rates 379, 579.1 and 778.8 ml/min.
- Pulse frequency 19, 22, 31 and 40 min^{-1}.
- Pulse amplitude 2.55 cm.
- Plate free area 24.4 %.
- Operating pH of the aqueous phase 10.06.

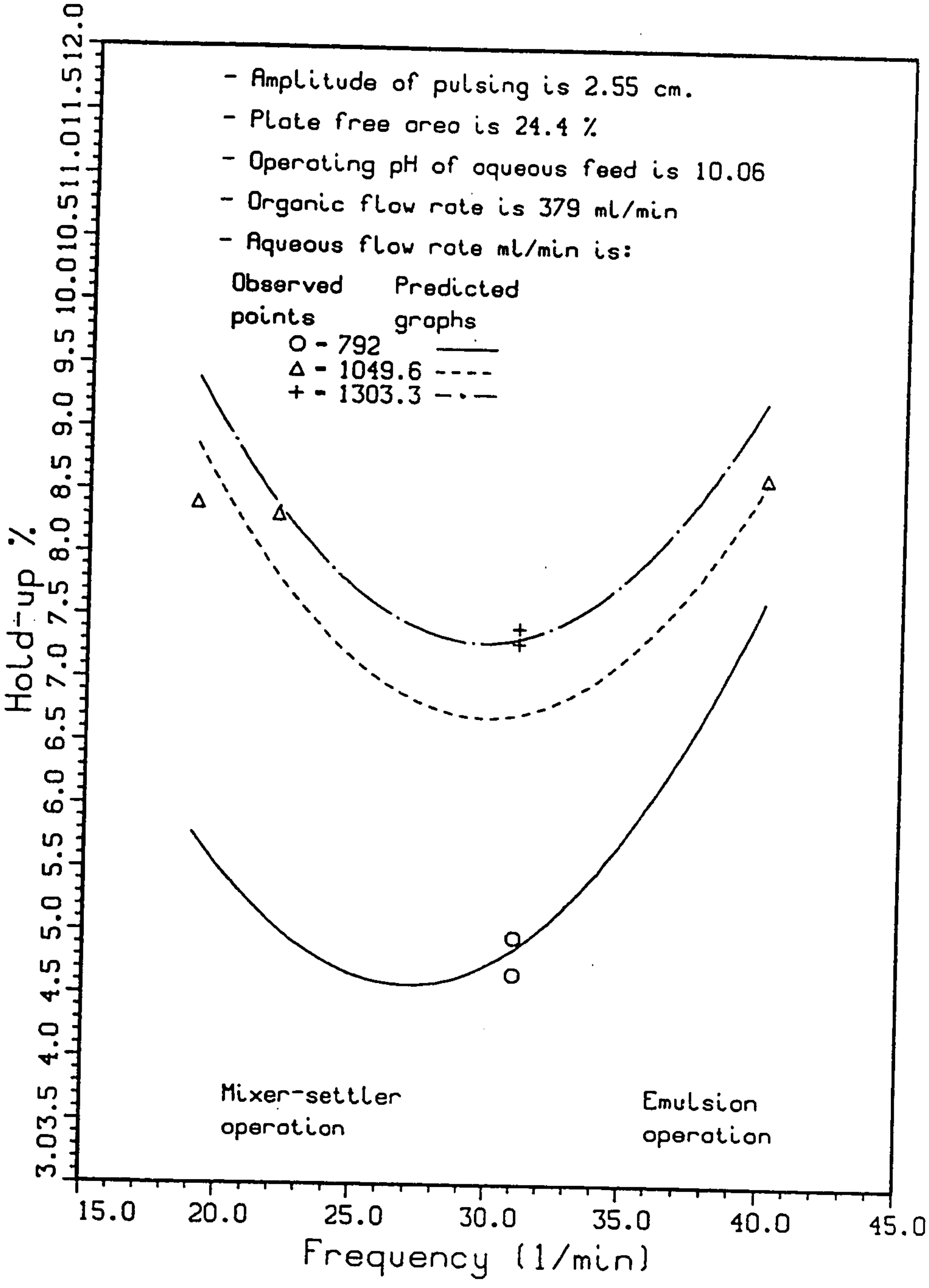

Figure B.9: Predicted and experimental hold-up % data of Set III, clarified aqueous dispersed phase (variable), organic continuous phase (constant).

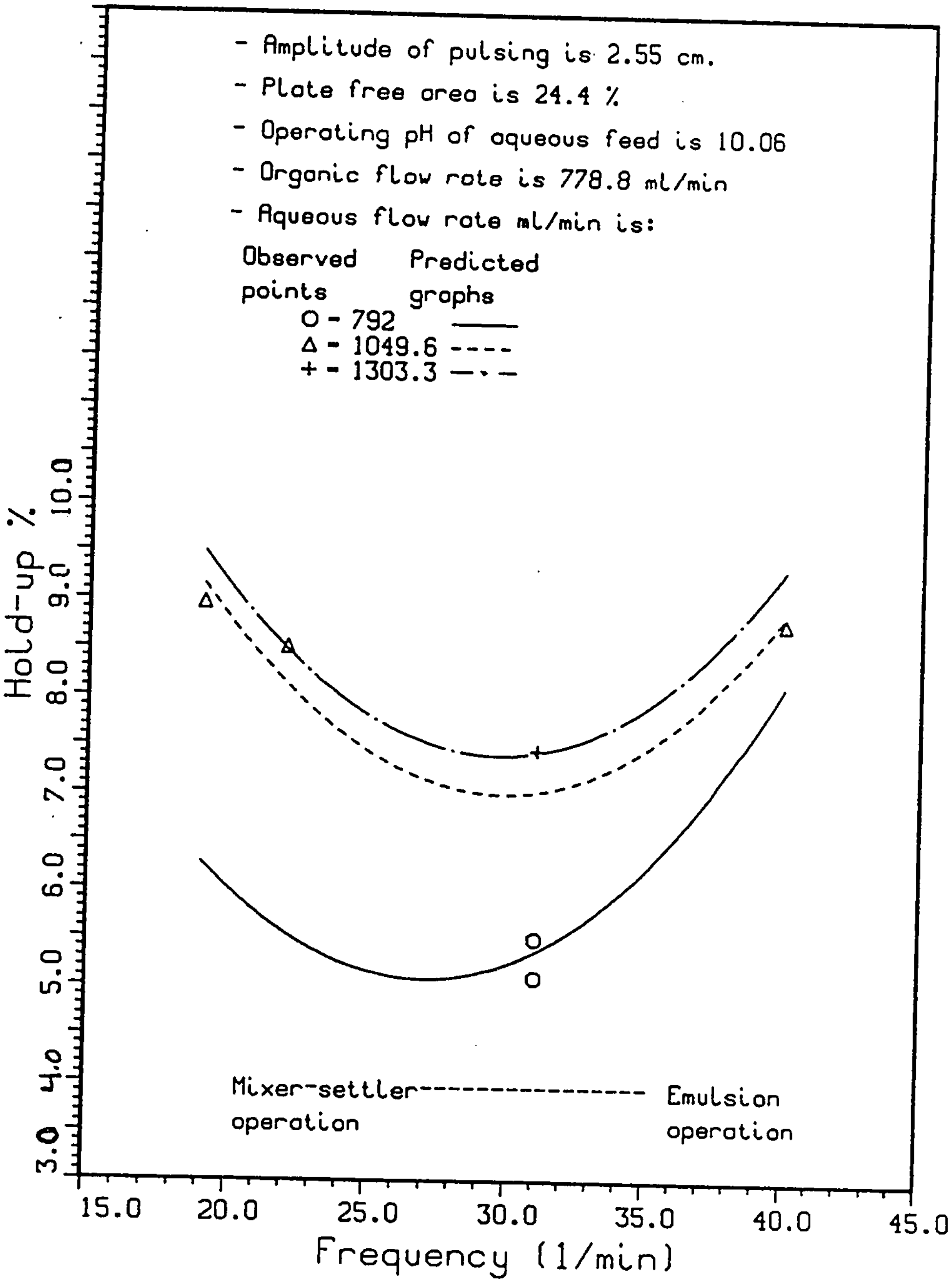

Figure B.10: Predicted and experimental hold-up % data of Set III, clarified aqueous dispersed phase (variable), organic continuous phase (constant).

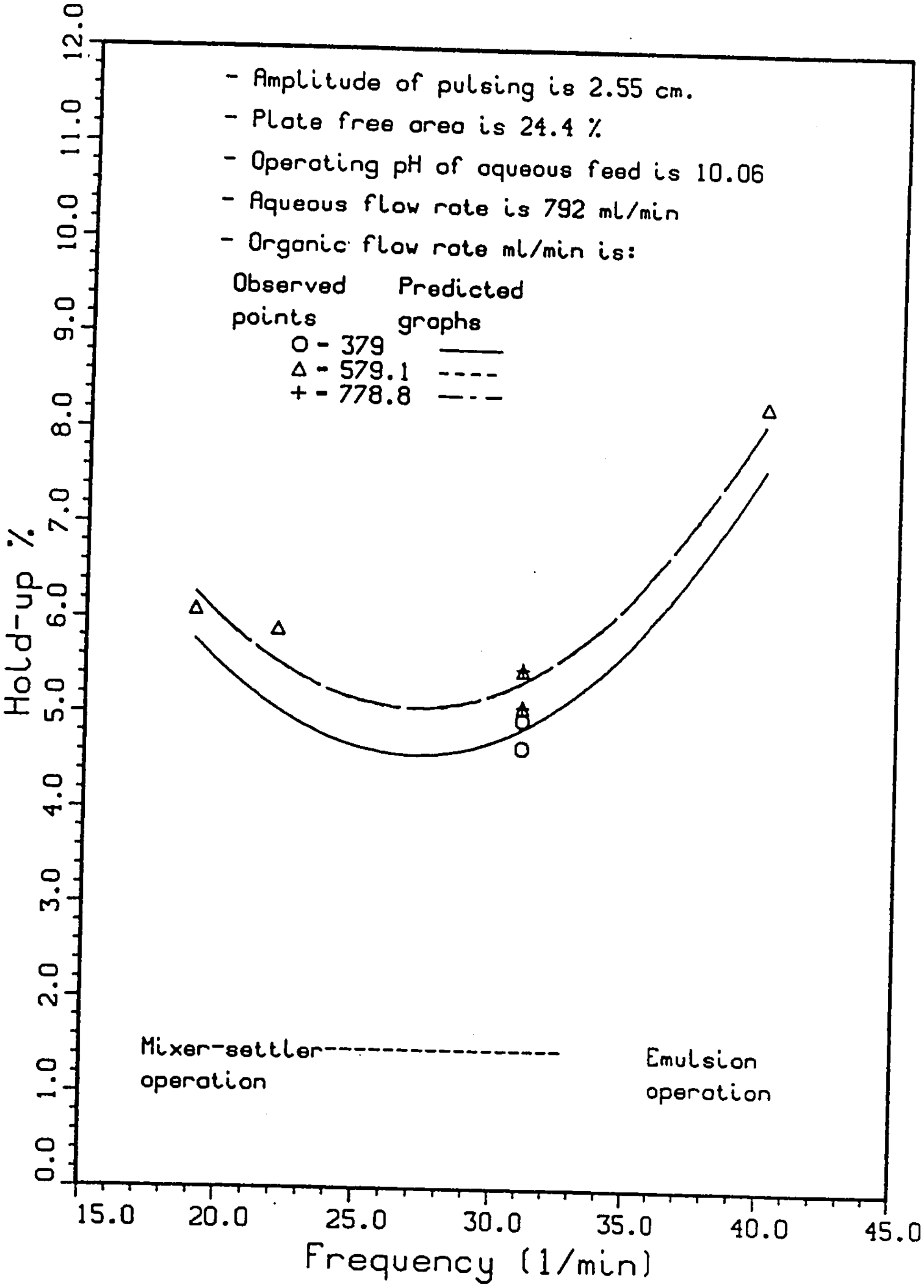

Figure B.11: Predicted and experimental hold-up % data of Set III, clarified aqueous dispersed phase (constant), organic continuous phase (variable).

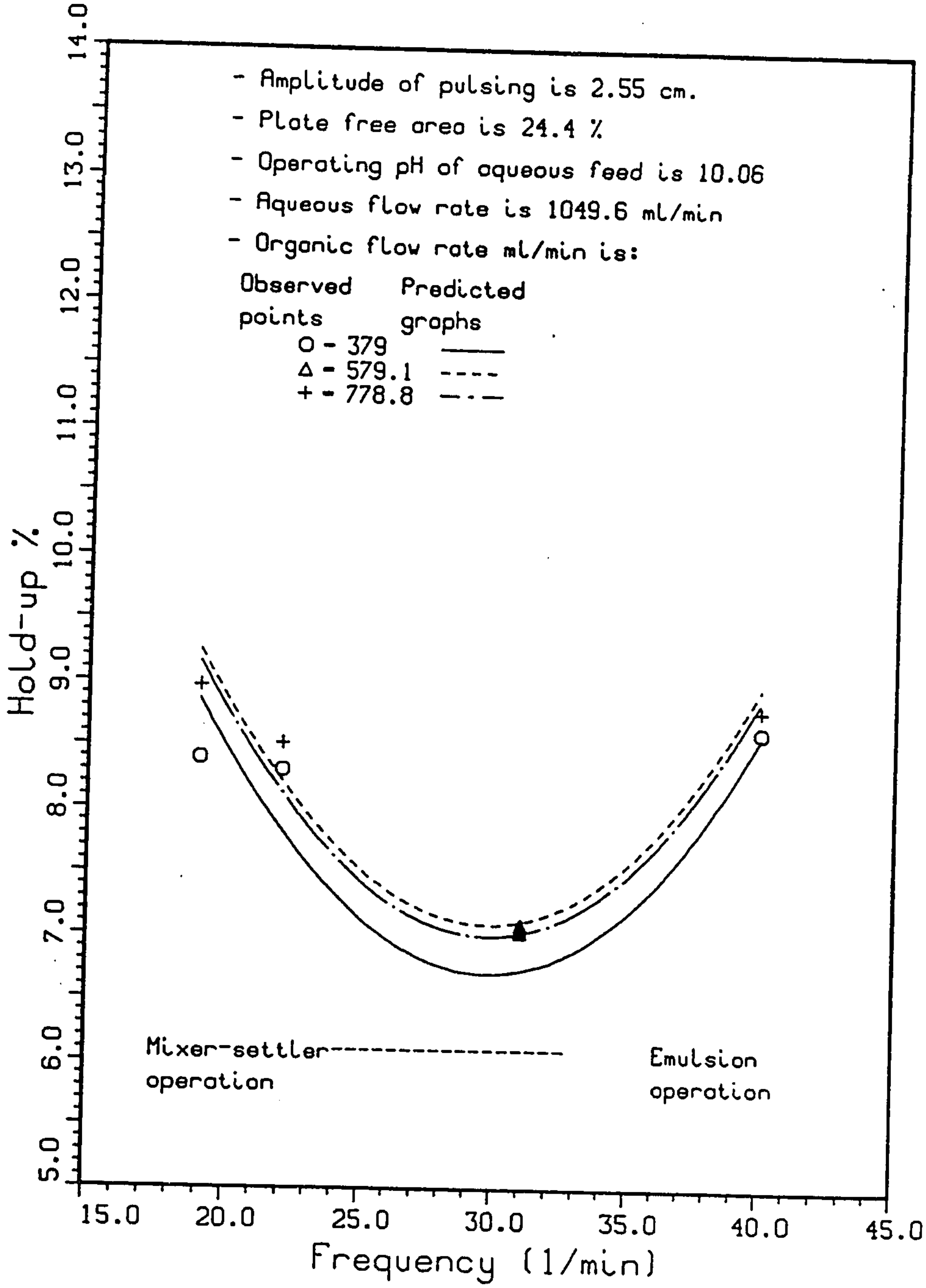

Figure B.12: Predicted and experimental hold-up % data of Set III, clarified aqueous dispersed phase (constant), organic continuous phase (variable).

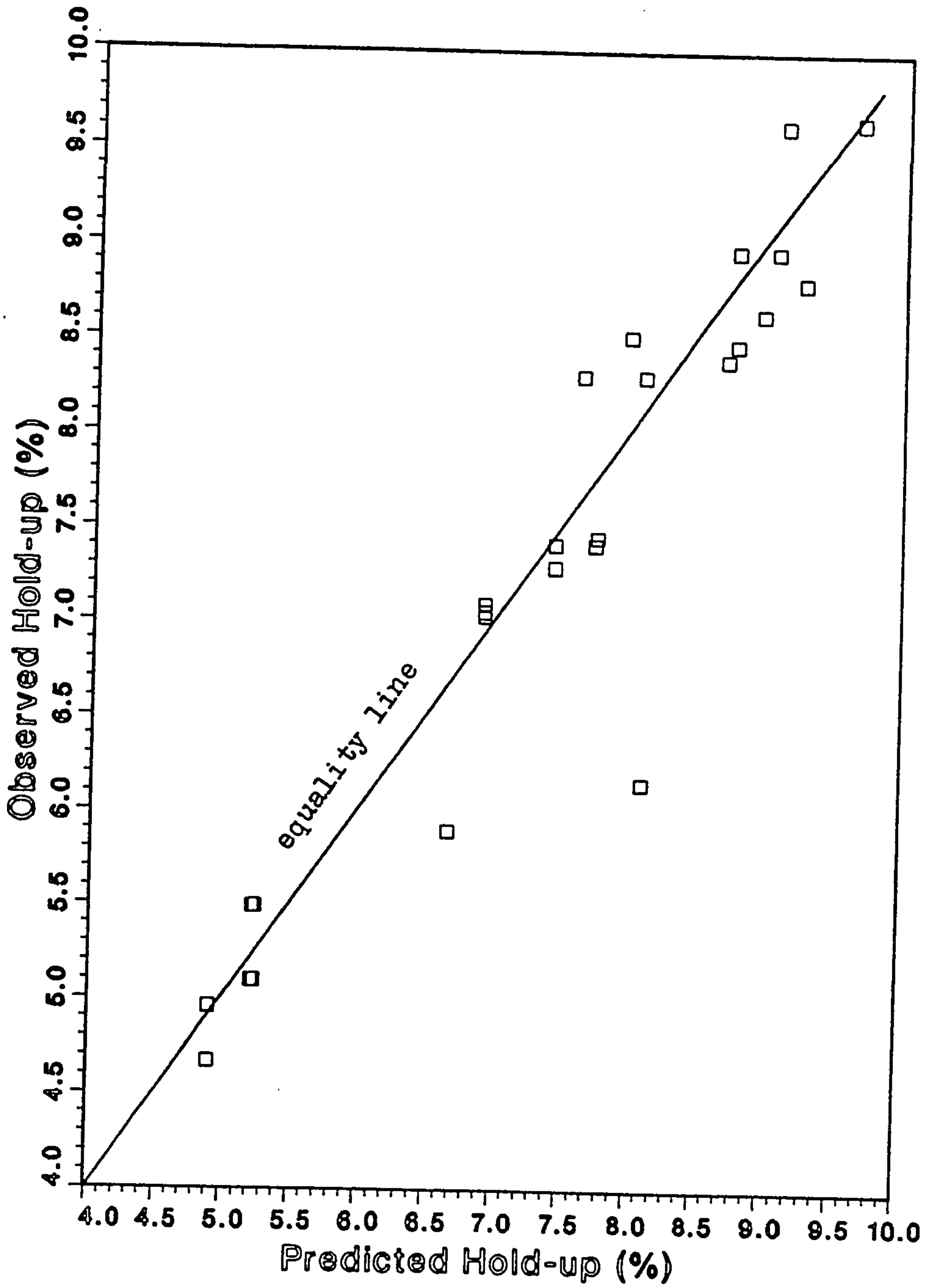

Figure B.13: Observed versus predicted hold-up % values of Set III.

SET IV

Conditions of Operation of Set IV

- Organic dispersed phase flow rates 350, 600 and 850 ml/min.
- Clarified aqueous continuous phase flow rates 1000, 1500 and 2000 ml/min.
- Pulse frequency 19, 22, 31 and 40 min^{-1}.
- Pulse amplitude 2.55 cm.
- Plate free area 24.4 %.
- Operating pH of the aqueous phase 10.06.

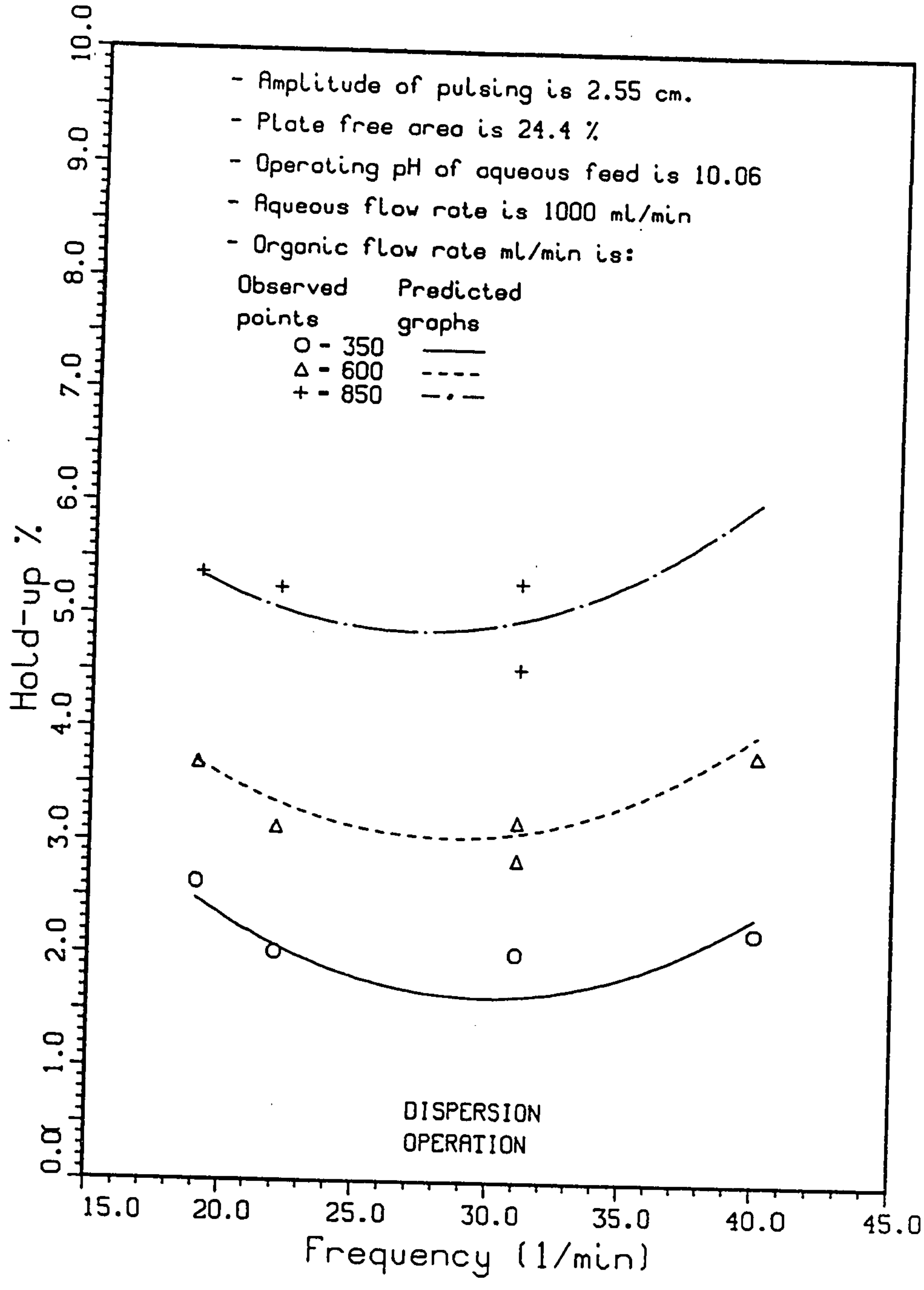

Figure B.14: Predicted and experimental hold-up % data of Set IV, clarified aqueous continuous phase (constant), organic dispersed phase (variable).

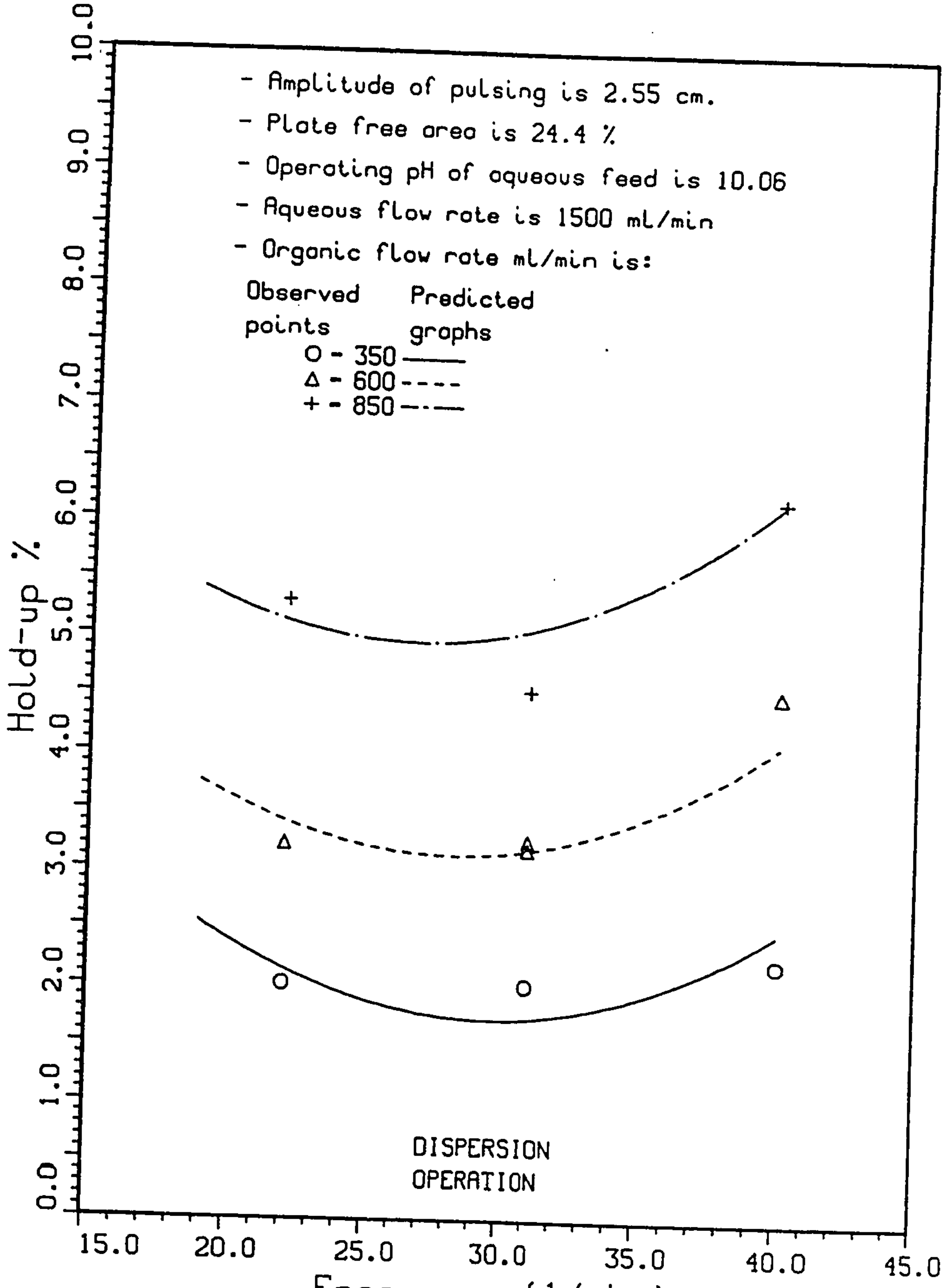

Figure B.15: Predicted and experimental hold-up % data of Set IV, clarified aqueous continuous phase (constant), organic dispersed phase (variable).

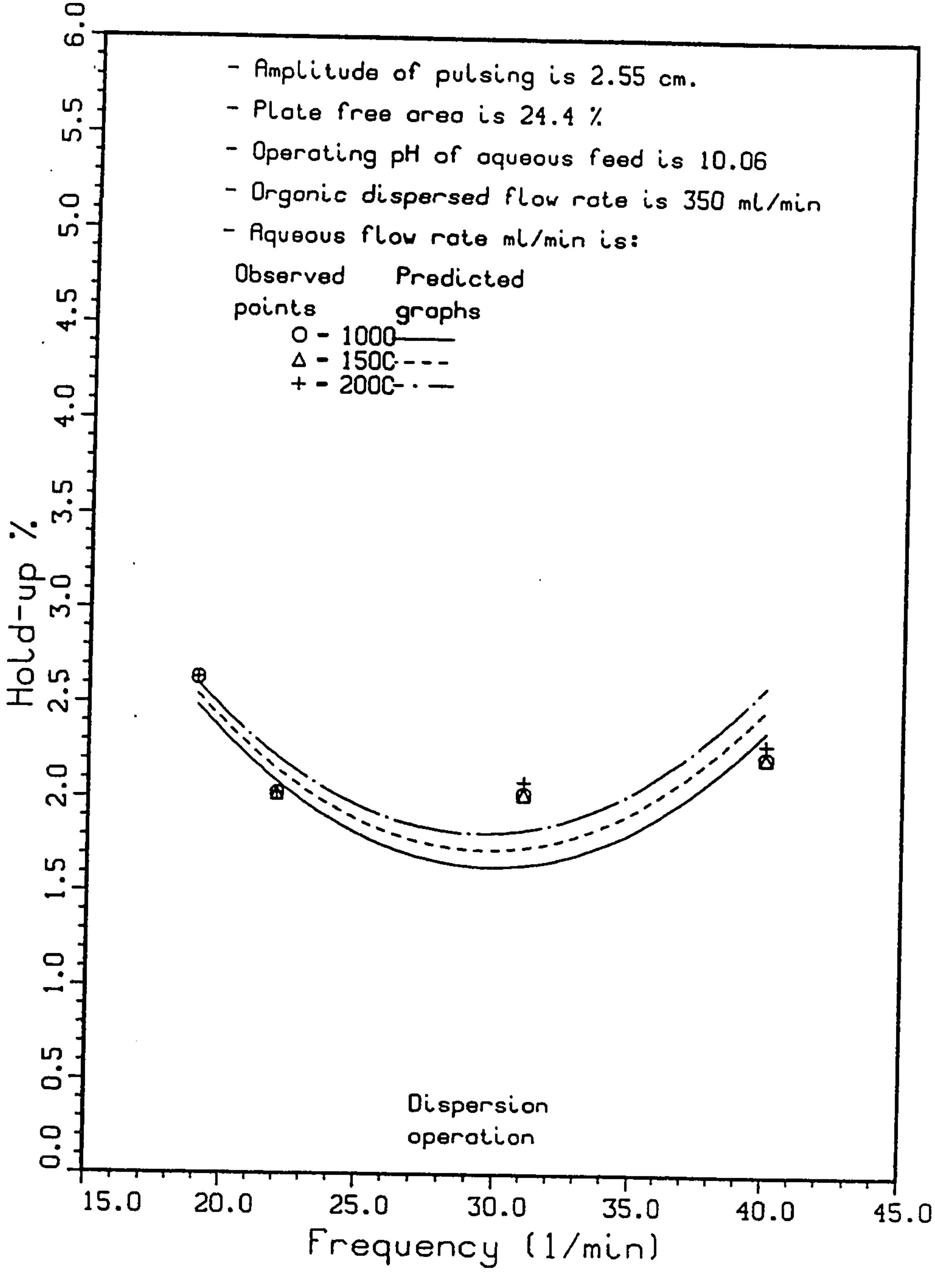

Figure B.16: Predicted and experimental hold-up % data of Set IV, clarified aqueous continuous phase (variable), organic dispersed phase (constant).

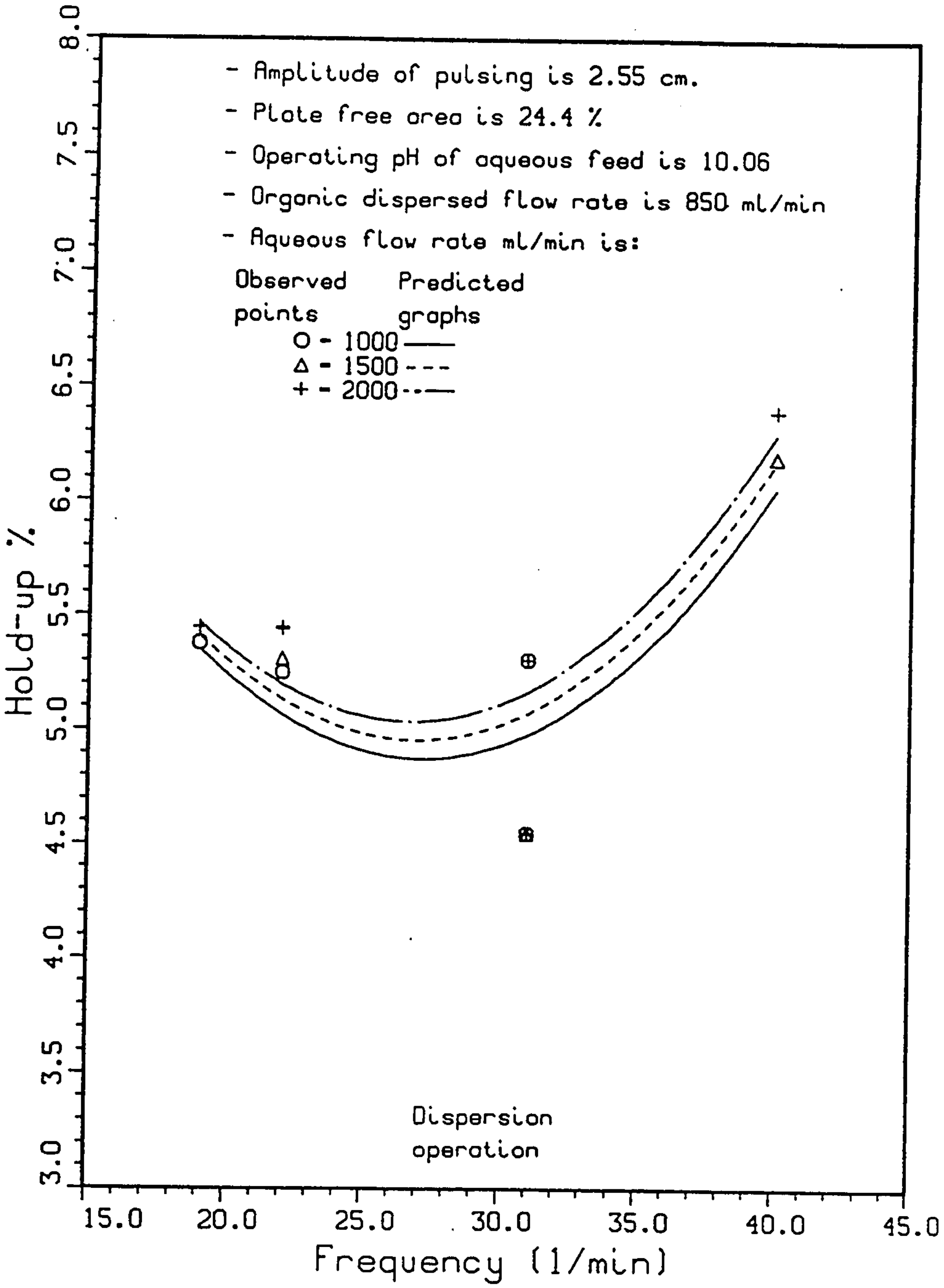

Figure B.17: Predicted and experimental hold-up % data of Set IV, clarified aqueous continuous phase (variable), organic dispersed phase (constant).

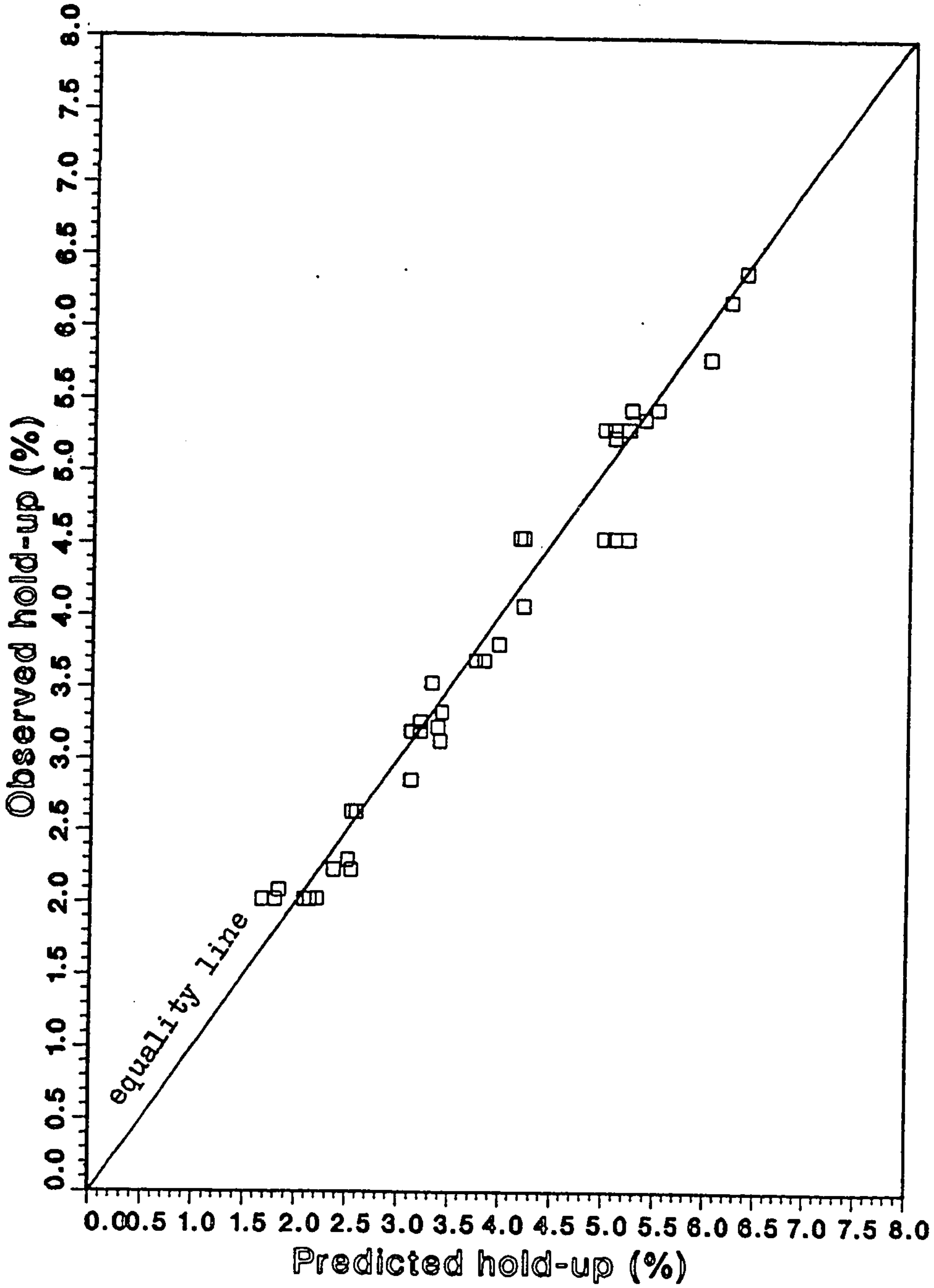

Figure B.18: Observed versus predicted hold-up % values of Set IV.

SET V

Conditions of Operation of Set V

- 10 % uranium tailings aqueous dispersed phase flow rates 792, 1049.6 and 1303.3 ml/min.
- Organic continuous phase flow rates 379, 579.1 and 778.8 ml/min.
- Pulse frequency 19, 22, 31 and 40 min^{-1}.
- Pulse amplitude 2.55 cm.
- Plate free area 24.4 %.
- Operating pH of the aqueous dispersed phase 4.04.

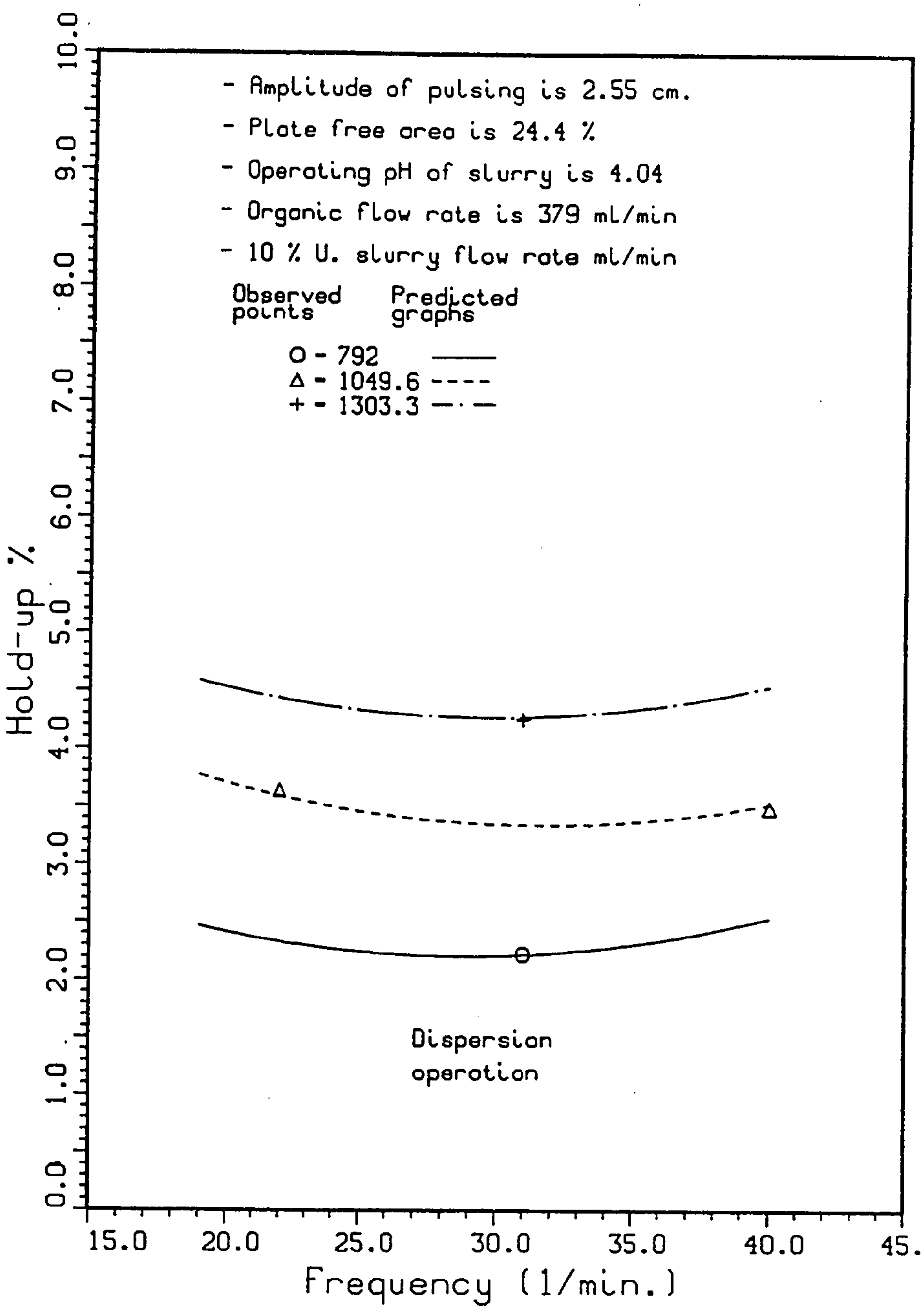

Figure B.19: Predicted and experimental hold-up % data of Set V, 10 % spent uranium tailings dispersed phase (variable), organic continuous phase (constant).

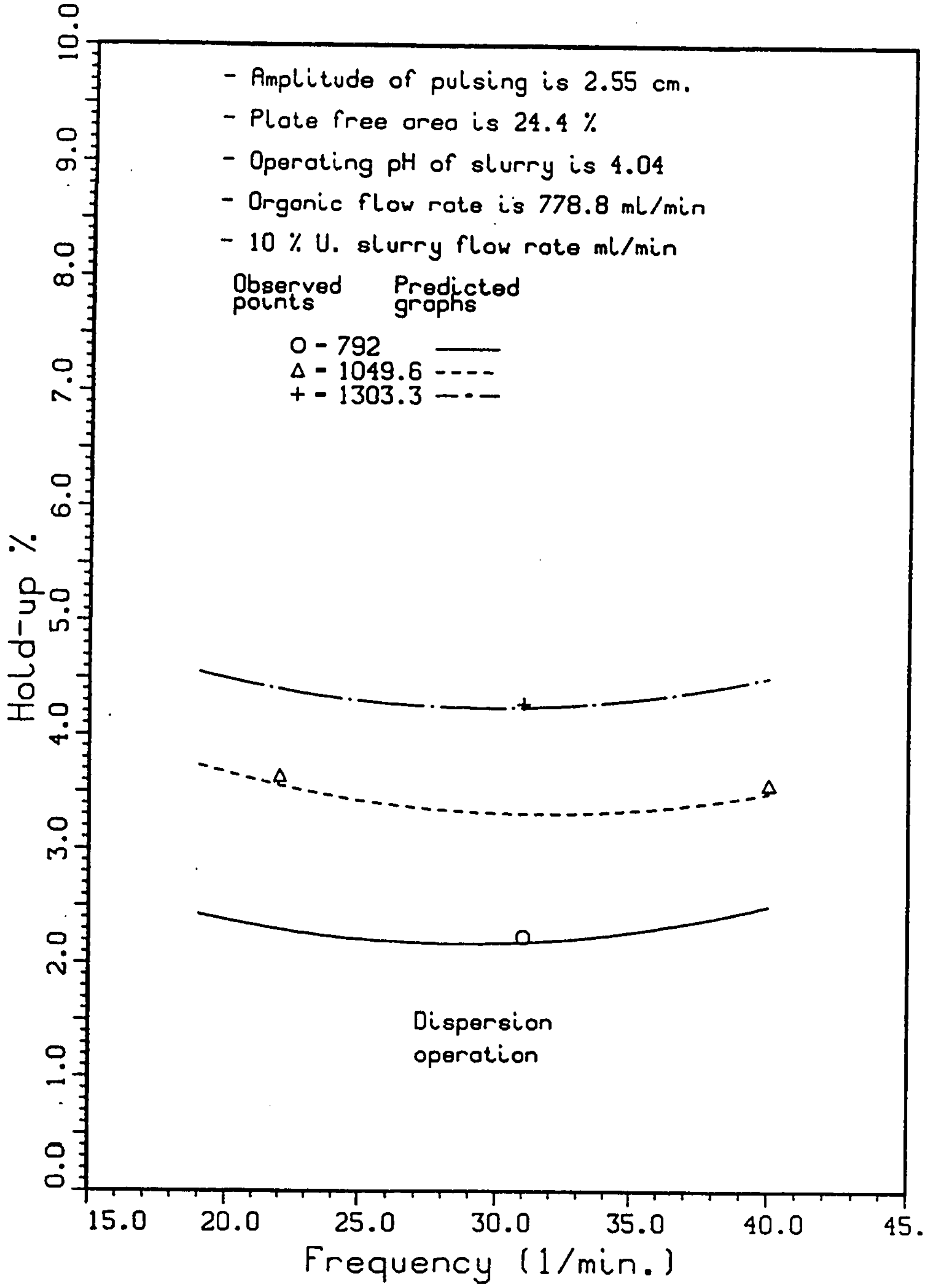

Figure B.20: Predicted and experimental hold-up % data of Set V, 10 % spent uranium tailings dispersed phase (variable), organic continuous phase (constant).

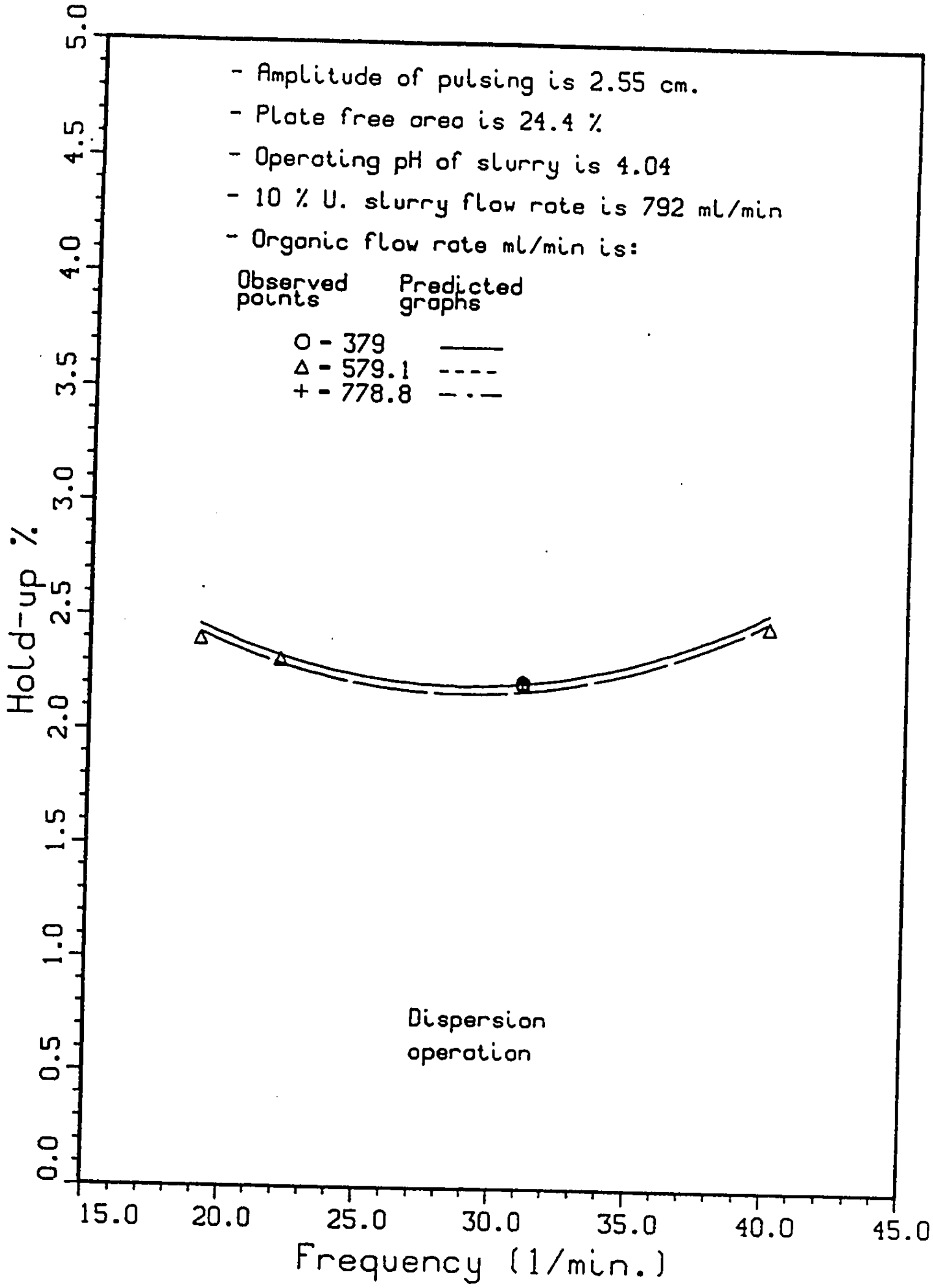

Figure B.21: Predicted and experimental hold-up % data of Set V, 10 % spent uranium tailings dispersed phase (constant), organic continuous phase (variable).

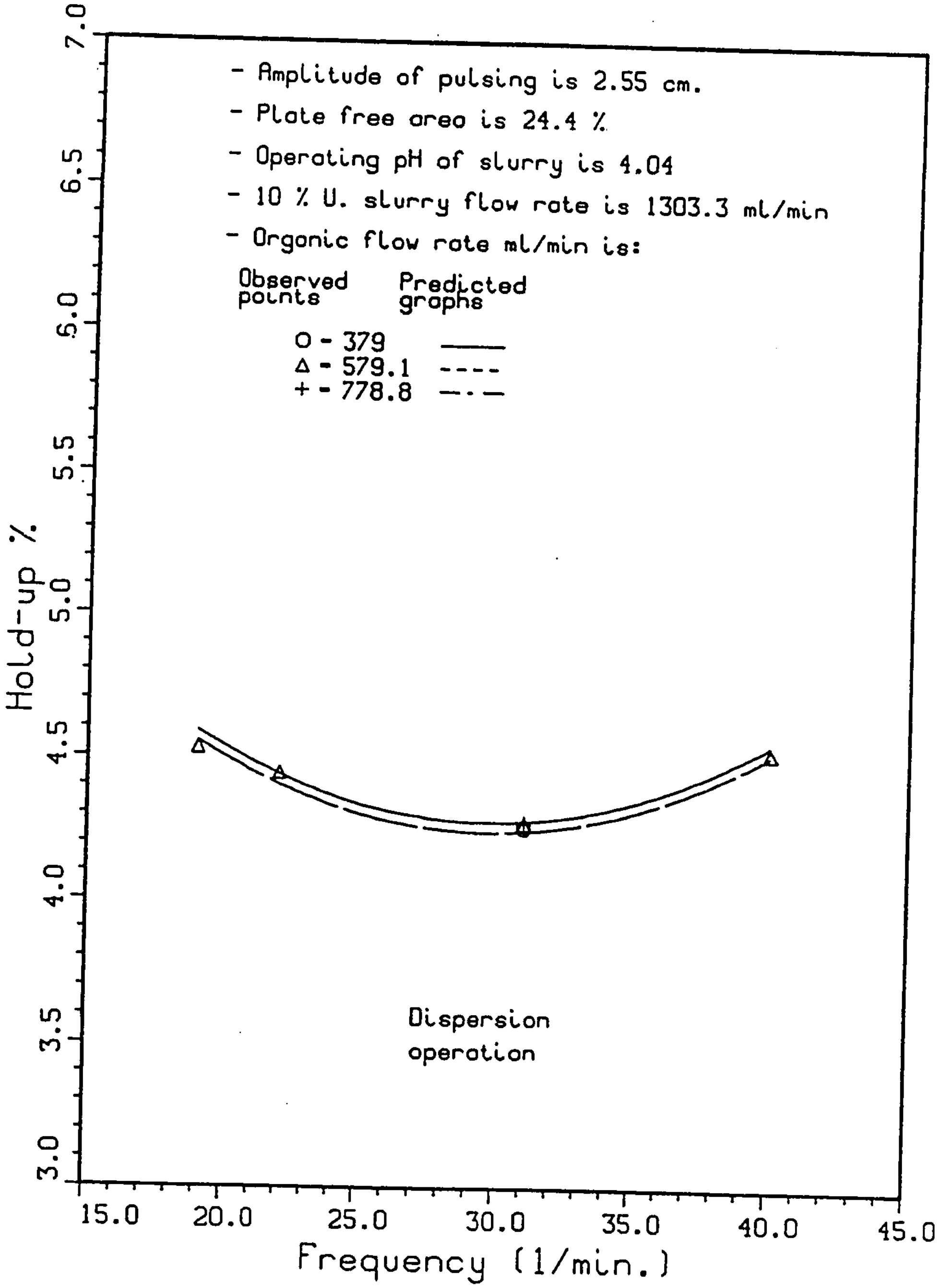

Figure B.22: Predicted and experimental hold-up % data of Set V, 10 % spent uranium tailings dispersed phase (constant), organic continuous phase (variable).

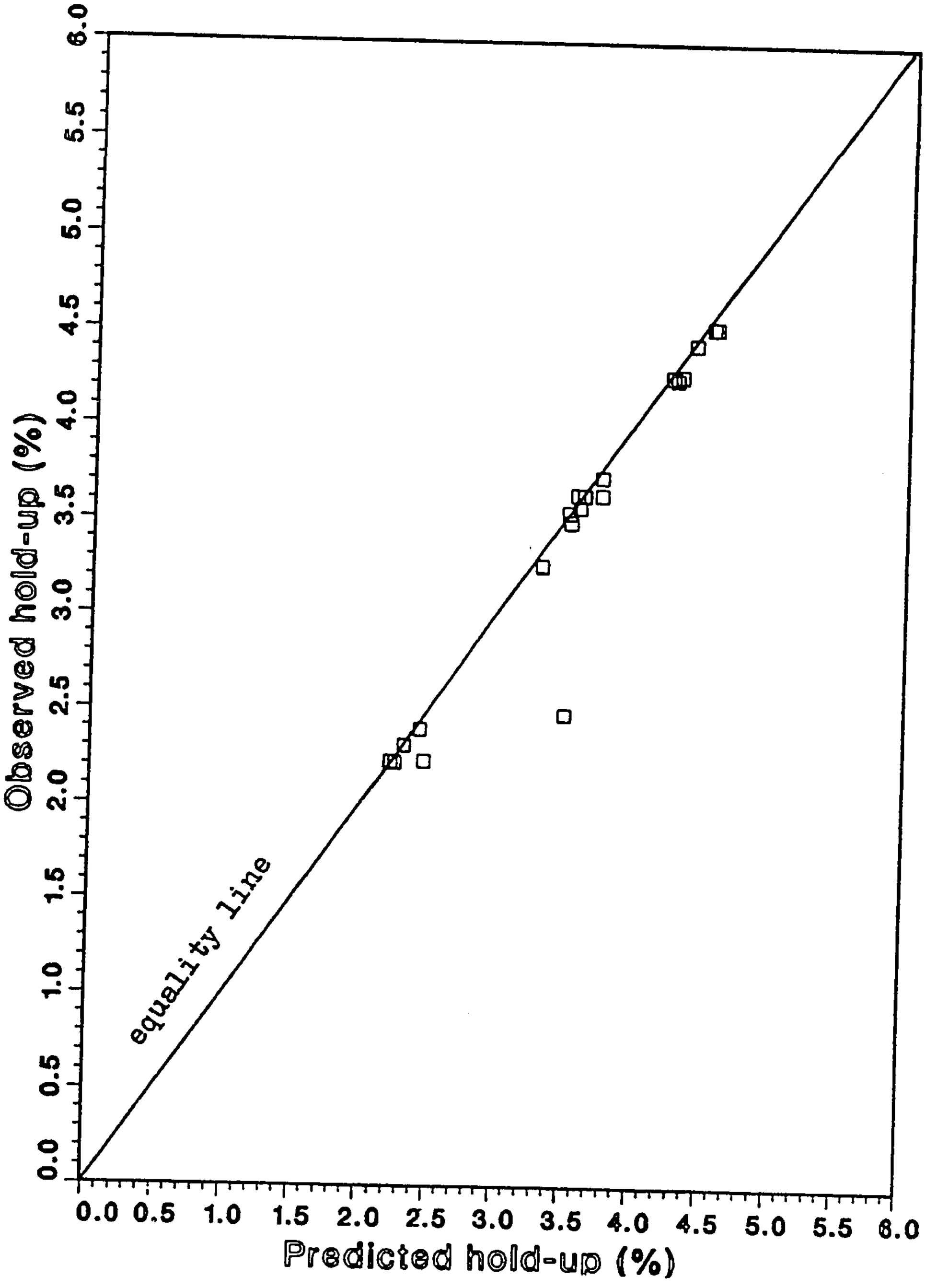

Figure B.23: Observed versus predicted hold-up % values of Set V.

SET VI

Conditions of Operation of Set VI

- 25 % uranium tailings aqueous dispersed phase flow rates 792, 1049.6 and 1303.3 ml/min.
- Organic continuous phase flow rates 379, 579.1 and 778.8 ml/min.
- Pulse frequency 19, 22, 31, 40, 51 and 60 min^{-1}.
- Pulse amplitude 2.55 cm.
- Plate free area 24.4 %.
- Operating pH of the aqueous dispersed phase 4.04.

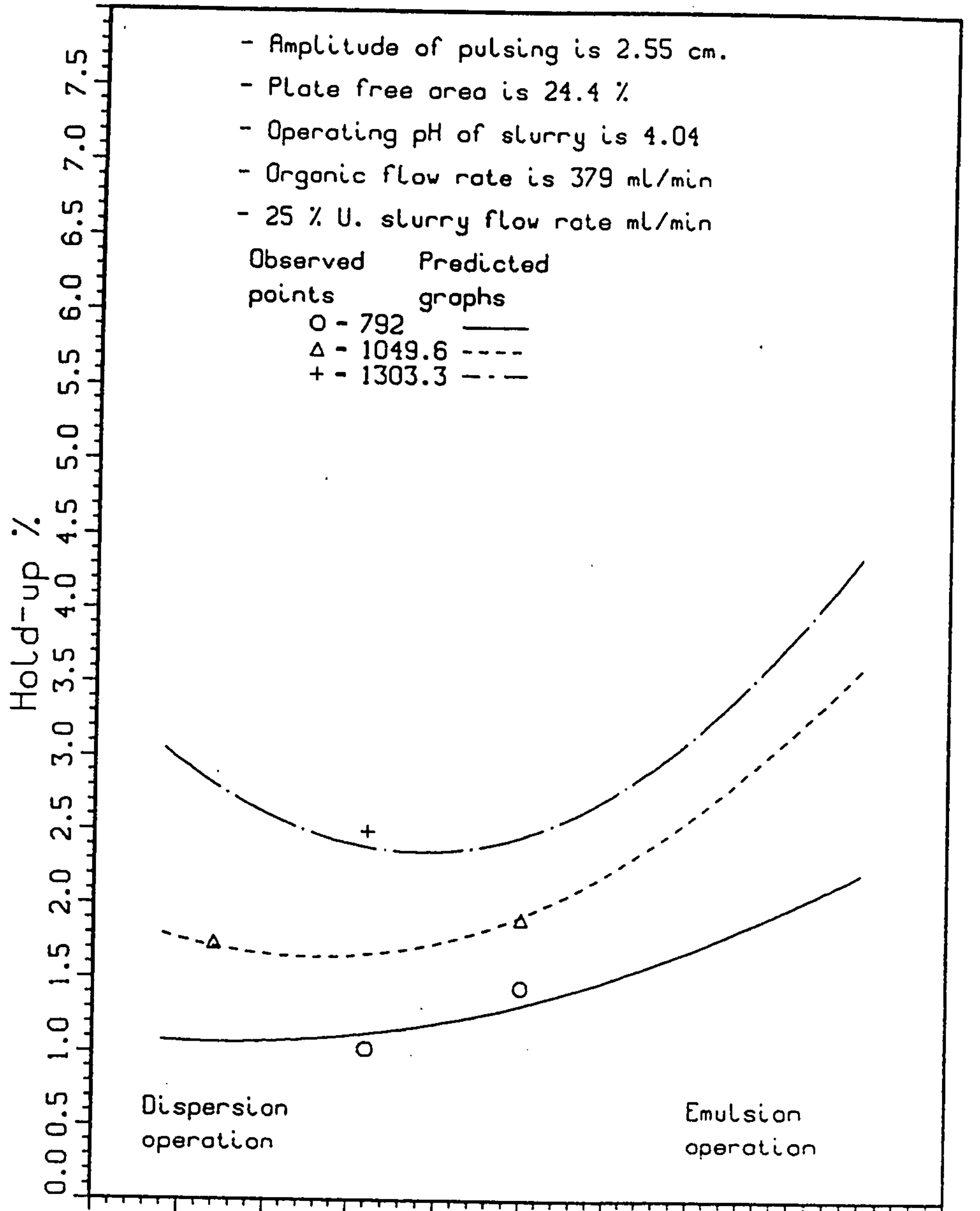

Figure B.24: Predicted and experimental hold-up % data of Set VI, 25 % spent uranium tailings dispersed phase (variable), organic continuous phase (constant).

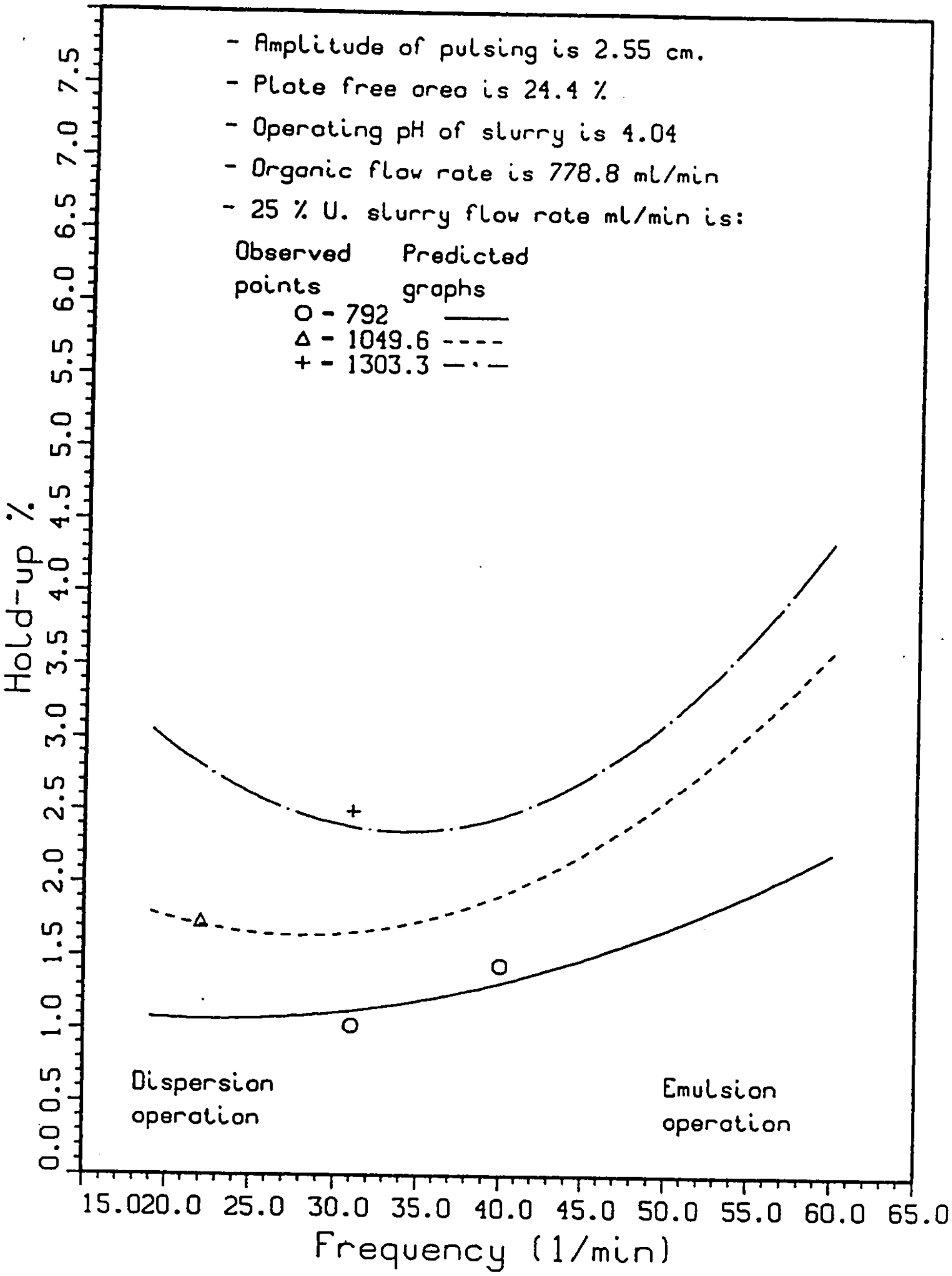

Figure B.25: Predicted and experimental hold-up % data of Set VI, 25 % spent uranium tailings dispersed phase (variable), organic continuous phase (constant).

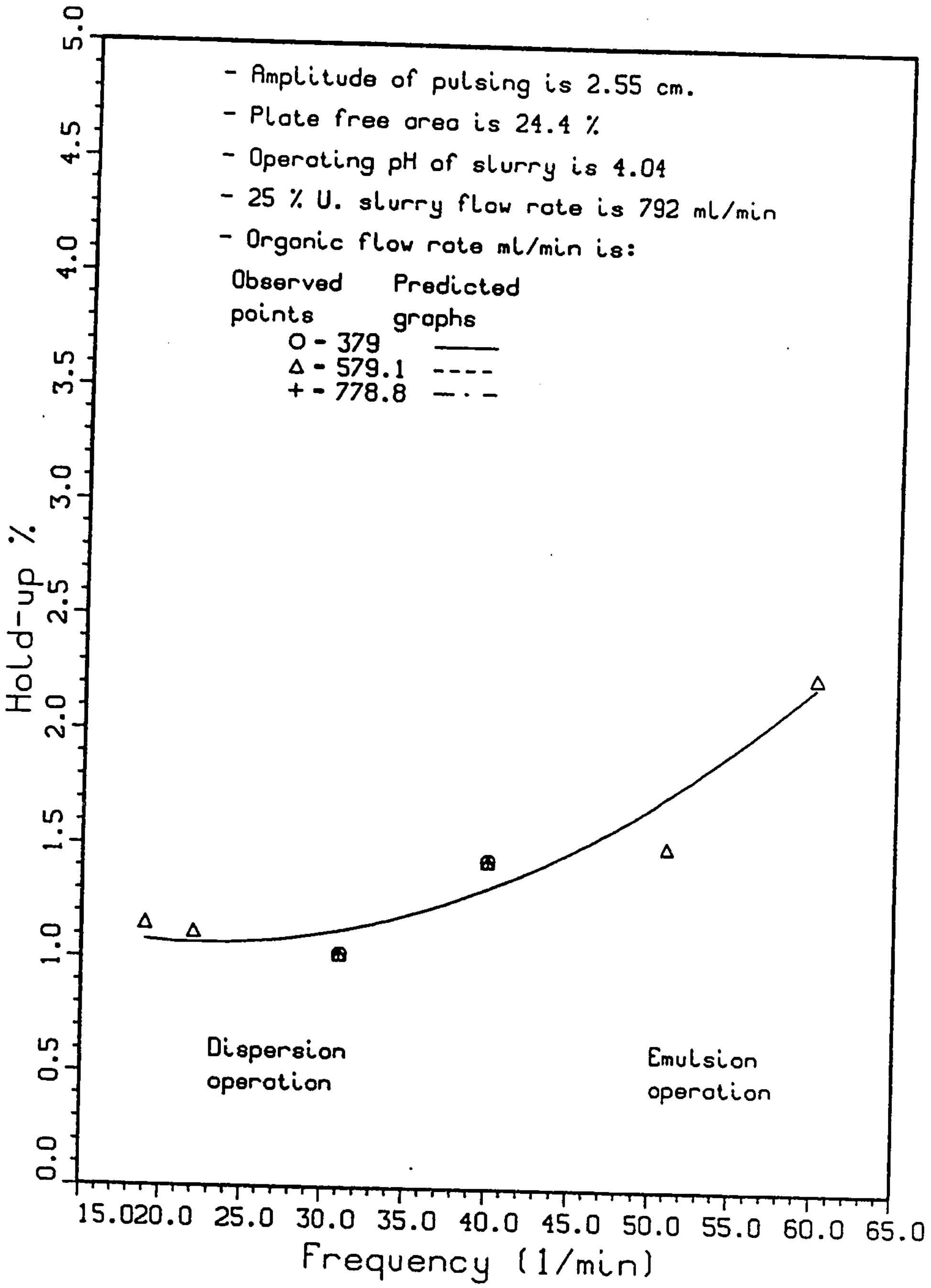

Figure B.26: Predicted and experimental hold-up % data of Set VI, 25 % spent uranium tailings dispersed phase (constant), organic continuous phase (variable).

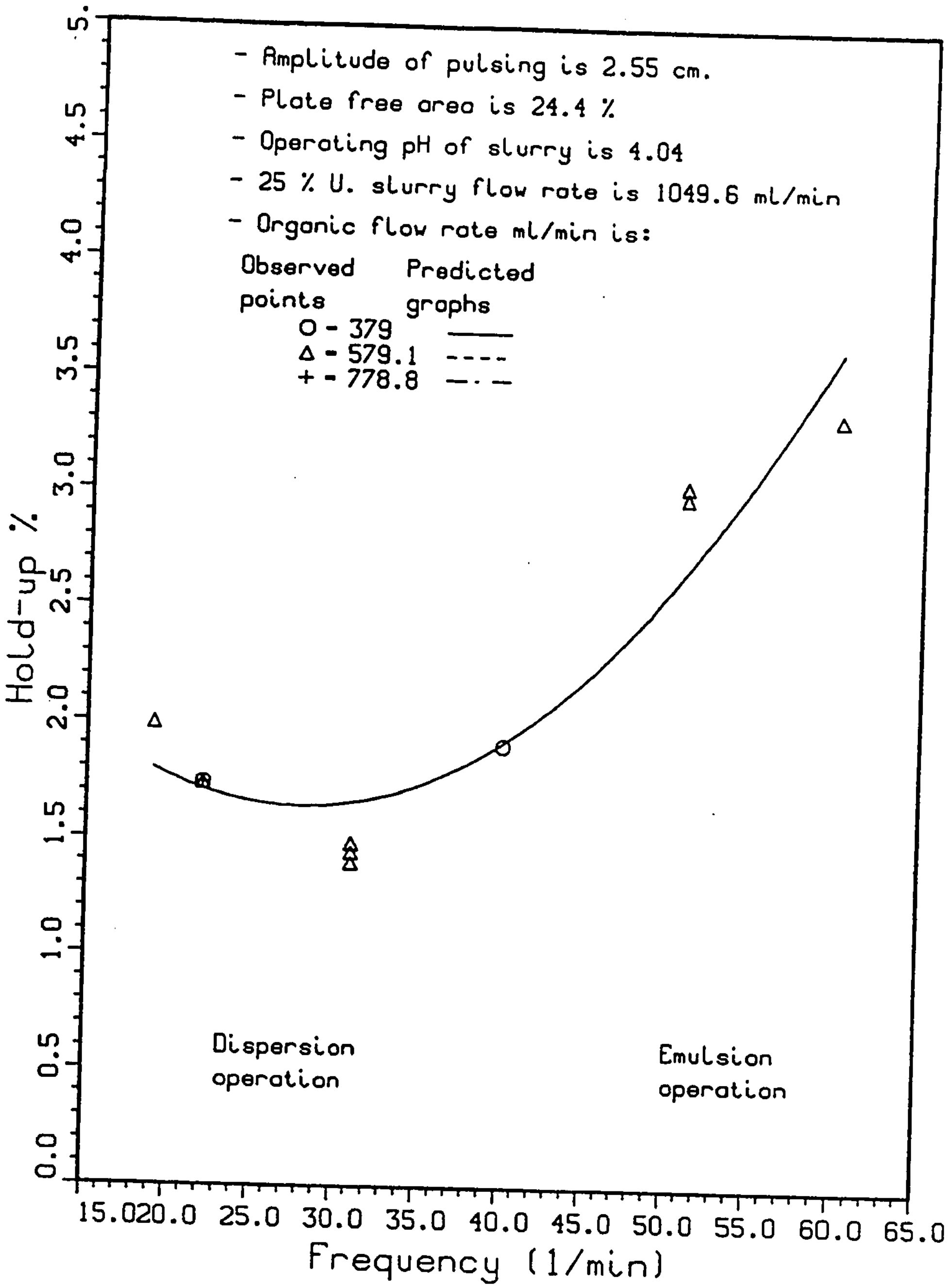

Figure B.27: Predicted and experimental hold-up % data of Set VI, 25 % spent uranium tailings dispersed phase (constant), organic continuous phase (variable).

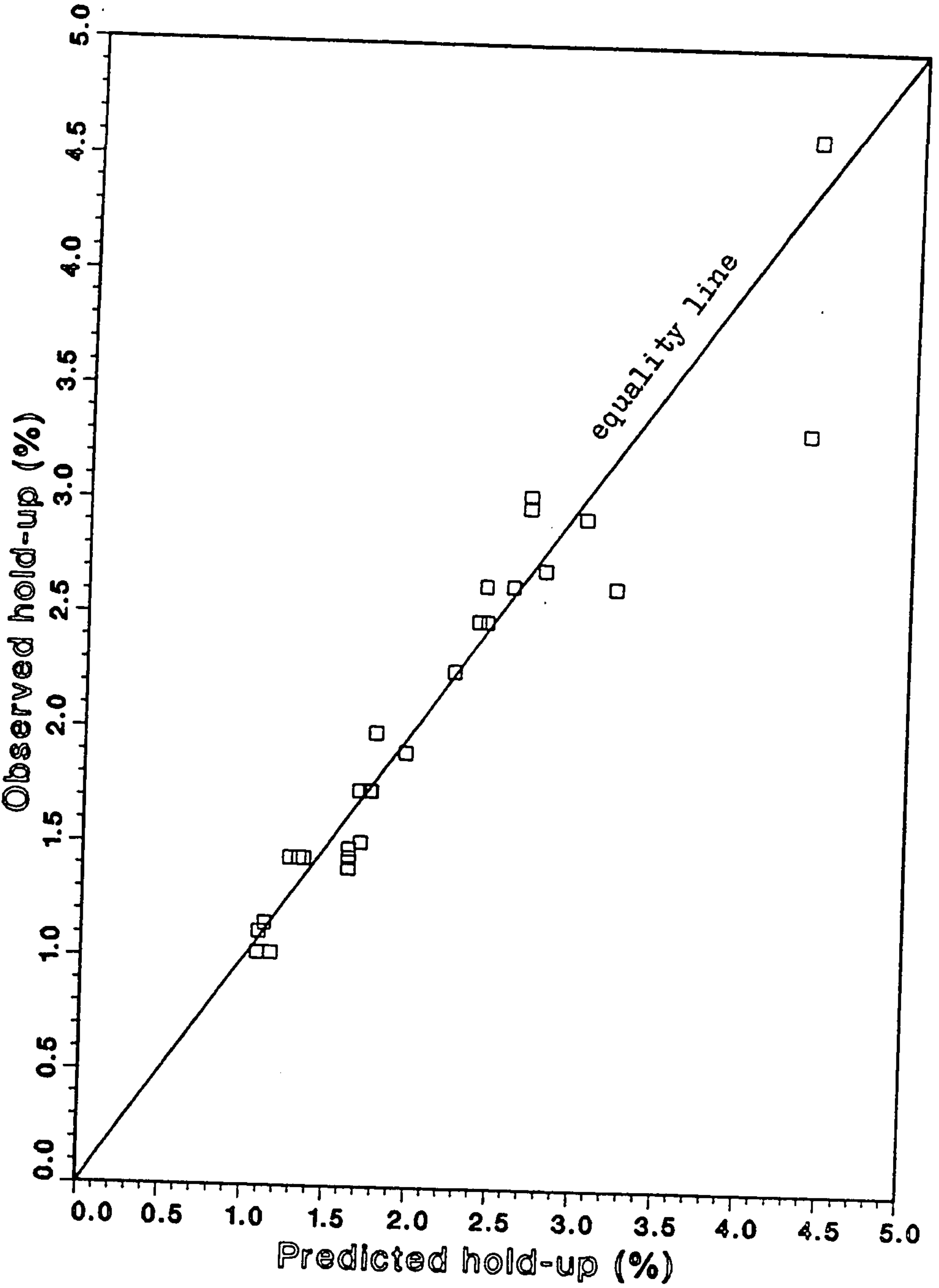

Figure B.28: Observed versus predicted hold-up % values of Set VI.

SET VII

Conditions of Operation of Set VII

- Organic dispersed phase flow rates 379, 579.1 and 778.8 ml/min.
- Clarified aqueous continuous phase flow rates 792, 1049.6 and 1303.3 ml/min.
- Pulse frequency 19, 22, 27, 31, and 40 min^{-1}.
- Pulse amplitude 2.55 cm.
- Plate free area 12.4 %.
- Operating pH of the aqueous continuous phase 10.1.

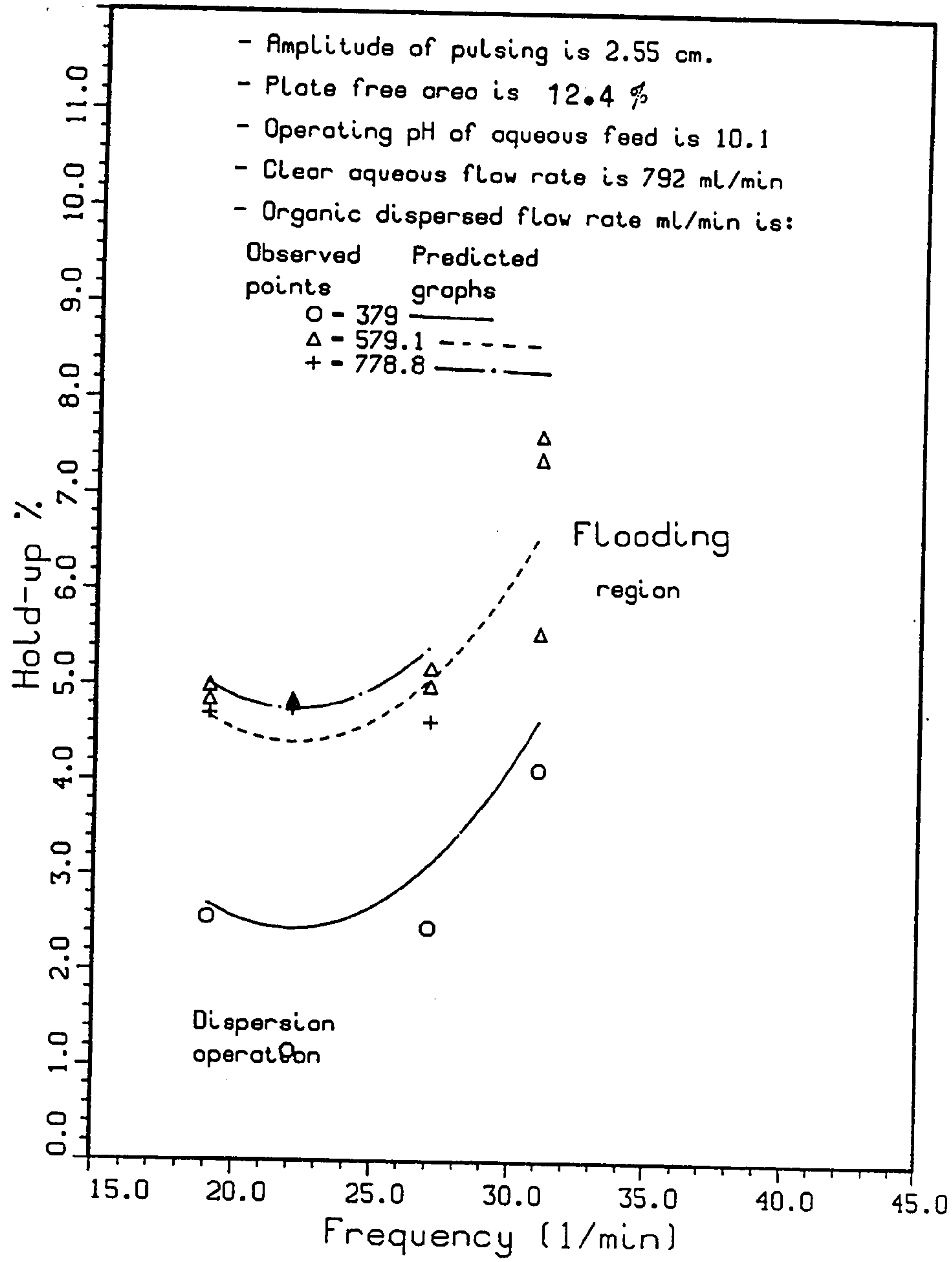

Figure B.29: Predicted and experimental hold-up % data of Set VII, organic dispersed phase (variable), clarified aqueous continuous phase (constant).

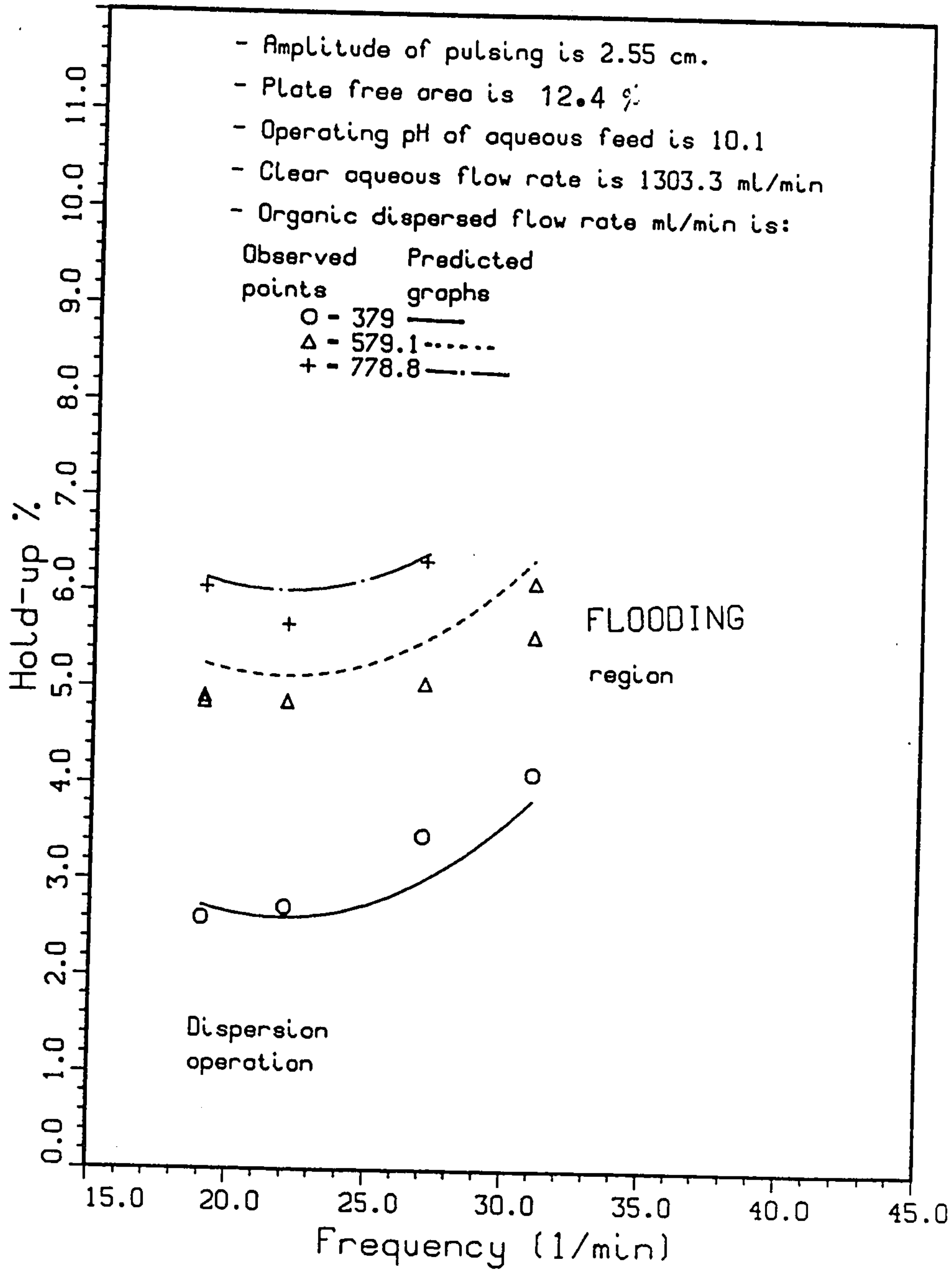

Figure B.30: Predicted and experimental hold-up % data of Set VII. organic dispersed phase (variable), clarified aqueous continuous phase (constant).

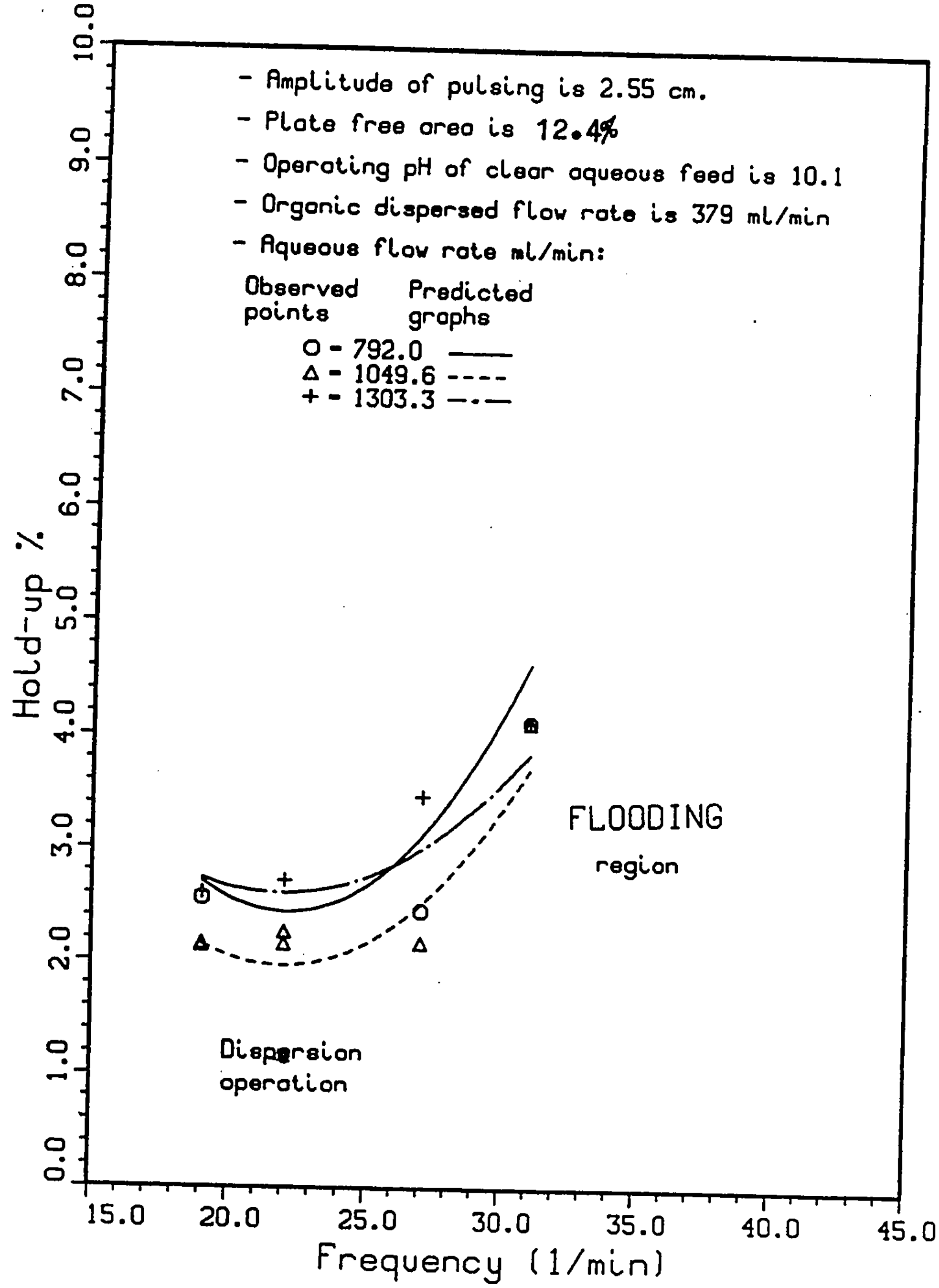

Figure B.31: Predicted and experimental hold-up % data of Set VII, organic dispersed phase (constant), clarified aqueous continuous phase (variable).

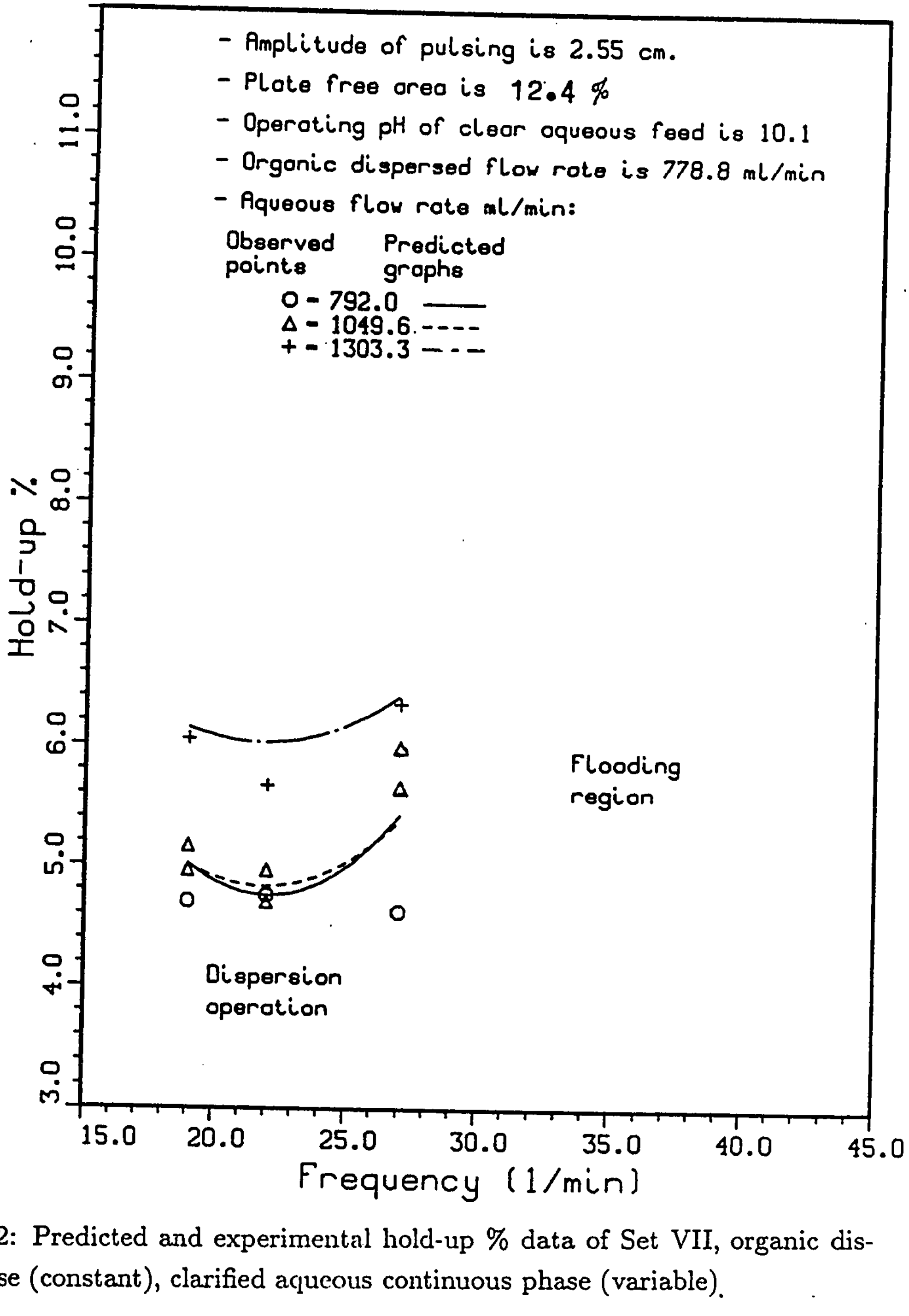

Figure B.32: Predicted and experimental hold-up % data of Set VII, organic dispersed phase (constant), clarified aqueous continuous phase (variable).