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*CONTINUOUS ION EXCHANGE
IN FLUIDIZED BEDS*

M. J. SLATER AND P. PRUDHOMME

EXTRACTION METALLURGY DIVISION

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CONTINUOUS ION EXCHANGE IN FLUIDIZED BEDS

by

M. J. Slater* and P. Prud'homme**

SUMMARY

A continuous ion-exchange plant using a multi-stage fluidized-bed extraction column coupled with moving-bed regeneration and wash columns has been built and tested. Hydrodynamic studies on resins and ore leach pulps have been made and a mathematical model has been developed to predict the performance of the extraction column.

Clarified solutions from the leaching of uranium ore and leach pulps of 5 and 10 w/o have been used with IRA 430 ion exchange resin.

The mathematical model requires refinement but its actual performance has been satisfactory for predicting the extraction of uranium.

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Direction des mines
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L'ÉCHANGE CONTINU D'IONS DANS DES
LITS FLUIDISÉS

par

M. J. Slater* et P. Prud'homme**

RÉSUMÉ

Les auteurs ont construit et ont mis à l'essai une installation d'échange continu d'ions en utilisant une colonne d'extraction à plusieurs étages de lit fluidisé; celle-ci étant couplée avec une colonne régénératrice et une colonne de lavage à lit mobile. Ils ont fait des études hydrodynamiques sur les résines et sur les fines de lixiviation des minerais; et ils ont développé un modèle mathématique pour prédire le rendement de la colonne d'extraction.

Ils ont employé des solutions clarifiées de la lixiviation du minerai d'uranium et des fines de lixiviation de 5 et 10 w/o*** avec la résine d'échange d'ions (IRA-430).

Les auteurs ont trouvé que c'était nécessaire d'améliorer le modèle mathématique mais que son rendement actuel était satisfaisant pour prédire l'extraction de l'uranium.

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***eau/organique

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INTRODUCTION

Continuous ion-exchange equipment is now being used industrially for water treatment^(1,2) and metals recovery⁽³⁾ on a large scale. In most applications, a moving packed bed of resin flows counter-currently to the solution in a cycle of operations rather than in a truly continuous manner. This has been found necessary to control the flow of resin.

The moving packed-bed type of equipment is not necessarily suitable for all ion-exchange processes. It is considered that a series of fluidized or otherwise agitated beds of resin may be a more economic approach to equipment design in two cases; the first is when ion exchange conditions are very favourable and few theoretical stages or transfer units are required for achieving specified product stream compositions, and the second is when solutions contain particulate material that is difficult or expensive to remove^(4,5).

A pilot plant (Figure 1) has been built comprising a multi-stage column of fluidized beds (based on a U.S. Bureau of Mines design⁽⁶⁾) coupled with intermittently moving-bed regeneration and wash columns. The plant was used to test methods of transferring resin between columns and to obtain experimental data on extraction of uranium from unfiltered ore leach pulps and of other metals from mine drainage waters.

The extraction column is operated with upflow of solution or pulp for a set time period, this is followed by drainage of resin and solution out of the base of the column

for a certain time during which solution feed is stopped. Flow of resin and solution is co-current downwards through each stage during the resin transfer. The resin is transferred to the top of the regeneration column. A level controller senses the increased resin inventory in the regeneration column as the resin from the extraction column is received. It allows transfer of resin from the base of the regeneration column, through the wash column and up to the top of the extraction column until the resin level in the regeneration column reaches a set point.

Under most conditions envisaged, the extraction ion-exchange process is rate-controlled by liquid film diffusion, and favourable but non-linear equilibrium isotherms are encountered.

To gain insight into the performance of the extraction column, hydrodynamic studies on resin and pulp in fluidized beds were made, and a simplified calculation procedure was developed for mass transfer performance. A simple experimental technique of measuring the mass transfer rate was used; this was based on the same assumptions concerning flow patterns and equilibria so that the derived mass transfer coefficient contained the effects of the inadequacies of the mathematical model compared to the real situation. Marchello has considered the case of a series of stirred vessels containing resin with continuous liquid flow; some of the basic equations used in that work are utilized here⁽⁷⁾.

CONSTRUCTION AND OPERATION OF THE PLANT (FIGURE 1)

The extraction column had seven 24 x 2-inch sections of Kimax glass tube. Between the sections were fitted polythene blocks with a 0.5-inch hole drilled centrally through each; these blocks act as liquid distributors between the stages. The top two sections did not have a polythene block between them.

Liquid or pulp was metered by means of a peristaltic pump or rotameter. A tee was placed next in line with an automatic valve on the side arm to dump feed to a tank instead of allowing it to flow into the column during resin removal. A one-way valve was fitted, vertically, immediately below where the feed entered the base of the column. The lines and fittings used were generally 0.5 inch in diameter.

The overflow of liquid or pulp from the top of the column passed into a small tank and then to drain. Liquid from this tank was used in the resin feed hopper.

Resin was drained from the extraction column through a 0.5-inch line into a tee. Water or feed solution was pumped into the tee through a screen to transport resin to the vibrating screen where resin and solution were separated. The solution was recycled. The size of resin increments removed and the removal frequency were controlled by timers operating an automatic ball valve on the resin removal line. This type of valve was used for expediency and is not recommended, although no serious attrition of resin was observed as a result of using it.

Resin feed to the top of the extraction column was achieved using an auxiliary vessel. Vacuum or air at 5 psig could be applied to the top of this vessel alternately through a three-way electrical solenoid valve receiving signals from the level controller on the regeneration column. Two more input lines were connected to the top: a resin feed line from the base of the wash column and another line for "dilution" liquid obtained from the product overflow holding-tank. One outlet line at the base of the vessel was fitted with an automatic ball valve operated simultaneously with the air/vacuum three-way valve. When the resin in the regeneration column rose above the level controller and blocked the light path for a set time, suction was applied to the resin hopper and the outlet valve was closed. Resin and liquid in suitable proportions were sucked into the vessel. Meanwhile the resin level in the regeneration column fell below the level controller and, after a set delay time, vacuum was cut off, air was applied, and the outlet valve on the resin hopper was opened. Resin was pushed out of the vessel and was followed by a rinse of solution so that the outlet valve did not crush resin while it was closing.

The resin was discharged to a funnel leading into the top section of the column for a distance of 12 inches, through a rigid 0.75-inch pipe.

The regeneration column was a 48 x 4-inch glass tube. The diameter was out of proportion to the extraction column because of the difficulty of handling large flows in a large-diameter extraction column. The regeneration column was merely

regarded as a source of highly regenerated resin and no attempt was made either to optimize the size of the column or to operate with minimum use of regenerant. (Recovery of regenerant was expected in most process applications).

Resin entered the top of the regeneration column from a vibrating 100-mesh screen and was flushed down a 1-inch pipe with either water or, preferably, the eluate being partly circulated to avoid eluate dilution. The resin in the column was in a packed condition. Resin was removed from the regeneration column through a 0.5-inch pipe directly into the wash column. The wash column was 2 inches in diameter and 24 inches high. Water entered the base of the wash column through a screen and flowed upwards into the regeneration column where it was joined by a stream of concentrated eluant. Both streams continued upwards to leave the regeneration column at the top. The resin in the wash column was generally in a loosely packed condition and, when suction was applied, it flowed freely downwards in the regeneration and wash columns and left at the base of the wash column.

Starting-up required manual adjustment of the amount of resin required in the various columns. The resin in the extraction column was allowed to settle completely in the bottom stages, then was expanded until a steady fluidized bed of resin was achieved, with the top reaching just into the top section so that a freeboard of about 24 inches was available. Operation was then switched over to automatic control and fine adjustments were made where necessary.

THE FLUIDIZATION OF ION EXCHANGE RESINS
WITH ORE PULPS

1. Background information

Two types of pulp were used. In early work, pressure-leached uranium ore solids from Consolidated Denison Mines were used in water. In later work involving extraction of uranium, ore leach pulp was obtained from Rio Algom Ltd., at Elliot Lake.

To calculate the performance of a multi-stage fluidized-bed ion exchange column, the particle size range of the solids and their fluidization (or sedimentation) characteristics were required. The maximum size of pulp solids transported upwards and the concentration of solids in pulp flowing upwards through the fluidized beds of resin could then be determined. Larger pulp solids, unsupported by upward pulp flow, would be expected to travel downwards with the resin and be discharged with the resin at the base of an extraction column.

The size distributions of pulps used are given in Tables 1 and 2.

2. Sedimentation and Fluidization tests

Fluidization and sedimentation experiments yield the same information on the velocities of solid particles relative to the surrounding liquid, as a function of concentration of solids and of range of particle size in the mixture. Sedimentation is easier in practice for pulp solids, but fluidization is easier for resin particles which have much higher falling velocities.

The pressure-leached solids were used for sedimentation tests. The solids were dried and pulps containing 5, 10, 20, and 30 wt % solids were prepared in water. Graduated cylinders were filled with pulp up to a given mark and then shaken to disperse the solids. The height of the solids/water interface was measured at various times until the solids had settled. For dense pulps of a close size range a sharp interface was seen but for dilute pulps and the unsieved solids the interface was less certain. Very obvious segregation took place with the unsieved pulp even at high pulp concentrations.

Plots of interface height versus time indicated a constant falling velocity until solids were slowed down in the region of dense packing just before complete settling. The composition of the falling pulp is assumed unchanged in the region of constant falling velocity.

The results were treated according to the method of Richardson and Zaki⁽⁸⁾ by plotting falling velocity against volume fraction of liquid (voidage) between the particles. The results are given in Table 3. Normally a straight line is obtained on logarithmic co-ordinates with a slope which is a function of the particle Reynolds number (Re_p) if the particle size is uniform (or at least not spread over a wide range). The intercept at zero solids concentration should be the terminal velocity (V_T) in an infinite medium which can be calculated from Figure 2. Reasonable agreement between extrapolated and calculated results is shown except for the 150 to 200-mesh fraction (Figure 3).

The equations of the sedimentation lines are, in theory, of the form

$$V_s = V_T e^n$$

where the correlations for n are

$$\begin{aligned} n &= (4.6 + 20 \frac{d}{D}) && \text{for } 0 < Re_p < 0.2 \\ n &= (4.4 + 18 \frac{d}{D}) (Re_p)^{-0.03} && \text{for } 0.2 < Re_p < 1.0 \\ \text{and } n &= (4.4 + 18 \frac{d}{D}) (Re_p)^{-0.1} && \text{for } 1 < Re_p < 200 \end{aligned}$$

The measured slopes are in agreement with theory allowing for the poor accuracy of slope measurement.

The slope of the line for particles finer than 325 mesh (44 μ) is high because of the wide particle size range from 44 μ to much less than 10 μ . This is substantiated by the curve for the mixture of pulp solids for which the interface level observed is that for the finest solids in the mixture. Extrapolation of this curve gives a terminal velocity appropriate to a size of about 20 μ .

The theoretical approach breaks down for pulps containing 5 wt % solids or less (2 vol % or less) because turbulence in the liquid at the beginning of an experiment slows the fall of particles. In denser pulps, such turbulence is damped out quickly.

Earlier work with ion exchange resins⁽⁹⁾ has given confidence that no experimental work is required to check the prediction of the velocity/voidage relationship for different sizes of resin. Experimental data for various resins are shown on Figures 4, 5, 6, 7, and 8 and in Tables 5 and 6; the data

may be closely matched by applying the equations to each size fraction, and the average velocity can be found for various void-ages. If the size distribution of resin is not large the equations may be applied, using the Sauter mean particle diameter and mean Reynold number. The terminal velocity, V_T , should be as high as possible for economic operation of a fluidized system. The velocity is dependent on resin size and density, and on pulp density and viscosity.

3. Terminal velocities and particle sizes

The terminal velocities of particles may be plotted as a function of particle diameter (Figure 9). The experimental work on pulp solids and resins has shown that existing theory may be used in these calculations.

The pulp solids will have lower velocities while surrounded by other solids (due to hindrance), so corrected velocities are shown on Figure 9 for a pulp (10 wt % solids), equivalent to 0.960 voidage between pulp particles. The correction to velocity varies with particle size but is small in all cases.

The terminal velocity of the smallest resin bead must not be exceeded by the liquid or pulp superficial velocity if loss by elutriation is to be avoided. In practice, resin is employed in fluidized beds in the pulp contactor proposed, and lower liquid velocities have to be used to maintain an adequate hold-up of resin in each stage. Earlier work has established that a voidage between resin beads of 65 to 85 % gives best mass transfer results; the fluidization velocity under these circumstances is shown on Figure 9 for IRA 430 resin in the chloride form, which

will limit the flow rate due to a density lower than that for resin in the uranium-loaded form.

A small range in resin bead diameter and a high bead loading is hydrodynamically desirable. However, an initial loading of virtually zero is required to ensure low losses of metal in the barren effluent.

4. Viscosity of pulps in a fluidized bed

The variation of viscosity of uranium ore pulps and slimes (after gold extraction) with solids concentration has been reported in several cases with widely differing results. The viscosity is very important in determining terminal velocities of resin beads in the pulp and, hence, flow rates of pulp possible for a given degree of expansion of a fluidized bed of resin.

The viscosity of a liquid-solids slurry has been expressed in various ways for dilute mixtures, subject to Newtonian hydrodynamics, and empirically as a power law function of concentration for dense, non-Newtonian slurries. At present, any pulp to be processed has to be investigated experimentally under conditions appropriate to the equipment used for extraction (by ion exchange or solvent extraction) to determine the relationship between viscosity and solids concentration. If shear conditions in a pulp that exhibits non-Newtonian flow characteristics are not matched when measuring viscosity, the result may be invalid.

A bed of ion exchange resin was fluidized with water, glycerol solutions and pulps of various concentration and solids size range. Only those experiments in which pulp solids were not

trapped in or under the resin bed (due to insufficient liquid velocity to support them) were used.

The wash column of the continuous ion exchange loop was used to simulate a 24-inch section of the extraction column. The terminal velocities were extrapolated from the data (Figures 5, 6), a pulp density was assumed and a pulp viscosity was then calculated (Table 6). It is evident from the results that viscosities are relatively low over the range of pulp densities that can be handled by normal resins.

The viscosity is not significantly affected by varying the size distribution (a large experimental error was incurred which disguises small variations). Other ores may not give the same results as shown here.

5. Critical pulp solids size

It is seen in Figure 9 that, for a given resin of known size range and density used in a fluidized bed with a specified voidage between resin particles, the maximum superficial velocity allowable to void elutriation of the smallest beads may be determined.

For pulps of any density and viscosity, the terminal velocities of the different particle sizes in the pulp may be calculated, and the maximum pulp particle size that can be supported by a given superficial pulp velocity can be determined.

In a multi-stage column operated cyclically, any pulp solids in the feed pulp, larger than can be supported at the superficial velocity used, will be trapped in the lowest bed of resin. The amount trapped in a given time of flow can be

calculated from the pulp flow rate, and the fraction of solids larger than the critical size can be estimated, as described above. The pulp solids trapped will be removed with resin periodically from the base of the column.

In the case of a Rio Algom pulp (10 wt % solids), particles larger than about 60 μ , comprising about 15 % of the pulp solids in the feed, should be trapped to be discharged with resin. However, at some point in the system, these coarse pulp solids must be removed and washed, otherwise build-up in the feed system will occur.

CALCULATION OF MASS TRANSFER PERFORMANCE OF THE EXTRACTION COLUMN

1. Mathematical model

The extraction column is to process dilute solutions, so the mass transfer rate will usually be controlled by the diffusion rate of ions through a liquid film surrounding each resin bead. In the case of extraction of large complex ions such as uranium sulphates, the rate may be partly controlled by diffusion in the resin bead: this case would be difficult to analyse.

If the liquid film controls the extraction rate, and the rate is assumed proportional to a linear function of a driving force, we may write the rate equation

$$\frac{dq}{dt} = K_{OL} a (C - C^*) \dots\dots\dots (1)$$

If the resin controls the rate, a similar equation cannot be written without serious error because the rate is

dependent on both resin composition, q , and liquid composition, C .

The equilibrium relationship is written as

$$q = mC^* \dots\dots\dots (2)$$

which will normally have to be obtained by experiment.

The third requirement for the analysis is a statement of the partial mass-balance relationship which depends on the flow patterns of both liquid and resin in the extraction column. The flow of liquid through a fluidized bed and the circulation of particles in the bed is complex. In order to analyse the situation, it is assumed that, for each stage, the liquid and resin are completely mixed. An assumption of plug flow of liquid through a mixed bed of resin is probably more accurate but more difficult to handle mathematically.

The mass balance proposed for one stage is therefore

$$V_R dq = L_f (C_{IN} - C_{OUT}) dt \dots\dots\dots (3)$$

Now from (1)

$$\frac{d^2q}{dt^2} = K_{OL} a \left[\frac{dC_{OUT}}{dt} - \frac{dC^*}{dt} \right] \dots\dots\dots (4)$$

From (3)

$$\frac{dq}{dt} = \frac{L_f}{V_R} (C_{IN} - C_{OUT}) \dots\dots\dots (5)$$

or

$$\frac{d^2q}{dt^2} = \frac{L_f}{V_R} \cdot \left[\frac{dC_{IN}}{dt} - \frac{dC_{OUT}}{dt} \right] \dots\dots\dots (6)$$

and from (2) and (3)

$$\frac{dC^*}{dt} = \frac{1}{m} \cdot \frac{dq}{dt} = \frac{L_f}{mV_R} (C_{IN} - C_{OUT}) \dots\dots\dots (7)$$

$$\frac{L_f}{V_R} \cdot \left[\frac{dC_{IN}}{dt} - \frac{dC_{OUT}}{dt} \right] = K_{OL} a \left[\frac{dC_{OUT}}{dt} \right] - \frac{K_{OL} a L_f}{m V_R} \cdot (C_{IN} - C_{OUT}) \quad \dots (8)$$

or

$$\frac{dC_{OUT}}{dt} \cdot \left[K_{OL} a + \frac{L_f}{V_R} \right] + \frac{K_{OL} a L_f}{m V_R} \cdot C_{OUT} = \frac{L_f}{V_R} \cdot \frac{dC_{IN}}{dt} + \frac{K_{OL} a L_f}{m V_R} \cdot C_{IN} \quad \dots (9)$$

or

$$\frac{dC_{OUT}}{dt} \cdot \left[1 + \frac{L_f}{K_{OL} a V_R} \right] + \frac{L_f}{m V_R} \cdot C_{OUT} = \frac{L_f}{K_{OL} a V_R} \cdot \frac{dC_{IN}}{dt} + \frac{L_f}{m V_R} \cdot C_{IN} \quad \dots (10)$$

or

$$(1 + \alpha) \cdot \frac{dC_{OUT}}{dt} + \beta C_{OUT} = \alpha \frac{dC_{IN}}{dt} + \beta C_{IN} \quad \dots (11)$$

For a multi-stage system, $C_{IN,n} = C_{OUT,(n-1)}$, therefore, if Equation (11) is written for every stage, the set of equations may be solved if boundary and initial conditions are specified. The initial conditions of liquid compositions leaving each bed of resin at the start of a flow cycle, after start-up transients have decayed, are not known immediately, so an analytical solution using a Laplace transform method is not practicable.

For each stage, the initial condition of the liquid outlet composition can be linked to the initial liquid inlet composition, which can be related back to the composition of the liquid feed to the first stage.

Using Equations (1) and (3)

$$K_{OL} a (C_{OUT} - C^*) = \frac{L_f}{V_R} \cdot (C_{IN} - C_{OUT})$$

For initial conditions for all stages,

$$C_{OUT\ i} \left(K_{OL} a + \frac{L_f}{V_R} \right) = \frac{L_f}{V_R} \cdot C_{IN\ i} + K_{OL} a \cdot C^*_{i} \quad \dots (12)$$

or

$$C_{OUT\ i} = \frac{\alpha C_{IN\ i} + C^*_{i}}{(1 + \alpha)} \quad \dots (13)$$

To calculate C^*_{i} , it is necessary to know the initial resin composition q_i . The value of q_i may be calculated using an over-all mass balance for a stage for contact time, θ_L . The integrated form of equation (3) is used.

$$q_p - q_i = \frac{L_f}{V_R} \int_0^{\theta_L} (C_{IN} - C_{OUT}) dt \quad \dots (14)$$

Equations (11), (13) and (14) have to be solved simultaneously for each stage in turn, starting from the point where liquid feed enters the column. A trial and error technique is required. To maintain the cycle, resin is removed from the base of the column. The amount removed is calculated in terms of a fraction, x , of the quantity of resin in one stage, V_R .

The resin compositions between two stages are related by

$$q_{i,n} = (1 - x) q_{p,n} + x q_{p,(n+1)} \quad \dots (15)$$

so that, after the initial resin composition is calculated for the stage from which resin is removed, the final resin composi-

tion in the stage above is easily obtained. The initial value, $C_{OUT\ i,n}$, should also be corrected for the mixing of liquid of composition, $C_{OUT\ p,(n+1)}$, in the same manner. To simplify the calculations it is assumed that this effect is negligible.

A computer programme was written to solve these relations using a Runge-Kutta/Milne predictor-corrector method to solve the differential equations and Simpson's rule to evaluate the integral equation (14); see Appendix 1^(10,11).

2. Input information

The resin and liquid feed and product compositions are to be specified. The mass transfer coefficient, K_{OL} , and the equilibrium line slope, m , are fundamental parameters to be determined by experiment. The liquid flow rate must be given and the consequent values of K_{OL} , the interfacial area, a , and resin volume, V_R , will then be calculated. The expansion of the fluidized beds, hence, the hold-up of resin in each stage is dependent on fluid velocity. For uniform-diameter beads, the surface area per unit volume of beads is

$$a_r = \frac{6}{d}$$

hence the surface area per unit volume of fluidized bed with voidage, e , is

$$a = \frac{6(1-e)}{d} \propto V_R \quad \dots (16)$$

The height of the expanded bed will normally be very close to the stage height, with minimum freeboard. The volume of resin per stage will be

$$V_R = V_{ST} \cdot \bar{y} \cdot (1-e) = H.A.y. (1-e) \quad \dots (17)$$

and the voidage will be some function of the liquid superficial velocity determined experimentally in a fluidized bed.

$$V_S = \frac{L_f}{A} = \phi(e) \quad \dots (18)$$

An over-all mass balance for the column determines the average resin flow rate, because the terminal compositions and liquid flow rate are specified. The average flow rate of the fraction, x , of each bed removed in a cycle and the cycle time, θ_c , i.e.,

$$\frac{x V_R}{\theta_c} = \frac{\bar{L}_f (C_f - C_e)}{(q_e - q_f)} \quad \dots (19)$$

$$\theta_c = \frac{x V_R (q_e - q_f)}{\bar{L}_f (C_f - C_e)} \quad \dots (20)$$

The cycle time, θ_c , comprises liquid contact time, θ_L , and resin flow time, θ_R , hence

$$\theta_L = \theta_c - \theta_R \quad \dots (21)$$

and
$$\bar{L}_f = \frac{L_f \theta_L}{\theta_c}$$

Specification of x and θ_R will enable the required number of stages to be calculated.

3. Equilibrium relationship

The computation is based on a linear equilibrium relationship (Equation (2)) and the actual equilibrium lines are curved (Figure 10), so a method of characterizing the data is required.

A linear approximation valid at low resin loading was not adequate, nor was a method based on the ratio q/C .

The best method proved to be a plot of the tangent to the equilibrium curve against resin composition. The equations used were

$$q = m C$$

$$m = \frac{m_0}{q}$$

$$\text{and } m_0 = 15$$

so that, in effect

$$q^2 = m_0 C$$

for q and C in eq/l wet-settled resin (wsr) and eq/l respectively. It is known⁽¹²⁾, from existing equilibrium data for various uranium solutions and resins, that the maximum resin loading is a linear function of pH from about pH 0.5 to pH 2.0. In the present case, the total ionic concentration is also being varied; both of these affect the equilibrium line.

The non-linearity of the equation renders the mathematics very difficult and it is necessary to use Equation (2) with varying value of 'm'.

4. Measurement of the mass transfer coefficient

The values of K_{OL} appropriate to the fluidized beds are best established in such apparatus, although assumptions about flow patterns have to be made. Nevertheless, as long as the same assumptions are made in further mathematical modelling for apparatus of the same scale, serious errors may be avoided.

Equations (1), (2), and (3) may be solved for the case of a single fluidized bed of resin with a constant-composition liquid feed, and resin of known initial composition; see Appendix 2. Using Equation (11)

$$\frac{dC_{OUT}}{dt} = \frac{\beta (C_f - C_{OUT})}{(1 + \alpha)} \quad \dots (22)$$

$$\text{when } t = 0, C_{OUT} = \frac{\alpha C_f + C_i^*}{(1 + \alpha)} \quad \dots (23)$$

$$\frac{C_f - C_{OUT}}{C_f - C_i} = \frac{1}{(1 + \alpha)} \cdot \exp - \left[\frac{\beta}{1 + \alpha} \right] \cdot t \quad \dots (24)$$

$$\text{and } C_i^* = \frac{q_i}{m}$$

By fitting experimental and theoretical curves the value of K_{OL} may be found. Measurement of the initial composition yields a value of α , and the slope function, $\beta/(1 + \alpha)$, then yields a value of β . In general, however, the equilibrium line slope varies with resin composition and, in the present case of uranium extraction, the liquid flow was gradually increased, as resin density increased, to maintain a constant voidage. These effects were incorporated into the calculation of output curves; the resulting curves are not exponential.

It is known that the film mass transfer coefficient can vary several times in magnitude for substantial changes in resin composition^(13,14), but it was calculated that this effect was not significant at low loading.

Experiments were undertaken with a single fluidized bed of the same design as one section of the multi-stage extraction column used in this work. Uranium ore pulps (5 and 10 wt % solids) and clarified uranium liquors were passed through the resin bed and the effluent was analysed for uranium until the resin was fully loaded. The fluidized bed was expanded to fill the stage for each experiment so that both voidage and the amount of resin in the stage varied according to the flow rate and pulp density and viscosity. The flow rate was adjusted to maintain constant voidage as resin density increased with loading.

Results for the single stage experiments are shown in Figures 11 to 15. The curves were fitted by adjusting the equilibrium line slope parameter, m_0 , after agreement at zero time had been established by varying the value of the mass transfer coefficient. The value of m_0 was less than that for the measured equilibrium curve because the assumption of perfect mixing was not strictly valid.

The predicted curves are very close to each other while the resin is approaching saturation with uranium. As a result, changing values of the mass transfer coefficient, as resin composition changes, are not detectable in this work. However, only a low degree of loading is used in the multi-stage extraction column, and mass transfer coefficients for lightly loaded resin can be ascertained from the early part of the experimental curves.

A measure of the mass transfer coefficient can be obtained on an area-free basis by means of Equation (16). The

value thus obtained is only apparent because it incorporates the effect of mixing patterns different to those assumed in the mathematical model; only the ratio $K_{OL} a/V_R$ is therefore reported to emphasize this point (Figure 16). The ratio a/V_R is constant. At high voidage, high relative velocities give a high value of $K_{OL} a/V_R$. At lower voidage, lower relative velocities are experienced, but increasing inter-action of particles reduces axial mixing of both phases, so the driving force for mass transfer is enhanced and the mass transfer coefficient is apparently higher.

The mass transfer coefficient is not significantly different in pulps (5 and 10 wt % solids) at a given voidage. The higher superficial velocity used for a 5 wt % solids pulp, which might give better hydrodynamic conditions, is perhaps compensated by a lower ionic diffusivity in solution at the lower total ionic concentration.

The mass transfer coefficients for clarified undiluted uranium solution of higher uranium content are very different. The value for run MT8 is suspect due to the difficulty of fitting the breakthrough curve (Figure 14).

Approximate values of mass transfer coefficients may be obtained from fixed bed data using Michael's method⁽¹⁵⁾. Breakthrough curves for uranium extraction on various anion resins from various clarified uranium solutions were analysed and mass transfer coefficients of 0.013 to 0.049 s^{-1} (0.8 to 3.0 min^{-1}) were found. The values found in this work are similar in magnitude.

5. Calculation of multi-stage column performance

A series of computer calculations were carried out using experimentally determined mass transfer coefficients and equilibrium equations to establish satisfactory operating conditions for pilot plant tests and to obtain an indication of the relative sizes of extraction columns operating under different conditions.

Pilot plant tests described below were used to confirm the accuracy of the calculation technique.

In calculations of performance, Equation (20) was used. Experimental values of the mutually dependent variables K_{OL} , V_R , L_f , and C_f were used. The liquid product composition, C_e , was set by requiring 99.5 % extraction of uranium, and the resin feed was set to zero. Various values of resin loading were assumed. The ratio x/θ_c was then fixed and a value of x was taken as 0.25 to determine θ_c . The number of column stages can be calculated for these conditions.

The volume of the extraction column required for every case considered can be calculated. The flow rate of pulp or solution and the allowable superficial velocity give the cross sectional area of the column, and the number of stages give the height. To obtain a relative measure of column volume, a common basis of a fixed number of pounds per unit time of uranium extracted is required. This is achieved by correcting the calculated volume with the dilution factors used in this work. The dilution factor is the ratio of uranium concentrations in the clarified, undiluted feedstock and the various pulps used.

The calculated column volume was examined with reference to the resin loading (Figure 17).

The column volume increases as resin loading increases because the driving force for mass transfer decreases. A low loading is compensated for by a higher resin flow rate which is achieved either by removing larger fractions of the bed or decreasing the liquid flow period.

A small change in resin composition gives better overall results than a large change, because regeneration of resin is slow if ions near the core of the head are to be removed. If a large density change occurs on loading, as with uranium, undesirable differences in bed voidage will occur in a multi-stage column. If a high degree of extraction is required, highly regenerated resin must be used.

If low loading is accompanied by a short liquid flow time, the frequency of valve operation will adversely affect valve life and resin attrition. A more serious factor envisaged was the possibility of high iron loading at low uranium loading. This was investigated during extraction column runs.

Contacting volume varies considerably with variation in voidage (Figure 18). A minimum volume is found at a voidage of 0.7 to 0.8 for nominal 5 and 10 wt % solids pulps.

The points for a 5 wt % solids pulp are not significantly different to those for a 10 wt % solids pulp but a large advantage in column size is gained by treating a clarified pulp which is undiluted, despite a lower mass transfer coefficient than for diluted pulps.

In the case of the pulps a larger column area resulting from dilution is fortuitously compensated for by fewer stages resulting from a more favourable equilibrium relationship; the mass transfer coefficients are similar.

The terminal velocity of pulp particles in a pulp, containing a given fraction of pulp solids of known specific gravity, can be calculated in the present case (confirmed by experimental sedimentation work) allowing consideration of the relationship of relative column size and maximum size of pulp solids which may be allowed in the feed (Figure 19).

As the voidage increases, the superficial pulp velocity and the allowable maximum particle size in the pulp increase. It appears that pulp particles larger than about 200 mesh will affect column size adversely.

6. Mixing of liquid and resin in the stages

It was known that mixing of resin and liquid in each stage was not perfect as assumed in calculations. Residence-time tracer tests were made using sodium chloride tracer in water passing through a single fluidized bed of resin in the chloride form. A variable quantity of resin was used so that the column section used was always full of expanded resin at each different flow rate. It was found that by-passing of liquid was severe at about 90 % voidage and that some mixing and recycling was occurring. Recycling increased and by-passing decreased as voidage decreased. However, at voidages of about 75 %, the residence-time distribution for the liquid crudely approximated that expected for perfect mixing. On scale-up, better distribution

of liquid, less recycling, and good mixing of resin could be expected.

COLUMN TRIALS AND MODEL TESTING

The assumptions made in the calculation procedure were investigated by comparing predicted results for a seven-stage column, processing a uranium leach slurry (10 wt % solids) and a clarified uranium leach liquor with IRA 430 resin in chloride form. The predicted performance utilized values of the mass transfer coefficients and equilibrium parameters measured experimentally in one stage as described above. The voidage, amount of bed transferred per cycle, cycle time, liquid feed and resin product compositions, and liquid flow rate were input to the programme. The predicted and observed results are shown in Figures 20 and 21.

The conclusion drawn at this time is that agreement can be obtained by using single-stage data, but the experimental errors involved, particularly on loaded resin sampling and voidage measurement during pulp trials, are sufficient to preclude comments on the validity of the assumptions made in the calculations. Two important assumptions to be tested are that final resin concentrations can be used to establish the slope of the equilibrium line and the neglect of back-mixing of liquid on resin transfer.

The imperfect prediction for run MT12 for pulp is probably due to variations in bed voidage and in the amount of resin in each stage, caused by operating difficulties described

below. The pulp used in column trials was 10 wt % ore that was 90 % minus 270 USS mesh. At the flow rate used, some sanding out occurred, as expected, and the barren pulp contained about 9 % solids. The conditions for this run are shown in Table 11. The pulp flow of 3.25 gpm/ft²* (0.26 cm/s) allowed 98.7 % uranium extraction in six 24-inch high stages. A 6.25-minute liquid flow was used with a 5-second resin flow. The resin loading is very low but this does not preclude a high degree of uranium extraction. An extraction of 99.9% could be achieved with two more stages.

A trial was made on a clarified uranium solution the composition of which is given in Table 8. A solution flow of 12.1 gpm/ft² gave 99.1 % extraction in six stages and an estimated 99.9 % extraction in eight or nine 24-inch high stages. The periods of liquid and resin flow were 3.0 minutes and 6 seconds respectively. Again the resin loading was relatively low, but good extraction was achieved.

OPERATING PROBLEMS

In the pulp trial described, a higher flow could have been used to allow higher resin bed voidage and upwards transport of larger pulp particles (up to 200 USS mesh). The maximum flow rate which can be used for a pulp (10 wt % solids) and a resin bed voidage of 0.86 is 4.8 gpm/ft²; above this limit, elutriation of the smaller unloaded resin beads at the top of

* Imperial measure unless otherwise stated.

the extraction column is unavoidable. To allow some margin, a lower flow than the calculated maximum must be used. If a higher voidage is used, mass transfer performance suffers; in the limit, for the impractical case of bed voidage of 1.0, a maximum flow of 7.5 gpm/ft² (0.6 cm/sec) is allowable.

In the early work with pulp, temporary one- or two-minute shut-downs (to clear a blocked line for example) allowed resin beds to settle with pulp particles in the interstices so that it was extremely difficult to re-fluidize the beds smoothly. The beds remained in a packed state and moved bodily as slugs up the column. High flow and low flow in rapid succession was required to break up the compacted beds. In contrast was the ease of start-up by expanding the settled resin with water.

The second problem concerned the flow of resin and pulp from the base of the extraction column. The small fraction of coarse pulp solids which settled out beneath the bottom resin bed joined the outflowing resin but, on occasion, the frictional resistance to the resin/coarse-solids mixture was sufficient to prevent flow. The removal of all solids greater than that size transported upwards must be recommended.

There were no operating problems of consequence while treating either clear solutions or those containing very little particulate material.

DISCUSSION AND CONCLUSIONS

By studying the fluidization of resins in water and ore leach pulps, a satisfactory design procedure may be established for the hydrodynamic aspects of resin-in-pulp fluidized systems. This work has shown the relationship between the important variables of pulp flow, pulp density, resin size and size distribution, resin density, and pulp particles size and density of pulp solids.

Experiments on the rate of mass transfer have indicated the best conditions to use for minimum capital cost of the extraction column. The calculation method used to assess the performance of the multi-stage extraction column described needs refinement, but reasonable agreement of the few observed and computed results has been achieved so far.

This work has confirmed the work of others that a series of fluidized beds can give high degrees of uranium extraction in a short column; this is not the prerogative of fixed beds of ion exchange resin. An advance has been made in modelling the system mathematically so that optimum conditions may be found at minimum expense and the interrelationship of the many variables more clearly understood. A system of inter-column resin transfer has been designed and tested. The system has worked well but resin attrition rates due to the system cannot be measured without prolonged operation.

The use of short liquid flow cycle times is desirable theoretically for efficient extraction, although it is undesirable from the mechanical point of view of valve operation. A further factor is that resin does not have time to segregate into layers

according to particle size and fine resin does not accumulate in the top stage, becoming progressively loaded, causing the barren effluent to be of inferior quality to that expected.

Although column start-up was easy in the 2-inch-diameter column, problems are envisaged on the large scale. The expansion of settled resin through perforated plates of low free area (less than 10 %) may lead to high forces exerted on the plates, particularly if bed expansion is hindered by having a wide range of resin beads or pulp particles in the settled resin bed. If beds are excessively expanded on flow surges during start-up or mal-operation, resin cannot settle back into lower stages unless flow is substantially reduced. However, the rate of settling of resin into lower stages is very low. Temporary reductions of flow may reduce bed expansion but have no serious consequences. Once resin is over-expanded and pushed into higher stages, normal cyclic operation will not allow gradual refilling of all stages. To combat these problems more free area on each perforated plate is recommended. It is further proposed that the rate of resin feed (during flow reversal) from a higher to a lower stage should slightly exceed the flow of resin out of the lower stage. If resin is pushed upwards out of a stage by excessive flow, a gradual refilling of the stage is possible. If each stage is already full of expanded resin, transfer upwards of any excess will always occur. To achieve this, the degree of bed expansion should decrease towards the top of the column, for example, by increasing the area available for liquid flow by increasing column diameter.

The fluidization data indicate that, for IRA 430 resin in a pulp (10 wt % solids), flow velocities no greater than 0.38 cm/sec (4.8 gpm/ft²) should be used. For minimum volume of extraction column, a flow of 0.26 cm/sec (3.25 gpm/ft², voidage 0.75) is proposed. However, the minimum capital cost of the extraction column may correspond to a value outside these limits.

REFERENCES

1. Bouchard, J., S.C.I. Symp. on Ion Exchange, London, 1969.
2. Higgins, I., Ind. Eng. Chem., 1961, 53, (8), 635.
3. Rosenbaum, J., George, d'A., S.C.I. Symp. on Ion Exchange, London, 1969.
4. Slater, M., Brit. Chem. Eng., 1970, (1), 41.
5. George, d'A., et al., Min. Eng., 1968, (1), 73.
6. George, d'A., et al., U.S.B.M. Rpt. Inv., 6928, 1967.
7. Marchello, J., Davis, M., Ind. Eng. Chem. Funds., 1963, 2, (1), 27.
8. Richardson, J., Saki, W., Trans. Inst. Chem. Eng., 1954, 32, (1), 35.
9. Slater, M., Ph.D. thesis, Univ. Bradford, U.K., 1971.
10. McCormick, J., Salvadori, M., Numerical methods in Fortran, pub. Prentice-Hall, 1964.
11. Arden, B., Astill, K., Numerical algorithms, pub. Addison-Wesley, 1970.
12. Grinstead, R., et al., PUAE, 1956, 8, P/522.

13. Turner, J., Snowdon, C., Chem. Eng. Sci., 1968, 23, 221.
14. Nachod, F., Schubert, J., Ion Exchange Technology, pub.
Academic Press, 1956.
15. Michaels, A., Ind. Eng. Chem., 1952, 44, (8), 1922.

TABLE 1

Wet Screen Analysis of Pressure Leached Solids

Mesh	Size, μ	w/o	cum. w/o
+ 65	+208	0.1	0.1
+100	+147	0.7	0.8
+150	+104	2.9	3.7
+200	+ 74	8.1	11.8
+270	+ 53	8.8	20.6
+325	+ 44	7.1	27.7
+400	+ 37	4.2	31.9
	+ 29	9.8	41.7
	+ 20	10.6	52.3
	+ 14	10.4	62.7
	+ 10	5.6	68.3
	- 10	31.7	100.0

TABLE 2

Screen Analyses of Rio Algom Pulp Solids

Mesh	Pachuca w/o	Cyclone Overflow w/o	Part Sedimented w/o	Part Sedimented w/o
+ 65	20.8	2.5	0.1	
+100	12.5	4.9	0.6	
+150	11.3	8.4	1.0	2.3
+200	10.6	10.1	2.8	6.3
-200	44.8	74.1		
+270			5.4	9.7
+325			0.8	8.1
-325			89.3	73.6

TABLE 3

Sedimentation of pressure leached solids

MIXTURE

<u>w/o Pulp</u>	<u>V_s cm/sec</u>	<u>v/o Pulp</u>	<u>Voidage</u>
5	0.01411	1.91	0.981
10	0.00941	3.95	0.960
20	0.00498	8.48	0.915
30	0.00315	13.70	0.863

- 325 MESH

<u>w/o Pulp</u>	<u>V_s cm/sec</u>
5	0.0500
10	0.0313
20	0.0125
30	0.0045

-150 +200 MESH

<u>w/o Pulp</u>	<u>V_s cm/sec</u>
5	0.459
10	0.777
20	0.645
30	0.540

- 200 + 270 MESH

<u>w/o Pulp</u>	<u>V_s cm/sec</u>
2	0.245
5	0.300
10	0.219
20	0.175
30	0.188

- 270 + 325 MESH

<u>w/o Pulp</u>	<u>V_s cm/sec</u>
5	0.244
10	0.188
20	0.144
30	0.107

TABLE 4

Pressure Leached Solids Data

Mesh Size	Re_p	n	n_{calc}
-150 +200	0.50	3.8	4.6
+270	0.21	4.7	4.6
+325	0.10	5.4	4.6
-325	0.06	8.2	

TABLE 5

Resin Properties, Fluidized in Water

Resin	V_T cm/sec	n	ρ_R	d_{SM} mm	n (calc.)
IRA 425 (C1)	2.0	3.0	1.069	1.118	3.5
IRA 430 (C1)	2.5	3.0	1.082	1.293	3.4
IR 120 (Na)	2.5	3.0	1.270	0.706	3.5
A 200C (Na)	2.5	3.0	1.250	0.904	3.5
IRA 430 (U)	-	-	1.185	1.091	-
IRA 430 (U)	-	-	1.160	1.070	-
XE 137 (C1)	0.96	2.9	1.095	0.495	3.9

TABLE 6

Resin Fluidized in Pulp

Resin	w/o Pulp	V_T cm/sec	μ , cp
XE 137 (C1)	0.0	0.87	1
XE 137 (C1)	3.2*	0.7	1
XE 137 (C1)	7.2*	0.6	1
IRA 430 (C1)	9 - 11 ⁺	0.6	2
IRA 430 (C1)	5 ⁺	1.2	2
IRA 430 (U)	9 - 11 ⁺	1.6	3

* Pressure leached solids
 + Rio Algom solids

TABLE 7

Primary Feed Liquor Composition

	MT 1 to MT 8, MT 12	MT 11
pH	1.15	1.9
U	1.51 g/l	1.25
SO ₄	8.99 g/l	4.64
Fe	2.76 g/l	0.095

TABLE 8

Actual Feed Compositions

	MT 1,2,2R	MT 3,4	MT 6,7	MT 8	MT 10	MT 11,13	MT 12
U gm/l	0.45	0.50	0.25	1.52	0.46	1.25	0.47
SO ₄ g/l	3.20	-	-	8.99	3.22	4.64	3.57
Fe g/l	-	-	-	-	0.84	0.095	0.88
pH	1.50	1.50	1.90	1.15	1.78	1.90	1.50

TABLE 9
Size Analyses of Pulp Solids

Mesh	MT 1		MT 2	MT 3		MT 6		MT 7		MT 12
	Feed	Product	Feed	Feed	Product	Feed	Product	Feed	Product	Feed
+ 65		0.1	0.1	1.2		0.2	0.9	0.6	0.9	
+100	0.2	0.4	0.7	3.5	0.1	1.0	2.3	1.2	1.8	2.8
+150	0.9	0.7	1.7	5.9	1.4	2.0	3.9	2.4	2.9	2.4
+200	2.4	2.3	4.5	8.8	3.9	3.4	6.1	4.1	4.9	10.8
+270	3.6	4.1	9.9	11.6	8.3	4.6	7.7	6.0	6.3	9.7
+325	5.3	5.3	10.7	11.6	8.3	5.0	7.7	6.2	6.5	0.7
-325	94.7	94.7	89.3	88.4	91.7	95.0	92.3	93.8	93.5	73.6

TABLE 10

Experimental Conditions for Single Stage

Run	w	L_p ml/min	L_f ml/min	e	V_R ml	C_f eq/l	K_{OL}^a min^{-1}	m_o eq/l	N^*	θ_L min
MT 1	0.115	320	305	0.716	485	0.00376	6	15	4	10.6
MT 2	0.100	120	115	0.613	551	0.00376	>10	15	3	32.0
MT 2R	0.085	120	115	0.524	835	0.00428	c.100	15	3	42.3
MT 3	0.107	460	440	0.886	202	0.00428	3	15	8	2.7
MT 4	0.104	350	335	0.818	350	0.00420	4	15	5	6.2
MT 6	0.056	520	509	0.776	400	0.00210	4	15	5	9.4
MT 7	0.054	440	431	0.647	580	0.00210	8	15	4	16.1
MT 8	0	900	900	0.740	400	0.01270	2	4	10	0.9
MT 13	0	1000	1000	0.814	350	0.01010	6	2	-	-

* for $q_e = 0.10$

TABLE 11

Experimental Conditions for Column Trials

Run	θ_L min	θ_R s.	x	L_P ml/min	L_f ml/min	V_R ml	e	C_f meq/ml	w
MT 10	6.25	5.0	0.395	300	287	380	0.750	0.00386	0.105
MT 11	3.0	6.0	0.194	1150	1150	567	0.685	0.01050	0.0
MT 12	3.0	5.0	0.124	310	298	563	(0.700)	0.00394	0.094

TABLE 12

Experimental Results for Column Trials

RUN	C_f eq/l	C_e eq/l	q_f eq/l	q_e eq/l	m_o	$K_{OL} a$ min^{-1}	N	\bar{x} %
MT 10	0.00386	0.000045	0.000167	0.0313	-	4.4	6	98.7
MT 11	0.01050	0.00010	0.0020	0.2730	4	2.8	6	99.1
MT 12	0.00394	0.00002	0.0005	0.0072	15	(6.5)	6	99.5

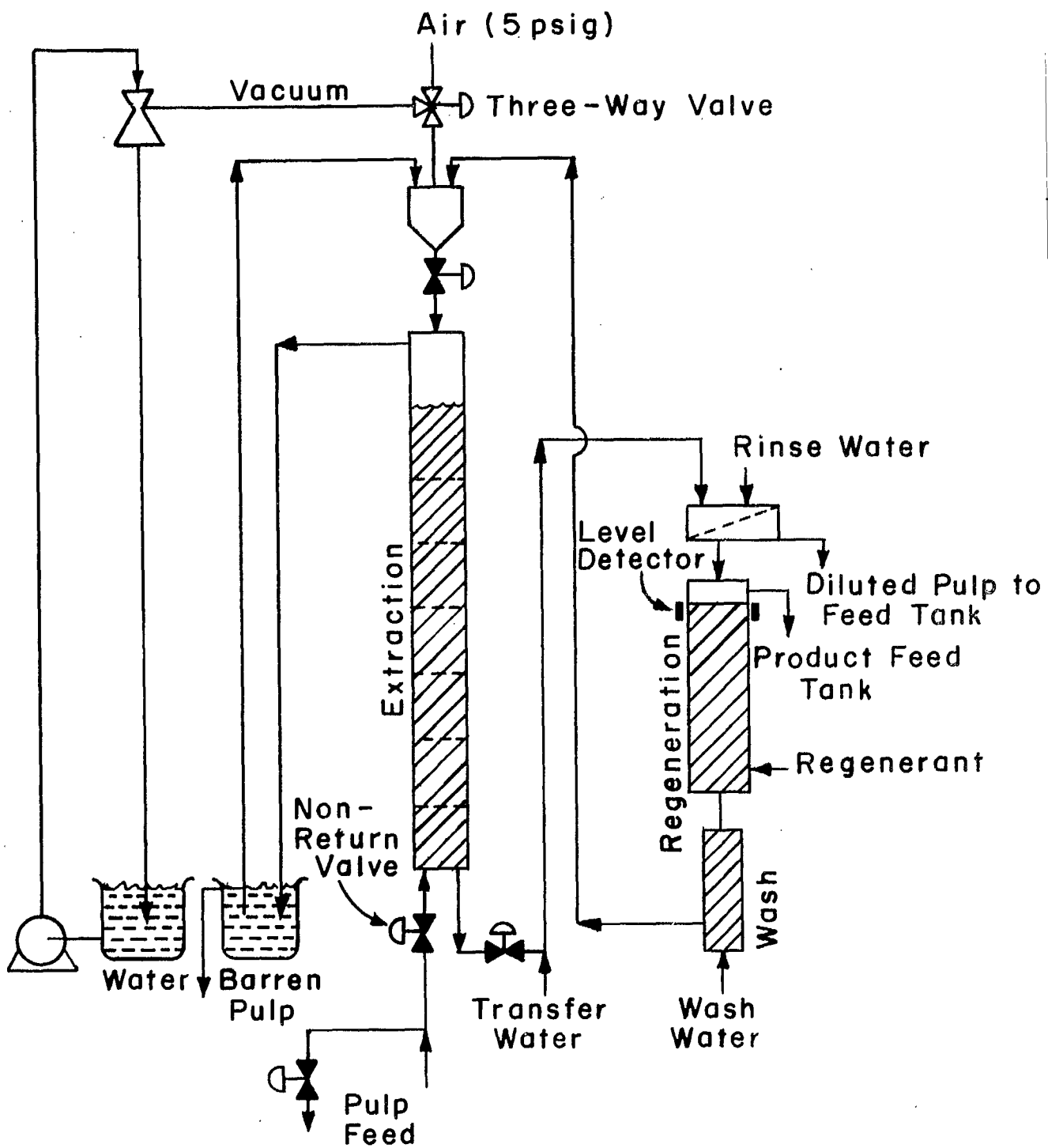
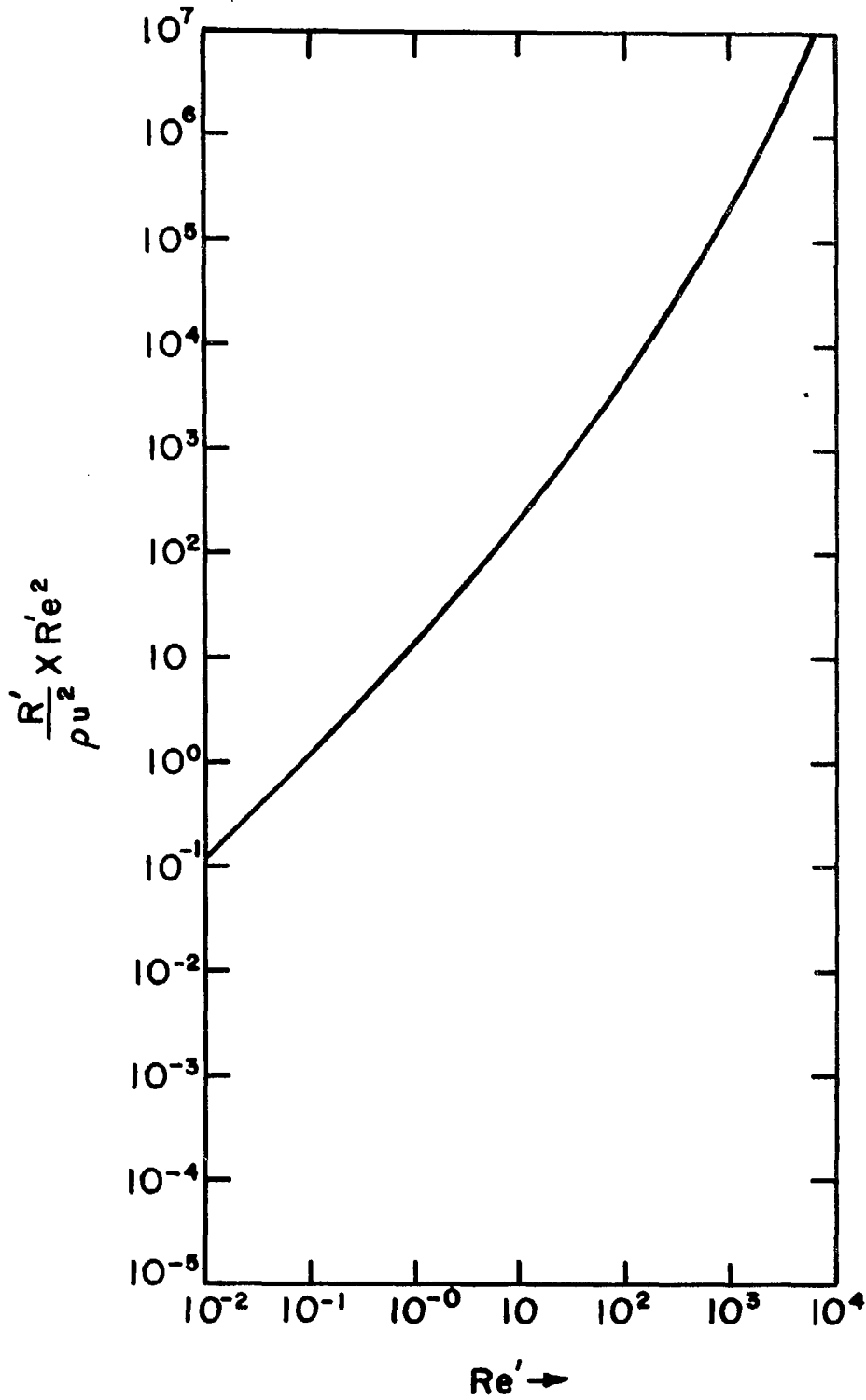


Figure 1. Ion Exchange Plant for Pulp Processing



$$Re' = Re_p$$

$$\frac{R'}{\rho u^2} \times Re'^2 = \frac{2 d^3 \rho_L g (\rho_R - \rho_L)}{3 \mu_L^2}$$

Figure 2. Terminal Velocity Chart

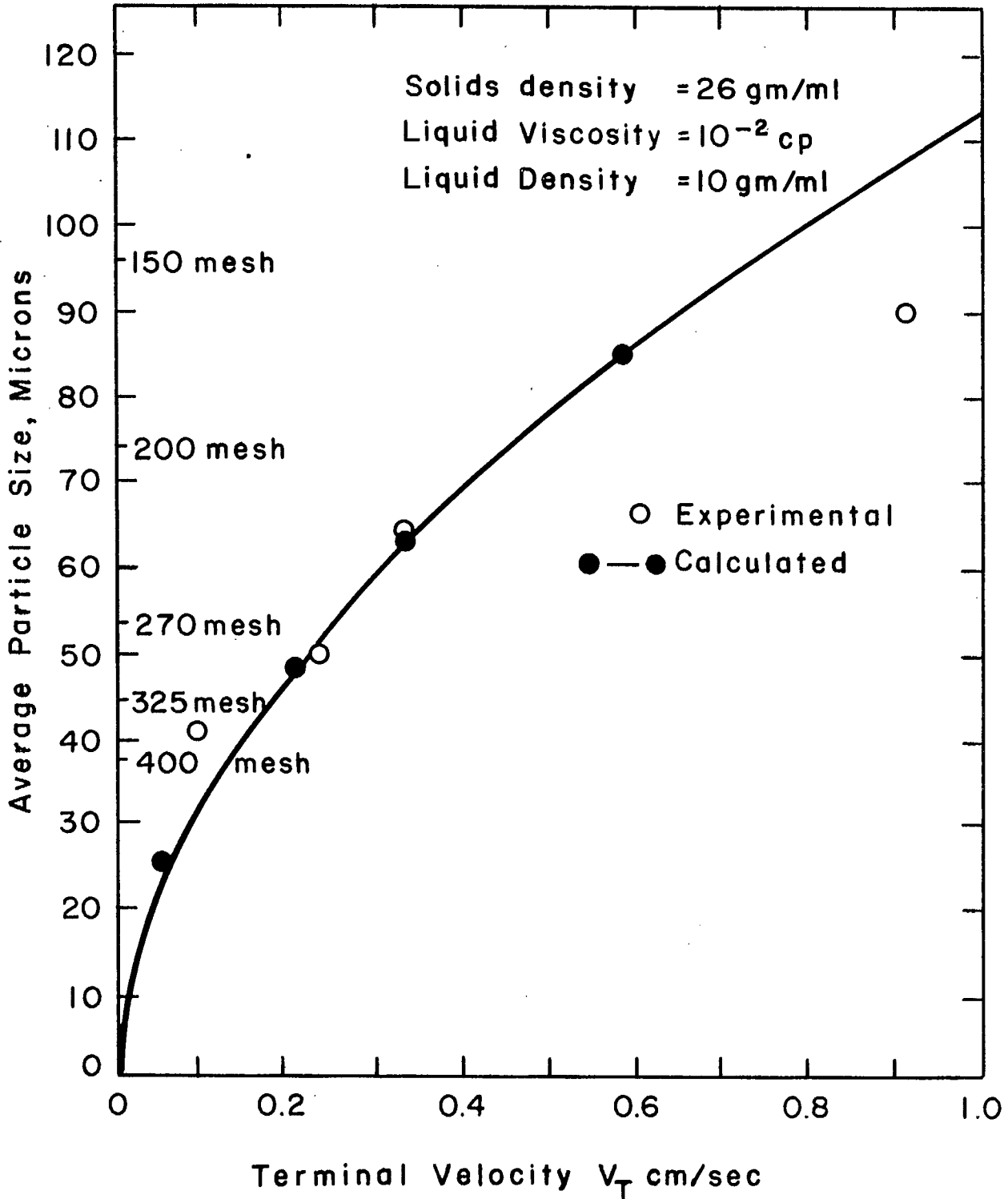


Figure 3. Terminal Velocities of Pulp Solids

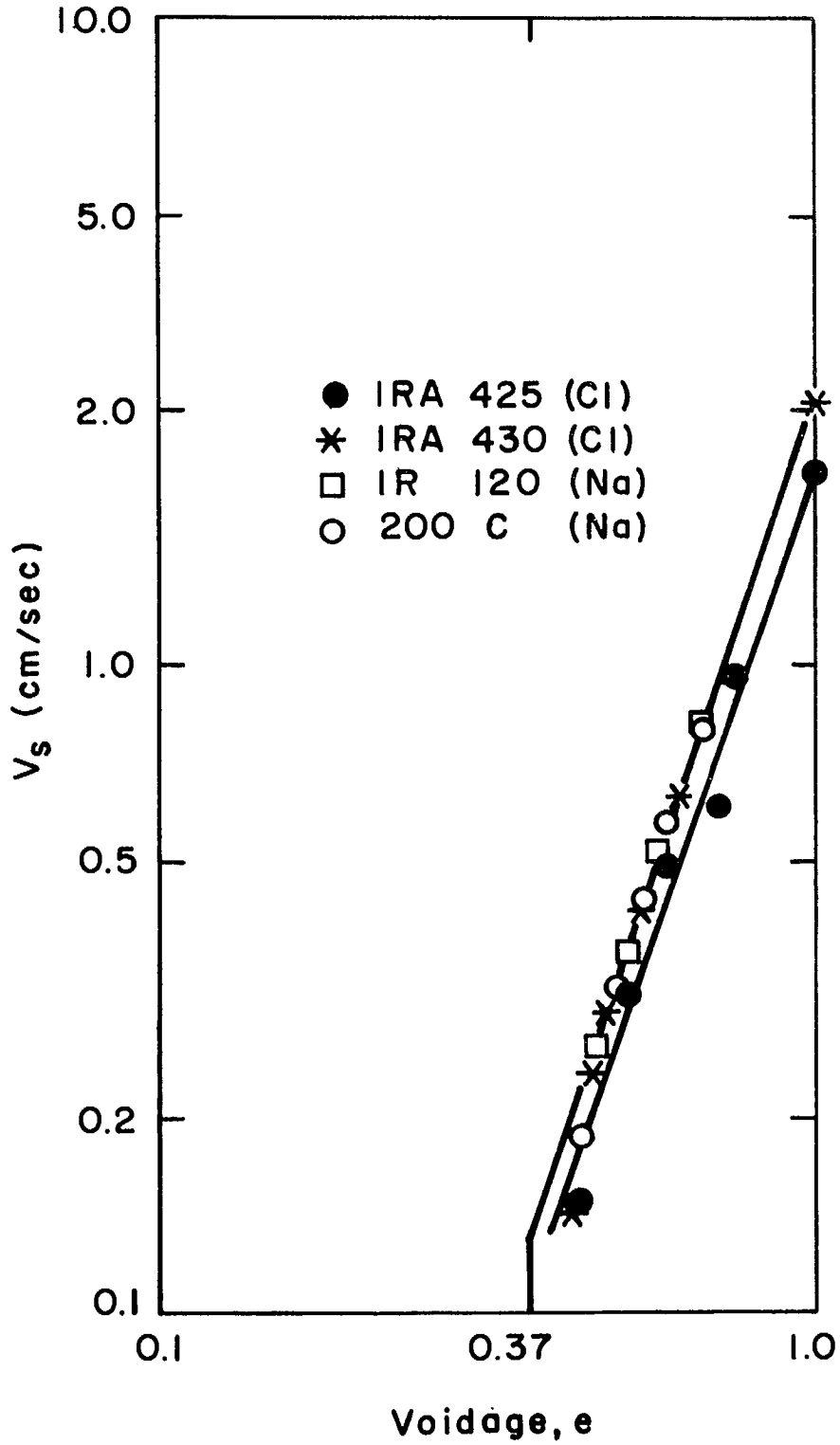


Figure 4. Resin Fluidization in Water

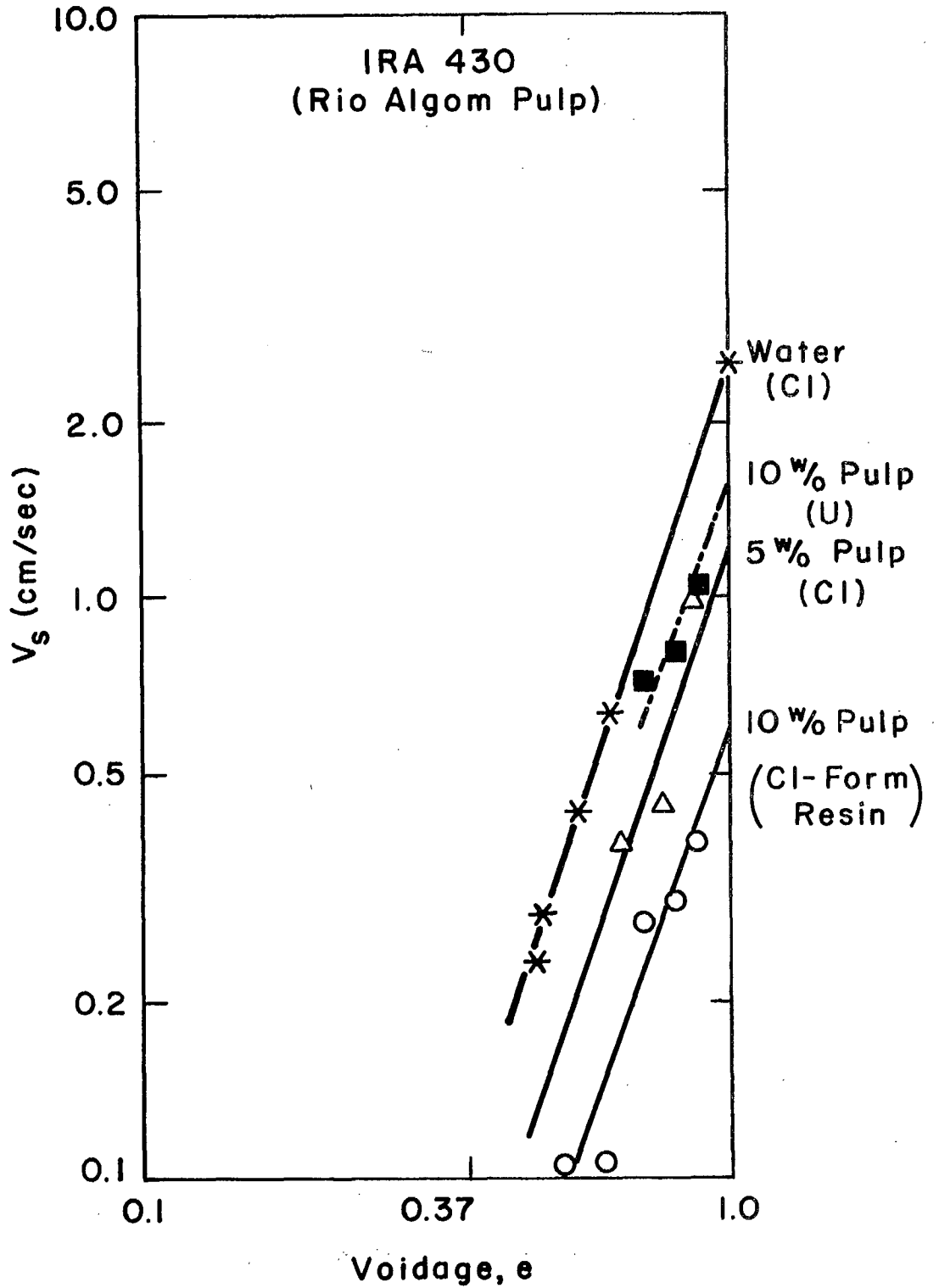


Figure 5. Resin Fluidization in Pulp

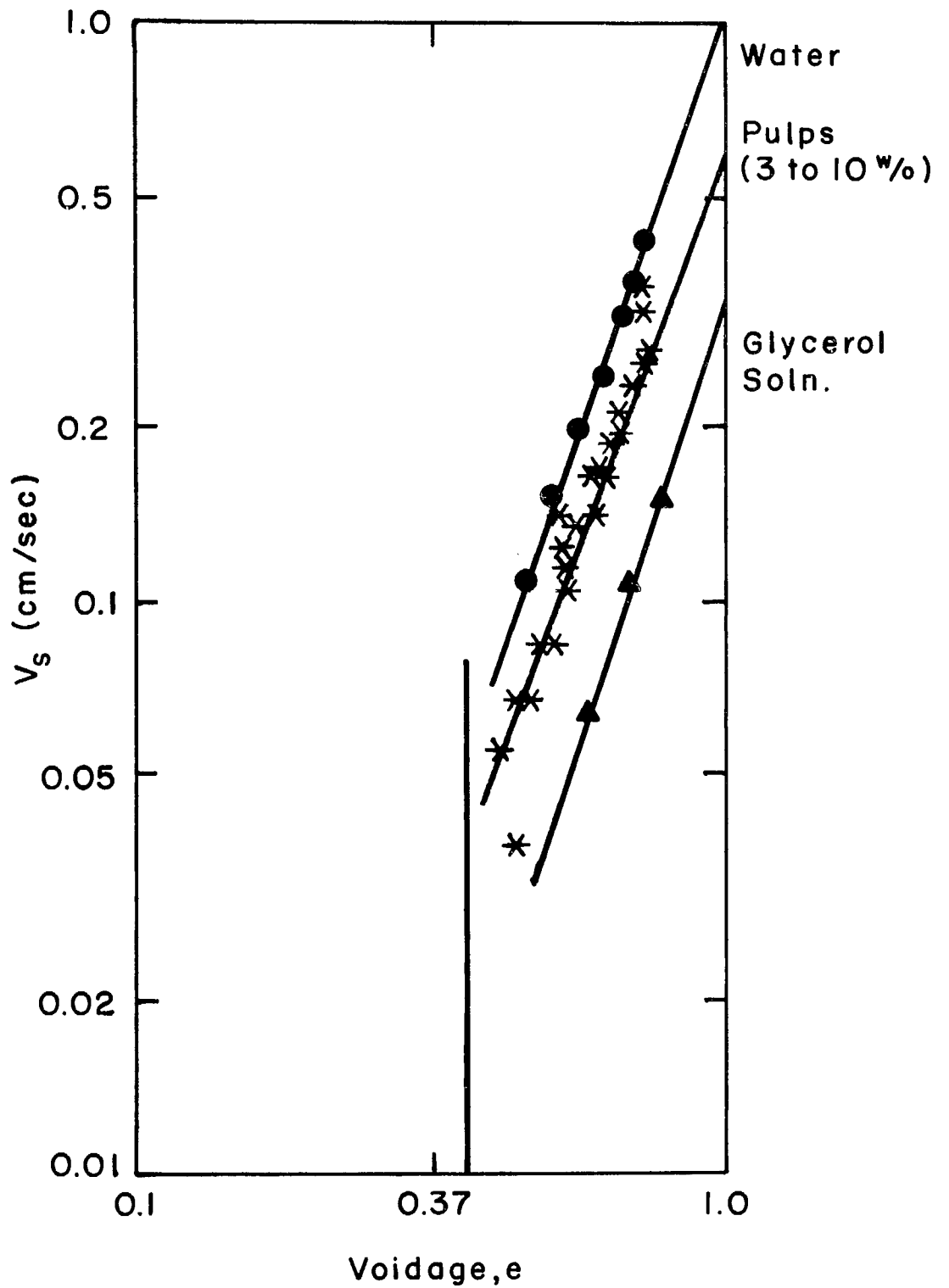


Figure 6. Fluidization of XE 137 Resin
(Pressure Leached Solids in Pulp)

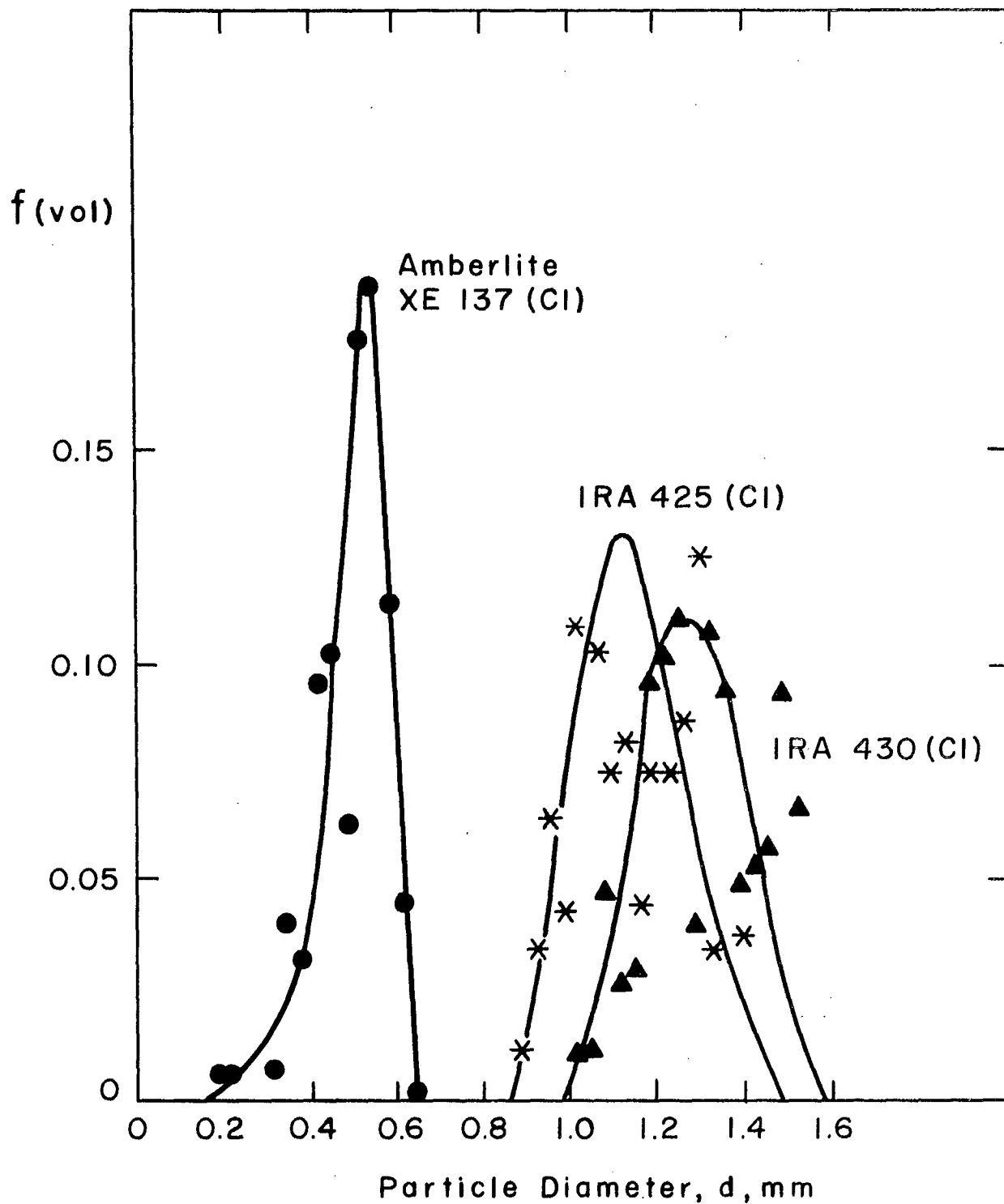


Figure 7. Resin Particle Size Distributions.

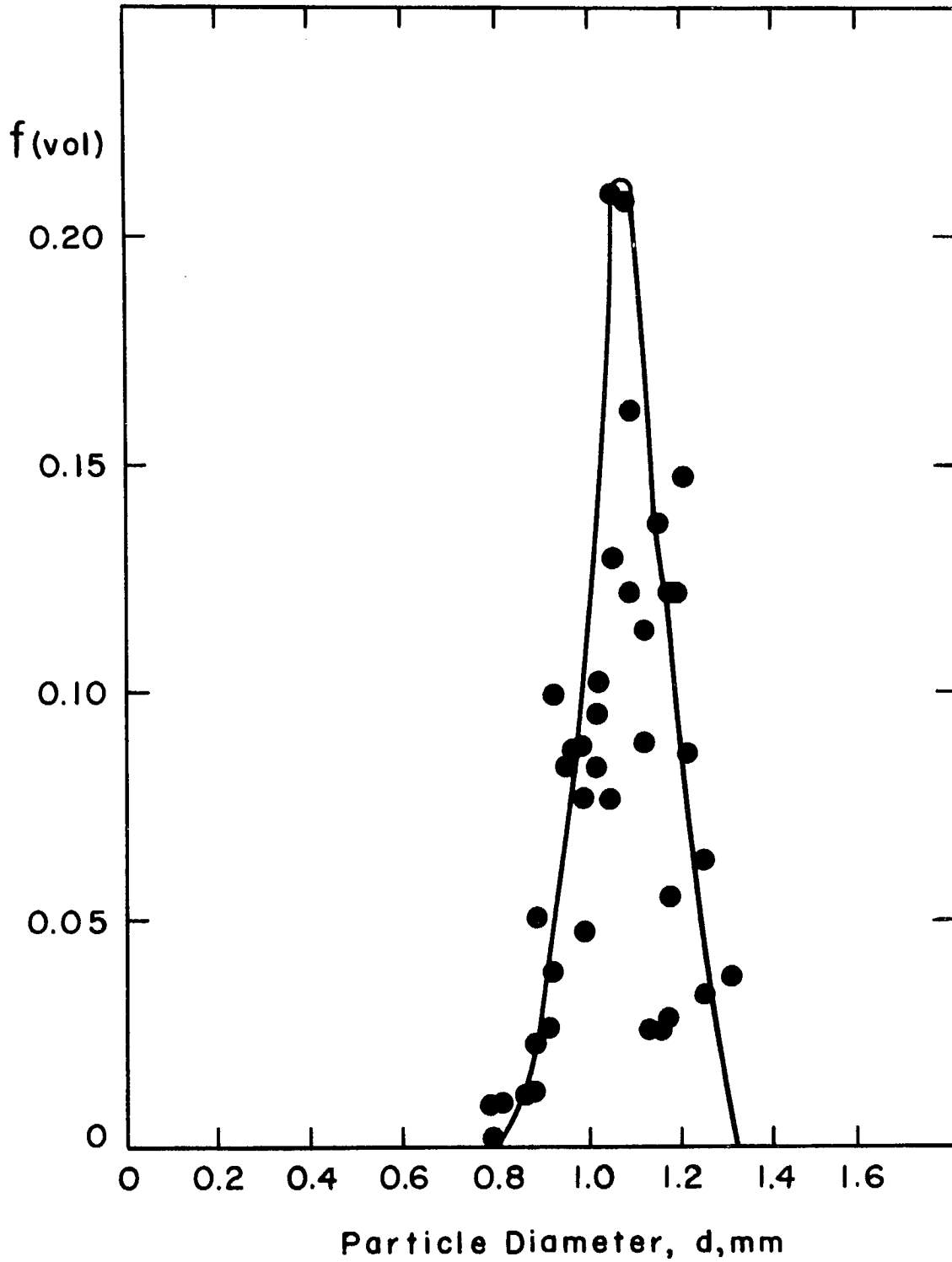


Figure 8. Particle Size Distribution for Uranium Loaded IRA 430 Resin.

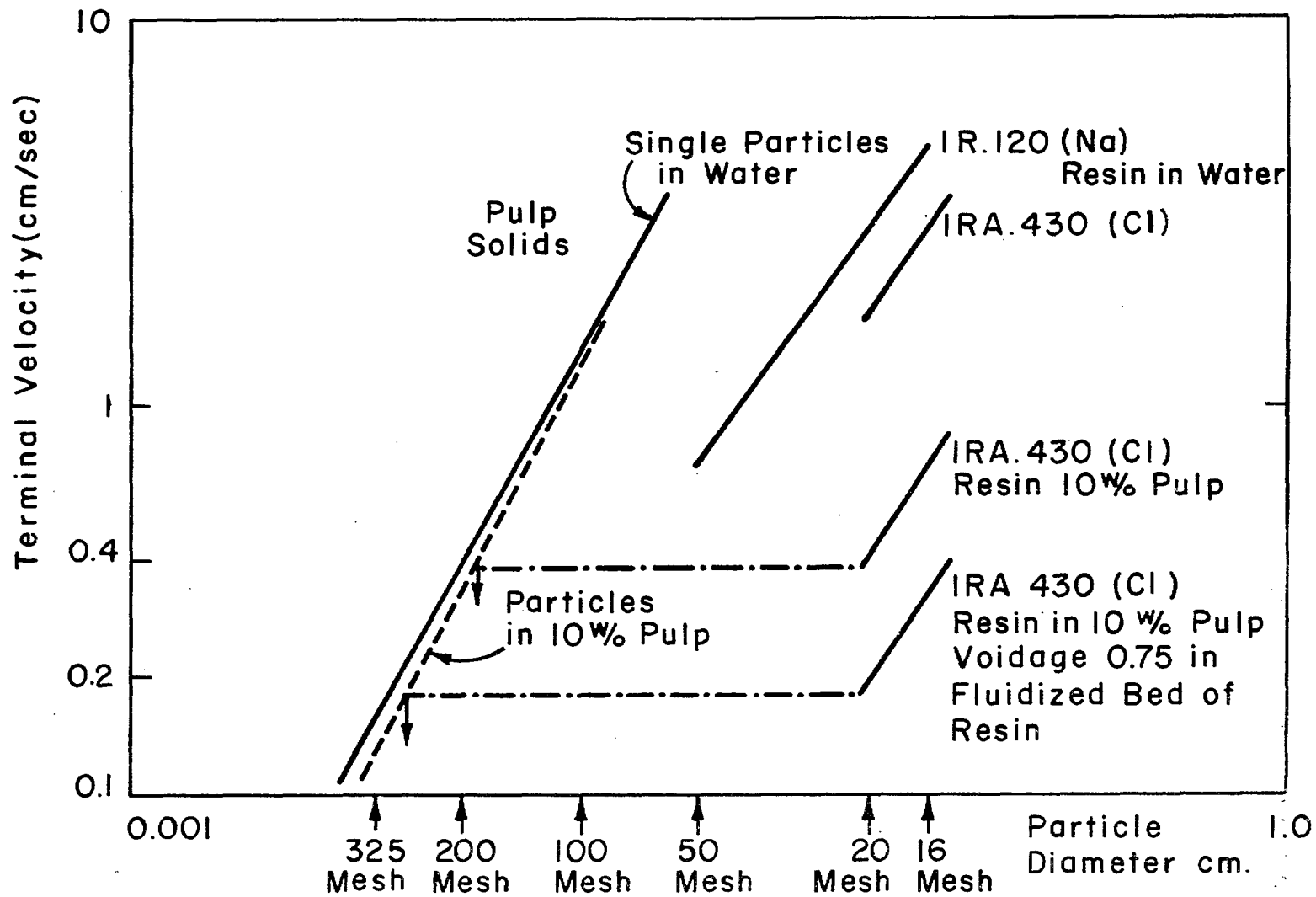


Figure 9. Terminal Velocities of Pulp Solids and Resin as a Function of Particle Size.

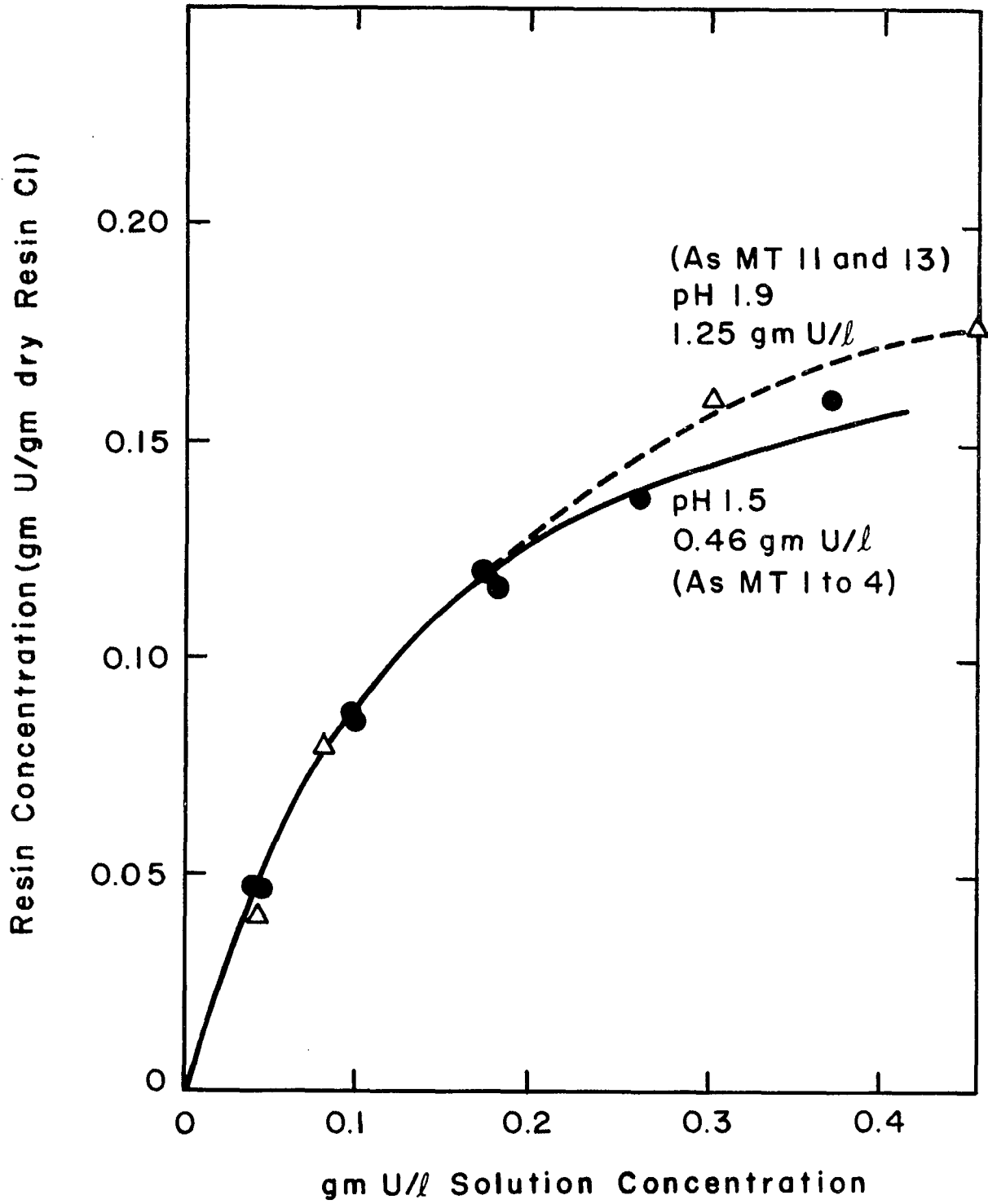


Figure 10. Equilibrium Curves

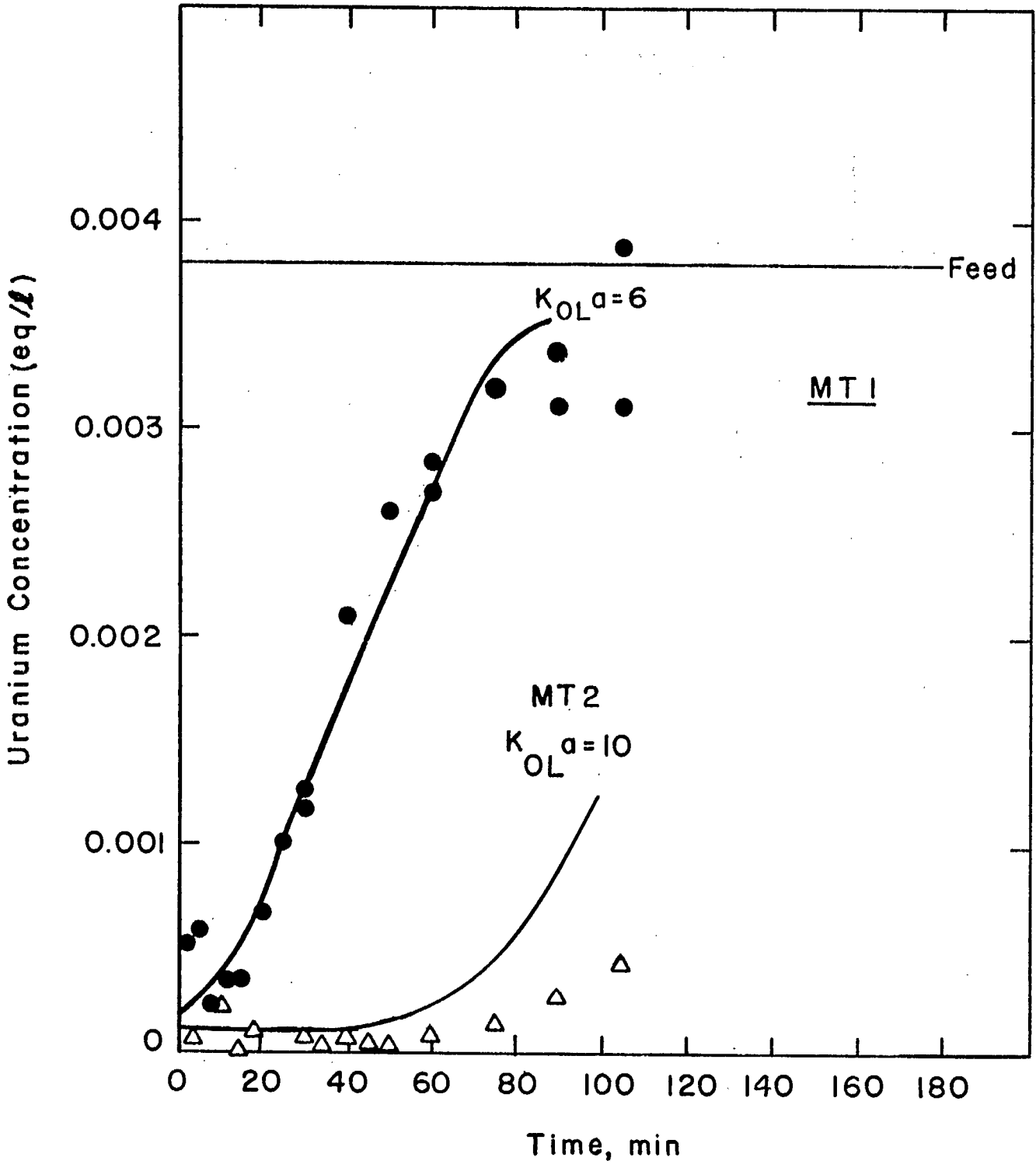


Figure II. Single-Stage Breakthrough Curves

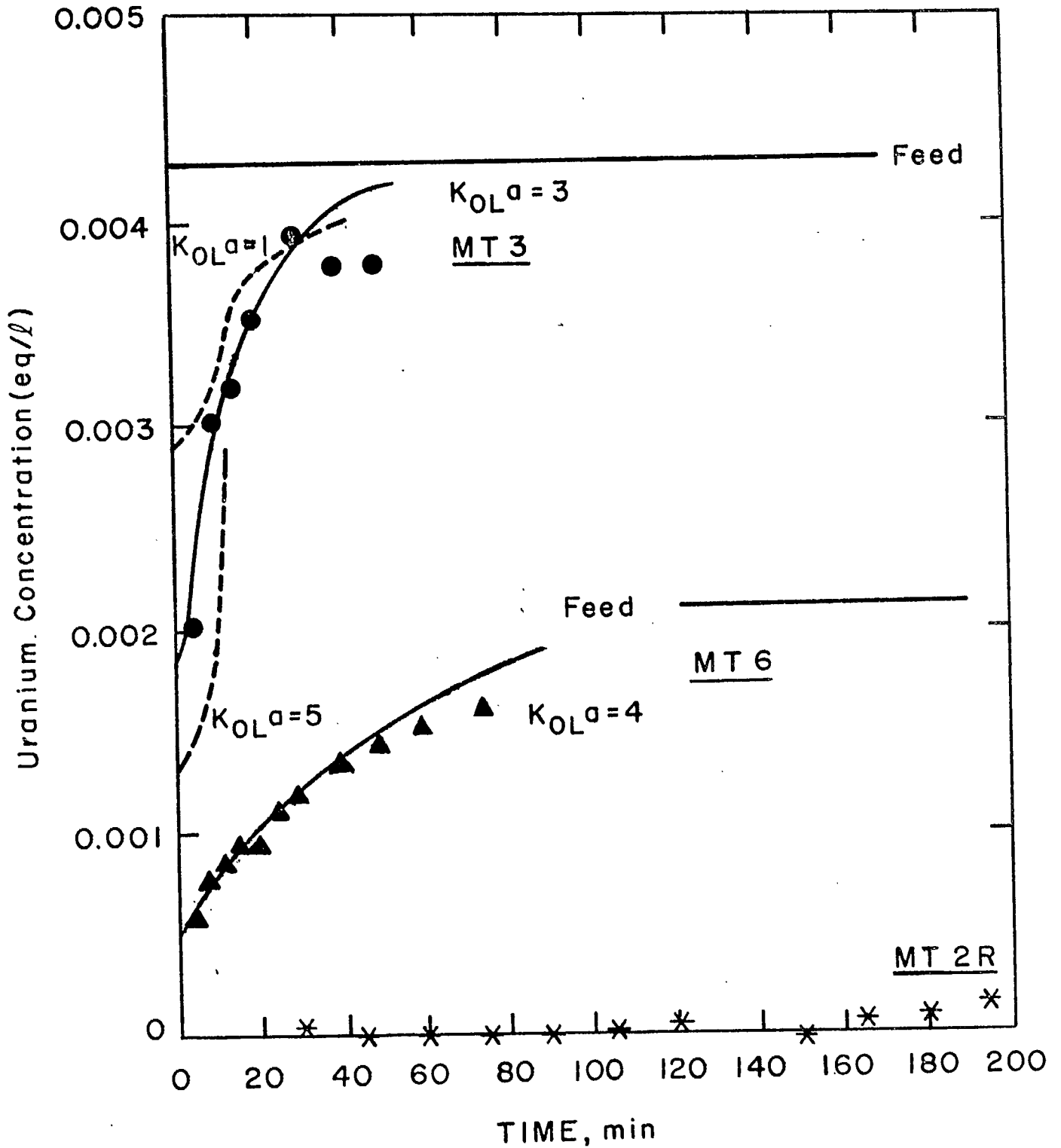


Figure 12. Single-Stage Breakthrough Curves

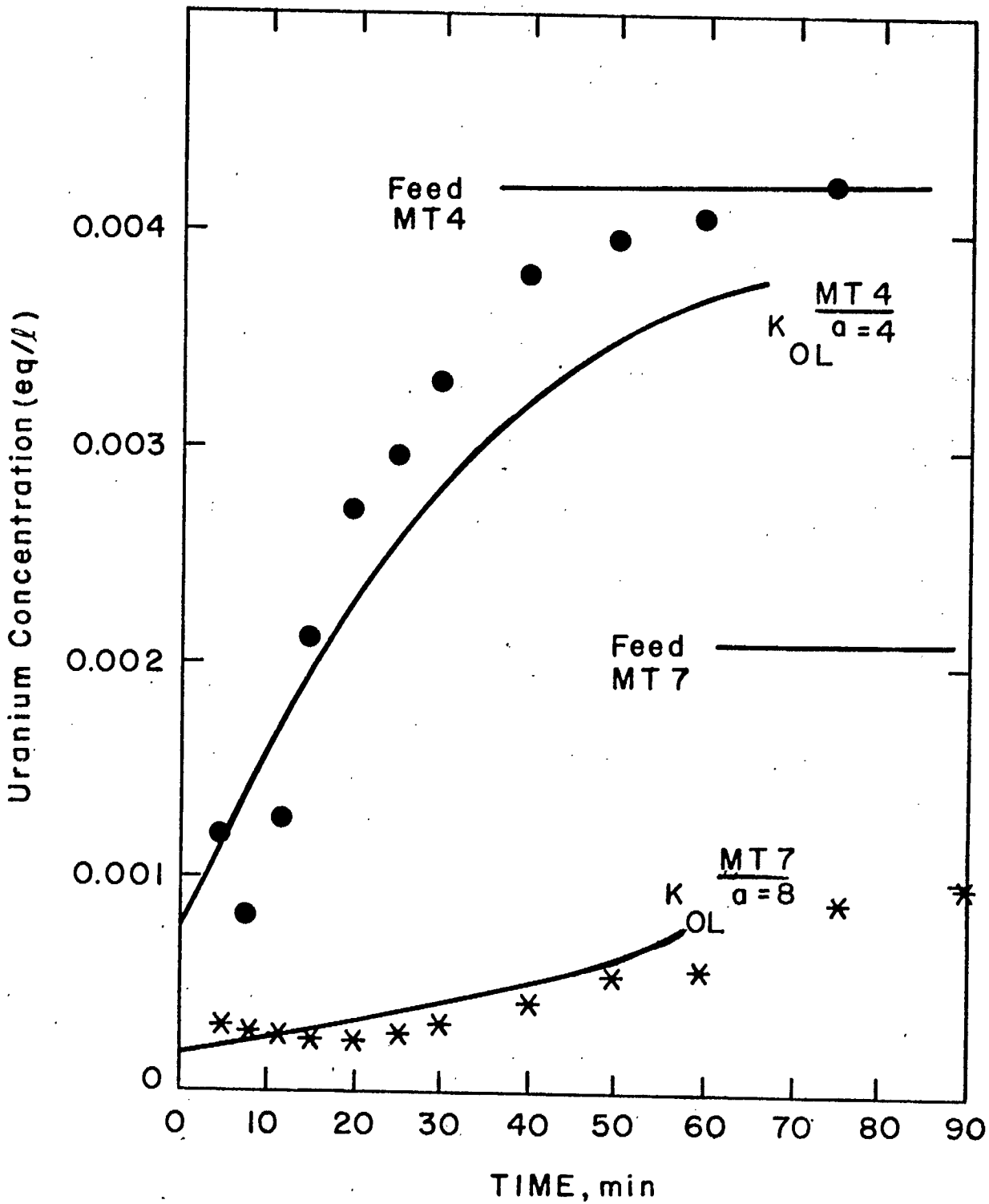


Figure 13. Single-Stage Breakthrough Curves

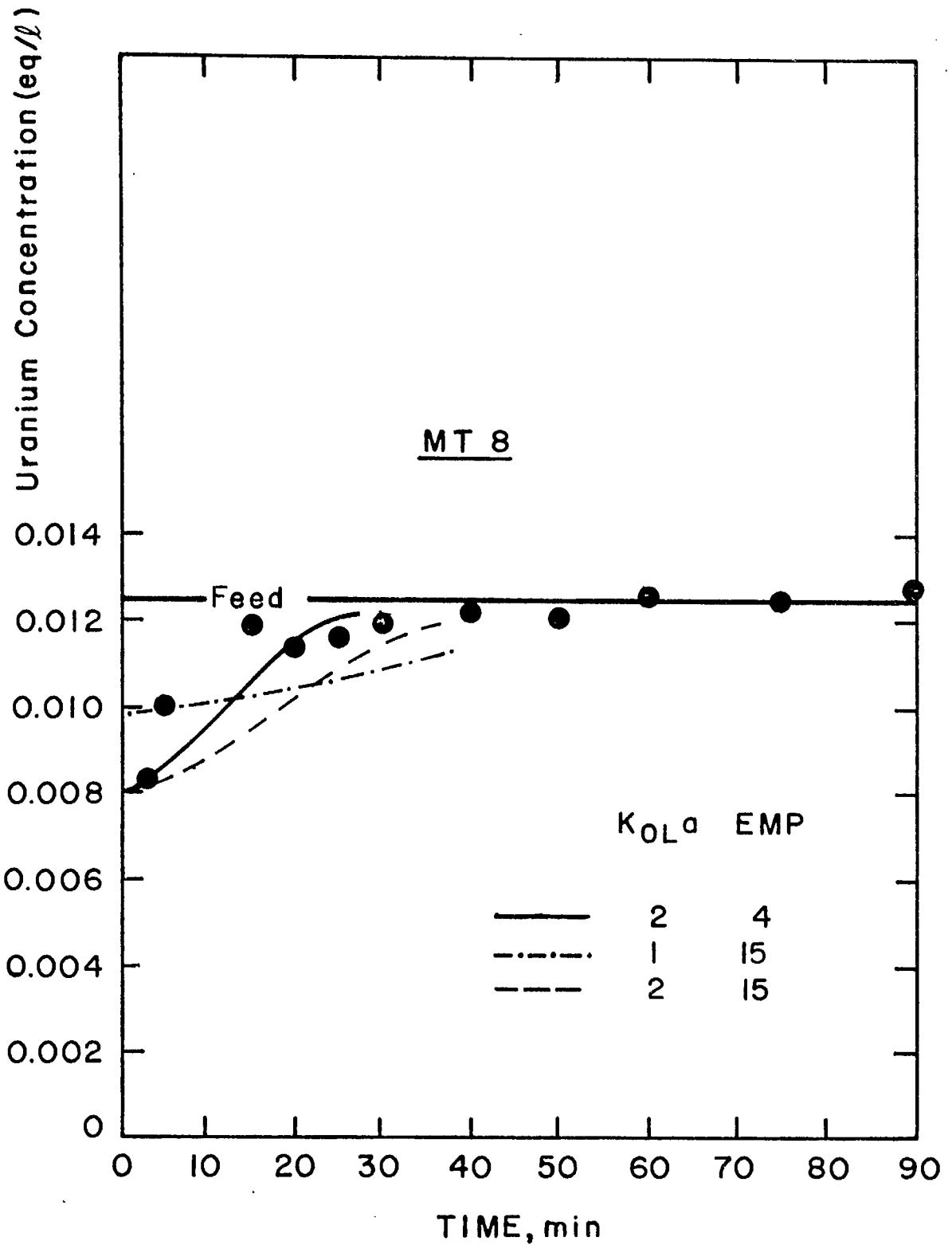


Figure 14. Single-Stage Breakthrough Curves

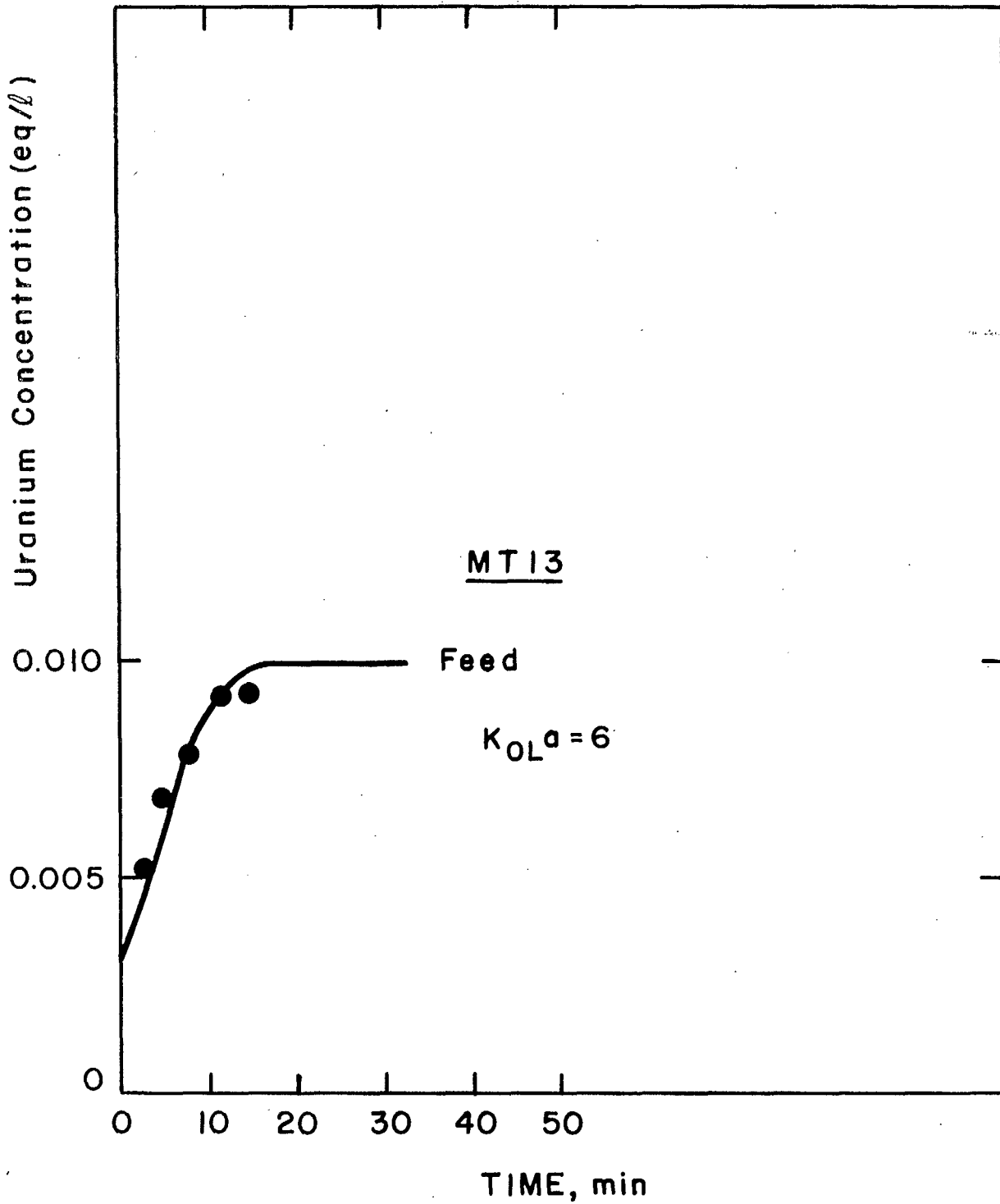


Figure 15. Single-Stage Breakthrough Curves

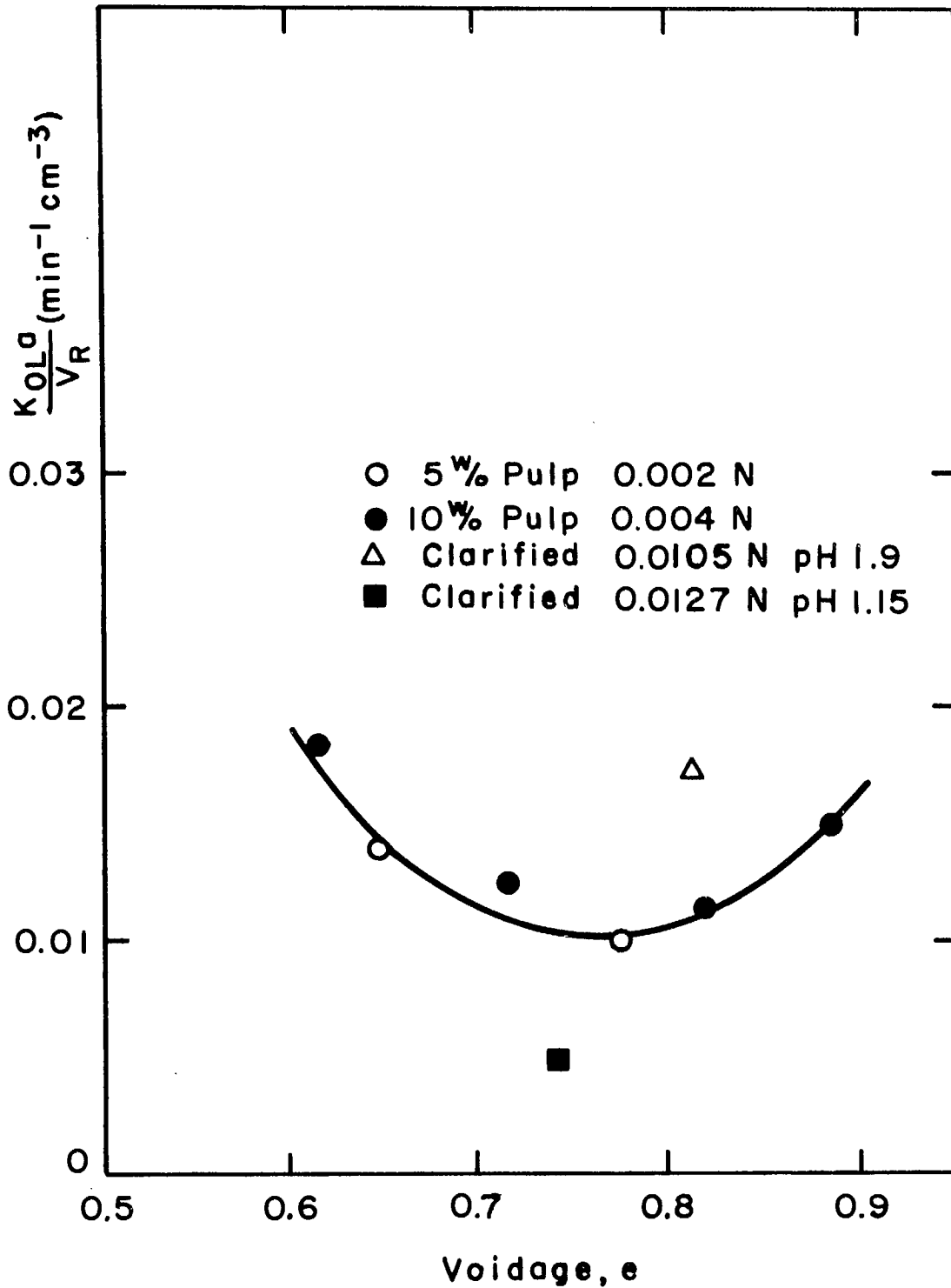


Figure 16. Variation of the Mass Transfer Coefficient with Voidage

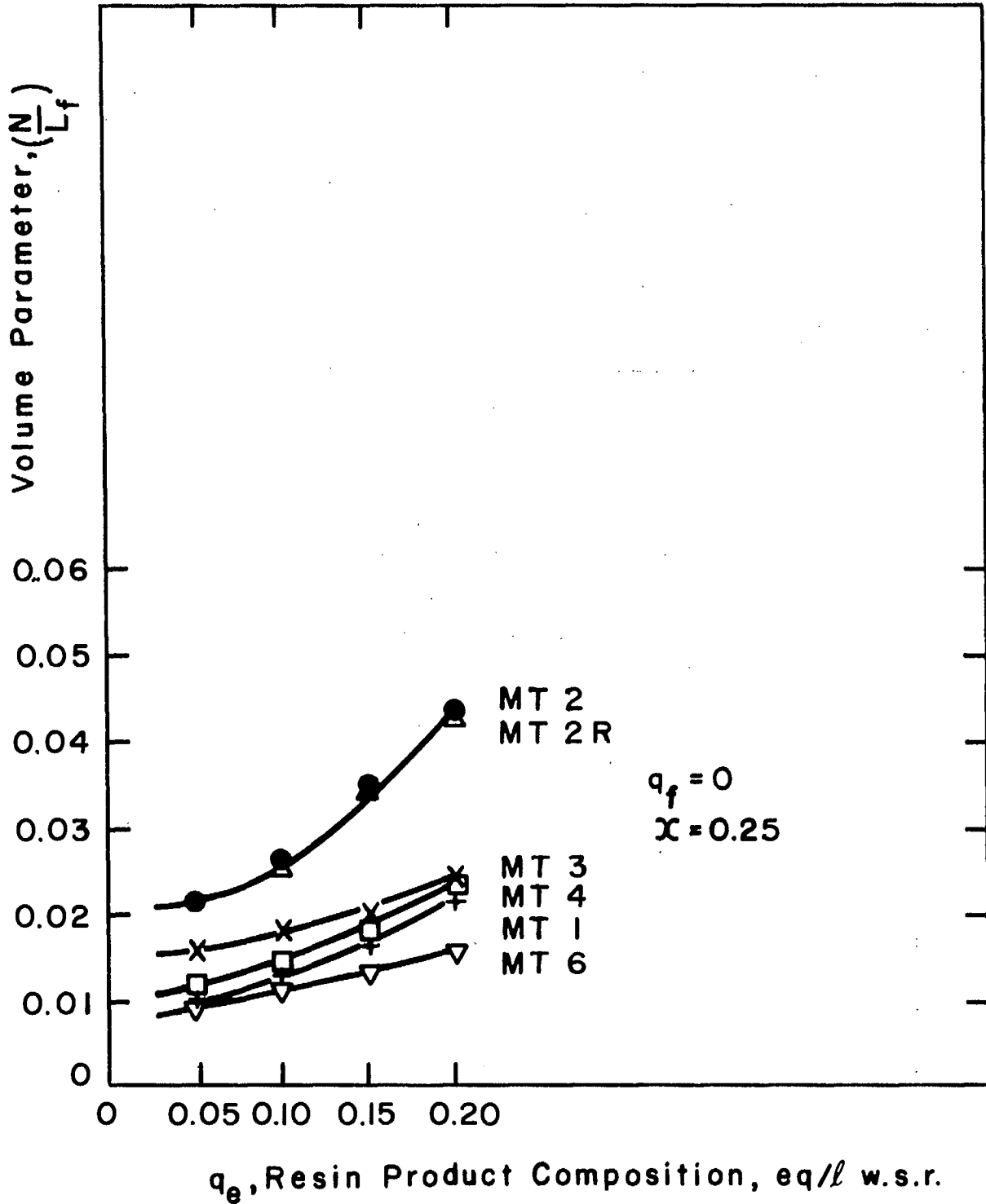


Figure 17. Variation of Column Size with Resin Loading

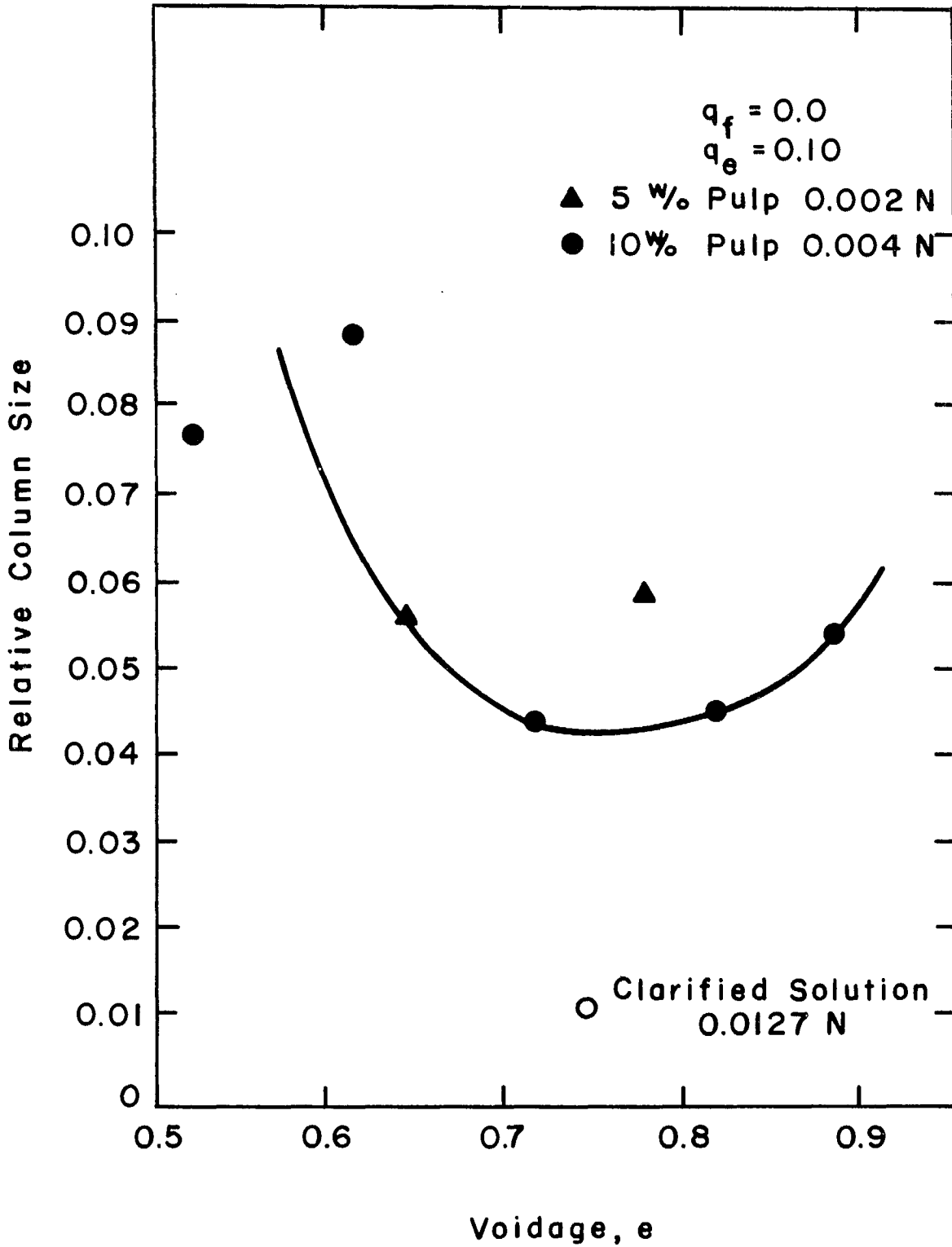


Figure 18. Variation of Column Size with Voidage

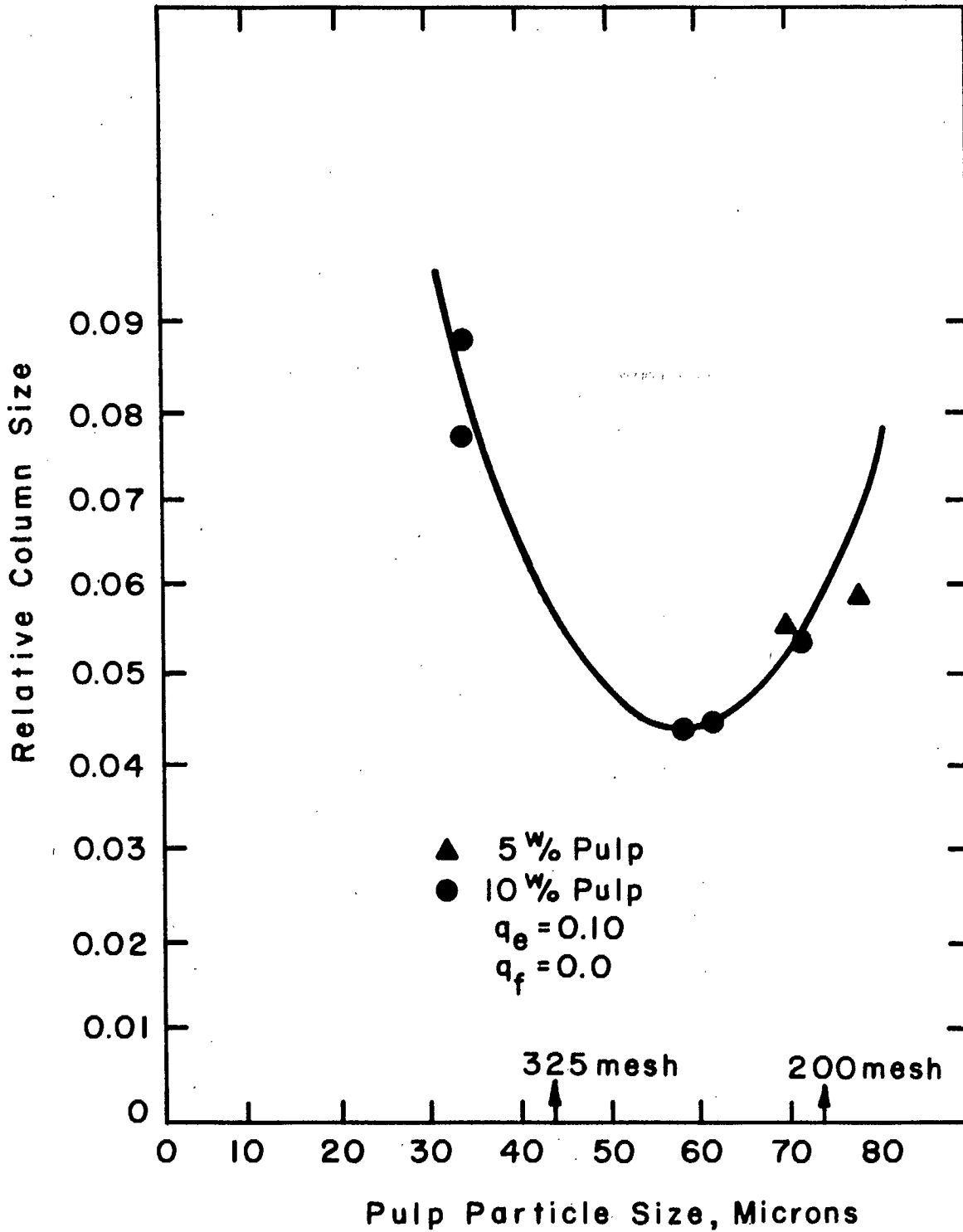


Figure 19. Variation of Maximum Pulp Particle Size with Column Size

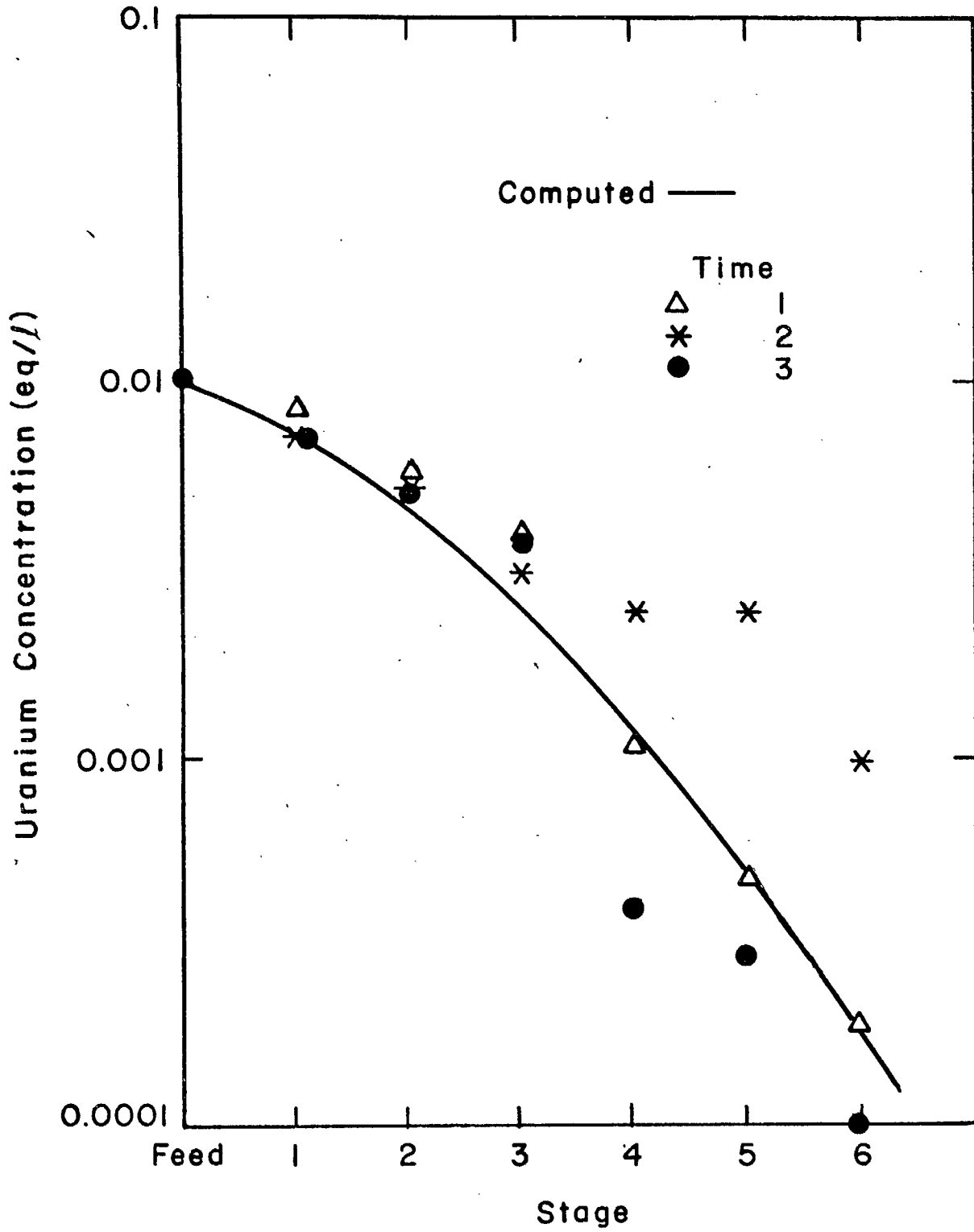


Figure 20. Liquid Composition Profile MT II (Clarified Solution)

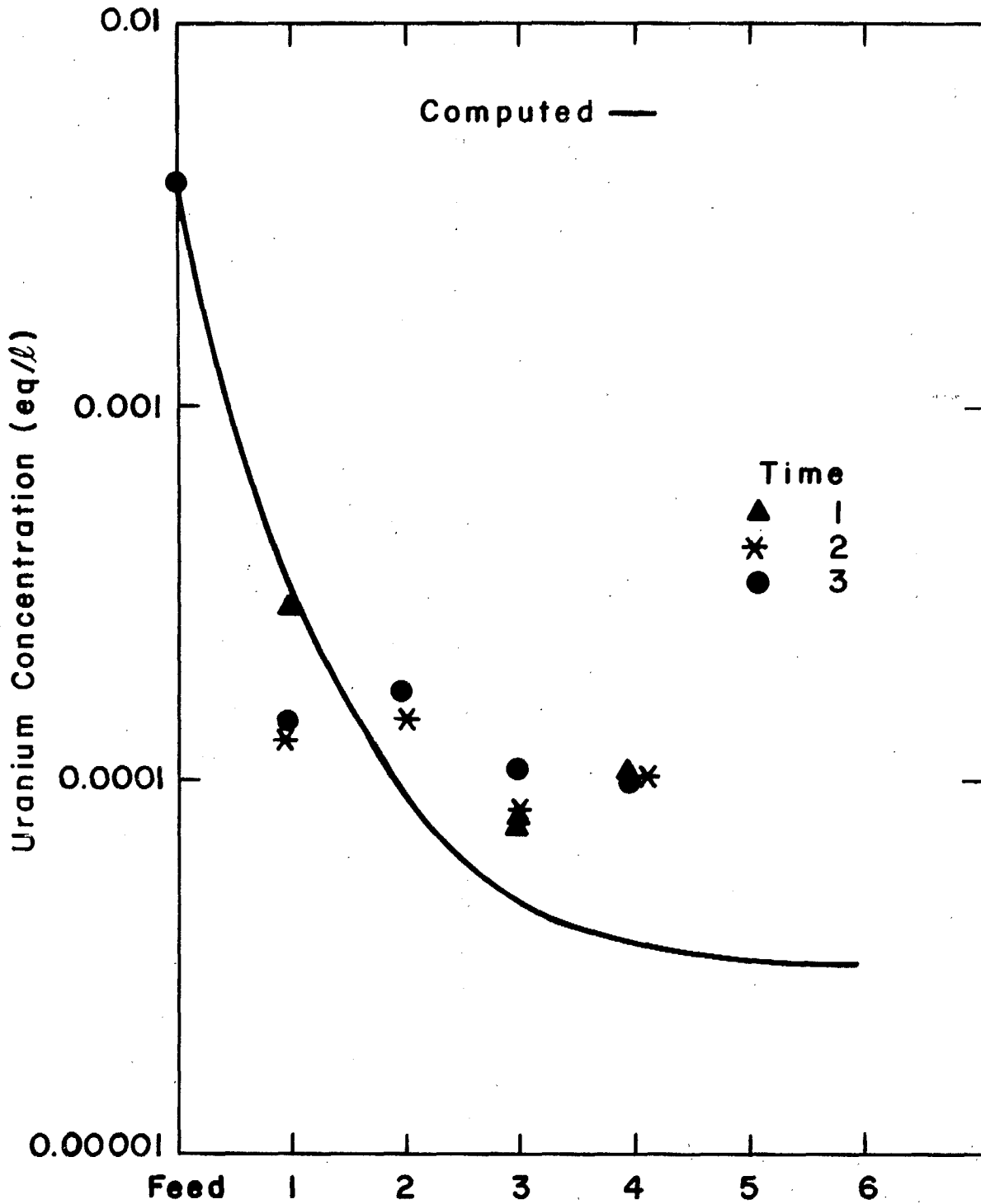


Figure 21. Liquid Composition Profile
MT 12 (10^w% Pulp)

NOMENCLATURE

Symbols in parenthesis are used in computer programme

a	interfacial area per volume of stage, cm^{-1}
a_r	interfacial area per unit volume of resin bead, cm^{-1}
A	cross sectional area of column, cm^2
C(COUT)	liquid composition, eq/l
C*	equilibrium composition in liquid, eq/l
d	particle diameter, cm
D	column diameter, cm
e	voidage (volume fraction liquid between particles)
f	percentage extraction
H	height of a stage, cm
K_{OL}	over-all liquid mass transfer coefficient, cm/min
$K_{OL} a$ (KL)	area based coefficient, min^{-1}
L_f (LF)	solution flow rate, ml/min
L_p (LP)	pulp flow rate ml/min
\bar{L}_f	average flow rate, ml/min
m(EM)	equilibrium line slope
m_o (EMP)	coefficient in equilibrium equation, eq/l.
n	slope or number of stage
N(L)	number of extraction column stages
q(Q)	resin composition, eq/l wet settled resin
Re_p	Reynolds number, $\rho_L dV_T/\mu_L$
t	time, minutes

NOMENCLATURE (Cont'd)

V_S	superficial liquid or pulp velocity, cm/sec
V_T	terminal velocity, cm/sec
V_R (VR)	volume or resin per stage, ml
V_{ST}	volume of a stage
w(WF)	weight fraction of solids in pulp
x(FR)	fraction of resin bed volume removed
y	fraction of stage occupied by resin

Greek

α	$L_f / (K_{OL} a \cdot V_R)$
β	$L_f / (m \cdot V_R)$
μ_L	liquid viscosity, p
μ_p	pulp viscosity, p
ρ_L	liquid density, gm/ml
ρ_R	resin density, gm/l
θ	time, minutes

Subscripts

C	cycle time
e	exit point
f	feed point
i	initial
IN	inlet to a stage
L	liquid flow time

Subscripts (Cont'd)

n	stage number
OUT	outlet from a stage
p	product at end of cycle or pulp
R	resin flow time
SM	Sauter mean

Miscellaneous

eq/l	equivalents per litre
v/o	volume per cent
w/o	weight per cent
wsr	wet settled resin (free, undisturbed settling in water)

APPENDIX 1

Programme for multi-stage extraction column

Programme COLUMN

Line

- 7 $F = \frac{dC_{OUT}}{dt}$, $QP = q_p$, $QS = q_i$, $Y = C_{OUT}$
- 8 $COUT = C_{OUT}$, DIF (see line 165)
- 10 $KL = K_{OL}a$, LF = flow rate of solution in pulp,
LP = flow of pulp
- 14 EMP = equilibrium line parameter, M_o
WF = wt. fraction of solids in pulp
PH = pH of feed pulp
- 17 CF = Feed concentration
- 20 L = number of stages, M = number of calculation
increments
- 23 HT = height of one stage, AREA = area of one stage
- 26 VOID = e, $VR = V_R$ (volume of wet settled resin)
- 30 DQ = range of loading on resin, QP(1) defined in line 7,
LP defined in line 10, FR = volume fraction of bed
moved per cycle
- 33 VOLP = volume fraction of solids in pulp
- 36 VS = superficial pulp velocity
- 38 THETA = liquid contact time in a cycle
0.995 = degree of recovery of solute
- 42 ALPHA = α
- 44 H = calculation time increment
- 45 SIMPA = average C_{OUT} from a stage (see line 197)
- 53 N = number of the stage being calculated
- 58 EM = equilibrium line slope parameter = EMP for
straight equilibrium line
- 59 BETA = β
- 66 Z = number of trial attempt to find correct initial
value of C_{OUT}

Line

67 P = index which equals 1 if trial for initial C_{OUT} is satisfactory

70 COUTO = subroutine for providing value of initial C_{OUT}

73 T = time

74 S = counter for printing instruction, line 109

75 MT = array subscript for COUT (N,MT) indicating time value

81 Y(N) is used to calculate COUT for each stage but values are stored in two dimensional COUT(N,MT)

82 K = counter for subroutine RKSUB

83 NO = storage capacity for subroutine PRED and CORR

85 Make sure initial values stored

125 Print every tenth value of COUT

131 Integrate using initial value of COUT obtained from subroutine COUTO

133 A = initial time value for integral

134 B = final value for integral

141 Increment has to be 2 x H because of halving

163 Check if two sides of equation, named EST1 and EST2 are equal using current values of initial COUT and integral SIMP

165 Obtain difference DIF between estimates to obtain better value of initial COUT by extrapolation

171 If initial COUT satisfactory calculate initial resin composition QS

173 Calculate resin product composition QP from stage above

177 Calculate average liquid outlet composition COUT over one cycle, called SIMPA

SUBROUTINE COUTO

5 INVAL = Current estimate of initial value of C_{OUT}
7 Start calculation with two estimates, INVAL(1) and
 INVAL(2)
10 R = fractional error in initial COUT assumed
12 Predict better estimate of INVAL
13 Check if error acceptable

SUBROUTINE RKSUB

Standard Runge-Kutta starting procedure

SUBROUTINE PRED AND CORR

Standard Milne routines

FUNCTION FUN

Function to be integrated in SIMP
(see line 136 in main programme)

```

PROGRAM COLUMN(INPUT,OUTPUT,TAPE1=INPUT,TAPE5=OUTPUT)
C CALCULATION OF ION EXCHANGE EXTRACTION COLUMN
C MULTISTAGE FLUIDIZED BED COLUMN OPERATED CYCLICALLY
C M. J. SLATER
5 C AUGUST 1972
C SEE MINES BRANCH OTTAWA TECH BULL TB158 FOR DETAILS AND NOMENCLATURE
  DIMENSION F(160),QP(41),QS(40),Y(160)
  COMMON COUT(40,102),DIF(50)
  INTEGER E,G,P,S,U,V,W,X,Z
10 REAL KL,LF,LP
C READ INPUT DATA
C EQUILIBRIUM LINE SLOPE, FRACTION SOLIDS IN PULP, PH OF PULP
C PUT WF=0.0 IF SOLUTION NOT PULP
  READ(1,101) EMP,WF,PH
15 101 FORMAT(3F8.3)
C LIQUID FEED COMPOSITION IN EQUIVALENTS PER LITRE
  READ(1,102) CF
  102 FORMAT(F8.5)
C NUMBER OF STAGES IN COLUMN , NUMBER OF INCREMENTS IN CALCULATION
20  READ(1,103) L,M
  103 FORMAT(2I5)
C HEIGHT OF A STAGE IN COLUMN, CROSS-SECTIONAL AREA OF COLUMN ,CM ANDCM2
  READ(1,104) HT,AREA
  104 FORMAT(2F6.3)
25 C VOIDAGE,VOLUME OF WET SETTLED RESIN IN A STAGE,MASS TRANSFER COEFF.
  READ(1,106) VOID,VR,KL
  106 FORMAT(3F8.3)
C DIFFERENCE IN RESIN COMPOSITION OVER COLUMN,RESIN LOADING, EQ/L OF WSR
C PULP OR SOLUTION FLOW RATE, ML/MIN, FRACTION OF A STAGE TRANSFERRED
30  READ(1,105) DQ,QP(1),LP,FR
  105 FORMAT(4F14.8)
C USE SOLIDS DENSITY TO GET SOLUTION FLOW RATE
  VOLP=WF/(2.67-1.67*WF)
  LF=(1.0-VOLP)*LP
35 C CALCULATE SUPERFICIAL VELOCITY
  VS=LP/(AREA*60.0)
C IF COMPUTING A THEORETICAL CASE SET DQ AND DC,THE DEGREE OF RESIN UTI
C LIZATION AND DEGREE OF EXTRACTION FROM THE LIQUID AND CALCULATE
C THE VALUE OF THETA, THE CYCLE TIME

```

```

40      C SET OPT=1.0
      C IF CHECKING A COLUMN TRIAL SET VALUE OF THETA USED
      C SET OPT=2.0
          OPT=2.0
          IF(OPT.GT.1.0) GO TO 999
45      DQ=0.95*QP(1)
          DC=0.995*CF
          THETA=FR*DQ*VR/(LF*DC)
          GO TO 998
999     CONTINUE
50      THETA=3.0
998     CONTINUE
          WRITE(5,66) QP(1),THETA,L,M,FR
          66  FORMAT(1H1,4X,7HQ(1) ,F14.8,2X,7HTHETA ,F14.8,2X,3HL ,I4,
          22X,2HM ,I5,2X, 3HFR ,F14.8)
55      ALPHA=LF/(KL*VR)
      C CALCULATE INCREMENT FOR CALCULATIONS
          H=THETA/M
          SIMPA=CF
          WRITE(5,65) ALPHA,WF,H
60      65  FORMAT(1H0,8X,9HALPHA IS ,E12.3,4X,6HWF IS ,E12.3,4X,5HH IS ,F6.3)
          WRITE(5,71) HT,AREA,DQ,VS,VOID,VR
          71  FORMAT(1H0,8X,3HHT ,F6.3,2X,5HAREA ,F6.3,2X,3HDQ ,F6.3//8X,3HVS ,
          1F6.3,2X,5HVOID ,F6.3,2X,3HVR ,F8.3,2X)
      C START CYCLE OF CALCULATIONS FOR REQUIRED NUMBER OF STAGES IN COLUMN
65      20  DO 34 N=1,L,1
          WRITE(5,30) N
          30  FORMAT(1H0,10X,13HSTAGE NUMBER ,I3)
      C CALCULATE EQ LINE SLOPE
      C EMP IS PARAMETER WHICH CAN BE ALTERED TO FIT EQUILIBRIUM CURVE
70      C WRITE EM=EMP FOR STRAIGHT EQUILIBRIUM LINE
          EM=EMP/QP(N)
          BETA=LF/(EM*VR)
          WRITE(5,64) LF,EM,KL,CF
          64  FORMAT(1H0,2X,4HLF ,F8.3,2X, 4HEM ,1PE15.7,2X,
          14HKL ,F6.3,2X,4HCF ,F8.5)
75      WRITE(5,25)
          25  FORMAT(1H0,5X,4HTIME,8X,4HCOU)

```

```

C OBTAIN INITIAL VALUES! TRIAL NUMBER IS Z
      Z=0
80      P=0
      10 Z=Z+1
C OBTAIN AN INITIAL VALUE OF LIQUID COMP. AND CHECK BY SIMPSON
90      CALL COUTO(N,Z,CF,P,Y)
C START BY USING RUNGE-KUTTA
85      C SOLVE DIFFERENTIAL EQUATIONS BY MILNE METHOD
91      T=0.0
      S=0
      MT=1
      IF(P.EQ.0) GO TO 40
80      WRITE(5,31) T,Y(N)
      31 FORMAT(1H0,5X,F6.3,6X,E10.3)
      IF(Y(1).GT.CF) GO TO 76
C TIME INCREMENT NUMBER IS MT
40      COUT(N,MT)=Y(N)
95      K=0
      NO=4*L
C START CALCULATION
      DO 55 E=1,N,1
100      55 Y(E)=COUT(E,1)
      MT=2
C EQUATION FOR FIRST STAGE
      F(1)=BETA*(CF-Y(1))/(1.0+ALPHA)
      IF(N.EQ.1) GO TO 60
      DO 8 X=2,N,1
105      C EQUATION FOR OTHER STAGES
      8 F(X)=(ALPHA*F(X-1)+BETA*Y(X-1)-BETA*Y(X))/(1.0+ALPHA)
60      I=3
      3 IF(I) 4,7,4
      4 DO 5 J=1,N,1
110      Y(J+I*N)=Y(J)
      5 F(J+I*N)=F(J)
      6 CALL RKSUB(N,K,INDEX,Y,F,T,H)
      GO TO (1,2), INDEX
      1 F(1)=BETA*(CF-Y(1))/(1.0+ALPHA)
115      IF(N.EQ.1) GO TO 6
      DO 9 W=2,N,1

```

```

          9  F(W)=(ALPHA*F(W-1)+BETA*Y(W-1)-BETA*Y(W))/(1.0+ALPHA)
            GO TO 6
          2  COUT(N,MT)=Y(N)
120      MT=MT+1
            I=I-1
            GO TO 3
C  PROCEED BY MILNE PREDICTOR-CORRECTOR METHOD
          7  F(1)=BETA*(CF-Y(1))/(1.0+ALPHA)
125      IF(N.EQ.1) GO TO 61
            DO 11 V=2,N,1
          11 F(V)=(ALPHA*F(V-1)+BETA*Y(V-1)-BETA*Y(V))/(1.0+ALPHA)
          61 CALL PRED(N,NO,Y,F,T,H)
            F(1)=BETA*(CF-Y(1))/(1.0+ALPHA)
130      IF(N.EQ.1) GO TO 62
            DO 12 U=2,N,1
          12 F(U)=(ALPHA*F(U-1)+BETA*Y(U-1)-BETA*Y(U))/(1.0+ALPHA)
          62 CALL CORR(N,NO,Y,F,T,H)
            COUT(N,MT)=Y(N)
135      IF(P.EQ.0) GO TO 63
            IF(T.GE.THETA) GO TO 95
C  ORGANIZE PRINTING OF RESULTS
            S=S+1
            IF(S.LT.10) GO TO 63
            S=0
140      95  WRITE(5,31) T,COUT(N,MT)
          63  MT=MT+1
            IF(T.LT.THETA) GO TO 7
C  INTEGRATE TO CHECK IF INITIAL VALUE IS CORRECT
145  C  (SIMPSONS RULE
            A=0.0
            B=THETA
            MT=101
            SUMB=FUN(N,CF,MT)
150      MT=1
            SUMA=FUN(N,CF,MT)
            SUM1=SUMA+SUMB
            SUM2=0.0
            T=A+H*2.0

```

```

155      MT=MT+2
      32 SUM2=SUM2+FUN(N,CF,MT)
         T=T+H*2.0
         MT=MT+2
         IF(T+H-B) 32,50,42
160      42 SUM4=0.0
         MT=1
         T=A+H
         MT=MT+1
      13 SUM4=SUM4+FUN(N,CF,MT)
         T=T+H*2.0
         MT=MT+2
         IF(T-B) 13,50,21
      21 SIMP=H/3.0*(SUM1+2.0*SUM2+4.0*SUM4)
50      CONTINUE
170      C OBTAIN NEW ESTIMATE OF INITIAL VALUE OF Y(N)
         IF(N.EQ.1) GO TO 81
         CIN=COU((N-1),1)
         GO TO 82
      81  CIN=CF
175      C INTERPOLATION PROCEDURE TO GET NEW VALUE
      82  EST1=EM*(1.0+ALPHA)*COU(N,1)-EM*ALPHA*CIN
         EST2=QP(N)-SIMP*LF/VR
         DIF(Z)=EST1-EST2
         IF(P.EQ.0) GO TO 10
180      WRITE(5,83) DIF(Z)
      83  FORMAT(1H0,5X,7HDIF IS ,E10.3)
      C  CALCULATE INITIAL RESIN COMPOSITION
      C  INTEGRAL EQUATION USED
         QS(N)=QP(N)-SIMP*LF/VR
185      C  TAKE TRANSFER OF RESIN INTO ACCOUNT
         QP(N+1)=(QS(N)-(1.0-FR)*QP(N))/FR
         WRITE(5,43) QS(N),QP(N)
      43  FORMAT(1H0,5X,29HINITIAL RESIN COMPOSITION IS ,F14.8/
          38X,27HFINAL RESIN COMPOSITION IS ,F14.8)
190      C AVERAGE LIQUID OUTLET COMPOSITION
         A=0.0
         B=THETA
         MT=1

```

```

195      SUM1P=COUT(N,1)+COUT(N,101)
        SUM2P=0.0
        T=A+H*2.0
        MT=MT+2
33      SUM2P=SUM2P+COUT(N,MT)
        T=T+H*2.0
200     MT=MT+2
        IF(T+H-B) 33,34,35
35      SUM4P=0.0
        MT=1
        T=A+H
205     MT=MT+1
36      SUM4P=SUM4P+COUT(N,MT)
        T=T+H*2.0
        MT=MT+2
        IF(T-B) 36,34,37
210     37      SIMPA=H/3.0*(SUM1P+2.0*SUM2P+4.0*SUM4P)/THETA
        WRITE(5,38) SIMPA
C      AVERAGE LIQUID COMPOSITION IS SIMPA
38      FORMAT(1H0,5X,22HAVERAGE LIQUID OUT IS ,E10.3)
34      CONTINUE
215     76      STOP
        END

```

C THIS SUBROUTINE PROVIDES A NEW ESTIMATE OF INITIAL LIQUID

SUBROUTINE COUTO(N,Z,CF,P,Y)

DIMENSION INVAL(50),Y(160)

COMMON COUT(40,102),DIF(50)

5

REAL INVAL

INTEGER Z,P

INVAL(1)=0.3*CF

INVAL(2)=0.1*CF

IF(Z.LE.2) GO TO 5

10

R=DIF(Z-1)/INVAL(Z-1)

SLOPE=(INVAL(Z-2)-INVAL(Z-1))/(DIF(Z-2)-DIF(Z-1))

INVAL(Z)=INVAL(Z-1)-SLOPE*DIF(Z-1)

IF(R.GT.0.01) GO TO 4

U=-R

15

IF(U.LT.0.01) GO TO 3

4 IF(Z.EQ.50) GO TO 3

GO TO 5

3 P=1

5 Y(N)=INVAL(Z)

20

RETURN

END


```

SUBROUTINE RKSUB( N,K,INDEX,Y,F,T,H)
DIMENSION CAPY(40),PO(40),Y(N),F(N)
K=K+1
GO TO (1,2,3,4,5), K
5      2 DO 12 I=1,N,1
      PO(I)=F(I)
      CAPY(I)=Y(I)
12     Y(I)=CAPY(I)+0.5*H*F(I)
11     T=T+0.5*H
10     1 INDEX=1
      RETURN
      3 DO 13 I=1,N,1
      PO(I)=PO(I)+2.0*F(I)
15     13 Y(I)=CAPY(I)+0.5*H*F(I)
      INDEX=1
      RETURN
      4 DO 14 I=1,N,1
      PO(I)=PO(I)+2.0*F(I)
20     14 Y(I)=CAPY(I)+H*F(I)
      GO TO 11
      5 DO 15 I=1,N,1
25     15 Y(I)=CAPY(I)+(PO(I)+F(I))*H/6.0
      INDEX=2
      K=0
      RETURN
      END

```

```

SUBROUTINE PRED(N,NO,Y,F,T,H)
DIMENSION Y(NO),F(NO)
DO 1 I=1,N,1
5      Y(I+N)=Y(I)
      Y(I+2*N)=Y(I+N)
      Y(I+3*N)=Y(I+2*N)
      YT=Y(I+3*N)
      Y(I)=YT+8.0*H*(F(I)-0.5*F(I+N)+F(I+2*N))/3.0
10     F(I+N)=F(I)
      1 F(I+2*N)=F(I+N)
      T=T+H
      RETURN
      END

```

```
SUBROUTINE CORR(N,NO,Y,F,T,H)
DIMENSION Y(NO),F(NO)
DO 2 I=1,N,1
YT=Y(I+2*N)+H*(F(I)+4.0*F(I+N)+F(I+2*N))/3.0
5 2 Y(I)=YT+(YT-Y(I))/29.0
RETURN
END
```

```
FUNCTION FUN(N,CF,MT)
COMMON COUT(40,102),DIF(50)
IF(N.EQ.1) GO TO 1
FUN=COUT((N-1),MT)-COUT(N,MT)
GO TO 2
1 FUN=CF-COUT(N,MT)
2 CONTINUE
RETURN
END
```

APPENDIX 2

Programme for single-stage calculations

Symbols as in Appendix 1

PROGRAM STAGE(OUTPUT,TAPES=OUTPUT)
C CALCULATION OF CONCENTRATION OUTPUT CURVES FOR PULP
C THROUGH ONE FLUIDIZED BED OF RESIN
C EQUILIBRIUM LINE SLOPE VARIATION WITH RESIN COMPOSITION

```
5      REAL LP,KL
      DIMENSION COUT(21)
      DATA PH,CF,LP,WF,VR,VOID/1.9,0.0101,1000.0,0.0,350.0,0.814/
      WRITE(5,3) PH,CF,LP,WF,VR,VOID
3      FORMAT(1H0,5X,3HPH=,F6.3,4X,3HCF=,F8.5,4X,3HLP=,F8.3,4X,
10     13HWF=,F6.3,4X,3HVR=,F8.3,4X,5HVOID=,F6.3)
      VOLP=WF/(2.67-1.67*WF)
      WRITE(5,5)
5      FORMAT(1H0,7X,4HCOUT,10X,5HTHETA,10X,2HQP)
      EMP=1.0
15     DO 20 M=1,4,1
      EMP=EMP+1.0
      KL=1.0
      DO 4 K=1,10,1
      LF=(1.0-VOLP)*LP
20     WRITE(5,6) KL, EMP
6      FORMAT(1H0,5X,3HKL=,F6.3,3X,4HEMP=,F4.1)
      N=1
      AREA=0.0
      QP=0.0
25     THETA=0.0
10     CONTINUE
      IF(N.LE.2) GO TO 13
      EM=EMP/QP
      GO TO 14
30     13 EM=200.0
14     CONTINUE
      ALPHA=LF/(KL*VR)
      BETA=LF/(EM*VR)
      X=-BETA/(1.0+ALPHA)
35     COUT(N)=CF*(1.0-(EXP(X*THETA)))/(1.0+ALPHA)
      WRITE(5,2) COUT(N),THETA,QP
2      FORMAT(1H0,5X,F8.5,5X,F8.3,5X,F8.5)
      IF(THETA.LT.18.0) GO TO 7
      LF=(1.0-VOLP)*(2130.0+6.1*THETA)
```

```

40      GO TO 1
7       LF=(1.0-VOLP)*(1000.0+70.0*THETA)
1       CONTINUE
        IF(THETA.GT.200.0) GO TO 4
        IF(THETA.LT.20.0) GO TO 8
45      THETA=THETA+20.0
        DTHETA=20.0
        GO TO 9
8       THETA=THETA+2.0
        DTHETA=2.0
50      9       CONTINUE
        IF(N.EQ.1) GO TO 11
        AINC=(CF-(COUT(N-1)+COUT(N))/2.0)*DTHETA
        AREA=AREA+AINC
        QP=LF*AREA/VR
55      11      CONTINUE
        N=N+1
        GO TO 10
4       KL=KL+1.0
20      CONTINUE
60      STOP
        END

```

