Prediction of Resistance in Constant-Pressure Cake Filtration

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Methods of predicting resistance in constant-pressure cake filtration have been classified into three groups, (1) basic particle and cake properties, (2) permeability tests, and (3) small-scale filtration tests. Methods 2 and 3 involve the concept of specific filtration resistance, a property characteristic of each unit mass of deposited cake, and these methods were extensively investigated in the laboratory. Specific resistances for three chemical slurries were determined by laboratory filtrations and filter- and compression-permeability tests. Results of these investigations showed that specific filtration resistance could be predicted from compression-permeability test data.

Filtration tests made on commercial-scale equipment operating on pearl cornstarch illustrated the correlation between predicted and actual specific resistance values, the resistance predicted from compression-permeability test data agreeing very well with the actual resistance of the prefilt.

Prediction of the characteristics of filtration usually involves consideration of some form of resistance. The problem of determining the resistance has long been the subject of theoretical and experimental investigations, and the methods used may be classified in decreasing fundamental order as those involving (1) basic particle and cake properties, (2) permeability tests, and (3) small-scale filtration tests.

In the first of these, the deposited filter cake is considered as a porous medium and the filtrate rate the resulting fluid permeability, expressible in terms of known particle-cake properties. This was qualitatively stated as early as 1908 by Hatschek (8) and more recently quantitatively treated through the development of the Kozeny-Carman and similar equations (1 to 5, 9, 12, 13, 17). Unfortunately, the determination of the necessary particle-cake properties is not a simple matter for cake-filtration materials. This and other complications of the basicproperties method led to the development of methods 2 and 3, which involve specific resistance, a property characteristic of each unit mass of deposited

The basic rate equation

$$\frac{1}{A} \frac{dV}{d\theta} = \frac{g_c \Delta P}{\mu (R_d + R_c + R_m + R_1)} \tag{1}$$

can be written in terms of specific resistance of the cake α by replacing R_c by $(W_c/A)\alpha$. In like manner the septum resistance R_m can be expressed in terms of an equivalent cake-solids weight W_m , and Equation (1) becomes

$$\frac{1}{A}\frac{dV}{d\theta} \tag{2}$$

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$$= \frac{g_c \Delta P}{\mu \left(\frac{W_c}{A} \alpha + \frac{W_m}{A} \alpha + R_d + R_1\right)}$$

Equations (1) and (2) are generally considered applicable to conditions of fixed cake weight (permeability) and variable cake weight (filtration). Deposition-zone resistance (R_d) is zero for the permeability conditions and of only theoretical interest for the filtration conditions; therefore, when apparatus resistance R_1 is negligible, Equation (2) reduces to

$$\frac{1}{A}\frac{dV}{d\theta} = \frac{g_c \Delta P}{\mu \left(\frac{W_c}{A}\alpha + \frac{W_m}{A}\alpha\right)}$$
(3)

The specific resistance α of all materials is a function of particle-to-particle compacting pressure, which is related to fluid pressure drop across the cake. The so-called "incompressible" materials result, simply, from a limitation of the pressure range. Such cases afford no problem since a single filtration or permeability test provides the specific resistance value which is applicable throughout the specified pressure range. For compressible materials, however, the problem of determining the relationship of specific resistance to compacting pressure or fluid pressure drop is not simple. For each material this determination must be made experimentally, and various tests have been applied with different degrees of success.

In the application of Equation (3) to filtration, the instantaneous filter cake weight W_c is directly proportional to filtrate volume, $W_c = w_c V$, and W_m may be written $W_m = w_c V_m$. Substituting these relationships into Equation (3) and integrating between V and $-V_m$ (volume limits) corresponding to θ and $-\theta_m$ (time limits) give the integrated form of the filtration equation:

$$(V + V_m)^2 = K(\theta + \theta_m) \tag{4}$$

where

$$K = \frac{2A^2g_c\Delta P(1 - ms)}{\mu\rho s\alpha}$$
 (5)

which is valid for proper cake filtrations. The particular form and treatment of Equations (3) and (4) used by various investigators appear to be a matter of individual preference. For utility the treatment of Ruth et al. (14, 15, 16) is considered desirable. Differentiation of Equation (4) gives

$$d\theta/dV = \frac{2}{K} (V + V_m) \qquad (6)$$

and when $\theta = \theta_{n+1} - \theta_n$ is taken as corresponding to $(V_{n+1} + V_n)/2$ then $d\theta/dV \equiv \Delta\theta/\Delta V$. This identity is of considerable utility in the treatment of test filtration data. For a consistent value of ΔV a plot of $\Delta\theta/\Delta V$ vs. $(V_{n+1} + V_n)/2$ is linear with slope 2/K. Specific filtration resistance α of a material, therefore, can be calculated by use of time-filtrate volume discharge data to evaluate the slope for use in Equation (5). Septum resistance is not needed for the calculation but is easily determined when desired by use of the $\Delta\theta/\Delta V$ vs. $(V_{n+1} + V_n)/2$ plot intercent.

Considering the filtrate passage through filter cakes as a phenomenon of flow through porous media of unknown basic properties led to the concept of permeability testing to determine specific resistance. Under certain assumptions it is intuitively apparent that filtration should be expressible in terms of fixedweight bed permeability tests. These data can be divided into two types: (1) filter permeability, in which hydraulic pressure drop (ΔP) produces variable compacting pressure across the cake thickness, and (2) compression permeability, wherein presumably constant compacting pressure is applied independently of the relatively low-magnitude fluid pressure drop. The presumption of constant compacting

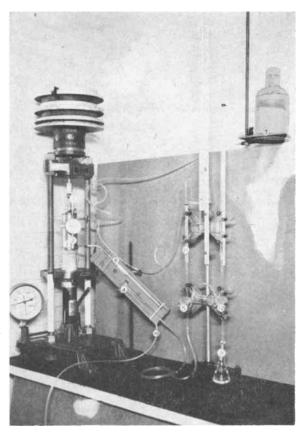


Fig. 1. Compression-permeability test-equipment assembly.

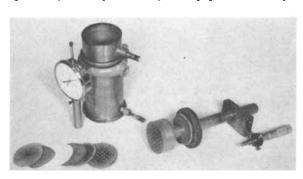


Fig. 2. Compression-permeability test cell.

pressure across the compression-permeability cake thickness was shown to be reasonably valid by Grace (5) for cakes of thickness-diameter ratio not greater than 0.6. Equation (3) is applicable to both types of permeability test, but the respective calculated α values do not bear the same significance. Filter-permeability tests have been advocated as theoretically valid for predicting filtration resistance, and Hoffing and Lockhart (10) reported successful results for a limited investigation of a single, relatively incompressible material.

The compression-permeability test, however, is more fundamental. In this test the cake solids are placed in mechanical compression by means of a stress-loaded perforated piston, and fluid permeability is determined after each stress-loading compaction. The test was first described and discussed in 1946 by Ruth (14). He used the expression

$$\alpha_{avg} = \frac{P - 0}{\int_{-\infty}^{P} \frac{dP_m}{dt}} \tag{7}$$

to relate compression-permeability specific resistance $[\alpha_p = f(P_m)]$ to predicted specific filtration resistance (α_{avg}) . A verification of Equation (7) was obtained in 1953 by Grace (5, 6, 7) in an extensive study of filter-cake resistance and compressibility.

Tiller (18, 19) showed that Equation (7) gives the ultimate resistance approached as $dV/d\theta$ becomes zero. He developed the expression

$$\alpha = \frac{P - \frac{\mu R_m}{g_c} \left(dV/d\theta \right)}{\int_{-R_m/g_o}^{P - (R_m/g_o) (dV/d\theta)} \frac{dP_m}{g_c}} \tag{8}$$

for the average filtration resistance at any

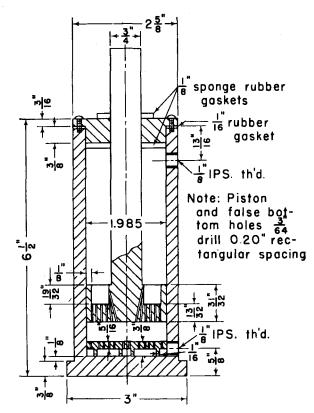


Fig. 3. Compression-permeability test-cell details.

time. The term $(\mu R_m/g_c)$ $(dV/d\theta)$ is the pressure at the cake-septum interface and represents the pressure drop through the septum when the apparatus resistance is negligible. Equation (8) reduces to Equation (7) when the term $(\mu R_m/g_c)$ $(dV/d\theta)$ approaches zero or when the filtration pressure is large compared with the pressure drop through the septum.

LABORATORY TESTS

The comparison among laboratory filtration tests and filter- and compression-permeability tests, with commercial-plant filtrations, is of theoretical interest as well as practical application. These tests were extensively investigated (11) to develop and illustrate the correlation between predicted and actual specific-resistance values. Laboratory tests were made on slurries of commercial-grade calcium carbonate, commercial-grade barium sulfate, and chemically pure titanium dioxide.

Compression-permeability tests were made in the apparatus shown in Figures 1 to 3. In these tests a thickened (settled and decanted) slurry of the test material was poured into the cell (cross-sectional area 3.095 sq. in.) and the cake-forming solids were retained between two filter papers backed by a fine (200-mesh) and a coarse (40-mesh) screen. The perforated piston of the cell was then inserted and successively loaded by weight increments to a compressive stress of approximately

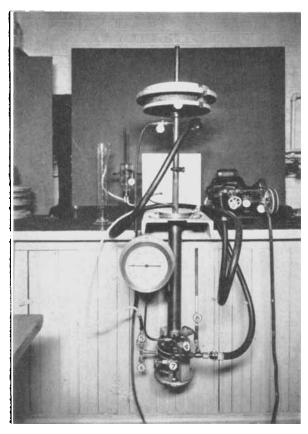


Fig. 4. Laboratory constant-pressure test filter assembly.

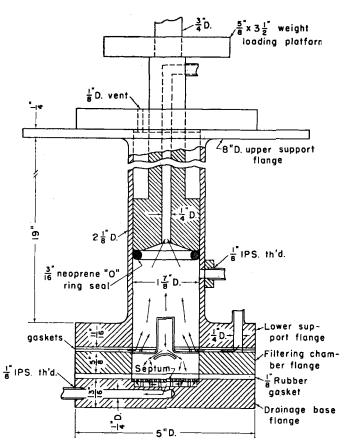


Fig. 5. Schematic of laboratory test filter.

82 lb./sq. in. and by a small Carver hydraulic press from 82 to 743 lb./sq. in. Load transfer from weights to hydraulic was accomplished without removing the weights. After cake compaction had developed at each compressive stress, the filtrate permeability of the cake was determined by use of a low-magnitude fluid-head driving force. From Equation (3), with the notation $W_t = W_c + W_m$ substituted, values of $(\alpha_p)(W_t)$ were calculated for the data of two tests of different cake weight W_{c_1} and W_{c_2} . The results were plotted as $\log (\alpha_p)(W_t)$ vs. $\log P_m$ (curves 1 and 2 of Figures 6, 7, and 8). These plots were used to determine α_p by the identical Equations (8) and (9).

$$\alpha_p = \Delta(\alpha_p)(W_i)/\Delta W \tag{8}$$

$$\alpha_{p} = g_{e} \left\{ \frac{A^{2}(\Delta P)_{2}}{\mu_{2} \frac{(dV)}{(d\theta)_{2}}} - \frac{A^{2}(\Delta P)_{1}}{\mu_{1} \frac{(dV)}{(d\theta)_{1}}} \right\}$$

$$\left\{ \frac{1}{W_{e_{2}} - W_{e_{3}}} \right\}$$
(9)

The calculation procedure indicated in Equation (9) is referred to as a differential analysis and compensates for cake-septum interaction resistance and fluid entrance and exit losses across the cake which occur in testing. The differential-analysis calculation is more realistic than the use of corrections for empty-apparatus flow resistance.

From plots of log α_p vs. log P_m (curves 3 of Figures 6, 7, and 8), relationships of the form $\alpha_p = hP_m^b$ were determined for

each test material. These were used analytically to obtain expressions for predicted specific filtration resistance $(\alpha_{asq}$ and $\alpha_{mean})$ by means of Equation (7) and the expression

$$\alpha_{mean} = \frac{\int_0^P \alpha_p \, dP_m}{P - 0} \tag{10}$$

Plots of α_{mean} and α_{avg} are shown as curves 4 and 5 of Figures 6, 7, and 8. Data and results from the compression-permeability testing of titanium dioxide are given in Tables 1 and 4* and are shown in Figure 6. Data for the tests on barium sulfate and calcium carbonate given in Tables 2 and 3* were treated similarly (11) and the results are shown in Figures 7 and 8.

The laboratory constant-pressure filtrations were made in the small test filter shown in Figures 4 and 5. Filter area for the apparatus was 2.768 sq. in.

For the laboratory filtration test approximately 800 ml. of test slurry was pumped into the apparatus and maintained throughout the test in a uniformly agitated condition by the recirculating pump and system shown in Figure 5. Filtration pressure was varied by changing weight loading on the hydraulic piston. From the time-filtrate-

volume-discharge data, plots of $\Delta\theta/\Delta V$ vs. $(V_{n+1} + V_n)/2$ were made and the slope (2/K) values were determined. Specific filtration resistance (α) values calculated with Equation (5) are plotted in Figures 6, 7, and 8.

The laboratory constant-pressure test filter apparatus was also used for making the filter-permeability tests, which consisted in placing a quantity of thickened (settled and decanted) slurry in the filter-chamber flange, assembling the apparatus, filling with clear filtrate, applying pressure by means of load weights, and determining the time-filtrate-volume-discharge data. Specific resistance (α) values calculated with Equation (3) are plotted in Figures 6, 7, and 8.

When the suitability of the compression- and filter-permeability tests for predicting specific filtration resistance was considered, the calculated results of the laboratory filtrations were taken as the criteria. This was justified by determining the average-specific-resistance-vs.time relationship for each material by the method proposed by Tiller (18, 19). For the three materials equilibrium compaction was apparently obtained within a very short time and the specific resistance was nearly constant for times greater than 3 min. Since the laboratory filtrations were carried out for periods of not less than 5 min., equilibrium compaction was undoubtedly obtained.

Comparison of laboratory-filtration

^{*}Tabular material has been deposited as document 5588 with the American Documentation Institute, Photoduplication Service, Library of Congress, Washington 25, D. C., and may be obtained for \$1.25 for photoprints or 35-mm. microfilm.

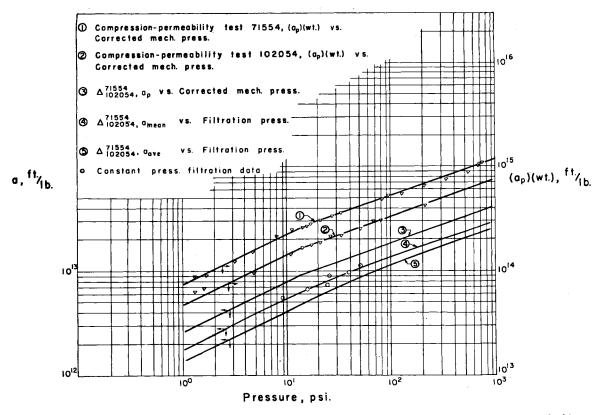


Fig. 6. Variation of specific resistance with corrected mechanical pressure for chemical-pure titanium dioxide.

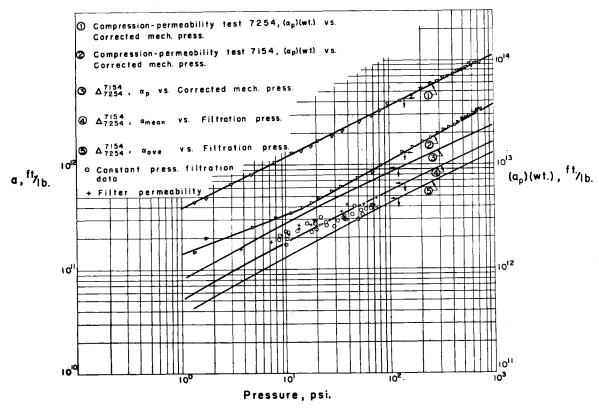


Fig. 7. Variation of specific resistance with corrected mechanical pressure for commercial-grade barium sulfate.

specific resistance, filter-permeability specific resistance, and compression-permeability predicted specific resistance $(\alpha_{asg}$ and $\alpha_{mean})$ is shown in Figures 6, 7, and 8. For all three materials the best

prediction of specific filtration resistance was obtained from the compression-permeability test data, and the values of α_{mean} calculated from Equation (10) were at least as good, if not better, then α_{avg}

calculated from Equation (7). Reasonable filter-permeability test results could not be obtained for cakes of titanium dioxide, since increasing fluid pressure drop resulted in decreasing specific resistance

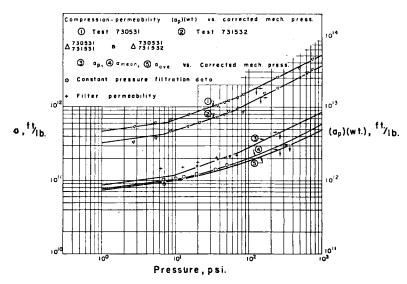


Fig. 8. Variation of specific resistance with corrected mechanical pressure for commercialgrade calcium carbonate.

due to cake cracking and channeling. The filter-permeability specific-resistance results obtained for the calcium carbonate were consistently and significantly higher than the actual filtration values. For the barium sulfate the agreement was better but the trend of high filter-permeability results was apparent.

COMMERCIAL PLANT TESTS

To demonstrate the correlation of filtration theory with industrial practice a series of five tests was made at different drum speeds of a continuous rotary-drum (FEINC) commercial-plant filter. The filter, operating on dewatering of a pearl cornstarch prefilt stream, was 8 ft. in diameter by 7 ft., 4 in. in face width. Samples of the cornstarch prefilt were also tested to obtain compression-permeability, filter-permeability, and laboratory constant-pressure filtration results.

In the plant filtration tests the filter vacuum, which constitutes the filtration pressure, ranged from 6.38 to 6.88 lb./ sq. in., the average value being 6.68. Specific filtration-resistance values calculated from the test data, with septum resistance neglected, ranged from 6.18 X 10^{10} to 10.05×10^{10} ft./lb.-mass. The average value corresponding to 6.68 lb./sq. in. of filtration pressure was 9.05×10^{10} ft./lb.-mass. A reasonable allowance for septum resistance of these tests was estimated as 20 per cent, or 1.67 × 1010 ft./lb.-mass. With this allowance, the specific filtration resistance of the 6.68 lb./sq. in. plant pearl cornstarch filtration was 7.38 × 1010 ft./lb.-mass.

From a differential analyses of the pearl cornstarch compression-permeability test data the relationship $\alpha_p = (6.83 \times 10^{10}) P_m^{0.0959}$ ft./lb.-mass was determined for pressures below 30 lb./sq. in. This relationship was integrated to obtain $\alpha_{q_{2}q} = (6.18 \times 10^{10}) P_m^{0.0959}$

ft./lb.-mass and $\alpha_{mean} = (6.23 \times 10^{10}) P_m^{0.0969}$ ft./lb.-mass. For predicting specific filtration resistance the two expressions are not significantly different for this material. They were used to calculate predicted specific filtration resistance for the plant operating pressure of 6.68 lb./sq. in. The calculated values were $\alpha_{avg} = 7.38 \times 10^{10}$ and $\alpha_{mean} = 7.52 \times 10^{10}$ ft./lb.-mass.

From the constant-pressure laboratory filtrations on the pearl-cornstarch-prefilt specific filtration resistances of 9.42×10^{10} and 7.89×10^{10} ft./lb.-mass were obtained for filtration pressures of 8.7 and 15.5 lb./sq. in. Laboratory filter-permeability test results for this material were 7.52×10^{10} , 7.76×10^{10} , and 7.93×10^{10} ft./lb.-mass, corresponding to pressures of 6.25, 12.0, and 20.0 lb./sq. in.

CONCLUSIONS

From the laboratory investigations of three chemical slurries it was found that specific resistances determined by compression-permeability tests could be used to predict the average specific resistance in filtration. Predictions made by using the expression

$$\alpha_{mean} = \frac{\int_0^P \alpha_p \, dP_m}{P - 0}$$

were in some cases better than predictions made by using the expression

$$\alpha_{avg} = \frac{P - 0}{\int_{0}^{P} \frac{dP_{m}}{\alpha_{m}}}$$

In other cases the predicted values of α by both methods were in good agreement. In all cases, however, the $\alpha_{\alpha r}$ values were lower than the laboratory-filtration specific resistances. Specific resistances

determined by filter-permeability tests were usually higher than the laboratory-filtration values.

From the results of the comparison of commercial-plant tests and laboratory tests made on pearl-cornstarch prefilt it was found that the plant filtrations, laboratory filtrations, and laboratory filter permeabilities were in agreement. It was also apparent that the specificfiltration resistance predicted from the compression-permeability test agreed very well with the actual specific filtration resistance of the prefilt. This agreement shows clearly that filtration resistance can be accurately predicted and that filtration theory can be satisfactorily correlated with industrial practice.

ACKNOWLEDGMENT

This paper is based on a Ph.D. thesis conceived and originally directed by B. F. Ruth of the Iowa State College Chemical and Mining Engineering Department, who contributed generously of his time, effort, and understanding of the filtration process until his death on January 1, 1954. The authors are grateful for having had the opportunity of working and associating with Dr. Ruth.

Appreciation is extended to the many chemical engineering students who took part in the experimental program; to Clinton Foods Incorporated, Clinton, Iowa, for its cooperation in the plant testing program; and to E. I. duPont deNemours and Company, Inc., Wilmington, Delaware, and Ethyl Corporation, Baton Rouge, Louisiana, for industrial fellowship support of the program.

NOTATION

 A = total filter area perpendicular to flow, sq. ft.

α = specific filtration resistance ft./lb.-mass

α_p = specific filtration resistance of a differential-thickness solids layer or a finite-thickness cake of particles in uniform mechanical compression, ft./lb.-mass

 α_{avp} = specific filtration resistance predicted from compression-permeability test data by the equation

$$(P-P_0) / \int_0^{P-P_0} \frac{dP_m}{\alpha_p}$$

 $\alpha_{mean} = egin{array}{l} (P_0 \ {
m is generally zero}), {
m ft./lb.-mass} \\ {
m specific filtration resistance predicted from compression-permeability test data by the equation} \end{array}$

$$\left\{\int_0^{P-P_0} \alpha_p \, dP_m\right\} / P \, - \, P_0$$

 $(P_0 \text{ is generally zero}), \text{ ft./lb.-mass}$ = numerical constant

= Differential analysis of compression-permeability tests n_1 and n_2 [Compare Equation (9)].

= Newton's gravitational constant 32.1740, (lb. mass)(ft.)(lb. force) (sec.2)

= numerical constant \boldsymbol{k}

= parabolic parameter, sec. or K min./sq. ft.

= ratio of mass of wet cake to dry

= filtrate viscosity, lb.-mass/(ft.) (sec.)

= filtration pressure, lb.-force/sq.

= hydrostatic pressure at any point P_x lb.-force/sq. in.

= mechanical pressure on cake particles or solids, lb.-force/sq. in.

= filtrate density lb.-mass/cu. ft.

resistance of cake solids to fluid R_c flow, 1/ft.

= resistance of filtration deposition R_d zone to fluid flow, 1/ft.

= resistance of equipment leads, \boldsymbol{R} fittings, etc. to fluid flow, 1/ft.

resistance of filter cloth or septum to fluid flow, 1/ft.

prefilt consistency, weight of oven-dry solids to prefilt weight

= filtration time, hr.

= time equivalent of V_m , hr.

= filtration rate, hr./cu. ft.

= filtration rate calculated as $(\theta_{n+1} - \theta_n)/\frac{1}{2}(V_{n+1} + V_n)$ when successive values of $V_{n+1} - V_n$ are equal, hr./cu. ft.

= fluid or filtrate volume, cu. ft.

= filtrate volume equivalent to filter cloth resistance, cu. ft.

dV= fluid or filtrate rate, cu. ft./hr.

= oven-dry weight of cake soilds deposited per cubic foot of filtrate discharged, lb.

= oven-dry weight of cake solids, lb.

= oven-dry weight of cake solids equivalent of R_m , lb.

 $= W_o + W_m$, lb.

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Individual Film Coefficients of Mass Transfer in Liquid-liquid Extraction

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A study has been made of the individual film coefficients of mass transfer for two binary liquid-liquid systems of differing physical properties, namely methyl isobutyl carbinolwater and methylethyl ketone-water, in a 4-in. diam. extraction column operated as a spray column and with 1/2-in. Raschig ring packing. The value of H_t for the dispersed phase was found to be a constant, C_1 , for a given system in a given column. The H_i values for the continuous phase could be correlated by the equation,

$$(H_t)_c = C_2(V_c/V_d)^n$$

Values of the constants C_1 , C_2 , and n are tabulated along with the values found by earlier investigators for other systems and column packings. The $oldsymbol{H_t}$ values have been reduced to area base coefficients by the expression for droplet surface area proposed by Gaylor

Presaturation of either phase was found to have no effect on mass transfer rates. There appears to be relatively little difference in the efficiency of spray and packed columns for systems of low interfacial tension, but for high interfacial-tension systems packed columns are considerably more efficient than spray columns.

While no definitive correlations for the effect of physical properties are proposed, there are some indications that n is a function of the viscosity ratio of the two liquid phases and that C_2 is a function of the 1/4 power of the groups $(d\Delta\rho\gamma/\mu^2_c)(\mu_c/\mu_d)$ and $(N_{Sc})_c$. No correlation was found for the effect of physical properties on $(H_t)_d$.

In recent years the unit operation of liquid-liquid extraction has assumed an increasing importance as the separation problems of modern process chemistry have become more complex. While many new forms of extraction equipment have been proposed recently, particularly those involving mechanical agitation to improve the two-phase contact, the

packed column remains one of the simplest and most economical extractors to build and operate. Such columns are easily and cheaply assembled from stock parts and are generally less expensive to operate than rotating disk contactors, pulsed columns, centrifugal extractors,

This paper is based on a thesis submitted in partial fulfillment of the requirements for the degree of Doctor of Philosophy at Carnegie Institute of Technology.

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