

or pore; ratio of scattered to geometrically obstructed radiation

k = effective total conductivity

k_c = effective conductivity for gaseous conduction, solid conduction, and natural convection only

k_r = equivalent conductivity for radiation

L = thickness of insulation

M = interception cross section per unit volume of insulation

m = index of refraction

N = back scattering cross section per unit volume of insulation

n = number of scatters per unit volume

P = absorption cross section per unit volume of insulation

q = total heat-flux density (a constant across insulation)

$q_r(x)$ = net radiant heat-flux density

R = pore radius

S_a = absorption (and emission) cross section per scatter

S_s = scattering cross section per scatter

$T(x)$ = absolute temperature

x = distance through insulation

y = mean thickness of solid associated with a pore

Greek Letters

β = exponential absorption coefficient of solid material

ϵ = emissivity of boundary surface of insulation

λ = wave length

ρ = bulk density

ρ_g = gas density

ρ_s = solid density

σ = Stefan Boltzmann constant

Subscripts

0 — at $x = 0$ boundary

L — at $x = L$ boundary

λ — monochromatic

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The Economic Design of Mixer-Settler Extractors

ROBERT E. TREYBAL

New York University, New York, New York

Relationships are developed for establishing the most economic values of the major variables of a liquid-extraction process, including the concentrations of solute in recycled solvent and rejected raffinate, the solvent-to-feed ratio, and for mixer-settlers certain of the design features. Consideration is given to costs of extraction and solvent recovery, as well as to the value of unextracted solute and lost solvent. For the design of mixer settlers scale-up relationships are developed to permit prediction of the stage efficiency of a large extractor from experimental data taken on a small scale. These are expressed in terms of a scale-up index relating the relative size of the mixer with the volumetric rates of liquid flow. It is shown that the cost of a multistage extractor increases with scale up in a different fashion from the stage efficiency, depending upon the scale-up index used. A detailed study of the costs for a typical case led to the development of economic scale-up indexes which, because they cover nearly a fivefold ratio of fixed to operating costs, are of fairly general utility. The common practice of scaling up with equal holding times on the large and small scale is shown to be amply safe from the point of view of stage efficiency to be realized on the large scale but usually uneconomic.

Simplification of the complete system of equations permits rapid estimation of the most economic circumstances for any type of countercurrent extractor of which the cost per stage is proportional to Q^a .

Despite the facts that mixer settlers are probably the oldest form of commercial liquid extractors and that new countercurrent tower devices are invented every year, mixer settlers have never

been wholly superseded. In recent years there has even been an increased tendency to use them. The many reasons for this need not be dwelt upon here. It is sufficient to note that it is generally believed

that small-scale experiments provide a reliable guide to the design of large-scale equipment.

Although there have been a number of new mixer-settler designs in the past few

years, the simplest arrangement consists of a combination of an agitated vessel (the mixer) through which the liquids to be contacted are passed and a settler in which the dispersion of liquids is settled by gravity. The two vessels constitute one stage, not necessarily ideal, and any number of stages may be joined to provide multistage, countercurrent effects. It is common procedure to determine the stage efficiency in a small-scale mixer and to scale up to a larger size by providing in the large scale equal agitator power per unit volume of liquids and a geometrically similar mixing vessel sized so as to provide equal holding time for both large and small scale. When the stage efficiency of the small vessel is essentially 1.0 (or 100%), this is said to give good results [Warner (12) for example]. It will be shown that these practices are almost certain to give good results but may be uneconomical.

What is really required is not merely a method of scale-up to produce a predictable performance but also a set of conditions for the large scale which results in the least cost of extraction. It is desirable not to be bound by the circumstances under which the small-scale experiment was run, since these may not necessarily be those which lead to the optimum large-scale plant. The scale-up procedure should therefore be adjustable so as to produce the optimum plant.

It is useful to consider the factors which enter into the cost of an extraction operation, the quantities which the designer may vary, and where the latter influence the costs. The total annual cost of extracting a solute from a feed solution may be considered to be the sum of six cost quantities, as outlined in Table 1. If a flow rate of feed solution, the concentration of contained solute, and the identity of the solvent to be used are stipulated, the quantities which may be set to produce an optimum arrangement are those listed in the lower part of Table 1, together with the cost items which each influences. A few of these have been studied in the recent literature (6, 9) without however full consideration of some important interrelationships which exist among them. It is the purpose here to present a more detailed and less restrictive study.

To minimize the total cost one must partially differentiate C_T with respect to each of the variable quantities while holding the others constant, set the resulting expressions equal to zero, and solve them simultaneously. The major assumptions made in the course of this development are listed in Table 2.

DESIGN OF EXTRACTOR STAGES

Item I, Table 1, is the only cost quantity which need be considered. The annual cost of a multistage, countercurrent extractor of n ideal stages and

E_0 over-all stage efficiency may be described as

$$I = nC_E/E_0 \quad (1)$$

where

$$n = \frac{\log \left[\frac{(c_F - c_S/m)}{(c_R - c_S/m)} (1 - 1/mR) + 1/mR \right]}{\log mR} \quad (2)$$

$$E_0 = \frac{\log [1 + E_M(mR - 1)]}{\log mR} \quad (3)$$

$$C_E = C_V \left(\frac{p}{Y} + b \right) + (P_A + P_P)WH \quad (4)$$

The number of ideal stages is quite independent of the stage design, whereas C_E and E_0 are affected, the latter through the stage efficiency of each stage E_M . Consequently

$$\frac{\partial I}{\partial (\text{design})} = \frac{n \partial (C_E/E_0)}{\partial (\text{design})} \quad (5)$$

and for optimum design

$$\frac{\partial (C_E/E_0)}{\partial (\text{design})} = 0 \quad (6)$$

Stage Efficiency

The discussion here is limited to baffled mixing vessels in the form of vertically disposed circular cylinders of height equal to diameter, with centrally located flat-blade turbines as the agitating devices. For these it has been shown (11) that it is possible to estimate the stage efficiency. The relationships required lead to two dimensionless quantities, $X = mk_c d_p / 2D_D'$ and $Z = 2mk_c \theta / d_p$, which together with the chart of Figure 1 permit estimation of the efficiency. Figure 1 is reproduced here in simplified form for purposes of demonstration later, and the earlier paper should be consulted for a working chart. Some of the relationships of that development are not well established, particularly several coefficients and the effective diffusivity. For this reason small-scale experiments

are carried out. When the relationships are used only in ratio for scale up in the same chemical system and under conditions to be described, the uncertain quantities cancel out.

It is undoubtedly sound practice to scale up by using geometrically similar vessels (4). The use of equal power per unit volume of liquids, theoretically (4) and also according to the limited data available (11), will provide for equal degrees of dispersion of one of the liquids in the other. There remains the question of holding time. As indicated earlier equal holding time on small and large scale is common practice. Provision for equal mass velocities of the liquids might also be considered a logical scale up basis and this will mean an increase in holding time on the large scale. Equal holding times means a reduced mass velocity. The use of unequal holding times and mass velocities is certainly a possibility.

To investigate the effect of scale up of rate of flow on stage efficiency when all extraction conditions except total flow rate remain constant, a scale-up index is defined by Equation (7):

$$\frac{T_1}{T_2} = \left(\frac{Q_1}{Q_2} \right)^x \quad (7)$$

Geometrically similar vessels and agitator turbines are specified so that

$$\frac{L_1}{L_2} = \frac{T_1}{T_2} \quad (8)$$

In order that the fraction of the vessel contents devoted to dispersed phase be constant equal power per unit volume of flowing liquids will be applied (11):

$$\frac{P_1}{Q_1} = \frac{P_2}{Q_2} \quad (9)$$

If the agitator Reynolds number is sufficiently large, the power number $Pg_c/\rho N^3 L^5$ remains constant. Under these circumstances it is possible to show* that the ratios of X and Z for scale up are

$$\frac{X_1}{X_2} = \left(\frac{Q_1}{Q_2} \right)^{0.322x + 0.066} e^{0.70(T_2 - T_1)} \quad (10)$$

$$\frac{Z_1}{Z_2} = \left(\frac{Q_1}{Q_2} \right)^{1.151x - 0.592} e^{0.70(T_1 - T_2)} \quad (11)$$

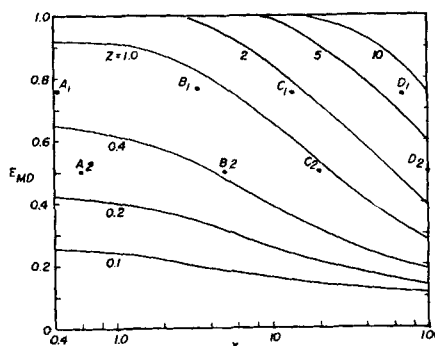


Fig. 1. Murphree dispersed-phase stage efficiency for mixer-settler extractors (11).

*Tabular material has been deposited as document 5968 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.

TABLE 1. EXTRACTION COSTS

$C_T = I + II + III + IV + V + VI$
C_T = annual total cost of extraction process
I = annual cost of multistage extractor
II = annual value of unextracted solute
III = annual cost of solvent recovery from the extract
IV = annual cost of solvent recovery from the raffinate
V = annual cost of lost solvent
VI = annual labor cost

Quantities to be optimized

Quantity	Cost term affected
1. Design of extractor stages	I
2. Concentration of solute in raffinate	I, II, III
3. Concentration of solute in recycled solvent	I, III
4. Solvent-to-feed ratio	I, III, V
5. Concentration of solvent left in product	III, V
6. Concentration of solvent in stripped raffinate	IV, V
7. Reflux ratio for solvent recovery from extract by distillation, or similar item if another operation is used	III
8. Reflux ratio for solvent recovery from raffinate by distillation	IV

TABLE 2. MAJOR ASSUMPTIONS

1. Constant annual labor cost.
2. Insolubility of solvent and feed.
3. Constant equilibrium distribution coefficient.
4. One solute extracted in a countercurrent multistage extractor.
5. On scale-up of mixer settlers,
 - a. Geometrically similar, baffled mixers, height = diameter.
 - b. Agitation with flat-blade turbine impellers.
 - c. Equal agitator power to liquids per unit volume flow rate of liquids.
6. Constant Murphree stage efficiency of all stages in any one multistage cascade.
7. Constant relative volatility of solvent and extracted solute.
8. Loss of solvent in distilled product and stripped raffinate negligible.
9. Solvent loss proportional to number of extraction stages and solvent circulation rate.

For equal mass velocities in small and large scale $x = 0.5$; for equal holding times $x = 1/3$. Figures 2, 3, and 4 were prepared from these equations for these values of x and also for $x = 0.25$.

To demonstrate the method of developing the scale-up charts the case for $x = 0.5$, $T_2 = 1$, $E_{MD2} = 0.5$, and $Q_1/Q_2 = 4$ is considered. It follows that $T_1 = 2$ [Equation (7)], $X_1/X_2 = 0.68$ [Equation (10)], and $Z_1/Z_2 = 1.97$ [Equation (11)]. When one refers to

Figure 1, it is clear that there are any number of combinations of X_2 and Z_2 corresponding to $E_{MD2} = 0.5$, but fortunately this does not have important influence on the final result. Thus when one starts with points A_2 , B_2 , C_2 , and D_2 at $E_{MD2} = 0.5$, Figure 1, the scale-up relations provide the points A_1 , B_1 , C_1 , and D_1 , where the corresponding values of E_{MD1} are 0.76, 0.77, 0.75, and 0.74 respectively, averaging 0.754. The same averaging procedure was used throughout

in establishing the scale-up charts, and nowhere were the variations in E_{MD1} any greater. On Figure 2 therefore point A corresponds to $E_{MD2} = 0.5$, $T_2 = 1$, and scale-up to $Q_1/Q_2 = 4$ provides B at $T_1 = 2$, relative volume rate = 4, $E_{MD1} = 0.754$.

The curves of Figures 2, 3, and 4 then become scale-up paths for the corresponding values of x . Thus it may be supposed that by experiment it is known that in passing 150 cu. ft./hr. of extracting liquids through a mixer 1.4 ft. in diam. a stage efficiency $E_{MD2} = 0.25$ is obtained. It is desired to estimate the stage efficiency for 1,500 cu. ft./hr. of liquids, scaled up according to the conditions established earlier with $x = 0.5$. The known conditions correspond to point C on Figure 2, where the relative volume rate of flow is 1.95. For a tenfold increase in flow rate the relative volume rate of flow is 19.5, the new conditions correspond to point D, and the new stage efficiency is $E_{MD1} = 0.94$. The corresponding tank diameter is 4.4 ft.

Figure 3 shows that scale up according to equal holding time from tanks 1-ft. diameter or larger always results in an appreciable improvement in stage efficiency. Constancy of stage efficiency on scale up is most nearly realized for $x = 0.25$ over the middle range of Figure 4. Truly constant stage efficiency would evidently require adjustment of the agitator power.

With the volume flow rate, the distribution coefficient, and the individual stage efficiency assumed the same for all stages, the over-all efficiency of the large extractor is given by Equation (3), with $E_M = E_{MD1}$. E_{o1} thus depends, through E_{MD} and Figures 2 through 4, upon Q_1/Q_2 and x .

Extractor Costs

The vessel sizes and hence the costs per real stage at a given scale-up ratio Q_1/Q_2 are less at low values of x , but

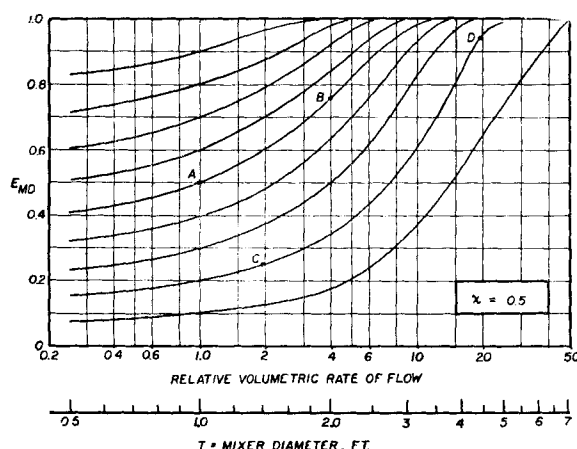


Fig. 2. Scale up at constant solvent/feed ratio, $x = 0.5$. Basis: geometrically similar vessels and equal power input per unit volumetric rate of flow.

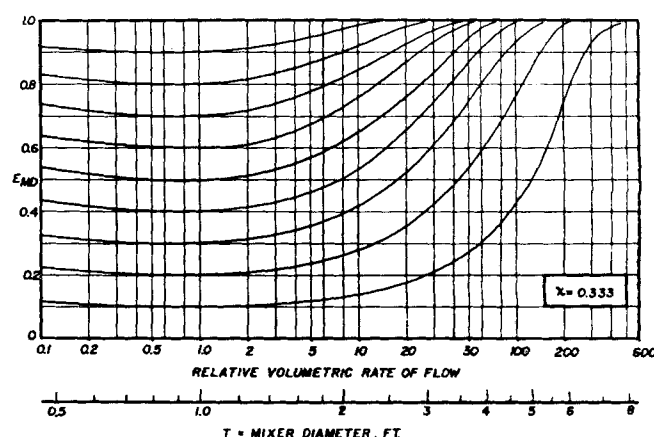


Fig. 3. Scale up at constant solvent/feed ratio, $x = 0.333$. Basis: geometrically similar vessels and equal power input per unit volumetric rate of flow.

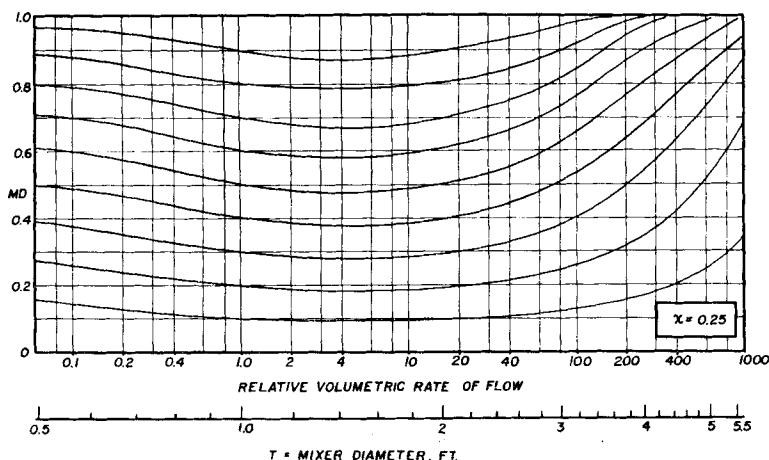


Fig. 4. Scale up at constant solvent/feed ratio, $x = 0.25$. Basis: geometrically similar vessels and equal power input per unit volumetric rate of flow.

since the stage efficiency is also less, more real stages are required. It follows that there should be a most economical x .

When one refers to Equation (4), the major equipment cost per stage is

$$C_V = C_M + C_A + C_S + C_P \quad (12)$$

Data on costs of equipment and their variation with size were taken for the most part from the recent books by Aries and Newton (1) and Peters (8). For the empty mixer

$$\begin{aligned} C_{M1} &= C_{M2}(V_1/V_2)^{0.55} \\ &= C_{M2}(Q_1/Q_2)^{1.65x} \end{aligned} \quad (13)$$

When one assumes that the settler increases in volume directly with that of the liquids to be handled

$$C_{S1} = C_{S2}(Q_1/Q_2)^{0.55} \quad (14)$$

For the pumps

$$\begin{aligned} C_{P1} &= C_{P2}(q_1/q_2)^{0.333} \\ &= C_{P2}(Q_1/Q_2)^{0.333} \end{aligned} \quad (15)$$

There may be either two pumps per stage if both liquids are pumped between

stages, one if one of the liquids flows by gravity, or none if both flow by gravity. The agitating equipment covered by C_A includes the impeller shaft, stuffing box, explosion-proof motor, geared speed reducer, and mounting and support. It was found that the costs of these unfortunately must be established for specific cases. For example the shaft diameter and length depend on the vessel size, power, and speed. No simple generalization of the sort used for the other costs was possible.

When one assumes that the efficiency of the transmittal of power to the agitated liquids remains constant,

$$P_{A1} = P_{A2}(Q_1/Q_2) \quad (16)$$

A few calculations for typical cases indicated that pump power for overcoming pipe friction was the only important consideration for this item. The power for pumping the dispersed liquid from stage to stage through piping is (?)

$$P_P = K(q_D \rho_D)^{2.84}/d^{4.84} \rho_D^2 \quad (17)$$

and the economical pipe diameter is (?)

$$d = K'(q_D \rho_D)^{0.448}/\rho_D^{0.312} \quad (18)$$

Combining Equations (17) and (18) and taking ratios at constant pump efficiency for the large and small scale, one gets

$$\begin{aligned} P_{P1} &= P_{P2}(q_{D1}/q_{D2})^{0.67} \\ &= P_{P2}(Q_1/Q_2)^{0.67} \end{aligned} \quad (19)$$

A similar relation applies to a pump for the continuous phase.

It was indicated above that the cost of agitating equipment must be estimated for particular cases. To come to some conclusions about the scale-up index, a set of conditions were chosen which are believed to be representative of good practice, and the costs were determined. From the study of a fivefold variation in the payout time, thus covering a wide variation in the ratio of fixed to operating costs, it is thought that the conclusions become reasonably general. In any case the relative importance of many of the variables can be demonstrated. Table 3 lists the details of the chosen situation.

In the case of the agitating equipment the costs for recommended equipment with the customary allowances for power overload and discount were determined for several values of Q and the corresponding speed, power, and mixer size at each value of x studied (5). The resulting costs are shown in Figure 5. Figure 6 shows the uninstalled cost of major equipment, less pumps per stage. For values of Q_1 from 500 to 5,000 cu. ft./hr. these may be closely represented by

$$\begin{aligned} C_{M1} + C_{S1} + C_{A1} &= 730(Q_1/100)^{0.520x+0.365} \end{aligned} \quad (20)$$

When one includes the single pump per stage, the costs are given closely by

$$C_{V1} = 1000(Q_1/100)^{0.468x+0.332} \quad (21)$$

Annual costs were then computed for $Q_1/Q_2 = 5$ to 100; $Y = 1, 2$, and 5; extraction factor $mR = 0.05$ to 20; E_{MD2} in the small scale = 0.3 to 1.0;

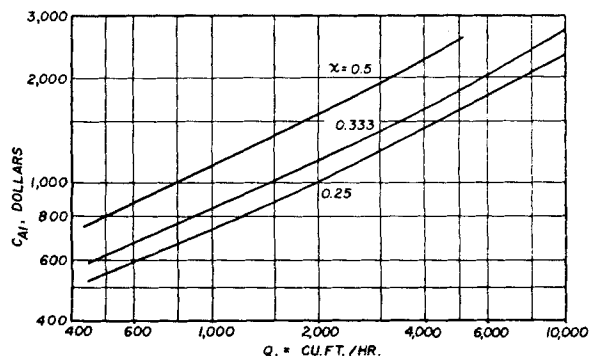


Fig. 5. Uninstalled cost of agitating equipment for one extraction stage. Basis: 12.5 gal./min. through a 1.5-ft.-diameter vessel, agitation intensity 1,000 (ft. lb./hr.)/(cu. ft./hr.).

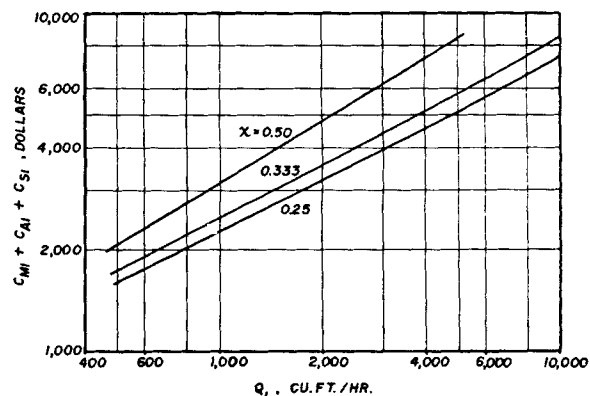


Fig. 6. Uninstalled cost of major equipment less pumps for one extraction stage. Basis: 12.5 gal./min. through a 1.5-ft.-diameter vessel, agitation intensity 1,000 (ft. lb./hr.)/(cu. ft./hr.).

TABLE 3. CONDITIONS FOR STUDYING THE ECONOMIC SCALE-UP INDEX

Small-scale conditions

Total liquid flow, solvent + feed = 12.5 gal./min.; $Q_2 = 100$.

Mixing Vessel: diameter $T_2 = 1.5$ ft., $C_{M2} = \$123$.

Settling vessel: 10 min. holding time, 16.7 cu. ft.; $C_{S2} = \$338$.

Agitator: power to liquids = 0.05 horsepower = 1,000 (ft.-lb./hr.)/(cu. ft./hr.) $T/L = 3$, Efficiency of gear reducer and stuffing box = 0.90; of motor 0.85.

$$P_{A2} = 0.05/(0.9)(0.85) = 0.066 \text{ hp.}$$

Pump: one per stage, pumping half the liquid (50 cu. ft./hr.).

$$C_{P2} = \$235; \text{ pump efficiency} = 0.50; \text{ motor efficiency} = 0.85.$$

Pipe: 1 in.; equivalent length including valves and fittings = 80 ft./stage; 2 enlargements 2 contractions/stage; change in elevation = 1 ft./stage. Pump power output = 0.051 hp., $P_{P2} = 0.051/0.50(0.85) = 0.12 \text{ hp.}$

Large-scale conditions

Cost of agitator C_{A1} : see Figure 5.

Cost of power = \$0.015/kw.-hr. $W = \$0.0112/(\text{hp.})(\text{hr.})$

Annual time of operation $H = 7,200 \text{ hr./yr.}$

Uninstalled cost of instruments = $0.15C_V$

Installation cost for vessels, agitators, pumps, instruments = $0.43(C_V + 0.15C_V)$

Installed cost of piping = $0.86C_V$

$$p = 1 + 0.15 + 0.43(1 + 0.15) + 0.86 = 2.50$$

Maintenance: $b = 0.06$

and $x = 0.25$ to 0.50 . Values of x outside this range were not considered. At values of x below 0.25 the intensity of agitator-power input into relatively small vessels makes agitator selection difficult, and since computed droplet size decreases, there is increased likelihood of forming emulsions difficult to settle. Values of x above 0.5 are of use only for cases of unreasonably poor small-scale efficiency.

Economic Scale-Up Index

The most economical x , providing for minimum cost, is given by Equation (6). Since the charts of Figures 2 through 4, which cannot be conveniently expressed algebraically, are involved in the evaluation of E_{01} , a graphical procedure was used. Thus one may plot C_{E1}/E_{01} against x and observe the minimum at the economic x . Alternatively

$$\frac{\partial(C_{E1}/E_{01})}{\partial x} = (p/Y + b) \cdot \frac{\partial(C_{V1}/E_{01})}{\partial x} + (P_{A1} + P_{P1}) \quad (22)$$

$$\frac{WH}{\partial x} \frac{\partial(1/E_{01})}{\partial x} = 0$$

from which

$$\frac{\partial(C_{V1}/E_{01})/\partial x}{\partial(1/E_{01})/\partial x} = \frac{\partial(C_{V1}/E_{01})}{\partial(1/E_{01})} \quad (23)$$

$$= \frac{-(P_{A1} + P_{P1})WH}{(p/Y) + b}$$

One may therefore plot C_{V1}/E_{01} and $1/E_{01}$ against x and determine the value of x when the ratio of the slopes of the two curves equals the right-hand side of Equation (23); one may determine the value of x where the slope of a curve of C_{V1}/E_{01} vs. $1/E_{01}$ has this value

The results are summarized in Figure 7, and the economical x is independent of Y in the range studied. As the stage efficiency in the small scale becomes

$$n_{dmin} = \frac{\log \left(\frac{\text{moles solvent/hr.}}{\text{moles solute/hr.}} \right)_{dist}}{\log \alpha} + \frac{\log \left(\frac{\text{moles solute/hr.}}{\text{moles solvent/hr.}} \right)_{botts}}{\log \alpha} - 1 \quad (27)$$

larger, the economical x becomes small. For $E_{MD2} = 1$ it will be 0.25 for all situations studied, since then $E_{01} = 1$, the costs per real stage and ideal stage are the same, and C_V is least for $x = 0.25$.

While these conclusions are necessarily dependent upon the circumstances chosen for study, it is believed that they may be generally useful as a guide to economical scale up for fairly wide variations in the quantity of liquid passed through the 1.5-ft.-diameter mixer, provided that the agitator power to the liquids in the 1.5-ft. tank is fixed at 0.05 hp. and that the abscissa of Figure 7 is considered as Q_1/Q_2 rather than $Q_1/100$.

It is fully recognized furthermore that these results represent only a limited consideration of the design features, those which are presently amenable to attack

$$\text{Econ. } c_s = \frac{m\gamma C_d(p/Y + b)(O)(1 + r)}{E_d G_d F q_F H \ln \alpha} = \frac{m\gamma C_d(p/Y + b)B}{E_d G_d F q_F H \ln \alpha} \quad (29)$$

through the scaling up of small-scale experiments.

CONCENTRATION OF SOLUTE IN RAFFINATE AND RECOVERED SOLVENT

For the establishment of the most economic values of the concentration of solute in the raffinate from the extractor

$$J = 1 + \frac{4C_E}{(c_F - c_s/m)F q_F H E_0 (mR - 1) \ln mR} \quad (31)$$

and in the recovered solvent the cost equations of Table 1 could be differentiated formally. For this purpose the cost of unextracted solute may be

described as

$$II = F q_F c_R H \quad (24)$$

The cost of distillation of the solvent was expressed in the manner of Happel (3) adapted to the present problem:

$$III = \frac{C_d n_d (p/Y + b)(O)(1 + r)}{E_d G_d} + \frac{C_h (p/Y + b)(O)(1 + r)}{G_h} + C_{hc}(O)(1 + r)H \quad (25)$$

where

$$n_d = \gamma n_{dmin} \quad (26)$$

The minimum number of ideal distillation trays can be obtained from the well-known Fenske equation. Where the solvent is the more volatile component of the extract

$$\log \left(\frac{\text{moles solvent/hr.}}{\text{moles solute/hr.}} \right)_{dist} + \log \left(\frac{\text{moles solute/hr.}}{\text{moles solvent/hr.}} \right)_{botts} - 1 \quad (27)$$

where *dist* and *botts* refer respectively to distillate and still bottoms. If the solute is the more volatile, these subscripts are interchanged. For the case shown above the amount of solute in the botts, which is the product of the entire extraction process, is essentially that in the extractor feed and is independent of c_s . Product-purity specifications ordinarily require negligible solvent to be found in the botts, and the numerator of the first term on the right is essentially the entire flow rate of solvent. Therefore

$$\left(\frac{\text{moles solvent/hr.}}{\text{moles solute/hr.}} \right)_{dist} = \frac{q_F R \rho_s / M_s}{q_F R c_s / M_E} = \frac{\rho_s M_E}{M_s c_s} \quad (28)$$

The ultimate results, for either type of volatility, are*

where $B = O(1 + r)$, to be elaborated upon later.

$$\text{Econ. } (c_R - c_s/m) = (c_F - c_s/m) \cdot \frac{(1 - mR)}{2} (1 \pm \sqrt{J}) \quad (30)$$

where

$$J = 1 + \frac{4C_E}{(c_F - c_s/m)F q_F H E_0 (mR - 1) \ln mR} \quad (31)$$

In Equations (30) and (46) the negative square root is used if mR exceeds unity, the positive if mR is less than unity. In

*See footnote on page 475.

Equation (31) it is usually satisfactory to neglect c_s/m in comparison with c_F .

SOLVENT-TO-FEED RATIO

The solvent-to-feed ratio R affects cost items I, III, and V. Therefore

$$\frac{\partial C_T}{\partial R} = \frac{\partial I}{\partial R} + \frac{\partial III}{\partial R} + \frac{\partial V}{\partial R} = 0 \quad (32)$$

Effect on Extractor

The flow rate of feed to be extracted, that to be used on the large scale, remains constant, and it follows that the total volume of liquid through the extractor varies with the flow rate of solvent. The stage efficiency and consequently the extractor cost is therefore dependent upon the scale-up index used to establish the varying size of the stages while allowing R to vary. Since R also enters into Equation (2), then

$$\frac{\partial I}{\partial R} = \frac{\partial(nC_E/E_0)}{\partial R} \quad (33)$$

In studying the effect of R on C_E and E_0 it will be assumed that the cost of a large-scale extractor and its stage efficiency is known at some solvent/feed ratio R_1 , and total liquid flow rate $Q_1 = q_F(R_1 + 1)$. These may be obtained, if necessary, from scale up of laboratory data in the manner described earlier. The variation of these with R on the large scale is then to be determined.

For geometrically similar mixers

$$\frac{T}{T_1} = \left(\frac{Q}{Q_1}\right)^x = \left(\frac{R+1}{R_1+1}\right)^x \quad (34)$$

If sufficient agitator power, approximately 1,000 (ft. lb./hr.)/(cu. ft./hr.) or more (11), is used, the holding time for both continuous and dispersed phases within the vessel are equal. When one assumes equal power/volumetric rate of flow for the mixing, as before, the relationships used earlier (11) provide the equations of Table 4. The over-all efficiency may then be computed through Equation (3). Table 4 also shows the variation of costs with R , except for the agitating device which cannot be generalized in this manner. However if the basis for Figure 6 and Equations (20) and (21) is suitable, these may be used to compute C_V .

The number of ideal stages is given by Equation (2), which, together with Equation (30), provides

$$\text{Econ. } n = \frac{\log [1 - 2/(1 \pm \sqrt{J})]}{\log mR} - 1 \quad (46)$$

It is noteworthy that the varying values of c_s and c_R which enter into Equation (46) alter the usual n vs. R relationship obtained when these concentrations are held constant very considerably.

TABLE 4. SCALE-UP FOR CONSTANT FEED RATE, VARYING SOLVENT/FEED RATIO, GEOMETRICALLY SIMILAR VESSELS, AND EQUAL POWER/VOLUME RATE OF FLOW

Feed (raffinate) continuous, solvent (extract) dispersed

$$q_F = q_C = \text{constant}, q_D \text{ varies}, R = q_D/q_C$$

$$\frac{X}{X_1} = \left(\frac{R}{R_1}\right)^{0.43} \left(\frac{R+1}{R_1+1}\right)^{0.322x-0.299} \left(\frac{\rho_D R + \rho_C}{\rho_D R_1 + \rho_C}\right)^{0.645} \cdot \left[\frac{R_1 \left(1 + \frac{1.5\mu_D}{\mu_C + \mu_D}\right) + 1}{R \left(1 + \frac{1.5\mu_D}{\mu_C + \mu_D}\right) + 1} \right]^{0.71} e^{0.70(T_1 - T)} \quad (35)$$

$$\frac{Z}{Z_1} = \left(\frac{R_1}{R}\right)^{0.43} \left(\frac{R+1}{R_1+1}\right)^{1.151x+0.246} \left(\frac{\rho_D R + \rho_C}{\rho_D R_1 + \rho_C}\right)^{0.302} \cdot \left[\frac{R_1 \left(1 + \frac{1.5\mu_D}{\mu_C + \mu_D}\right) + 1}{R \left(1 + \frac{1.5\mu_D}{\mu_C + \mu_D}\right) + 1} \right]^{0.71} e^{0.70(T - T_1)} \quad (36)$$

Feed (raffinate) dispersed, solvent (extract) continuous

$$q_F = q_D = \text{constant}, q_C \text{ varies}, R = q_C/q_D$$

$$\frac{X}{X_1} = \left(\frac{R+1}{R_1+1}\right)^{0.322x-1.009} \left(\frac{\rho_D + \rho_C R}{\rho_D + \rho_C R_1}\right)^{0.645} \cdot \left[\frac{\frac{1}{R} \left(1 + \frac{1.5\mu_D}{\mu_C + \mu_D}\right) + 1}{\frac{1}{R_1} \left(1 + \frac{1.5\mu_D}{\mu_C + \mu_D}\right) + 1} \right]^{0.71} e^{0.70(T_1 - T)} \quad (37)$$

$$\frac{Z}{Z_1} = \left(\frac{R+1}{R_1+1}\right)^{1.151x-0.464} \left(\frac{\rho_D + \rho_C R}{\rho_D + \rho_C R_1}\right)^{0.302} \cdot \left[\frac{\frac{1}{R} \left(1 + \frac{1.5\mu_D}{\mu_C + \mu_D}\right) + 1}{\frac{1}{R_1} \left(1 + \frac{1.5\mu_D}{\mu_C + \mu_D}\right) + 1} \right]^{0.71} e^{0.70(T - T_1)} \quad (38)$$

Applicable to both cases:

$$C_M = C_{M1} \left(\frac{R+1}{R_1+1}\right)^{1.65x} \quad (39)$$

$$C_S = C_{S1} \left(\frac{R+1}{R_1+1}\right)^{0.55x} \quad (40)$$

$$C_P = C_{P1} \quad \text{for raffinate} \quad (41)$$

$$C_P = C_{P1}(R/R_1)^{0.333} \quad \text{for extract} \quad (42)$$

$$P_A = P_{A1} \left(\frac{R+1}{R_1+1}\right) \quad (43)$$

$$P_P = P_{P1} \quad \text{for raffinate} \quad (44)$$

$$P_P = P_{P1}(R/R_1)^{0.67} \quad \text{for extract} \quad (45)$$

values of n , C_E , and E_0 appropriate to the stripping operation.

Solvent Recovery by Stripping

If the solvent is regenerated by extraction of the extract, the effect of R is given by Equation (33), with of course

Solvent Recovery by Distillation

Equation (25) may be written

$$III = A(O)(1+r) = AB \quad (47)$$

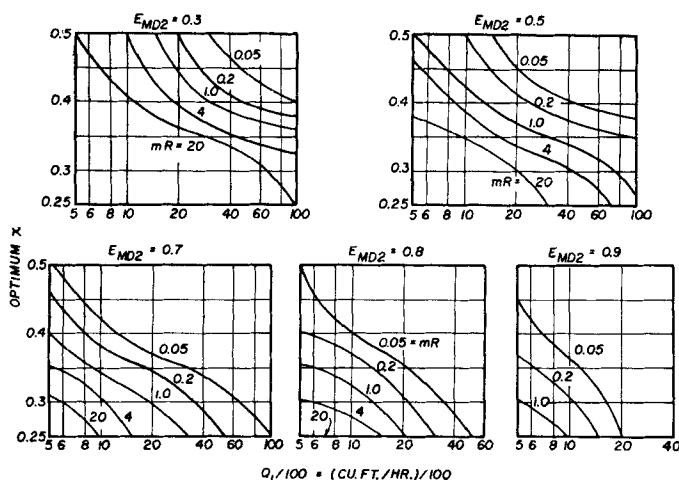


Fig. 7. Optimum x , constant solvent/feed ratio, for $Y = 1$ to 5 yr. For E_{MD} = extract efficiency, $mR = (\text{equilibrium } c_E/c_R) (\text{extract rate/raffinate rate})$. For E_{MD} = raffinate efficiency, $mR = (\text{equilibrium } c_R/c_E) (\text{raffinate rate/extract rate})$. Basis: 12.5 gal./min. through a 1.5-ft.-diameter vessel, agitation intensity 1,000 (ft. lb./hr.)/(cu. ft./hr.).

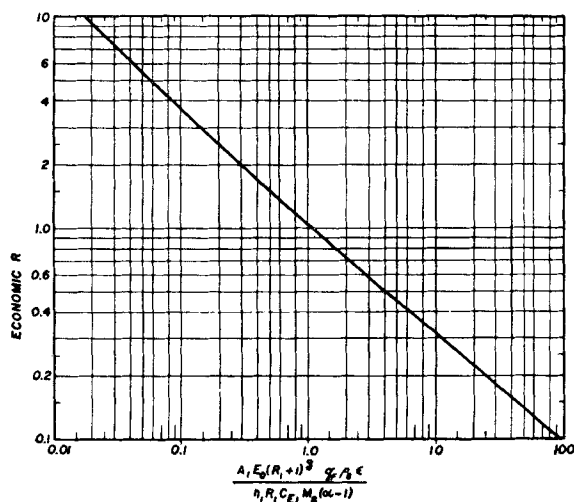


Fig. 8. Economic R for $g = 0.45$. Practically useful for $g = 0.3$ to 0.8 .

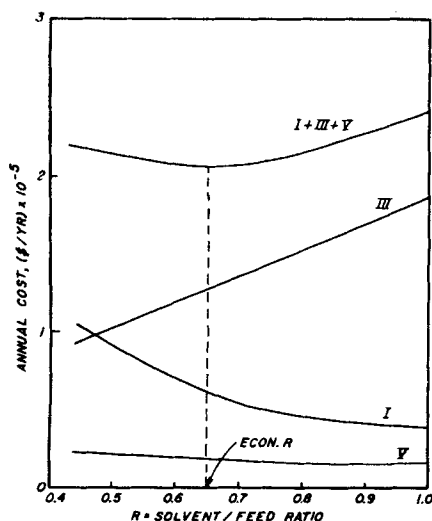


Fig. 9. Determination of optimum solvent/feed ratio.

where

$$A = \left(\frac{C_d \gamma n_{dmin}}{E_d G_d} + \frac{C_h}{G_h} \right) \left(\frac{p}{Y} + b \right) + C_{hc} H \quad (48)$$

It can be shown* that for cases where the solvent is more volatile than the extracted solute

$$B = O(1 + r) = \frac{R q_F \rho_S}{M_S} \cdot \left(1 + \frac{\beta}{\alpha - 1} \right) + \frac{\beta U}{\alpha - 1} \quad (49)$$

and if the extracted solute is the more volatile

$$B = O(1 + r)$$

$$= U \left(1 + \frac{\beta}{\alpha - 1} \right) + \frac{R q_F \rho_S}{M_S (\alpha - 1)} \quad (50)$$

where

$$U = q_F (c_F - c_R) / M_E \quad (51)$$

Cost of Lost Solvent

The losses contributing to item V may be considered as those occurring in the extractor stages, the solvent remaining in the stripped raffinate, and the solvent in the final product. The last two are considered negligible. The first of these is assumed to be proportional to the number of extractor stages and the solvent recirculation rate:

$$V = \frac{n q_F R L S H}{E_0} \quad (52)$$

Economic R

The operation required by Equation (32), if attempted formally, is made difficult by the variation of E_0 with changing R . For a complete computation therefore the best procedure is to plot the sum (I + III + V) against R and locate the minimum in the curve. In the computation of these cost items the economic x , c_R , c_S , and n should be used. A sample calculation demonstrates the method.

Short-cut Method

If some reasonable approximations are made, Equation (32) can be made to yield the economic R fairly quickly. Equation (46) is such that the product nR is nearly constant over modest ranges of R , unlike the situation when c_S and c_R are held constant. Further E_0 is also nearly constant over quite wide ranges of R . If operating costs for the extraction stages are neglected in comparison with annual equipment costs, then

$$C_E = C_V (p/Y + b) = C_{E1} C_V / C_{V1} \quad (53)$$

For most situations Equation (21) is a special case of the more general relation

$$C_V = K'' Q^g = K'' [q_F (R + 1)]^g \quad (54)$$

The first differentiation of Equation (32) then becomes

$$\frac{\partial I}{\partial R} = \frac{-n_1 R_1 C_{E1}}{E_0 (R_1 + 1)^g} \cdot \left[\frac{1 + (1 - g)R}{(R + 1)^{1-g} R^2} \right] \quad (55)$$

It was also noted that n_{dmin} remains remarkably constant with fairly large

*See footnote on page 475.

variations in R and so may be computed at any value R_1 . Substituting Equations (28) and (29) in Equation (27) one gets

$$n_{dmin1} = \frac{\log \left[\frac{2.3 \rho_s M_E E_d G_d F q_F H k \log \alpha}{M_s m \gamma C_d (p/Y + b) B_1} \right]}{\log \alpha} - 1 \quad (56)$$

where the subscript 1 indicates that B and n_{dmin} are evaluated at R_1 . This value of n_{dmin} yields A_1 in Equation (48), Equations (48), (49), (50), and (47) then yield

$$\frac{\partial III}{\partial R} = \frac{A_1 q_F \rho_s \epsilon}{M_s (\alpha - 1)} \quad (57)$$

where $\epsilon = \alpha + \beta - 1$ for solvent more volatile, and $\epsilon = 1$ for solute more volatile.

If nR is considered constant, then $\partial V/\partial R = 0$. Equation (32) then provides the economic R :

$$\frac{1 + (1 - g)R}{R^2(R + 1)^{1-g}} = \frac{A_1 E_0 (R_1 + 1)^g q_F \rho_s \epsilon}{n_1 R_1 C_{E1} M_s (\alpha - 1)} \quad (58)$$

Equation (58) should be reasonably useful for any countercurrent extractor, either tower or mixer settler, of which the cost per stage is proportional to Q^0 . If Equation (21) is acceptable and $x = 0.25$, as it frequently will be, $g = 0.45$ and Figure 8 provides the solution to Equation (58). Actually the nature of Equation (58) is such that, for all practical purposes, Figure 8 may be used for any value of g from 0.3 to 0.8. In any case the economic c_s and c_R are taken into consideration.

The procedure to be followed in using Equation (58) or Figure 8 is

1. Choose any reasonable value for R_1 .
2. Compute C_{v1} and $C_{E1} = C_{v1} (p/Y + b)$. C_{v1} is obtained through Equation (54) or in any convenient fashion, such as Equation (21).
3. Choose E_0 . In most cases it can be taken as 0.90 or 0.95.
4. Compute B_1 [Equations (49) or (50)].
5. Compute J_1 [Equation (31)]. In most cases c_s/m may be neglected but is obtained through Equation (29) if desired.
6. Compute n_1 [Equation (46)].
7. Compute n_{dmin1} [Equation (56)] and A_1 [Equation (48)].
8. Determine economic R by Equation (58) or Figure 8.
9. The economic values of n , c_R , and c_s may then be obtained by Equations (46), (30), and (29).

Some of the items of Table 1 have not been studied. The annual cost of solvent recovery from the raffinate, IV, is set in part by the solubility of the solvent in the raffinate, in turn dependent upon the

chemical system used, and the concentration of solvent allowed to remain in

the stripped raffinate. The complete economic design of this equipment may be attacked by the method of Sherwood and Pigford (10). Economic reflux ratios may be chosen through the correlations of Happel for this equipment (3).

The methods described here may also be extended to cases of countercurrent extraction with reflux to determine the best reflux ratio. It may be of interest in this connection to note that for systems of constant selectivity the relation among the number of extraction stages, the minimum number, the extraction reflux ratio, and the minimum reflux ratio has been found to follow quite well the familiar Gilliland chart prepared for distillation (2). The extractor computations may then be made reasonably quickly. They may also be extended to fractional extraction of two solutes with two solvents for determining the optimum solvent ratio and reflux ratios. In such processes the calculations predict different over-all stage efficiencies, if E_{MD} is not unity, for each of the solutes being extracted, although this apparently has not yet been reported from experiment.

SAMPLE COMPUTATION

A solute is to be extracted from a dilute aqueous solution in a countercurrent mixer-settler plant, the solute concentration in the feed being $c_F = 5$ lb./cu. ft. The aqueous volume will be $q_F = 1,000$ cu. ft./hr. The solvent, insoluble in the aqueous solution, provides a distribution coefficient $m = 2.0$ at the prevailing concentrations. In the mixers the solvent will be dispersed, the aqueous phase continuous. The various physical properties are as follows: mole wt. solute = $M_E = 60$; mole wt. solvent = 100; viscosity of solvent = $\mu_D = 0.6$ cp., of aqueous solutions $\mu_C = 1.0$ cp.; density of solvent = $\rho_D = \rho_S = 50$, of aqueous $\rho_C = 62.4$ lb./cu. ft. The solvent will be recovered by distillation, where the solvent is the more volatile, and $\alpha = 2.5$. The finished product is to be 99.9 mole % extracted solute. A laboratory test in a 1.5-ft.-diameter mixer, with equal volume rates of feed and solvent, 100 cu. ft./hr. total flow, and agitator power 1,000 (ft. lb./hr.)/(cu. ft./hr.), showed a stage efficiency $E_{MD} = 0.7$.

The costs of Figure 6 will be assumed valid, and one pump per stage, for the aqueous, will be used. Solvent will flow by gravity between stages. Other items are as follows: $p = 2.5$, $b = 0.06$, $Y = 2$ yr., $H = 7,200$ hr./yr., $W = \$0.0112$ /hp.-hr., $S = \$6$ /cu. ft. (or 12¢/lb.) for the solvent, $F = \$0.50$ /lb. for the solute, $l = 0.00005$ fractional solvent loss/stage. The following distillation values are suggested by Happel (3): $C_d = \$20$ /sq. ft., $G_d = 15.0$, $G_a =$

0.10, $C_h = \$1.5$ /sq. ft., $C_{hc} = \$0.0093$ /lb. mole, $E_d = 0.90$, $\beta = 1.5$, $\gamma = 2.5$.

Complete solution

The computation will be made for $x = 0.25$, to be confirmed later. With a volumetric-rate-of-flow scale up of 20 from laboratory to plant at $R = 1$ Figure 4 shows the large scale conditions $R_1 = 1$, $Q_1 = 2,000$, $q_F = q_C = 1,000$, $q_{D1} = 1,000$, $T_1 = 3.17$, $E_{MD1} = 0.86$, with $P_{A1} = 1.32$ hp. [Equation (16)]. Computations for $R = 0.7$ follow.

Cost item I

Equation (34): $T = 3.17 (1.7/2)^{0.25} = 3.04$ ft. Equation (35): $X/X_1 = 0.86$. Equation (36): $Z/Z_1 = 1.29$. Figure 1: $E_{MD} = E_M = 0.94$, the average of values varying from 0.93 to 0.95. Equation (3): $E_0 = 0.946$. $Q = q_F(R + 1) = 1,700$ cu. ft./hr. Figure 6: $C_M + C_S + C_A = \$2,960$. For 50 cu. ft./hr. of liquid the basic pump cost is (Table 3) \$235 and pump power 0.12 hp. Equation (15): for 1,000 cu. ft./hr. raffinate $C_P = \$640$. Equation (19): $P_P = 0.90$ hp. Equation (43): $P_A = 1.12$ hp. Equation (12): $C_V = \$3,600$. Equation (4): $C_E = \$4,873$ /stage (yr.).

When one neglects c_R with respect to c_F , Equation (51): $U = 83.3$ lb.-moles/hr. Equation (49): $B = 783$. Equation (29): $c_S = 0.00231$ lb./cu. ft. Equation (31): $J = 1.0856$. Equation (46): $n = 10.5$ ideal stages. Equation (1): $I = \$54,100$ /yr.

Cost item III

Equation (28): (moles solvent/hr.)/(moles solute/hr.) in distillate = $50(60)/100(0.00231) = 13,000$. (Moles solute/hr.)/(moles solvent/hr.) in botts. = $99.9/0.1 = 999$. Equation (27): $n_{dmin} = 17.9$ ideal trays. Equation (26): $n_d = 44.7$ ideal trays. Equation (48): $A = 173.3$. Equation (47): $III = \$135,800$ /yr.

Cost item V

Equation (52): $V = \$16,800$ /yr.

Therefore $I + III + V = \$206,700$ /yr. In similar fashion values of this sum were computed for other values of R and the results plotted in Figure 9. A minimum occurs at $R = 0.65$ cu. ft. solvent/cu. ft. feed. At this R , $E_{MD} = 0.95$, $T = 3.02$ ft. for 1,650 cu. ft./hr. total flow rate. When one refers to Figure 4 and scales down to the 1.5-ft. laboratory mixer, the latter would have provided a stage efficiency $E_{MD2} = 0.87$ at 100 cu. ft./hr., $R = 0.65$. Figure 7, at $mR = 2(0.65) = 1.3$, $Q_1/100 = 16.5$, confirms the use of $x = 0.25$.

The economical conditions are therefore $R = 0.65$, 12 ideal or 13 real extractor stages, $c_S = 0.00216$, $c_R = 0.00658$ lb./cu. ft., and a percentage recovery of extractable solute of 99.87%.

It is of interest to note that increasing the solvent loss to two and four times the amounts used above shifted the economic R to 0.67 and 0.70 respectively, indicating that R is relatively insensitive to this quantity.

Other computations made with different dollar values of the solute, which is a quantity of major importance, resulted in variations of the extractor cost which

were unexpected, at least to the author. Thus for a solute value of \$0.25/lb., half that used above, the economic R becomes 0.51 and the extractor cost is increased (14 stages), although of course the total cost is reduced, largely through changes in c_s and c_R and their effect on the cost of the solvent recovery system.

Approximate solution

Choose $R_1 = 1.0$, as in the laboratory test; hence $Q_1 = 2,000$. Choose $x = 0.25$. Equation (21): $C_{V1} = \$3,850$. $C_{E1} = C_{V1}(p/Y + b) = \$5,050$. Equation (31): $J_1 = 1.0171$. Equation (46): $n_1 = 6.87$. Equation (49): $B_1 = 1,083$. Equation (56): $n_{dmin} = 17.6$. Equation (48): $A_1 = 172.5$. Abscissa of Figure 8 = 2.15, and economic $R = 0.7$.

ACKNOWLEDGMENT

The author is very pleased to acknowledge the assistance of David MacLean, Turbo-Mixer Division of General American Transportation Corporation, who provided the cost data for the agitation equipment and advice in its selection, and the helpful suggestions of John Happel.

NOTATION

A = factor defined by Equation (48)
 b = annual maintenance cost as a fraction of equipment cost
 B = factor defined by Equations (49) and (50)
 C_A = uninstalled cost of agitating equipment, \$/yr.
 C_d = uninstalled cost of a distillation tray and accompanying shell, \$/sq. ft. tray
 C_E = annual cost of a real extraction stage including equipment and operating cost, \$/yr.
 C_h = uninstalled cost of heat transfer equipment, \$/sq. ft.
 C_{hc} = cost of steam and coolant, \$/lb. mole distillate
 C_M = uninstalled cost of empty mixing vessel, \$/stage
 C_P = uninstalled cost of pump, \$/stage
 C_S = uninstalled cost of settler, \$/stage
 C_T = total annual cost of a complete extraction process, \$/yr.
 C_V = uninstalled cost of major equipment of an extractor, \$/stage
 c_F = concentration extractable solute in feed, lb./cu. ft.
 c_R = concentration extractable solute in raffinate, lb./cu. ft.
 c_S = concentration extractable solute in recycled solvent, lb./cu. ft.
 D = molecular diffusivity, sq. ft./hr.
 D' = effective molar diffusivity, sq. ft./hr.
 d = pipe diameter, ft.
 d_p = drop diameter, ft.
 E_d = fractional over-all tray efficiency for distillation

E_M = fractional Murphree extract stage efficiency
 E_{MD} = fractional Murphree dispersed-phase stage efficiency
 E_o = fractional over-all stage efficiency for extraction
 e = 2.7183
 F = value of extractable solute, \$/lb.
 G_d = allowable mass velocity of vapor in distillation, lb.-moles/(hr.) (sq. ft.)
 G_h = vapor handling capacity of heat transfer equipment, lb.-moles/(hr.) (sq. ft.)
 g = constant, Equation (54).
 g_c = conversion factor, (ft./hr.²) (lb.-mass/lb.-force)
 H = time of operation, hr./yr.
 J = quantity defined by Equation (31)
 K, K', K'' = constants
 k = mole ratio, solute/solvent, in distilled product
 k_c = mass transfer coefficient for continuous phase, ft./hr.
 L = agitator turbine diameter, ft.
 l = fraction of solvent flow lost/real stage
 \ln = natural logarithm
 \log = common logarithm
 M_E = mole wt. extractable solute, lb./lb.-mole
 M_S = mole wt. solvent, lb./lb.-mole
 m = equilibrium distribution coefficient, (concentration in extract)/(concentration in raffinate), (lb./cu. ft.)/(lb./cu. ft.)
 Note: if raffinate is dispersed, m for Figure 7 only must be (concentration in raffinate)/(concentration in extract)
 N = agitator speed, rev./hr.
 n = number of ideal extraction stages
 n_d = number of ideal distillation trays
 n_{dmin} = minimum number of ideal distillation trays
 O = rate of distillate flow, lb.-moles/hr.
 P = agitator power applied to liquids, ft.-lb./hr. (stage)
 P_A = agitator power, input to agitator drive, hp./stage
 P_P = pump power, input to pump drive, hp./stage
 p = (cost of instruments, piping, and installation as a fraction of major equipment cost) + 1
 Q = volume rate of total liquid flow = $q_D + q_C$, cu. ft./hr.
 q = volume rate of flow of a phase, cu. ft./hr.
 q_F = volume rate of feed flow to extractor, cu. ft./hr.
 R = volumetric ratio of flow rates, extract/raffinate or solvent/feed. Note: if raffinate is dispersed, R for Figure 7 only must be raffinate rate/extract rate.
 r = reflux ratio for distillation, lb.-mole/lb.-mole distillate

r_{min} = minimum reflux ratio for distillation
 S = value of solvent, \$/cu. ft.
 T = diameter and height of mixing vessel, ft.
 U = rate of extraction, lb. mole solute extracted/hr.
 V = volume of mixing vessel, cu. ft.
 W = cost of power, \$/hp.
 X = dimensionless parameter
 x = scale-up index, defined by Equation (7)
 Y = payout time, yr.
 Z = dimensionless parameter
 I, II, III, IV, V, VI = contributions to the cost of an extraction process, defined in Table 1, \$/yr.

Greek Letters

α = relative volatility
 β = r/r_{min}
 γ = n_d/n_{dmin}
 $\Delta\rho$ = difference in densities, lb./cu. ft.
 ϵ = $\alpha + \beta - 1$ if solvent is more volatile than solute
 = 1 if solute is more volatile than solvent
 θ = holding time for dispersed phase in mixer, hr.
 μ = viscosity, lb./ft. hr.
 ρ = density, lb./cu. ft.
 φ = volume fraction of a phase in the mixer

Subscripts

C = continuous phase
 D = dispersed phase
 S = solvent
 1 = large-scale plant
 2 = small-scale model

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