Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

Design, Scale-Up, and Applications of the Reciprocating Plate Extraction Column

A. E. Karr[a]
[a] Chem-Pro Equipment Corp., Fairfield, New Jersey

To cite this Article Karr, A. E.(1980) 'Design, Scale-Up, and Applications of the Reciprocating Plate Extraction Column', Separation Science and Technology, 15: 4, 877 — 905
To link to this Article: DOI: 10.1080/01496398008076276
URL: http://dx.doi.org/10.1080/01496398008076276

SEPARATION SCIENCE AND TECHNOLOGY, 15(4), pp. 877-905, 1980

DESIGN, SCALE-UP, AND APPLICATIONS OF THE RECIPROCATING PLATE EXTRACTION COLUMN

A. E. Karr
Chem-Pro Equipment Corp.
27 Daniel Road
Fairfield, New Jersey 07006

ABSTRACT

The Reciprocating Plate Extraction Column has been scaled up successfully from small diameter (1" and 2") test columns to many production units up to 40 inches in diameter. Optimization of the extraction process in the small diameter test column is the key to the economic optimum scale-up. The optimization procedure includes maximizing volumetric efficiency as well as selecting the best plate spacing variation. The latter can be calculated from the physical properties of the system and the throughputs in different sections of the column.

Design features, principles of design, and scale-up procedure will be reviewed. Several typical applications will be discussed.

INTRODUCTION

The first paper on an open type Reciprocating Plate Extraction Column (RPEC) was published about twenty years ago.(1) In that paper it was shown that a three-inch diameter column exhibited high volumetric efficiencies compared to other small diameter columns on the market for both low and high interfacial tension systems. Volumetric efficiency is defined as follows:

$$\text{Volumetric Efficiency} = \frac{\text{Total Throughput}, \frac{m^3}{hr.\times m^2}}{\text{HETS} \quad m} = \frac{1}{hr.}$$

Subsequently, high volumetric efficiencies were confirmed in a 12-inch diameter column (2) and a 36-inch diameter column.(3), (4)

The RPEC is now being employed extensively in laboratories, pilot plants, and industry around the world.

What is a Reciprocating Plate Extraction Column? Basically, it consists of a multiple of perforated plates with relatively large holes and a high percentage of open area assembled in the form of a plate stack which is reciprocated vertically inside a shell.

Fig. 1 shows a typical perforated plate.

Fig. 2 shows a typical baffle plate. Baffle plates are used periodically in the plate stack to minimize axial mixing.(2), (3), (4)

Fig. 3 shows a typical plate stack. This plate stack is nominally 40 inches in diameter, 40 feet

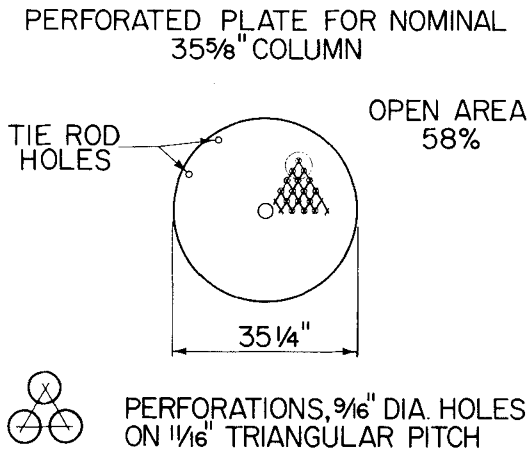

FIGURE 1. Typical perforated plate.

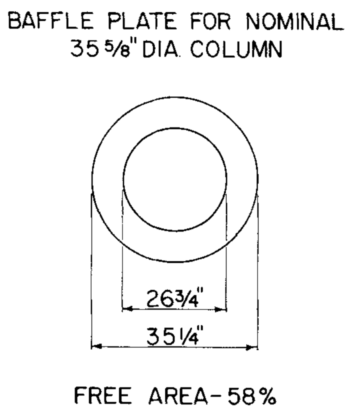

FIGURE 2. Typical baffle plate.

long. The plate stack is built in four sections with couplings between each section. Fig. 4 shows one of the end spider plates, and one can see the ends of the tie rods which pass through the plate

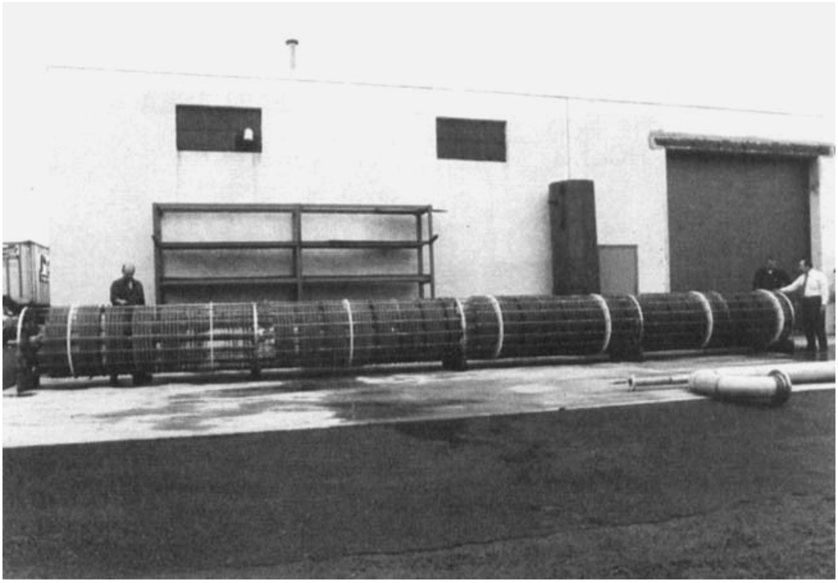

FIGURE 3. Typical plate stack.

stack. The plates are kept separated by spacers which fit over the tie rods. The combination of spider plates, tie rods, and spacers provide for a rigid construction of the plate stack. Fig. 5 shows the arrangement of perforated plates and baffle plates. The white baffle plates are Teflon and are slightly larger in diameter than the stainless steel perforated plates and baffle plates. This prevents metal-to-metal contact of plates and shell.

Fig. 6 shows a typical coupling arrangement.

FIGURE 4. Plate stack showing end spider plate.

DESIGN OF RECIPROCATING PLATE EXTRACTION COLUMN

The design characteristics of a reciprocating plate extraction column are as follows:

1. Large open area - typically 55%
2. Large diameter holes - typically 9/16".
   The large open area and hole size contributes to the high throughputs achieved. The large open area eliminates the need to maintain a close clearance between the plates and shell.

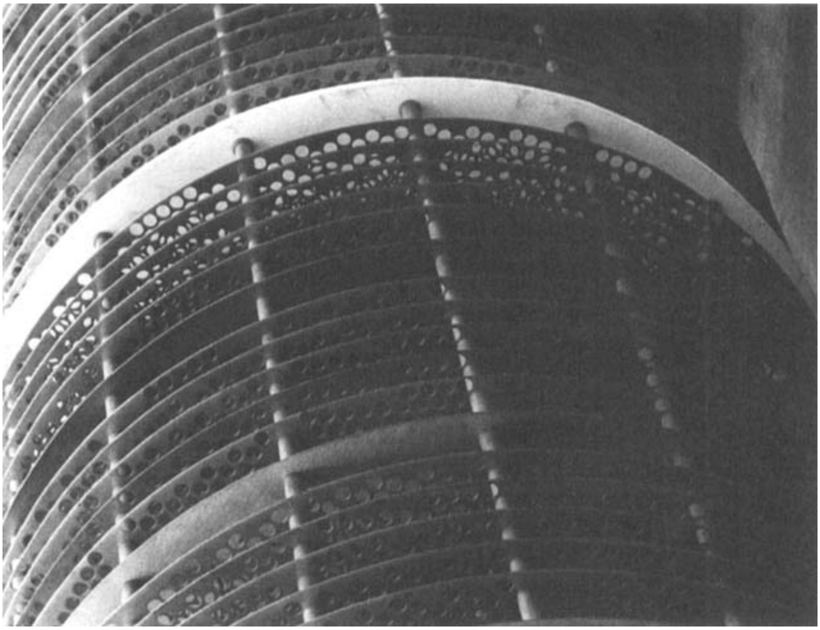

FIGURE 5. Arrangement of perforated plates and baffle plates.

3. Stroke length - typically 3/4" (range 1/2" to 1").
4. Reciprocating speed - typically 150 RPM (range 10 to 400).
5. Plate spacing - 1" to 8" - typically 2". However, frequently plate spacing should be varied in different portions of the column in accordance with the relative values of density difference and interfacial tension. This is discussed later.
6. Baffle plates - these are advisable in

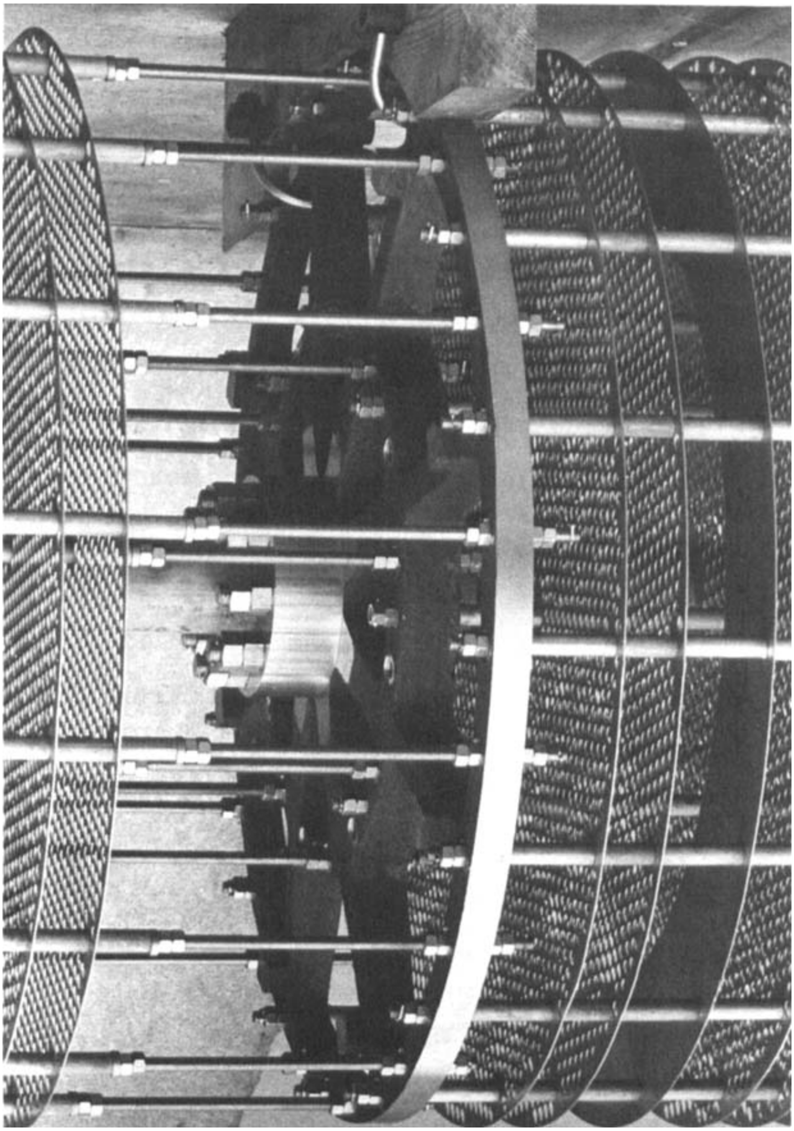

FIGURE 6. Typical coupling arrangement.

columns greater than 3 inches in diameter to minimize axial mixing. Fig. 7 shows a typical perforated plate and baffle plate arrangement.

Let's briefly discuss the state of the art of designing internally agitated extraction columns. Although a great deal of work has been done on the design of extraction columns from basic principles, to my knowledge no manufacturer of extraction equipment will provide a guaranteed design without some test work. Designing an extraction column from basic principles would require knowledge of at least the following variables:

1. Mass transfer coefficient dispersed phase, k_d
2. Mass transfer coefficient continuous phase, k_c
3. Drop size)) Calculate area
4. Holdup)
5. Axial mixing coefficients E_c, E_d
6. Flooding

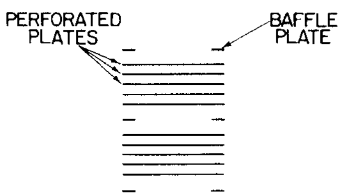

FIGURE 7. Typical perforated and baffle plate arrangement.

Above depend on

1. Physical properties
2. Design and arrangement of internals
3. Direction of extraction
4. Dispersed phase
5. Type of agitation, agitation intensity
6. Solvent ratio

Empirical correlations are available for some of these variables for some types of equipment, but usually there is some or a great deal of information lacking. Thus, one inevitably resorts to test work and then an empirical or semi-empirical scale-up procedure is employed. With good planning, test work required for scale-up can be minimized and performance optimized with relatively few runs.

Dr. M. H. I. Baird and his co-workers at McMaster University in Hamilton, Ontario have made a great deal of progress in delineating some of the fundamental hydrodynamic characteristics of the reciprocating plate extraction column. They have studied and correlated flooding, holdup, drop size, and axial dispersion.(5), (6), (7) For four different systems in which the interfacial tension was approximately 30 dynes/cm. Baird showed:

$$(U_D + 0.67U_C) = 24.2\ Af^{-1.2} \rho_c^{-\frac{1}{3}} (\rho_D - \rho_c)^{2/3} \left(\frac{\mu_c}{\mu_{co}}\right)^{-\frac{1}{3}} \qquad \text{(Eq. 1)}$$

For the more general case - based partly on theoretical considerations.

$$(U_D + 0.67U_C) = 2.24 \times 10^{-2} \left(\frac{\sigma^3}{\psi^2 \bar{\rho}}\right)^{0.2} \left(\frac{g^2 \Delta\rho^2}{\rho_c \mu_c}\right)^{1/3} \qquad \text{(Eq. 2)}$$

The $(U_D + 0.67U_C)$ function has recently been modified.(8)

We have made good use of these equations, as an excellent guide, but we still do not depend entirely upon them because we find in the presence of mass transfer adjustments to the correlation may be necessary.

From the equations it is obvious that if physical properties, especially density difference and interfacial tension, vary in different parts of the column, then agitation should also be varied to prevent one section of the column from severely limiting throughput. Since agitator speed and amplitude are fixed throughout the column, the only way to vary agitation intensity is to vary plate spacing.

Power input per unit volume, ψ is inversely proportional to plate spacing, ℓ. From (Eq. 2) one can derive that for practical purposes by considering only the most important physical properties of $\Delta\rho$ and σ the optimum relative plate spacing in the different parts of the column is given by the following equation:

$$\ell \propto \frac{1}{(\Delta\rho)^{5/3}\,(\sigma)^{3/2}} \qquad \text{(Eq. 3)}$$

Where ℓ is the optimum relative plate spacing in any location in the column

$\Delta\rho$ = density difference in the corresponding column location

σ = the interfacial tension in the corresponding column location

We have used the above equation successfully in optimizing plate spacing. The net result is a design which optimizes the degree of agitation throughout the column, thereby maximizing throughput and extraction efficiency and, therefore, minimizing investment.

As mentioned before, we still rely to a great extent on test work for efficiency and throughput data. The economic design of the production unit can be only as good as the optimization job done in obtaining the pilot scale data. These are recommendations for obtaining the pilot scale data.

1. Estimate Throughput, Agitator Speeds, and Test Column Height

   A preliminary estimate of the allowable range of throughputs and agitator speeds can be gleaned from the correlations of Baird (5), (8) or from previous experience. The height of plate stack selected for the tests will depend on the number of theoretical stages required.

2. Estimate Plate Spacing Variation

   If the density difference and interfacial tension are known to vary from one end of the column to the other, it is possible to estimate optimum plate spacing variation from Equation 3. The minimum plate spacing to be used is 2 inches, unless the plate spacing variation is very large, in which case the minimum plate spacing should be 1 inch.

3. Determine Minimum HETS at Three Throughputs

Runs are made on at least three different throughputs and HETS is determined as a function of RPM. Since the minimum HETS frequently occurs close to the flood point in small diameter columns, it is usually sufficient to determine the agitator speed at which the column will just flood, and then reduce the agitator speed 5 or 10%. The determined value of HETS will be close to the minimum.

4. Plot Minimum HETS vs. Throughput

The minimum value of HETS is plotted against throughput and normally HETS will increase with throughput.

5. Plot Volumetric Efficiency vs. Throughput

Maximum volumetric efficiency at each throughput is then plotted against throughput. If sufficient data are gathered, the volumetric efficiency will pass through a maximum, which is usually close to the economic optimum design throughput for scale-up.

6. Extraction Process Should be Demonstrated

If possible the extraction process should be demonstrated. There should be sufficient stages in the test column to obtain the product desired in the required yield. There could be pitfalls in determining the height of an equivalent theoretical stage and calculating the height of the column based on the number of theoretical stages required. The possible pitfalls are inadequate distribution data or HETS data in the area not tested.

7. Ready for Scale-up

You are now ready for scale-up. With good preparation the entire procedure described above can be carried out in two or three days. It is time well spent. Because of the relative simplicity of carrying out the tests it will probably be a long time before it is feasible to replace them entirely with calculation procedures alone.

SCALE-UP PROCEDURE

The scale-up procedure for the Reciprocating Plate Extraction Column is based on performance data obtained in 1", 3", 12", and 36" diameter columns.(1), (2), (3), (4)

1. Data are obtained in a 1", 2" or 3" diameter column. The diameter selected is frequently determined by the availability of material. Many successful scale-ups have been done directly from the 1" diameter column. The 2" and 3" pilot plant columns may be used for scale-up data development where the production column is to be in excess of 2 feet in diameter and where unusual conditions are involved.
2. The optimum performance of the pilot column is determined. The criteria for optimum performance is maximum volumetric efficiency in a column having optimum plate spacing.
3. When scaling up from the pilot data the following parameters are held constant:

   Plate spacing

   Stroke length

   Throughput per $ft.^2$
4. The expected minimum HETS in the large diameter column is calculated from the following equation:

$$\frac{(HETS)_{D_2}}{(HETS)_{D_1}} = \left(\frac{D_2}{D_1}\right)^{0.38}$$

5. The corresponding reciprocating speed required is calculated from the following equation:

$$\frac{(SPM)_{D_2}}{(SPM)_{D_1}} = \left(\frac{D_1}{D_2}\right)^{0.14}$$

6. Suitable baffle plates in design and spacing are provided.

The above procedure has been used to scale-up thirty-five production columns, all successfully. The largest column built so far is 40 inches in diameter having 40 feet of plate stack. We are now prepared to scale-up to 6 feet in diameter or larger.

APPLICATIONS

A few applications of the Reciprocating Plate Extraction Column follow.

Fig. 8 shows a 40" diameter stainless steel column containing 32 feet of plate stack operating on the purification of an organic compound. The column was scaled-up from data obtained in a 2" diameter column. Specification products were produced in the first runs at design conditions.

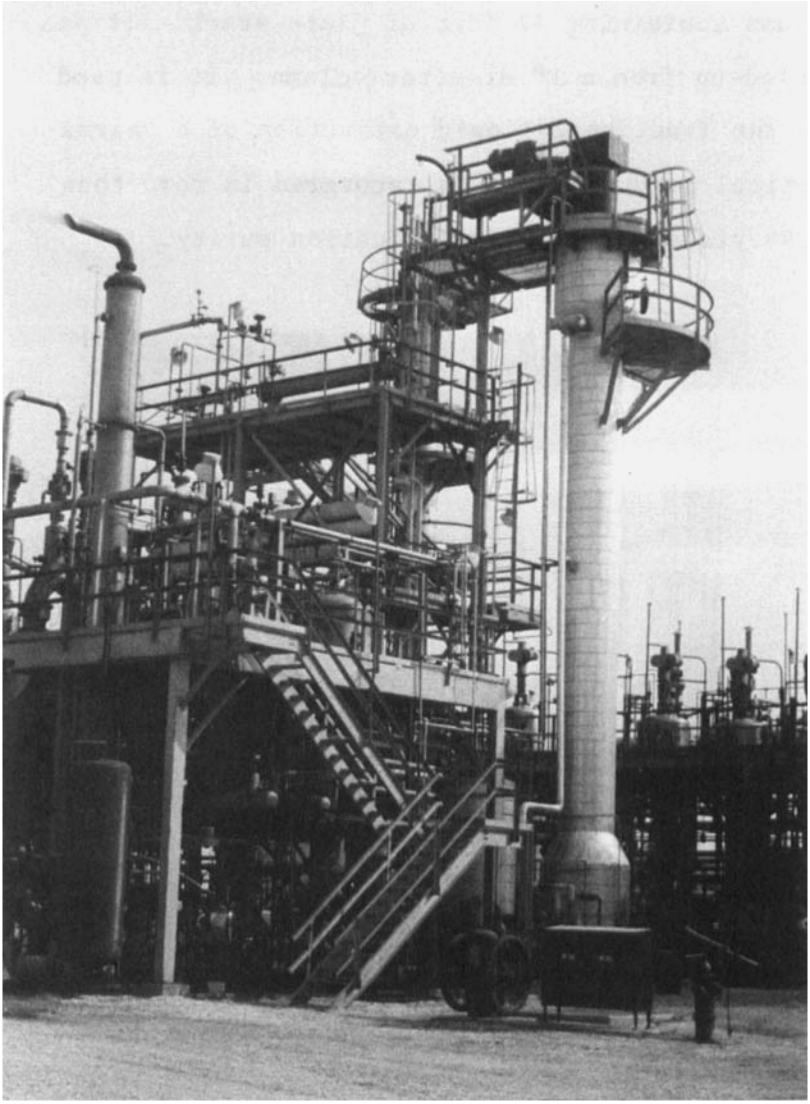

FIGURE 8. 40-inch diameter column containing 32 feet of plate stack.

Fig. 9 shows a 30" diameter stainless steel column containing 42 feet of plate stack. It was scaled-up from a 1" diameter column. It is used for the fractional liquid extraction of a pharmaceutical product which is recovered in more than 99.9% yield and with specification purity.

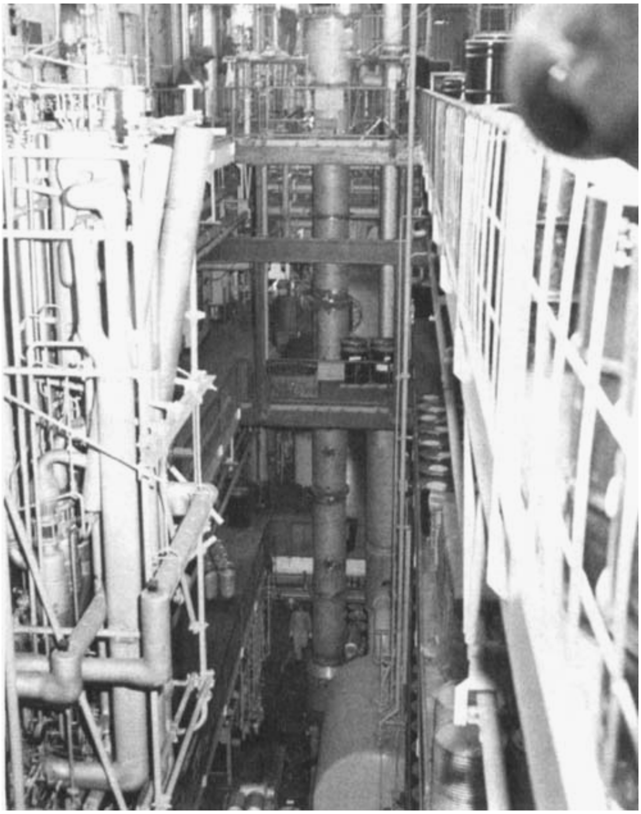

FIGURE 9. 30-inch diameter column containing 42-feet of plate stack.

Fig. 10 shows the lower portion of a 12" diameter column used for the extraction of a whole fermentation broth containing about 5% unfiltered mycelia. Recently we scaled up a similar application from a 1" diameter column to 18" in diameter. For many years the latter application was being processed in a centrifugal extractor, but the operation was very tedious in that the broth first had to be given a preliminary batch extraction, followed by three passes through the centrifugal extractor. The whole broth was processed in the reciprocating plate extraction column in a single pass through the column and with no preliminary batch extraction. Solvent usage was reduced 65%, and investment and maintenance was significantly reduced.

From the above experiences, the reciprocating plate extraction column should be considered for the processing of whole or filtered fermentation broths. Emulsions are avoided because of the uniform agitation over the cross-sectional area of the column.

Fig. 11 is a view of a 24" diameter column operating on the solvent system hexane and water.

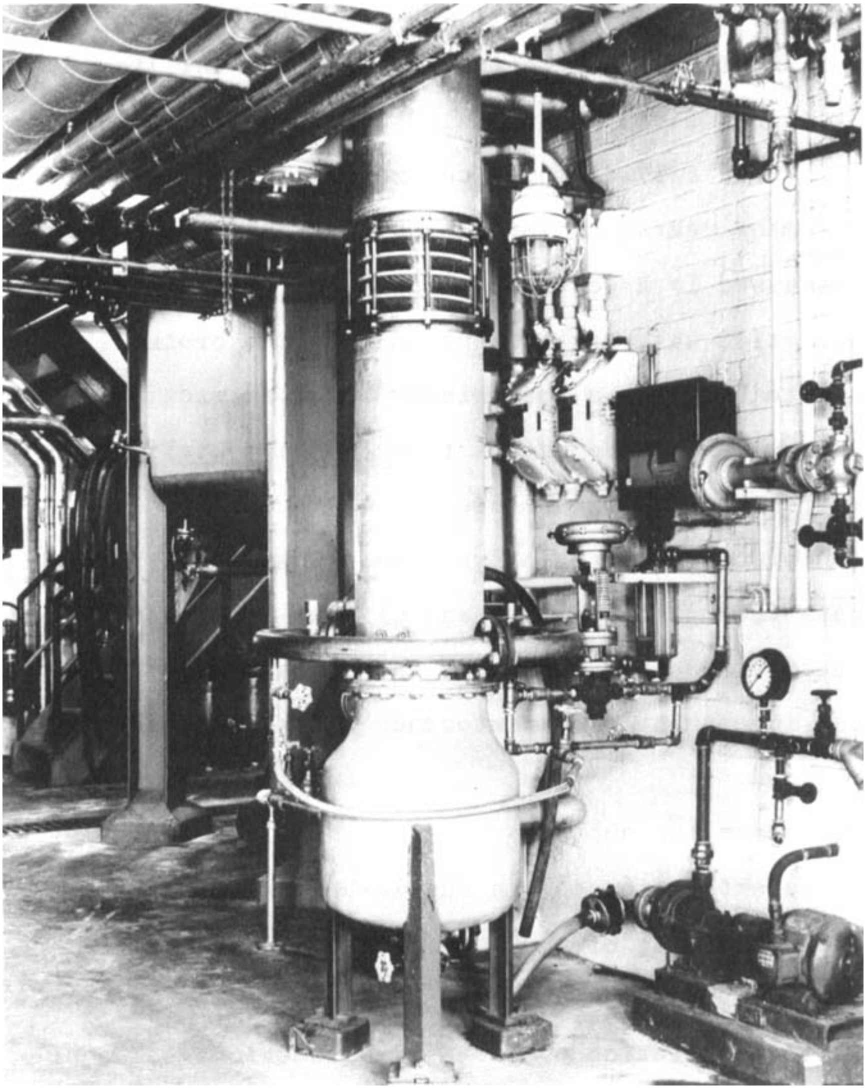

FIGURE 10. 12-inch diameter column for extracting whole fermentation broth.

FIGURE 11. 24-inch diameter column for high interfacial tension system.

This system has a very high interfacial tension, approximately 50 dynes/cm. and, therefore, requires a high degree of agitation, around 350 strokes/min., and 3/4" stroke length.

Fig. 12 shows a view of a 30" diameter Teflon plate stack 30 feet long for use in a glass-lined shell. The spider plate shown here is of Hastelloy construction and so are the tie rods and central shaft. Teflon-lined shells can also be used with a Teflon plate stack. In fact, a wide selection of corrosion resistant materials of construction can be used in the reciprocating plate column. For example,

Fig. 13 shows a view of a polypropylene plate stack.

Fig. 14 shows some possible materials of construction.

Fig. 15 shows an installation for the recovery of phenol from a saturated aqueous stream. The concentration of phenol in the raffinate is less than 10 ppm. Units can be designed to discharge raffinates with less than 1 ppm phenol.

The reciprocating plate extraction column is

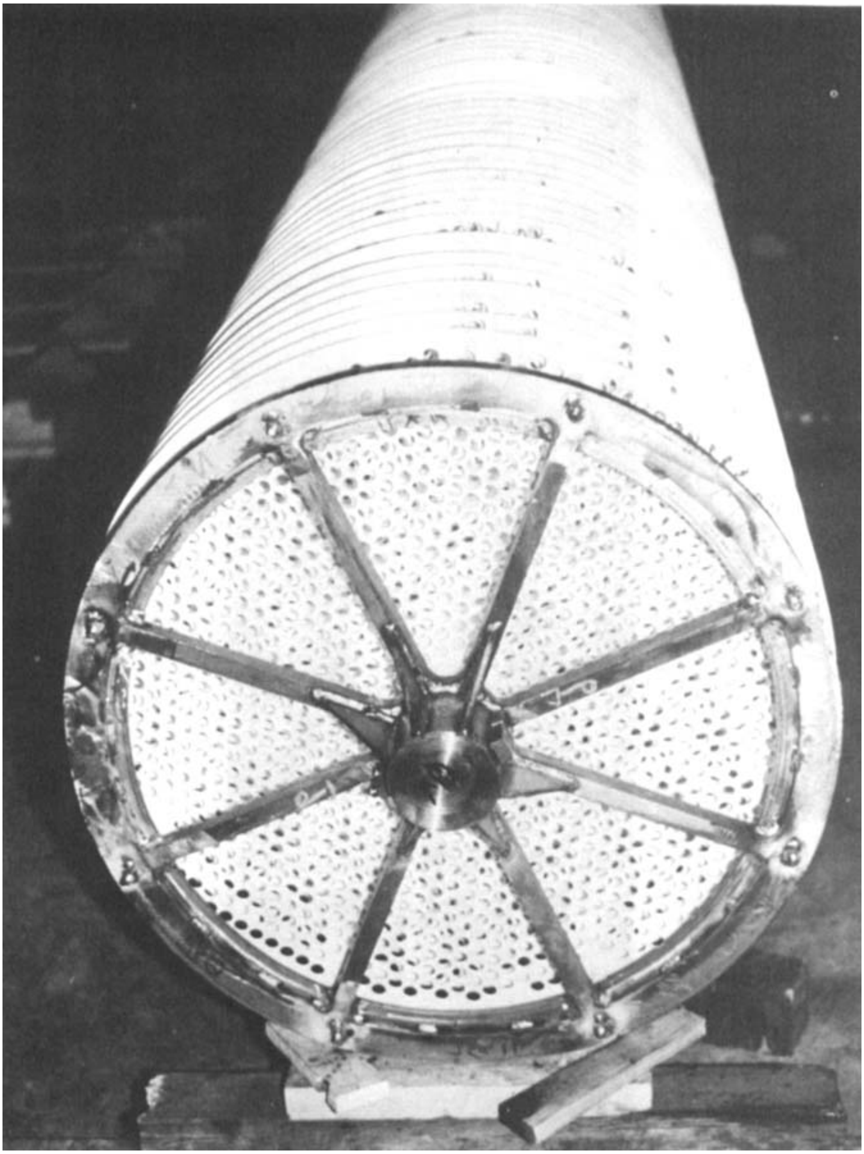

FIGURE 12. 30-inch diameter Teflon plate stack.

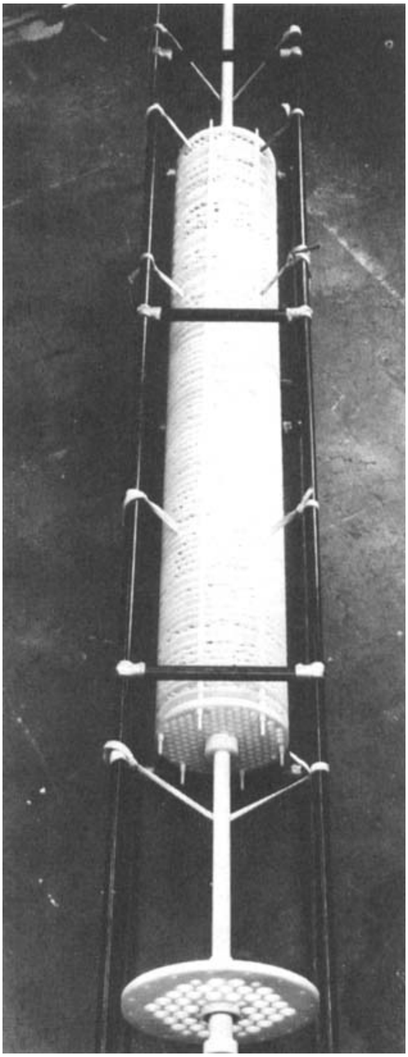

FIGURE 13. Polypropylene plate stack, 18 inches diameter.

MATERIALS OF CONSTRUCTION TO DATE

SHELLS	PLATES	SPIDERS SHAFTS, TIE RODS
FRP	TFE	TFE SHEATHED
FEP LINED	S.S.	FEP SHEATHED
KYNAR LINED	CARBON STEEL	HASTELLOY C
GLASS	TITANIUM	S.S.
GLASSED STEEL	POLYPROPYLENE	CARBON STEEL
S.S.	HASTELLOY C	TITANIUM
C.S.		GLASS FILLED TFE
Ti		

FIGURE 14. Materials of construction.

also being used industrially for the recovery of acetic acid from aqueous streams. The solvent to be employed and the processing flowsheet depends upon the acid concentration in the feed stream.

The column has also been used to extract organics and color bodies from aqueous waste streams. Extraction should be considered for such applications especially at concentrations greater than 2000 ppm.

Many other extraction operations are being carried out in the reciprocating plate extraction column in the pharmaceutical, chemical, food, petrochemical and hydrometallurgical industries.

Recently several applications for the reciprocating plate extraction column as a cocurrent

FIGURE 15. Phenol recovery installation.

contactor have been developed. Fig. 16 shows a unit being used as a plug flow type mixer as in a mixer-settler operation. The advantages are high throughputs, reduced settler volume, and reduced overall cost. The cocurrent mixer configuration has also been used effectively as a plug flow reactor for two liquid phases.

ADVANTAGES

Some of the advantages of the Reciprocating Plate Extraction Column are as follows:

1. Low HETS) High volumetric
) efficiency
2. High throughput)
3. Handles emulsifiable materials
4. Handles solids

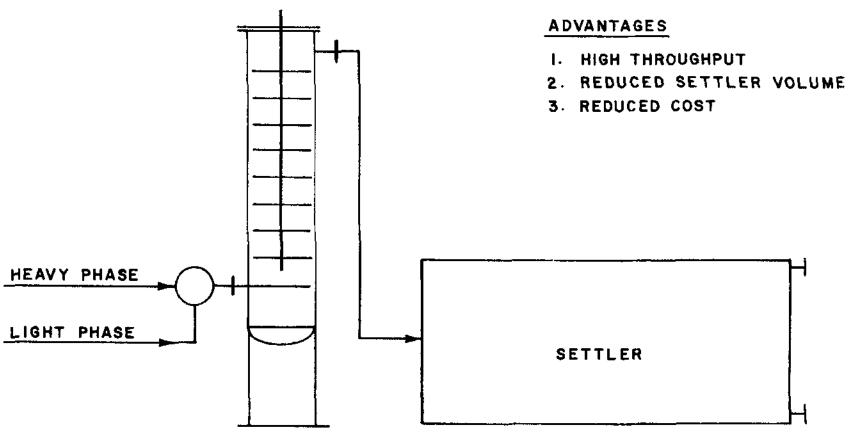

FIGURE 16. Karr Column as cocurrent mixer.

5. Wide range of materials of construction including glass and Teflon
6. Reliable scale-up procedure
7. Flexibility permits optimization of lab or pilot plant unit
8. Low cost and low maintenance

NOMENCLATURE

A Amplitude, cm.

f Frequency, Hz.

g Gravitational acceleration cm./sec.^{2}

Plate spacing, cm.

U Superficial velocity at flooding point, cm./sec.

Density difference $(\rho_D - \rho_C)$, g/cm.^{3}

Viscosity, poise

co Reference viscosity 0.01 poise

Energy dissipation per unit volume, ergs/cm.^{3}sec.

Density, grams/cm.^{3}

Average density

Interfacial tension, dyne/cm.

Subscripts

C - Continuous phase

D - Dispersed phase

REFERENCES

(1) A. E. Karr, A.I.Ch.E. Journal 5, 446 (Dec. 1959)

(2) A. E. Karr and T. C. Lo, Proceedings of the International Solvent Extraction Conference, Society of Chemical Industry (London), Vol. 1, 299 (1971)

(3) A. E. Karr and T. C. Lo, Chemical Engineering Progress 72, 68 (Nov. 1976)

(4) A. E. Karr and T. C. Lo, Proceedings of the International Solvent Extraction Conference, Society of Chemical Industry, Toronto, Sept. 1977

(5) M. H. I. Baird, R. G. McGinnis, and G. C. Tan, Proceedings of the International Solvent Extraction Conference, Society of Chemical Industry, The Hague, April 1971, p. 251-259

(6) M. H. I. Baird and S. J. Lane, Chem. Eng. Sci. 28, 947 (1973)

(7) S. D. Kim and M. H. I. Baird, Can. J. Chem. Eng. 54, 81 (1976)

(8) M. M. Hafez, M. H. I. Baird, and I. Nerdosh, Can. J. Chem. Eng. 57, 150 (1979)