PERFORMANCE OF CENTRIFUGAL EXTRACTORS

David B. Todd and Gordon R. Davies
Baker Perkins Inc.
Saginaw, Michigan, USA 48601

ABSTRACT

The design and operation of Podbielniak type centrifugal liquid-liquid extractors are described. Performance data on extraction efficiency, capacity, and residence time distribution are presented on a new more compact, more portable, and more versatile pilot extractor, which is capable of operation with centrifugal forces up to 10 000 G's. These data are compared with similar information on commercial centrifugal extractors, covering the combined throughput range from 0.1 to 100 m^3/hr.

INTRODUCTION

Centrifugal extractors, although widely used, are probably equally widely poorly understood. Some of the mystery about their operation stems from a lack of visibility. It is not possible to make a glass or plastic centrifugal extractor that will operate with the centrifugal forces commercially encountered. Nor is is convenient to obtain intermediate samples required for study of discrete stage phenomena. Thus, as may be common with many chemical reactors, conclusions are drawn about what is happening internally by the observation of output responses to input changes.

The very compactness of centrifugal extractors has posed some challenging questions for both the manufacturer and the user. Relatively high capacities are achieved in a small geometric space. The short residence time (usually less than one minute) makes it possible to study many process variables, since steady state is so rapidly approached. In commercial operation, an upset in the inlet conditions is reflected so rapidly in the extract and raffinate that corrective actions can be initiated equally rapidly without jeopardizing production or producing a sizeable inventory of off-spec product.

Although centrifugal extractors exist in which the axis of rotation is vertical (1), this discussion is confined to the horizontal axis species, generally known as the Podbielniak type contactor.

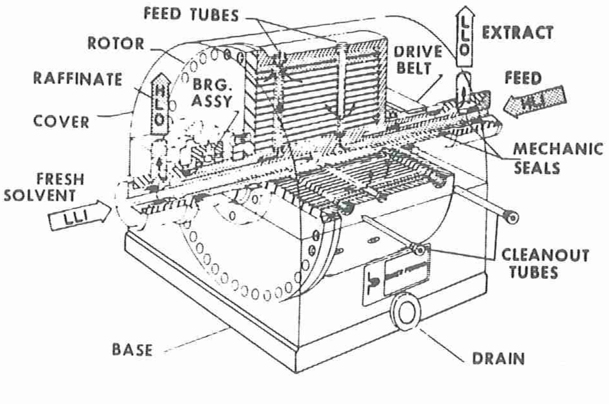


FIGURE 1. CUTAWAY OF CENTRIFUGAL EXTRACTOR

DESIGN AND OPERATION

The centrifugal extractor is a perforated plate extraction tower (without downcomers) that has been wrapped around a shaft, which in turn is rotated to create a centrifugal force field that allows a great reduction in the height and the time required to achieve a countercurrent contacting process result. The typical features of the commerical size continuous countercurrent centrifugal extractor are illustracted in Figure 1. All fluids enter and leave the rotor via mechanical seals and shaft passageways. Radial conduits connect the shaft passageway to the appropriate collection or distribution points within the rotor.

The extraction process may be described by assuming a typical extraction of a solute from an aqueous feed with a less dense organic solvent. The organic solvent is introduced into the rotor through distributors located near the periphery. Aqueous feed is introduced similarly through distributors located nearer the shaft. The heavier liquid is centrifuged outward, causing a displacement of the lighter phase inward. The countercurrent flow of the two phases through the compartments effects a series of intimate contacts. Clarification zones are provided inboard of the feed inlet and outboard of the solvent inlet. The extract collects at the shaft. The raffinate collects at the periphery, and then is led back through conduits to the shaft passageway.

Mechanical features provided include access ports to the rotor interior (ASCO tubes) which can be used for inspection or cleaning. Disassembly is not required for cleaning, although one rotor end plate can be removed. The internal contacting trays are now made with such precision that an existing centrifugal extractor can be retrayed in the field should the intended service be changed. The radial position of the feed ports may be altered, without disassembly, by reworking or replacing the appropriate radial conduits. The rotor is driven via a belt connected to a fluid coupling variable speed drive. Power requirements are low since the fluids enter and leave at the same axial "ground-stage," and only windage and friction must be overcome.

The comparision with the perforated place tower is more readily visualized by looking at the radial section in Figure 2. The centrifugal force accelerates the separation process, and allows greatly reduced plate spacings (in the order of 6 mm), with the resultant compaction of tower height and greatly reduced settling times.

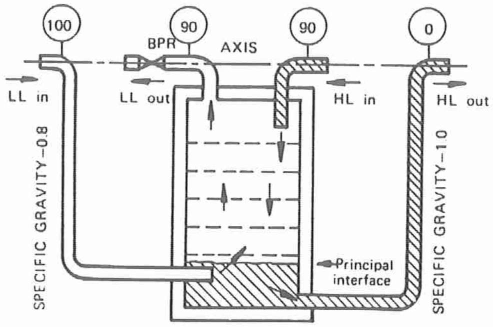


FIGURE 2. TOWER ANOLOGY OF A CENTRIFUGAL EXTRACTOR

The positioning of the principal interface between the two phases is accomplished by imposing a back pressure on the light liquid effluent. That is, the selection of the predominantly dispersed and the predominantly continuous phase may be accomplished in a manner similar to that used for liquid level control in a tower. Although the "height" may be very small, and the specific gravity difference may be slight, the product of these two terms multiplied by the G-forces yields a satisfactory pressure differential. The inlet and outlet pressures can be interpreted in terms of legs of a manometer, and the position and nature of the principal interface within the centrifugal extractor can be deduced from the pressure interrelationships (2).

A head of coalesced dispersed phase accumulates above or below each tray depending on which phase is continuous (i.e. a head of heavy liquid exists inboard of the tray for light liquid continuous). The depth of this phase (h), or the depth of continuous phase displaced, and the associated centrifugal force, provide the pressure drop for the liquids through the orifices. In the most simplistic of terms, this may be respresented by:

$$2\,(pH - pL)\,RW^2h = pC\,(Q_C/A_C)2 + pD\,(Q_D/A_D)^2 \qquad (1)$$

where R is the radius, W is the angular velocity, pH, pL, pC and pD are the densities of the heavy, light, continuous (light or heavy) phase and dispersed phase respectively, Q_C and Q_D is the volumetric flow rate of each phase, and A_C and A_D, the flow cross-sectional area, may be related to the actual open area by orifice coefficients.

PILOT CENTRIFUGAL EXTRACTORS

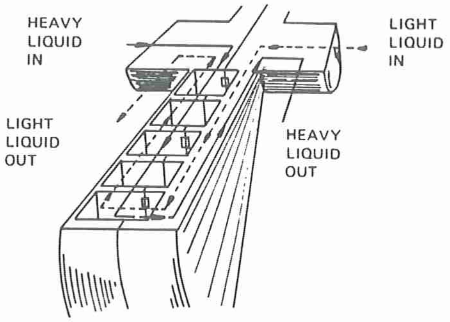
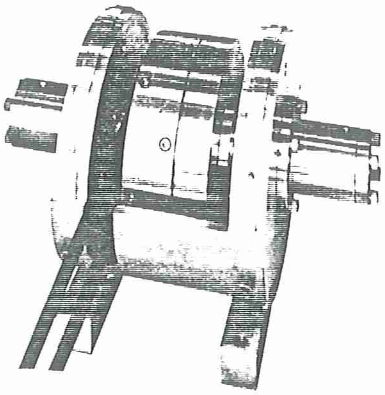


FIGURE 3. SCHEMATIC DIAGRAM OF PUP EXTRACTOR

The early pilot centrifugal extractor was the Podbielniak Pup. Most of the extraction performance data which have been published (3, 4, 5, 6) have been obtained on these units. The construction consisted of a series of milled annular grooves, as compartments, with two milled slots between each pair of grooves schematically illustrated in Figure 3. The two 430 mm diameter end plates were held together with 72 bolts, many of which passed through the extraction zone. The rotor interior was only about 6 mm wide. The machined design, as well as the small internal dimensions, precluded the study of geometric variables, such as tray spacing, hole size (which may be a large as 12 mm in commercial units), hole shape, and hole patterns. The maximum speed was 5000 RPM, and a typical maximum capacity was about 0.5 liters per minute. Although only a pilot device, the rotor and base weighed about 95 kg. The heavy weight, coupled with the complications of the closing bolts, made this pilot equipment not readily serviceable by laboratory personnel.

Recently, a new pilot centrifugal extractor, the Model A-1 as shown in Figures 4 and 5, has been developed which is even more compact, and yet has features much more in common with the commercial counterparts. The 216 mm outside and 178 mm inside diameter rotor is constructed with a removable end plate, and fourteen removeable trays, spaced at 4 mm. This offers the adaptability to investigate various tray designs, and to a limited extent, the effect of tray spacing. The trays are gasketed in each end plate. This positive seal eliminates possible leaks around the trays, and the possible misinterpretation of data where end effects might be accounting for a large portion of the capacity. The internal width is 25 mm, and the total internal volume is about 0.5 liter.



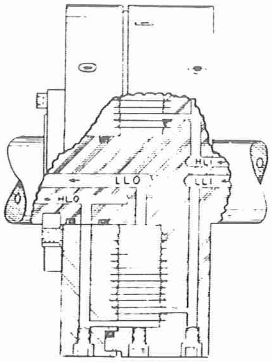


FIGURE 5. CROSS-SECTION OF PILOT EXTRACTOR

Both heavy and light fluids enter and leave via three radial distributors. Typically, the heavy liquid enters at a radius of 40 mm, and the lighter liquid at a radius of 88 mm, with ten cylindrical contacting trays in between. Two coalescing trays are provided outboard of the countercurrent contacting zone for clarification of the heavy phase. Similarly, two coalescing trays are provided inboard of the contacting zone for clarification of the light phase. The ratio of contacting to clarifying trays may be varied. Rim plugs at each of the radial inlet and exit channels, as well as around the tray section may be removed to allow cleaning and inspection.

The stainless steel rotor and aluminium housing weigh only 46 kg., and are mounted with a variable speed drive on a 70 cm by 110 cm aluminium base plate, permitting desk top operation. The light weight, coupled with the ease of disassembly, including a simple split key at the shaft to accomplish end-plate closure, facilitates the retraying of the unit to study alternate designs. Depending upon the internal tray design, maximum capacity ranges from 0.5 – 5 liters per minute. The maximum operating speed is 10 000 RPM, at which speed 10 000 G's (multiples of gravity) are developed at the rim. With only a fraction-of-a-minute holdup time, steady state is rapidly approached, and a multitude of process conditions can be tested in a very short period.

PILOT PLANT PERFORMANCE

Extraction. The pilot plant centrifugal contactor was installed in the test loop as schematically illustrated in Figure 6. Temperature and pressure gauges were installed at each of the inlets and outlets. Spring loaded, diaphragm type pressure control regulators were located in both the light liquid (methyl isobutyl ketone) and heavy liquid (deionized water) effluent lines. The feed system for each phase consisted of a feed pump, back pressure regulator (by-pass valve), water cooled heat exchanger, calibrated rotameter, and needle flow control valve. The pump by-pass valves served to divert the unused portion of the feed capacity, as well as adjust the pressure such that a small pressure drop existed over the rotameter and needle valve flow regulating combination. This leads to more stable operations with minimal drift of rotameter float. Distribution coefficients for acetic acid partitioned between

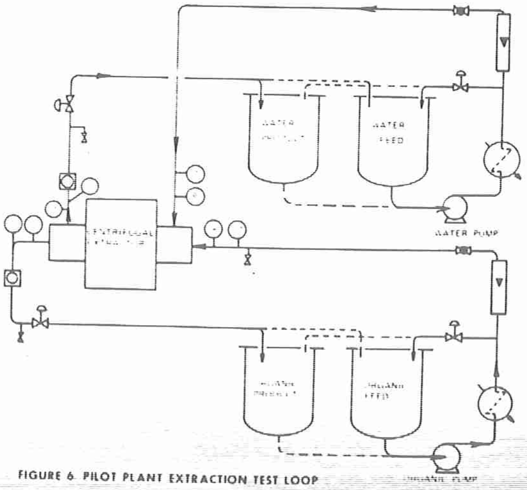


FIGURE 6. PILOT PLANT EXTRACTION TEST LOOP

methyl isobutyl ketone (MIBK) and water were determined for three concentration levels over the range of 26 to 38°C, employing standard titration techniques. The data proved to be linear and quite insensitive to temperature over the region of interest. The equilibrium curve is shown in Figure 7. A feed solution containing approximately 5% acetic acid in the methyl isobutyl ketone was prepared. Typical performance data are shown in Table 1 for three different internal designs. For this system, both speed of rotation and location of principal interface are important. Maximum efficiency was obtained at the maximum operating speed. Performance was also better when the entire countercurrent contact zone was filled with the low flow aqueous phase, in agreement with the general rule that the liquid with the greater flow rate should be selected as the dispersed phase. The McCabe-Thiele plot for the best run is illustrated in Figure 7, with 5.4 theoretical stages.

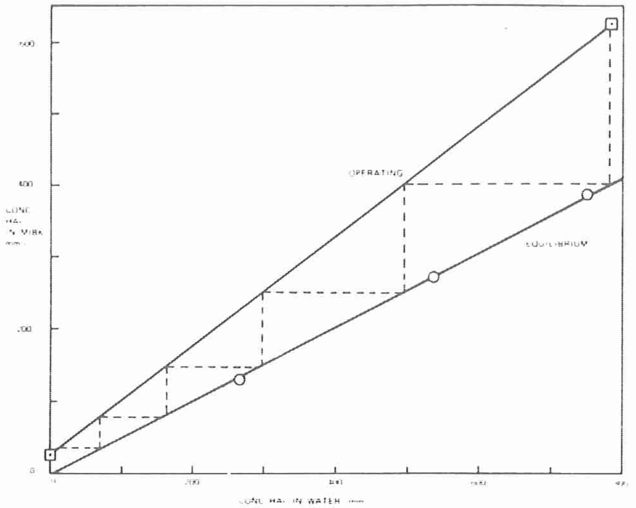


FIGURE 7 EQUILIBRIUM AND THEORETICAL STAGES. MIBK-HAC-WATER

TABLE 1

Performance of Model A–1 Laboratory Extractor
Extraction of Acetic Acid from MIBK into Water

Internal Set	Rpm	Liter/min. MIBK	Liter/min. Water	Interface R/R Max.	No. of Stages
A	10 000	0.755	0.551	0.40	5.4
	10 000	0.755	0.551	0.55	4.5
B	10 000	0.755	0.551	0.55	4.2
	10 000	0.755	0.551	0.40	5.0
	7 000	0.635	0.525	0.40	3.9
C	10 000	0.755	0.551	0.40	3.7

The model system of methyl isobutyl ketone-acetic acid-water with extraction from the organic to the aqueous phase has been used to illustrate the applicability of general extraction rules, for column extractors, to the centrifugal contactor. Both the choice of the continuous phase, and the approach to flooding conditions, each of which relate to the available interfacial area for mass transfer, have indicated a marked difference in extraction efficiency. An overall extraction efficiency of 60% per compartment has been demonstrated for this system. In actual practice, other circumstance may influence the preference for the continuous phase, and extraction efficiency may be sacrificed. An undesirable density gradient caused by solute transfer may preclude choice of the low flow liquid as the continuous phase. The tendency toward emulsification is usually greater at the feed-extract end than at the solvent-raffinate end. Emulsification difficulties are often altered by entering one of the liquids into a large body of the other. In general, interfacial emulsions for the formation of a steady-state interfacial "rag" are more readily accommodated at the rotor light-liquid-in port where a higher centrifugal force exists. In the case of explosive or radioactive materials, minimizing the inventory of one of the phases, may be predominant in the selection of the dispersed phase.

Capacity. The flooding of the centrifugal contactor is similar to columns in that three types are possible. Light liquid (or shaft) flooding, observed by entrainment of heavy liquid into the light liquid effluent, may occur, even at low capacities, by moving the major interface through the light liquid clarifying zone, and out of the equipment. At higher capacities this type of flooding may occur, even with the major interface positioned in the contacting zone, when the clarifying section residence time and settling forces are insufficient to achieve coalescence and disengagement of droplets generated in the contacting zone. Heavy liquid (or rim) flooding, observed by entrainment of light liquid into heavy liquid effluent, may occur in an analogous manner at the other extreme of the column or centrifugal contactor. Capacity flooding is observed by simultaneous flooding (entrainment) at both ends of the unit. This flooding is initiated in the contact zone, and occurs when an excessive head (h) of coalsesced dispersed phase is required to generate the pressure drop for flow of the liquids through the orifices. The tray design in the contacting section should be selected such that flooding occurs in each compartment at approximately the same capacity for maximum extraction efficiency, and the avoidance of a capacity bottleneck in a portion of the section. The pilot centrifugal contactor was operated in the test loop (Figure 6) on the system kerosene (specific gravity approximately 0.8)-water. Typical flooding envelope capacity curves, for two internal designs, at three speeds and two phase ratios are presented in Figures 8 and 9. The ordinate $\bar{P}_{LLO}$ (=$P_{LLO}-P_{HLO}$) represents the range of back pressures on the exiting light phase (LLO) over which the extractor may be operated. The 3x set of internals was specifically designed to provide a threefold increase in capacity.

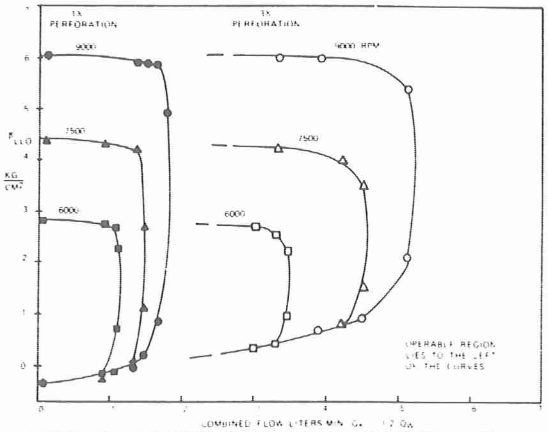


FIGURE 8. FLOODING ENVELOPES AT Q_k=0.5 Q_w (KEROSENE-WATER SYSTEM)

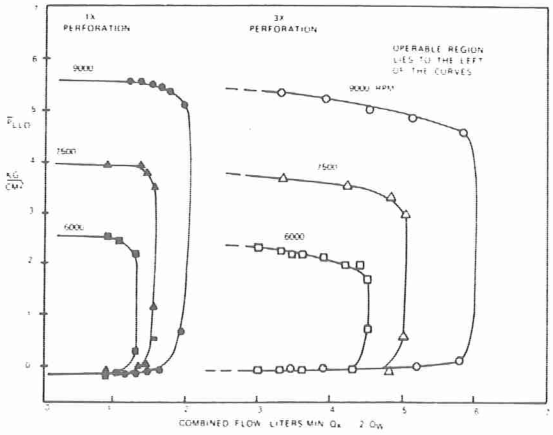


FIGURE 9. FLOODING ENVELOPES AT Q_k= 2.0 Q_w (KEROSENE-WATER SYSTEM)

The approach to capacity flooding, as distinct from shaft or rim flooding, is extemely rapid, and definition of the curve at these points is difficult. The maximum abcissa lines represent inoperable capacity flooding for the respective conditions, whereas the plotted points represent a full range of operable major interface posititions from light liquid flooding to heavy liquid flooding. The approximate flood point capacity, for each of the curves, is shown in Table 2.

TABLE 2

Maximum Capacity (Flooding Point) Kerosene—Water
Model A—1 Extractor

Internal Set	QK/QW	Combined Flow, liters/min., at Rotor RPM		
		6000	7500	9000
1X	0.5	1.14	1.50	1.75
	1.0	1.18	1.53	1.80
	2.0	1.31	1.58	2.03
3X	0.5	3.50	4.53	5.16
	1.0	3.98	4.65	5.48
	2.0	4.51	5.03	6.00

The data are in general agreement with Equation 1; namely, that the combined flow is proportional to the first power of speed. This test also confirmed the attainment of the threefold capacity increase. The total capacity appears to be higher when the ratio of light-to-heavy phases is increased.

Residence Time Distribution. The distribution of residence times for the flow of water alone was determined by injection of a pulse of concentrated $NaNO_3$ solution, and measuring the cumulative amount collected in the effluent as a function of time. The tracer was charged to a small injection loop. A quarter turn of a 4-way valve permitted injection of the tracer without any interruption in flow. The amount of $NaNO_3$ in sequential samples was determined by measuring the electrical conductivity. The cumulative distributions for five difference flow rates are shown in Figure 10, compared with the theoretical curve for 13 well-mixed stages in series. Except for the 300 and 600 ml./min. rates, the data points all lie close to the theoretical curve. One can conclude from these data that each compartment is well mixed, and that there is essentially no backmixing across the trays at high net flows. The amount of backmixing can be deduced from the shape of the cumulative distribution curve (7). Where the data coincide with the theoretical curve for the same number of compartments, the amount of backmixing is negligible. For the 300 ml./min. flow, the amount of backmixing is estimated to be 175 ml./min. At 600 ml./min., this had dropped to 150 ml./min. At 1080 ml./min., no further backmixing could be detected.

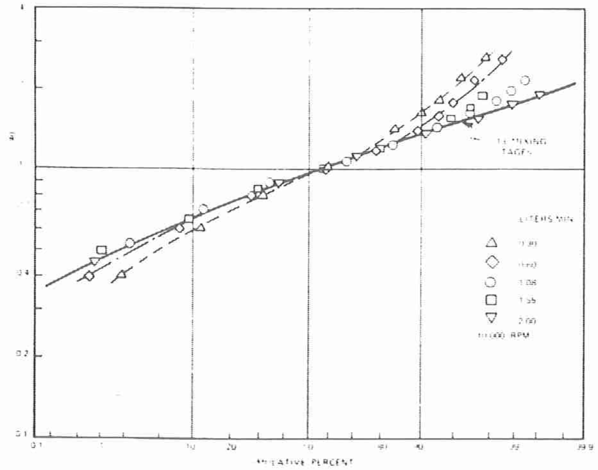


FIGURE 10. RESIDENCE TIME DISTRIBUTIONS FOR THE PILOT CENTRIFUGAL EXTRACTOR

COMMERCIAL SCALE PERFORMANCE

Users of commercial centrifugal extractors are generally most reluctant to release their operating data. The commercial units, once installed, are generally run to meet production needs, and are not available at the investigator's whim to demonstrate performance over a wide range of variables. The manufacturer of the equipment is limited in his ability to develop the data without constructing a complete solvent recovery plant. Despite these limitations, some data have been obtained with several of the smaller commercial Model B–10 extractors (capacity about 7 m^3/h) and with an intermediate size Model D–18 (capacity about 23 m^3/h). Dimensions of these units are listed in Table 3.

TABLE 3

Dimensions of Centrifugal Extractors

Model	Rotor ID, mm	Rotor Width mm	Holdup Liters	RPM at 1.0 S.G.	Max. G's	Rated Capacity M^3/H(a)
A–1	180	25	0.5	10 000	10 030	0.1
B–10	580	250	57	3200	3340	7
D–18	860	450	220	2100	2130	34
D–36	860	900	440	2100	2130	68
E–48	1140	1200	980	1600	1630	136

(a) For typical multi-stage extraction.

Pressure Drop. The pressure drop for single phase flow is shown in Figure 11 for a Model B–10 extractor. The total pressure drop can be divided into extrance and exit losses, and a loss through the contacting zone. The data indicate a significant contribution of rotation above that at zero rpm. This additional energy dissipation is one of the factors controlling droplet size.

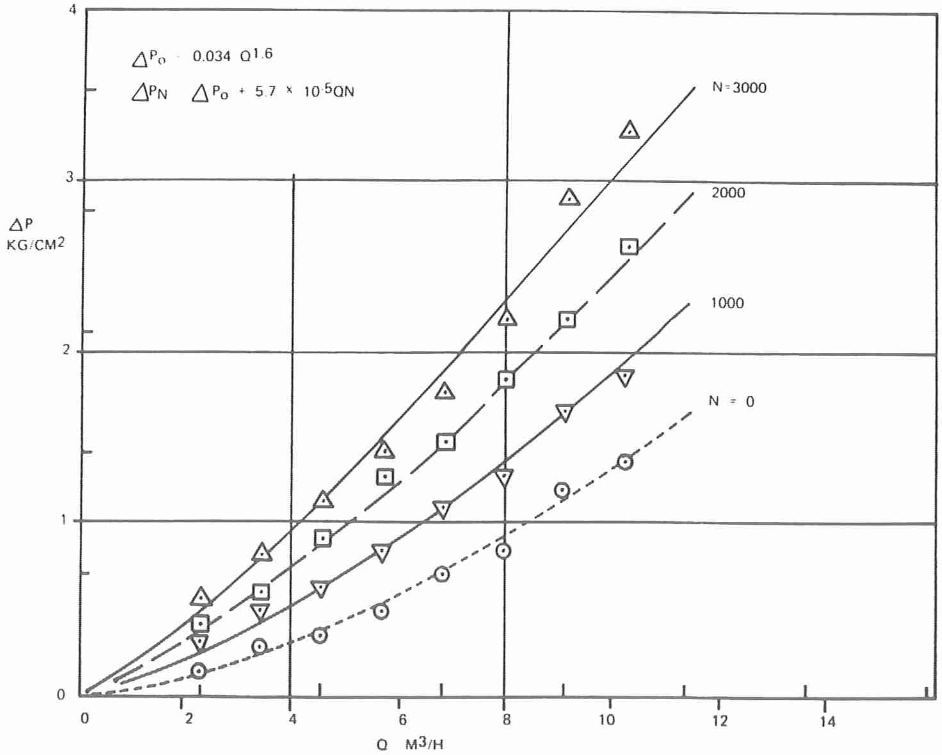


FIGURE 11. PRESSURE DROP THROUGH A MODEL B-10 CENTRIFUGAL EXTRACTOR

For centrifugal extractors, the data can generally be correlated by a power equation for zero rpm and an additive term for the effect of rotor speed. The pressure drop at zero rpm (P_o) is dependent upon the geometry and flow rate Q:

$$P_o = ZQ^a \tag{2}$$

The additive term for rotation depends upon both flow rate Q and rotor speed N:

$$P_N = P_o + BQN \tag{3}$$

Z and B in the above equations are constants depending upon the system and extractor. The curves in Figure 11 are plotted on the basis of the constants noted.

The effect of countercurrent flow is demonstrated in Figures 12 and 13 for the water-kerosene system in the same Model B–10 contactor. The water phase pressure drop is determined under conditions wherein the principal interface is held inboard of the heavy phase inlet (contact zone filled with water). The kerosene phase pressure drop is similarly determined, with the interface held just outboard of the light phase inlet (contact zone filled with kerosene). As either flow is increased, the required pressure increases, probably due to both an increased holdup of the dispersed phase and the competition of both fluids for the available flow passageways within the rotor.

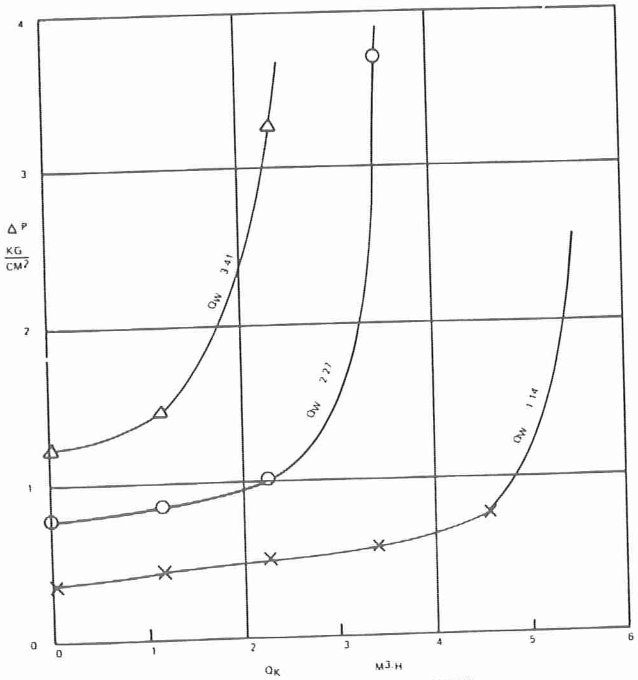


FIGURE 12. EFFECT OF KEROSENE FLOW ON WATER PHASE PRESSURE DROP (MODEL B-10 AT 3000 RPM)

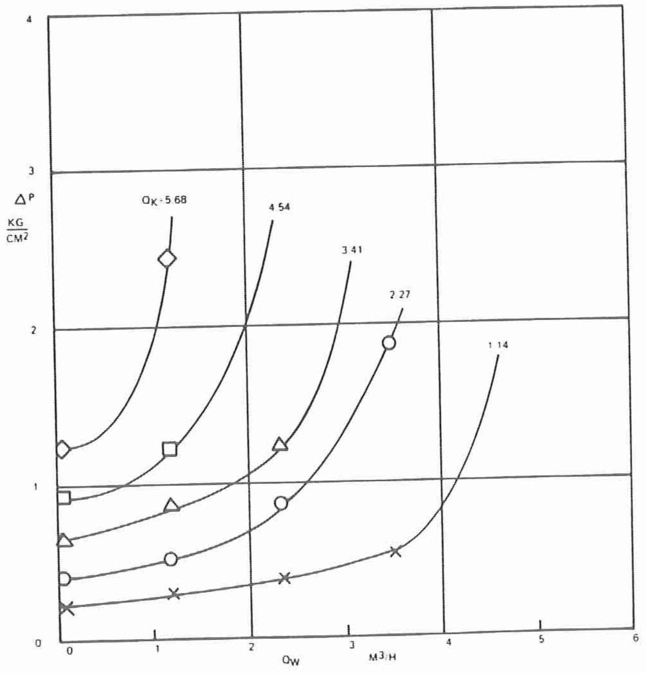


FIGURE 13. EFFECT OF WATER FLOW ON KEROSENE PHASE PRESSURE DROP (MODEL B-10 AT 3000 RPM)

Curves similar to Figures 11, 12 and 13 have been generated for the other sizes of centrifugal extractors.

Extraction. The extraction of n-butyl amine from kerosene with water was studied in both the Model B–10 and D–18 extractors listed in Table 3. Typical extraction data for the Model B–10 extractor operating at 3000 rpm with 3.4 m^3/h of kerosene and 1.7 m^3/h of water are shown in Table 4 and Figure 14.

TABLE 4

Extraction of n Butyl Amine from Kerosene with Water
Model B–10 Extractor, 3000 RPM, 3.4 M^3/H Kerosene
1.7 M^3/H Water

Run	$\frac{R}{R\ Max}$	Concentration (mm/l)				No. of Stages
		Feed	Raffinate	Solvent	Extract	
105	0.40	596	105	0	910	6.6
107	0.82	38	9.5	6	62	6.7
109	0.36	39	5.5	3	62	6.3
118	0.36	52	20	4	74	5.3
119	0.40	56	25	3.5	71	3.6
120	0.69	66	26	3.5	94	6.4
122	0.97	64	18	3	91	4.0
124	0.34	169	44	2	241	4.0

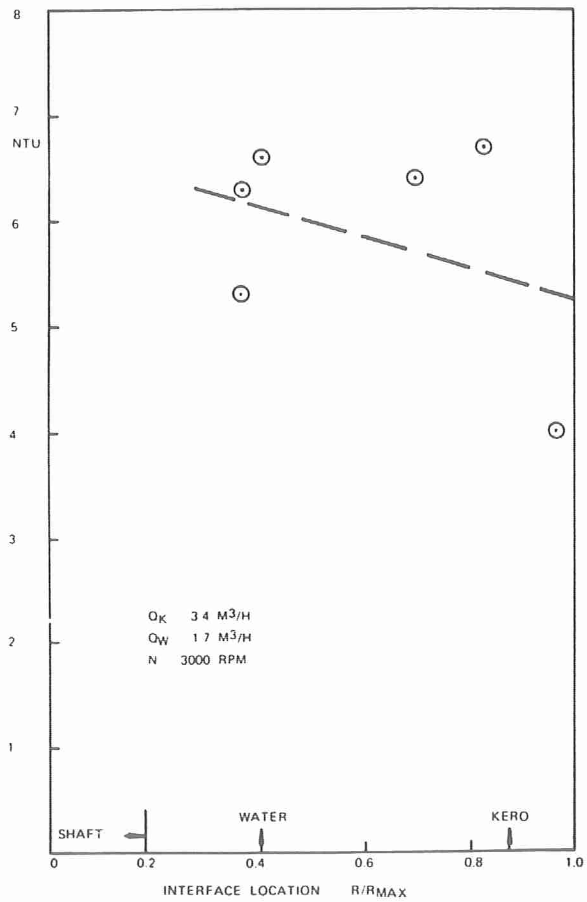


FIGURE 14. EXTRACTION STAGES STAGES FOR KEROSENE-n BUTYLAMINE-WATER (MODEL B-10)

An attempt was also made to obtain direct contact heat transfer data for this extractor, as longer steady state runs could be obtained without as much testing difficulty. The number of heat transfer stages, as shown in Figure 15 parallel the data for mass transfer. For this series, there were 10 compartments between the warm kerosene and the cold water feeds.

Although the Model D–18 extractor has been designed for 23 m^3/h, it was tested on the same kerosene - n-butyl amine - water system at the maximum rates obtained with the existing pumping and metering system (11 m^3/h). The observed five theoretical stages (Figure 16) compare favorably with the data from the smaller units.

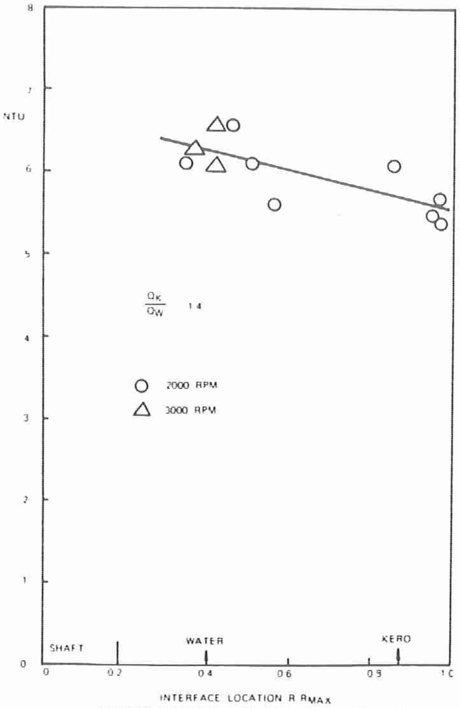


FIGURE 15. DIRECT HEAT TRANSFER STAGES KEROSENE-WATER (MODEL B-10)

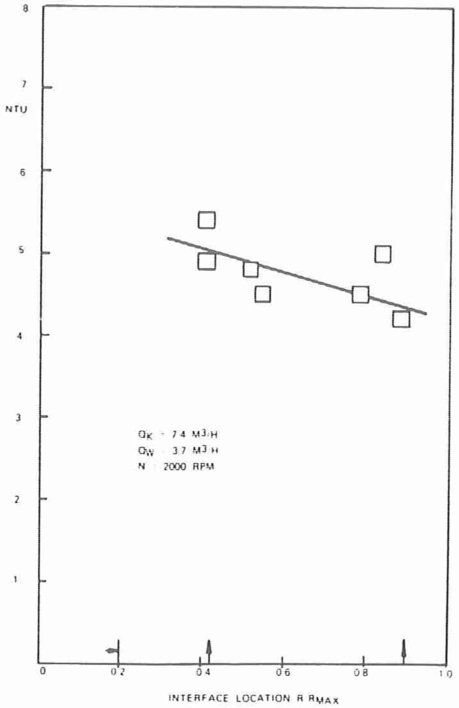


FIGURE 16. EXTRACTION STAGES FOR KEROSENE-n BUTYL AMINE-WATER (MODEL D-18)

Capacity. The flooding envelopes which define the capacity limits for commercial centrifugal extractors are very similar in form to those for the pilot extractors. Typical kerosene-water flooding envelopes for a Model B–10 extractor are shown in Figure 17. As was noted with the Model A–1 in Figures 8 and 9, the maximum combined flow is higher as the ratio of light-to-heavy phases is increased.

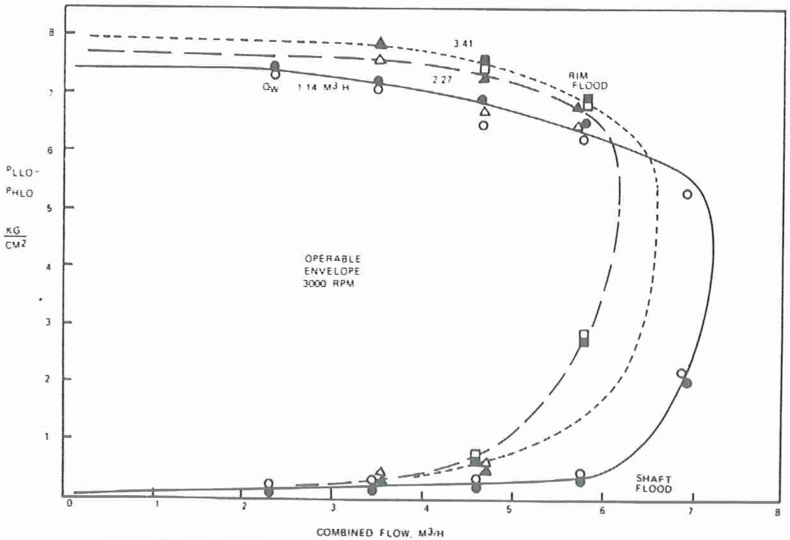


FIGURE 17. CAPACITY ENVELOPE FOR MODEL B-10 EXTRACTOR (KEROSENE-WATER)

As with the pilot units, the ultimate capacity is generally approximately proportioned to rotor speed (7). Some exceptions are encountered in the very readily emulsified systems. In these, the pressure differential over which the extractor may be operated is expanded as the rotor speed is increased, but the capacity flood point is not increased proportionately. Typical flooding limits as observed in a Model E–48 centrifugal extractor operating on the extraction of aromatics from lubricating oil with phenol as shown in Figure 18.

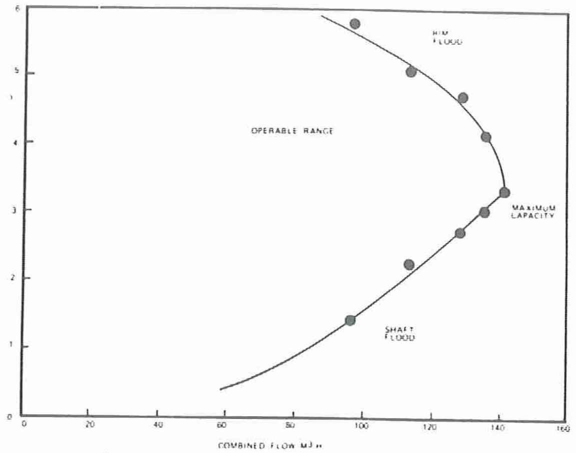


FIGURE 18. CAPACITY ENVELOPE OF MODEL E-48 EXTRACTOR. PHENOL-LUBE OIL 1600 RPM

Backmixing. The residence time distributions were determined for the aqueous phase with varying amounts of countercurrent kerosene flow in a Model B–10 extractor. The operating conditions were specifically chosen to exaggerate any backmixing effect – namely, a very low continuous phase superficial velocity and high countercurrent flows. The cumulative distributions are shown in Figure 19 for four flow conditions, and are compared with the curve for 25 theoretical well mixed stages. The amount of backmixing was deducted from the deviation between the data and the theoretical curve. Table 5 shows the calculated amount of backmixing. Apparently, increasing the counter flow of kerosene does not induce or entrain increasing quantities of water. As the water rate is increased the relative amount backmixing decreased, but the absolute quantity increased. This may reflect the increasing turbulence within the compartment.

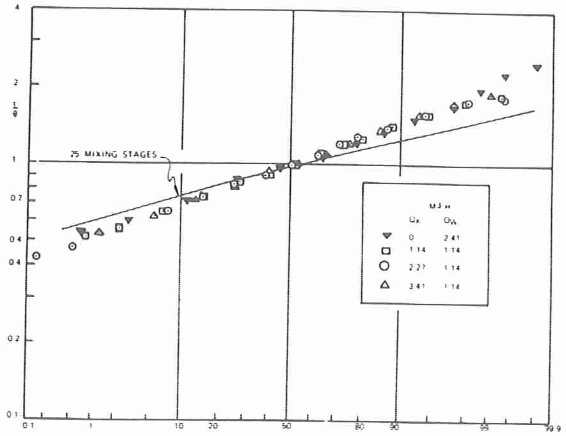


FIGURE 19. RESIDENCE TIME DISTRIBUTIONS FOR THE MODEL B-10 CENTRIFUGAL EXTRACTOR

TABLE 5
Effect of Dispersed Kerosene Flow on Backmixing in a Model B–10 Extractor, 3000 RPM

Flows (M^3/H)		
Kerosene Q_K	Net Water Q_W	Backmixing Q_B
0	2.41	0.85
1.14	1.14	0.53
2.27	1.14	0.61
3.41	1.14	0.59

CONCLUSIONS

Although differing in many respects from single gravity tower operation, the performance of centrifugal extractors bares a lot of similarities to conventional extraction towers. Operation close to flooding is frequently desireable to maximize mass transfer area. The ability to vary this point by varying the centrifugal force is an advantage that centrifugal extractors possess. However, the major advantages of centrifugal extractors evolve from either their compactness (I) or the use of mult-gravitational separating forces (II).

I. Process dependent upon compactness:

   a. Extraction of unstable ingredients, such as penicillin from acidified broth.

   b. Extraction where an expensive inventory of solvent would otherwise to tied up, such as in liquid ion exchange processes.

   c. Extraction or separation where expensive alloy materials of construction are required, such as in various acid treating processes.

   d. Extraction where space is at a premium, such as in existing buildings or on oil drilling rigs at sea.

II. Processes dependent upon superior separation:

   a. Extraction processes which involve easily emulsified systems, such as certain pharmaceutical extractions, vegetable oil refining or lube-oil refining with phenol.

   b. Extractions with very little specific gravity difference between the phases, such as plasticizer washing.

   c. Extractions where one or both phases are very viscous, such as catalyst removal from polymer solutions.

   d. Extractions where completely clarified effluent streams are mandatory.

The development of a new laboratory centrifugal extractor built along the same lines as the commercial units now permits better modeling studies and more assured scaleup.

REFERENCES

1. Todd, D. B. and Podbielniak, W. J., "Advances in Centrifugal Extraction," *Chemical Engineering Progress*, *61*, No. 5, 69 (1965).

2. Todd, D. B., "Improving Performance of Centrifugal Extractors," *Chemical Engineering*, *79*, No.16, 152 (1972).

3. Barson, N., and Beyer, G. H., "Characteristics of a Podbielniak Centrifugal Extractor," *Chemical Engineering Progress*, *49*, 243 (1953).

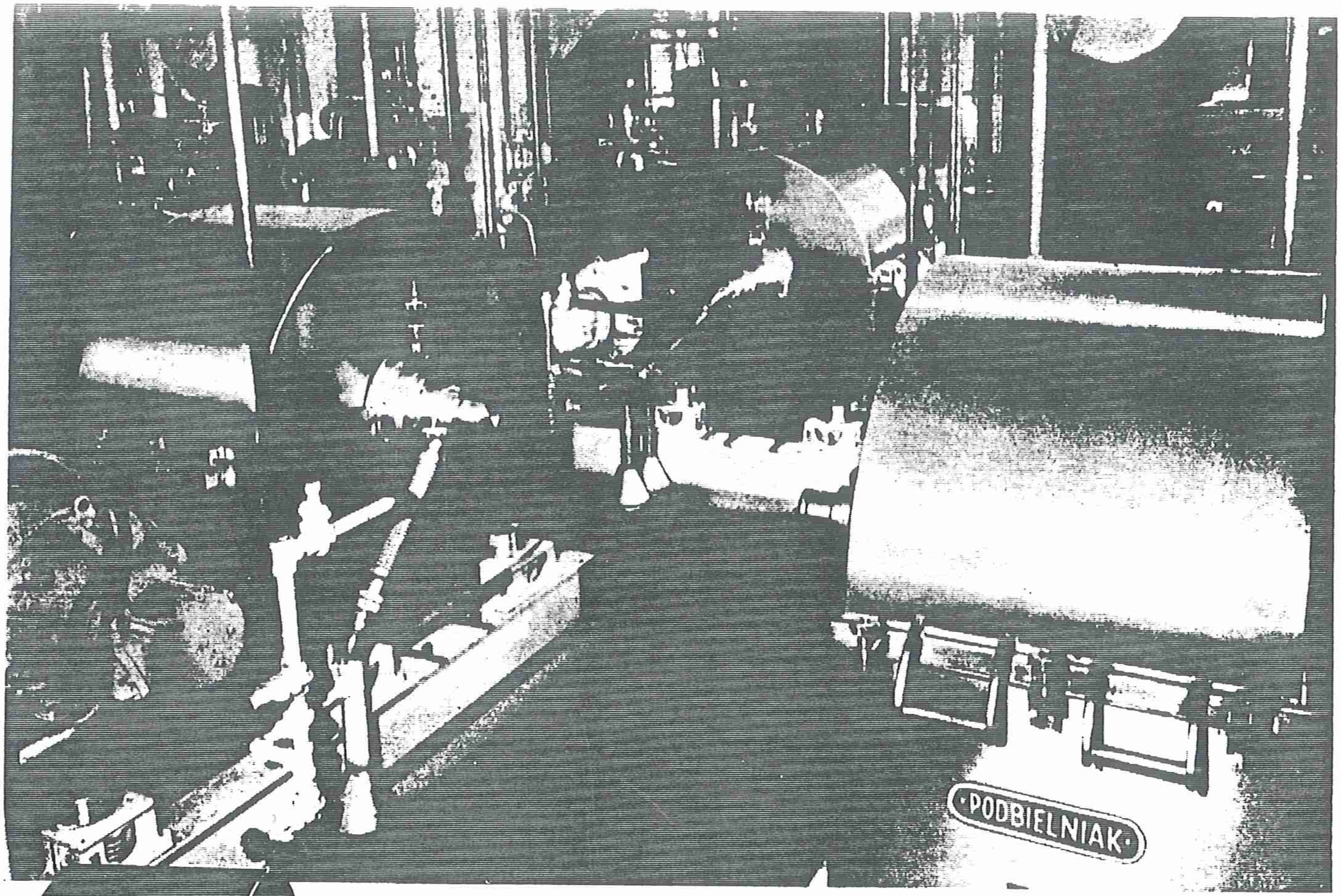
4. Alexandre, M., and Gentilini, P., "Determination de L'Efficacite d'un Appareil Semi-industriel d'Extraction par Solvents, Le Super-Contacteur Podbielniak." *Revue IFP II*, No. 3, 389 (1956).

5. Jacobsen, F. M., and Beyer, G. H. "Operating Characteristics of a Centrifugal Extractor," *AIChE Journal*, *2*, 283 (1956).

6. Fox, J. M., "Centrifuge Compared for Phenol Extraction of Lube," *Hydrocarbon Processing*, *42*, No. 9, 113 (1963).

7. Todd, D. B., "Multiple Functions in a Centrifugal Extractor," *Chemical Engineering Progress*, *62*, No. 8, 119 (1966).

Reprinted from the Proceedings of the International Solvent Extraction Conference, Lyon, Vol.3, 2380 – 2398 (September 1974).



CENTRIFUGES IN ACTION

Reprinted from CHEMICAL ENGINEERING PROGRESS, May 1965

Advances in centrifugal extraction

The centrifugal extractor greatly extends the field of application of extraction processes. Centrifugal extractors, varying in size from 17 to 60 in. in diameter, and in capacity from 0.06 to 600 gal./min. combined throughout, are now becoming a more frequent item in the CPI.

D. B. TODD *and*
W. J. PODBIELNIAK*
Podbielniak Division
Dresser Industries
Franklin Park, Ill.

ALTHOUGH MORE THAN 500 CENTRIFUGAL liquid extractors are in commercial operation at this time, many chemical engineers are still unfamiliar with the principle and operation of this distinctive type of processing equipment. Except for the theories proposed by Barson and Beyer (*1*), Jacobsen and Beyer (*2*), and Fox (*3*), based on experiments with small laboratory units, there has been little exposition of the operating mechanism of the centrifugal extractor beyond that available in the basic patents (*4-6*) and in manufacturer's literature (*7, 8*).

On commercial performance, there is a paucity of data on true countercurrent commercial applications of the centrifugal extractor, although several papers have been published on use of centrifugal contactors in vegetable oil refining, where the contactor is usually used as a cocurrent settler or clarifier. The scarceness of commercial data is attributable mostly to the natural reluctance of commercial users to divulge data regarded as confidential to their process. Thus, while there are now over a score of centrifugal extractors in phenol lube oil service at this time, no detailed

---

*Consultant.

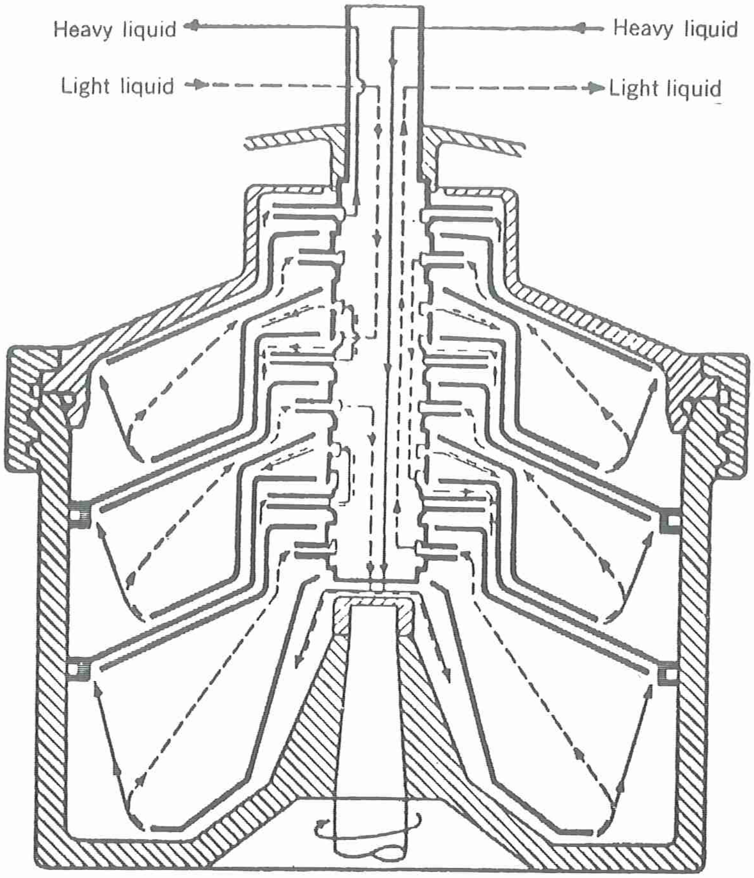


Figure 1.
Luwesta centrifugal extractor.

performance data have been published. Some limited plant-scale results have been reported, however, for chlormycetin production *(9)*, streptomycin production *(10)*, dephenolization of coke oven liquors *(11)*, lube oil refining with furfural *(12)*, and concentration of uranium *(13)*.

Except in centrifugal extractors, all extraction process equipment depends upon gravity settling, although mixing may be achieved by mechanical agitation. In the centrifugal extractor, high centrifugal force is used both for dispersion and generally countercurrent contacting of liquids and for repeated coalescence of the dispersed liquids. The fine dispersion which can be produced in a properly designed apparatus permits a very rapid extraction, and yet the dispersion is equally rapidly settled. Thus, one obtains a high capacity in a short time and in a small volume, even with a small density difference and with easily emulsified liquids.

Types of centrifugal extractors

The known centrifugal extractors consist of a drum on a rotating shaft. The Luwesta and DeLaval extractors are examples of units rotating on a vertical shaft, whereas horizontally rotating units are of the Podbielniak type.

(A) Luwesta extractor (Figure 1): The Luwesta extractor is characterized by a vertical flexible (operating above first critical speed) shaft, by annular weirs with or without skimmers used for liquid withdrawal according to usual centrifugal clarifier practice, and by the use of three completely discrete mechanical stages to mix and settle liquids in countercurrent flow. The rotating speed is generally high, in the range of 3,500 to 5,000 rev./min. Liquids enter at relatively low pressures at or near shaft center.

The Luwesta is analogous to three gravitational mixers plus three gravitational settling tanks with connections to make the system countercurrent. Perhaps be-

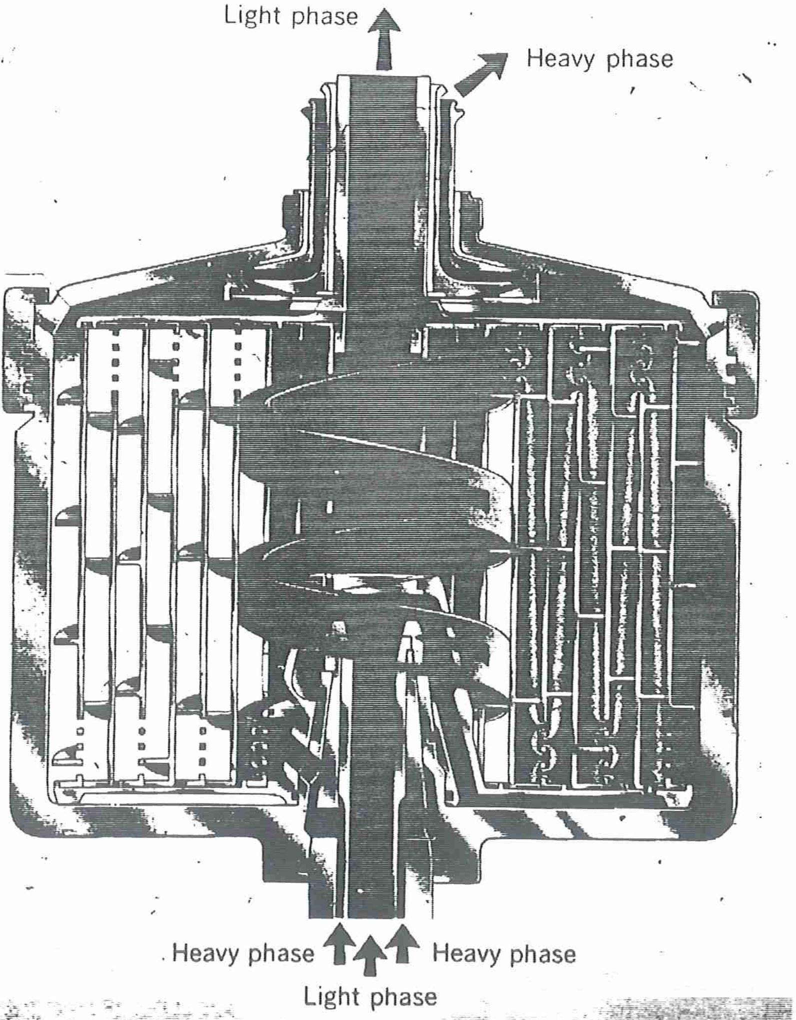


Figure 2.
DeLaval centrifugal extractor.

cause of limitations of its vertical flexible shaft suspension system, the throughput capacity of the Luwesta is limited to about 50 gal./min. Stage number is limited to three, times the machine's efficiency factor, which varies with the system.

A similar extractor to the Luwesta, but with only two mechanical stages, has recently been described in a Soviet journal (*14*).

(B) DeLaval extractor (Figure 2): This has been developed abroad and recently introduced in this country (*5, 8*). It may be considered a hybrid between the Podbielniak extractor and the Luwesta, in that it uses a vertical shaft, either mechanical seals or annular overflow weirs and skimmers, and other construction of conventional centrifugal clarifiers. It incorporates in the rotor a series of cylinders, perforated at ends similar to those used in the Podbielniak extractor described below, but with the addition of helical vanes between cylinders to cause prolonged helical countercurrent flow of the liquids. Like the Luwesta, it generally is provided with a constant speed drive. Data on applications and performance are not available.

(C) Podbielniak extractor (Figure 3): This is the most widely used type of centrifugal extractor. While not limited to a horizontal axis, many practical considerations have favored the adoption of a horizontal shaft, especially for long, high capacity rotors. Contrary to centrifugal clarifier practice, the shaft is made extremely rigid, and rotor speeds employed are low (down to 1,400 in 48-in. diameter commercial units), thus, permitting the use of mechanical shaft seals for all liquid entries and exits, and true hermetic operation.

What appeared to be copies of early Podbielniak antibiotic extractor models have been reported to be in use at a Czechoslovakian penicillin plant, and the Soviets have recently announced the availability of a horizontal centrifugal extractor (*15*). The patent literature contains examples of other variants of centrifugal extractors, but none are known to be operating.

Internal contacting elements

As noted previously, the major difference between the three types of extractors described is in the internals used. Essentially, the Podbielniak-type centrifugal extractor can be visualized as an extraction column, such as a perforated tray or other gravitational extractor, wrapped around a shaft and whirled to expose its contents to centrifugal force. Like the countless variations in tray design which have been invented and used for gravitational fractionation columns, many variants of contacting elements have been tried out, or proposed, for the centrifugal extractor. The original extractor used unperforated long spiral sheet passageways, succeeded by perforated spiral sheets, then for reasons of greater flexibility of design and ease of fabrication, with perforated concentric cylinders. Means for varying degree of perforations have been disclosed (*16*), and claims have been made for improved performance by particular arrangements and shapes of perforations (*17, 18*). Means have also been devised to permit externally changing the radial position of feed and solvent inlets (*19*), also to permit cleaning without dismantling (*6*).

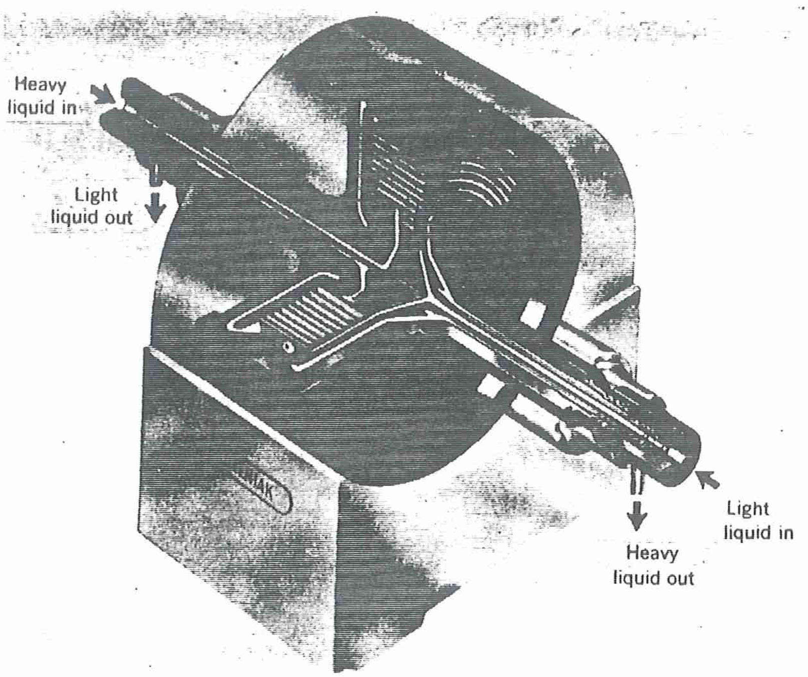


Figure 3. Podbielniak centrifugal extractor.

Pressure balance

In any annular weir and skimmer liquid exit seal extractor, the heavy and light liquids are entered into the rotor at or near shaft center, with little pressure required. The weir overflow or skim-off radius for the heavy liquid must be mechanically adjusted according to density difference of the two liquids; the overflow radius for the light liquid must similarly be adjusted mechanically both according to density difference and according to desired location of principal interface within the rotor. Effluents issue with little pressure.

Introduction and withdrawal of all liquids through mechanical shaft seals results in a hermetically tight rotor. The advantages of hermetic operation can be appreciated from a recent extraction performed between a compound melting at about 250°F and water. Thus, from this liquid-liquid extraction, above 250°F, the raffinate solidified as it left the extractor, and the extract vaporized.

Figures 4 and 5 illustrate the pressure balance over the centrifugal extractor for the cases of inner and outer position of the principal interface, giving typical illustrative required pressures. The bottom half of centrifugal extractor, Figure 4, may be visualized as corresponding to a gravity tower, but in which all fluids enter, leave, and have their pressures measured at the closed top of the tower.

It should be clear that to introduce the light liquid into the rotor, sufficient pressures must be provided, as illustrated, to balance

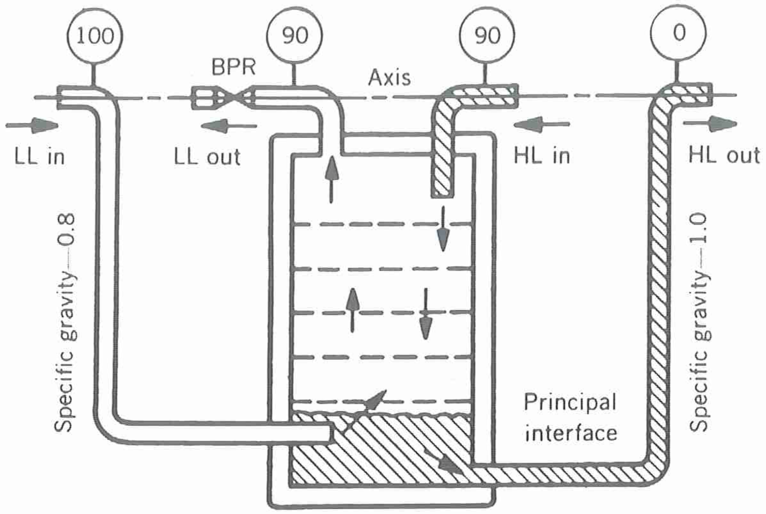


Figure 4. Pressure balance on centrifugal extractor; high back pressure, light liquid continuous, heavy liquid dispersed.

out the manometer formed by light liquid-in line and the heavy liquid-out line. In the centrifugal extractor, centrifugal head acting on a small radius compares with gravity head acting on tower height. Usually, heavy liquid out leaves the extractor at near atmospheric pressure, and light liquid in is pumped into extractor to overcome the centrifugal head caused by the difference in densities. Thus, the light liquid-in pressure is determined more by centrifugal head requirements than by liquid flow rate and interface position.

Principal interface

The location of the principal interface is important in determining optimum performance for capacity, entrainment-free effluents, and stage number efficiency. One general rule, applicable both to the centrifugal extractor and to most gravitational extractors, is to fill the extractor with the slower moving fluid, by suitable interface positioning. Obviously this rule loses its meaning when the solvent ratio is near one to one.

The principal interface position is determined by back-pressure control of light liquid-out flow, as illustrated in Figures 4 and 5. The above-cited rule for varying interface position plus experience with a given system are used in deciding where to carry the interface. Obviously, the process designer cannot freely replace a gravity extractor with a centrifugal extractor in his flow sheet without considering the difference in pressure requirements. Note that light liquid out usually leaves the centrifugal extractor under sufficient pressure to save pumping to the next process step, but the heavy liquid will leave at a pressure lower than that of the light liquid (which is opposite from the pressure relationship on the effluent liquids from a gravity column).

Use of temperature gradient

One of the limitations of the centrifugal extractor is the difficulty of providing or removing heat during the extraction, except through difference in the temperatures of the entering feed and solvent. An imposed temperature profile may be desirable to change mutual solubility or to generate an internal recycle.

The effect of an imposed temperature gradient on an extraction process is difficult to determine in pilot centrifugal extraction equipment, as the fluids assume the temperature of the metal. In commercial equipment, where the surface-to-volume ratio is quite low, a considerable temperature gradient can be maintained. Heat pickup through the seals and bearings is inconsequential except for laboratory equipment. For example, a 100 gal./min. extractor will generally draw less than 15 hp, thus, even if all of the mechanical energy is converted to process heat, the temperature will rise less than 1°F. Incorporation of an additional seal on the shaft does permit return of a recycle stream, such as in some furfural-lube oil installations or in aromatics recovery via sulfolane extraction. Return of a recycle stream does allow further control of the temperature gradient.

Handling of slurries and pastes

In general, a centrifugal extractor can handle process streams which contain solids that are nongranular, or are lighter than either of the process fluids, or are not much heavier than the heavy liquid so that continued flushing can occur. If the solids form a pasty slime, or if the heavy liquid is copious, a fair amount of solids can be tolerated since the solids remain free flowing.

Internal fluid phenomena

Exactly what goes on inside a whirling centrifugal extractor drum is difficult to ascertain. Designers and students of gravity extractors have the advantage of visually examining and photographing drop phenomena and flow patterns in glass or transparent plastic equipment under normal operating conditions. Centrifugal extractor drums which are subject to high stresses obviously cannot be constructed of such materials.

Pilot extractors have been made with a transparent plastic disc on one side, to operate at considerably lower than normal operating speeds. When illuminated with stroboscopic flashlight, and using suitably colored liquids, it is possible to follow the position of principal and minor interfaces, and some idea of flooding behavior, but not of dispersion phenomena. To operate at normal rotating speeds and still permit visibility, discs of glass or clear plastic, small enough to resist centrifugal stresses, may be mounted into metal sideplates, thus, permitting visual observation of operation under stroboscopic light. The first of this type of experimental extractor was built many years ago, with approximately 1-in. diameter quartz discs set in line, in many locations, on both sideplates, with stroboscopic light shining through the rotor.

Visual study of internal phenomena is believed to be extremely helpful in further development of the centrifugal extractor and to minimize pilot testing of new systems. Accordingly, the plastic side pilot extractor has recently been studied both with stroboscopic light and ultrahigh-speed motion picture photography. The results were not as informative as expected, principally because stroboscopic light shows only one view of any one spot, per 360° revolution of rotor, during which revolution, dispersion patterns change and blur. A rotor constructed with small plastic disc windows has been built to permit higher speeds and to take advantage of the new optical methods which have recently been developed to follow dispersion patterns over more than just one flash per revolution.

Process advantages

The major process advantages accruing to use of a centrifugal extractor derive from either its centrifugal separating force or its compactness. The first factor is of particular advantage when dealing with systems with a very small density difference, or high viscosities.

Compactness is important under many conditions. Expensive solvents need no longer be dismissed from possible application, as the total solvent inventory and consequent capital investment can be greatly reduced by use of a centrifugal extractor. Nor need corrosive feeds or solvents be excluded, as the compact centrifugal extractor can be readily fabricated in a variety of alloys without greatly increasing its cost.

The small inventory is also important when feedstocks are being changed frequently. This feature of instant raffinate is particularly appreciated in refining of a variety of lube oils with furfural or phenol. The short residence time was a major factor in the early commercialization of centrifugal extractors, as it permitted extraction of antibiotics in seconds under such conditions where the half-life of the active species was only a few minutes.

Both compactness and centrifugal separating force are important in the washing of catalysts from polymer solutions, thus, permitting more accurate control of the molecular weight of the reacting polymer leaving the reactor.

Present status

As chemical technology has advanced, more stringent requirements have been placed on separation and purification techniques. Distillation continues to be the most important unit operation, and is usually an integral part of an over-all extraction process as well. Nevertheless, extraction is a vital part of many manufacturing sequences, since advantage can be taken of differences in chemical affinities when differences in physical properties are insufficient to support an economic recovery or purification system based on distillation alone.

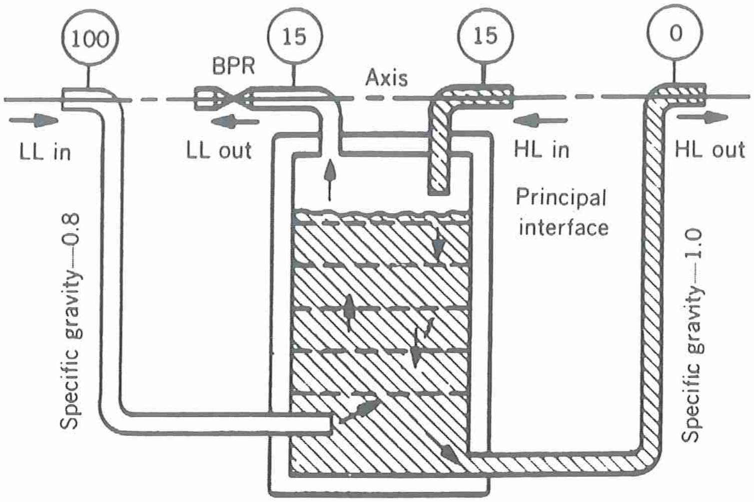


Figure 5. Pressure balance on centrifugal extractor; low back pressure, heavy liquid continuous, light liquid dispersed.

Although the centrifugal extractor, as a precision piece of equipment may cost more than a bare gravity tower of equivalent design duty, the former requires only a minimum of space, no ladders, scaffolding, elaborate insulation, piping, etc., and of course only a minute fraction of solvent and material inventory. Costs should, thus, be compared on the basis of a complete installation, including the cost of solvent inventory. Mechanical reliability as regards bearings and shaft seals, the only rubbing parts, has been improved to a point where their maintenance is now comparable with the maintenance of other chemical processing equipment.

The advent of the centrifugal extractor greatly extends the field of application of extraction processes. Centrifugal extractors, varying in size from 17 to 60 in. in diameter, and in capacity from 0.06 to 600 gal./min. combined throughput, are now becoming a more frequent item on the industrial scene. With machines designed to match industry's mounting needs, centrifugal extraction will likely continue to grow apace. #

LITERATURE CITED

1. Barson, N., and G. H. Beyer, *Chem. Eng. Progr.*, *49*, p. 243 (1953).
2. Jacobsen, F. M., and G. H. Beyer, *A.I.Ch.E. Journal*, *2*, p. 283 (1956).
3. Fox, J. M., *Hydrocarbon Process. Petrol. Refiner*, *42*, No. 9, p. 133 (1963).
4. Podbielniak, W. J., U. S. Patent 2,758,783 (1956); U. S. Patent 2,880,929 (1959).
5. Palmqvist, F. T. E., and S. Beskow, U. S. Patent 3,108,953 (1963).
6. Angelo, N. J., U. S. Patent 2,652,975 (1953).
7. Podbielniak Brochure P-100.
8. DeLaval Bulletin ABE 216.
9. Anderson, D. W., and E. F. Lau, *Chem. Eng. Progr.*, *51*, p. 507 (1955).
10. Bartels, C. R., and G. Kleiman, *Chem. Eng. Progr.*, *45*, p. 589 (1949).
11. Carbone, W. E., R. N. Hall, H. R. Kaiser, and G. C. Bazell, *Blast Furnace Steel Plant*, *46*, No. 5, p. 471-477 (1958).
12. Ishigaki, S., and H. R. Kaiser, *Petro/Chem. Engr.*, *35*, No. 5, p. 40 (1963).
13. Banks, N. K., *Mining Congr. J.*, *45*, No. 1, p. 44-47 (1959).
14. Lyskovtsov, I. B., *Khim. Machinostroenie*, *4* (1961).
15. *Soviet Export*, *32*, p. 38 (1964).
16. Podbielniak, W., U. S. Patent 3,116,246 (1963); U. S. Patent 3,132,100 (1964).
17. Redlich, O., U. S. Patent 2,619,280 (1952).
18. Claridge, E. L., Canadian Patent 679,104 (1964).
19. Doyle, C. M., and M. J. Costello, U. S. Patent 3,050,238 (1962).

Todd

Podbielniak

David B. Todd is manager of engineering for Podbielniak. He received his B.S. and M.S. degrees from Northwestern and his Ph.D. degree from Princeton. He also spent one year in the Netherlands under a Fulbright fellowship. Before joining Podbielniak in early 1963, he was employed at Shell Development Co., where he headed a process engineering group.

Walter J. Podbielniak, now a consultant engineer, is the author of numerous patents in the gas distillation and centrifugal extraction field. After undergraduate work at the University of Buffalo he received his Ph.D. in chemical engineering at the University of Michigan. In 1929 he founded Podbielniak, Inc., which became a division of Dresser Industries in 1960.

Reprinted from CHEMICAL ENGINEERING PROGRESS, August 1966

equipment
feature

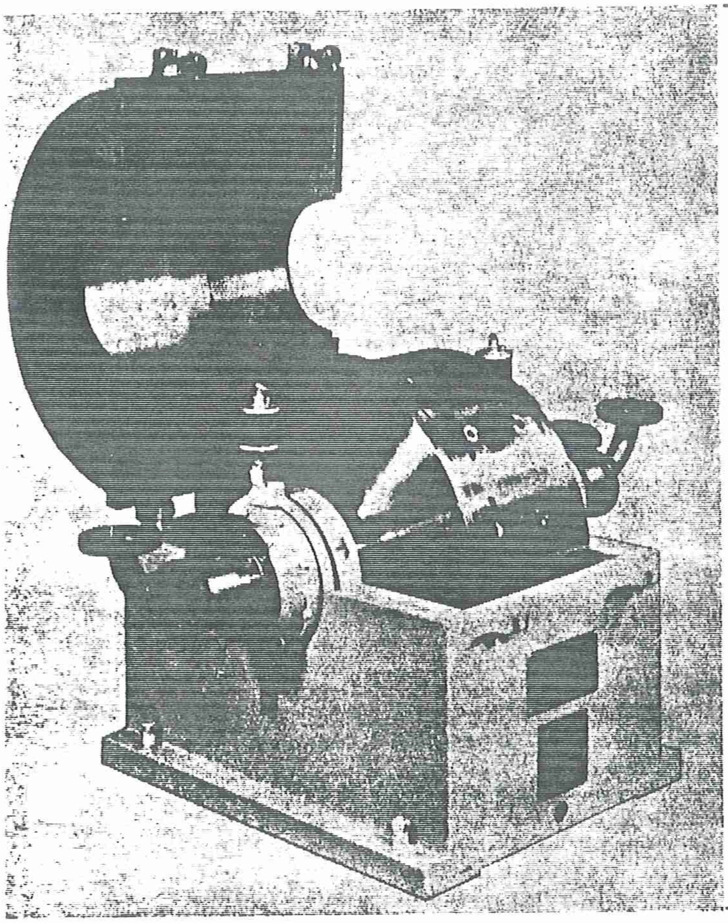


Figure 1. Podbielniak Model B-10 centrifugal extractor.

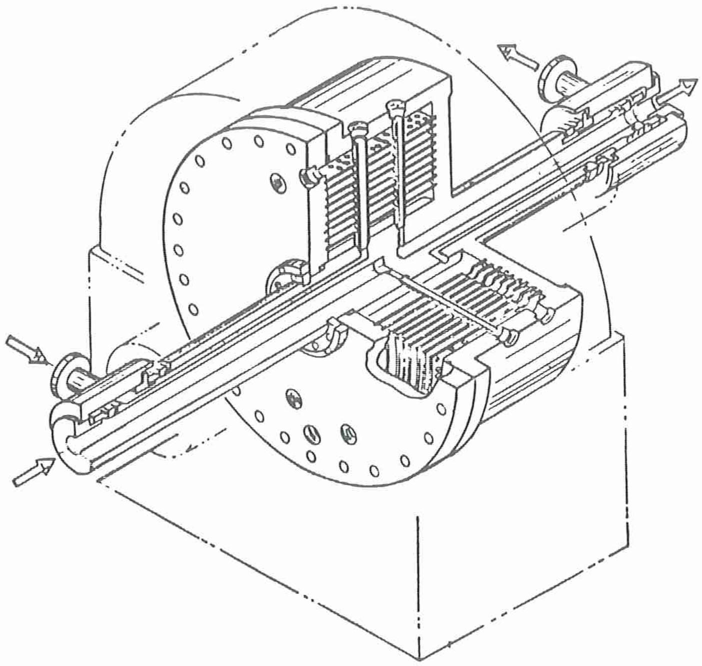


Figure 2. Cutaway view showing features of a centrifugal extractor.

Multiple functions in a centrifugal extractor

D. B. TODD *Podbielniak Div. Dresser Industries Franklin Park, Ill.*

A compact flexible extractor which can be readily converted allows for both optimum and maximum utilization. Providing means for changing feed location, seal arrangement and internal elements results in flexibility.

THE CENTRIFUGAL EXTRACTOR, IN addition to performing the clarification function common to other centrifuges, enjoys the additional process advantage of being able to perform an extraction function by repeated countercurrent contacting. The simple centrifugal extractor has the flexibility of varying rotor speed and, therefore, both mixing and settling forces, not common to its gravity tower counterpart. This flexibility is sufficient to allow a given extractor to operate well over

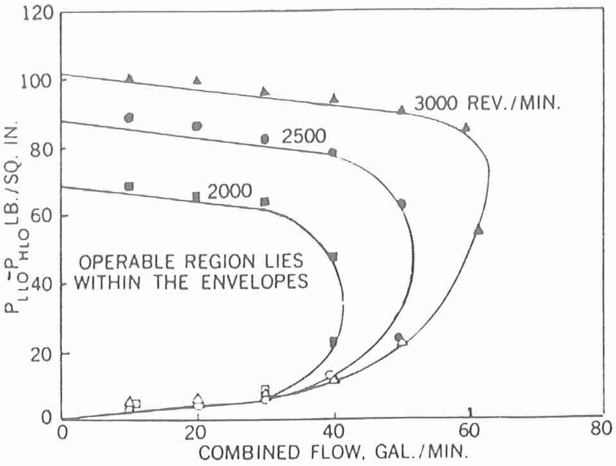


Figure 3. Capacity envelopes; water-kerosene at 1/1 phase ratio.

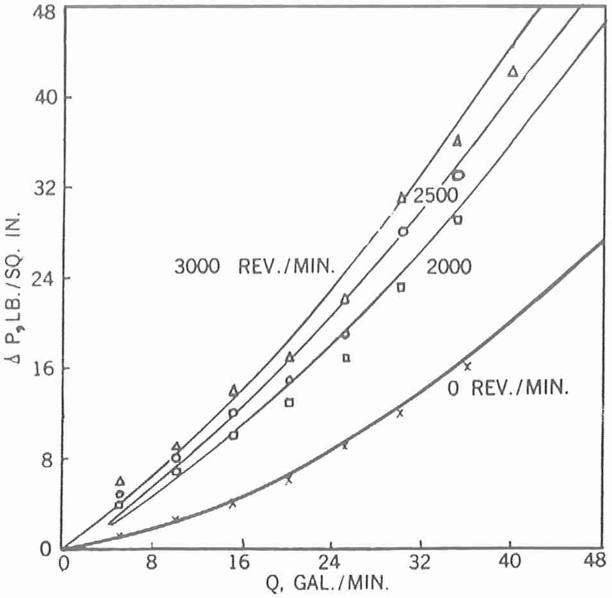


Figure 4. Pressure drop for water flow through a B-10 centrifugal extractor.

a wide range of flow rates for a specified process, although an extractor designed for one service may well not be the best one for a different service.

It can readily be seen that the usefulness of a centrifugal extractor can be enhanced if its versatility and flexibility can be increased (at low cost). What aspects of flexibility are required? Discussed herein are features involving:

1. flexibility in feed port location,
2. flexibility in seal arrangement,
3. accessibility for inspection and cleaning, and
4. ability to provide for rapid change of internal design.

The most prevalent type of centrifugal extractor consists of a horizontally rotating drum with balanced mechanical seals for entry of feed and solvent, and exit of raffinate and extract, through passageways in the rigid shaft. Within the rotating chamber, the heavier liquid is introduced near the shaft, and is hurled outward by centrifugal force. The lighter liquid is introduced nearer the periphery, and is displaced inward by the outmoving heavy liquid. In transit, the two fluids pass countercurrently through a series of mixing and calming zones. Traditionally, these zones are created by a series of perforated concentric cylinders, much as in a conventional perforated plate tower.

In the extraction function, it is desirable (a) to create a large amount of surface for mass transfer, (b) to maximize the gradient for mass transfer by limiting the degree of backmixing of both phases, and (c) to overcome stagnation within each phase. The large surface is created by making a fine dispersion, backmixing is limited by appropriate baffling, and the effects of stagnation can be offset by repeated coalescence and redispersion. Concurrently with the above, there is the additional requirement of achieving these goals while handling enough total flow in equipment of small enough size and cost to be economically justified.

The specific extractor described here is a Model B-10. This extractor, shown in Figures 1 and 2, has a 25-in. outer diameter and a 10-in. internal width, with an internal volume of about 14 gal. Operating speed is 3,000 rev./min. with a liquid of unit specific gravity. It is driven by a 7.5 hp motor through a fluid drive, permitting operation at any lower speed.

Feed port location

Before extract and raffinate leave the extractor it is desirable that each be as free as possible of contamination with the other phase, otherwise, such entrainment will appear as lower recovery or poorer extraction. How much entrainment does occur depends upon the stability of the dispersed droplets, and the relative probability of their being coalesced to a size large enough to separate before being swept out with the continuous phase. The lifetime of a drop is dependent upon transient phenomena occurring at its interface as well as upon the physical obstructions which might promote coalescence. In a given extractor, the depth of the clarification zone can be changed by changing the radial location of the feed and solvent ports.

The radial location is readily changed by removing the rim plugs over the six feed or six solvent inlets, and replacing the six distributors with different tubes. Thus, the rotor volume apportionment between clarification and contacting zones may be easily varied.

Seal arrangement

In a given extraction system, contact of one of the fluids with air may lead to formation of undesirable polymers or oxidized products. The balanced mechanical seals prevent air incursion. Since there are two fluid passageways in each end of the shaft, a small seal is used to separate the central liquid from the annular liquid, and a large seal to separate the annular liquid from air. At the seal surface, a minuscule amount of fluid passes from the high to the low pressure side, which keeps the seal surface lubricated. Normally, it is desirable to arrange the flows such that the higher pressure liquid exists inside the central passageway to minimize the pressure drop across each seal. Sometimes, however, process reasons exist which call for the low pressure liquid to be absolutely shielded from air, even at the seal. For example, a polymer solution being handled near the boiling point of

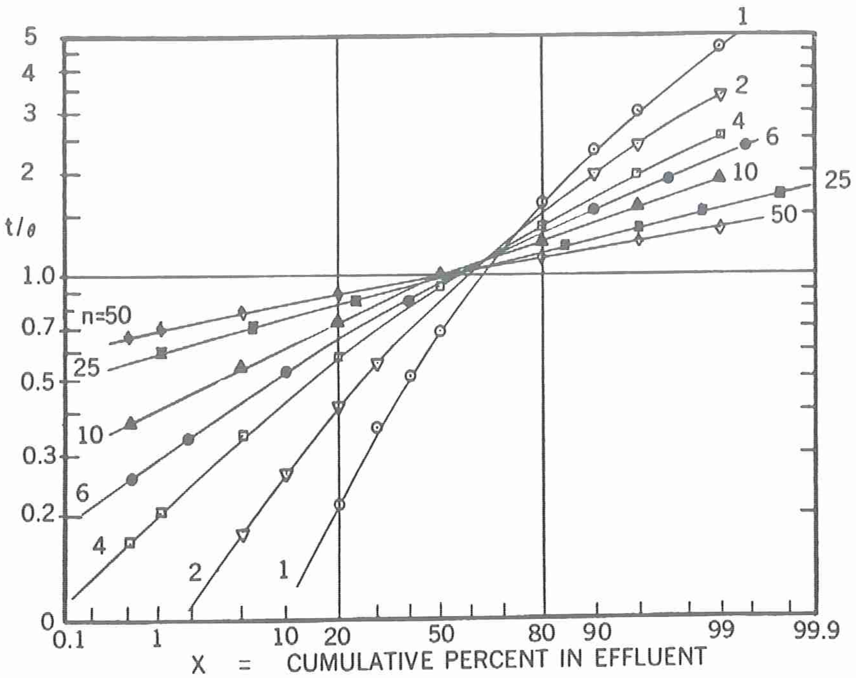


Figure 5. Cumulative distribution curves for equally sized well-mixed vessels.

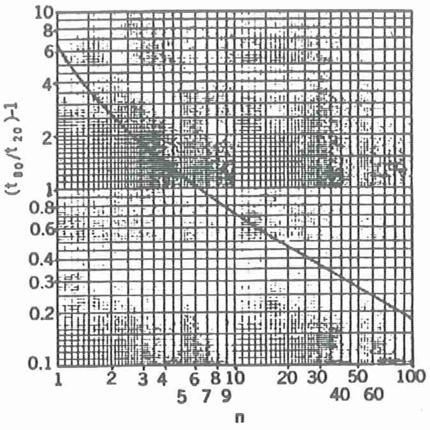


Figure 6. Slope of cumulative distribution curves.

the solvent might tend to vaporize at the large seal, leaving a residue of polymer which could interfere with the sealing action. Thus, it becomes desirable to have freedom of selection as to which fluid is introduced through which shaft passageway.

In the Model B-10, each of the four shaft passageways connects with a row of six holes through which the feed distributors and heavy liquid offtake are attached. Over each of the six radial passageways for the light liquid offtake, no distributor is needed, but a cavity exists radially outward. These cavities are filled by a plug, removal of which provides ports for inspection or cleaning. Thus, there are four sets of holes distributed axially in the shaft, each set containing six holes arranged radially. If it becomes desirable to change from any given flow pattern around the seals to a different one, this can be done by the switching of one set of six distributor tubes with any other set.

The small seal between the central and annular shaft passageways is also designed to be partially balanced irrespective of the pressure gradient across the seal. Consequently, the inlet and outlet connections may be freely interchanged without requiring a new seal because of the pressure reversal.

Accessibility

The high stresses developed in a centrifugal extractor dictate that the drum be of quite solid construction. The ability to have some access to the interior for inspection and cleaning is a desirable feature, especially if these tasks can be achieved without dismantling or removing the rotor from its base.

The cleanout plugs which fill the cavities radially over the connections in the shaft have already been mentioned. These do permit a boroscope inspection, or a cleanout by flushing with a lance.

In addition to these cleanout facilities, the rotor is provided with a removable end plate. Normally, this need never be removed, but should the need arise, removal can readily be accomplished at the installation site.

Internals

The ideal extractor will have physical features which maximize mass transfer and produce clarified effluents. The most satisfactory internal design for one process would only fortuitously be the best design for another. With the removable end plate and replaceable feed tubes, the internals can be completely removed from the rotor. Since the internals are made with great precision, it is now possible to supply replacement internals which are completely interchangeable. The interchange can be made in one day. Thus, the means are provided for the extractor to be readily converted to a modified process or to a completely different one.

The Model B-10 extractor described above occupies only 16 sq. ft. of floor space, and weighs only 2,500 lb. This small size is particularly convenient for indoor location. Should the originally intended process usage be markedly changed or abandoned, the extractor may be easily reinstalled in another location. Since the internals may also be readily changed, this inherent flexibility means that this capital investment need never remain idle.

This extractor is currently in use in processes such as recovery of phenol from waste water, recovery of sterols and tocopherols from soap stock, removal of catalyst residues from polyterpene resins, and various water washing operations.

Multifunction use

Certain combination processes can readily be accomplished in equipment having the features described above. If the effluents from two processing steps can be combined without detriment, one extractor can combine two functions. A frequent example of such dual functionality is caustic neutralization followed by water washing of the dissolved soap or salts. This is a common practice in caustic refining of low-gum vegetable oils to remove the free fatty acids simultaneously with water washing to remove the newly formed soap (*1*). The same procedure may be used in the synthesis of esters such as plasticizers to remove unreacted precursor acids.

Similarly, mineral acid catalysts may be removed from organic products, and phenolic polymerization inhibitors may be removed from monomers. Also, the product formed in one processing operation can sometimes be washed as it is separated from the original mixture, such as occurs in the continuous acidulation of vegetable oil soapstock to spring fatty acids (*2*).

Performance

Most commercial extraction processes involve multicomponent systems, and frequently are not easily identified as to number of theoretical stages. Instead the user is concerned with an adequate rejection or extraction of a given material over the range of flow rates likely to be encountered. Important to the effectiveness of extraction is the limitation of backmixing, or end-to-end mixing of the streams within the extractor.

Presented below are performance data on one of the Model B-10 centrifugal extractors, including capacity, pressure drop, and back-mixing, for a kerosene-water system.

Capacity

The pressure balances around a centrifugal extractor have been described previously (*3*). As throughput is increased, the centrifugal extractor will reach a flood point much as a gravity tower extractor will. The operating limits are conveniently described by determining the upper and lower limits of light liquid out (LLO) back pressure which cause excessive entrainment. Representative data for kerosene-water are shown in Figure 3 for three different speeds. To a first approximation, the maximum capacity is proportional to rotor speed. For this extractor:

$$Q = 0.02N \qquad (1)$$

where Q is given in gallons per minute and N in revolutions per minute. With a different arrangement of internals, it is possible to get either more or less capacity than that obtained above.

With some extraction systems, it is possible to obtain better performance by reducing the speed if the throughput is decreased considerably. By operating closer to flooding, the holdup and presumably the mass transfer area are increased. The lower G separating forces are offset by the greater residence time in the clarification zones.

Since any one bottleneck can be the limiting factor on capacity, it is not safe to extrapolate from one extractor to another without some confirming data. For example, with simple perforated cylinders as the contacting elements, doubling the number of perforations does not double the capacity.

Pressure drop

Although the pressures required for introduction of the feed and solvent are primarily determined by the centrifugal head arising from the density difference and interface location, frictional pressure drop also becomes important as flow rate is increased.

The centrifugal head requirement arises from the fact that the heavy liquid is conducted from the periphery back to the shaft, in essence establishing a centrifugal head of heavy liquid. The light liquid is introduced near the periphery, and must buck this head of heavy liquid. The centrifugal head pressure required to introduce the light liquid (P_{LLI}) above that existing on the heavy liquid effluent (P_{HLO}) is determined by the rotational speed (N), the radius of the light liquid inlet (R) and the difference in the specific gravity ($\Delta\rho$) between light liquid in and heavy liquid out:

$$P_{LLI} - P_{HLO} = 0.513 \times 10^{-6} R^2 N^2 \Delta\rho \qquad (2)$$

with R in inches, N in revolutions per minute, and pressure difference in pounds per square inch.

In addition to the frictional drop through the hardware, there is also an interacting contribution from speed and flow. Figure 4 shows the pressure (ΔP_o), data for water alone through a stationary Model B-10 extractor.

$$\Delta P_o = 0.055 Q^{1.6} \qquad (3)$$

where Q is given in gallons per minute.

The total pressure drop (ΔP_N) at operating speeds can be correlated by adding a term to the zero speed pressure drop:

$$\Delta P_N = \Delta P_o + 0.0002\, NQ \qquad (4)$$

For a first rough approximation, the frictional contribution for this extractor is about 0.5 to 1 (lb./sq. in)/(gal./min.). The pressure drop obviously will be a function of the mechanical construction.

Effectiveness characterization

In extraction, the age old battle is to find the optimum balance between the desirable dispersive forces which provide the large mass transfer area associated with fine droplets and the undesirable dispersive forces which decrease the

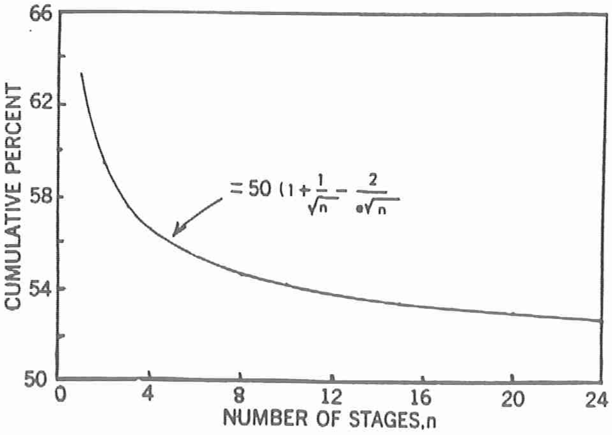


Figure 7. Cumulative percent effluent at unit residence time.

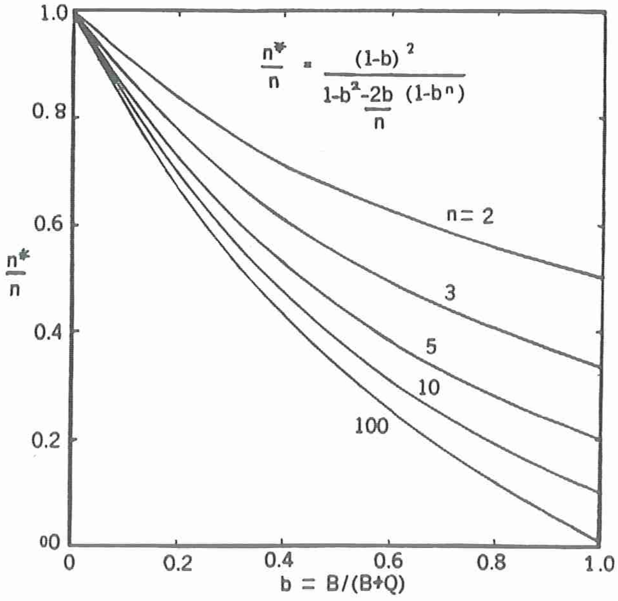


Figure 8. Effect of backmixing on number of stages.

4-way valve, the tracer was smoothly introduced into the heavy liquid inlet (HLI) line. Commencing with the valve turning, the total (or a large fraction at high rates) aqueous effluent was continuously collected in gallon cans, with the time of filling each can being noted. At the end of a run, each can was mixed well, and the $NaNO_3$ concentration determined by measuring the electrical conductivity. After correcting for the conductivity of the carrier water, the cumulative tracer could, thus, be readily determined as a function of time.

Representative data are shown in Figure 9. From the slopes of these curves, the number of equivalent mixing stages (n^*) was calculated, and is shown in Figure 10. Two curves have been drawn; one for equal flows of 5 gal./min. each of kerosene and water, and the other for combined flows at about 60% of the maximum capacity, still at a phase ratio of unity. The backmixing data confirm two frequently observed phenomena with centrifugal extractors; namely, that at low throughputs it is usually advantageous to reduce the speed of rotation, and that efficiency is usually higher at speeds when capacity is being pushed.

The inferred holdup and the inferred volumetric amount of backmixing are shown in Table 1. The amount of the dispersed phase flow has little if any effect on the backmixing of the continuous phase. Although the backmixing in this centrifugal extractor is low, it would become increasingly important as flow rate is reduced. The tracer tests were all performed with the principal interface at the heavy liquid inlet radial position. No measurement has as yet been made of the distribution of residence times of the dispersed phase.

Comparison with towers

Very few data have been published on the amount of backmixing occurring in commercial gravity extraction towers.

Strand, Olney and Ackerman (*9*) have reported Peclet numbers for a 42-in. diameter rotating disc contactor (RDC). Additional axial dispersion data for small diameter pilot size RDC's (2, 6, and 8 in.) have also been presented (*9-12*). These data generally indicate that axial mixing effects are severe at high rotation rates or at low continuous phase throughputs.

Table 1. Summary of residence time tests.

	FLOW, GAL./MIN.				
N, REV./MIN.	WATER	KEROSENE	n^*	HOLDUP, GAL.	BACKMIXING, GAL./MIN.
2,000	5	5	15.4	11.4	2.1
2,000	10	10	11.8	9.7	7.9
2,500	5	5	12.5	10.0	3.6
2,500	20	5	16.6	10.6	7.0
2,500	17	17	13.5	11.0	9.6
3,000	5	5	12.0	11.5	3.6
3,000	20	5	16.6	11.2	7.0
3,000	20	20	15.6	10.6	8.0

With an Oldshue-Rushton type column, Gutoff (*13*) reported excessive axial mixing for a single phase flow at a relatively low throughput. In the standard column, the backmixing was high enough to reduce the number of seven actual stages down to less than two effective stages. Even with screens between the stages in the stator plate openings, the maximum number of effective stages obtained was 3.0. Other physical modifications caused such an increase in backmixing to reduce the effective number of stages to only 1.1. Although Gutoff's superficial flow rate of only 2 cm./min. is well below that frequently encountered industrially, such low flow rates do occur when the solvent/feed ratio differs greatly from unity, and the low phase volume fluid is held as the continuous phase.

Longitudinal dispersion in small pulsed perforated plate columns has been extensively studied by Miyauchi and Oya (*14*). Their results indicate a decreasing Peclet number (increasing backmixing) as amplitude or frequency of pulsing is increased, or as throughput is decreased. The same conclusions can be inferred from the results of Gelperin and Neustroev (*15*).

Conclusions

1. Since there are diverse functions to be achieved in extraction processes, having a compact flexible extractor which can be readily converted allows for both optimum and maximum utilization.

2. Flexibility for process change or for other process use can be built into a centrifugal extractor by providing means for changing feed location, seal arrangement, and internal elements.

3. For a given centrifugal extractor, the maximum capacity is proportional to speed of rotation.

4. A simple experimental technique has been evolved for conducting and evaluating residence time distribution tests. From a log-probability plot of the cumulative distribution curves, the equivalent number of physical mixing stages can be deduced, along with the dispersed phase holdup and the degree of backmixing.

5. The backmixing in stirred column extractors is more sensitive to rotor speed than it is in centrifugal extractors. #

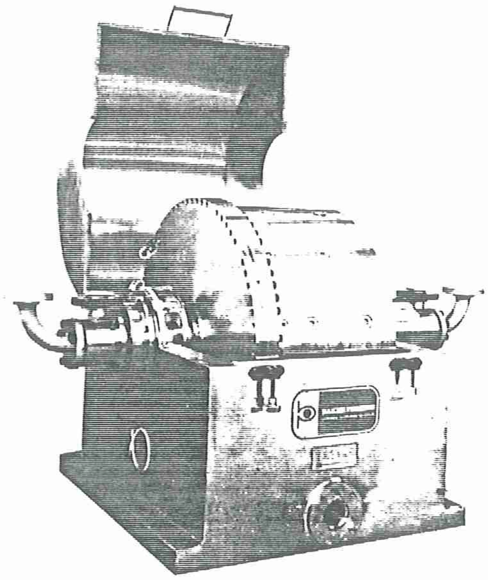
ACKNOWLEDGMENT

The development of the particular centrifugal extractor described herein has been made possible by the contributions of many fellow employees, particularly M. Bender, M. J. Costello, and N. Stanchiu. The assistance of M. F. Gannon, C. A. Hopper and H. R. Kaiser in obtaining and analyzing the data is especially appreciated.

LITERATURE CITED

1. Morren, J. E., S. G. Sourelis, and H. R. Kaiser, *J. Am. Oil Chem. Soc.*, *39*, No. 6, p. 278 (1962).
2. Todd, D. B., and J. E. Morren, *J. Am. Oil Chem. Soc.*, *42*, No. 4, p. 172A (1965).
3. Todd, D. B., and W. J. Podbielniak, *Chem. Engr. Progr.*, *61*, No. 5, p. 69 (May, 1965).
4. Olney, R. B., and R. S. Miller, Chapter on "Liquid-Liquid Extraction" in "Modern Chemical Engineering, Volume 1, Physical Operations," A. Acrivos, Ed., Reinhold Publishing Corp., New York (1963).
5. Sleicher, C. A., Jr., *A.I.Ch.E. Journal*, *5*, p. 145 (1959).
6. Miyauchi, T., U.S. Atomic Energy Comm., UCRL-3911 (August, 1957).
7. Buckler, E. J., and L. Breitman, *Can. J. Chem. Eng.*, *37*, p. 25 (February, 1959).
8. Van der Laan, E. T., *Chem. Eng. Sci.*, *7*, p. 187 (1958).
9. Strand, C. P., R. B. Olney, and G. H. Ackerman, *A.I.Ch.E. Journal*, *8*, p. 252 (1962).
10. Westerterp, K. R., and P. Landsman, *Chem. Eng. Sci.*, *17*, p. 363 (1962).
11. Westerterp, K. R., and W. H. Meyberg, *Chem. Eng. Sci.*, *17*, p. 373 (1962).
12. Kagan, S. Z., T. S. Volkova, and M. E. Aerov, *Khim. Prom.*, No. 12, p. 39 (1961).
13. Gutoff, E. B., *A.I.Ch.E. Journal*, *11*, p. 712 (1965).
14. Miyauchi, T., and H. Oya, *A.I.Ch.E. Journal*, *11*, p. 395 (1965).
15. Gelperin, N. I., and S. A. Neustroev, *Khim. Prom.*, No. 5, p. 360 (1964).

Todd



Improving Performance of Centrifugal Extractors

Factors can be varied to improve the performance of centrifugal extractors. But before attempting any adjustments, it is essential that operators have a clear picture of what goes on inside the machines.

DAVID B. TODD, Baker Perkins Inc.

Proper operation of centrifugal extractors often poses a problem, because the persons in charge do not fully understand how the~~se~~ machines work. But once this becomes clear, operating problems can be diagnosed and corrected—and the efficiency of the units increased—through the correct interpretation of a good set of pressure readings.

Pressure Balance

To understand the significance of the pressures around a centrifugal extractor and their effect on the relative amounts of material in each phase within the extractor, it is helpful to think of the inlet and outlet circuits for each phase as legs of manometers.

In the bottom half of a centrifugal extractor (Fig. 1), Channel A leads the heavy liquid out (*HLO*) of the rotor, after the liquid has been centrifuged outward toward the rim. The light-liquid-out (*LLO*) conduit is Channel B, through which the liquid exits after it has been displaced inward and collected at the shaft. Channel C is the light-liquid-in (*LLI*) passage to the rotor chamber—near the periphery but not outboard as far as *HLO*. Channel D is the heavy-liquid-in (*HLI*) conduit to the rotor chamber, somewhat outboard of the shaft but nearer the shaft than the rim. As the heavy liquid is centrifuged towards the rim, the light liquid is displaced inward towards the shaft, thus producing countercurrent flow between the two phases.

Space inboard of the heavy-liquid-in region (R_{HLI} in Fig. 2) is provided for clarification of the effluent light liquid (*LLO*). This area is called the light-liquid clarification zone. The space between R_{HLI} and R_{LLI} is the

Reprinted from CHEMICAL ENGINEERING, July 24, 1972. Copyright © 1972 by McGraw-Hill Inc.
1221 Avenue of the Americas, New York, N.Y. 10020

two-phase contact zone, while the space outboard of R_{LLI} is provided for clarification of the heavy liquid leaving the clarification zone.

Interface Location

A centrifugal extractor may be run with either phase (light or heavy) dispersed in the other, whichever is better for the specific process. The line of demarcation between the regions where the light and the heavy phases are respectively continuous is called the *principal interface*, whose location is controlled by imposing a back pressure on the *LLO* stream. Since the conduit leading to Channel A is filled with heavy liquid, the pressure at R_{HLO} is:

$$P_H = (0.513)(10^{-6})R^2N^2G_H \qquad (1)$$

where R = conduit length, in.; N = rpm.; and G_H = specific gravity of the heavy liquid. This equation represents one leg of the imaginary manometer (Fig. 3a).

The pressure at the rim outboard of R_{LLO} (the light-liquid outlet connection) must also be equal to the pressure calculated in Eq. 1. This can be considered as the second leg of the manometer. Both legs are equal as far back as the principal interface. Inboard of that level in the B leg, an additional pressure—just sufficient to offset the differences in specific gravity between the two phases—must be imposed at B (P_{LLO}). Imposing a larger pressure can cause the rotor to fill with a continuous phase of light liquid. Similarly, reduction of P_{LLO} to a low value will allow the rotor to fill with the heavy phase.

Inlet Pressure Requirements

The pressure needed to pump the light liquid into the rotor (P_{LLI}) can also be calculated on the basis of a similar manometer (Fig. 3b).

Nomenclature

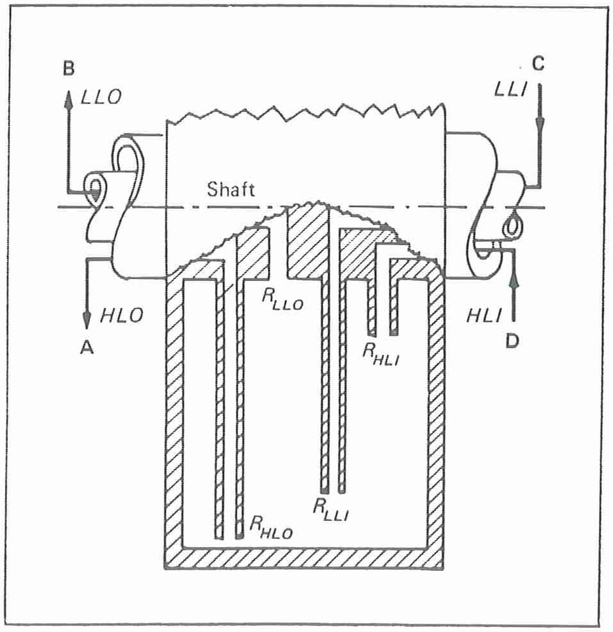
G	Specific gravity
H	Heavy phase
HLI	Heavy liquid in
HLO	Heavy liquid out
L	Light phase
LLI	Light liquid in
LLO	Light liquid out
N	Rotor speed, rpm.
R	Radius, measured from shaft center line, in.
P	Pressure, psig.
u	Pressure of *LLO* at which heavy-phase liquid entrains in *LLO*
v	Pressure of *LLO* at which light-phase liquid entrains in *HLO*
x	Pressure of *LLO* at which principal interface is at R_{HLI}
z	Pressure of *LLO* at which principal interface is at R_{LLI}

If the principal interface is inboard of R_{LLI}, the column of light liquid in the conduit must have a pressure imposed sufficient to overcome the differences in head caused by the differences in specific gravities ($G_{HLO} - G_{LLI} = \Delta G$). This is analogous to trying to force a liquid into a centrifugal pump near the periphery, where a centrifugal head must be bucked. This pressure can be calculated as before:

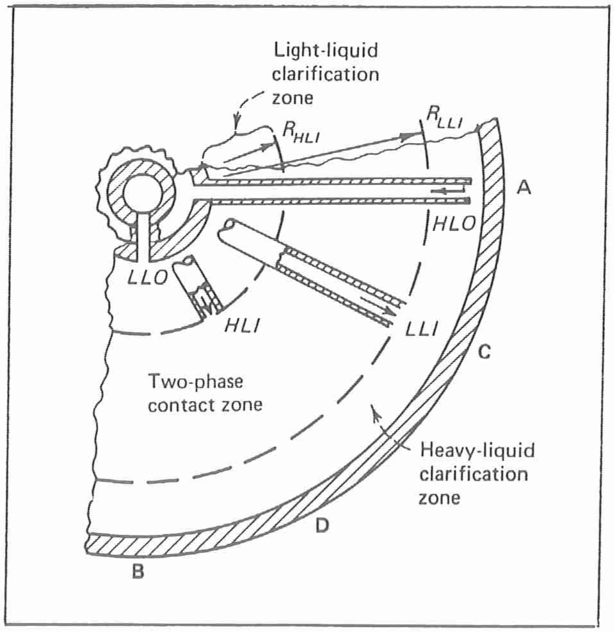
$$P_{LLI} - P_{HLO} = (0.513)(10^{-6})(R_{LLI})^2N^2(\Delta G) \qquad (2)$$

Note that P_{LLI} is insensitive to the location of the principal interface as long as this interface is inboard of R_{LLI}. As the interface is moved outboard—by imposition of more pressure at P_{LLO}—the length of the conduit of the less-dense phase is extended, and P_{LLI} increases exactly as P_{LLO} increases.

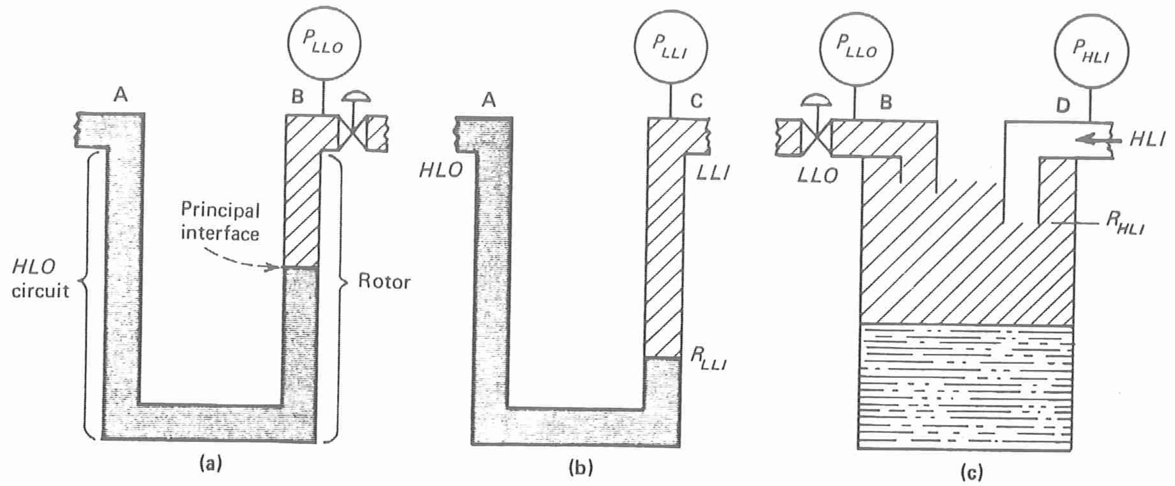
On a plot of P_{LLI} versus P_{LLO} (Fig. 4a), the horizontal



INLET AND OUTLET channels of centrifugal extractor—Fig. 1



ZONES of a centrifugal extractor—Fig. 2



MANOMETER ANALOGIES for determining (a) interface location, (b) pressure required to pump light liquid into the rotor, and (c) pressure needed to pump heavy liquid into the rotor_Fig. 3

line will normally break to a 45-deg. line at the point $P_{LLO} = z$, where the principal interface is precisely at the radial position of the light-liquid inlet ports (R_{LLI}). If the interface is moved too far outboard beyond this point, light liquid will start entraining out of the rotor with the heavy liquid (this is called rim flooding), at which point $P_{LLO} = v$.

The pressure required to pump the heavy liquid into the rotor (P_{HLI}) can be estimated again by the manometer analogy. The easiest manometer to visualize is the one with *LLO* as one of the legs. If the principal interface is outboard of R_{HLI}, the two legs of the manometer will be essentially equal, or $P_{HLI} = P_{LLO}$ (Fig. 3c).

If the interface is allowed to move inboard of R_{HLI}, P_{HLI} will no longer be sensitive to its location. Thus, a plot of P_{HLI} versus P_{LLO} will show a break from the 45-deg. slope to horizontal at the point $P_{LLO} = x$ (Fig. 4a) when the principal interface is at R_{HLI}.

Flooding Limits

If the interface is allowed to move too far inboard, heavy liquid will start entraining out with the light liquid (shaft flooding) at $P_{LLO} = u$ (Fig. 4a). Thus, at any flowrate, the shaft-flood point, *u*, and rimflood point, *v*, define the operable pressure range over which the centrifugal extractor may be operated.

If these points are plotted as a function of throughput, *Q*, a *flooding envelope* is defined (Fig. 4b), which depends upon rotor speed, mechanical design, and system properties. The practical operating flowrate will be somewhat less than the maximum.

Which Phase Continuous?

It may be obvious from the process which phase should be continuous. When not evident, it has generally been found expedient to disperse the major-phase flow into the minor one. Under these conditions, it is probably best to run with the interface in the vicinity of the radial entry point of the minor-phase flow. Noting the change from a horizontal to a 45-deg. slope of both inlet pressures against P_{LLO} (i.e., where $P_{LLO} = x$ and $P_{LLO} = z$) helps the operator define this location (Fig. 4a).

Frictional Pressure Drop

Because the pressure gages are located outside of the rotor chamber, they reflect line frictional drop, especially at high flowrates. A rough estimate of this internal friction drop can be obtained with completely immiscible systems from the pressure data (Fig. 4c).

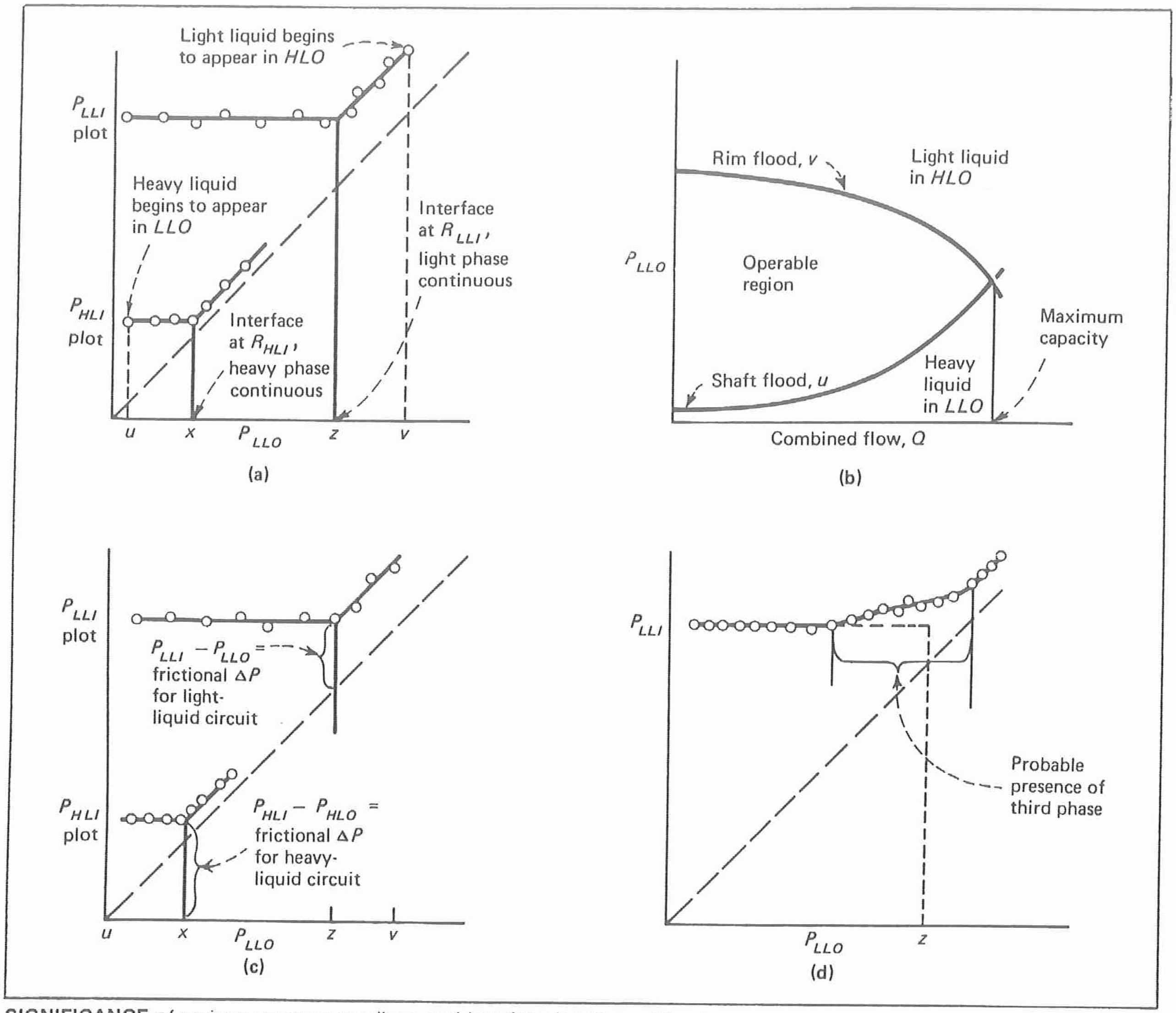
The difference between inlet and outlet light-liquid pressure ($P_{LLI} - P_{LLO}$) in the 45-deg. slope area is representative of all the frictional drop, because the centrifugal heads balance each other in that region. Similarly, the heavy-liquid-circuit frictional pressure drop can be obtained from the observed P_{HLI} at the horizontal portion of its plot versus P_{LLO}.

Any pressure imposed on the exiting heavy liquid disrupts the pressure balance and the location of the principal interface, unless the same pressure is also imposed on *LLO* (both P_{LLI} and P_{HLI} will automatically increase an equivalent amount). For correctness, the accompanying sketches are all based on subtracting P_{HLO} from all the other pressures.

Frequently, P_{HLO} will be so close to atmospheric pressure that it can be ignored. In many instances, operation of the centrifugal extractor is smoother if a small, constant back pressure is imposed on *HLO*.

Fuzzy Interface

The pressure balances just discussed assume clean-breaking phases. In many processes, however, an emulsion that is stable over some period of time may form. Its presence in the extractor can frequently be



SIGNIFICANCE of various pressure readings and interface locations—Fig. 4

detected by carefully noting the response of P_{LLI} to small changes in P_{LLO} (Fig. 4d). A portion of a P_{LLI} pressure curve with a slope that is neither 45 or 0 deg. indicates the presence of a third phase of intermediate density. An asymptotic curve thus denotes an interfacial region with a density gradient.

Two-Phase Feed

When the feed is a mixture of two phases, the apparent density will be that of the mixture, unless phase separation occurs in the feed conduit, in which instance the feed will appear as if the light phase is continuous. With an emulsion feed, the effective density may be higher than that of the clarified light phase, so it is possible for P_{LLO} to exceed P_{LLI}.

Improving Performance

The operator should explore minor variations in P_{LLO}, and perhaps in rotor speed, to see what change in performance occurs. The changes should be incremental. Because of the low holdup, the extractor will adjust to its new conditions within minutes.

A drastic change in P_{LLO} may give a false flooding indication, because the displacement of the amount held will momentarily increase the exiting flow of the displaced phase. Proper positioning of the interface can result in a 10 to 40% increase in extraction efficiency. Defining the flooding limits may allow a significant increase in capacity. ■

Meet the Author

David B. Todd is technical director of Chemical Machinery Div., Baker Perkins Inc., Saginaw, MI 48601, where he has technical responsibility for chemical-process equipment. Before, he was manager of engineering at Podbielniak, then part of Dresser Industries, Inc. He holds B.S. and M.S. degrees in chemical engineering from Northwestern University, and a Ph.D. from Princeton. He is experienced in equipment design, extraction, fluidization and polymer technology. A licensed professional engineer in Illinois and Michigan, he belongs to AIChE, ACS, American Oil Chemist's Assn. and other professional societies.