Liquid-liquid Extraction in Continuous-flow Agitated Extractors

A. W. Flynn and R. E. Treybal
New York University, New York

Extraction studies were made with the systems toluene water benzoic acid and kerosene water benzoic acid in two dimensionally similar cylindrical extractors, one 6 in. in diameter by 6 in. high, the other 12 in. in diameter by 12 in. high. Each extractor contained four radial baffles, of width equal to 16.7% of the extractor diameter. The extractors were agitated by six-bladed dimensionally similar turbine impellers, the diameters of which were in each case one third the diameter of the extractor.

When the extractors were full of liquid, i,e., in the absence of an air-liquid interface, the power required for a given impeller speed was independent of the rate of flow of liquid. However, such operation required more power than the equivalent open-tank agitator, and more baffling was required to produce fully turbulent flow.

Flow rates were varied from 0 to 4,000 lb./hr. and solvent-to-water mass ratios from 3:1 to 1:6. Extraction stage efficiencies were found to be appreciable even at zero agitator speed.

The additional stage efficiency produced by agitation was found to be a function of the power input per unit volume of liquid flowing. This relationship permitted the correlation of all extraction efficiency data for a given system at a constant solvent-to-water ratio over eight-fold range of power input. It was found possible to predict the performance of an extractor in continuous flow from batch-extraction measurements through the methods of MacMullin and Weber.

In the case of transfer of material between two immiscible liquid phases by agitation in a vessel, the relationship between rate of mass transfer and power required for agitation is of interest. The power consumption of agitators operating in a single liquid phase has been investigated by Rushton and coworkers (*12* and *13*) and Mack and his associates (*4, 5, 6*). They have presented methods of predicting the power consumption of an agitator based on size, speed, and liquid properties. Miller and Mann (*9*) studied the operation of agitators in various systems composed of two immiscible liquid phases. In all these studies there was no flow of liquid through the tank.

In the case of mass transfer, batch systems were studied by Mack and Marriner (*7*) and Hixson and Smith (*2*), and more recently continuous-flow systems were studied by Oldshue and Rushton (*10*), Hein (*1*), Karr and Scheibel (*3*), and Overcashier and his associates (*11*).

The present study was undertaken to determine the effect of agitator speed and power, vessel size, rate of flow, and phase ratio on the stage efficiency of agitated extractors and to confirm if possible the published procedures for predicting continuous-flow performance from batch-operation tests.

EQUIPMENT

Two dimensionally similar extraction vessels, as shown in Figure 1, were made for the tests. Constructed of commercially available glass tubing clamped between two flat metal plates, each vessel was provided with four radial baffles each 16.7% of the

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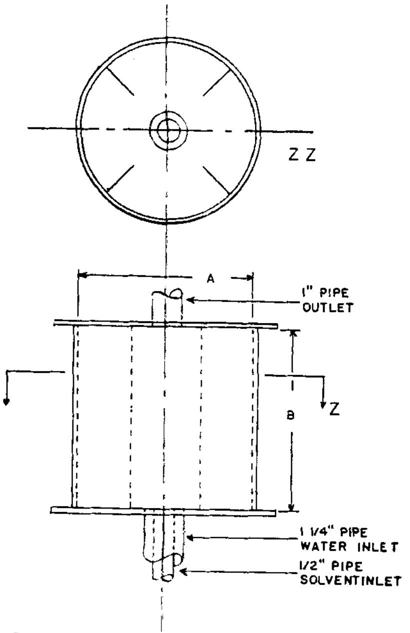
A. W. Flynn is at present with Nuclear Development Associates, White Plains, New York.



SIZE	A	B
6" EXTRACTOR	5 3/4"	6 1/8"
12" EXTRACTOR	11 1/8"	12 1/4"

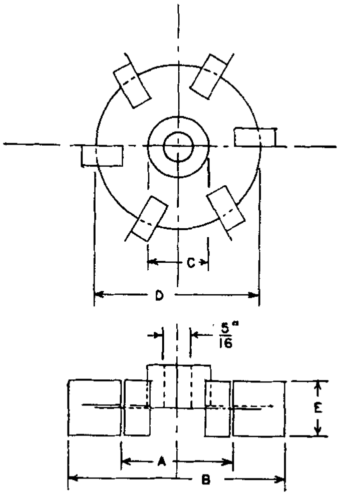
Fig. 1. Extraction vessel.

vessel diameter in width. The agitator impeller, the diameter of which was one third the vessel diameter, was driven by a shaft passing through the vertical outlet pipe and was located at the center of the vessel. The agitator impellers, as shown in Figure 2, were flat-bladed radial turbines constructed by welding six flat plates to a circular plate and stiffening the assembly by welding small reinforcing tabs to the backs of the radial blades.

The tests were performed in the equipment arrangement shown in Figure 3. All extraction studies were made with the top cover and wall baffles in place and with all entrapped air removed from the equipment. There were four parts to the investigation: (1) power measurements, (2) measurements of the phase ratio of the liquid in the vessel, (3) extraction measurements during continuous flow, and (4) batch-extraction measurements.

POWER MEASUREMENTS

The power input to the impellers was determined as a function of speed, phase ratio of liquids, and rate of flow. In all cases the baffles were in place and the liquid level was maintained in the "chimney" to prevent aeration of the liquid. The power measurements were taken by varying the speed of the agitator from 300 to 1,100 rev./min. for the 6-in. extractor and 100 to 350 rev./min. for the 12-in. and reading the tachometer and dynamometer. Power was determined electrically at low inputs where the dynamometer scale read less than 1 lb.



SIZE	A	B	C	D	E
2" IMPELLER	1	2	1/2	1 1/2	13/32
4" IMPELLER	2	4	1	3	13/16

Fig. 2. Agitator impeller.

The determination of small power values of agitator power input required a number of precautions to ensure accuracy. The drive motor was a small D.C. shunt motor with oil-lubricated bearings. Before each test the bearings were freshly oiled and the motor operated for between 45 and 60 min. to permit stabilizing the bearing and winding temperatures. The motor was operated at constant field current to eliminate that variable from consideration. The armature resistance was determined at the normal operating temperature and the armature input power was measured by meters of 0.5% accuracy. The motor-power output was determined by subtracting the armature resistance losses and the stray power losses from the armature power input. The no-load power input was checked frequently to detect any change in the motor or tachometer friction. The agitator power measurements are believed accurate to within ±5%.

Physical properties of the various liquids used in the tests are listed in Table 1.

The measured power data were used to calculate the power numbers and the Reynolds numbers. Figure 4 shows the individual power numbers for agitation of single liquids plotted against the corresponding Reynolds numbers for 16.7% baffles.* This type of plot was previously used by Rushton, Costich, and Everett(*13*). Whereas at high Reynolds numbers the water, toluene, and sucrose-solution power numbers lie on a single straight line for both sizes of vessel, those for kerosene do not.

TABLE 1.—PHYSICAL PROPERTIES OF LIQUIDS USED

Liquid	Density, lb./cu.ft.	Viscosity, centipoises
Water	62.2	0.95
Toluene	54.0	0.59
Kerosene	51.2	2.52
Sucrose-water solution	69.3	3.38
Motor oil	55.8	87.5

The width of the baffles in the 6-in. extractor was then increased to 25% of the vessel diameter and the tests were repeated. In Figure 5 the impeller power numbers are plotted against Reynolds number for the 25% baffles. Identical curves were obtained for the sucrose solution, kerosene, and water, but not for the more viscous motor oil. When, however, the tests were repeated after the liquid level in the vessel was lowered until a small air space was formed under the top cover, all the liquids provided a single curve, as shown by the dotted curve in Figure 5.

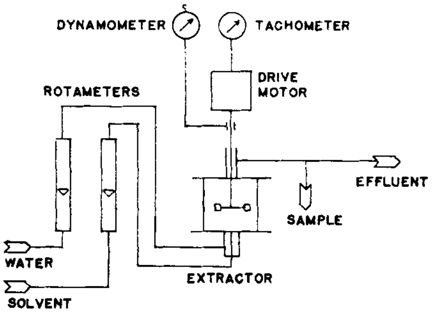


Fig. 3. Flow diagram.

Rushton(*12*) has shown that for reliable prediction of the performance of a large-scale agitator from measurements on a small-scale one it is necessary that both operate fully baffled. In addition when a given agitator is operated fully baffled the power number is a function solely of the Reynolds number. For open vessels operated with an air-liquid interface, a fully baffled condition (or "standard" baffling) is provided by four radial baffles each one-twelfth the tank diameter in width. From Figures 4 and 5 it can be seen that neither arrangement of agitator and baffles meets the condition of full baffling when vessels are operated without an air-liquid interface. This condition has not been reported in the literature as existing

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*Complete tabular data are obtainable as document 4653 from the Photoduplication Service, American Documentation Institute, Library of Congress, Washington 25, D.C., for $1.25 for photoprints or microfilm.

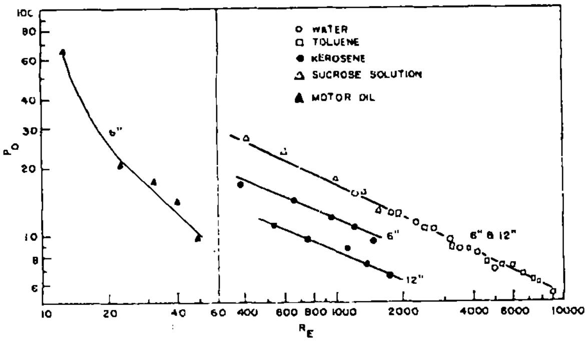

Fig. 4. Power number correlation; 6- and 12-in. extractors, 16.7% baffles.

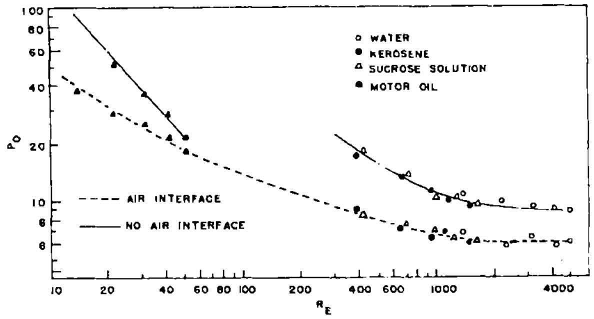

Fig. 5. Power number correlation; 6-in. extractor, 25% baffles.

in large commercial-size vessels although the baffles in such vessels are much narrower than the ones employed in these tests. The need for more baffling to provide for a fully baffled condition when the agitators are operated full of liquid may be a characteristic which is limited to small-scale vessels. The present conclusion is that more work must be done on these matters, and they are being explored further.

The effect of rate of liquid flow through the equipment on the power required for a given agitator speed was too small to be detected.

The power required for agitation of the single liquids water, toluene, or kerosene is so nearly the same for each that reliable measurements of the effect of mixing two insoluble phases could not be made. For the toluene-water mixtures encountered in extraction measurements, the power requirement was calculated from Figure 4 by use of the average viscosities and densities of Miller and Mann (*10*). Because the behavior of kerosene was different from water and did not produce a common power-number–Reynolds-number curve with 16.7% baffles, it was felt that the Miller and Mann interpolation should not be used with mixtures of kerosene and water. Experimentally the power required for agitating these mixtures could scarcely be distinguished from that for water alone, and the latter is used in reporting the subsequent extraction data.

PHASE RATIO OF VESSEL CONTENTS

A comparison was made between the ratio of the insoluble liquids in the feed to the extraction vessel during continuous operation and the ratio in the vessel contents at steady state. This was done by operating at fixed rate of flow of liquids, phase ratio, and impeller speed until steady state was reached. The liquid flow and the impeller rotation were then suddenly stopped, the liquids in the vessel allowed to separate into two layers, and their relative volumes measured. Tests were made only on the 12-in. vessel because its larger dimensions permitted greater accuracy in the measurements. The results are presented in Table 2. The holdup of organic liquid was very small at zero agitator speed. With a rotating agitator, at all but the slowest speeds the ratio of the phases in the vessel approached that in the feed mixture.

CONTINUOUS EXTRACTION

The equipment in Figure 3 was operated by extracting the benzoic acid from the organic phase into

TABLE 2.—ORGANIC LIQUID HOLD-UP

12-in. Extractor, 16.7% Baffles

Flow rate, lb./hr.		Solvent holdup, % of feed composition			
		Impeller speed, rev./min.			
Solvent	Water	0	150	250	350
		Solvent = toluene			
250	250	3.92	86.0	100	100
500	500	4.75	97.0	100	100
1,000	1,000	9.60	100	100	100
1,500	1,500	12.5	100	100	100
2,000	2,000	14.4	100	100	100
250	750	8.90	100	100	100
500	1,500	11.0	100	100	100
750	2,250	12.2	100	100	100
285	1,715	15.4	100	100	100
400	2,400	19.3	100	100	100
750	250	50.0	100	100	100
1,500	500	90.0	100	100	100
		Solvent = kerosene			
250	250	3.38	88.0	98.0	98.0
500	500	5.16	79.5	91.2	98.5
1,000	1,000	6.75	77.8	88.0	98.0
1,500	1,500	7.60	77.6	88.0	98.0
2,000	2,000	8.50	77.6	88.0	98.0
250	750	8.50	95.0	98.0	100
500	1,500	8.50	87.9	100	100
750	2,250	8.50	81.0	95.0	100
285	1,715	8.50	90.0	100	100
400	2,400	11.80	82.5	100	100
750	250	4.50	97.0	100	100
1,500	500	6.80	96.0	100	100

pure water. The effluent stream was sampled at the extractor outlet by collection of the mixed effluent in a 500-ml. graduate. The sample was allowed to separate into two layers. Samples for analysis were immediately pipetted from the settled liquids and titrated with fresh sodium hydroxide solution, phenolphthalein being used as an indicator. Alcohol was used with the organic layer to produce a single-phase mixture. In many of the runs the concentration change in the organic liquid was so small that it was difficult to check material balances exactly. In a typical run, for example, 9.25 cu.ft./hr. of a toluene–benzoic acid solution containing 0.0148 lb. mole of acid/cu.ft. was contacted with 9.01 cu.ft. of water/hr., acid free. The effluent water analyzed 0.000900 lb. mole of acid/cu.ft. The effluent toluene analyzed 0.0141 lb. mole of acid/cu.ft., which represents a discrepancy of 11% between the acid transferred from the toluene and that transferred to the water. On the other hand, a perfect material balance requires the toluene effluent to analyze 0.01402, which agrees with the observed value within the error of analysis. Similar agreement of observed and calculated effluent analysis of the organic liquid was obtained in 91% of the runs made. The remaining rates were not included in the final correlations. Mass transfer rates were calculated on the basis of water analyses owing to the larger percentage change in composition for that liquid.

A total of 149 extraction measurements was made on these systems, covering a range of from 500 to 4000 lb./hr. total flow, solvent-to-water ratios from 3:1 to 1:6, and dimension changes of 2:1.* Stage efficiencies were expressed as Murphree efficiencies based on water concentrations,

$$E = 100(C_{W2} - C_{W1})/(C_{We} - C_{W1}) \quad (1)$$

and since in every case C_{W1} equaled zero, this expression becomes simply $E = 100\ C_{W2}/C_{We}$. The stage efficiency E_0 at zero agitator speed was found to be appreciable even at low flow rates. This efficiency without agitation varied from 44.5% on the 6-in. extractor passing 250 lb./hr. of water and 250 lb./hr. of toluene to 92% for the same extractor passing 250 lb./hr. of water and 750 lb./hr. of toluene. Similar but lower values were found with kerosene. It was necessary to correct for this large initial efficiency in order to study the effect of agitation. This was done through the expression

$$E_A = 100(E - E_0)/(100 - E_0) \quad (2)$$

where E_A is the stage efficiency attributable to agitation, E is the over-all stage efficiency at any agitator speed, and E_0 that at zero agitator speed for the same liquid flow rate.

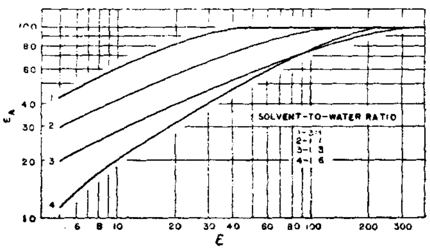

Fig. 7. Extraction efficiency summary, toluene water benzoic acid.

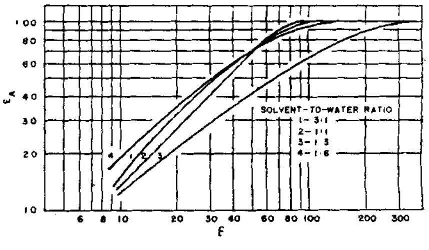

Fig. 8. Extraction efficiency summary, kerosene water benzoic acid.

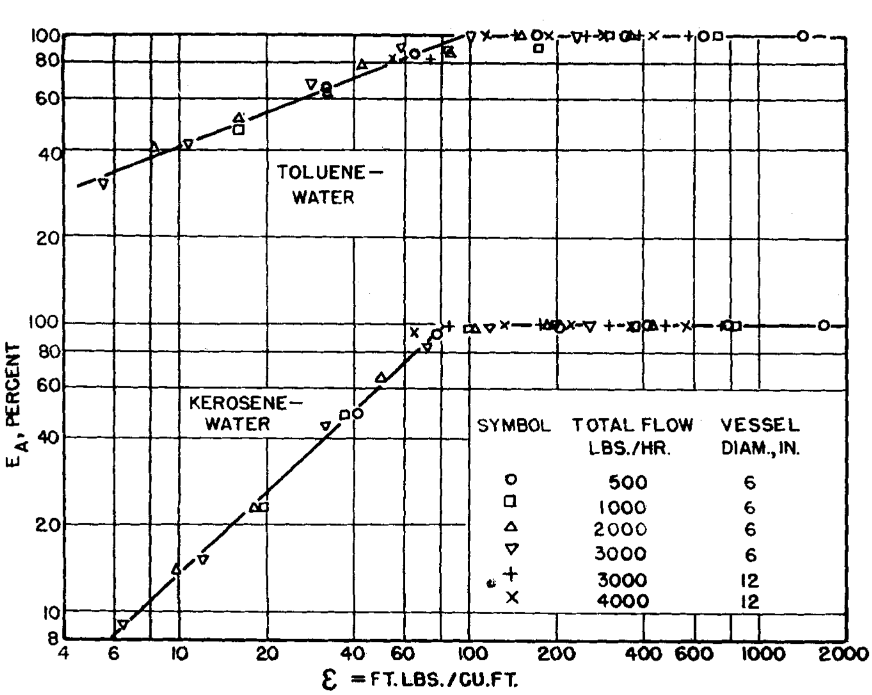

Fig. 6. Extraction of benzoic acid for equal rates of flow of organic and aqueous liquids.

The efficiency attributable to agitation for each system and each phase ratio when plotted against the agitator energy per unit volume of liquid flowing through the vessel, $\varepsilon = Pt/V$, gave rise to a single curve for all flow rates and for both vessel sizes. Figure 6, for example, shows the curves for the one-to-one flow ratio which result for the two systems. The remaining data are summarized in Figures 7 and 8. This provided the best correlation among the data and suggests a means of predicting the performance of larger scale equipment which is dimensionally similar.

In general greater solvent-to-water ratios increased the extraction efficiency, in opposition to the findings of Miller and Mann (9) and Hixson and Smith (2), who worked only with batch systems. The difference may be explained by the fact that in continuous operation the feed streams were directed into the impeller, which then did not have to lift the heavy liquid from the bottom of the vessel in order to mix it into the dispersion. In a batch operation the agitator must lift the heavy liquid, and when the proportion of heavy liquid is small, any of it which remains on the bottom of the vessel unmixed with the dispersion lowers the measured agitator efficiency. This is true whether the

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*See footnote, p. 325.

efficiency is indicated by the mixing index of Miller and Mann(*9*) or the extraction rate as described by Hixson and Smith (*2*).

The continuous-flow extractor operates with complete and uniform mixing once the agitator speed exceeds a certain low value. Where the solvent-to-water ratio is high, the work required to produce the amount of interfacial area required for a given efficiency is less than when the ratio is low. This may be due to the fact that the degree of initial dispersion in the vessel is more a function of solvent than of water flow rate. This is confirmed by a study of many photographs made of the vessel in operation. When the initial dispersion is good, the initial E is high and only a small amount of work is required from the agitator to raise E to 100%. This small increase in E, however, represents a change in E_A between zero and 100%. Therefore, with large solvent-to-water ratio, less agitator power is required to increase E_A from zero to 100% than for the case of small ratios, where E is low. The exceptional behavior of the 6:1 solvent-to-water ratio mixtures in both systems may be related to the beginnings of emulsification which were observed with this ratio.

It was not found possible to produce a good correlation between the data for the two systems studied.

BATCH EXTRACTION

The effect of time on the extraction efficiency in batch operation was determined. Measured quantities of solvent containing benzoic acid and water were placed in the vessel without mixing. The agitator was then started and run for a measured length of time at constant speed. The time at which the clear layers of water and solvent disappeared was recorded as the time at which thorough agitation was attained. At the end of the agitation time the agitator was stopped and the mixture allowed to separate into two liquid layers. Samples of the solvent and water layers were then analyzed. All batch measurements were made in the 12-in. extractor because it held a sufficient quantity of material to ensure accuracy.

Over-all extraction efficiencies were calculated and used to predict the extraction efficiency of the equipment in continuous flow through the use of the method of MacMullin and Weber(*8*). As an example of the results for a flow of 2000 lb./hr. of toluene and 2000 lb./hr. of water the E_A measured in continuous flow was 79% while that calculated from batch data was 80%. For the kerosene system at the same flow rates the measured value was 92.4% and the calculated value was 88%. The good agreement between the calculated over-all efficiencies and the efficiency attributable to agitation indicates that it is possible to predict the extraction efficiency in continuous flow from batch measurements if all the extraction in both cases is attributable to agitation.

SUMMARY

1. It is tentatively concluded that an agitated vessel without an air-to-liquid interface may not behave as a fully baffled vessel even though it contains baffles considerably larger than those required by the same vessel operating with an air-to-liquid interface.
2. The extraction efficiency attributable to agitation is a function of the agitator energy input per unit volume of total liquid flowing. This relationship may be used to predict the performance of dimensionally similar equipment at a constant solvent-to-water ratio. This has been verified over a range of 8 to 1 in flow rate, 2 to 1 in dimensions, and 100 to 1 in agitator power input, with the same solvents and solute.
3. It is possible to predict the performance of the extraction equipment in continuous flow from batch measurements through the method of MacMullin and Weber provided that all extraction is attributable to agitation.

NOTATION

C = concentration of benzoic acid, lb.mole/cu.ft.
d = diameter of impeller, ft.
E = extraction stage efficiency, %
E_A = extraction stage efficiency attributable to agitation, %
g_c = conversion factor, 32.2 ft. (lb. force)/(sec.^{2}) (lb. mass)
n = impeller speed, rev./sec.
P = agitator power, ft.lb./sec.
P_0 = power number = $Pg_c/\rho n^3 d^5$, dimensionless
Q = flow rate, cu.ft./sec.
R_E = Reynolds number = $d^2 n\rho/\mu$, dimensionless
t = holdup time, sec.
V = extractor volume, cu.ft.

Greek Letters

ε = specific energy, ft.lb./cu.ft.
μ = viscosity, lb./ft.sec.
ρ = density, lb./cu.ft.

Subscripts

K = kerosene
S = organic solvent
T = toluene
W = water
e = equilibrium condition
0 = zero impeller speed
1 = initial condition
2 = final condition

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