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PROGRESS REPORT OF PROCESS TEST SECTION

FOR AUGUST, 1956

A. D. Ryon
K. O. Johnsson

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CHEMICAL TECHNOLOGY DIVISION

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A. D. Ryon and K. O. Johnsson

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DYNAMIC KINETIC STUDIES

R. R. Wiethaup

Abstract

A study has been made of the rates of extraction and stripping with a Dapex-type organic and a high-uranium, high-vanadium liquor. The purpose of the investigation was to confirm design data for this particular system, as well as to examine the effect of impeller speed and flow rate on extraction efficiency under the extreme concentration conditions.

The tests were conducted in the six-inch mixer cell equipped with a three-inch diameter, four flat-blade turbine-type impeller, as described previously.[1/] [2/] The organic extractant consisted of di(2-ethylhexyl)phosphoric acid and tributyl phosphate in kerosene. The aqueous phase was a synthetic leach liquor. These solutions are described in Table 1.

Extraction Isotherm

In order to evaluate the performance of the mixer, it is necessary to know the equilibrium distribution of the uranium between the two phases. This distribution was determined by equilibrating the head solutions at 24 to 25°C in phase ratios ranging from 1:10 to 10:1 so as to cover a wide concentration range.

The experimental data are shown in Fig. 1 where they are plotted as rectangles which define the 95% confidence intervals of the data. The data are correlated within the confidence intervals by the equation:

$$\ln x = 3.0 \ln \left(\frac{\sqrt[3]{y}}{12.38-y} \right) + 2.7$$

where x and y = concentration of uranium in grams per liter in the aqueous and organic phases, respectively.

1/ Progress Report of Pilot Plant Section, p. 16, ORNL-2026, March 1955.

2/ Progress Report of Engineering Section, ORNL CF-56-4-79, January 1956.

The constants 3.0 and 2.7 were determined by applying the method of least squares.

TABLE 1. DESCRIPTION OF FEED SOLUTIONS

Aqueous Feed

Density at 25°C	1.090 g/ml
pH	0.32

Concentrations, g/liter

U	5.3 ± 2% (95% C.I.)		
V (total)	4.8	SO_4	94
V (V)	3.4		
Fe (III)	0.52	PO_4	3.0
Al	2.7	Cl	0.98
Mo	0.02	F	0.28

Organic

Density at 25°C	0.813 g/ml
Di(2-ethylhexyl)phosphoric acid	0.156 molar
Tributylphosphate	30 g/liter
Diluent	kerosene

Procedure

The procedure for the extraction tests was essentially the same as described previously. A series of consecutive runs was made at the same flow rate and continuous phase, with varying impeller speeds. Samples were taken by collecting the mixed effluent in a beaker fitted with a drain cock in the bottom. The mixture was allowed to coalesce (about 1 minute) and the separate phases filtered to remove entrainment.*

The loaded organic phase was stripped three times by batchwise contacting with aqueous sodium carbonate and acidified by contacting with dilute sulfuric acid. This acidified organic was then used in the next run.

*Samples were also taken directly from the mixture in the cell through fritted glass and Teflon diaphragms so as to minimize the extraction that occurred as a result of sampling. This work is not complete and will be presented in a future report.

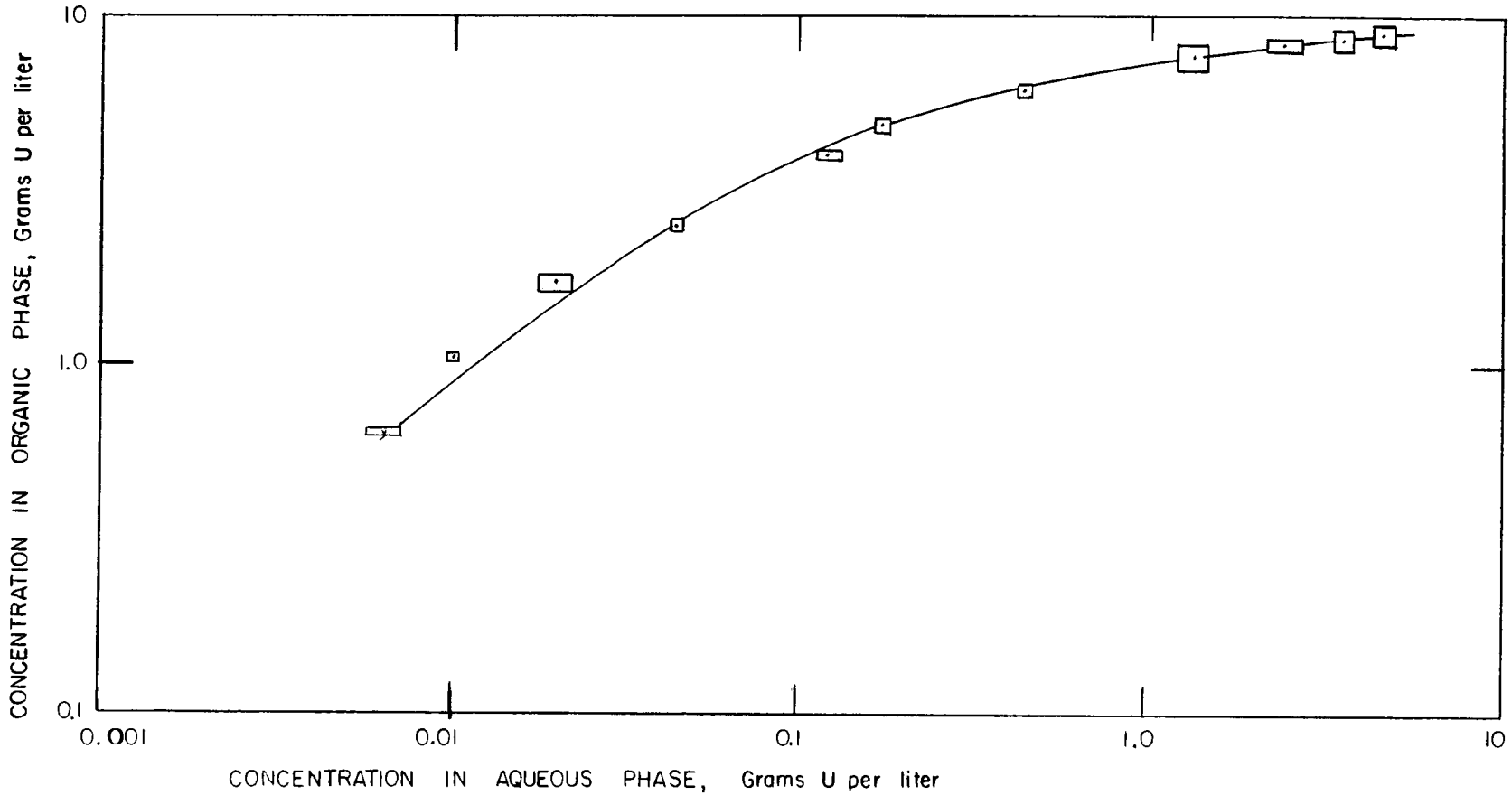

FIGURE 1 – EQUILIBRIUM DISTRIBUTION OF URANIUM AT 25°C

Results

The operating conditions are given in Table 2. The detailed results are presented in Table 3. In Fig. 2 the results are presented as the Murphree stage efficiency, E_M, based on the aqueous phase compositions.[1] This figure shows that E_M increased with impeller speed up to a point, then leveled off. Some extraction occurred when the impeller was not turning due to both the mixing caused by flow through the cell and to mixing during collection of the effluent sample. The choice of the continuous phase appears to have practically no effect upon E_M so single curves have been drawn for the present graph.

Fig. 3 is a cross-plot of Fig. 2 and shows that E_M decreased as the flow rate increased (i.e. as the nominal residence time[2] decreased) even at the highest speeds tested. However, Murphree efficiencies of 90% or better can be attained by operating at a nominal residence time of 1.5 minutes and impeller speeds of 500 rpm or more in a 6-inch mixer. This speed is equal to 0.012 horsepower per gallon of mixer contents.

In Fig. 4, the results are correlated by the method of Flynn and Treybal.[3] Here, a correction is made for the extraction that takes place when the impeller is not turning. The corrected efficiency, E_A, is computed by:

$$E_A = \frac{E_M - E_O}{100 - E_O} \times 100$$

where: E_O = Murphree efficiency at 0 rpm.

E_A is a function of the energy expended per volume of fluid processed, termed the specific energy.

1/ $E_M = \frac{C_1 - C_2}{C_1 - C^*} \times 100$

C_1 = concentration of uranium in aqueous feed.
C_2 = concentration of uranium in aqueous effluent.
C^* = concentration of uranium in aqueous effluent that would be in equilibrium with the organic effluent.

2/ Defined as the volume of fluid in the vessel divided by the total volumetric throughput.

3/ Flynn, A.W., and R. E. Treybal, "Liquid-Liquid Extraction in Continuous-flow Agitated Extractors," AIChE Journal, Vol. 1, No. 3, p 324-28 (Sept. 1955).

TABLE 2. OPERATING CONDITIONS

Total flow rate, gpm	0.5, 1.0, 2.0
Flow ratio	1:1
Temperature °C	24 to 25
Average density of effluents, g/ml	
Aqueous	1.085
Organic	0.823
Volume of fluid in mixer, gal	0.734

$$\text{Specific energy} = \frac{P\Theta}{V}$$

P = power input to impeller
Θ = nominal residence time
V = volume of fluid in mixer.

Rather than measure the impeller power, it was calculated using the equation:

$$P = \frac{K}{g} N^3 D^5$$

K = 4.5 for the impeller used
g = gravitational constant
N = impeller speed
D = impeller diameter

Fig. 4 shows that, at each residence time, E_A increased as the specific energy was increased up to 1000-2000 ft lb/ft^3. Above this, E_A was essentially constant.

In the work done by Flynn and Treybal, the lines at different residence times coincided. In previous work on the Amex system[1] the data for a range of residence times were

1. Progress Report of Engineering Section, ORNL-CF-56-5-134, February 1956.

TABLE 3. SUMMARY OF RESULTS

Run	Total Flow in (gpm)	Nominal Holdup Time (min)	Flow Ratio (aq/org)	Continuous Phase	Conc., grams U/liter Aq in	Aq out	Org in	Impeller Speed (rpm)	Power Per Mixer Volume (hp/1000 gal)	Specific Energy (ft lb/ft³)	Extraction Efficiency (%) E_M	E_A
541	0.49	1.50	1.05	aqueous	5.3	4.8	0	0	0	0	9.4	0
542	"	"	"	"	"	3.7	"	210	0.86	320	30	30
543	"	"	"	"	"	0.72	"	420	6.9	2,600	89	88
544	"	"	"	"	"	0.45	"	630	23	8,700	95	95
545	"	"	"	"	"	0.40	"	840	55	20,000	96	96
546	"	"	"	"	"	0.38	"	950	80	30,000	96	96
551	0.50	1.46	0.89	organic	"	0.66	0.149	420	6.9	2.500	90	88
552	"	"	"	"	"	0.53	"	630	23	8,300	92	92
553	"	"	"	"	"	0.42	"	840	55	19,800	95	94
554	"	"	"	"	"	0.40	"	950	80	28,000	95	94
555	0.99	0.74	1.01	aqueous	5.3	4.9	0.050	0	0	0	7.5	0
556	"	"	"	"	"	3.9	"	210	0.86	160	26	20
557	"	"	"	"	"	1.12	"	420	6.9	1,270	81	79
558	"	"	"	"	"	0.86	"	630	23	4,300	86	85
559	"	"	"	"	"	0.66	"	840	55	10,200	90	89
560	"	"	"	"	"	0.62	"	950	80	14,700	91	90
564	0.89	0.82	1.04	organic	"	1.11	0.079	420	6.9	1,410	81	79
565	"	"	"	"	"	0.72	"	630	23	4,800	89	88
566	"	"	"	"	"	0.59	"	840	55	11,300	92	91
567	"	"	"	"	"	0.60	"	950	80	16,300	92	91
577	1.93	0.38	1.00	aqueous	5.3	5.3	0.026	0	0	0	0	0
573	1.96	0.37	1.04	"	"	3.2	0.073	300	2.5	230	40	40
574	"	"	"	"	"	1.88	0.073	400	6.0	550	65	65
575	"	"	"	"	"	1.31	"	600	20	1,860	77	77
578	1.93	0.38	1.00	"	"	1.39	0.026	800	48	4,500	75	75
579	"	"	"	"	"	1.34	"	900	68	6,400	76	76
576	"	"	"	organic	"	3.2	"	300	2.5	230	40	40
580	1.99	0.37	0.95	"	"	2.3	"	400	6.0	540	57	57
581	"	"	"	"	"	1.27	"	600	20	1,820	78	78
582	"	"	"	"	"	0.96	"	800	48	4,300	84	84
583	"	"	"	"	"	0.93	"	900	68	6,200	84	84

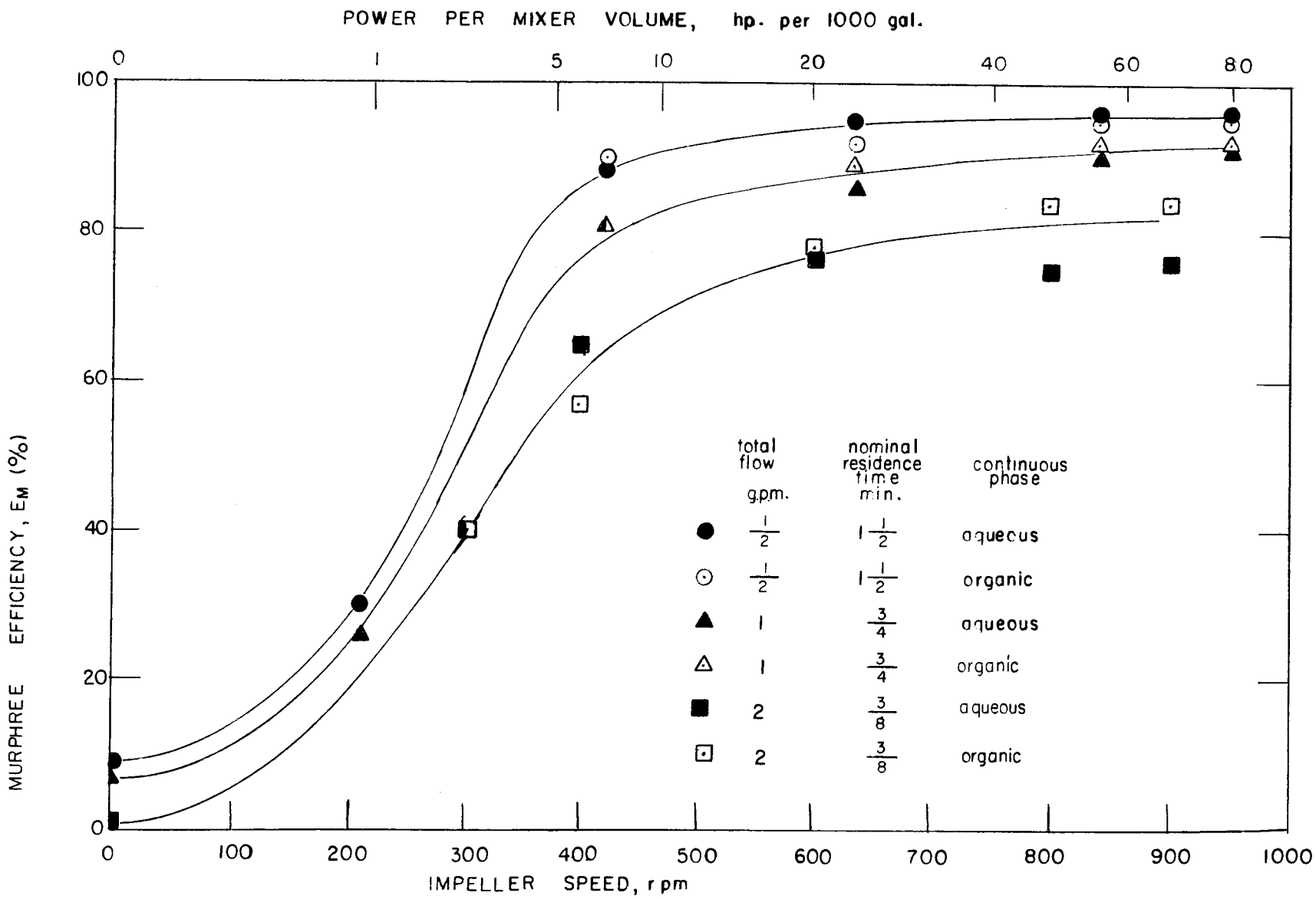

FIGURE 2 – PERFORMANCE OF A BAFFLED MIXER

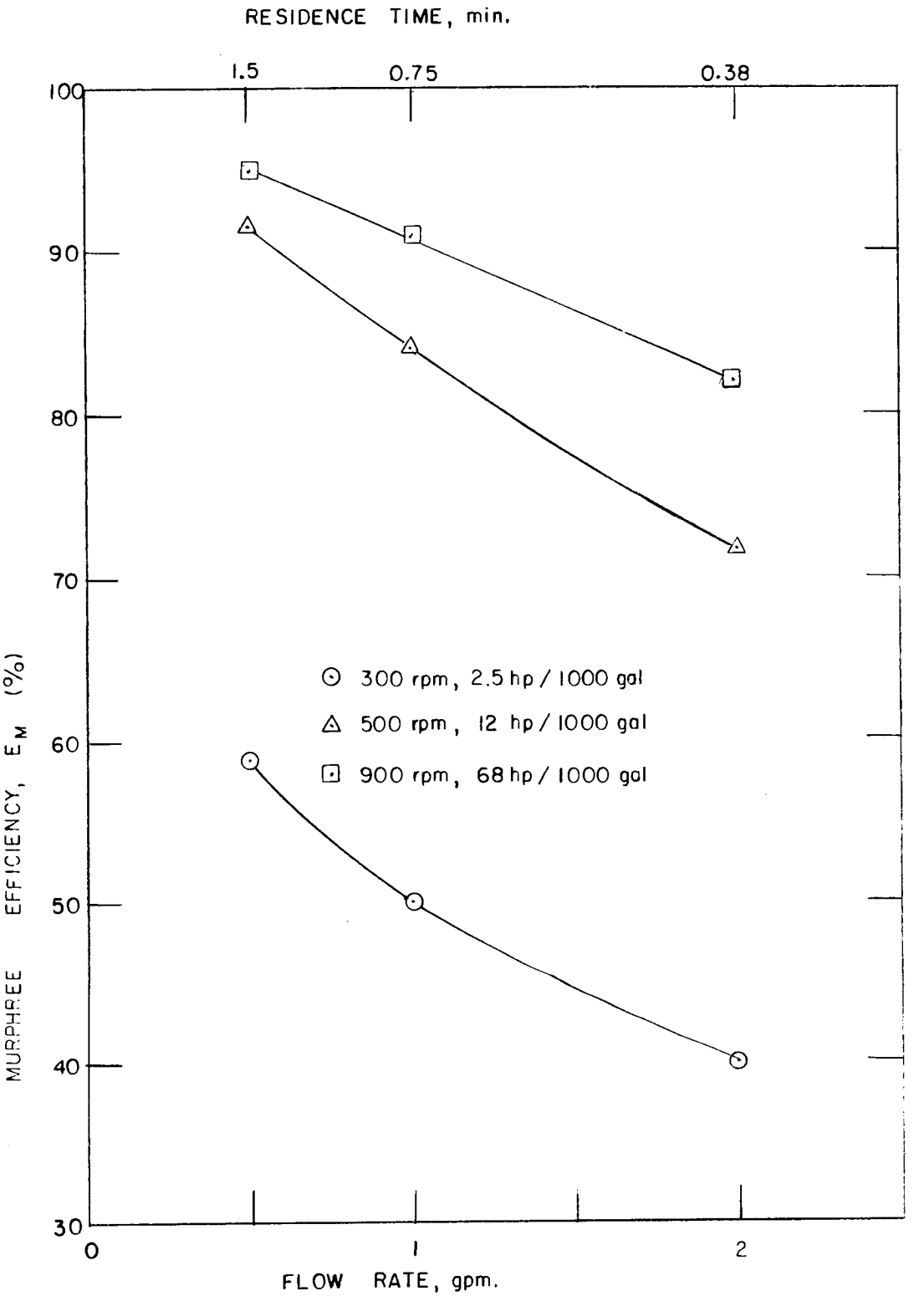

FIGURE 3 – PERFORMANCE OF A BAFFLED MIXER

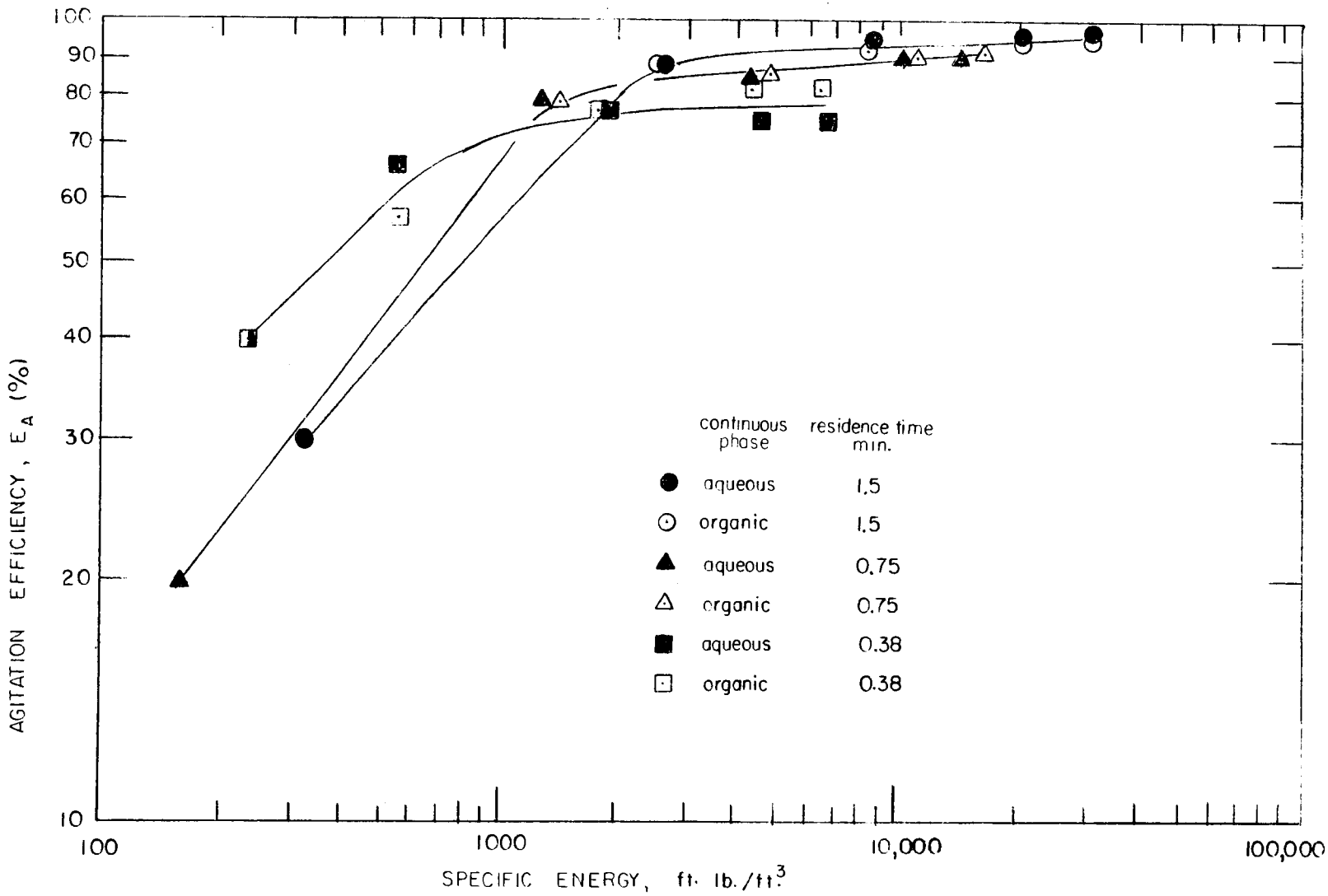

FIGURE 4 — FLYNN-TREYBAL CORRELATION

correlated by a single line. However, this was not the case in the present work. At the same specific energy, E_A decreased as residence time decreased. The correlation of Flynn and Treybal does not provide an adequate correlation of the data.

Future Work

The rate of extraction from the same liquor but with a lower uranium concentration will be studied so as to simulate the performance of the second mixer in a countercurrent extractor.

The rate of stripping will be studied in a manner analogous to the extraction studies.