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Operation with Three Liquid Phases in a Staged Liquid-Liquid Contactor

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ABSTRACT

Operation with three liquid phases was demonstrated in a staged liquid-liquid contactor. The possibility that three liquid phases could be handled in a liquid-liquid contactor normally used with two liquid phases was initially established using a laboratory batch test. The three liquid phases were obtained using a thorium flow sheet having high concentrations of both acid and thorium. To analyze the batch test, the concept of a dimensionless dispersion number for use with two liquid phases was extended so that it could be applied to three liquid phases.

Based on the batch tests, continuous flow tests were run in a staged liquid-liquid contactor used for solvent extraction. A critical factor in the success of these tests was determining the position of the liquid-liquid interface in the contactor. Thus, a contactor was used which allows the position of the liquid-liquid interface to be adjusted. Actual three-phase operation was demonstrated using a 4-cm annular centrifugal contactor, albeit with a somewhat greater (3-4 vol. %) aqueous-phase contamination of the organic exit stream than normal (<1 vol. %).

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INTRODUCTION

Under certain conditions, three liquid phases can occur during solvent extraction. For example, a second organic phase has been observed when testing flowsheets for separating thorium from its fission products and from other heavy metals (1,2,3). This second organic phase occurs when both the thorium concentration and the acid concentration are high. This second organic phase is a complex of thorium nitrate and tri-n-butyl phosphate which has low solubility in the organic solvent, n-dodecane.(3). High thorium concentrations are desired because they increase the decontamination of thorium from fission products. Some concern has been expressed that operation with three liquid phases might be unstable (4) or, at least, cause serious difficulties (5). In spite of this, successful thorium separations with a second organic phase present have been reported both for pulsed columns (6) and for mixer-settlers (7). However, it has been stated by others that "the ANL centrifugal contactor probably will not work with a third phase" (8).

The focus of our experimental tests was to show that the ANL centrifugal contactor (9) can indeed work with three phases, and work quite well. This claim was demonstrated in both single-stage and multistage tests by showing that the two organic phases flow out one exit of a contactor stage while the aqueous phase flows out the other exit. In addition, it was required that the phases flow easily from one stage to the next. As a part of these tests, a design criterion for the centrifugal settling zone was proposed and checked. In addition, it was shown that contactor throughput can be estimated from a simple batch test.

Although the work presented here is for a mixer-settler with a centrifugal separating zone (9,10,11), the results should also apply to other centrifugal contactors, to mixer-settlers with a gravity settling zone (12,13,14), and to extraction columns which have a series of discrete separating zones such as the Hanson-Kaye design (15). For units where the position of the dispersion band in the separating zone can not be changed, e.g., the Robatel centrifugal contactor (11), the theory presented here still applies. However, it must be applied to a unit before the unit is built which can represent a major inconvenience.

BATCH SEPARATION TESTS

Definition of Dispersion Number for Three Phases

It has been shown that, for two liquid phases, the separating zone volume can be related to the stage throughput by the dimensionless dispersion number N_{Di} (16). For batch separation tests, the two phases are dispersed and the time-to-break t_B is measured as the two phases separate under gravity g. If the dispersion container has a uniform cross section with a height ΔZ, the dispersion number is given by

$$N_{Di} = \frac{1}{t_B} \sqrt{\frac{\Delta Z}{g}}$$

For three dispersed phases, there are two dispersion numbers: $N_{Di,M}$, which uses the time-to-break between the two more dense phases $t_{B,M}$, and $N_{Di,L}$, which uses the time-to-break between the two less dense phases $t_{B,L}$. As before, ΔZ is the total height of all the liquid phases and the separating zone has a cross section that does not vary with vertical height.

In some cases a dispersion does not form at one interface, but tiny droplets move to the interface and change the interface position as they reach the interface and coalesce there. For these cases, the position of the interface is measured as a function of time. The time-to-break, then, becomes the time for the interface to be 1% or less from its final position or, in some cases, to be only 5% from its final position. The dispersion number will vary, depending on which criterion is chosen for measuring the time-to-break. It is expected that, if the design criterion chosen is to have the interface only x% from its final position, the use of the resulting dispersion number to design a continuous-flow unit would give a unit that has x% carry over of the other phase or phases in a given separating zone effluent.

Thorium Tests

Using thorium nitrate, nitric acid, water, tri-n-butyl phosphate (TBP), and, in all but one case, n-dodecane (nDD), seven multiphase systems were prepared. The initial concentrations and volume of each phase are given in Table 1. The physical properties of these systems after being equilibrated are given in Table 2, and their dispersion numbers are listed in Table 3. In three cases, the viscosity of the viscous more-dense organic phase was measured. The results indicate that the

TABLE 1

Composition of Test System Components before Equilibration

	Initial Conditions in the Aqueous Phase			Initial Conditions in the Organic Phase		
System Number	Vol, mL	Thorium Conc, M	Free Acid Conc, M	Vol, mL	TBP Conc, %	nDD Conc, %
1	25	0.5	1.0	25	30	70
2	25	1.0	1.0	25	30	70
3[a]	25	1.0	1.0	25	30	70
4	25	1.5	1.0	25	30	70
5	25	2.0	1.0	25	30	70
6	25	2.0	1.9	25	30	70
7	25	1.0	1.0	7.5	100	0

[a]Duplicate test of System Number 2.

TABLE 2

Physical Properties of Equilibrated Thorium Systems

System Number	Phase[a]	Vol, mL	Density[b], kg/m^3	Thorium Conc, M	Free Acid Conc, M	Viscosity,[c] mPa·s
1	A	24	1150.8	0.306	d	d
	HO	6.25	1088	0.463	d	12.4[e]
	LO	19.75	837.7	0.092	d	d
2	A	23	1307	0.686	1.02	d
	HO	9	1235	0.807	0.21	d
	LO	18	787.9	0.0406	0.046	d
3	A	24.75	1290.8	d	d	d
	HO	8.5	1234	d	d	53[f]
	LO	16.75	789.5	d	d	d
4	A	24	1466	1.108	d	d
	HO	9.5	1301	0.953	d	127
	LO	16.5	775.3	0.025	d	d
5	A	23.2	1659	1.637	1.01	d
	HO	9.7	1342.6	1.05	0.81	d
	LO	17.2	767.6	0.014	0.017	d
6	A	24.2	1647.9	1.57	1.51	d
	HO	9.3	1338.9	1.037	0.23	d
	LO	16.5	766.6	0.013	0.023	d
7	A	24	1298	0.664	d	d
	HO[g]	8.5	1312	0.971	d	d

[a] A is aqueous phase; HO is heavy organic phase; and LO is light organic phase.

[b] Density of pure nDD is 759 kg/m^3; that of 30% TBP in nDD, 826 kg/m^3.

[c] The viscosity of the aqueous phase is 1.0 mPa·s. The viscosity of the light organic phase is essentially that of nDD, that is, 1.9 mPa·s. All systems were measured at 26°C except as noted.

[d] Not measured.

[e] Measured at 25°C.

[f] Measured at 25.5°C.

[g] No third liquid phase formed in this test system. Note that the organic phase is more dense than the aqueous phase.

TABLE 3

Dispersion Numbers of Test Systems

System Number	$N_{Di,M} \times 10^4$	Residual[a] A in O (O in A), Vol %	$N_{Di,L} \times 10^4$	Residual[b] O_H in O_L, Vol %
1	10.8	0.0	14.4	1.3
3	6.2	1.0	6.2	1.5
4	4.3	0.4	2.9	0.0
5	3.0	6.9	2.0	c
6	3.3	5.0	2.0	c
7	3.0	(2.0)	--	--

[a] A indicates aqueous phase; O indicates organic phase.

[b] O_H indicates heavy (more dense) organic phase; O_L indicates light (less dense) organic phase.

[c] Not measured.

viscosity increases and the dispersion number decreases as the thorium concentration increases. The relatively high value of the dispersion numbers, especially for the lower three-phase thorium concentrations, indicates that satisfactory contactor performance can be expected in continuous-flow separation tests up to relatively high throughputs.

During the batch settling tests for system 2 in Table 1, the three-phase dispersion was observed as it coalesced to determine which phase was continuous. One criterion, which is usually valid, is that the dispersed phase will be the clear phase. Since only the light organic phase was clear, it appears to have been the dispersed phase. A second criterion is that, for the final separation of a batch settling test where the dispersion band is very distinct, the overall continuous phase will have the least volume in the dispersion band. Using this second criterion indicates that the aqueous phase was the overall continuous phase. Thus, it appears that the light organic phase is dispersed in the heavy organic phase which in turn is dispersed in the aqueous phase. Such a structure would explain why, early in the separating process, large droplets of heavy organic phase appeared at the interface between the dispersion layer and the light organic phase. Over a period of time, the drops would break free from this interface and drop down through the dispersion layer to the heavy organic phase layer.

CONTINUOUS SEPARATION TESTS

Continous separation tests of two- and three-phase liquids were made using an annular centrifugal contactor, often called the ANL centrifugal contactor. This design features an annular mixing zone, which promotes excellent mixing even at very short residence times, and a centrifugal separating zone as shown by Bernstein *et al* (9). While an air-controlled aqueous weir (*i.e.*, a double aqueous weir) could have been used if the unit were larger, the small size of the rotors (4-cm diameter) in these tests made it easier to build rotors with a single aqueous weir. A schematic of the rotor with a single aqueous weir was given in an earlier paper (11).

Single Stage Operation

Single-stage continuous separation tests were made to evaluate criteria proposed for the design of the centrifugal separating zone. For these tests, the aqueous feed was 0.70 *M* thorium nitrate in 1.15 *M* nitric acid and the organic feed was 30 vol. % TBP in nDD. At an O/A flow ratio of 3.0, the concentration of thorium in the organic phase is low enough that no second organic phase forms. The aqueous and organic phases have densities of 1129 and 868 kg/m^3, respectively. However, at an

O/A ratio of 1.0, there is a second organic phase. The aqueous phase, the heavy organic phase, and the light organic phase have densities of 1222, 1162, and 792 kg/m^3, respectively. These two flow ratios were used in the single stage tests reported here.

In a centrifugal contactor, such as used in this test, the interface between the organic and aqueous phases in the centrifugal separating zone of the rotor must be balanced between the organic weir and the aqueous underflow to the organic weir. For an air-controlled aqueous weir (17), this balance can be adjusted by changing the air pressure over the inner aqueous weir. For a single aqueous weir, which cannot be controlled by air pressure, the interface position is adjusted by changing the aqueous weir radius. For the contactor used in these single and multistage tests, each stage has a single aqueous weir. The aqueous weirs are removable so that it is easy to test various aqueous weir radii.

Two alternative criteria were proposed for the design of the centrifugal separating zone. The one criterion assumes that the two organic phases can be treated as a single organic phase with an average density of 891 kg/m^3 based on the amount and density of each of the two organic phases. The alternative criterion assumes that the densities used must be those of the two phases between which a separation is desired. In the tests reported here, the separation is to be made between the aqueous phase and the heavier organic phase. Thus, using the alternative criterion, the densities of these two phases should be used in making the hydraulic balance that determines the interface position in the centrifugal separating zone of the rotor.

The results of the single-stage thorium tests are listed in Table 4. Test 1 has only two phases, and the aqueous weir radius of 9.75 mm was chosen as being close to optimum for this test. If the first design criterion applies for three phase operation, test 2 should be near its optimum aqueous weir radius so that the aqueous phase should all come out one exit and the two

TABLE 4

Single-Stage Thorium Test Results

Test[a]	Total Flow Rate, L/min	O/A Ratio	Aqueous Weir Radius, mm	Effluent Flow Rate, L/min		Effluent Temp, °C		Phase Composition of Effluents[b]				Notes
				Organic Exit	Aqueous Exit	Organic Exit	Aqueous Exit	Organic Exit		Aqueous Exit		
								Phase	Amt Present, vol %	Phase	Amt Present, vol %	
1	0.6	3.3	9.75	0.452	0.140	26.6	25.8	O	100	O	0.7	c
										A	99.3	
2	0.6	1.0	9.75	0.240	0.357	26.5	26.5	LO	94.4	LO	0.9	d
								HO	5.6	HO	17.2	
										A	81.9	
3	0.2	3.3	8.59	0.156	0.0472	25.7	25.6	O	100	O	0.4	c
								A	trace[e]	A	99.6	
4	0.2	1.1	8.59	0.105	0.0950	26.4	26.5	LO	71.5	O	0.1	g
								HO	25.2	A	99.9	
								A	3.3[f]			
5	0.6	1.1	8.59	0.332	0.282	26.0	26.0	LO	69.9	O	0.5	g,i
								HO	26.5	A	99.5	
								A	3.6[h]			

[a] Aqueous feed contained 0.7 $\underline{M}$ Th and 1.15 $\underline{M}$ HNO_3. Organic feed was 30 vol % TBP in nDD with no thorium or nitric acid. Both feeds were prepared the day of the tests. Rotor speed was 3568 ± 9 rpm. Tests used in stage 3 of the 4-stage centrifugal contactor.

[b] O indicates the organic phase for a two-phase system and the total organic phase for a three-phase system. A indicates the aqueous phase. LO indicates the light organic phase. HO indicates the heavy organic phase.

[c] No second organic phase.

[d] Heavy organic phase slowly separated from the organic effluent sample.

[e] Some aqueous droplets were on wall of the cylinder containing the organic effluent, but no aqueous phase accumulated at the bottom.

[f] Aqueous phase appeared slowly. After 36 minutes, the aqueous volume had become constant at this volume percent.

[g] As noted, the three phases in the organic effluent take some time to separate out.

[h] Aqueous phase appeared slowly. At 0.0 minutes after the test, there was no aqueous phase; 4.5 minutes, 0.6%; 10 minutes, 2.4%; 28 minutes, 3.6%; 34 minutes, 3.6%.

[i] After test, rotor and pumps were stopped together. Contents from the rotor and the mixing zone, drained into a 150-mL beaker with 10-mL markings, included 90 mL of aqueous phase, 10 mL of heavy organic phase, and 40 mL of light organic phase.

organic phases should come out the other exit. This is not the case. Considerable organic phase came out the aqueous exit. Thus, the second criterion seems to apply.

To test this further, a smaller aqueous weir radius, 8.59 mm, was chosen based on the alternative criterion. With this weir radius, the maximum throughput for satisfactory two-phase operation is much less, about 0.2 L/min. This result is because the radius is far from the optimum radius used in test 1. As tests 3 and 4 show, both two- and three-phase operation works well at this reduced flow rate. At this aqueous weir radius, the alternative criterion predicts that three phase operation should be nearly optimum, making flow rates up to 0.6 L/min possible. This was checked in test 5 and found to be true; however, somewhat more aqueous phase (3 to 4 vol. %) than normal (<1 vol. %) was found in the organic effluent. This entrapment of 3 to 4 vol. % of the aqueous phase in the two organic phases corresponds to the 0.0 to 6.9 vol. % residual aqueous phase in the organic phase for $N_{Di,M}$ of the batch gravity-settling tests given on Table 3.

From these single-stage contactor tests, we see that, using the second design criterion, both organic phases can be made to come out one exit while the aqueous phase comes out the other exit. Based on these encouraging results, four-stage contactor tests were made.

Multistage Operation

The flow sheet to demonstrate annualar centrifugal contactor operation with three liquid phases is shown in Fig. 1. It was expected that the two middle stages (stages 2 and 3) would have three liquid phases. However, with only a single scrub stage (stage 4) and with the high acid concentration in the organic liquid going to that stage, it seemed possible that stage 4 might have three liquid phases and that stage 2 might

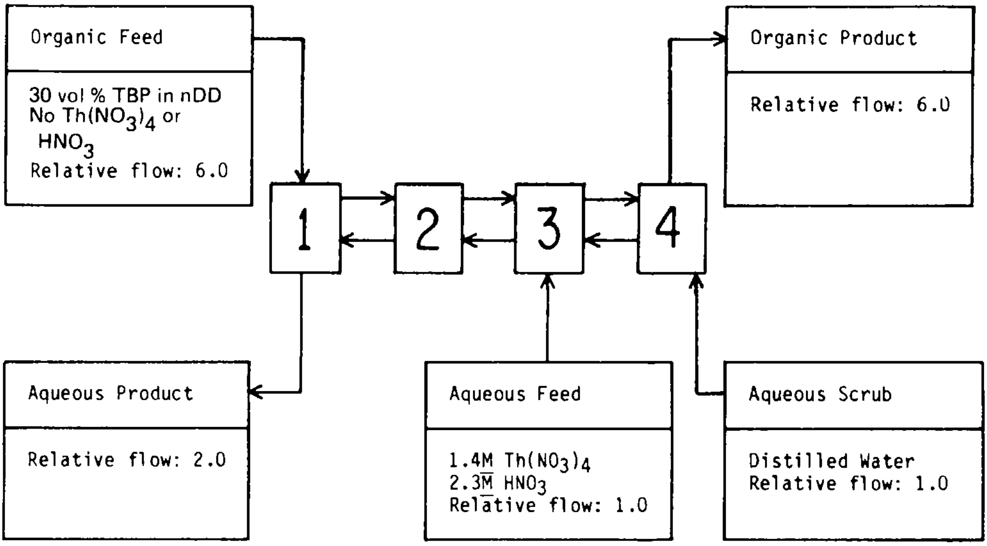

FIGURE 1. Flow Sheet for Multistage Thorium Test.

have only two liquid phases. Because of the uncertainty over which stages would have three liquid phases, the first test of the four-stage contactor was run at a low flow rate (200 mL/min), using the smaller weir radius in each stage. Under these operating conditions, the contactor can handle the flow sheet with either two or three phases in a stage. During this test (test 6), the number of liquid phases present in each stage was determined. The results, given in Tables 5, 6, and 7, show that there were three liquid phases in stages 3 and 4 and two liquid phases in stages 1 and 2.

Using the same flow sheet, the contactor was operated at a high throughput of 600 mL/min. This is close to the maximum throughput of 800 mL/min calculated from a dispersion number of 6.1×10^{-4}. For each stage where only two liquid phases were present, the weir with the larger radius was installed in place of the smaller weir. Start-up was seen to be a potential problem because, at this high flow rate, the stages with weirs

TABLE 5

Contactor Specifications for Multistage Thorium Test

Stage No.[a]	Aqueous Weir Radius, mm		Vane-to-Rotor Gap,[b] mm
	Test 6	Test 7	
1	8.59	9.75	0.87
2	8.61	9.75	1.0
3	8.59	8.59	0.96
4	8.62	8.62	0.4

[a] Four-stage 4-cm contactor was used with the flowsheet as shown in Fig. 1. Rotor speed was 3568 ± 9 rpm. In addition, rotors 1 and 4 required Teflon tape beneath the aqueous weirs to prevent leakage. (To measure rotor leakage, the amount of liquid in the rotor was measured after several minutes at no-flow conditions.) After the tape was added, the liquid in the rotor after 2 to 3 minutes of no flow was 60 mL for stage 1, 65 mL for stage 2, 75 mL for stage 3, and 70 mL for stage 4. For this test, only aqueous phase was used.

[b] These gaps were set for test 6 listed in Table 6. Vane-to-rotor gaps were not measured for test 7.

designed to operate with two phases cannot handle three phases, and vice versa. In the actual test (test 7), start-up was not a problem. As the results in Tables 6 and 7 show, operation was quite satisfactory with the flow of the organic phases maintained countercurrent to the flow of the aqueous phase. Thus, three-phase operation is seen to be satisfactory in annular centrifugal contactors, both at intermediate stages and at feed stages with external feed and product flows.

For the multistage tests, it was necessary to establish the number of liquid phases in each stage. This was done by draining out the liquid in each stage after the test. These results are given in Table 7. Both tests showed good operation, with the heavy organic phase staying with the light organic phase as

TABLE 6

Multistage Thorium Test Results

	Test No. 6	Test No. 7
Aqueous Feed Rate, mL/min	25	75
Liquid Temp, °C		
Organic Exit	24.3	24.6
Aqueous Exit	24.5	24.6
Amt of Each Phase in Effluent[a]		
Organic Exit		
Amt of LO, %	87.2[b]	79.5[c]
Amt of HO, %	12.8	20.5
Aqueous Exit		
Amt of O, %	0.9	0.7
Amt of A, %	99.1[d]	99.3[c]
Effluent Flow Rate[e], mL/min		
Organic Exit	168.5	454
Aqueous Exit	54.5	147
Time Allowed to Reach Steady State, min	18	6

[a] O indicates the organic phase for a two-phase system and the total organic phase for a three-phase system. A indicates the aqueous phase. LO indicates the light organic phase. HO indicates the heavy organic phase.

[b] Values when the sample was held overnight (20 h). At that time, the heavy organic phase volume was clear near the top but still cloudy below. This indicates some trace amount of aqueous phase was present. The light organic phase was very clear. The two phases slowly separated so that, 20 minutes after the sample was taken, there was 90.8% light organic phase and 9.2% heavy organic phase.

[c] Volumes were measured 28 min after the samples were taken. No second organic phase was visible at first in the organic exit effluent. After six minutes, two distinct organic phases were visible. The light organic phase was clear which suggests it was the dispersed phase. The heavy organic phase was opaque as it still contained 5% of the light organic phase. After 13 minutes, the two phases were completely separated. Even after 28 minutes there was no visible aqueous phase.

[d] Value when sample held overnight (20 h). Twenty minutes after the sample was taken, there was only a trace of organic phase floating on the aqueous phase.

[e] Total volumetric flow rate for all phases leaving the specified exit.

TABLE 7

Liquid Volume in Each Stage after Multistage Thorium Tests

Four-stage 4-cm contactor.
Flow sheet as shown in Fig. 1.
Feed pumps and rotor motors were stopped at the end of the test. Then all liquid was drained from each stage.
Volumes were measured in 250-mL cylinders with 2-mL markings after the samples had been allowed to separate from 17 to 20 h.

Test	Stage	Total Liquid Volume, mL	Phase Volume, mL (%)		
			Aqueous	Heavy Organic	Light Organic
6	1	95	67.6(71.2)	trace[a]	27.4(28.8)
	2	95.7	67.0(70.0)	trace[b]	28.7(30.0)
	3	132	70.0(53.0)	18(13.6)	44(33.4)
	4	103	46.4(45.0)	31.6(30.7)[c]	25(24.3)
7	1	105	52.5(50)	trace[a]	52.5(50)
	2	145	53(36.5)	8.5(5.9)	83.5(57.6)
	3	193	92(47.7)	36(18.6)	65(33.7)
	4	136	35(25.7)	51.5(37.9)	49.5(36.4)

[a] Appears as dirty white droplets at the liquid-liquid interface.

[b] Appears as droplets at the liquid-liquid interface. More droplets than for stage 1.

[c] 16 min after the sample was taken and the phases were still separating, cloud-like lumps were noted at the interface between the heavy and light organic phases.

intended. Since the interstage lines were transparent polyvinyl chloride tubing, the heavy organic phase could be seen moving from stage 3 to stage 4 in the organic interstage line.

To avoid potential problems in the start-up of test 7, the following procedure was used. First, the aqueous scrub (distilled water) was started and run alone until it was flowing out the aqueous product exit. Then the organic feed was started.

One minute later, when the organic phase reached stage 3, the aqueous feed containing the thorium nitrate and nitric acid was started. In this way, start-up went very smoothly to steady-state operation. No attempt was made to disturb steady-state operation to see if the contactor could recover.

CONCLUSIONS

Operation with three liquid phases was demonstrated using a thorium flow sheet. The results show that the annular centrifugal contactor can be designed to handle three liquid phases and that the presence of three liquid phases in several stages of a multistage unit does not interfere with multistage operation.

The criterion to be used in the design of the centrifugal separating zone is as follows. Only those two phases closest in density, between which a separation is desired, are considered. A hydraulic balance is established such that the theoretical interface between these two phases is located near the middle of the separating zone. A contactor operated in this way will be at or near its maximum throughput.

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