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DEVELOPMENT OF A SOLVENT EXTRACTION PROCESS FOR CESIUM REMOVAL FROM SRS TANK WASTE

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ABSTRACT

An alkaline-side solvent extraction process was developed for cesium removal from Savannah River Site (SRS) tank waste. The process was invented at Oak Ridge National Laboratory and developed and tested at Argonne National Laboratory using single-stage and multistage tests in a laboratory-scale centrifugal contactor. The dispersion number, hydraulic performance, stage efficiency, and general operability of the process flowsheet were determined. Based on these tests, further solvent development work was done. The final solvent formulation appears to be an excellent candidate for removing cesium from SRS tank waste.

INTRODUCTION

The Savannah River Site (SRS) currently has a large quantity of high-level waste solutions stored in underground tanks. About 22% of the cesium in the waste is ^{137}Cs, which is highly radioactive and must be removed prior to disposal of the bulk of the liquid waste as grout in a near-surface disposal facility. To remove the cesium, an in-tank precipitation (ITP) process was built (1). However, because the ITP process had technical problems, it was abandoned, and SRS is now evaluating several alternatives for removing cesium from the waste. One of the alternatives being considered is an alkaline-side solvent extraction process for cesium removal, which was developed at Oak Ridge National Laboratory (ORNL) (2). The process can both separate the cesium from the waste with the required decontamination factor (D.F.) of 40,000 and concentrate it by 12 times in an aqueous stream that contains mainly $CsNO_3$ in very dilute HNO_3, so that the removed cesium can be fed directly to the glass plant for incorporation in the glass waste form. The salt solution fed to this alkaline-side cesium extraction (CSEX) process has previously had strontium and long-lived alpha-emitting nuclides removed by the addition of monosodium titanate slurry and filtration. After the cesium is removed by the CSEX process, the salt solution is sent to a grout facility for final disposal.

At Argonne National Laboratory (ANL) and ORNL, we have worked closely with SRS personnel to take this alkaline-side cesium extraction (CSEX) process from the test-tube to the pilot-plant stage, the first step in process scale up. This work involved developing and testing process flowsheets in a multistage 2-cm centrifugal contactor. Contactor size refers to the diameter of the spinning rotor in each stage. A multistage contactor operates as follows. The two immiscible phases flow by gravity between stages in opposite directions. They enter into a stage at the annular mixing zone around the rotor as shown in Fig. 1. The dispersion created flows down the mixing zone and enters the rotor through a hole in the bottom. While the residence time in the mixing zone is short, typically a few seconds, the mass transfer efficiency is high, typically greater than 95%. The high centrifugal forces inside rotor quickly separate the dispersion into two phases and, using baffles and weirs, the two phases exit separately. They are slung into their respective collector rings where, typically, the momentum of each liquid phase carries it out the appropriate exit port. From there, the two phases continue their countercurrent flow to the adjacent stages (3). Multistage contactor tests using tracer levels of radioactive ^{137}Cs were carried out in a glovebox. Further information about centrifugal contactors and their advantages can be found in (4-9).

The cesium extractant for the alkaline-side CSEX solvent was the calixarene-crown ether: calix[4]arene-*bis*(*tert*-octylbenzo-crown-6), designated BoBCalixC6. The structure of BoBCalixC6, which has a molecular weight of 1149.52 g/mol, is shown in Fig. 2. It was manufactured by IBC Advanced Tech-

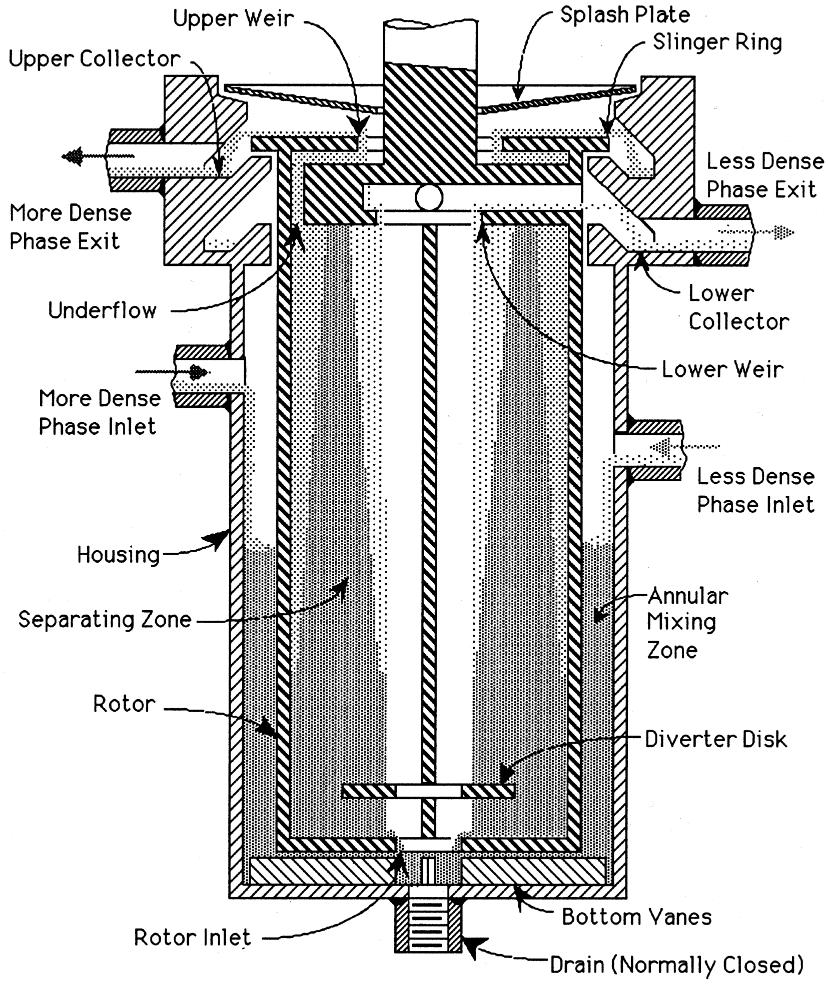

Figure 1. Schematic of operating contactor stage.

nologies, Inc. (American Fork, UT) using a procedure developed at ORNL. Several solvent modifiers were also synthesized, developed, and tested at ORNL. In the contactor tests, the modifier used was an alkyl aryl polyether: 1—(1,1,2,2-tetrafluoroethoxy)-3-[4-(*tert*-octylphenoxy)-2-propanol, designated Cs-3. The structure of Cs-3, which has a molecular weight of 380.43 g/mol, is shown in Fig. 3. The diluent, Isopar®L, is manufactured by Exxon Corp. (Houston, TX). It is composed largely of saturated paraffins with highly branched molecular structures that have an approximate boiling range from 188 to 207°C. The CSEX solvent

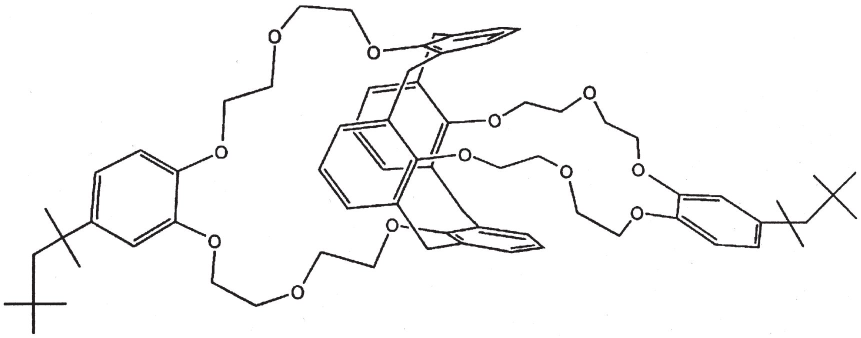

Figure 2. Structure of the BoBCalixC6 extractant.

composition for the contactor tests was 0.01 *M* BoBCalixC6 and 0.20 *M* Cs-3 in Isopar®L and is designated Cs3/120L.

This paper covers process and solvent development for the alkaline-side CSEX process as well as issues related to scale-up from a laboratory unit to a plant-scale contactor. The process was evaluated on the basis of the dispersion number, hydraulic performance, stage efficiency, and general operability in single-stage and multistage contactor tests. This evaluation led to further solvent development work and the final solvent composition. Using data based on the final solvent composition, a flowsheet appropriate for cesium removal from SRS tank waste was developed. Because the BoBCalixC6 is expensive, it will be important to limit solvent losses. For that reason, a method for solvent recovery is outlined.

PROCESS DEVELOPMENT

For process development, the proposed solvent was evaluated in a number of ways. Its ability to be mixed and separated at each stage was determined by measuring (1) the dispersion number in batch tests and (2) the hydraulic performance in a series of single-stage tests and in three multistage contactor tests during which the CSEX process itself was also evaluated. The ease with which the metal ion (Cs^+) is extracted by the solvent was determined by measuring the stage efficiency during single-stage and multistage contactor operation. General pro-

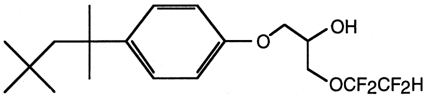

Figure 3. Structure of the Cs-3 modifier.

cess operability was evaluated during these single-stage and multistage contactor tests. When problems were uncovered, the solvent was modified. In doing these tests, two simulants were used, designated SRS#2 and SRS#4. These simulants, whose compositions are given in Table 1, approximate the average composition of the alkaline tank waste at SRS.

Dispersion Number

The dispersion number test evaluates the general ability of a two-phase dispersion to separate quickly and, therefore, to give good hydraulic performance in a solvent extraction process. Details for measuring the dispersion number are given in (10). Good performance in the centrifugal contactor is expected when the dispersion number is greater than 8 x 10^{-4}. The dispersion number for the Cs3/120L solvent was determined at room temperature (24 ± 2°C) using the aqueous phases of the alkaline-side CSEX process: SRS#2 simulant for the extraction section, 0.05 *M* HNO_3 for the scrub section, 0.0005 *M* HNO_3 plus 0.0001 *M* $CsNO_3$ for the strip section, and 0.5 *M* NaOH for the solvent rinse section. The dispersion number was 8 x 10^{-4} or greater for all but two cases, leading us to expect generally good hydraulic performance for this solvent. The two worst cases occurred in contacts with the strip solution. At an organic-to-aqueous ratio (O/A)

Table 1. Simulant Compositions for SRS Alkaline Tank Waste

	Concentration, *M*	
Ionic Species	SRS#2	SRS#4
Na^+	7	6.44
Al^{3+}	0.40	0.44
K^+	0.02	0.017
Cs^+	0.0007	0.00027
$^{137}Cs^+$	4E-08	1E-08
OH^- (total)	3.5	3.28
OH^- (free)	1.9	1.52
NO_3^-	2.7	2.7
NO_2^-	1	0.82
SO_4^{2-}	0.22	0.20
CO_3^{2-}	0.20	0.20
Cl^-	0.10	0.10
F^-	0.05	0.05
CrO_4^{2-}	0.015	0.015

of 1.0, the dispersion number was 7.3×10^{-4}, still a fairly good value. At an O/A of 0.33 (an unlikely condition as this CSEX process requires high O/A flow ratios in the strip section), the dispersion number was 1.3×10^{-4}, which is poor. However, no dispersion band formed in this dispersion number test, so phase separation was controlled by the movement of the other-phase droplets. Since the movement of other-phase droplets will be to the first power under the accelerational forces in the centrifugal contactor, not to the 1/2 power as is the case for the dispersion number, this case should not be a problem in the centrifugal contactor. Hydraulic performance tests confirmed this conclusion. Overall, the dispersion number tests predicted that the alkaline-side CSEX process should work well in the centrifugal contactor.

Hydraulic Performance

While the dispersion number test is a quick indicator of how a two-phase pair will perform in solvent extraction equipment, some performance characteristics for the two-phase pair are specific to the equipment. To get this additional information, hydraulic performance tests must be done in the type of equipment that will be used. Since centrifugal contactors are to be used in carrying out the alkaline-side CSEX process, hydraulic performance for the Cs3/120L solvent was determined in single-stage and multistage 2-cm contactors.

Single Stage

Single-stage hydraulic performance tests were carried out at room temperature (26 ± 2°C) using the same aqueous phases that were used in the dispersion number tests. The rating for hydraulic performance is as follows. “Excellent” is for no problems. “Very good” is for no problems, but at least one phase is very cloudy; if any other-phase carryover is observed, that is, visible discrete droplets of the other phase, it is less than 1%. “Borderline” is for 1 to 2% other-phase carryover. “Unacceptable” is for >2% other-phase carryover. This designation may also indicate a phase-inversion problem. When a phase-inversion problem occurs, liquid from the mixing zone rises into the liquid-level tube connected to the mixing zone, sometimes overflowing this tube. To check for the possibility of phase-inversion problems at each O/A flow ratio, the mixing zone is started with only one phase flowing through it. Then the flow of the other phase is started. In this way, contactor operation is initially made either aqueous continuous or organic continuous. If no problems occur for either start-up condition, then the contactor is free of phase-inversion problems for the immiscible liquid pair and O/A flow ratio tested.

The hydraulic performance for the scrub, strip, and rinse sections was very good to excellent at all O/A flow ratios (0.33, 1, and 3). This level of performance was observed even for the strip section at low O/A flow ratios where the dispersion number was low, a result that demonstrates how the centrifugal contactor can expand operability into regions unavailable in gravity-settling systems.

Hydraulic performance in the extraction section was also very good to excellent at low and medium O/A flow ratios (0.33 and 1). At a high O/A flow ratio (3.0), hydraulic performance was unacceptable because phase inversion occurred. The dispersion would not convert easily from aqueous- to organic-continuous operation. Instead, the dispersion became very viscous and could not flow into the rotor. As the dispersion backed up in the mixing zone, liquid was observed at the top of the liquid level tube (standpipe) of the contactor, sometimes overflowing this tube. In addition, we often found greater than 1% aqueous phase in the organic effluent. Fortunately, high O/A flow ratios are not needed in the extraction section of this process. When operation at high O/A flow ratios is needed or occurs by accident, the single-stage hydraulic-performance tests indicated a method to solve this problem. If the rotor motor is turned off long enough for the rotor to stop spinning and is then restarted, the phases invert and the problem disappears. Note that, if the process wanders back into an operating region that gives aqueous-continuous operation, the problem will reoccur when the process returns to high O/A flow ratios that require organic-continuous operation.

Multistage

Multistage hydraulic performance tests were not carried out separately, but were part of the multistage tests of the alkaline-side CSEX flowsheet using the 2-cm contactor, tests CS2, CS18, and CS19. The flowsheet for test CS2 is given in Fig. 4. The flowsheet for tests CS18 and CS19 is given in Fig. 5. For test CS18, the solvent was not recycled; for test CS19, the solvent was recycled, starting with the used solvent from test CS18.

As with the single-stage tests, the hydraulic performance for the multistage strip and rinse sections was very good to excellent. However, a phase-inversion problem was observed in the extraction section of test CS2. During startup of this test, the aqueous (DF) feed pump failed and caused an excursion into the high O/A flow ratio in the extraction section. The contactor was shut down, the pump was replaced, and the test restarted. At that point, the interstage lines to stages 2 and 3 filled up, and some organic phase overflowed the standpipe at stage 1. Turning off the organic pump for five minutes seemed to solve the problem. However, we continued to see a significant amount of aqueous phase flowing in the organic interstage lines from stages 1-3 throughout the test. As single-stage tests of hydraulic performance later showed, the process could probably have recovered fully if the

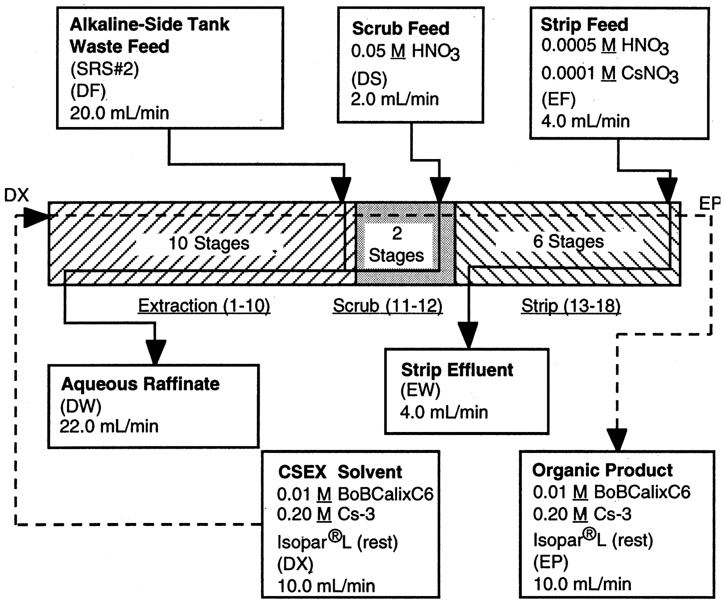

Figure 4. Flowsheet for test CS2.

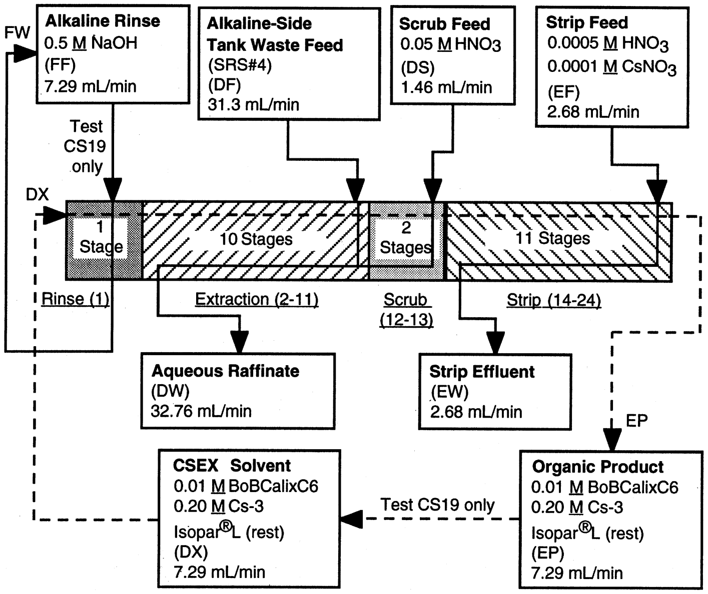

Figure 5. Flowsheet for tests CS18 and CS19.

rotor motors for these stages had been turned off long enough for the rotors to stop spinning, and then restarted.

A hydraulic-performance problem was observed during the startup of test CS18. In one of the scrub stages, liquid was observed backing up in the aqueous interstage line. Following lessons learned from the single-stage hydraulic-performance tests, the rotor motor for that stage was turned off for several seconds and restarted. This operation allowed the liquid to flow out of the interstage line. This operation had to be repeated several times before the stage operated properly and maintained the same low liquid level observed in the other interstage lines.

In summary, the hydraulic performance was generally good to excellent. The only problem observed was some resistance to phase inversion. This problem should disappear in plant-scale centrifugal contactors as it has only been observed in the smallest (2-cm) units. As discussed earlier (11), this behavior has never been observed in contactors that range from 4 to 25 cm with throughputs from 0.4 to 120 L/min. In addition, we have learned how to correct phase inversion problems when they do occur in the 2-cm contactor. More information on the dispersion number and hydraulic performance tests is given in (11).

Stage Efficiency

Stage efficiency is defined as the amount of a component transferred from one phase to the other in a stage relative to the amount that would be transferred if the two phases were fully equilibrated. In centrifugal contactors, stage efficiency is very high. From large laboratory-scale (4 cm) to full plant-scale (25-cm) units, stage efficiency is normally greater than 95% and is typically between 98 and 100%. The experimental basis for this statement is summarized in (11). Because stage efficiency is very high, contactor scale-up and flowsheet design will be easier since each physical stage is essentially a theoretical stage. This is true whether a stage is used alone or in a multistage operation.

Single Stage

A review of all the one-stage efficiency tests made in the 2-cm contactor using either cesium, technetium, or americium indicated these units have a stage efficiency close to their design value of 98% when the O/A flow ratio is close to 1.0 (11). However, at O/A flow ratios much greater than 1.0 or much less than 1.0, stage efficiency seems to drop off. A correlation of the data gives

$$E_a = 1 - 0.15 \cdot | \log_{10} R | \qquad (1)$$

where E_a is the fractional stage efficiency and R is the O/A flow ratio, which ranged from 0.18 to 6.1. The data, given in Fig. 6 along with Eq. 1, show consider data scatter. The data scatter increased as contactor operation moved away from an R of 1.0. The increase was larger when $R > 1$ than when $R < 1$. At $R > 1$, most of the data were for cesium mass transfer in single-stage tests that simulate conditions in the scrub and strip sections of the CSEX process. In the scrub section, the amount of cesium that is back-extracted is low, so data scatter will be greater. In the strip section, there was considerable uncertainty and variability in the distribution ratio of Cs (D_{Cs}); therefore, the data scatter will be greater. Also, for $R > 1$, the aqueous phase, which has a higher surface tension than the organic phase, is the phase with the lower flow rate. In these tests, the data scatter is such that at all O/A flow ratios for fractional E_a values < 1.0, the data lie within error bars of ± 0.10 from the fractional E_a value given by Eq. 1.

The decrease in the single-stage efficiency is attributed to the liquid entering the mixing zone of the contactor stage as discrete slugs. Because the contactors including the inlet tubes are made of stainless steel, these slugs were not actually observed. However, as noted below, slugs were observed in the translucent fluorocarbon interstage lines (Teflon PFA tubing) during multistage operation and likely occurred in the single-stage tests as well. The slugs in the one-stage tests would be the result of surface tension causing the liquid to drip into the mixing zone even though (1) the liquid was pumped to the inlet of the mixing zone at a relatively constant rate and (2) the translucent Teflon PFA tubing from the pump to the inlet tube was full. This effect will be most pronounced when a phase has a flow rate that is low, that is, below 5 to 10 mL/min. Since the 2-cm contactor has a nominal throughput of 40 mL/min, the flow rate of one phase or the other will drop into this flow regime as the O/A flow ratio moves away from 1.0. When one phase is in the slug flow regime, some of the other phase can pass through the mixing zone after only minimal contact with the first phase. When this happens, the apparent or measured stage efficiency drops.

Multistage

Multistage efficiency measurements made as part of tests CS2, CS18, and CS19 using cesium showed a stage efficiency of 80% for a multistage 2-cm contactor at an O/A flow ratio of 1.0. As with the single-stage tests, as the O/A flow ratio moved away from 1.0, the stage efficiency appeared to drop off. A correlation of the data, including earlier multistage 2-cm contactor results for Sr from acid, Tc from base, and U from acid (11), gives a fractional stage efficiency of

$$E_a = 0.80 - 0.17 \cdot | \log_{10} R | \tag{2}$$

where R ranged from 0.16 to 2.6. In these tests, the data scatter was great at all O/A flow ratios, and all data lie within error bars of ± 0.15 from the fractional E_a

value given by Eq. 2. The data used to get Eq. 2, along with the curve given by the equation, are shown in Fig. 6.

This lowering of stage efficiency is attributed mainly to slug flow of the liquid in the interstage lines between contactor stages. Slug flow was also observed in the effluents from the contactor, but, of course, this would have no effect on stage efficiency. Slug flow could be observed in the interstage lines of the 2-cm contactor since the lines were made of a translucent Teflon PFA tubing with an inside diameter of 5/16 in. (0.8 cm). In single-stage tests, the liquid flow to the stage was steady so that the only place slug flow could occur was at the final 90° drop into the contactor mixing zone. In multistage tests, liquid can be held up in the collector ring of the previous stage. This is only true in very small contactors such as the 2-cm contactor where the momentum of the liquid exiting the spinning rotor is dissipated in the collector ring. When this is the case, the liquid level in the collector ring rises and liquid release as slugs is controlled by the balance between the liquid height and the surface tension. In addition, the interstage lines have an effective slope of only 10 to 15°, so gravity forces on the flowing liquid are reduced. Thus, slugging in interstage flow can occur at higher flow rates, up to 50 to 100 mL/min. Since, the 2-cm contactor has a nominal throughput of 40 mL/min (both phases), it cannot be operated outside of the slug flow regime.

At 32 mL/min, the slug flow of the aqueous phase through the interstage

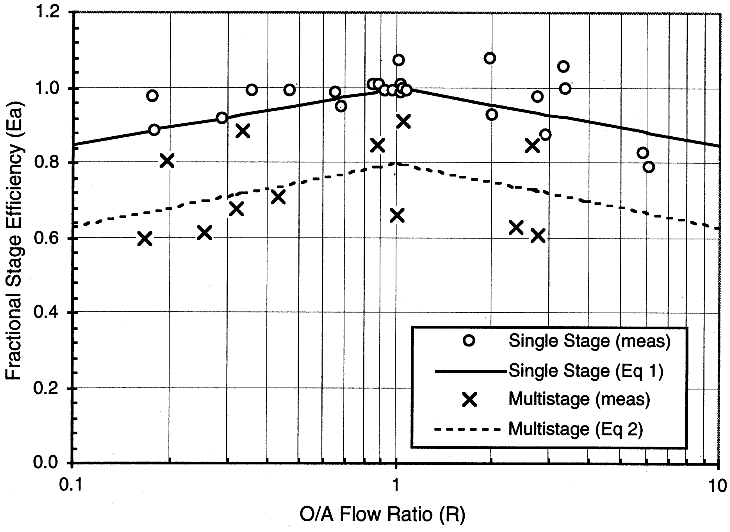

Figure 6. Correlation of stage efficiency in 2-cm contactor with O/A flow ratio.

lines of the 2-cm contactor had a period between consecutive slugs that ranged from 1 to 25 s. The average period for a slug was 10 ± 9 s. In many cases, a rivulet of liquid followed a slug and continued to flow for several seconds. Then, surface tension forces collapsed the rivulet, and no further flow occurred until the next slug. Since the residence time in the mixing zone of the contactor is short, in the range from 1 to 5 s, this liquid slugging between stages degrades the apparent or measured stage efficiency.

At higher flow rates in the 100 to 200 mL/min range, which are available in larger contactors, for example, a 4-cm contactor, the inertial forces of the liquid thrown from the spinning contactor rotor override the surface tension and gravity forces, and the liquid flows smoothly from stage to stage. This has been observed previously in a video of liquid flowing through glass interstage lines of a 4-cm contactor that has a nominal throughput of 600 mL/min (12). Thus, stage efficiencies for the 4-cm contactor have typically been 95 ± 5% (11). However, in two 4-cm contactor tests, the single-stage efficiencies were 85% and 88%, respectively (13). For these two tests, which were done at very high and very low O/A flow ratios, 33 and 0.01, respectively, such a low flow rate would be required for one phase or the other that slug flow would develop for that phase. At these extreme O/A flow ratios, slug flow can also occur in the 4-cm contactor and, so, the reduced stage efficiencies are not a surprise. In larger contactors, where even the lowest flow rates will not drop below 100 mL/min, there will be no slug flow and no reduction in stage efficiency.

In summary, for solvent extraction flowsheets tested in multistage 2-cm contactors, stage efficiencies range from 60 to 90%. These low efficiencies are attributed to intermittent (slug) flow between contactor stages. Thus, a multistage 2-cm contactor evaluation is a severe test of a process. If a process runs successfully in this apparatus, it can be run as well or better and with fewer stages in larger centrifugal contactors that have higher flow rates. However, even with slugging and stage-efficiency problems, 2-cm contactor tests are useful since they minimize the volume of both the feed and solvent needed to perform the test and the space required. For multistage operation in larger contactors (from 4 cm and on up to 25 cm or more) with flow rates greater than 100 to 200 mL/min, liquid momentum forces are greater than gravity or surface tension forces. This creates smooth flow in the interstage lines and, so, gives the expected stage efficiencies, which are between 95 and 100%. More information on the stage efficiency tests is given in (11).

Overall Operability

Given the constraints on the hydraulic performance and stage efficiency for solvent extraction tests in the multistage 2-cm centrifugal contactor, the three alkaline-side CSEX tests, CS2, CS18, and CS19, went very well and showed good overall process operability. Each effluent stream was sampled every 15 minutes during the test to get its flow rate and the concentration of Cs-137. Key operating conditions and results for each of the three tests are summarized in Table 2. These results include (1) [Cs,DF]/[Cs,DW], which is the concentration of ^{137}Cs in the aqueous (DF) feed to the extraction section relative to that in the aqueous (DW) raffinate (effluent) from the extraction section, that is, the cesium decontamination factor for the extraction section; (2) [Cs,EW]/[Cs,DF], which is the amount that cesium is concentrated in the aqueous (EW) effluent from the strip section relative to the cesium in the aqueous (DF) feed to the extraction section; (3) q_{DF}/q_{EW}, which is the flow rate of the aqueous feed to the extraction section relative to the flow rate of the aqueous effluent from the strip section; (4) [Cs,EP]/[Cs,DF], which is the concentration of ^{137}Cs in the organic (EP) effluent from the strip section relative to that in the aqueous (DF) feed to the extraction section; (5) cesium recovery material balance around the whole process; (6) $D_{Cs,Ex}$, which is D_{Cs} for the extraction section determined by carrying out batch tests with the same feeds as those used in the countercurrent contactor test); (7) $D_{Cs,Sc}$, which is D_{Cs} for the scrub section (determined by carrying out sequential batch tests with the same feeds as those used in the countercurrent contactor test); (8) $D_{Cs,St,Avg}$, which is the average D_{Cs} for the strip section determined from the experimental data using the Spreadsheet Algorithm for Stagewise Solvent Extraction (SASSE) worksheet; and (9) $D_{Cs,St,Batch}$, which is D_{Cs} for the strip section (determined by carrying out sequential batch tests with the same feeds as those used in the countercurrent contactor test). Further details are available in (11).

Process modeling using the SASSE worksheet (14) with appropriate D_{Cs} values for each section, the actual flow rates, and realistic values for stage efficiency and other-phase carryover gave good agreement with experimentally mea-

Table 2. Key Results from Multistage Tests in 2-cm Contactor

Test:	CS2	CS18	CS19
[Cs,DF]/[Cs,DW]	1840	280	24
[Cs,EW]/[Cs,DF]	5.7	13.0	9.7[a]
q_{DF}/q_{EW}	5.5	11.5	10.2
[Cs,EP]/[Cs,DF]	0.022	0.20	0.161
M.B. for Cs recovery, %	106	118	104
$D_{Cs,Ex}$	10.5	11.4	8.2
$D_{Cs,Sc}$	1.07	0.94	1.00
$D_{Cs,St,Avg}$	0.088	0.32	0.27
$D_{Cs,St,Batch}$	0.32	0.27	0.23

[a] This ratio is based on the maximum concentration in the aqueous effluent from the strip section which occurred at 45 min into test CS19. By the end of the test (180 min), this ratio had dropped to 7.5 and, as a result, the material balance had dropped to 83%.

sured effluent concentrations. In the strip section, where D_{Cs} was most variable, $D_{Cs,St,Avg}$ calculated by SASSE is compared with $D_{Cs,St,Batch}$ determined using sequential batch tests. Based on experimental results at ORNL before test CS2, $D_{Cs,St}$ was expected to be between 0.034 and 0.088. The multistage test gave $D_{Cs,St}$ of 0.088 for test CS2, and the ANL sequential batch tests done later were much higher, 0.32. As Table 2 shows, this high $D_{Cs,St}$ value was confirmed by the two later multistage tests, CS18 and CS19. As discussed below, this finding led to further work at ORNL to understand the reason for the high $D_{Cs,St}$ value and, based on this knowledge, develop a second-generation solvent. The SASSE model agrees with the experimental data from test CS19, in which the solvent had been recycled four times. This agreement demonstrates that (1) the process can be operated under full solvent recycle and (2) solvent recycle can be modeled in the SASSE worksheet.

The cesium material balances listed in Table 2 for the three multistage tests show considerable variability, with cesium recovery values ranging from 83 (see footnote in Table 2) to 118%. This range is attributed to the low flow rate of the aqueous strip (EW) effluent. Because this flow rate is low, it is hard to measure based on the change in liquid level of the tank containing the aqueous strip (EF) feed. In addition, the slugging interstage flow in the strip section creates large flow variations in the strip effluent, making this effluent flow rate hard to measure accurately as well. When the large fluctuations in the aqueous phase flow rate for the strip section are coupled with the high cesium concentration in this effluent, the variability in the cesium material balance error becomes understandable.

At the end of the solvent recycle test (CS19), the cesium decontamination factor for the extraction section was 12 times less than on the test without solvent recycle (CS18). This was true even though the two flowsheets are almost identical. The reasons for this lower decontamination factor are as follows. (1) Because of low stage efficiency and high $D_{Cs,St}$ values, the strip section did not remove the cesium from the recycled solvent sufficiently. (2) The solvent rinse was operated at full recycle. Therefore cesium accumulated there and the cesium concentration in the aqueous rinse solution rose steadily throughout the test. This pattern resulted in a corresponding increase in the cesium concentration of the solvent going to the extraction section, which increased the cesium concentration in the aqueous raffinate. (3) The solvent for test CS19 already contained cesium because it was reused from test CS18. Thus, the cesium decontamination factor for test CS19 was much lower than that for test CS18. In a fully developed process, the cesium concentration in the strip section would be reduced to an appropriately low value. In addition, if a rinse section were needed, the aqueous rinse solution would not be recycled so many times that the cesium concentration in the solvent would be increased significantly.

The scrub section has little effect on the cesium decontamination factor for the extraction section because, as shown in Table 2, D_{Cs} for the scrub section is about 1.0 while the O/A flow ratio is high, 5.0. Thus, the extraction factor (D_{Cs} times the O/A flow ratio) for cesium is greater than 1.0 and the cesium can move easily through the scrub section. However, other metal salts with D values much less than that for cesium are effectively scrubbed out of the solvent (15). The more scrub stages, the more effectively the other metal salts will be removed. Since the D values for the other metal salts in the aqueous (DF) feed appear to be low, two scrub stages should be adequate.

Because the SASSE worksheet was able to model tests CS2, CS18, and CS19 discussed above, we were comfortable in using it to generate improved alkaline-side CSEX flowsheet for the new solvent that is discussed next.

SOLVENT DEVELOPMENT

After the three multistage contactor tests, the solvent was developed further. In particular, the results of the three multistage tests were analyzed to determine why the D_{Cs} value was sometimes higher than expected in the strip section. It was postulated that if organic-soluble acidic anions were present, they could keep the cesium cations from being stripped from the solvent. Since the concentration of the cesium cations is low, the concentration of these anions could be at trace levels. Work at ORNL showed that undecyl and dodecyl sulfonate were likely present at parts-per-million levels in those aqueous (DF) feeds to the extraction section that subsequently created problems in the strip section (16). These two compounds are present in the detergent (Joy) used to clean the glassware in which the DF feeds at ANL were prepared. To confirm this finding, batch contacts were done to show that, when dodecyl sulfonate is added to the Cs3/120L solvent, D_{Cs} in the strip section is high. To solve this problem, trioctyl amine (TOA) was added to the solvent. The amine will remain neutral in the alkaline extraction section but will be converted to its trioctyl ammonium nitrate salt in the organic phase as it is contacted with the acidic scrub solution. In addition to providing a nitrate concentration high enough to prevent ion dissociation, this amine suppresses the effect of the organic-soluble acidic anions on the cesium cations in the solvent. It was found that, with only a small concentration of TOA in the solvent (0.001 M), good cesium stripping is obtained in the presence of the undecyl and dodecyl sulfonates in a 0.001 M HNO_3 stripping solution (16). Thus, with TOA in the solvent we do not anticipate organic-soluble acidic anions to be a problem in either lab- or plant-scale operations.

Further solvent work focused on developing a new modifier. The solvent modifier inhibits the formation of a second organic phase and helps solvate the anion associated with the cesium cation. The Cs-3 modifier showed significant (50%) degradation after tests in which the solvent was kept in contact with SRS#2 simu-

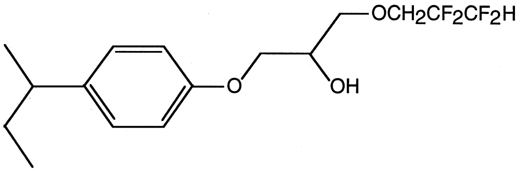

Figure 7. Structure of the Cs-7SBT modifier.

lant for 3.4 weeks (570 h) at 53 ± 2°C (2). The BoBCalixC6 in the solvent was not degraded during this test. Work to develop a new modifier that was more stable at increased temperature resulted in an alkyl aryl polyether, 1-(2,2,3,3,-tetrafluoropropoxy)-3-(4-*sec*-butylphenoxy)-2-propanol, also called Cs-7SBT. The structure of this modifies is shown in Fig. 7. The polyether R group to the right of the benzene ring in the Cs-7SBT modifier was changed from that in the Cs-3 modifier so that the modifier is more stable (17). In addition, the *tert*-octyl group connected to the 4-position of the benzene ring in the Cs-3 modifier was replaced by a *sec*-butyl group in the Cs-7SBT modifier. Thus, thermal or irradiation damage to this portion of the Cs-7SBT modifier will give degradation products with a shorter chain length (*sec*-butyl rather than *tert*-octyl). Since *sec*-butyl degradation products should come out of the solvent more easily than *tert*-octyl degradation products, solvent cleanup should be easier when the Cs-7SBT modifier is used.

Based on this solvent development work, the appropriate solvent composition for the SRS#2 simulant was determined to be 0.01 *M* BoBCalixC6, 0.50 *M* Cs-7SBT, and 0.001 *M* TOA in Isopar®L, designated Cs7SBT/150LT. Batch tests of this solvent with the SRS#2 simulant gave D_{Cs} for the extraction and scrub sections as

$$D = \{-(D_o x/T + 1 - D_o R) + [(D_o x/T + 1 - D_o R)^2 + 4D_o R]^{0.5}\}/(2R) \qquad (3)$$

where D is the actual D_{Cs} value in the stage, D_o is the D_{Cs} value with no cesium loading of the solvent, R is the O/A ratio in the stage, T is the molar concentration of the extractant (BoBCalixC6) in the solvent, and x is the molar concentration of all the cesium entering the stage, assuming that it is equally distributed by concentration through all the entering aqueous phases with nothing in the entering organic phases. For the extraction section, the D_o for this solvent with the SRS#2 simulant was extrapolated from the experimental data at 25°C and found to be 14.4; for the scrub section with 0.05 *M* HNO_3, 1.49. For the strip section with 0.001 *M* HNO_3, no correlation was developed. Instead, the batch results for the Cs7SBT/150LT solvent taken at an O/A ratio of 3 were used directly with a D_{Cs} of 0.175 for the first strip stage, 0.0998 for the second, 0.064 for the third, and 0.050 for subsequent strip stages.

A flowsheet was developed using the SASSE worksheet for the

Cs7SBT/150LT solvent with these feeds. The flowsheet is designed to meet the three process goals required at SRS: (1) achieve a cesium decontamination factor of 40,000 in the aqueous raffinate, (2) concentrate the cesium in the aqueous strip effluent by a factor of 12, and (3) minimize the number of contactor stages. The SASSE model includes feed flow rates, other-phase carryover, solvent recycle, different D_{Cs} values in each stage, and stage efficiency. The final flowsheet, shown in Fig. 8, meets all three design goals using process parameters that appear to be very realistic. No solvent rinse section is shown as it is not known at this time if such a section will actually be needed.

Because of the many factors that are included in the SASSE calculations, it was possible to consider a new concept, process robustness. It is defined as the calculated decontamination factor for the extraction section divided by the desired decontamination factor A SASSE evaluation of this new flowsheet showed that, if the variation in flow rate is ±10%, then a process robustness of 3 to 5 is required for the specified operating conditions, that is, stage efficiency and other-phase carryover. If the variation in flow rate is ±7%, then a process robustness of 2 to 3 is required. The process robustness is shown in Table 3 over the range of operating

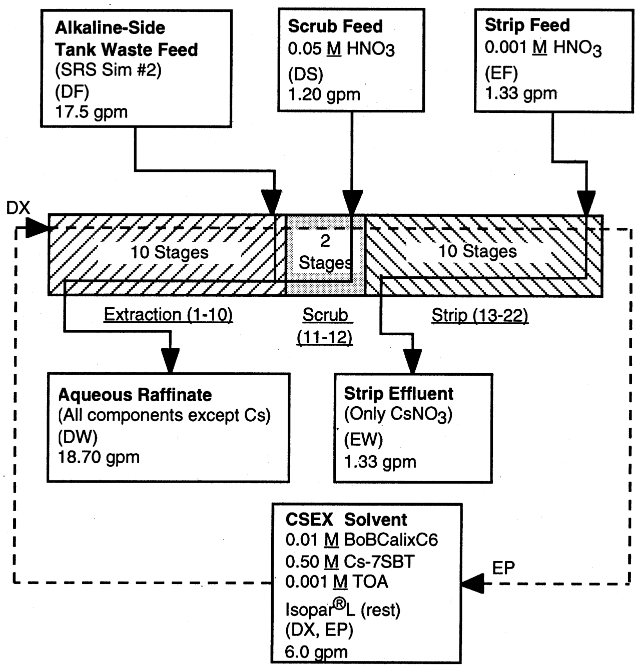

Figure 8. Alkaline-side CSEX flowsheet for new Cs7SBT/150LT solvent in a plant-scale centrifugal contactor.

Table 3. Robustness of the New Flowsheet for the Cs7SBT/150LT Solvent

Fractional Stage Efficiency	Fractional Amount of Other-Phase Carryover	Decontamination Factor (D.F.)	Robustness	Notes
0.95	0.01	1.30E + 05	3.25	Lowest expected operation in plant-scale contactor
0.98	0.01	2.88E + 05	7.19	Near lower end for expected operation in plant-scale contactor
0.99	0.0004	8.58E + 05	21.4	Typical operation expected in plant-scale contactor (case shown on Fig. 8).
1.00	0.005	7.87E + 05	19.7	Near upper end for expected operation in plant-scale contactor
1.00	0.0004	1.24E + 06	30.9	Upper end for expected operation in plant-scale contactor

conditions that can be expected in plant-scale centrifugal contactors. The final design specification was set to get good process operation over this range of operating conditions. Thus, we specified that, at the lowest expected operating condition for a plant-scale centrifugal contactor, that is, 95% stage efficiency and 1.0% other-phase carryover, the process should have a robustness of 3. As Table 3 shows, the flowsheet for the new Cs7SBT/150LT solvent just satisfies this criteria. For typical operation expected in a plant-scale contactor, that is, 99% stage efficiency and 0.04% other-phase carryover, the process robustness is 21. Thus, the flowsheet shown in Fig. 8 represents a conservative design that should be easy to operate and maintain for the range of perturbations that might occur over years of process operation.

The flow rates for the alkaline-side CSEX flowsheet shown in Fig. 8 were obtained by the following procedure. Since all flows on the flowsheet are relative, the aqueous (DF) flow rate was set at the rate needed at SRS, 17.5 gal/min (66 L/min). The scrub (DS) flow rate was set equal to one-fifth of the solvent (DX) flow rate. The strip (EF) flow rate was set equal to one-twelfth of the DF flow rate. This EF flow rate was then reduced another 10% to allow for flow variations of ±10%. Finally, for a given number of extraction, scrub, and strip stages, the DX flow rate was varied to maximize the cesium decontamination factor in the extraction section for the lowest (worst) expected operating conditions in a plant-scale contactor.

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SOLVENT RECOVERY

Because of the cost of the solvent, especially the extractant (BoBCalixC6), it is likely that we will want to recovered it from the aqueous effluents. The very small fraction of the BoBCalixC6 and the other solvent components that exit with the aqueous raffinate and the aqueous strip effluent must do so either dissolved in the aqueous phase or in any solvent that is dispersed in the aqueous phase. Both types of solvent loss can be minimized by contacting each aqueous effluent with two stages of the diluent (Isopar®L). In this diluent-contact method, the first stage extracts most of the solvent components, the second stage is a guard stage that removes almost all residual solvent. The effective concentration of the solvent in the aqueous effluents determines the size of the system required to process the diluent from the first diluent-contact stage.

Details of the diluent-contact method for recovery of the BoBCalixC6 and the other solvent components are as follows. The aqueous effluent is fed in series to two centrifugal contactor stages acting as recovery stages. The organic feed to each recovery stage is the diluent for the solvent (Isopar®L). This diluent is operated at full recycle for each stage and should recover 98% or more of the solvent from the aqueous phase as it passes though the stage. When the BoBCalixC6 concentration in the first-recovery-stage diluent increases so that it is 1 to 5% or more of its concentration in the working solvent, this diluent would be sent to a vacuum distillation unit. There, Isopar®L would be recovered as the overhead product since it is more volatile than the extractant, modifier, or suppressant. The extractant, modifier, and suppressant, now concentrated in Isopar®L at the bottom of the vacuum distillation unit to about the same concentrations they have in the working solvent, would be subjected to a final concentration adjustment and returned to the process. It is possible that additional purification would be required. The organic (diluent) feed for the second recovery stage would be moved to the first recovery stage, and the processed (distilled) diluent would be returned to the second recovery stage. In this way, it is estimated that more than 99.9% of the solvent in the aqueous effluents would be recovered and recycled. In addition, the recovery medium, Isopar®L, is itself recovered and reused.

No work has yet been done to demonstrate the effectiveness with which Isopar®L can remove the components of the process solvent from the aqueous effluent in a centrifugal contactor stage. However, the principle of using an organic liquid to remove organic components from an aqueous phase has been used in batch contacts with good results. For example, concentration of BoBCalixC6 and Cs-3 in an aqueous phase was determined by contacting the aqueous phase with dichloromethane to extract these organics. When the dichloromethane was evaporated to dryness, the residual weight gave the total mass of solvent that was in the aqueous phase. This residue was then redissolved in deutero chloroform containing a known concentration of hexamethyl benzene as an internal standard. The concentration of BoBCalixC6 and Cs-3 in the deutero chloroform was determined by nuclear magnetic resonance (2). Since plant-scale centrifugal contactors have high stage efficiencies and since an organic phase is commonly used to recover organic molecules from an aqueous phase, solvent recovery from the aqueous effluents using the process diluent should be straightforward.

An alternative method, which would recover only the dispersed solvent, would be to physically separate the dispersed solvent from the aqueous phase by sending each aqueous effluent to a settling tank or centrifugal settler. This method is simpler than diluent recovery and it would guard against large solvent losses that could occur if the aqueous phase became very cloudy or had visible droplets of organic phase in it or both.

DISCUSSION

While major progress was made in the development of an alkaline-side CSEX process, further work is needed. In particular, the flowsheet for the new Cs7SBT/150LT solvent needs to be tested in a centrifugal contactor. The solvent also needs to be tested with actual waste. The rate at which the solvent in contact with the various aqueous phases degrades at high temperatures and in high radiation fields (β((γ) must be determined, along with the degradation products formed. Once the degradation products are known, a plan for solvent cleanup can be devised. Some degradation products may not be extracted by the diluent and will exit in the aqueous effluents. Other degradation products may be recovered as a separate stream during vacuum distillation in solvent recovery. Any polymerized solvent may form a crud that can be removed by passing the recycled solvent through a coarse stainless steel mesh filter.

The rate of solvent loss can be estimated from single-stage 2-cm contactor tests reported in (11). The aqueous (DW) raffinate should range from hazy to very clear, and the aqueous strip (EW) effluent should be very clear. Thus, most of the lost solvent will be carried in the DW raffinate since (1) its flow rate is much higher than that for the EW effluent and (2) the amount of dispersed solvent is higher, perhaps as high as 100 ppm (hazy). Since the concentration of BoBCalixC6 in the solvent is 11.5 g/L (0.01 *M* BoBCalixC6), 100 ppm of solvent in the aqueous phase gives it an effective BoBCalixC6 concentration of 1.15 ppm. The concentration of BoBCalixC6 dissolved in the aqueous phase can be calculated using the partition ratio for BoBCalixC6, which is $>4 \times 10^4$ (2). Thus, the maximum aqueous-phase concentration for dissolved BoBCalixC6 is 0.29 ppm. Combining these two BoBCalixC6 concentrations, the total (effective) BoBCalixC6 concentration in the aqueous raffinate will be 1.44 ppm or less.

Using this total BoBCalixC6 concentration for the aqueous effluents from the extraction and strip sections, the cost of solvent loss without solvent recovery and the processing capacity of the solvent recovery system can both be calculated for the SRS flowsheet shown in Fig. 8. Since the total flow rate for the two aqueous effluents will be 20 gal/min (76 L/min), the loss of BoBCalixC6 without any solvent recovery will be 0.109 g/min (57 kg/y). The cost of this BoBCalixC6 loss would be $8.6 million per year based on a BoBCalixC6 cost of $150 per gram. With 99.9% recovery of the BoBCalixC6, the cost of this loss drops to $8,600 per year. At this efficiency, the recovery system would recover 0.6 L/h of solvent. If the diluent used for recovery is loaded with 1% of the solvent, the recovery system would need to process 60 L/h of diluent; if 5%, 12 L/h; if 50%, 1.2 L/h. This calculation shows that the size of the system for solvent recovery depends on (1) the extent to which solvent can be loaded in the diluent and (2) the concentration of BoBCalixC6 in each aqueous effluent. In any future work on the alkaline-side CSEX process, values for these two quantities should be refined so that process economics can be better assessed.

The loss rate can be calculated if the concentrations of the solvent dispersion and of the dissolved extractant are known or can be estimated. Based on the cloudiness of the aqueous effluents, the dispersed solvent concentration can be estimated. For the Cs3/120L solvent, we found the concentration of dispersed solvent in the aqueous phase can range from 20 ppm (very clear) to 400 ppm (very cloudy), that is, 0.23 to 4.6 ppm of BoBCalixC6. For this solvent,

Because of the low stage efficiency found during multistage operation of the 2-cm contactor (60-90%), ways to improve this stage efficiency were reviewed. One method, which looks particularly attractive, would increase the effective stage efficiency by having more liquid volume in the mixing zone of each stage. With more liquid, the deleterious effect of slug flow on effective stage efficiency is reduced. We would not increase the 1.6-mm (1/16-in.) annular gap of the mixing zone, as this increases the possibility of phase-inversion problems (8). Instead, we would increase the diameter of the rotor inlet so that the rotor is no longer fully pumping. When this is done, some minimum liquid level will be maintained in the mixing zone, even at very low flow rates. The diameter of the rotor inlet must be made large enough so that sufficient liquid stays in the mixing zone to improve efficiency in multistage operation. However, it must not be so large that the liquid in the mixing zone overflows into the lower (less-dense phase) collector ring of the rotor housing. With the current design, the liquid level is 24 mm or less with air mixed in with the liquid (18). Since the length of the mixing zone is 57 mm, this liquid level can be raised somewhat, which should improve the effective stage efficiency during multistage operation.

SUMMARY AND CONCLUSIONS

Single-stage and multistage tests in a 2-cm centrifugal contactor were used to evaluate an alkaline-side CSEX process for cesium removal from SRS tank waste. Based on the results of these tests, a new solvent was developed. Using the D_{Cs} values for the new solvent and the hydraulic performance and stage efficiencies seen with the original solvent, a new flowsheet was developed. The flowsheet calculations were guided by a new definition for process robustness that should ensure good operation in plant-scale centrifugal contactors over the normal range of process perturbations. Because of the high cost of the solvent, the conceptual design of a recovery system using process diluent was outlined. This system, which still needs to be tested experimentally, should keep solvent costs at a reasonable level. While further work needs to be performed, especially in the area of solvent degradation and polymerization, the work presented here shows that the Cs7SBT/150LT solvent is a very attractive candidate for removing and concentrating cesium from the SRS alkaline-side tank wastes.

The three multistage tests reported here, as well as similar tests done previously, indicate that stage efficiencies for multistage 2-cm contactors range from 60 to 90% with the lower efficiencies occurring as O/A flow ratios move away from 1.0. This effect is attributed to the slug flow of the liquids in the interstage lines. Larger laboratory and plant-scale units, which have higher flow rates, do not exhibit slug flow in the interstage lines. Thus, for larger centrifugal contactors, stage efficiencies in multistage operation will be the same as that for a single stage, that is, 95 to 100% in a well-designed contactor.

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