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HYDRAULIC PERFORMANCE AND MASS TRANSFER EFFICIENCY OF ENGINEERING SCALE CENTRIFUGAL CONTACTORS

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Annular centrifugal contactors (ACCs) are being evaluated for process scale solvent extraction operations in support of Advanced Fuel Cycle Initiative (AFCI) separations goals. Process scale annular centrifugal contactors have the potential for high stage efficiency if properly employed and optimized for the application. Hydraulic performance issues related to flow instability and classical flooding in mini-contactors are likely less prevalent, especially for units with high throughputs. However, annular mixing increases rapidly with increasing rotor diameter under similar operating regimes.

For large contactors, elevated rotor speeds and/or throughput rates, can lead to organic phase foaming at the rotor discharge collector area. Foam buildup can aspirate vapor from the contactor housing resulting in a complete loss of separation equilibrium. Proper venting of larger contactors is required to balance pressures across individual stages and prevent vapor lock due to foam aspiration.

Testing of commercial 5 and 12.5 cm contactors to determine two phase hydraulics and operating ranges on several solvent pairs was completed. Evaluation of performance on the extraction, scrub, and strip sections of a Transuranic Extraction (TRUEX) process demonstrated good phase separation within the operating range. Mass transfer testing using stable cerium and europium was completed providing single stage extraction efficiencies above 95%.

I. INTRODUCTION

Centrifugal contactors are being evaluated by performing liquid-liquid solvent extraction technology development experiments to support the selective partitioning of actinide, lanthanide, and fission products, from dissolved Light Water Reactor (LWR) nuclear fuel solutions. Solvent extraction processes utilizing centrifugal contactors realize several important advantages over traditional extraction equipment. First, the units are capable of high throughputs requiring less height or smaller footprint when compared to pulse columns or mixer settlers. Secondly, the short residence times associated with contactor processing minimizes negative solvent effects such as hydrolysis and radiolysis when processing nuclear fuel solutions. Lower in process volume is required which requires less time to process equilibrium. However, shorter residence times and differences in mixing dynamics can impact mass transfer rates; hence, the efficiency of various sized contactors must be experimentally measured and their operating conditions defined.

Commercialization of annular centrifugal contactor technology in the U. S. began eleven years ago with the technology transfer of a patent from the Department of Energy's Idaho National Engineering Laboratory.[1] Since that time, a number of design enhancements have been made and patented that led to a device better suited for a wide range of liquid-liquid processes. Multiple sizes were designed to provide total throughput ranging from 0.2 to 800 liters per minute and interchangeable heavy phase weir rings were incorporated into the rotor to allow separation of a wide range of density pairs.[2, 3] A low-mix sleeve was added to aid in the separation of viscous and/or shear sensitive liquid mixtures.[4] Clean in place (CIP) capability was achieved by adding a hollow central shaft with spray nozzles to the rotor design to enhance use as a clarifier and to improve utility in both hands-on and remote applications.[5]

Commercially available, mass produced, contactors are currently utilized in a wide range of separations processes in both the industrial and government sectors. Evaluation of these mass-produced units in support of the various nuclear fuel cycle and radioactive waste management goals is continuing at the Idaho National Laboratory (INL) and other national laboratories within the DOE complex.

Hydraulic and mass transfer test results for the CINC V-02 (5 cm) model unit, including evaluation of the low mix and high mixing options, were reported in September 2005.[6] The Clean in Place (CIP) test results for a CINC V-05 (12.5 cm) model unit were reported in June 2006 followed by the hydraulic and reliability test results for the 12.5 cm unit reported in September 2006.[7,8] Additionally, the design of a system based on V-02 contactors, modified for remote installation in a radiation facility, is currently being pursued. Most recently the mass transfer testing of a single stage V-05 unit for the TRUEX process was completed. [9]

II. PURPOSE AND SCOPE

Hydraulic and mass transfer testing of candidate flowsheets employing pilot and production scale annular centrifugal contactors is being accomplished to supply important data to the design of future AFCI process facilities. Such specific information as process specific maximum and minimum flow rates and mass transfer data for each size unit is important input to guide process design.

In support of these goals, efforts were focused upon the selection, purchase, and installation of commercially available 5 cm and 12.5 cm ACC s into a pilot scale testing facility at the INL. The contactor test lab is located at the IEDF Facility in space W-4. Testing in this facility included hydraulic and non-radioactive elemental mass transfer performance under varied conditions. In addition, contactor design modifications to further enhance AFCI applications is being evaluated including performance testing of remote handled versions of the contactor equipment. This report will focus on the performance evaluation of the commercial 5 and 12.5 cm ACCs with regard to simple two-phase flow parameters and then progress to hydraulic and mass transfer testing of candidate separations processes.

III. EQUIPMENT DESCRIPTION

All testing was performed on commercially available 5 cm and 12.5 cm diameter rotor ACC s, Model V-02 and V-05, purchased from CINC Manufacturing, Carson City, NV.[10] Both models had incrementally interchangeable heavy phase weirs to allow centering of the interface within the rotor under various operating conditions. Variable frequency drives provided excellent rotor speed control.. The units were also equipped with optional clear polymer housings to aid in observing mixing, flooding, and discharge of separated liquid phases.

Masterflex peristaltic tubing pumps, Cole Parmer System Model 7553-70, were used to supply feeds to the 5 cm contactors. Two magnetic drive Micropump Series 5000 gear pumps with individual frequency drive controllers fed solutions to the 12.5 cm contactor inlet ports. A picture of the laboratory test assembly for the contactors is shown in Figure 1.

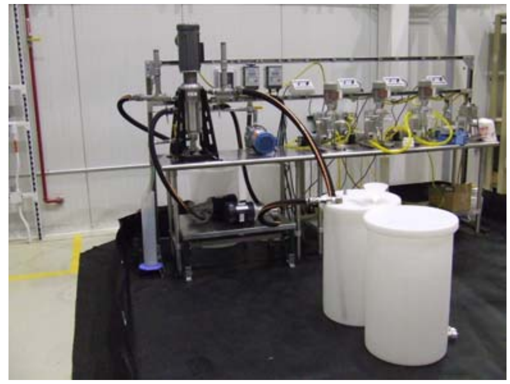

Fig 1. Centrifugal contactor test assembly

IV. EXPERIMENTAL AND RESULTS

IV. A. Lamp Oil/ Water Tests

Two-phase hydraulic testing was initially accomplished using tap water and commercial grade of blue dyed lamp oil. Lamp oil is a blend of normal paraffin hydrocarbons, from C_{14} to C_{16}. The color aids in the observing of mixing in the contactor annulus and also in seeing carryover in separated samples. Its physical and chemical properties, provided in Table 1, make it convenient and safe to use for basic hydraulic contactor performance studies.

TABLE 1. Lamp Oil Properties

Specific Gravity 0.773@ 16^0 C
Viscosity: 2.5-2.7 cSt @ 40^0 C
Vapor Pressure (mm Hg): 0.05 @ 20^0 C
Flashpoint: 121^0 C
Solubility in water: negligible
Odor: very mild hydrocarbon

Initial tests of the 5 cm ACC were conducted with the 0.975” aqueous (heavy phase) weir to determine the throughput maximum for this size rotor and liquid-liquid phases. An organic/aqueous (O/A) ratio of 1 was selected and total flow rates of 1.6 to 3.0 L/min were studied. Good phase separation was observed up to 2.5 L/min but 3% aqueous carryover into the discharged organic was seen at 3 L/min. The rotor speed was set at 2990 rpm and no visual indications of annular flooding, mixed phases rising up to contaminate the light phase collector, were observed even at the maximum feed rate. The measured rotor volume varied from 166 to 176 mL, total of organic and aqueous volumes. The variance is primarily due to inconsistent feed liquid hold-up in the bottom vane area when flow to the contactor is terminated. The separation residence time, defined as rotor volume divided by feed rate, for this test varied from 6.4 to 3.4 seconds. Annular mixing residence times estimated at 2-3 seconds.

Two adjacent sized weirs were tested, 0.925 and 1.025" diameter, at O/A ratios of 0.2 to 4 at 2990 rpm and a constant feed flow rate of 2 L/min. No other phase carryover was observed during this testing. Further testing with a 0.975" weir at varied rotor speed, O/A ratio, and increasing flow rates yielded only slight carryover of aqueous in the organic at 2.5 L/min and rotor speeds of 2000 to 4000 rpm. Three percent aqueous carryover was observed at 3 L/min flow rate under most conditions. No significant carryover of organic into the aqueous discharge was observed, however. A close up view of the 5 cm contactor with clear housing during lamp oil/water testing is shown in Figure 2.

Fig. 2. 5 cm Clear Housing Contactor View

The initial testing of the 12.5 cm contactor with lamp oil/water established an operating range by running total flow rates of 2L/min to 24 L/min at an O/A of 1 and a rotor speed of 2000 rpm. The point at which other phase carryover was observed gives good indication of the maximum amount of solution that can be processed with the V-05 unit when equipped with a proper diameter heavy phase weir. A calculation to center the interface between the light phase weir and the heavy phase underflow of the rotor was used to select the 2.600" weir at an O/A ratio of 1.

An unexpected observation occurred at the start of the testing with the clear polymer housing. A small fraction of the organic discharge from the rotor was observed cascading down the interior walls of the clear polymer housing at the initial 2 L/min total flow rate. This phenomenon decreased until it was not visible at the 10 L/min total throughput level.

After disassembly of the ACC, it was noted that the rotor discharge slots for the light phase effluent were aligned approximately ¼ inch below the center of the light phase collector ring opening. This misalignment allows the light phase discharge stream to partially contact the angled lip of the collector ring causing some of the solution to not be collected and gravity flow back down into the mixing annulus area. This misalignment of the light phase discharge slots results in a small recycle of light phase flow to the mixing annulus at low flow rates. It was not considered a significant problem and hydraulic testing was continued as planned. The discharge slots in the rotor for the heavy phase effluent were perfectly centered with the heavy phase collector ring opening.

Other phase carry over, aqueous in the organic, was observed at higher throughput levels, 0.5% at 22 L/min and 2.5% at 24 L/min. At most, only a slight sheen of organic was observed on the top of aqueous samples at 20 L/min and higher throughputs. The maximum V-05 flow rate for lamp oil/water without measurable carryover at an O/A of 1 is 18 L/min at 2000 rpm. The mixing height in the annulus increased from 1 to 3.5" while varying total throughput from 2 to 20 L/min. Views of the annulus height at two flow rates are shown in Figures 3 and 4.

Fig.3. Mixing Annulus in V-05, 2L/min.

Fig.4. Mixing Annulus in V-05, 20L/min.

The V-05 rotor volume was also measured by turning off the feed, stopping the rotor, and draining the mixture from the unit. Measured values were 0.84 L organic and 1.86 L aqueous, for a total of 2.7 L, including a small amount of liquid remaining in the bottom mixing plate of the housing. Calculated rotor volumes of 0.75L and 1.95 L totaling 2.7 L are in good agreement with measurements.

Once the maximum throughput for lamp oil/water with the 2.600 inch weir was determined, the next step was to examine the operating range for the V-05 at varied O/A ratios and rotor speeds. Total flow rates of 10, 12, and 16 L/min were selected. Various O/A ratios ranging from 0.2-5.0 were tested with four rotor speeds of 1500, 2000, 2500, and 3000 rpm.

The majority of the test conditions indicated acceptable phase disengagement and less than volumetrically measurable phase carryover. A flow rate of 16 L/min at 3000 rpm and at an O/A ratio of 0.2 was the only condition that provided a measurable carryover of A in O. Higher rotor speeds provided clearer phase samples especially at O/A >0.5. A in O was the predominant carryover observed for this testing matrix. The organic level present on the surface of the aqueous phase samples was too small to visually quantify throughout the lamp oil/water testing.

IV.B. UREX Solvent Tests

The next goal was to characterize 30% TBP/n-dodecane (UREX) solvent/ dilute nitric acid two phase hydraulic performance over a wide range of V-05 operating conditions. Sampling during testing using these solvents required a modified sampling technique from that used for lamp oil/water tests. All sampling during UREX testing was performed using the 3/8" diameter sample taps installed on both effluent lines as shown in Figure 6. The volume of sample taken was normally 70-100 mL of each phase. Carryover results obtained from the sample taps were periodically verified versus 1-2 L samples taken directly from outlet discharge hoses.

The first testing performed was to determine maximum throughput of the two-phase UREX solvent/nitric acid system following the same test scheme as was completed for the lamp oil/ water maximum throughput testing. Flow rates of 14-26 L/min at an O/A of 1 at 2000 rpm were tested. An observation of abnormal operation occurred at high flow rates that led to a modification to our test assembly. Significant phase carryover was observed in both effluent samples at a total flow rate of 24 L/min. At 26 L/min increased carryover was measured and cascading of light phase solution was observed running down the clear polymer housing from the collector ring area. Moreover, intermittent phase discharge flow was seen and belching sounds were heard coming from the contactor outlets.

These tests showed that, at high throughputs, foam formation in the light phase collector ring and outlet caused vapor lock resulting in flooding of one or both collector rings resulting in severe other phase carryover. The CINC operating manuals suggest that large diameter vents be installed in the outlet lines to a height of 12" to prevent such occurrences. They suggest that outlet hoses or pipes not be submerged in the collection tanks for the same reason. In cases of severe foaming it is also suggested than an inlet should also be vented in similar fashion, to allow added vapor into the housing.

Vents (1 ¼" dia. x 12" h) were then added to each V-05 outlet, as near as possible to the contactor discharge. Following addition of the vents, further throughput testing of the UREX two-phase system was completed. Flow rates of up to 28 L/min at an O/A of 1 were completed without measurable carryover observed in either phase effluent. No further evidence of vapor lock or abnormal operation was observed following addition of the outlet vents at tested flow rates up to 32 l/min.

Testing of the UREX solvent/nitric acid couple at 2000 RPM continued at various O/A ratios and flow rates. Less than measurable carryover levels at flows up to 28 L/min were observed for extraction section O/A ratios of 1,2,3; scrub ratios of 4,6,8; and strip ratios of 0.5,0.7, and 1.0.

IV.C. TRUEX Solvent Testing

Hydraulic testing of the TRUEX solvent, 0.2M CMPO, 1.4M TBP in n-dodecane was done in the 5 cm V-02 contactor, weir 0.975, following individual pre-equilibration for the extraction, scrub, and strip sections. Rotor speeds of 2500-4500 rpm were examined at various feed flow rates.

First, TRUEX solvent/2.5 M nitric acid at an O/A ratio of about 0.5 was tested for the extraction section. Foaming at the light phase collector ring was observed at rotor speeds of 3500 rpm and higher for all tests. However, no abnormal performance, vapor lock or flooding, of the V-02 was seen. Phase separation was excellent up to 1.5L/min. at all rotor speeds. Measurable A in O carryover occurred at 2L/min, starting at 5% at 2500 rpm and decreasing to 1% at 4500 rpm. Foaming in the light phase collector ring was seen at 2 L/min for all rotor speeds tested. Foam formation appears to be more prevalent at higher flow rates and also at higher rotor speeds.

Scrub tests using dilute nitric acid at 1 and 1.5 L/min and O/A ratios of 1 and 2 gave satisfactory phase separation at rotor speeds from 2500 to 4000 rpm. Another scrub test employing nitric acid oxalic acid at an O/A of 0.8 and 1 L/min was also successful at all tested rotor speeds.

Strip solutions consisting of lactic acid and DTPA were processed through the V-02 at various rotor speeds and several flow rates. The results are presented in Table 2.

TABLE 2. TRUEX Strip Testing Results

RPM	Flow (L/min)	O/A	Carryover
2500	1.0	1.2	2%O in A <0.5%A in O
3000	1.0	1.2	slight O in A <0.2%A in O
3500 `	1.0	1.2	slight O in A <0.1%A in O
4000	1.0	1.2	No O in A <0.5%A in O
2500	0.6	1.3	slight O in A 0.2% A in O
3000	0.6	1.3	No O in A 0.2% A in O
3500	0.6	1.3	No O in A 0.2% A in O
4000	0.6	1.3	No O in A 0.5% A in O

It is apparent from this data that the TRUEX strip has the longest disengagement time in this process. As such, increased rotor speed aids in reducing the carryover levels of aqueous in the organic but only to a point. At 4000 rpm the trend reverses indicating over mixing may be occurring. Loss of extractant to the aqueous phase, however, is low under almost all tested conditions.

IV. D. TRUEX Mass Transfer Testing

IV.D.1. V-02 Tests

A total of 1.4 L of TRUEX solvent was prepared, purity checked via the ^{241}Am extraction method,[11] carbonate washed, and subsequently pre-equilibrated with 3.2 M nitric acid prior to use. For the extraction test, an O/A ratio of 0.5 and total flow of 1 and 1.4 L/min were fed into the contactor operating at both 3000 and 4000 RPM. The aqueous feed solution was 3.3 M nitric acid containing 1.02 g/L and 0.24 g/L of stable cerium and europium, respectively. Temperatures of the discharged phases were 22- 23^{0}C.

Samples of the discharged phases were collected while at operating equilibrium, after displacing at least 3 rotor volumes at start-up. After each operating parameter change, equilibrium was reestablished by waiting three rotor volumes before sampling again. Samples were analyzed by ICP-MS by direct injection of the aqueous samples. Organic samples were stripped of Ce and Eu, the strips combined, and then analyzed.

The loaded organic from the extraction section was thrice scrubbed using dilute nitric acid at an O/A of 1.9 in discreet passes at a total flow rate of 0.7 L/min. and at 3000 rpm. No samples were taken for the scrub section.

Cerium and europium were stripped from the solvent by a solution of lactic acid containing DTPA at an O/A of 1.3 and flow rates of 0.6 and 0.8 L/min. and at both 3000 and 4000 rpm. As in the scrub section, flow rates selected for this section testing were determined by the highest carryover free organic flow rate found the extraction section. Samples of the discharged phases were taken and analyzed as in the extraction section testing.

Mass transfer efficiencies for extraction and strip section test parameters were calculated using the ICP-MS sample results taken from each test parameter and the re-equilibrated samples. The mass transfer equation;

$$\eta = \frac{(X - X_{in})}{(X_{eq} - X_{in})} * 100 \qquad (1)$$

where X is the metal concentration of the effluent, X_{in} is the inlet metal concentration, and X_{eq} is the metal concentration of the effluent following re-equilibration was employed. Efficiency can be calculated based on aqueous or organic phase concentrations. Aqueous phase concentrations were typically used for all extraction calculations and organic phase concentrations for strip calculations since these phases contained the Ce and Eu.

V-02 stage efficiencies for Ce and Eu extraction ranged from 93-99%, with the higher efficiencies observed at 4000 rpm. Stage efficiencies for the Ce and Eu strip test ranged from 95-99%. However, no clear trends were seen based on total flow rate or rotor speed for the strip section.

IV.D.2. V-05 Tests

V-05 TRUEX mass transfer tests were conducted in similar fashion to those for the V-02 contactor. However, only one dilute nitric acid scrub contact was used prior to stripping the Ce and Eu from the loaded organic in the V-05 testing. We determined that a significant amount of the extracted Ce and Eu was lost during the three discreet scrubs used in the V-02 tests. Also, a second strip was conducted and sampled to evaluate the mass transfer efficiency of both the first and second stages of the strip section. This evaluation was done in two separate tests to study a variety of process parameters and allow for sampling in each experiment.

Samples were taken at each set of parameters after allowing a short equilibration time equal to about 3 rotor volumes or about 9 L, total throughput. Samples of about 70-100 mL were taken for each phase and examined for visual carryover before archiving for analysis. There was no observable other phase carryover in any of the collected samples at the time of collection. A small droplet of aqueous was found in the bottom of organic samples upon standing. The process temperature during this test was 21-22^{0}C. Note that at lower process temperatures, <18^{0}C, phase disengagement slows and small percent level aqueous entrainment is often found in the TRUEX organic output, especially at high throughputs.

V-05 extraction test parameters were studied at four flow rates; 3, 5, 10, 15 L/min at an O/A of 0.5 and two rotor speeds, 1750 and 2250 rpm. Scrubbing of the loaded TRUEX organic was a single contact dilute nitric acid at an O/A of 1. No scrub samples were taken. Both strip tests were studied at four flow rates; 2.5, 5.7, 8.5, 13 L/min at an O/A of 1.3 and at 1750 and 2250 rpm.

Forward distribution coefficients for Ce and Eu in the extraction stage ranged from 13 to 19 at all throughputs and rotor speeds tested. Trends indicate that the higher rotor speeds and throughput levels tested had higher distributions. The first strip stage distribution results were different for Ce and Eu. Distributions for Ce ranged from 1.4 to 0.5 while those for Eu were measured at 0.8 to 0.1. In every set, Eu stripping was at least twice as effective as Ce. Overall, the strip distributions trend positively, i.e. better stripping, with higher rotor speed and throughput. However, higher efficiency stripping was expected in the first strip stage based on modeling.

Measured acid concentrations of the scrubbed organic entering the strip section were slightly higher than those predicted by the AMUSE model. A single scrub contact may be insufficient to remove enough acidity to allow efficient stripping in the first stage. For this reason, a second stage strip study was conducted.

The TRUEX solvent was carbonate washed after the first strip study and recycled for another bulk extraction, scrub, and first strip process to prepare it for the second stage strip study. Fresh Ce and Eu feed was prepared for the second TRUEX solvent loading and fresh scrub and strip solutions were used. The extraction, scrub, and first strip process was repeated, but with only a few bulk process verification samples, and once stripped organic was collected for the second strip stage testing. For the second strip stage test, the same parameters as for the first stage strip were repeated and each set sampled.

Distribution coefficients for the second strip stage were measured from .003 to .001 for Ce and .004 to .002 for Eu for all test parameters. No trends were observed due to flow rate or rotor speed as very efficient stripping occurred in this set of tests.

The mass transfer efficiencies for the V-05 were calculated for the extraction, first strip stage and second strip stage. For all parameters studied, the forward extraction efficiency was essentially 100%. The first strip stage efficiencies ranged from 75 to 93% with the lowest stripping at the lowest, 2.5 L/min, flow rate. The second stage strip mass transfer efficiency was also essentially 100%, even for the lowest flow rate studied.

V. SUMMARY AND CONCLUSIONS

Hydraulic testing of annular centrifugal contactors for use in pilot and production processes provides valuable data and guidance for plant and process design. Each flowsheet section used for elemental partitioning contains unique solvents that need to be evaluated on the desired size ACC prior to radioactive testing. This preliminary optimization provides a range of operating parameters, verifies unit efficiency, and thus provides focus, reliability, and confidence that subsequent radioactive testing will be successful.

Operating ranges and maximum throughput levels for simple two phase systems, UREX solvents and TRUEX solvents have been established. Testing of 5 and 12.5 cm rotor commercial contactors demonstrated the differences that are obtained when evaluating such operating hydraulics as maximum throughput and foam formation for different organic solvent systems. Due to the increased mixing obtained with large-scale contactors, phase separation becomes one of the most important efficiency issues. Over mixing can cause difficult to resolve dispersions or even emulsions that must be avoided in processes requiring high decontamination factors. The use low mixing units may be useful for terminal stages and/or solvent recycle processes. Overall, within the established operating range, carryover of aqueous entrainment into the organic phase was < 0.1 % and organic carryover in all aqueous phase samples was about an order of magnitude lower. Thus, solvent loss to the aqueous streams, an expensive and waste intense issue, was quite low under a wide range of operating conditions.

Mass transfer efficiency for a single stage 5 cm V-02 contactor was greater than 93 % in all cases and did not change measurably over the throughput range of 50-75% of hydraulic maximum for the TRUEX process. Likewise, most tested rotor speeds did not affect performance, either in terms of mass transfer or phase separation. Selection of the optimum rotor speed for a given unit size and throughput should be governed by the lowest rpm that provides good phase disengagement and mass transfer, a function of mixing. Over mixing does not lead to efficient separations due to increased carry over between phases.

Two mass transfer tests were performed using a single stage commercially available 12.5 cm V-05 centrifugal contactor. Mass transfer efficiency measurements of ≥ 98 % for both Ce and Eu in the TRUEX extraction section indicated the commercial 12.5 cm contactor is also a very capable tool.

The first strip contact mass transfer efficiencies were lower than those determined for the extraction section. Efficiencies determined for Ce and Eu ranged from 75 to 93% increasing slightly with higher rotor speed and throughput. Overall, the rotor speeds tested had little impact on efficiency or carryover for all experimental parameters.

A second mass transfer test was performed to measure distributions and mass transfer efficiencies of Ce and Eu in the 2nd strip stage. Ce and Eu distribution coefficients decreased dramatically compared to the first stage values. Mass transfer efficiencies for all 2nd strip stage test conditions were > 99 % for both Ce and Eu.

High single stage mass transfer efficiency and a good hydraulic operating range for both 5 and 12.5 cm centrifugal contactors makes this technology a viable option for solvent extraction processes in the advanced fuel cycle. However, in designing robust multi-stage processes, one must not design to operate at throughput maximums. Testing indicated that process throughputs of 50-70% of contactor maximum provides reliable separation efficiency, even over a wide range of operational variables. Annular centrifugal contactors have a very good turn down, that is, they operate well even at 10% of maximum flow. Mixing at low flows may not be optimal but added stages can be used to achieve desired results. Rotor over speed can lead to loss of performance due to over mixing, especially in large contactors.

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