INL/CON-07-12942
PREPRINT

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ISEC 2008

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September 2008

HYDRAULIC AND CLEAN-IN-PLACE EVALUATIONS FOR A 12.5-CM ANNULAR CENTRIFUGAL CONTACTOR AT INL

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ABSTRACT

Commercially available, Annular Centrifugal Contactors (ACC) are currently being evaluated for processing dissolved nuclear fuel solutions to selectively partition integrated elements using solvent extraction technologies. These evaluations include hydraulic and clean-in-place (CIP) testing of a commercially available 12.5 cm unit. Data from these evaluations is used to support design of future nuclear fuel reprocessing facilities.

Hydraulic testing provides contactor throughput performance data on two-phase systems for a wide range of operating conditions. Hydraulic testing results for a simple two-phase stable kerosene (oil)/water system followed by a 30 % tributyl phosphate in N-dodecane (UREX)/nitric acid pair are reported. Maximum total throughputs for this size contactor ranged from 18 to 32 L/min without significant other phase carryover.

A relatively new contactor design enhancement providing Clean-in-Place capability for ACCs was also investigated. Spray nozzles installed into the hollow central rotor shaft enable the rotor internals to be cleaned, offline. Testing of the solids capture of a diatomaceous earth/water slurry feed followed by CIP testing was performed. Solids capture efficiencies of > 95% were observed for all tests and short cold water cleaning pulses proved successful at removing solids from the rotor.

INTRODUCTION

The United States Department of Energy's Global Nuclear Energy Partnership (GNEP) was formed to expand economical, carbon free nuclear energy to meet growing electricity consumption demands. The development of advanced separation technologies to close the fuel cycle by processing dissolved nuclear fuel solutions while promoting nuclear non-proliferation is an integral part of this partnership.

Advanced separation technologies include the selective partitioning by solvent extraction (SX) of bulk uranium for reuse in nuclear fuel fabrication and minimizing heat generating fission products sent to a repository. Annular centrifugal contactors (ACC) have important advantages over historically used equipment. First, the units are capable of processing plant scale throughputs with a smaller in-process volume compared to pulse columns or mixer settlers. Secondly, the short resonance times using ACCs minimizes solvent effects such as hydrolysis and radiolysis. In addition, ACCs will tolerate wide ranges of organic/aqueous (O/A) ratios and can maintain separation equilibrium during flow interruptions. However, shorter resonance times and differences in mixing dynamics can impact mass transfer rates; hence, the hydraulics and total throughputs of these contactors must be experimentally measured to define operating conditions. One potential disadvantage is the inherent ability of contactors to efficiently separate entrained particulates from process feed streams. These particulates can be captured in the separation zone, and over time, form cake-like layers which can significantly impede phase separation and ultimately reduce process performance. Clean-in-place (CIP) capability was added by using a hollow central rotor shaft fitted with spray nozzles to wash solids from rotor internals, offline. A second disadvantage is the complexity of the units, each constructed with numerous moving components which may require additional maintenance in a radioactive operating environment.

This paper discusses hydraulic testing results for two independent two-phase systems; kerosene (Oil)/ water, and 30 % tributyl phosphate in N-dodecane (UREX)/nitric acid. The paper also includes results from a CIP test reporting solids capture and cleanout using a 0.1 wt. % diatomaceous earth in water slurry feedstock.

EXPERIMENTAL

Equipment Description for Hydraulic and CIP Evaluations

The experimental setup for both tests consisted of a commercially available model V-05 CINC annular centrifugal contactor with a 12.5 cm (5 inch) diameter rotor fitted with a 2.500 inch heavy phase weir. Additional heavy phase weirs are available from 2.300 to 3.000 inches in 0.050 in. increments. It was also fitted with an optional clear polymer lower housing to view two-phase mixing and measure mixing annulus height during operation.

A Seepex® progressive cavity pump (BN#2) was used to pump the diatomaceous earth slurry to the contactor inlet for CIP testing. A Gould G&L series centrifugal pump was installed to pump cleaning solution at 40 psig and flow rates of 25 L/min to the spray nozzles.

Stable Kerosene (oil)/water hydraulic testing

The initial testing focus was to examine two phase mixing and determine the maximum throughput 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 is observed to be measurably present gives good indication of the maximum amount of solution that can be processed with the V-05 unit equipped with any chosen heavy phase weir. Three weir sizes were tested to help define the best fit for the overall testing objective. The 2.600 inch weir was chosen based on final rotor volumes measured at selected test conditions.

To further examine the operating range for the V-05, total flow rates of 10, 12, and 16 L/min, O/A ratios ranging from 0.2 to 5.0, and rotor speeds of 1500, 2000, 2500, and 3000 rpm were tested. Samples were visually inspected for phase carryover at each set of parameters.

30% Tributyl phosphate in N-dodecane/nitric acid hydraulic testing

The maximum throughput for the TBP solvent/nitric acid pair was experimentally determined following the oil/water test scheme. Flowrates of 14-26 L/min at an O/A of 1 at 2000 rpm were tested. Stated flowrates were within 5 %, based on previous measurements at pump controller settings.

Following the initial throughput testing, two equipment modifications were made to the contactor. Vents (1¼ in. I.D. x 12 in. ht.) were installed as shown in Figure 1 on both phase outlets to prevent vapor lock that leads to excessive other phase carryover at high throughput rates. Sample taps were also installed on both discharge lines to facilitate safe and convenient performance monitoring.

A simplified flowsheet was employed consisting of an extraction, scrub, and strip section. The extraction section was performed at O/A ratios of 1, 2, and 3 at an initial rotor speed of 2000 rpm. Beginning at a total flowrate of 16 L/min, the flowrate was increased by 2 L/min intervals until measurable either phase carryover was observed in the samples. At that point, the rpm was increased to 2250 and finally to 2500 rpm to determine the impact on carryover. The scrub section was performed at O/A ratios of 4, 6, and 8 each beginning with a rotor speed of 2000 rpm and an initial total flowrate of 16 L/min. Again, the rotor speed was increased once measurable carryover was observed in phase samples. The strip section was performed at O/A ratios of 0.5, 0.7, and 1.0 following the same test scheme as done for the extraction and scrub sections.

Clean-in-Place (CIP) Evaluation

The initial phase of the CIP testing was to determine percent capture of solids in the contactor rotor by processing a 0.1 wt. % diatomaceous earth feed slurry at flowrates ranging from 1.9 L/min to 11.4 L/min at rotor speeds of 1750 to 3500 rpm. The 0.1 wt. % feed slurry was prepared by adding diatomaceous earth (mean particle size 80 micron and a density of approximately 2.4 g/mL) to normal tap water under agitation. Diatomaceous earth was not selected as a surrogate for fines normally encountered in nuclear fuel reprocessing but rather as a non-hazardous material for a first time CIP scoping evaluation. Before testing began, a feed slurry sample was analyzed for solids concentration. During testing, four liter samples were collected at each test condition then vacuum filtered through Nalgene 0.2 micron (CN) disposable filters. Percent capture was calculated from the following equation:

$$\text{Percent Capture} = [1 - (\text{solid conc. in sample} / \text{solid conc. in feed slurry})] * 100\ \%$$

The CIP evaluation was initiated by pumping the feed slurry to the contactor at a flowrate of 7.5 L/min, the rotor speed was set to 2300 rpm. After the feed slurry (~ 110 L) had been pumped through the contactor, the pump was turned off and the contactor rotor was shut down. The drain valve was opened slowly to minimize solids loss during draining. The contactor was then carefully disassembled to observe collected solids. Photographs of the solids captured in the rotor and vane package are shown in Figures 1 and 2.

After cleaning the rotor and reassembly, loading tests were repeated for two CIP tests using water as the cleaning solution. The rotor was stopped, slowly drained, then with the drain valve open, water was pumped through the spray nozzles at 25 L/min and

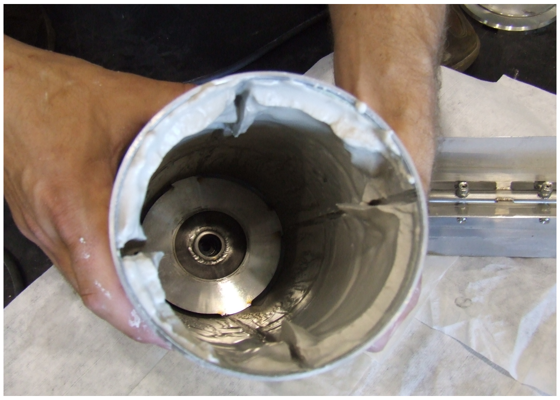

Figure 1. Caked solids on rotor interior.

Figure 2. Solids collected in vane package.

40 psig for 10 and 15 seconds, three times, while visually monitoring the discharged fluids. After each third wash pulse, the effluent appeared free of solids. The contactor was again disassembled and inspected.

RESULTS

Hydraulic Evaluations

The maximum V-05 throughput for all solvent pairs was noted as the final total flowrate tested where either phase carryover was not measurable and defined to be < 0.1 % aqueous in organic (A in O) or organic in aqueous (O in A). Table 1 includes the maximum throughput obtained for (oil)/water and 30%TBP/1M nitric acid pairs and is denoted with an asterisk.

Table 1. Maximum throughput without vents at O/A of 1 and rotor speed of 2000 rpm.

(Oil)/water		**30% TBP/1 M nitric acid**	
Flowrate (L/min)	Carryover	Flowrate (L/min)	Carryover
2-16	Not measurable	14-20	Not measurable
18*	Not measurable	22*	Not measurable
20	0.3 % A in O, A is ok	24	0.2 % A in O, 4 % O in A
22	0.5 % A in O, A is ok	26	3 % A in O, 12 % O in A

Maximum throughputs for both solvent pairs agree favorably with the manufacturers rating of 20 L/min. The throughputs listed in Table 1 were measured before the vents were installed on discharge lines. Carryover determinations were then continued at changing flowrates, rotor speeds, and O/A ratios again with (Oil)/ water. Flowrates of 10, 12, and 16 L/min were tested with rotor speeds of 1500, 2000, 2500, and 3000 rpm and at O/A ratios of 0.2, 0.5, 1.0, 2.0, and 6.0. Overall, phase samples from this entire test matrix revealed < 0.1 % phase carryover excluding one test condition; at 16 L/min with a rotor speed of 3000 rpm at an O/A ratio of 0.2.

30% TBP flowsheet testing provided maximum throughput carryover results for changing flowrates, rotor speeds, and O/A ratios for three different solvent/nitric acid pairings. The test matrices started at 16 L/min at 2000 rpm and increasing flowrate at 2 L/min intervals. Rotor speed was increased when carryover was measured to examine rotor speed increase impact on carryover. Tables 2, 3, and 4 include the carryover measurements for the three solvent pairs at respective O/A ratios tested.

Table 2. 30% TBP Extraction section tests.

O/A = 1		O/A = 2		O/A = 3	
Flowrate (L/min)	Carryover	Flowrate (L/min)	Carryover	Flowrate (L/min)	Carryover
16-30	Not measurable	16-34	Not measurable	16-28	Not measurable
32	Not measurable	36	Not measurable	30-34	0.5 % A in O, A is OK

Table 3. 30% TBP Scrub section tests.

O/A = 4		O/A = 6		O/A = 8	
Flowrate (L/min)	Carryover	Flowrate (L/min)	Carryover	Flowrate (L/min)	Carryover
16-30	Not measurable	16-28	Not measurable	16-26	Not measurable
32	Not measurable	30	Not measurable	28	Not measurable
34	0.3 % A in O, A is OK	32	0.7 % A in O, A is OK	30	0.1 % A in O, A is OK

Table 4. 30% TBP Strip section tests.

O/A = 0.5		O/A = 0.7		O/A = 1	
Flowrate (L/min)	Carryover	Flowrate (L/min)	Carryover	Flowrate (L/min)	Carryover
16-20	Not measurable	16-24	Not measurable	16-30	Not measurable
22	Not measurable	26	Not measurable	32	Not measurable
24	0.1 % A in O, 0.3 % O in A	28	O is ok, 0.5 % O in A	34	1.3 % A in O, 0.6 % O in A

It is clear that the addition of the vents on the discharge lines resulted in a dramatic increase of throughput for the 30% TBP solvent/nitric acid pair in comparing carryover at an O/A ratio of 1. In all cases, increased rotor speed reduced overall carryover at maximum throughput. However, over mixing associated with increased rotor speeds can occur so care must be used when applying this technique.

Percent Capture and CIP Capability

Percent capture results were compiled and are presented in Figure 6. The percent capture result at 1.9 L/min with a rotor speed of 3500 is omitted due to pump failure during testing but is expected to be > 99 % as related to those reported at the three lower rotor speeds.

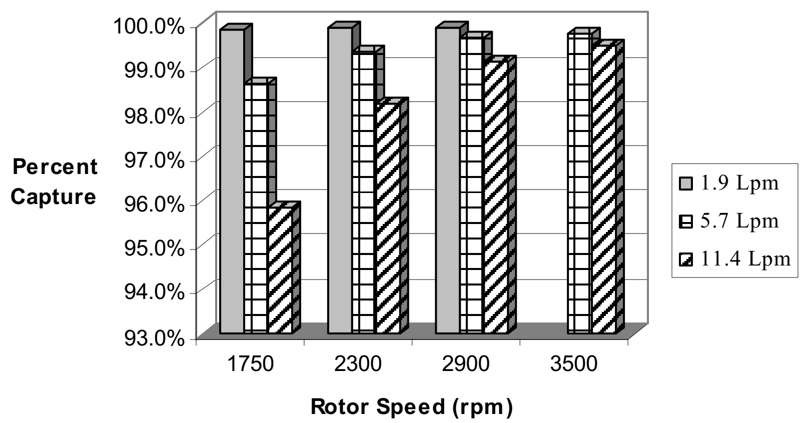

Figure 3. Percent capture as a function of flowrate and rotor speed.

The graph in Figure 3 illustrates that the percent capture is dependent on both flowrate and rotor speed. As expected, as rotor speed is increased, the percent capture increases and conversely, if flowrate is decreased, percent capture increases. Centrifugal contactors equipped with cleaning nozzles are effective at removing captured solids with small wash volumes. Three 10 second or three 15 second pulses performed at 25 L/min and 40 psig effectively cleaned the contactor internals.

CONCLUSIONS

Maximum hydraulic throughputs were confirmed using kerosene/water and 30 % tributyl phosphate in N-dodecane/nitric acid. Test results indicate that maximum throughputs of 18 to 32 L/min., dependant on solution pair characteristics, can be processed with the V-05 contactor without measurable phase carryover. The addition of vents on the discharge lines dramatically increased throughput compared to initial testing without vents.

Annular centrifugal contactors are very effective at capturing solids. Greater than 95 % of solids were captured in the rotor at all conditions tested. The CIP testing indicated captured solids can be effectively removed from the rotor interior using either three 10 second or three 15 second pulses.

A future CIP evaluation with a more representative solids material is recommended.