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Pilot-Scale TRUEX Flowsheet Testing for Separation of Actinides and Lanthanides from used Nuclear Fuel

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Testing of a TRUEX flowsheet has been performed using a thirty-stage, 5-cm centrifugal contactor pilot plant. This testing was performed using a non-radioactive feed surrogate and data were collected and analyzed to evaluate removal efficiencies of the lanthanides, mass transfer efficiency of the lanthanides in the extraction and strip sections of the flowsheet, and the temperature profile of the process solutions throughout the centrifugal contactor pilot plant. Results indicate >99.9% separation for all lanthanides and mass transfer efficiencies typically ranging from 85% to 100%. Energy input into the process under flowsheet conditions resulted in only a moderate temperature rise and did not appear to have a large effect on separations performance. The general process performance is also described.

Keywords actinide extraction; centrifugal contactor; lanthanide extraction; TRUEX

INTRODUCTION

Recent research sponsored by the U.S. Department of Energy Nuclear Energy Fuel Cycle Research and Development Program has resulted in the development of several solvent extraction options for the recycling of used nuclear fuel. These options include a uranium/plutonium extraction process, followed by several processes for the separation of the minor actinides from the used nuclear fuel. The separated minor actinides would be recycled to a nuclear reactor where they would be consumed and, therefore, not require disposal in a geological repository.

One proposed method of accomplishing the separation of minor actinides is to first separate the actinides and lanthanides using the TRUEX process and then separate the actinides from the lanthanides using the TALSPEAK process (1,2). This advanced reprocessing flowsheet has been previously demonstrated in laboratory-scale centrifugal contactors and mixer-settlers using actual used nuclear fuel (2). Flowsheet testing using laboratory-scale contacting equipment introduces several limitations, including inconsistent solution flow resulting in reduced stage efficiencies and the inability to sample individual stage solutions without disrupting steady-state conditions. The reduced stage efficiencies result in the demonstration of flowsheets that have more stages than would be required in a production-scale facility. With pilot-scale contactor equipment (e.g., approximately 4-cm centrifugal contactors and larger), stage efficiencies are expected to be much larger, approaching 100%, and results directly scalable to production-scale equipment. Also, with pilot-scale equipment, individual stage samples can be taken without disrupting steady-state conditions due to the much higher flowrates.

TRUEX flowsheet testing was performed at the Idaho National Laboratory with the goal to evaluate the effectiveness of the separation of actinides and lanthanides from used nuclear fuel using pilot-scale, commercially available, centrifugal contactor equipment. This evaluation was performed using a non-radioactive feed surrogate solution. This testing also was performed to measure stage efficiencies of the pilot-scale centrifugal contactors in the extraction and strip sections of the flowsheet to provide mass transfer efficiency data to support future flowsheet and centrifugal contactor design efforts. Finally, the solution temperature profile throughout the TRUEX flowsheet was measured to evaluate process temperature increases resulting from contactor operation. There can be significant energy input in the contactors resulting from the motor operation, but also a relatively short residence time for the process solutions in the equipment. Testing at this scale is useful in determining the magnitude of temperature rise and its impact on separations performance. These data are helpful in evaluating the need for temperature control during operation.

PROCESS EQUIPMENT DESCRIPTION

The centrifugal contactor pilot plant consists of 30 inter-connected 5-cm centrifugal contactors all mounted on a stainless steel support structure. The centrifugal contactors are off-the-shelf V-02 units manufactured by CINC Industries, Inc. The 30-stage pilot plant is shown in Fig. 1.

The pilot plant consists of thirty 5-cm centrifugal contactors with variable speed drives for each contactor,

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FIG. 1. Thirty Stage centrifugal contactor pilot plant.

five liquid supply pumps, four process solution heaters with controllers, multiple thermocouples, and feed flow meters. The centrifugal contactors are constructed of 316 L stainless steel and utilizes 1.6 cm I.D. chemically compatible Tygon for interconnective tubing.

The contactors have an operating range of 0.1 to 2.0 L/min total throughput (aqueous plus organic solution flowrate) and have a variable rotor speed range of 0 to 6000 rpm. The fan-cooled 1/3 HP motors are XP rated and operate on 208 V three-phase power. Motors are controlled via Yaskawa V7 N frequency drives enabled with DeviceNet^{TM} communication for PC remote control operation.

The pumps employed for the fluid feed systems are double diaphragm chemical metering pumps obtained commercially from Madden Manufacturing, Inc. The rated liquid flowrate range for these pumps is 0.226 to 2.26 L/min with an accuracy of ±5%. The pump heads are made of stainless steel and the diaphragms are made of Teflon®-faced viton. The pumps utilize a $\frac{1}{2}$ HP motor and operate on 110 V single phase power. Motor speed is controlled with a Franklin Electric IDMS controller. The controller is operated with a 0 to 10 VDC analog signal. Turbine type flowmeters were used to measure flow on each feed system and were obtained from FTI Flow Technology^{TM}, Inc. The flowmeters have a flow range of 0.303 to 3.03 L/min with a published accuracy of ±1% using water at 20°C. The flow signal is processed through a Linear Link linearizer and the signal output is communicated using a 4 to 20 mA analog signal. To reduce pulse flow generated from the diaphragm pumps, pulse dampeners and back pressure valves were installed. The pulse dampeners are made by Blacoh Fluid Control^{TM}, Inc. One 164 cubic cm and four 590 cubic cm dampeners were installed in the five fluid feed systems. The larger volume units are much more effective at reducing pulsing flow. The back pressure valves were acquired from Griffco Valve^{TM}, Inc. The back pressure valves provide back pressure on the downstream side of the pumps to assist with pulse dampener operation and eliminating siphoning effects that may prompt re-priming of the pumps between shutdown and restart. Fluid circulation heaters were acquired from Watlow. The heater models are the Cast × 2000 series and can operate at a maximum output of 6000 watts. They operate on 208 V single phase power and are controlled by the EZ-ZONE^{TM} PM heater controllers. Each heater has its own J-type thermocouple embedded in the heater for process control. The controllers also communicate via DeviceNet^{TM}. Stainless steel Swagelok® in-line filters were installed to protect the flowmeters from particulates. Filters are nominally rated at 100 micron and can be easily removed for cleaning. Pressure gauges were installed on each side of the filters to monitor for restrictions as particulate buildup occurs. The flow path from tanks through all components to the contactors is constructed with 0.95-cm stainless steel tubing and various Swagelok® fittings. A photograph of a typical fluid feed system is found in Fig. 2.

Type T thermocouples used for all temperature monitoring in the system, were obtained from Omega® Engineering. All contactor inlet thermocouples are 0.8 mm diameter and 15.2 cm in length. The drain and lab temperature thermocouples are 1.6 mm in diameter and also 15.2 cm in length. The contactor surface temperatures were monitored with a surface type thermocouple consisting of a thin junction captured in an adhesive tab. Type T thermocouples have a temperature range of −250°C to 350°C and a tolerance of ±1%.

Feed and product tanks were positioned directly below the 30-stage cascade. A total of nine tanks are available for use with seven having a capacity of 378 liters and two

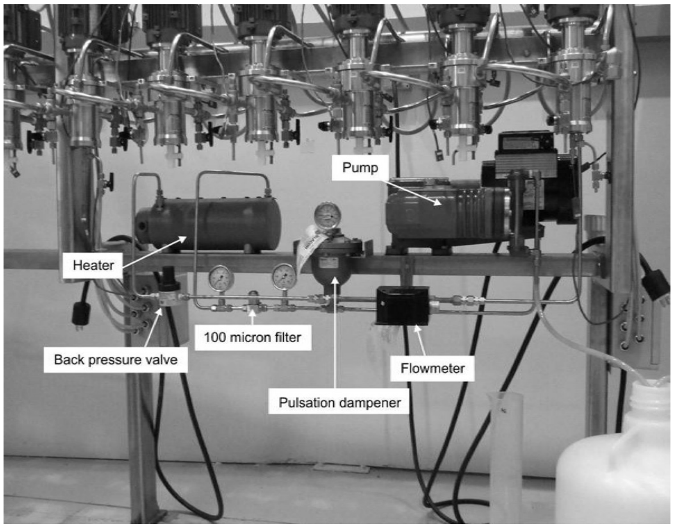

FIG. 2. Typical fluid feed system with components.

with a 265 liter capacity. The tanks are constructed of heavy-walled 0.95-cm HDPE with a 15.2 cm diameter cleanout port installed on top.

The off-gas system consists of a 7.6 cm diameter common vent header mounted on the support structure directly behind the contactors. Stainless steel 1.3-cm vent tubes equalize pressure and vapor flow on the system by connection to the vent header. The vent header is attached to an existing facility exhaust duct.

An automated process control and data acquisition system was installed to support pilot plant operation. Hardware supporting the automated process control configuration and data acquisition system was purchased from National Instruments^{TM} and housed in a metal cabinet adjacent to the pilot plant. This hardware consists of chassis and boards for analog and digital input/output signals, connector blocks for shielded cable connection, and a master/scanner interface for DeviceNet^{TM} communications. The thermocouple terminal block modules and amplifiers for thermocouple connectivity and communications are also housed in the cabinet. LabVIEW^{TM} version 8.2 was incorporated as the graphical user interface for process signal to automated control. The automated process control system provides full control of the pump speed for flowrates in either manual or automatic mode, contactor on/off and rpm adjustment, solution temperature heating/control via the heater controller, and real-time process monitoring of temperature and flowrates.

EXPERIMENTAL

TRUEX Flowsheet Preparation

The TRUEX flowsheet tested was the same as the flowsheet tested at Oak Ridge National Laboratory (ORNL) in 2008 as part of the Coupled End to End (CETE) demonstration except for an adjustment to the number of stages (3). The TRUEX flowsheet tested is presented in Fig. 3.

The TRUEX solvent used for this test consisted of 0.2 M octyl (phenyl)-N,N-diisobutylcarbamoyl-methylphosphine oxide (CMPO) + 1.4 M tri-butyl phosphate (TBP) in an n-dodecane hydrocarbon diluent. The CMPO was acquired from Eichrom Technologies, Inc. (Darien, IL). The reagent grade TBP and n-dodecane were received from Aldrich Chemical Co. (Milwaukee, WI). Before use, the solvent was washed with sodium carbonate and reacidified with dilute nitric acid. The washed and reacidified TRUEX solvent composition and purity was confirmed using an Am-241 purity test and the results of the purity test indicated that the solvent was suitable for use (4).

The aqueous feed solution used was a ~4.4 M nitric acid solution adjusted with ~0.07 M oxalic acid to complex the zirconium, thus minimizing extraction. The composition of the stable lanthanides and fission product surrogates added to the aqueous feed including the final acid concentrations are listed in Table 1. All chemicals used for aqueous feed preparation were reagent grade, acquired from Aldrich Chemical Co., and used as received.

Process Sampling

The design and construction of the pilot plant provide sampling points for the aqueous and solvent inlet/outlet streams for each stage as well as sampling points for all flowsheet effluent streams. Samples of the strip product were taken at intervals of 2, 3, 4, 5, and 7 minutes after the start of the feed simulant to evaluate the approach to steady-state. Samples were then taken from each effluent stream every 20 minutes. Additionally, the aqueous and organic effluent from each stage was sampled at 140 minutes to determine the concentration profile at steady-state conditions and calculate individual stage efficiencies.

Flowsheet Testing

Flowsheet testing proceeded as follows. The centrifugal contactors were started at 3750 rpm. The raffinate, strip product, wash effluent, and solvent effluent stages all utilized low-mix sleeves to improve phase disengagement and minimize other phase carryover in the effluent streams. The low mix sleeve is a sleeve installed around the spinning rotor to prevent the incoming solutions from contacting the spinning rotor, thus reducing mixing significantly (5). These stages were operated at 4000 rpm. Aqueous feed solutions were pumped to their input stages and allowed to flow through their entire section prior to starting the

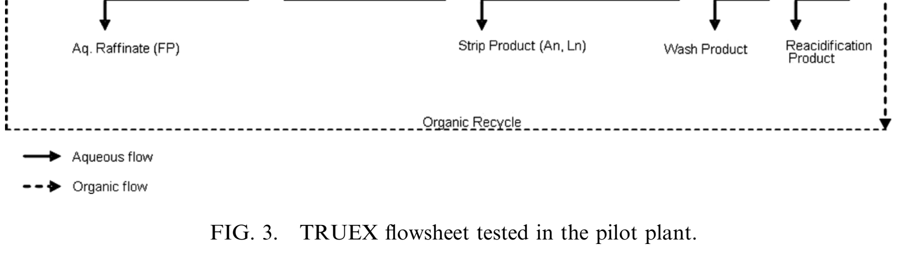

FIG. 3. TRUEX flowsheet tested in the pilot plant.

TABLE 1
Aqueous feed target and actual compositions

Component	Target concentration ($\underline{M}$ or g/L)	Actual concentrations ($\underline{M}$ or g/L)	Parent chemical
Oxalate	0.070 $\underline{M}$	0.07 $\underline{M}$	Oxalic acid
H^+	4.2 $\underline{M}$	4.4 $\underline{M}$	Nitric acid
La	0.15	0.17	$La(NO_3)_3 \cdot 6H_2O$
Ce	0.34	0.35	$Ce(NO_3)_3 \cdot 6H_2O$
Nd	0.48	0.51	$Nd(NO_3)_3 \cdot 6H_2O$
Eu	0.030	0.030	$Eu(NO_3)_3 \cdot 5H_2O$
Cs	0.22	0.23	$CsNO_3$
Sr	0.50	0.52	$Sr(NO_3)_2$
Zr	0.21	0.22	$ZrO(NO_3)_2$

solvent flow. All aqueous solutions used for initial startup were actual test solutions excluding the aqueous feed which was a 4.4 M nitric acid solution without any metal salts or oxalate added. A solvent feed solution was then initiated. Substantial solvent foaming was observed in all lighter phase inter-stage tubing of the extraction, scrub, and strip sections; however, the solvent flow appeared stable. Solvent foam formation is related to solvent composition but also may result from high rotor speed causing air to be mixed with the solvent as it is discharged into the collector ring. Foaming can also occur from high solvent flowrates or if the contactors are inadequately vented.

All rotor speeds were reduced to 3500 rpm to minimize foaming. The foaming persisted so the rotor speeds were further reduced to 3350 rpm and finally 3000 rpm for the remainder of the testing. At 3000 rpm, the solvent flow remained steady; however, some foaming was still observed.

When the process solvent had begun exiting stage 30, the actual test feed solution was initiated (time zero). The flowsheet testing lasted for a total of 172 minutes. At this time, all solution feed pumps and contactor rotors were de-energized simultaneously. Solutions were collected from each contactor drain and archived.

RESULTS AND DISCUSSION

General Observations

The flowsheet test lasted for a total of 172 minutes. The flowsheet operated very well with the only issue being the observation of foaming in the solvent phase inter stage tubing of the extraction, scrub and strip contactors for the duration of the test. This resulted in some other phase carryover in the raffinate and strip product streams. However, it did not impact the solvent flow between stages and foaming was not observed in the heavy phase inter-stage tubing. Samples taken during testing indicated a carryover of organic solution into the aqueous effluent streams (approx. 2% in aqueous raffinate, 0.8% in the strip product and 0.02% in the reacidification product). Solvent foaming in the organic phase collector ring was the most likely cause of this carryover. The centrifugal contactor aqueous phase collection ring is not sealed from the lower organic phase collection ring so when the organic in the lower collection ring foams, the foam can reach the upper ring and contaminate the aqueous phase. Organic phase carryover into the carbonate wash effluent as well as aqueous phase carryover in the solvent was not measurable. The use of low-mix sleeves at the raffinate and strip product outlets did not alleviate solvent phase carryover which supports the conclusion that the carryover foam forming in the lower collection ring of the contactors contributed to the carryover.

Steady-State Confirmation

Test samples were taken at 20 minute intervals for the raffinate (Fig. 4) and at 2,3,4,5 and 7 minutes for the strip product (Fig. 5) and were used to evaluate the approach to steady state for the metals during the test. The steady-state plots shown in Fig. 4 indicate that cesium, strontium, and zirconium reach steady-state conditions in the raffinate within 20 minutes. The approach to steady-state plot shown in Fig. 5 indicates that the lanthanides reached steady-state in the strip product within 20 minutes as well. Twenty minutes of operation corresponds to approximately three solvent turnovers through the centrifugal contactor equipment.

Removal Efficiencies

Sample results taken at 160 minutes (12 minutes prior to shutdown) were used to calculate the percentage of metals found in respective product streams and the removal efficiencies for those metals. The removal efficiency for the extractable species (lanthanides) and non-extractable species (Cs, Sr, Zr) were calculated as follows:

$$RE_{lanthanides} = 1 - \frac{Conc._{Raff} \times Flowrate_{Raff}}{Conc._{Feed} \times Flowrate_{Feed}} \bullet 100\%$$

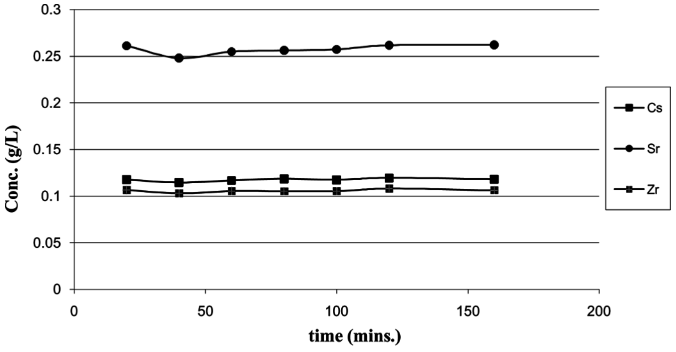

FIG. 4. Aqueous raffinate approach to steady-state.

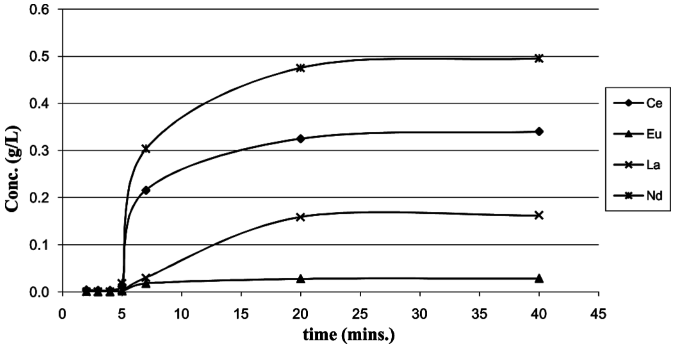

FIG. 5. Strip product approach to steady-state.

$$RE_{Cs,Sr,Zr} = 1 - \frac{Conc._{strip\ product} \times Flowrate_{strip\ product}}{Conc._{Feed} \times Flowrate_{Feed}} \bullet 100\%$$

Table 2 shows the calculated results for the product streams. In addition, analysis of the individual extraction section stage samples at shutdown were used to verify removal efficiencies.

Removal efficiencies indicated very good performance of the tested TRUEX flowsheet with >99.9% extraction of the lanthanides without extraction of the cesium and strontium and with minimum zirconium extraction (0.6%). The extraction section stage data indicate the detection limits for the lanthanides were actually reached several stages prior to the raffinate stream which supports the overall removal efficiencies of >99.9%. The concentrations of metals in the wash, rinse, and solvent effluents were below detection limits for all but Sr; however, the amounts of Sr detected in these streams were extremely low. Overall, these results indicated the flowsheet performed well.

The overall material balance for the lanthanides ranges from 90% to 93%. For the Cs, Sr, and Zr, the mass balances ranged from 96% to 102%. These material balances are within the expected margin of error based on ICP-MS analysis and flowrate measurement uncertainty. The lower material balances for the lanthanides indicate a greater flowrate inaccuracy for the strip product.

Mass Transfer Stage Efficiency

Individual stage efficiencies were calculated to further evaluate the overall performance of the centrifugal contactors with the TRUEX flowsheet. The aqueous and organic samples taken from each stage at 140 minutes were re-equilbrated by heating these samples to the test temperatures recorded at 140 minutes, then contacting appropriate volumes to match O/A ratios performed during the flowsheet. The phases were then separated. Analyzed metal concentrations from the original aqueous samples from each stage and from the re-equilibrated and separated aqueous samples from the corresponding stage were used to calculate stage efficiencies. The mass transfer stage efficiencies were calculated for selected extraction and strip stages using the Murphree efficiency equation as defined below:

$$\eta = \frac{(X - X_{\ln})}{(X_{eq} - X_{\ln})} * 100\%$$

Where X is the metal concentration of the non re-equilibrated effluent from a given stage, X_{in} is the inlet metal concentration to that stage, and X_{eq} is the metal concentration of the effluent from the same stage following re-equilibration. Calculations are based on the aqueous phase analyses since they were analyzed directly by ICP-MS, whereas organic phase samples require indirect measurement resulting in reduced accuracy.

Lanthanide stage efficiency results for five extraction section stages (feed stage is extraction stage one) and three strip section stages (strip product is stage one) are included in Table 3. Stage efficiencies are not included for the remaining stages due to sample results with lanthanide concentrations below detection limits or stages where there is minimal mass transfer occurring, such as the scrub section, and stages where the low-mix sleeve was installed. Thus, fission product mass transfer efficiency data are also omitted.

Individual stage efficiencies in the extraction section range from 85% to 95% excluding the feed stage −2 results for cerium and lanthanum and the feed stage −1 result for Ce. These low results are likely due to erroneous analytical

TABLE 2
Distribution of metals in flowsheet effluents

Effluent stream	Ce (%)	Eu (%)	La (%)	Nd (%)	Cs (%)	Sr (%)	Zr (%)
Raffinate	<0.002	<0.014	<0.007	<0.002	101.5	98.1	95.5
Strip Product	90.0	91.2	91.5	92.7	<0.001	<0.001	0.6
Wash Product	<0.0004	<0.003	<0.0006	<0.0004	<0.00004	0.0002	<0.02
Reacidification product	<0.0006	<0.004	<0.0008	<0.0006	<0.00007	0.0001	<0.03
Solvent effluent	<0.002	<0.013	<0.003	<0.002	<0.0002	0.0002	<0.085
Removal efficiency	>99.99	>99.98	>99.99	>99.99	<0.001	<0.001	0.6%

TABLE 3
Individual stage efficiencies for selected stages

Stage #	Ce (%)	Eu (%)	La (%)	Nd (%)
Fifth Extraction Stage	88.9	93.6	93.3	92.0
Fourth Extraction Stage	93.3	91.8	94.5	93.5
Third Extraction Stage	61.4	88.5	29.4	89.2
Second Extraction Stage	~100[1]	85.1	92.5	88.3
Extraction Feed stage	90.2	90.0	89.5	89.9
Second Strip Stage	60.0	70.6	59.0	62.7
Third Strip Stage	~100[1]	84.7	~100[1]	99.5
Fourth Strip Stage	~100[1]	97.9	~100[1]	~100[1]

[1] Calculated Efficiency was greater than 100% likely as a result of analytical and/or experimental uncertainty.

results. Good agreement was obtained for the individual lanthanide stage efficiencies. As centrifugal contactor sizes are increased from laboratory-scale to pilot and production-scale, the stage efficiencies are expected to increase and approach 100%. Centrifugal contactors of the size tested (5-cm) typically achieve 95% to 100% efficiency. There are several potential reasons for the measured stage efficiencies being lower than expected. First, the centrifugal contactor rotor speed was decreased from the initial speed of 3750 rpm to 3000 rpm to reduce the amount of foaming of the organic phase. This reduced speed will result in a reduced mixing intensity which could have impacted stage efficiency. Second, the carryover of solvent into the aqueous phase from the foaming contaminates the aqueous phase samples which could result in lower measured stage efficiencies. Even with the reduced stage efficiencies, good separation was achieved as indicated by the overall removal efficiency results.

The strip section stage efficiencies range from 59% to 100% with the lower efficiencies being on the first strip stage and the efficiencies increasing to near 100% on later strip stages. It is unclear why the early strip stage efficiencies are lower. The main difference in conditions on the early strip stage versus the remaining stages is the presence of nitric acid in the solvent that is being back extracted by the aqueous phase.

Temperature Profiles

Temperature profile data recorded with the data acquisition system were plotted for the duration of the flowsheet testing. The temperature of the aqueous phase throughout the extraction, scrub, and strip sections prior to shutdown are shown in Fig. 6 along with the temperature range across each of the sections of the flowsheet. Ambient temperature during the test was 27°C. The feed solutions were not heated or cooled during the test. The extraction, scrub, and strip section temperatures were elevated from the ambient temperature, with the highest temperature recorded being 7°C above ambient. This corresponds with previous temperature profile testing performed in this equipment (6). The solution temperatures that are below ambient temperature in Fig. 6 correspond to feed stages where the temperatures of the large containers of feed solutions did not increase as quickly as the ambient temperature which increased from 24°C to 27°C during the test. Based on the results of the flowsheet testing, the observed temperature variation did not adversely impact flowsheet performance.

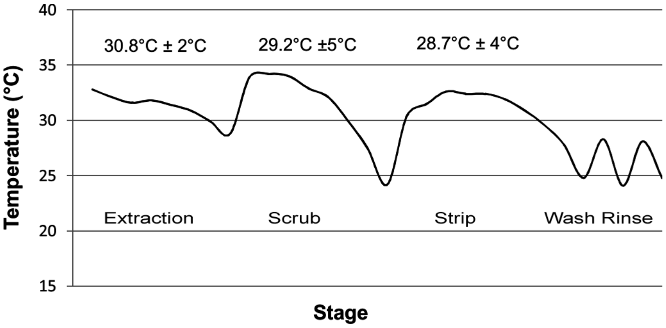

FIG. 6. Aqueous phase temperature profile.

CONCLUSIONS

An evaluation of the performance of the TRUEX process in a 30-stage centrifugal contactor pilot plant has been completed. The overall equipment operation, actual flowsheet performance including steady-state data, removal efficiencies, individual stage mass transfer efficiency, as well as temperature profiles have been measured and reported.

The overall flowsheet performance was very good. Sample results indicate the cascade achieved steady-state conditions rapidly, within about twenty minutes. Twenty minutes of operation corresponds to approximately three solvent turnovers through the centrifugal contactors. The lanthanide mass balance was a little low, ranging from 90% to 93% and the reasons not completely understood; however, analytical and flowrate uncertainties would explain these results. Removal efficiencies, based on raffinate samples and individual stage samples, indicate >99.9% of the Ce, La, Eu, and Nd was separated from the feed simulant. The cesium and strontium separation was negligible as these metals were not extracted by the TRUEX solvent. The calculated removal efficiency for zirconium indicates that ~0.6% of the metal may have extracted.

All equipment associated with the pilot plant operated as designed. Fluid pump systems and the data acquisition system performed without incident. There was foam generation observed in all inter-stage solvent phase tubing of the extraction, scrub, and strip stages, which resulted in slight solvent in aqueous carryover in the raffinate and strip product outlets. An estimated solvent in aqueous carryover of ~2% was measured in the raffinate while ~0.8% carryover was measured in the strip product. There was no foaming observed in the aqueous phase tubing. The possibility of foaming suggests that further evaluation of pilot plant operating conditions, solvent pair behavior, and/or equipment design, specifically the installed off-gas ventilation tubing and the solution flow path design, is warranted.

Individual stage mass transfer efficiency results ranged from 85% to 95% in the extraction stages and from 85–100% in all but the first measured strip stages. The first measured strip stage had efficiencies ranging from 59% to 71%.

Temperature profile data for the TRUEX flowsheet indicate process solution temperatures increased above the ambient temperature of 27°C, with a maximum temperature of 34°C in the scrub section. Extraction stage temperatures were 31°C ± 2°C, scrub stage temperatures 29°C ± 5°C, and strip stage temperatures 29°C ± 4°C. The results obtained indicate the TRUEX flowsheet employed with this scale of centrifugal contactor operated well without any cooling or heating of the feed solutions or without the use of heat exchangers on the individual centrifugal contactor stages.

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