Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

Thirty Stage Annular Centrifugal Contactor Thermal Profile Measurements

D. H. Meikrantz[a]; T. G. Garn[a]; J. D. Law[a]
[a] Idaho National Laboratory, Idaho Falls, ID, USA

Online publication date: 12 February 2010

To cite this Article Meikrantz, D. H. , Garn, T. G. and Law, J. D.(2010) 'Thirty Stage Annular Centrifugal Contactor Thermal Profile Measurements', Separation Science and Technology, 45: 3, 310 — 321
To link to this Article: DOI: 10.1080/01496390903484792
URL: http://dx.doi.org/10.1080/01496390903484792

Separation Science and Technology, 45: 310–321, 2010

ISSN: 0149-6395 print/1520-5754 online
DOI: 10.1080/01496390903484792

Taylor & Francis
Taylor & Francis Group

Thirty Stage Annular Centrifugal Contactor Thermal Profile Measurements

D. H. Meikrantz, T. G. Garn, and J. D. Law
Idaho National Laboratory, Idaho Falls, ID, USA

A thirty stage 5 cm annular centrifugal contactor cascade was assembled and tested to obtain thermal profiles during both ambient and heated input conditions of operation. Thermocouples were installed on every stage as well as feed inputs, and real-time data was taken during experiments lasting from two to eight hours at total flow rates of 0.5 to 1.4 liters per minute. Ambient temperature profile results show that only a small amount of heat is generated by the mechanical energy of the contactors. Steady state temperature profiles mimic the ambient temperature of the lab but are higher toward the middle of the cascade. Heated inlet solutions gave temperature profiles with smaller temperature gradients, more driven by the temperature of the inlet solutions than ambient lab temperature. Temperature effects of solution mixing, even at rotor speeds of 4000 rpm, were not measurable.

Keywords centrifugal contactor; CINC V-02; pilot plant; solvent extraction equipment; temperature profile

INTRODUCTION

An annular centrifugal contactor (ACC) pilot plant consisting of 30 stages of commercial 5 cm units has been built and operated at the Idaho National Laboratory (INL) during the past year. The purpose is to evaluate the performance of a large number of pilot-scale centrifugal contactors. Operational data needed to support solvent extraction system design includes—temperature profiles across the cascade under both ambient and elevated feed solution temperatures, hydraulic performance, and stagewise efficiency at various locations during start-up and at equilibrium process conditions.

Centrifugal contactors are being studied and employed for many liquid-liquid processes, world wide. Numerous designs have been developed with the annular mixing type, developed by Bernstein in the early 1970s (1), being employed by numerous countries for both nuclear and non-nuclear processes (2–10). In the United States, commercialization of annular centrifugal contactors began in 1993 with the license of an INL patent for rapid, continuous separation of hydrocarbon/water mixtures (11). During the next decade, a number of design improvements were implemented to meet a wide range of industrial liquid-liquid processing needs (12–16). Included are methods for replacing the heavy phase weir to meet a broad range of applications, clean-in place capability, a low mixing option, and rotors able to be disassembled for inspection purposes. Annular centrifugal contactors with rotor diameters up to 50 cm are thus commercially available from CINC manufacturing in Carson City, NV.

Recent pilot scale commercial ACC studies at INL have been limited to single-stage performance measurements. The contactor sizes tested to date, 5 and 12.5 cm rotor units, exhibit good performance in the total flow rate range of 0.2 to 30 L/min (17). In addition, single unit mass transfer efficiencies are quite high for processes with rapid kinetics, nearly 100% (18). However, as most flowsheet tests on actual spent fuel feed solutions are limited to 1 or 2 cm mini-contactors, reported stage efficiencies are somewhat lower due to flow inconsistencies related to geometric limitations. In addition, stagewise sampling at low flows can upset process equilibrium so it is avoided in mini-contactor testing. These factors can lead to an over-estimation of the number of stages required for a particular flowsheet. Non-radioactive pilot scale testing can provide design data for steady-state operating parameters and stagewise efficiency for various flowsheets and temperatures.

The CINC V-02-based pilot plant has been assembled to test those fuel cycle flowsheets employing stable surrogates to provide operating performance data for model input and validation. Detailed temperature profiles provide information needed to validate solvent extraction flowsheet performance predictions from existing dynamic models. Mechanically generated heat from bearings, seals, and motors added to two phase mixing in the annular region outside the rotor must be considered in flowsheet development. In general, forward extraction is usually enhanced by lower temperatures while stripping or back extraction is favored by higher temperatures. As such, testing was also done to provide data on the capability of temperature control via in-line inlet solution heaters.

Received 23 January 2009; accepted 29 October 2009.
Address correspondence to D. H. Meikrantz, Idaho National Laboratory, 2525 Fremont Ave., Idaho Falls, ID 83415-38701, USA. Tel.: 208 526 4636; Fax: 208 526 2930. E-mail: david.meikrantz@inl.gov

EXPERIMENTAL

Design and assembly of the annular centrifugal contactor pilot plant began in November 2007 and was completed in nine months. The support stand was designed to accommodate the contactors in one level extending almost 25 feet in length. The pilot plant is sectioned into five identical modules each supporting 6 stages and a fluid feed system. It provides mounting provisions for metering pumps, in-line feed solution heat exchangers, contactor variable frequency drives (VFDs), and the system off-gas vent header. Contactor system process control and data acquisition are under computer control. Thermocouples are used to monitor each contactor inlet for both aqueous and organic phases, solution feeds to the system, contactor housing surfaces, rotor inlet streams of selected units, and ambient lab temperatures at both ends of the cascade. In addition, data for feed flow rate, individual contactor rotor speed, and drive motor amperage level are collected by a data acquisition system at selected time intervals. Operation of all contactors, pumps, and heaters is centralized by the computer program with control via keyboard input. A photo of the completed pilot plant is shown in Fig. 1. For reference purposes, the contactors were numbered 1–30 with number 1 being the contactor on the far left end of the assembly as viewed in the figure.

Preliminary testing was conducted with all 30 contactors interconnected for continuous counter-current flow. System operating tests and initial temperature profile measurements were completed in this configuration prior to temperature profile testing of two discreet sections that required added feed and discharge connections. Lamp oil, a commercially available hydrocarbon mixture of C14 to C18 chains, and tap water adjusted to pH 2 were the organic and aqueous phases for all testing.

FIG. 1. Thirty stage V-02 annular centrifugal contactor pilot plant.

Experimental Equipment

The pilot plant includes; thirty 5 cm centrifugal contactors with frequency drives, five liquid supply pumps, four process solution heaters with controllers, multiple thermocouples, and assorted fluid flow components.

Centrifugal contactors are constructed of stainless steel (316 L) and have housings with incorporated phase collector rings and inlet/outlet flow tubes. Stainless steel 0.5 inch O.D. tubes and various pipe and compression fittings were used to interconnect to contactor 3/8" schedule 40 pipes and install thermocouples, vents and sample taps. Each contactor has a 5 cm diameter rotor with a light/heavy phase weir package all attached to a shaft and bearing assembly connected to the motor. The ACCs were obtained commercially from CINC Manufacturing, Inc. They are rated for a total liquid flow rate of 0.1 to 2.0 L/min and have a rotor speed range of 2000 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™ communication for PC remote control operation. Each motor was monitored by a separate VFD and amperages were individually logged continuously by the data system. Current draw was measured from 0.6 A at 4000 rpm to 0.8 A at 3500 rpm, empty. About 0.05 A additional draw was measured for flow rates of 0.5 to 1 LPM. Current usage typically varied by no more than 0.1 A across the 30 units under a given set of test conditions.

The feed pumps are double diaphragm chemical metering pumps obtained from Madden Manufacturing, Inc. The rated liquid flow rate 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 ½ HP motor and operate on 110 V single phase power. Motor speed is controlled with a Franklin Electric IDMS controller operated by the PC through a 0 to 10 VDC analog signal.

Additional components included in the fluid feed system are flow meters, pulse dampeners, back pressure valves, heaters and in-line filters. The turbine type flow meters were obtained commercially from FTI Flow Technology™, Inc. They have a range of 0.303 to 3.03 L/min with an accuracy of ±1%. The flow signal is processed through a Linear Link linearizer and the signal output is communicated to the PC using a 4–20 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™, Inc. One 10 cubic inch and four 36 cubic inch volume units 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™, Inc. The back pressure valves provide back pressure on the downstream side of the pumps. Watlow fluid circulation heaters, Cast X 2000 series, operate at a maximum output of 4500 watts at 208 V single phase power and are controlled by the EZ-ZONE™ PM heater controllers. Each heater has its own J-type thermocouple embedded in the heater. The heater controllers also communicate with the PC via DeviceNet™. Stainless steel Swagelok® in-line filters were installed to protect the flow meters. Pressure gauges were installed on each side of the filters. The remainder of the liquid flow path from tanks through all components to the contactors is constructed with stainless steel 3/8" tubing and Swagelok® fittings. A typical fluid feed system is detailed in Fig. 2.

Type T thermocouples, used for all temperature monitoring in the system, were obtained from Omega® Engineering. Three sizes/types of type T thermocouples were installed throughout the process. All contactors were instrumented with two solution inlet thermocouples of 1/32" diameter and 6" in length. The drain and lab temperature thermocouples are 1/16" in diameter and also 6" in length. 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%. All thermocouples communicate with the computer through a National Instruments™ thermocouple chassis.

Automated Process Control and Data Acquisition

Programs generated in LabVIEW™ enable a wide variety of process conditions and configurations to be selected. The software programming directs the user to an initial setup screen to select desired flowsheet configurations including pumps and heaters as well as drain and surface stage thermocouple location. The setup screen allows for five operations i.e., extraction, scrub, strip, to be selected and uniquely identifies each with a different color for stages selected for each operation for process flexibility. Once the setup screen has been finalized the user is directed to a process control screen to control all process parameters and monitor process conditions. With the process control screen in operation, the data acquisitioning and the rate at which data is acquired can be set. The user can also select graphical representations of desired pump charts for real-time flow rate monitoring.

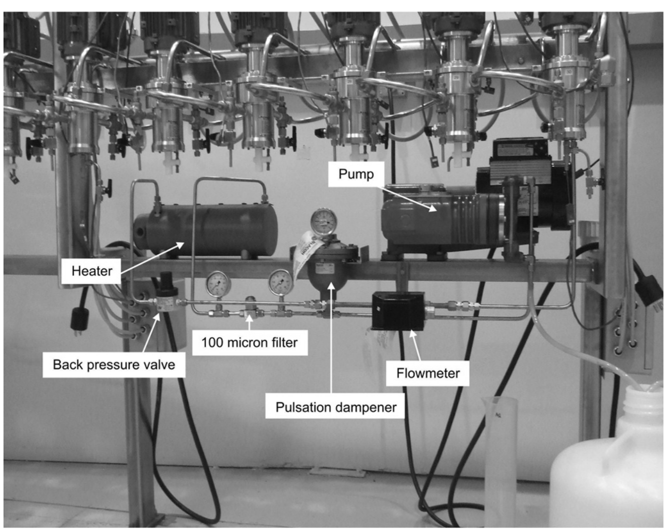


FIG. 2. Typical fluid feed system with components.

TEMPERATURE PROFILE EXPERIMENTAL TEST SUMMARY

Thermocouples used to measure inlet/outlet solution temperatures for each stage are installed through the top of cross fittings and extend down into the solution flow path. Thermocouple positioning for the temperature profile testing is as follows; at both phase inlet tubes for each contactor, on stage 1 aqueous outlet and stage 30 organic outlet, two thermocouples positioned at each end of the cascade directly behind stage 4 and stage 27 at contactor height for ambient lab temperature measurement. The drain thermocouples are inserted upward through contactor lower housing drain lines and extend ¼ inch above the interior housing base to the top of the curved vanes on the hi-mix bottom plates. This puts the thermocouples in direct contact with mixed solutions just before they enter the rotor. The adhesive tab surface thermocouples are attached to the outside of the housings at approximately 1 inch above the interior housing base. This distance allows for all mixing heights to be at or above the thermocouple positions. Surface and drain thermocouples are attached to stages 1, 7, 13, 19, 25, and 30.

The organic feed tank was filled with 35 gallons of lamp oil and the aqueous feed tank filled with 50 gallons of tap water adjusted to pH 2. Pump stroke settings were set to values previously evaluated in system operation testing. All inlet/outlet lines were vented at the end nearest contactor outlet, the high point of the interconnection. The vents were connected to a 3 inch header that was maintained at 0.01 inches, negative pressure versus atmosphere. The pressure is not important as we only wanted to move vapors to the ventilation system. The vents aid in balancing the pressure across each contactor discharge and inlet to stabilize the separation interface within the rotor.

Single-Phase Testing

Multiple temperature profile experiments were performed with the 30-stage pilot plant. Preliminary testing with a single-phase aqueous only pH 2 solution established baseline temperature profiles at ambient temperature. Two baseline tests were performed at 0.5 and 1.0 L/min at rotor speeds of 3500 and 4000 rpm, respectively. A heated single-phase test regime followed. Two tests were run with the

TABLE 1
Summary of temperature profile test conditions

Phases tested	Feed solution temperature (°C)	O/A ratio	Total flow rate (L/min)	Contactor rpm	Recycle	Test duration (hrs.)
Aq only	Ambient (24)	n/a	0.5	3500	No	3.3
Aq only	Ambient (23)	n/a	1.0	4000	No	2.5
Aq only	37	n/a	0.5	3750	No	4.0
Aq only	35	n/a	1.0	4000	No	2.7
Aq only	38	n/a	0.5	3750	Yes	8.0
Aq only	45	n/a	0.5	3750	Yes	7.0
Aq/Org	Ambient (25)	1.0	1.4	4000	No	2.7
Aq/Org	Both phases 40	0.8	1.3	4000	Yes both phases	7.5
Aq/Org	Both phases 50	0.8	1.3	4000	Yes both phases	8.0
Aq/Org	Ext. Aq ambient (24) Strip Aq 50	Ext. 0.9 Strip 0.9	Ext. 1.3 Strip 1.3	4000	No	2.5

Aq = pH 2 tap water; Org = Lamp oil.

solution inlet temperature heated to ~40°C at flow rates of 0.5 and 1.0 L/min and at rotor speeds of 3750 and 4000 rpm, respectively. Aqueous solution was passed once through the cascade for each of these four tests. The average runtime was approximately 2.5 to 4 hours dependant on the flow rate.

Next, two extended time experiments using heated inlet solution of 40°C then 50°C were completed. Flow rates for both tests of 0.5 L/min were processed at a rotor speed of 3750 rpm. Solution exiting stage 1 was collected in the receiver tank and then pumped back to the feed tank for recirculation back through the heater and on to the cascade to stage 30. The duration of these two runs was ~8 hours, each.

Two-Phase Testing

Two-phase testing was performed using tap water adjusted to pH 2 and lamp oil. Lamp oil is a paraffinic hydrocarbon, a mixture of alkanes of C_{14} to C_{18}, with low flammability and viscosity. All two-phase testing was completed at O/A ratios of ~1. Aqueous solution entered the cascade at stage 30 and organic solution entered at stage 1. Three tests were performed in this configuration. The first test provided a baseline ambient temperature profile, followed by two extended time runs with both aqueous and organic phases heated to 40°C and 50°C, respectively. The baseline ambient temperature test was performed as a once-through process while both phases were re-circulated throughout the duration of the extended tests. The ambient temperature test duration was ~3 hours while the two extended tests were ~8 hours each. The total flow rate was set at 1.5 L/min with a rotor speed of 4000 rpm for all three tests. Actual O/A ratios were determined from measured effluent flow rates during two-phase testing.

A final two-phase heated test was completed by dividing the cascade in half. It was performed to evaluate a temperature profile across a 15 stage extraction section and a 15 stage strip section, individually. The aqueous strip solution heated to 50°C entered stage 30 while the extraction aqueous feed was at ambient temperature and entered stage 15. The strip solution exited the cascade at stage 16 and the aqueous feed exited at stage 1. The organic solution traveled the entire length of the cascade, entered stage 1, unheated, and exited at stage 30. The target total flow rate for both extraction and strip sections was 1.5 L/min. There was no solution recycle hence the test lasted for 2.5 hours. Table 1 provides a summary of the test parameters used for all single and two-phase temperature profile testing.

RESULTS AND DISCUSSION

Single-Phase Testing

Temperature profiles can be measured to monitor mechanically generated heat transferred to process solutions and housings during contactor operation for various operating conditions. Ambient solution temperature testing with aqueous only was performed at two flow rates and contactor speeds. Aqueous solution was pumped into stage 30 and exited the cascade from stage 1. Data acquired was used to construct temperature profile graphs for evaluation. Figure 3 are two graphs constructed to show the temperature profiles of the inlet stage (stage 30) and the outlet (stage 1) with the averaged ambient lab temperature for the two aqueous only tests at ambient temperature.

These graphs serve two purposes—to show the temperature change of aqueous solution between inlet and outlet

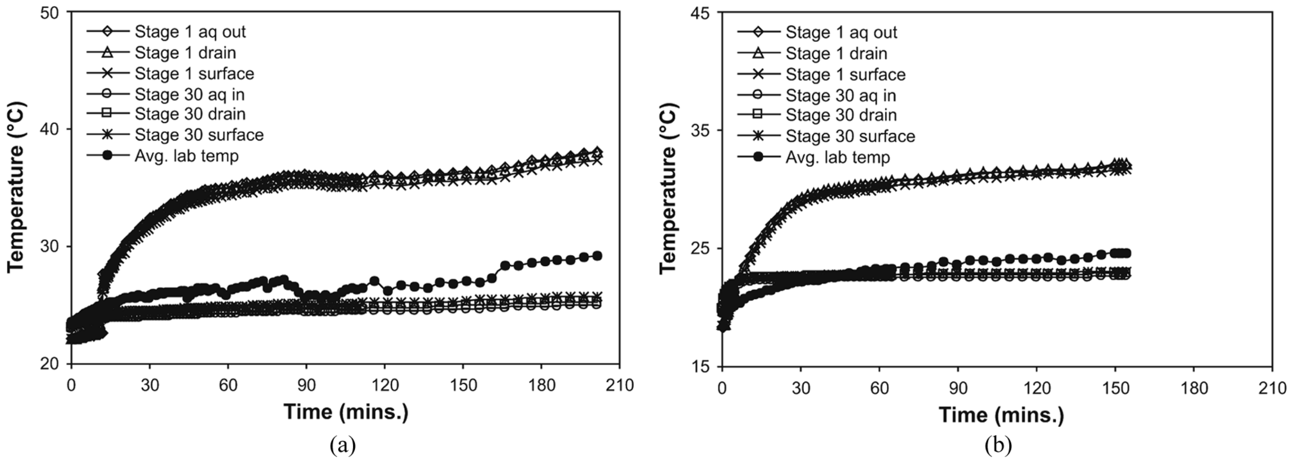


FIG. 3. Solution inlet/outlet temperature profile at (a) 0.5 L/min and rotor speed of 3500 rpm and (b) 1 L/min and rotor speed of 4000 rpm with no recycle. Drain temp refers to temperature measurement of process solution entering at rotor inlet.

stages over time, and more importantly, to show that the aqueous solution temperatures agree with the surface and drain temperatures measured for each stage. This shows that the surface temperature readings provide good estimates of the process solution temperatures, at the rotor inlet, for respective stages. The data points for the averaged lab temperature curve are the average of the near and far thermocouples located behind the contactor assembly at a given time. The averaged lab temperature curve in Fig. 3b fluctuates due to the constant changing of the ambient lab temperature. The distance between data points is directly related to the assigned data intervals during testing. For example, as indicated in Fig. 4, data taken during the first 20 minutes and the last ten minutes of testing were taken at faster intervals than those taken from 60 to 140 minutes.

The data in Fig. 3 indicate that a steady-state operating condition is reached within 60 minutes of operating time for both tests and that the continued temperature increase at the outlet is more influenced by ambient lab temperature changes. The temperature difference between inlet and outlet is slightly higher when the total flow rate is lower even with increased rotor speed at the higher flow rate. This shows that higher flows provide more heat removal/transfer from/to the system, independent of the heat capacity of the fluids. However, aqueous fluids, having higher heat capacity than organic fluids, transfer more heat to/from the system at a given flow rate.

To further evaluate the temperature profile across the 30-stage cascade at the two ambient temperature test conditions, graphs were constructed to show profiles of selected stages. Figure 4 include the contactor surface

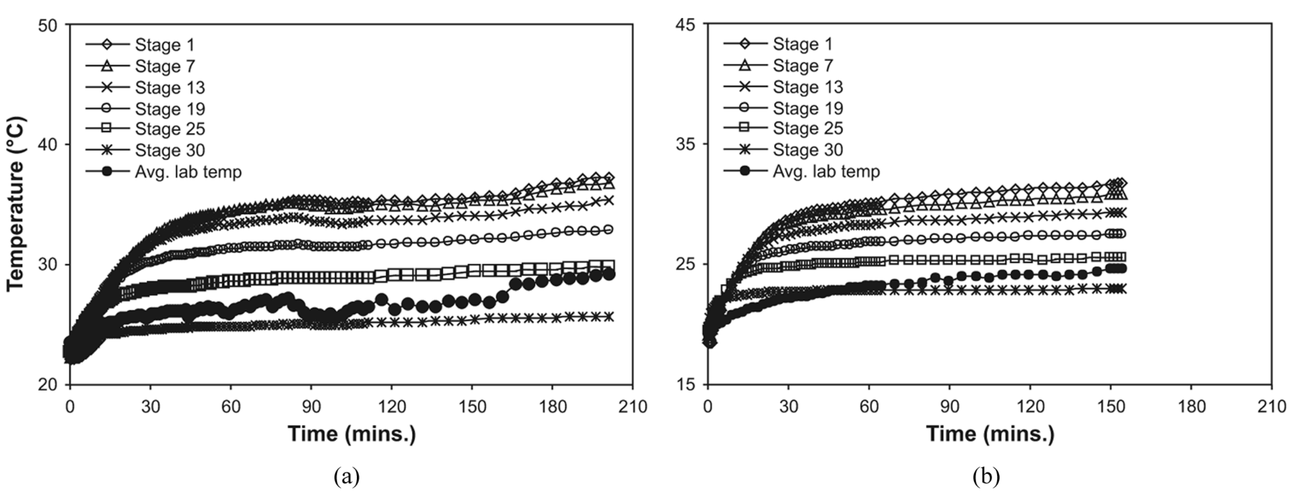


FIG. 4. Surface temperatures of selected stages at 0.5 L/min and rotor speed of 3500 rpm and (b)1 L/min and rotor speed of 4000 rpm.

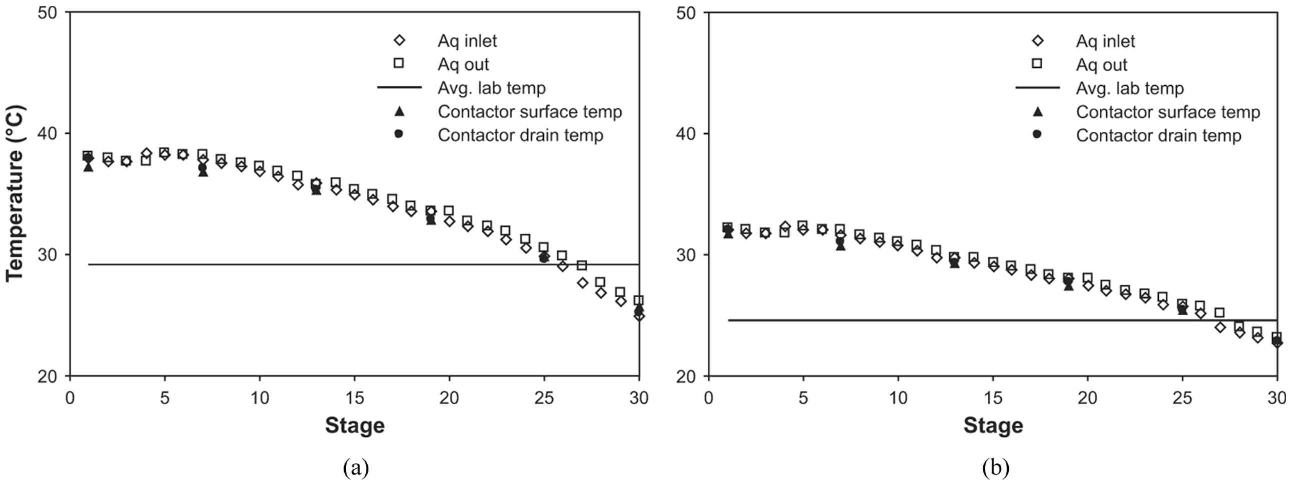


FIG. 5. Stage temperatures at shutdown at (a) 0.5 L/min and rotor speed of 3500 rpm and (b) 1 L/min and rotor speed of 4000 rpm.

temperature of the instrumented stages and average lab temperature over time for each test condition.

Figure 5 include inlet/outlet solution temperatures with surface and drain temperatures for selected stages at shutdown. The averaged lab temperature shown was the average of the near and far lab thermocouple readings at shutdown. These temperature profiles show that an increase in the total flow rate provides more heat removal from the system, even at higher contactor rotor speed.

Heated Single-Phase Testing

Four single-phase tests were completed with heated aqueous solution feed to the system. In the first two tests, solutions were heated to ~40°C as once-through tests, with no recycling of the solution. During these tests, the heater controller setpoint was set to the desired operating temperature, but because the setpoint responds directly to the thermocouple positioned in the heater, the final temperature of the solution entering at stage 30 was typically a few degrees lower than the setpoint. Figure 6 includes selected stage surface temperature profiles with associated averaged lab temperature for the two heated tests performed without solution recycle.

The increased flow rate associated with the 1 L/min test profiled in Fig. 6b removes more mechanical heat from the system. Also, the stage temperatures are nearly equal to the inlet solution temperature whereas in Fig. 6a, all stage surface temperatures excluding stage 30 are greater than the inlet solution temperature.

The graphs of a final view of stage temperatures at shutdown for the single-phase heated tests with no recycle are given in Fig. 7. They show that the lower flow rate

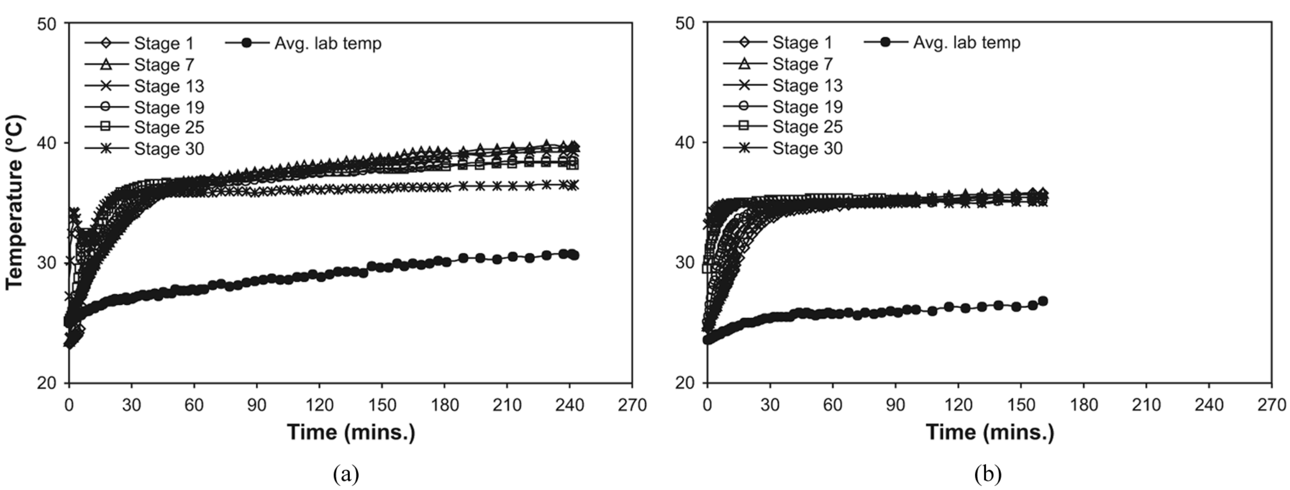
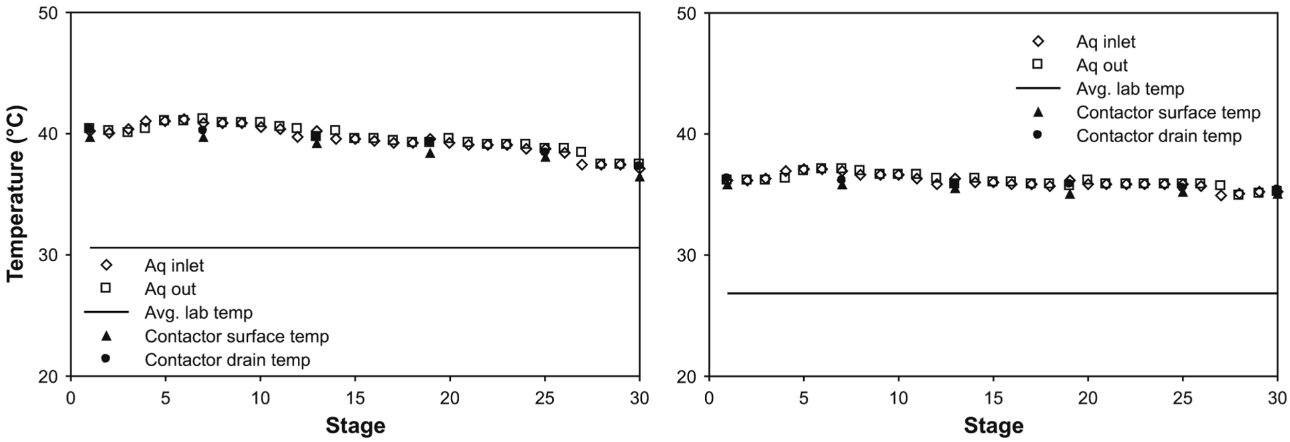


FIG. 6. Surface temperature profile for (a) 0.5 L/min and rotor speed at 3750 rpm with solution inlet temperature of 37°C and no recycle and (b) 1 L/min and rotor speed at 4000 rpm with solution inlet temperature of 35°C and no recycle.



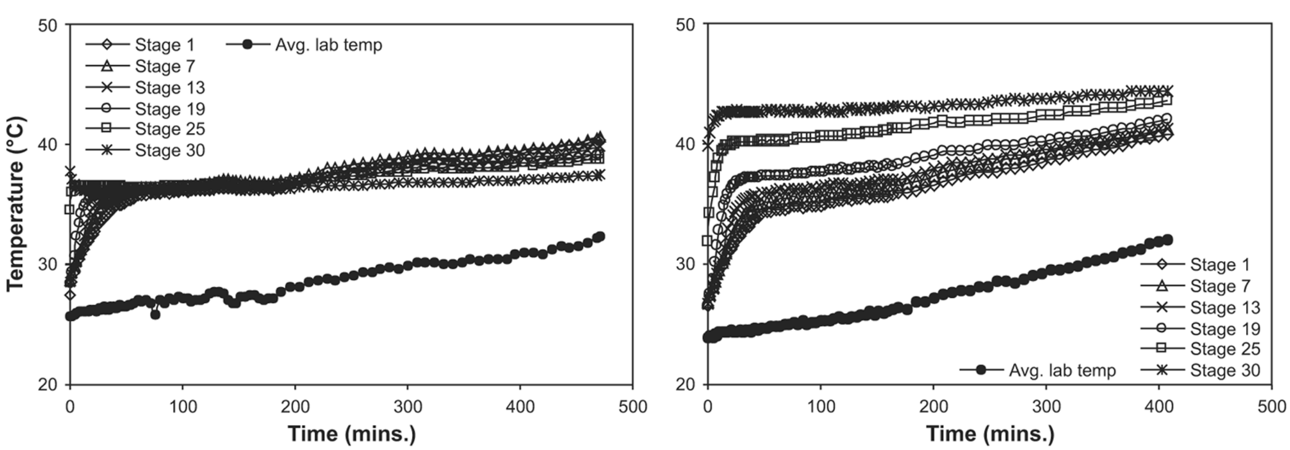
(a) (b)

FIG. 7. Stage temperatures at shutdown at (a) 0.5 L/min and rotor speed of 3750 rpm with inlet solution temperature of 37°C and (b) 1 L/min and rotor speed of 4000 rpm with inlet solution temperature of 35°C.

testing provides less heat removal from the system even at elevated heated solution temperatures. However, both of these temperature profiles are more consistent across the contactor cascade showing less temperature variance than those profiles taken at ambient temperature.

The next single-phase tests consisted of two extended time runs with inlet solutions heated to 38°C and 45°C, respectively. Solutions were recycled from the receiver to the feed tank, to the inline heater, and back to stage 30 for continuous operation. Stagewise surface temperature profiles were graphed for each test with the averaged lab temperature and are shown in Fig. 8. Stage temperatures continually increased relative to the lab temperature increase for the 38°C test, whereas in the 45°C inlet test, relative contactor profiles were decreasing from stage 30 to stage 1. At both inlet temperatures, the variance at shutdown across all stages is less than 4°C. However at 0.5 L/min flow, at the higher inlet feed temperature, thermal equilibrium was more slowly approached due to radiant heat loss across the contactor assembly.

Figure 9 are final views of the two heated single-phase tests at shutdown. As before, excellent thermal agreement is seen between the contactor housing surface and the drain position thermocouples. The profile shown in Fig. 9a at shutdown indicates that most stages reached temperatures slightly higher than the heated inlet solution temperature. In Fig. 9b, at the higher inlet temperature, no stage reached the temperature of the heated inlet solution even after 7 hours at a flow rate of 0.5 L/min. However, at higher flow rates, the overall cascade profile would be closer to the inlet feed temperature, as more heat would be transferred to the system.



(a) (b)

FIG. 8. Stage surface temperature profile at (a) 8 hours with inlet solution heated to 38°C at 0.5 L/min and a rotor speed of 3750 rpm and (b) 7 hours with inlet solution heated to 45°C at 0.5 L/min and a rotor speed of 3750 rpm.

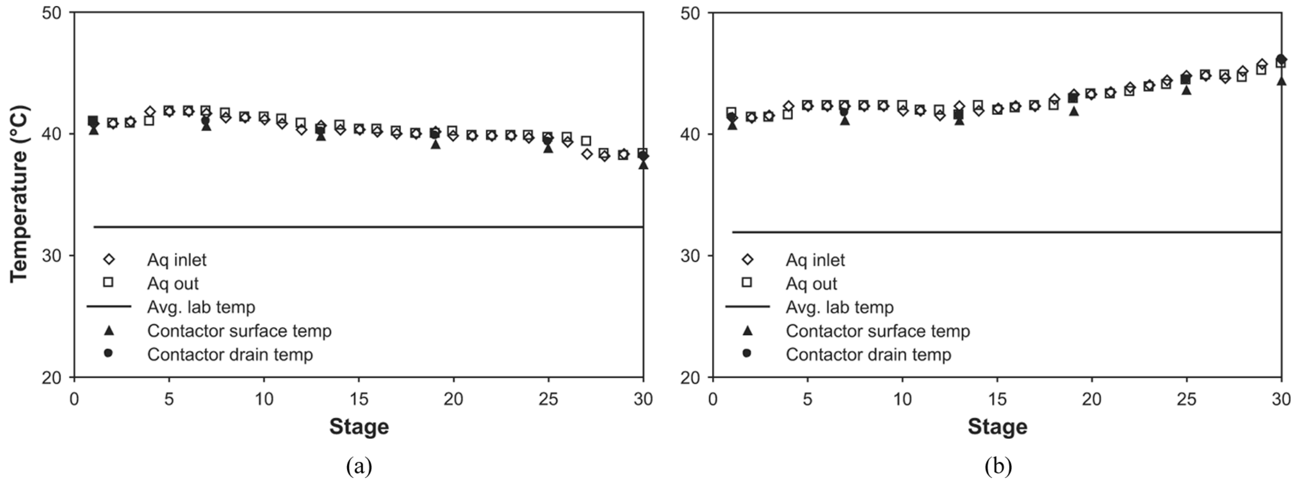


FIG. 9. Stage temperatures and averaged lab temperature at shutdown at 0.5 L/min at a rotor speed of 3750 rpm with inlet solution heated to (a) 38°C and (b) 45°C.

Two-Phase Testing

A total of four two-phase tests were completed using lamp oil and pH 2 tap water. Total flow rate was 1.3–1.4 L/min at an O/A ~1 for all tests. Ambient temperature tests were again followed by tests with heated input solutions. The cascade was then partitioned into two equal sections so that stages 1–15 were operated as the cooler extraction section and stages 16–30 were operated as the heated strip section.

Ambient Two-Phase Testing

Solution inlet temperatures for both phases were at ambient for this test. Lamp oil was fed to stage 1 and exited stage 30 while the pH 2 water was fed to stage 30 and exited the cascade at stage 1. Both phase effluent flow rates were 0.7 L/min resulting in an O/A ratio of 1. Contactor rotor speeds were set at 4000 rpm with no solution recycle. Figure 10 shows the instrumented stage surface temperatures and the average lab temp over time for the ambient two-phase test.

A comparison of the two-phase ambient temperature profiles in Fig. 10 with corresponding single-phase profiles (Fig. 4) show little difference. Final stage temperatures at shutdown for the aqueous and organic solutions are plotted in Fig. 11, respectively. Apart from the first 3 stages from the organic inlet, the final aqueous and organic temperature profiles are overlapping. Final peak temperatures of 32–33°C were observed near stage 6 in both single-phase and two-phase ambient temperature tests at flow rates of 1–1.4 L/min. The heat capacity of water is about twice that of the lamp oil solvent. As such, for two-phase flow tests at ambient, peak temperatures occur at stages 5 through 10 when the heat accumulated in the aqueous phase is no longer lost to the organic phase.

Solution temperature trends for both aqueous and organic stage solution temperatures at shutdown for the two-phase ambient temperature tests are in good agreement. Two-phase mixing does not appear to add measurable heat to the system. An overall cooler temperature trend across the 30-stage cascade was observed, especially near the organic inlet, when compared to single phase ambient temperature profiles. This is due to the additional heat capacity of the ambient temperature organic phase entering the cascade at stage 1. Rapid heating of the organic in the first two operating stages occurs as the result of the heat load of the aqueous phase being rapidly transferred to the ambient temperature organic entering at stage 1. This heat transfer is quite efficient due to the annular mixing providing intimate contact of the two phases at each stage. The aqueous phase temperature is decreasing in

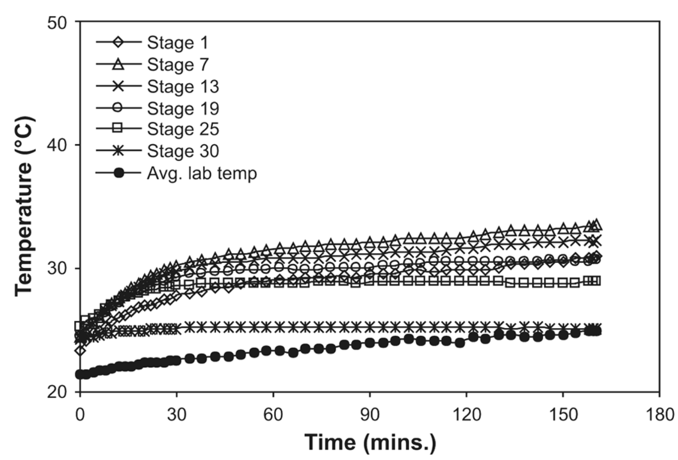


FIG. 10. Contactor surface temperatures of selected stages and average lab temperature at 1.4 L/min and rotor speed of 4000 rpm at an O/A ratio of 1.

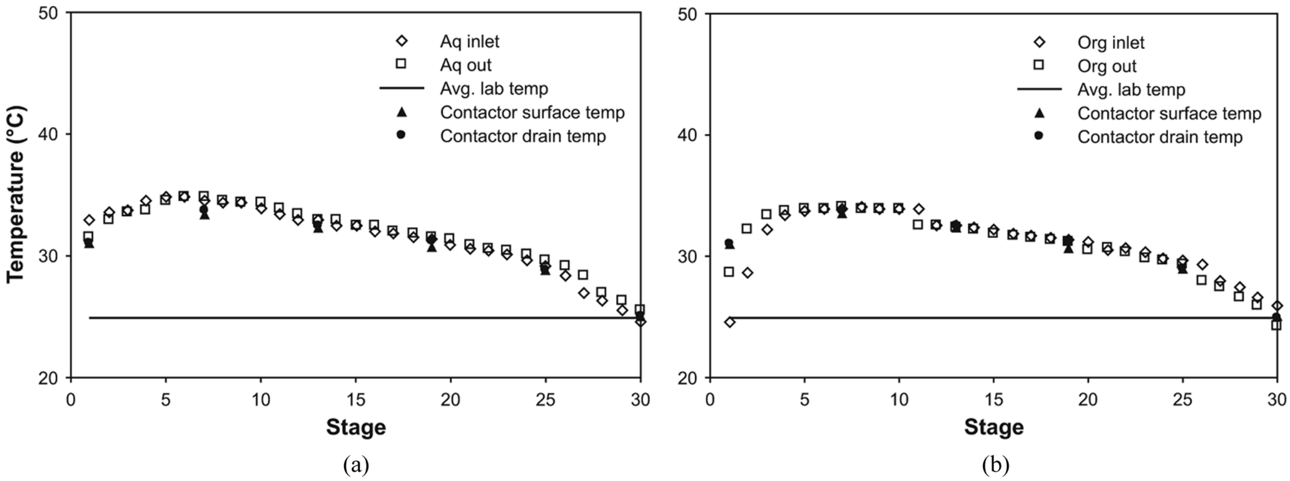


FIG. 11. Aqueous phase solution temperatures (a) and organic phase solution temperatures (b) with surface and drain temperatures for each stage at shutdown for ambient two-phase testing.

proportion to the organic phase heat-up in stages 1, 2, and 3. Thermal equilibrium between aqueous and organic is reached by stage 4, the point at which no further heat transfer to the organic phase occurs.

Heated Two-Phase Testing

Two tests were completed for heated two-phase testing. Both aqueous and organic inlet solutions were heated for each test, the first test to 40°C and the second to 50°C. Both phases were continuously recycled and reheated for the duration of each test. The total flow rate for each test was 1.3 L/min at contactor rotor speed of 4000 rpm and at O/A ratios of 0.8.

Contactor surface temperatures with average lab temperatures were plotted as a function of time for both tests and are shown in Figs. 12 and 13. The stage surface temperature profile for the 50°C shown in Fig. 13 shows a larger temperature spread for stages 1, 7, 13, and 25 than that of the previous test. The gradual temperature increase over the test duration still mirrors the average lab temperature trend.

Figures 14–15 depict individual stage inlet/outlet aqueous and organic solution temperatures and surface and drain temperatures along with the average lab temperature at shutdown for the heated two-phase tests. Aqueous solution stage temperatures are in good agreement with one another. The maximum temperature reached with 40°C heated two-phase testing is about 44°C (Fig. 14a at stages 6 and 7) when the ambient lab temperature reaches 34°C. The organic solution stage temperatures in Fig. 14b are less uniform than that of the aqueous. Pulsing observed of the organic effluent exiting stage 30 may have caused some of

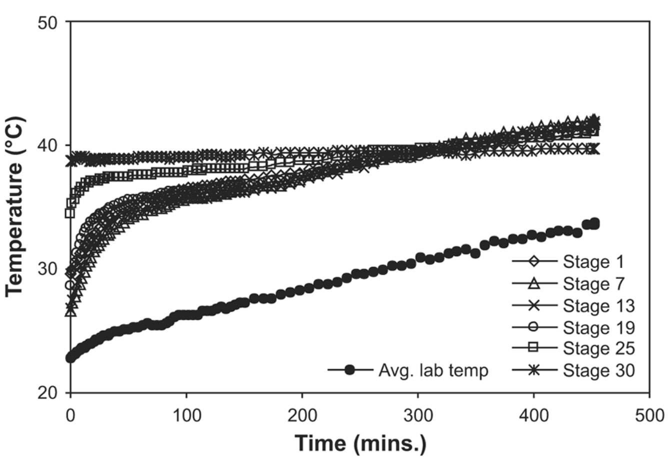


FIG. 12. Contactor surface temperatures and average lab temperature plotted with time. Both phases heated to 40°C at 1.3 L/min at rotor speed of 4000 rpm and O/A of 0.8.

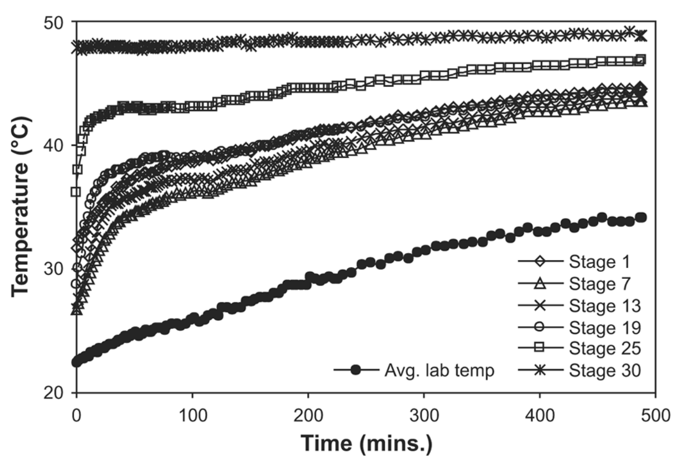


FIG. 13. Contactor surface temperatures and average lab temperature plotted with time. Both phases heated to 50°C at 1.3 L/min at rotor speed of 4000 rpm and O/A of 0.8.

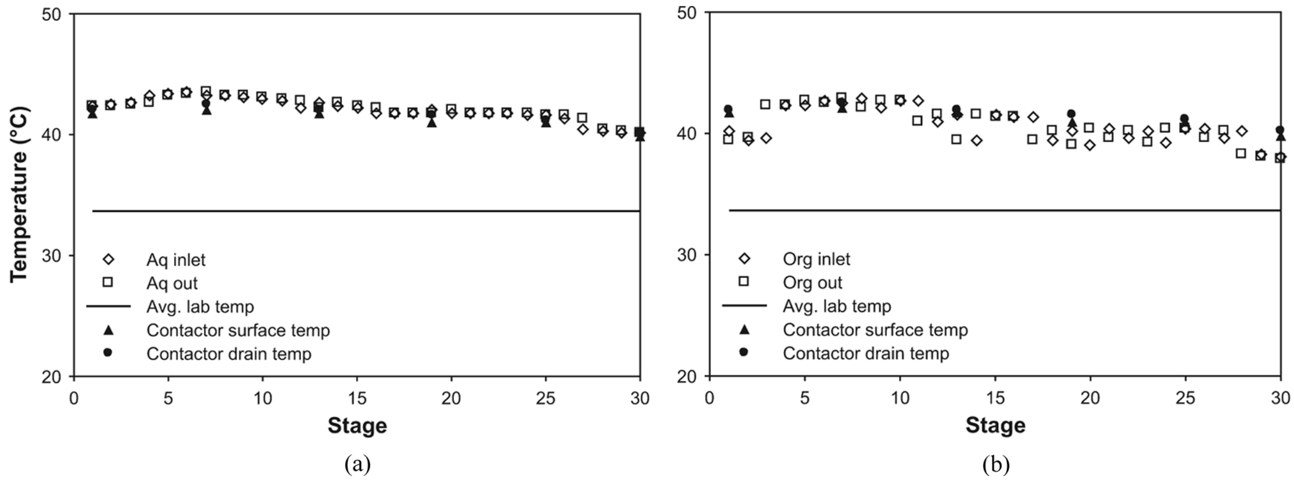


FIG. 14. Aqueous solution inlet/outlet stage temperatures (a) and organic solution inlet/outlet stage temperatures (b) and stage surface and drain temperatures at shutdown. Both phases heated to 40°C at 1.3 L/min at rotor speeds of 4000 rpm at an O/A ratio of 0.8 for 7.5 hours.

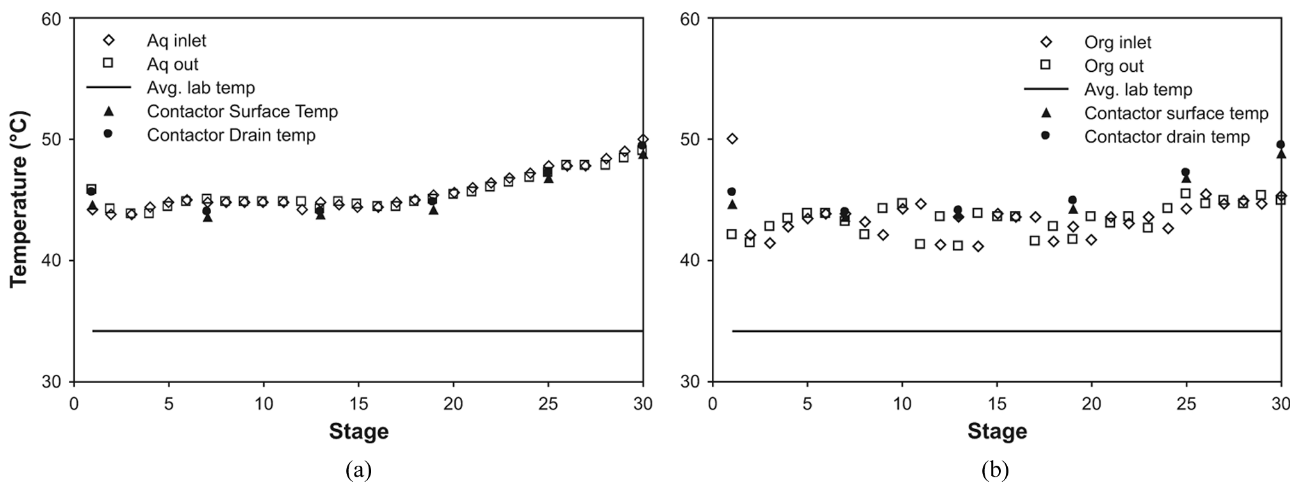


FIG. 15. Aqueous solution inlet/outlet stage temperatures (a) and organic solution inlet/outlet stage temperatures (b) and stage surface and drain temperatures at shutdown. Both phases heated to 50°C at 1.3 L/min at rotor speeds of 4000 rpm at an O/A ratio of 0.8 for 8 hours.

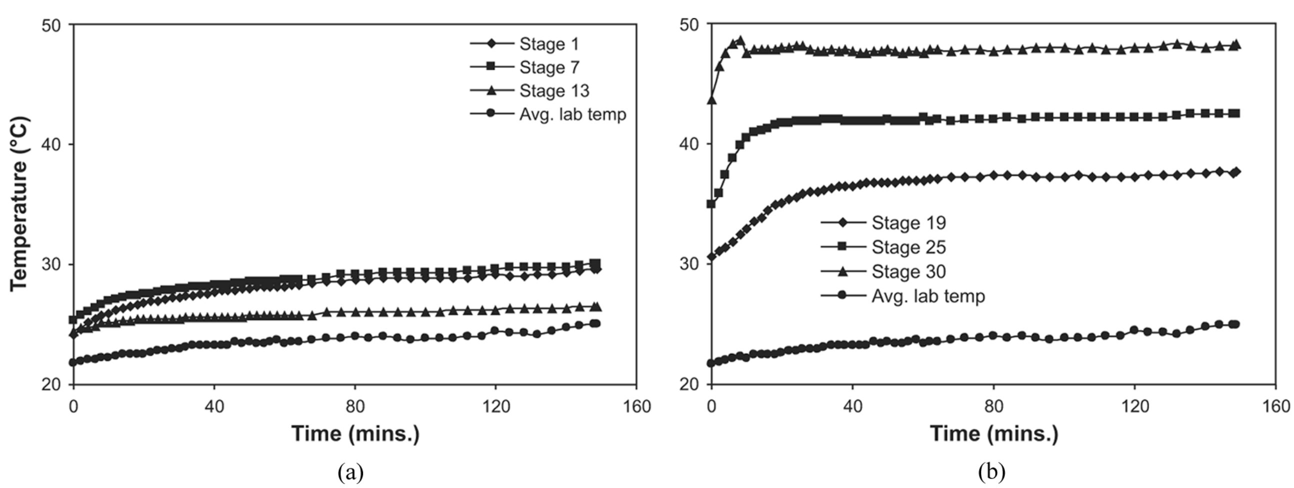


FIG. 16. Selected stage surface temperatures for the (a) extraction and (b) strip sections.

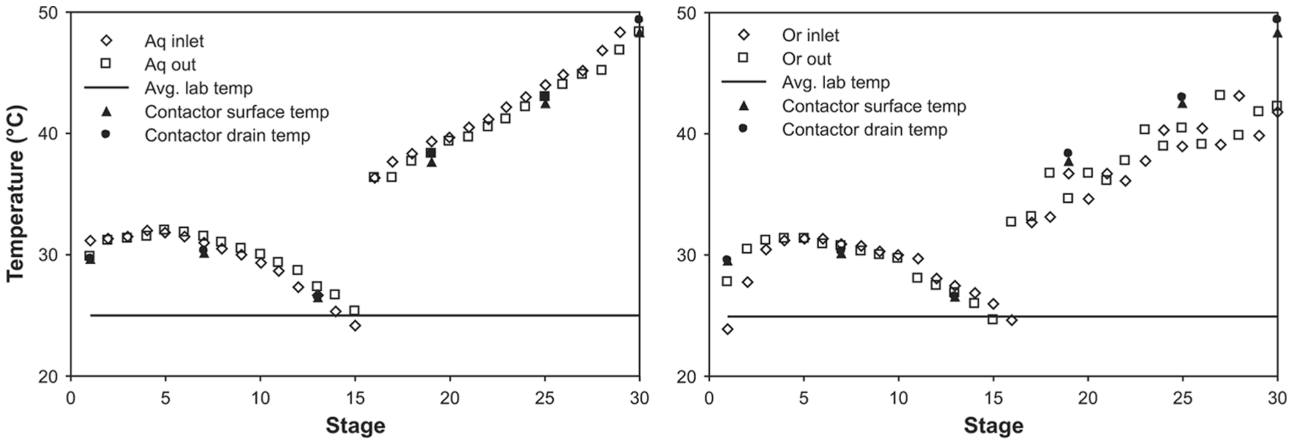


FIG. 17. Aqueous phase (a) and organic phase (b) solution temperatures for each stage for both the extraction and strip section with average lab temperature and surface and drain temperatures at shutdown.

the data spread but the temperature deviations are still quite small. Heat loss at 50°C is higher across the cascade with no large deviation in stage temperatures. The organic stage solution temperatures vary more between readings and both inlet and outlet temperatures are cooler than the rotor inlet solution temperatures or the aqueous inlet temperature.

Heated Strip Testing

This testing used a 15 stage extraction section and a 15 stage strip section. The unheated aqueous solution for the extraction section entered stage 15 and exited from stage 1. Aqueous strip solution heated to 50°C entered stage 30 and exited from stage 16. The ambient temperature organic solution entered stage 1 and exited at stage 30. All solutions were processed once-through with no recycle. The total flow rate for both extraction and strip sections was 1.3 L/min at an O/A ratio of 0.9. The contactor rotor speeds were set to 4000 rpm.

Figure 16 shows selected stage surface temperatures with average lab temperature as a function of time for the extraction and heated strip sections, respectively. Profiles of the extraction section show slight temperature increases in the three instrumented stages, over time. The heated strip section shows a cooling trend, overall. The heated strip section appears to reach steady-state after about one hour of operation.

Solution temperatures for each stage at shutdown for the heated strip testing are shown in Fig. 17. The aqueous temperatures at shutdown provide an excellent view of the profile of a 15 stage heated strip section and a 15 stage unheated extraction section operated at the same time. The 50°C heated aqueous strip solution does not significantly impact stage temperatures in the extraction section.

CONCLUSIONS

Single-phase testing demonstrated that water could be successfully processed by the 30 stage contactor assembly at rpm speeds from 3500–4000 rpm and total flow rates of 0.5–1.0 L/min. The need for higher rotor speed and some residual slight pulsing of the organic discharge indicated minor flow restriction between stages occurred. No other phase carryover was observed, however. The inter-connective tubing and fittings between contactors should be enlarged for enhanced flow through the 30 stage V-02 assembly.

Contactor surface thermocouples were in good agreement with process solution temperature measurements for all tests. Temperatures across the cascade stabilized within 30–60 minutes of startup using both heated and unheated feeds. Thereafter, gradual changes followed the ambient changes in laboratory temperature. Higher flow rates provide more rapid heat removal from the system even with increased contactor rotor speeds. Heat addition attributed to two-phase mixing was not measurable in these tests.

Heated strip testing for about 2.5 hours showed that steady-state temperatures are reached for all stages in about 30 minutes. No significant affect to the extraction section temperature profiles were observed due to heating of the strip solution.

At flow rates of 1 L/min, the temperature of the cascade is primarily driven by the aqueous feed temperature and secondarily by the ambient lab temperature. Therefore, it is likely that solvent extraction processes requiring chilled or heated inputs for increased efficiency can be operated using in-line solution heat exchangers for temperature control. The addition of thermal jacketing to individual contactors could be employed if more precise stagewise temperature control is required. However, insulation of the lower contactor housing alone may be sufficient for better control of heated or chilled stages. Mechanical heat contribution to the extraction system from motors, bearings, and seals appears to be minor as is the added heat from mixing of low viscosity fluids in the annular region of the contactor. These effects are readily neutralized by the input solution temperature adjustments as needed to attain the desired operating conditions. In addition, we believe that as contactor sizes and flow rates are increased, temperature profiles will not change appreciably. The added heat input of larger contactors, seals, and bearings will be offset by the increased heat transfer of the greater flow rate through the system. Flattening of the profiles is likely to occur and the thermal equilibrium of the contactor bank will be controlled by the input solution temperatures, to an even greater extent than shown in this study.

An analysis of heat transfer pathways in centrifugal contactors based on surface areas, motor temperatures, and equipment design specifics of centrifugal contactors is being performed by Argonne National Laboratory (ANL) (19). This analysis is being used to support the development of a model to predict solution temperatures of process stream in centrifugal contactor flowsheets. Data from the experiments reported herein are being used by ANL to support this model development.

ACKNOWLEDGEMENTS

The authors wish to thank Mitch Greenhalgh for his assistance with the assembly of the contactor cascade, Randy Bewley for software to drive the data acquisition and control system, and Bob Ristrem for much of the electrical assembly to power the whole system. We also wish to thank Janice Crank for her assistance in the organization and formatting of this manuscript and Christine White for graphics layout and design.

REFERENCES

1. Bernstein, G.J.; Grosvenor, D.E.; Lenc, J.F.; Levitz, N.M. (1973) A high capacity annular centrifugal contactor. *Nucl. Technol.*, 20: 200.
2. Leonard, R.A.; Bernstein, G.J.; Ziegler, A.A.; Pelto, R.H. (1980) Annular centrifugal contactors for solvent extraction. *Sep. Sci. Technol.*, 15 (4): 925.
3. Duan, W.; Song, C.; Wu, Q. (2005) Development and performance of a new annular centrifugal contactor for semi-industrial scale. *Sep. Sci. Technol.*, 40 (9): 1871.
4. Takeuchi, M.; Washiya, T.; Nakabayashi, H. et al. (2005) Engineering test of stripping performance by multi-centrifugal contactor system for spent nuclear fuel reprocessing. *13th Int. Conf on Nucl Eng.*, May 16–20, Beijing, China.
5. Okamura, N.; Takeuchi, M.; Ogino, H. et al. (2007) Development of centrifugal contactors with high reliability. Global, September 9–13, Boise, ID.
6. Geeting, M.W.; Brass, E.A.; Brown, S.J.; Campbell, S.G. (2008) Scale-up of caustic-side solvent extraction process for removal of cesium at Savannah River site. *Sep. Sci. Technol.*, 43 (9&10): 2786.
7. Zhou, J.; Duan, W.; Zhou, X.; Zhang, C. (2007) Application of annular centrifugal contactors in the extraction flowsheet for producing high purity yttrium. *Hydrometallurgy*, 85: 154.
8. Duan, W.; Zhou, X.; Zhou, J. (2006) Extraction of Caffeine with annular centrifugal contactors. *Solvent Extr. Ion Exch.*, 24 (2): 251.
9. Meikrantz, D.H.; Meikrantz, S.B.; Macaluso, L.L. (2001) Annular centrifugal contactors for multiple stage extraction processes. *Chem. Eng. Comm.*, 188: 115.
10. Meikrantz, D.H.; Macaluso, L.L.; Flim, W.D. et al. A new annular centrifugal contactor for pharmaceutical processes. *Chem. Eng. Comm.*, 189: 1629.
11. Meikrantz, D.H. Method for separating disparate components in a fluid stream. U.S. Patent 4,959,158, September 25, 1990.
12. Meikrantz, D.H.; Federici, A.G.; Macaluso, L.L.; Harris, P.J.; Sams, H.W. III Rotor sleeve for a centrifugal separator. U.S. Patent 5,571,070, November 5, 1996.
13. Meikrantz, D.H.; Macaluso, L.L.; Sams, H.W. III.; Schardin, C.H. Jr.; Federici, A.G. Centrifugal separator. U.S. Patent 5,591,340, January 7, 1997.
14. Meikrantz, D.H.; Macaluso, L.L.; Sams, H.W. III.; Schardin, C.H. Jr.; Federici, A.G. Centrifugal separator. U.S. Patent 5,762,800, June 9, 1998.
15. Macaluso, L.L.; Meikrantz, D.H. Self-cleaning rotor for a centrifugal separator. U.S. Patent 5,908,376, June 1, 1999.
16. Sheldon, B.V.; Flim, W.D.; Mendoza, G.; Macaluso, L.L. Method of making an easily disassembled rotor assembly for a centrifugal separator. U.S. Patent 6,363,611, April 2002.
17. Garn, T.G.; Meikrantz, D.H.; Mann, N.R.; Law, J.D.; Todd, T.A. (2008) Hydraulic and clean-in-place evaluations for a 12.5 cm annular centrifugal contactor at the INL. *Proc. Int. Solvent Extr. Conf.*, ISEC 2008, Tucson, AZ, 733.
18. Meikrantz, D.H.; Garn, T.G.; Law, J.D.; Mann, N.R.; Todd, T.A. (2008) Mass transfer testing of a 12.5 cm rotor centrifugal contactor. *Proc. Int. Solvent Extr. Conf.*, ISEC 2008, Tucson, AZ, 727.
19. Leonard, R.A. (2009) Personal communication, Argonne National Laboratory, July 2009.