INL/EXT-08-14861

Temperature Profile Measurements in a Newly Constructed 30-Stage 5 cm Centrifugal Contactor Pilot Plant

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The INL is a U.S. Department of Energy National Laboratory
operated by Battelle Energy Alliance

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GNEP-SEPA-PMO-MI-DV-2008-000133

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Prepared for the
U.S. Department of Energy
Office of Nuclear Energy
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

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September 30, 2008 iii

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Acknowledgements

The authors wish to acknowledge the contributions of Randy Bewley and Robert Ristrem for their efforts. Mr. Bewley’s expertise in LabVIEW™ programming, as well as electronics and process control systems, was instrumental in the completion of the automated process control and data acquisitioning systems. Mr. Ristrem’s tireless efforts to complete the wiring for the controllers and power boxes for all process electrical systems and completing the vast number of thermocouple terminations are greatly appreciated. The support and dedication of these two individuals towards the successful completion of this work was invaluable.

This work was performed under the auspices and financial support of the United States Department of Energy, Office of Nuclear Energy, Science and Technology through contract DE-AC07-05ID14517.

Summary

An annular centrifugal contactor pilot plant incorporating 30 stages of commercial 5 cm CINC V-02 units has been built and operated at Idaho National Laboratory (INL) during the past year. The pilot plant includes an automated process control and data acquisitioning system. The primary purpose of the pilot plant is to evaluate the performance of a large number of inter-connected centrifugal contactors and to evaluate the ability to control process temperature by controlling the temperature of the process solutions. Additional solvent extraction flowsheet testing using stable surrogates is also being considered for future testing.

Preliminary hydraulic testing was conducted with all 30 contactors interconnected for continuous counter-current flow. Hydraulic performance and system operational tests were conducted successfully but higher single-stage rotor speeds were found necessary to maintain steady interstage flow at flowrates of 1 L/min and higher. Initial temperature profile measurements were also completed in this configuration studying the performance during single aqueous and two-phase counter-current flow at ambient and elevated inlet solution temperatures. Temperature profile testing of two discreet sections of the cascade required additional feed and discharge connections. Lamp oil, a commercially available alkane mixture of C14 to C18 chains, and tap water adjusted to pH 2 with nitric acid were the solution feeds for all the testing described in this report.

Numerous temperature profiles were completed using the newly constructed 30-stage centrifugal contactor pilot plant. The automated process control and data acquisition system worked very well throughout testing. Temperature data profiles for an array of total flowrates (F_T) and contactor rpm values for both single-phase and two-phase systems have been collected with selected profiles and comparisons reported. The F_T ranged from 0.5-1.4 L/min with rotor speeds from 3500-4000 rpm. Inlet solution temperatures ranging from ambient up to 50° C were tested.

Ambient temperature profile results show that a small amount of heat is generated by the mechanical energy of the contactors. Heated inlet solution testing provides temperature profiles with smaller temperature gradients which are more influenced by the temperature of the inlet solutions than the ambient lab temperature. The temperature effects of solution mixing, even at 4000 rpm, were insignificant for all of the studies conducted using lamp oil and water.

Results indicate it is feasible that solvent extraction processes utilizing the CINC V-02 centrifugal contactors and requiring chilled or heated temperatures for increased efficiency can be operated using in-line feed solution heat exchangers for adequate temperature control. The addition of thermal jacketing to individual contactors would only be required for processes needing precise stage temperature control. Therefore, the added cost and complexity of thermal jackets and associated connections may not be necessary.

These data will be provided to Argonne National Laboratory (ANL) to support development of a computer model which predicts temperature profiles in centrifugal contactor flowsheets.

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TABLES

ACRONYMS

AMUSE	Argonne Model for Universal Solvent Extraction
ANL	Argonne National Laboratory
CIP	Clean-in-Place
F_T	total flowrate (also considered as total throughput)
HDPE	high-density polyethylene
IEDF	INL Engineering Demonstration Facility
INL	Idaho National Laboratory
IRC	Idaho Research Complex
LI	Laboratory Instruction
LWP	Laboratory-wide Procedure
MCP	Management Control Procedure
MFC	Materials and Fuels Complex
O/A	organic to aqueous flowrate ratio
PC	personal computer
PID	proportional integral derivative
R&D	research and development
SO	system operability
STC	Science and Technology Campus

SEPARATIONS CAMPAIGN TEMPERATURE PROFILE MEASUREMENTS IN A NEWLY CONSTRUCTED 30-STAGE 5 CM CENTRIFUGAL CONTACTOR PILOT PLANT

1. INTRODUCTION AND BACKGROUND

An annular centrifugal contactor pilot plant consisting of 30 stages of commercial 5 cm CINC V-02 units has been built and operated at Idaho National Laboratory (INL) during the past year. The purpose is to evaluate, on a pilot plant scale, the performance of a large number of pilot scale centrifugal contactors during operation. Operational data to support solvent extraction system design; temperature profiles across the cascade under both ambient and elevated feed solution temperatures, hydraulic performance, and stage wise efficiency at various locations during start-up and at equilibrium process conditions are needed.

Recent pilot scale centrifugal contactor studies at INL have been limited to single-stage performance measurements. To date, test reports for CINC V-02 (5 cm) and V-05 (12.5 cm) contactors have been published that provide both hydraulic and mass transfer data as well as reliability and Clean-in-Place (CIP) capability of these commercially available units.[1,2,3,4] Combined contactor sizes tested exhibit good performance in the total flowrate (F_T) range of 0.2 to 30 L/min. In addition, single unit mass transfer efficiencies are quite high for processes with rapid kinetics, nearly 100%. However, as most flowsheet tests performed with 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, stage-wise sampling at low flows can upset process equilibrium so it is avoided in mini-contactors. These factors can lead to an over-estimation of the number of stages required for a particular flowsheet. Pilot scale testing provides design data for steady-state operating parameters and stage-wise efficiency for flowsheets where stable element surrogates can be used. This 30-stage V-02 cascade has been assembled to test those fuel cycle flowsheets with stable surrogates, to provide performance data for models such as Argonne Model for Universal Solvent Extraction (AMUSE), and to study detailed stage-wise temperature profiles under varied operating conditions. Detailed temperature profile studies provide information needed to enhance solvent extraction flowsheet performance. This testing will also assess solution temperature control options such as in-line inlet solution heaters and their ability to maintain solution temperatures across a given number of stages as compared to other solution heating equipment such as thermal jackets on individual stages.

Design and assembly of the 30 stage V-02 cascade began in early FY-08. The support stand was designed to accommodate the contactors in one level extending almost 25 feet in length. In addition, it provides mounting provisions for metering pumps, in-line feed solution heat exchangers, contactor variable frequency drives, 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 and rotor inlet streams of selected units, and ambient lab temperatures at both ends of the cascade. In addition, data for feed flowrates, individual contactor rotor speed and amperage level, are collected by a data acquisition system at selected time intervals. Operation of all contactors, pumps and heaters is also centralized by the computer program with control via keyboard input.

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 alkane mixture of C14 to C18 chains, and tap water adjusted to pH 2 with nitric acid were the feeds for all the testing described in this report.

1.1 System Description

A modular centrifugal contactor pilot plant has been constructed at the INL. The pilot plant is sectioned into five identical modules each supporting 6 stages and a fluid feed system. The entire cascade consists of 30 inter-connected stages all mounted on a stainless steel support structure. All fluid process equipment is contained within a spill containment system. The pilot plant also has automated process control capabilities coupled with a data acquisition system. A photo of the completed pilot plant is shown below in Figure 1.

Figure 1. Completed 30-stage pilot plant.

1.1.1 Mechanical Equipment

The mechanical equipment making up 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.

The centrifugal contactors are constructed of stainless steel (316L) and have housings with incorporated phase collector rings and inlet/outlet flow tubes. 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 contactors were obtained commercially from CINC Processing Equipment, Inc. They are rated for a F_T operating range 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 V7N frequency drives enabled with DeviceNet™ communication for personal computer (PC) remote control operation. Contactors were installed as received from the manufacturer with no additional heat exchanging equipment such as thermal jackets added.

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 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 by the PC through a 0 to 10 VDC analog signal. Additional components included in the fluid feed system are flowmeters, pulse dampeners, back pressure valves, heaters and in-line filters. The turbine type flowmeters used to measure flow on each feed system were obtained commercially from FTI Flow Technology™, Inc. The flowmeters have a flow 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 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™, Inc. One, 10 cubic inch, and four 36 cubic inch volume 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™, 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 X 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™ PM heater controllers. Each heater has its own J-type thermocouple embedded in the heater for optimum process control. The controllers also communicate with the PC via DeviceNet™. Stainless steel Swagelok® in-line filters were installed to protect the flowmeters from damaging particulates. Filters are nominally rated at 100 micron and can be easily removed for cleaning by sonication. 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 stainless steel 3/8-in. tubing and various Swagelok® fittings. A photograph of a typical fluid feed system is found in Figure 2.

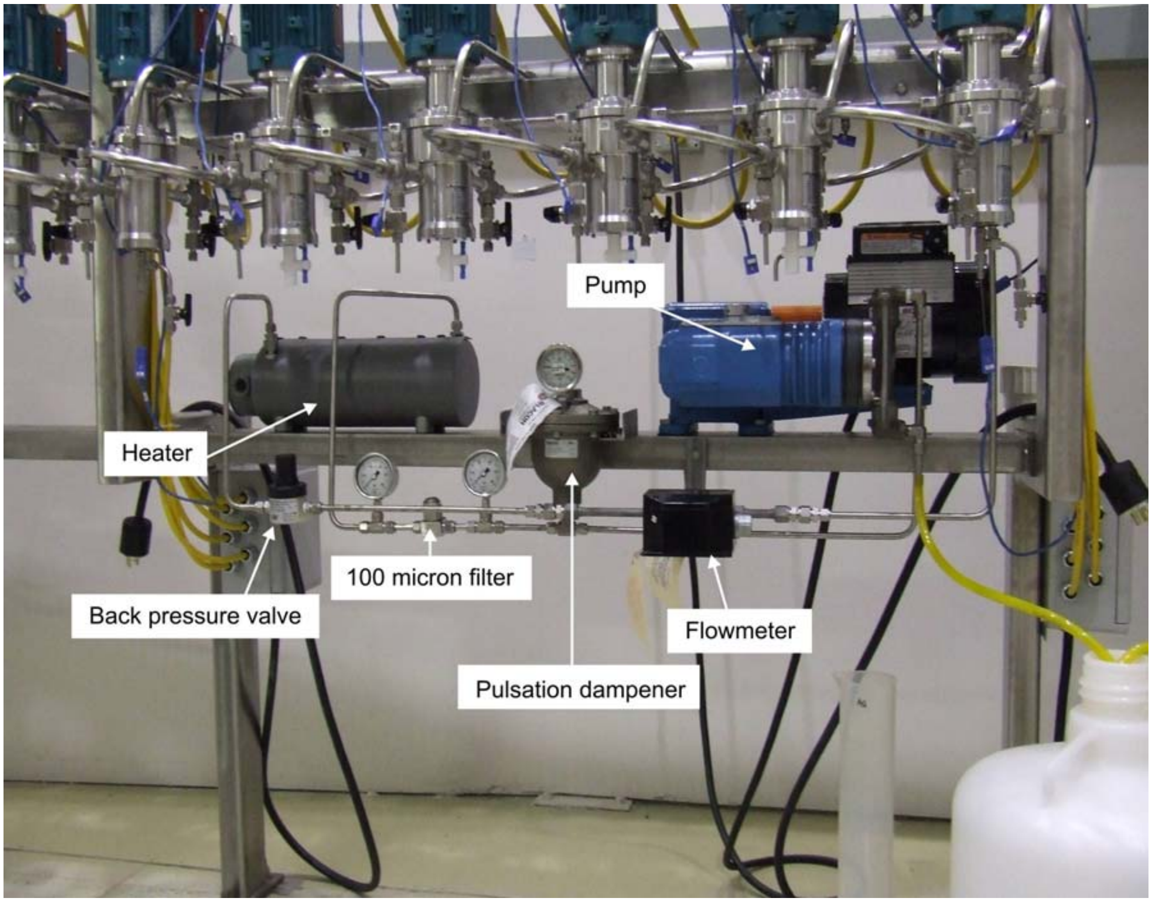

Figure 2. Typical fluid feed system with components.

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 contactor inlet thermocouples are 1/32-in. diameter and 6-in. in length. The drain and lab temperature thermocouples are 1/16-in. diameter and also 6-in. 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 narrow temperature range of -250º C to 350º C and a tolerance of ± 1% making them the proper choice for this application. All thermocouples communicate with the PC through a National Instruments™ thermocouple chassis.

1.1.2 Automated Process Control and Data Acquisition

Hardware supporting the automated process control configuration and data acquisition system was purchased from National Instruments™™ and housed in a metal cabinet adjacent to the pilot plant. 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™ communications. The thermocouple terminal block modules and amplifiers for thermocouple connectivity and communications are also housed in the cabinet. LabVIEW™ version 8.2 was incorporated as the graphical user interface for process signal to automated control.

The functionality of the automated process control consists of full control of pump speed for flowrates in either manual or automatic mode, contactor on/off and rpm adjustment, solution temperature heating via the heater controller, and real-time process monitoring of temperature and flowrates.

Programs generated in LabVIEW™ enabled 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 positioning. The setup screen allows for five processes i.e. extraction, scrub, strip, etc. to be selected and uniquely identifies each with a different color for stages selected for each process, allowing for maximum flexibility of process configuration selections. There is also a window to allow users to input comments into the data file header. Once the setup screen has been finalized the user is directed to a process control screen that is used 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 initiated. The user can also select graphical representations of desired pump charts for real-time flowrate monitoring. Figure 3 and Figure 4 include pictorial representations of a typical setup screen and process control screen. This theoretical flowsheet would include 10 extraction stages, 6 scrub stages, 10 strip stages, 2 wash and 2 reacidification stages. Each stage icon provides real-time data associated with that stage. The pump and heater icons are also color coordinated to reflect process affiliation. Stages 1, 7, 13, 19, 25, and 30 are designated to have drain and surface thermocouples installed in the representation.

1.1.3 Miscellaneous Equipment

Feed and product tanks were positioned directly below the 30-stage cascade. A total of 9 tanks are available for use with seven having a capacity of 100 gallons and two with a 70 gallon capacity. The tanks are constructed of heavy-walled 3/8-in. high-density polyethylene (HDPE) with a 6-in. diameter cleanout port installed on top. Inlet/outlet supply lines were inserted into the tanks via stainless steel fittings.

The off-gas system consists of a 3-in. diameter common vent header mounted on the support structure directly behind the contactors. Stainless steel 1/2-in. 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.

The electrical supply consists of a transformer, power panel, wiring and outlets that provide power circuitry exclusively to the pilot plant electrical components.

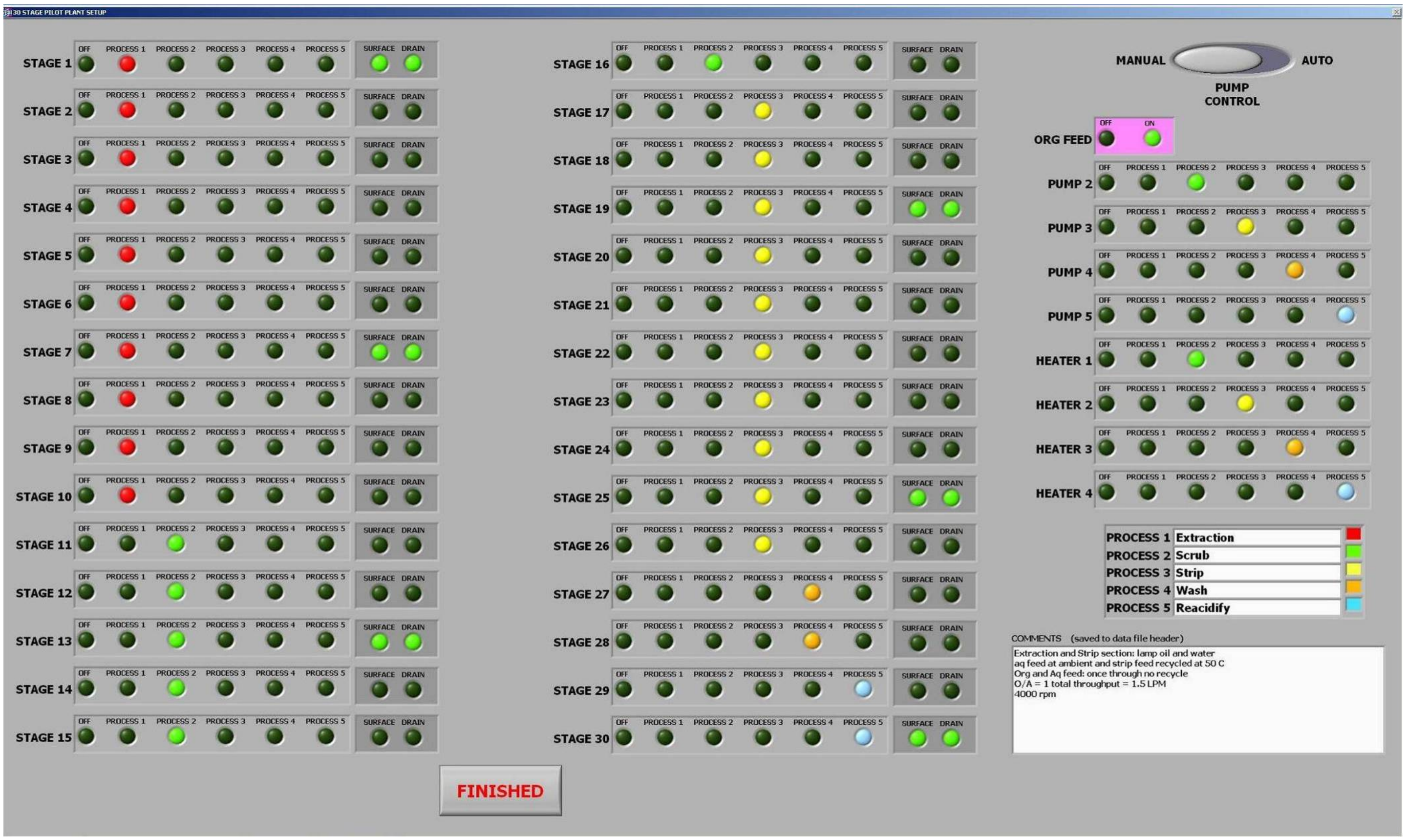

Figure 3. Typical setup screen.

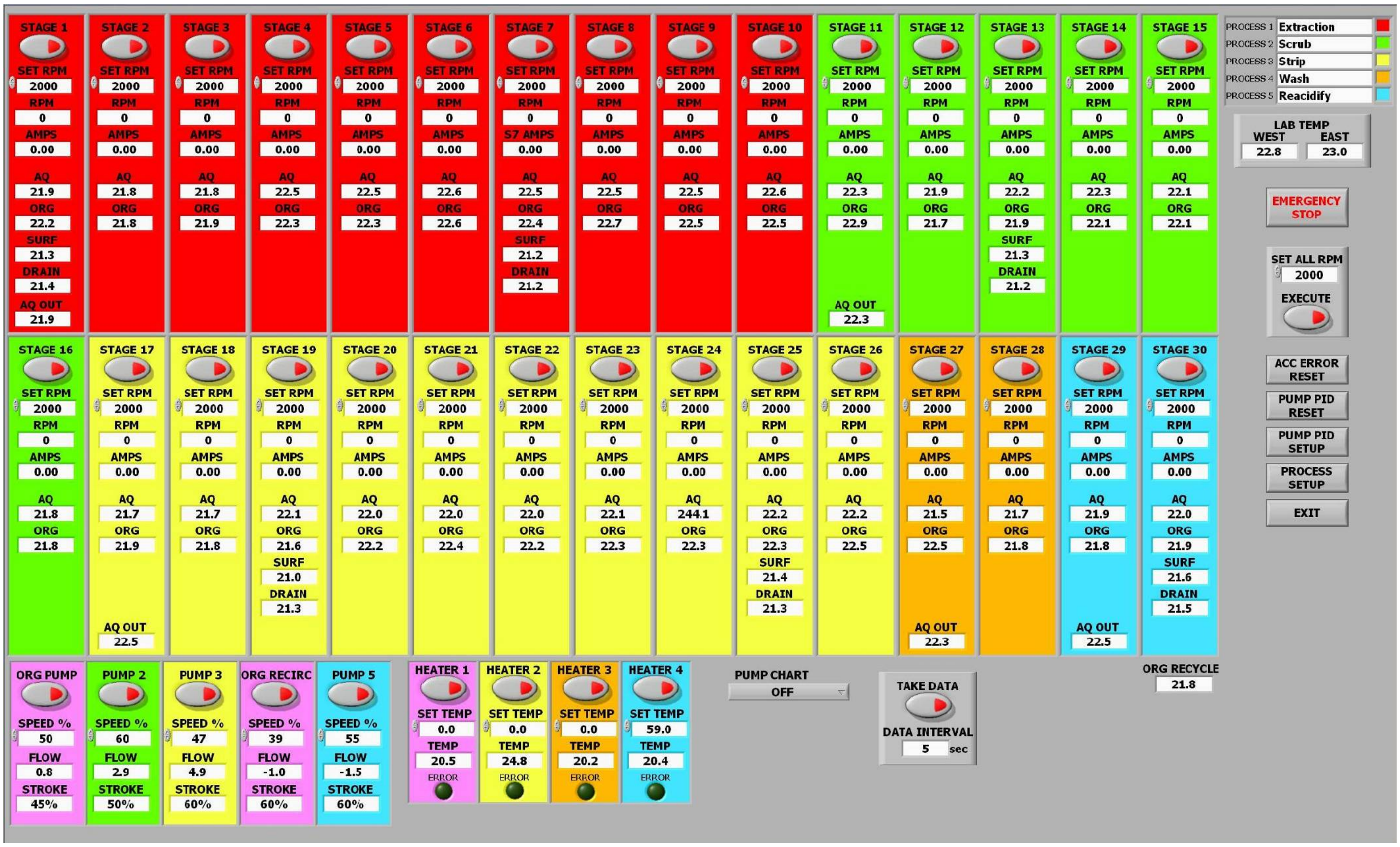

Figure 4. Typical control panel**.**

1.1.4 Quality Assurance

This research and development (R&D) activity is defined as Applied Research per INL procedure Laboratory-wide Procedure (LWP)-13016, “Applying Quality Assurance Requirements to Research and Development Activities.” The Quality Level of this activity was evaluated using the Quality Level Determination Process (Science and Technology Campus [STC]-000171). Several activities have been determined to be Quality Level 2 including measurement of flow rate input to contactors and temperature measurements taken at various locations on the pilot plant.

As a result of this analysis, the following controls were implemented for this research:

1. Testing was documented in controlled Laboratory Notebook # Lab-1125 in accordance with requirements of Management Control Procedure (MCP)-2875, Maintaining Laboratory Notebooks.
2. The work has received independent peer review.
3. The completed Laboratory Notebook is controlled as a Quality Record.
4. Temperature and flow measurements were performed using calibrated instruments. Calibration methods are documented in Laboratory notebook and paperwork retained in the Project file.
5. The experimental work was performed within the work control defined in Laboratory Instruction (LI)-1236-07-IEDF/IRC “Centrifugal Contactor Testing.”

1.2 Pilot Plant SO Testing

1.2.1 Fluid Feed Systems and Contactors

The first phase of system operability (SO) testing was to evaluate fluid feed system component performance. Water was pumped through each system to determine optimum pressure settings on back pressure valves and pulse dampeners to obtain desired flow ranges with minimum pulse flow. During this testing, all plumbing and components were thoroughly leak checked. Pump calibrations were completed using flowrate measurements at a multitude of pump speed and stroke settings. Flowmeter accuracies were also confirmed to be within manufacturers’ reported ± 1% of actual flow by manually measuring the flowrate at the flowmeter outlet with graduated glassware.

The next phase of SO testing included the operation of the 30 contactor stages. Water was pumped through the entire cascade at flowrates ranging from 0.3-1.5 L/min while rotor speeds were adjusted from 3000-4500 rpm. Flow exiting the last stage was visually observed for pulsing. It became evident early that there was some issue with the contactor operation. Nearly every test condition provided pulsing flow at the cascade outlet and water presence was noticed in several stages in the light phase inter-connective tubing in the sample ports. Examination of the cascade revealed that the contactor support rails were not adequately supporting the units in a horizontal position. A 1 × 2-in. stainless steel leveling bar was attached to the support legs of the structure allowing for leveling adjustments to be made to individual stages as needed. Resulting tests indicated the leveling bar modification did reduce the outlet pulse flow for most test conditions but did not alleviate it entirely. In addition, a secondary cause to pulse flow is the reduced inside diameter of the outlets on each contactor due to fitting connections for inter-connective tubing between stages. Higher than normal single-stage operational rotor speeds were found necessary to over come this resistance to discharge flow at the contactor outlets. At the end of this testing phase, a final determination of minimum speed of 3200 rpm for flowrates below 0.5 L/min and a maximum speed of 4000 rpm for F_T ranges from 0.5 to 1.5 L/min was required to minimize or eliminate pulsing in the cascade discharge flows. Slight pulsing of the organic discharge was still observed but no other-phase entrainment resulted during these tests.

1.2.2 Automated Process Control

Automated process control SO testing was completed in a step-wise fashion. As a sub-program was written for each component, that component would then be tested. Thermocouple readouts were first evaluated. Each thermocouple was checked for proper operation and indicator position within the control panel. Additionally, a quality check was made on all thermocouples to confirm the accuracy reported in the calibration standard accompanying the thermocouples from the manufacturer.

The next component to be checked for proper communication with the control system was the contactors. The contactors were all remotely energized and speed adjustments made to ensure the DeviceNet™ communication connections were correct. Since the heater controllers were also DeviceNet compatible, those were next SO tested. Contactors and heater controls were found to be operating as desired.

SO testing continued with the communications and control for the liquid feed pumps and associated flowmeters. Each fluid feed system was tested in both automatic and manual mode. The automatic mode included the selection of appropriate proportional integral derivative (PID) settings to provide optimum flow patterns for broad flow ranges. The fluid feed system incorporating the 10 cubic inch pulse dampener had high pulsing flow at the outlet. The 10-in. pulse dampener was inadequately sized for this fluid feed system. As a result of this higher pulsing, the pump control loop could not be operated satisfactorily in auto mode and thus actual testing would require manual mode pump control.

The next system to be SO tested was the data acquisition system. Numerous flowsheet configurations were selected and operated for short times to ensure the process variable values and flowsheet configurations were properly positioned in the intended data file. At the completion of the SO testing, the sub-programs were all incorporated into one all-encompassing program and made into an executable program.

To confirm the operation of the completed executable program, a final SO test was performed. All components were energized intermittently while water was processed through the cascade. Each fluid feed system was energized including heaters to ensure proper operation. Data was collected throughout this test. At the conclusion of the SO test, the data file was thoroughly investigated for accuracy and that all data values were properly positioned in the file. This test confirmed that the pilot plant was ready to begin temperature profile experimentation.

1.3 Temperature Profile Experimental Test Description

Thermocouples used to measure inlet/outlet solution temperatures for each stage are installed through the top of cross fittings installed at each contactor inlet 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 the west and east end of the cascade directly behind stage 4 and stage 27 and at contactor height for ambient lab temperature measurement. The drain thermocouples are inserted upward through housing drain lines and extend 1/4-in. 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 in. above the housing base. This distance allows for all mixing heights associated with test flowrates 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 SO testing. The off-gas vent header was plumbed into the facility exhaust system and flow adjusted to provide adequate venting of the contactor cascade and feed tanks.

1.3.1 Single-Phase Testing

Multiple temperature profile experiments were performed with the 30-stage pilot plant. Preliminary testing with the pH 2 water 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 solution inlet temperature heated to ~40° C at two flowrates of 0.5 and 1.0 L/min at rotor speeds of 3750 and 4000 rpm, respectively. Aqueous solution was passed once-through the cascade for each of these four tests. Average runtime was approximately 2.5 to 4 hours, dependant on flowrate.

To finalize the single-phase testing, two extended run experiments while heating the inlet solution to ~40° C then ~ 50° C were completed. Flowrates of 0.5 and 1.0 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 cascade. Temperature profile data was collected throughout the extended runs. Runtime for these two extended runs was ~ 8 hours.

1.3.2 Two-Phase Testing

Two-phase testing was performed using tap water, adjusted to pH 2 with nitric acid, and lamp oil. Lamp oil is a paraffinic hydrocarbon that is a liquid at room temperature. Aqueous solution entered the cascade at stage 30 and organic solution entered at stage 1. Three tests were performed with this configuration. The first test provided a baseline ambient temperature profile, followed by two extended 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 recirculated throughout the duration of the extended tests. The ambient temperature test lasted for about 3 hours while the two extended tests were ran for nearly 8 hours. The (F_T) was ~1.5 L/min with rotor speed of 4000 rpm for all three tests. All two-phase testing was completed at organic to aqueous flowrate ratio (O/A) ratios of ~1. Actual O/A ratios were determined from measured effluent flowrates during two-phase testing.

A final two-phase extraction/heated strip test was also completed. This test was performed to evaluate a temperature profile across both a 15 stage extraction section and a 15 stage strip section. The same two-phase test solutions were used in both sections with the aqueous strip solution heated to 50° C before entering stage 30 while the extraction aqueous feed was at ambient temperature before entering 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 before exiting stage 30. The target F_T for both extraction and strip sections was ~1.5 L/min. There was no recycle of solutions for the extraction/heated strip test, hence the test lasted for only 2.5 hours.

Table 1 provides a summary of the test parameters used for all single and two-phase temperature profile testing.

Table 1. Summary of temperature profile test conditions.

Phases Tested	Feed Solution Temperature (° C)	O/A Ratio	Total Flowrate (F_T) (L/min)	Rotor Speed (rpm)	Recycle	Test Duration (hrs.)
Aqueous only	ambient (24)	n/a	0.5	3500	No	3.3
Aqueous only	ambient (23)	n/a	1.0	4000	No	2.5
Aqueous only	37	n/a	0.5	3750	No	4.0
Aqueous only	35	n/a	1.0	4000	No	2.7
Aqueous only	38	n/a	0.5	3750	Yes	8.0
Aqueous only	ambient (23)	n/a	1.0	4000	No	2.5
Aqueous only	37	n/a	0.5	3750	No	4.0
Aqueous only	35	n/a	1.0	4000	No	2.7
Aqueous only	38	n/a	0.5	3750	Yes	8.0
Aqueous only	45	n/a	0.5	3750	Yes	7.0
Aqueous/ Organic	ambient (25)	1.0	1.4	4000	No	2.7
Aqueous/ Organic	Both phases 40	0.8	1.3	4000	Yes both phases	7.5
Aqueous/ Organic	Both phases 50	0.8	1.3	4000	Yes both phases	8.0
Aqueous/ Organic	Ext. Aqueous ambient (24) Strip Aqueous 50	Ext. 0.9 Strip 0.9	Ext. 1.3 Strip 1.3	4000	No	2.5

Aqueous = pH 2 tap water   Organic = Lamp oil

2. RESULTS AND DISCUSSION

2.1 Single-Phase Testing

Temperature profiles can be used 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 solution flow was performed at two F_T and two rotor 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 5 and Figure 6 include 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; one to show the temperature change of aqueous solution between inlet and outlet stages over time, and more importantly, to show that the aqueous solution temperatures agree within thermocouple error (± 1.0° C) with the surface and drain temperatures measured for each stage. This demonstrates that the surface and drain temperatures provide good estimates of the solution temperatures for respective stages. The data points for the averaged lab temperature curve represent the average of the west end and east end temperatures at given time. The averaged lab temperature curve in Figure 5 fluctuates due to the constant changing of the ambient lab temperature as a result of neighboring exterior lab roll-up doors being opened and closed while testing was performed. The distance between data points is directly related to the assigned data collection intervals during testing. For example, as indicated in Figure 6, data collected during the first 60 minutes and the last ten minutes of testing were collected at faster intervals than those taken from 60 to 140 minutes.

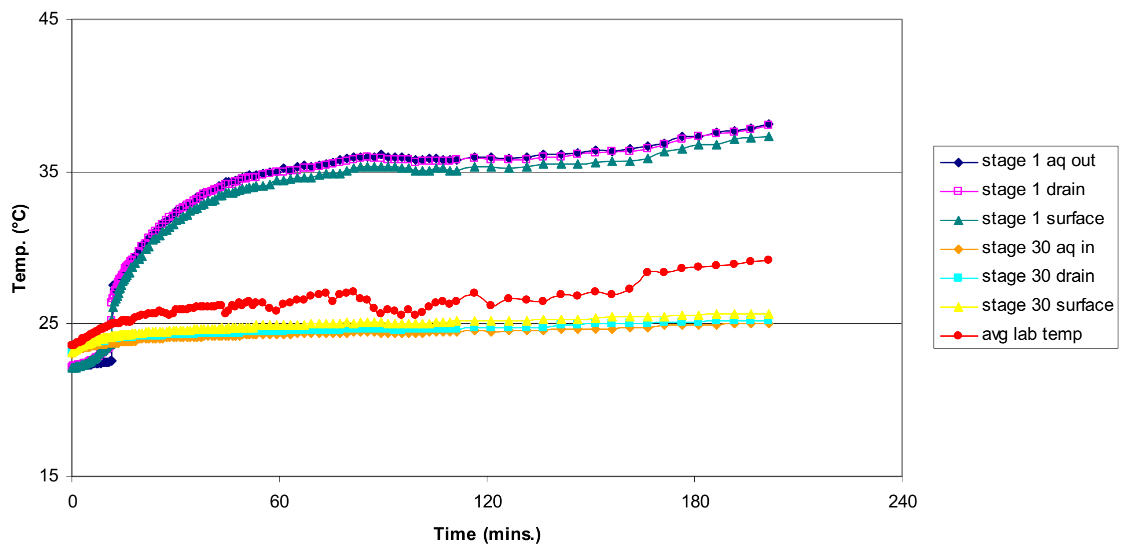

Figure 5. Solution inlet/outlet temperature profile at F_T of 0.5 L/min and rotor speed of 3500 rpm with no recycle. Drain temp refers to temperature measurement of process solution entering at rotor inlet.

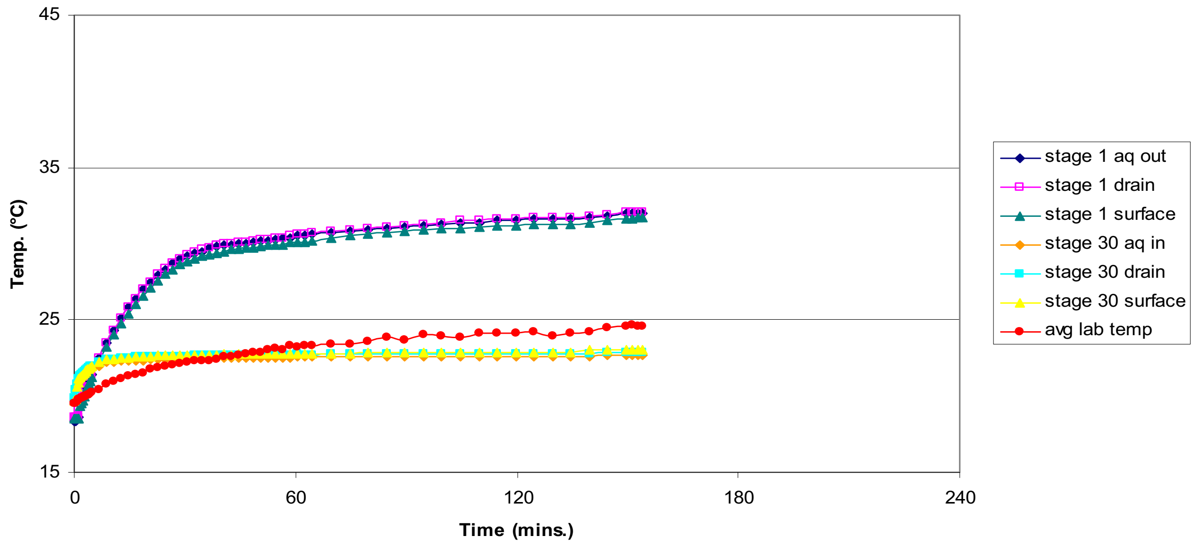

Figure 6. Solution inlet/outlet temperature profile at F_T of 1 L/min and rotor speed of 4000 rpm with no recycle. Drain temp refers to temperature measurement of process solution entering at rotor inlet.

The data in Figure 5 and Figure 6 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. Also of importance to note is that the differential temperatures between inlet and outlet is slightly higher when the F_T is lower even with increased rotor speed at the higher flowrate. This indicates that higher flows provide more heat removal from the system.

To further evaluate the temperature profile across the 30-stage cascade at ambient temperature test conditions, graphs were constructed to show profiles of selected stages across the cascade over time. Figure 7 and Figure 8 include the contactor surface temperature of the selected stages and average lab temperature over time for each test condition.

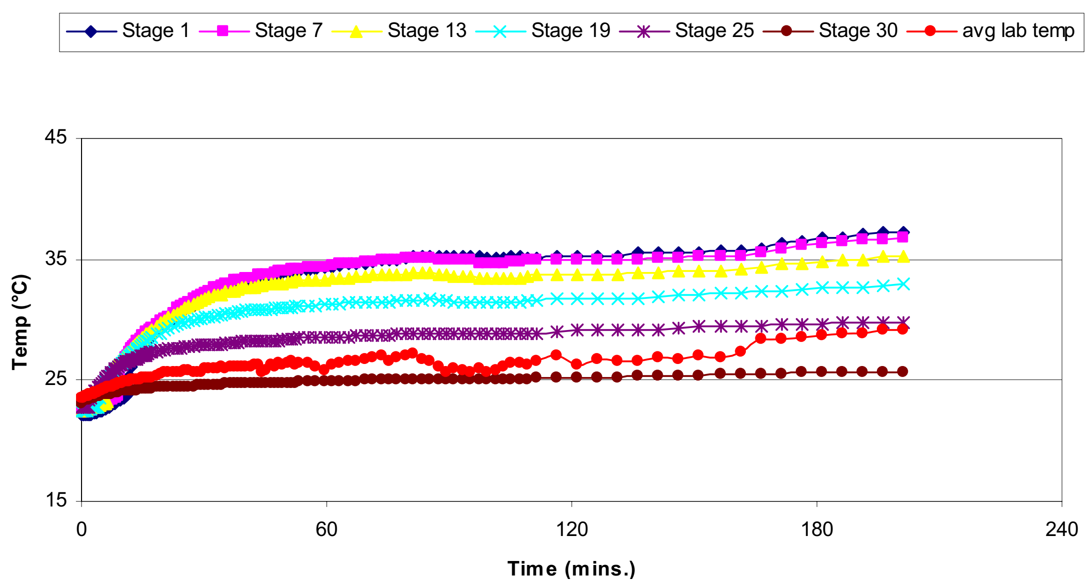

Figure 7. Surface temperatures of selected stages at F_T of 0.5 L/min and rotor speed of 3500 rpm.

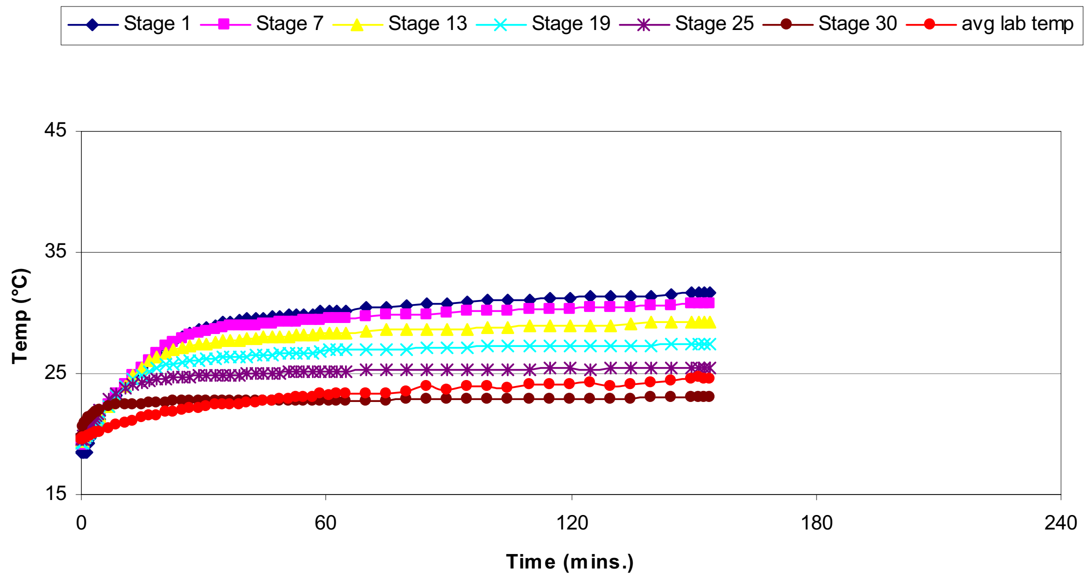

Figure 8. Surface temperatures of selected stages at F_T of 1 L/min and rotor speed of 4000 rpm.

Another set of graphs were constructed to provide final temperatures for all stages at shutdown for both test conditions. Figure 9 and Figure 10 include inlet/outlet solution temperatures with surface and drain temperatures for all stages at shutdown. The averaged lab temperature shown was the average of the east and west lab thermocouple readings at shutdown.

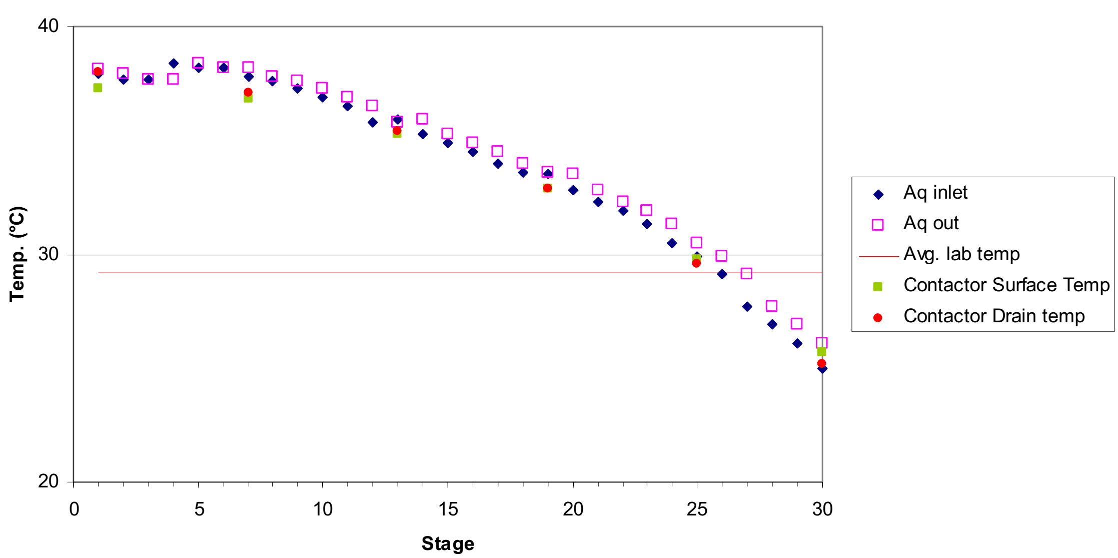

Figure 9. Temperature profile for all stages at shutdown at F_T of 0.5 L/min and rotor speed of 3500 rpm.

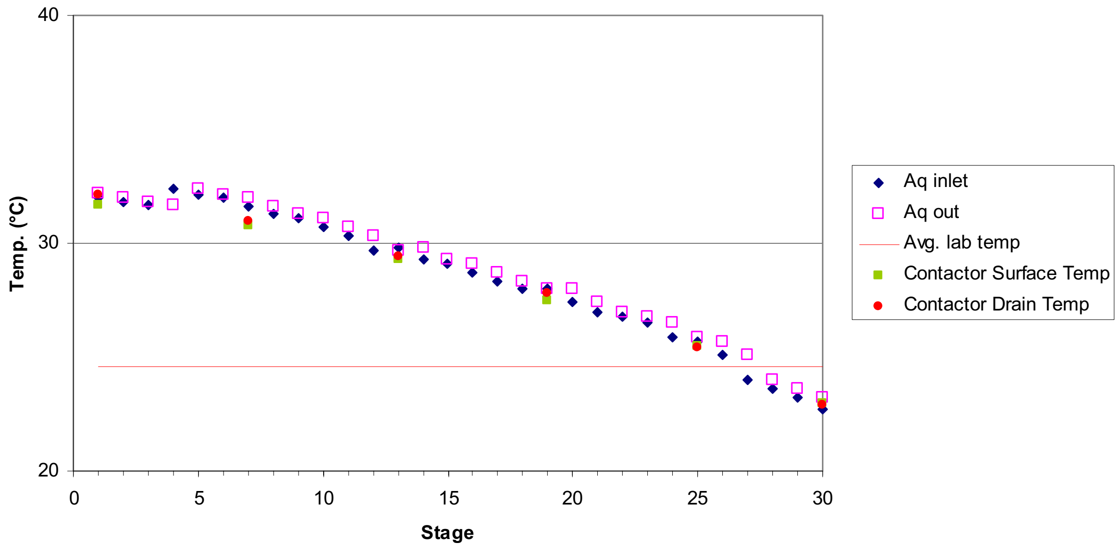

Figure 10. Temperature profile for all stages at shutdown at F_T of 1 L/min and rotor speed of 4000 rpm.

Temperature profiles provided in Figures 9 and 10 show that the increase in F_T provides more rapid heat removal from the system even with increased contactor rotor speeds.

2.2 Heated Single-Phase Testing

Four single-phase tests were completed with heating the aqueous solution. The first two test solutions were heated to ~ 40° C and considered once-through tests with no recycling of the solution. The final two were extended run tests where the solution was collected in a product tank and recycled back to the feed tank for continuous operation. The latter test solution was heated to nearly 50° C. 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 desired (as documented in

Table 1). Figure 11 and Figure 12 include selected stage surface temperature profiles with associated averaged lab temperature for the two heated tests performed with no recycle.

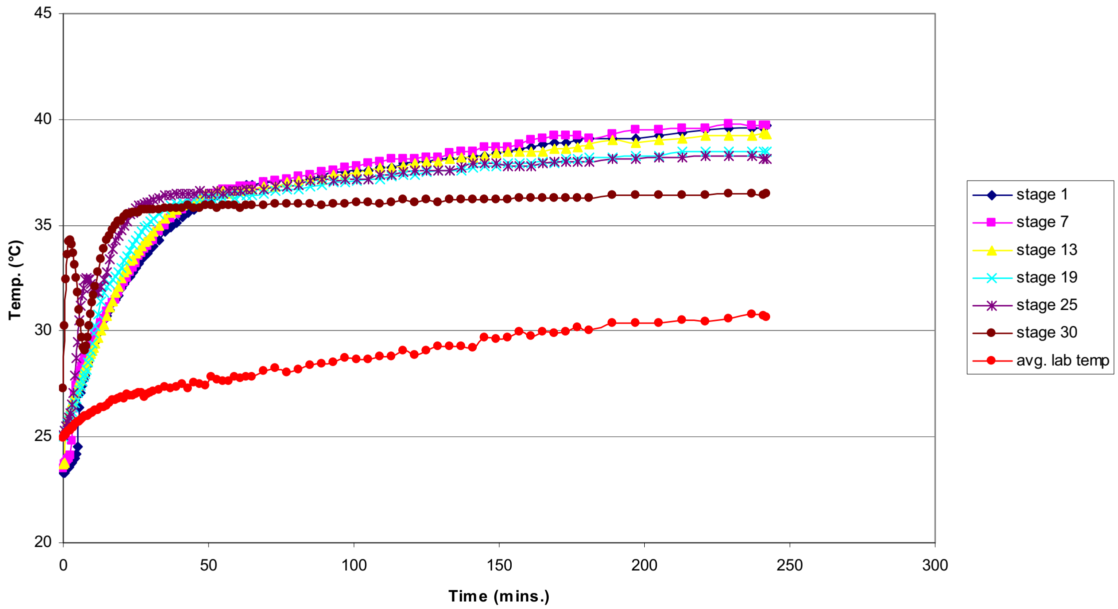

Figure 11. Surface temperature profile for F_T of 0.5 L/min and rotor speed at 3750 rpm with solution inlet temperature of 37° C and no recycle.

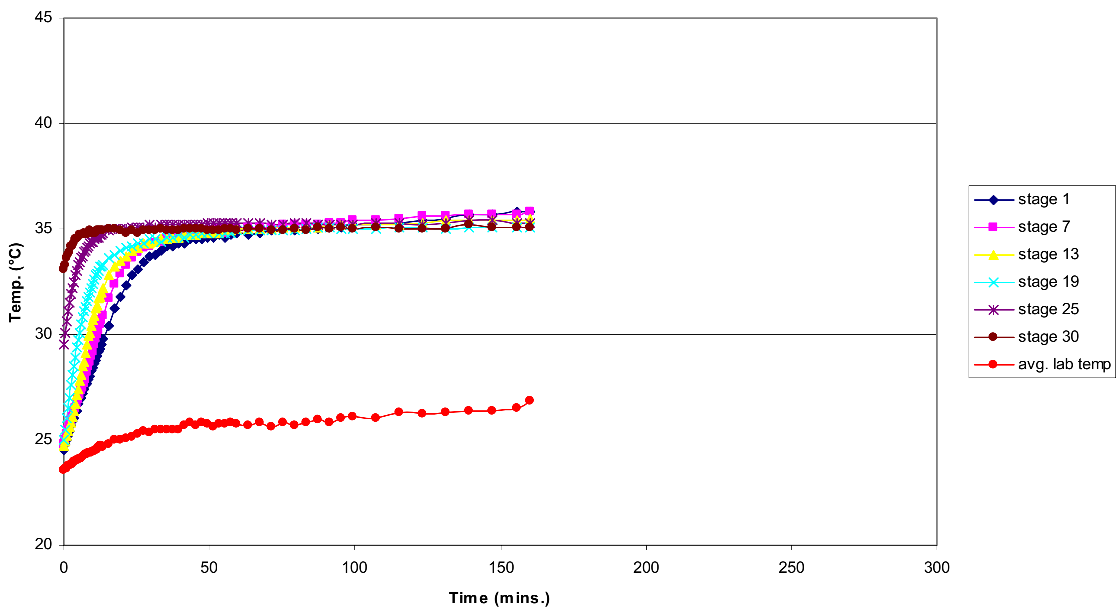

Figure 12. Surface temperature profile for F_T of 1 L/min and rotor speed at 4000 rpm with solution inlet temperature of 35° C and no recycle.

In comparing graphs in Figure 11 and Figure 12, it is quite evident that the increased flowrate associated with Figure 12 removes more heat from the system and the stage temperatures are nearly equal to the inlet solution temperature whereas in Figure 11, all stage surface temperatures, excluding stage 30, are greater than the inlet solution temperature.

Two graphs to illustrate all stage temperatures at shutdown for the single-phase heated tests with no recycle are included in Figure 13 and Figure 14.

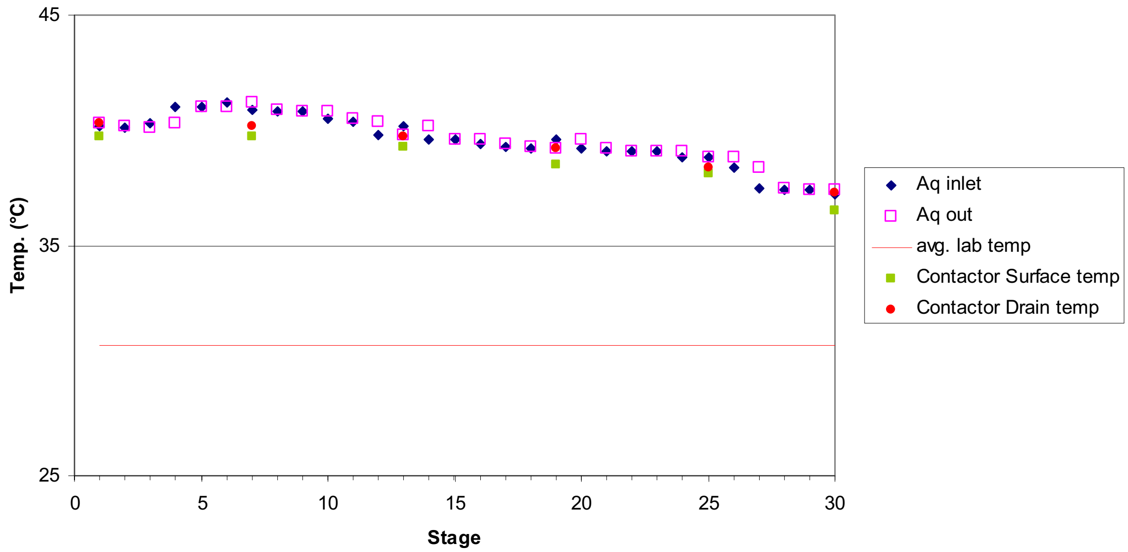

Figure 13. Temperature profile for all stages at shutdown at F_T of 0.5 L/min and rotor speed of 3750 rpm with inlet solution temperature of 37° C with no recycle.

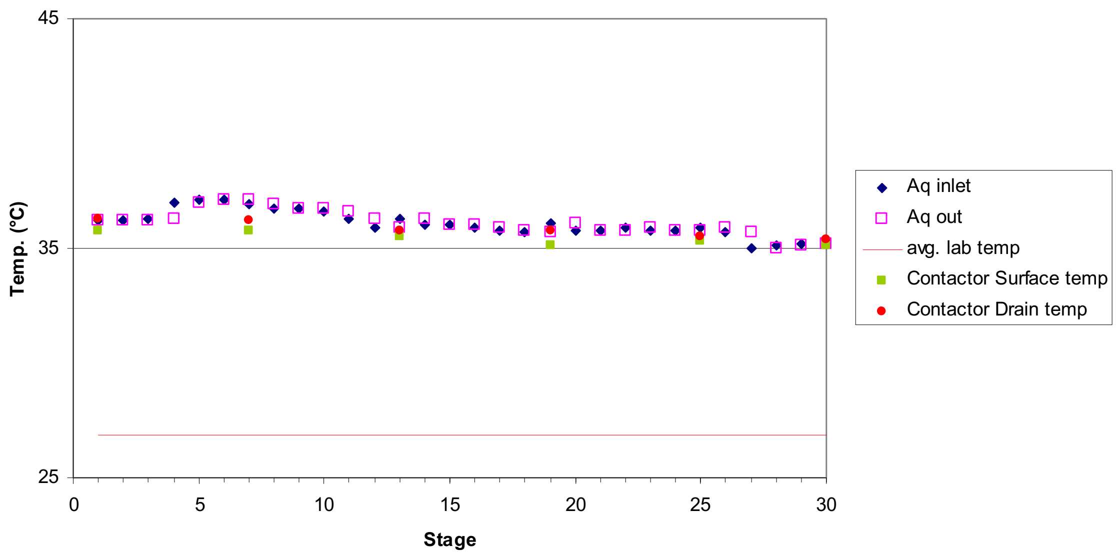

Figure 14. Temperature profile for all stages at shutdown at F_T of 1 L/min and rotor speed of 4000 rpm with inlet solution temperature of 35° C with no recycle.

The graphs at shutdown of the heated single-phase testing with no recycle show that the lower flowrate testing will provide less heat removal from the system even at elevated heated solution temperatures.

However, it should be noted that both of these temperature profiles are more flat across the contactor cascade and contains less temperature variance than those shown for ambient temperature testing, previously described.

The final single-phase testing regimen was to run two tests for a longer period of time while heating the inlet solutions to ~ 40° C and ~ 50° C at 0.5 L/min and at a rotor speed of 3750 rpm. Stage surface temperature profiles were graphed for each test with the averaged lab temperature and are shown in Figure 15 and Figure 16. These two graphs indicate the stage temperatures continually increased relative to the lab temperature increase.

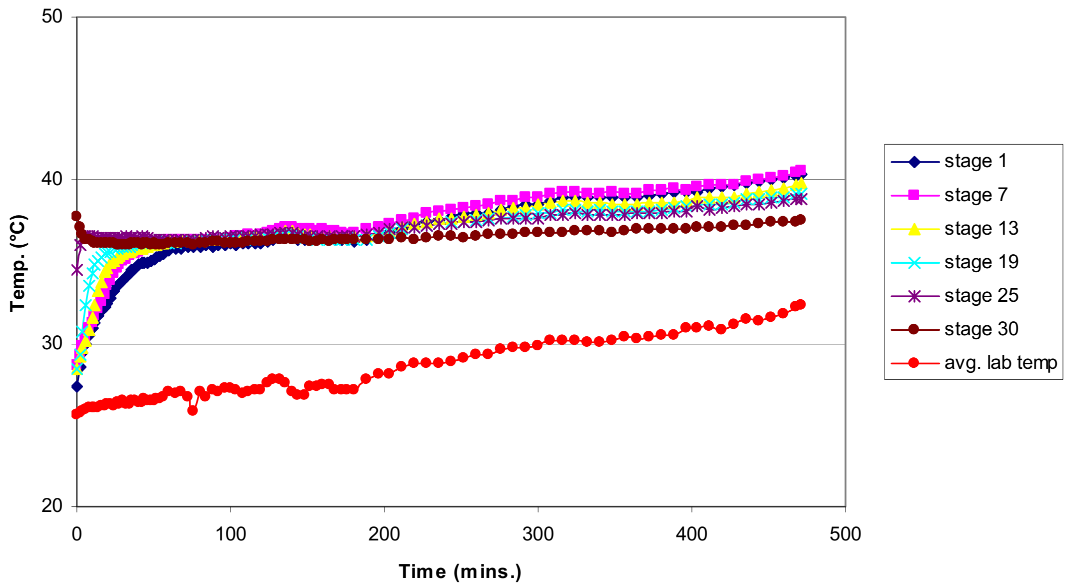

Figure 15. Stage surface temperature profile at 8 hours with inlet solution heated to 38° C at F_T of 0.5 L/min and a rotor speed of 3750 rpm, with continuous recycle.

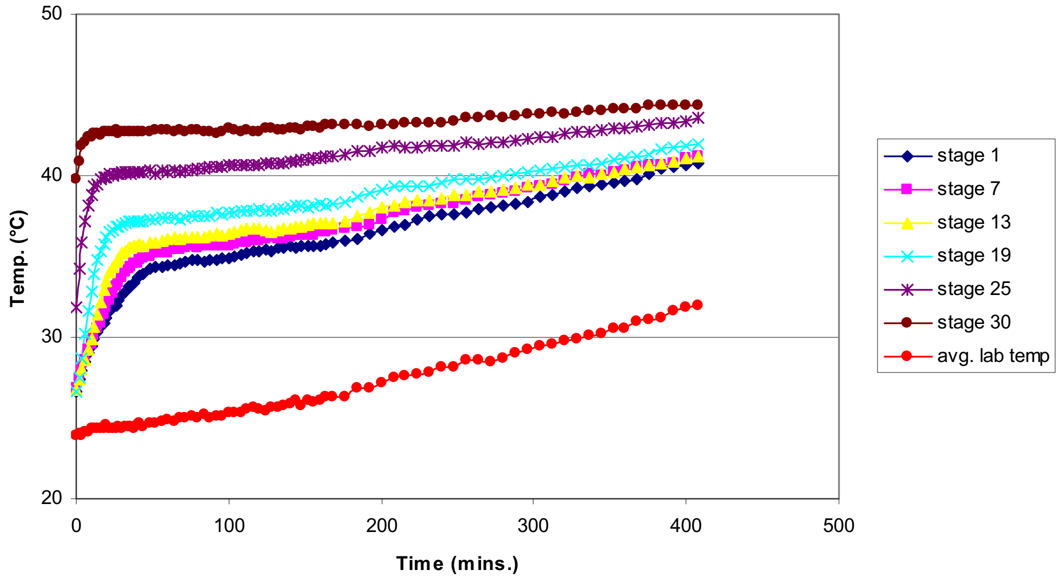

Figure 16. Stage surface temperature profile at 7 hours with inlet solution heated to 45° C at F_T 0.5 L/min and a rotor speed of 3750 rpm, with continuous recycle.

Figure 17 and Figure 18 include temperature profiles for the final two heated single-phase tests at shutdown. The graphs include all stage temperatures with the averaged lab temperature.

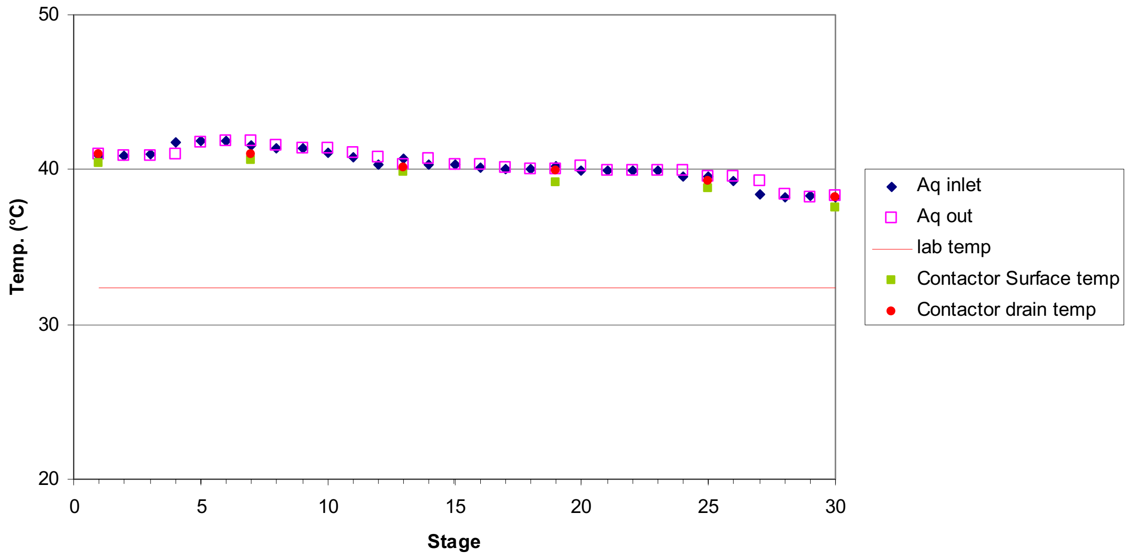

Figure 17. Temperature profile for all stages and averaged lab temperature at shutdown at F_T of 0.5 L/min at a rotor speed of 3750 rpm with inlet solution heated to 38° C with continuous recirculation.

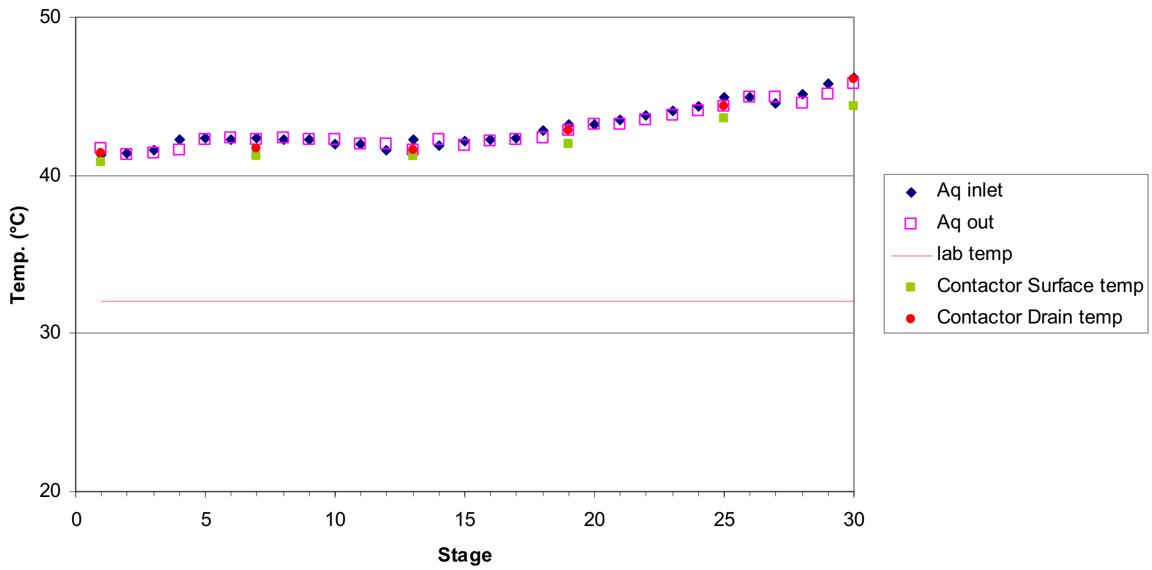

Figure 18. Temperature profile for all stages and averaged lab temperature at shutdown at F_T of 0.5 L/min at a rotor speed of 3750 rpm with inlet solution heated to 45° C with continuous recirculation.

The stage temperatures shown in Figure 17 at shutdown, indicate the stages lower than 28 actually reached temperatures slightly higher than the heated inlet solution temperature. In Figure 18, no stage reached the temperature of the heated inlet solution even after 7 hours, indicating the maximum stage solution temperatures expected for a 30-stage cascade operated at these conditions should not exceed 45° C.

2.3 Two-Phase Testing

A total of four two-phase tests were completed using lamp oil and pH 2 tap water. The F_T = 1.3-1.4 L/min at an O/A ~ 1 for all tests. Solution inlet temperatures were the changing variable for this testing. Ambient temperature tests were followed by tests with heated input solutions at 40° and 50° C. The cascade was then partitioned into two equal sections where stages 1-15 were operated as the extraction section and stages 16-30 were operated as the strip section. Temperature profile graphs for all tests were then generated from the acquired data.

2.3.1 Ambient Two-Phase Testing

Lamp oil was pumped to stage 1 and exited stage 30 while the pH 2 water was pumped to stage 30 and exited the cascade at stage 1. Both phase effluent flowrates were measured at 0.7 L/min resulting in an O/A ratio of 1. Contactor rotor speeds were set at 4000 rpm with no solution recycle performed. Figure 19 includes a graph of instrumented stage surface temperatures and the average lab temp over time for the ambient two-phase test.

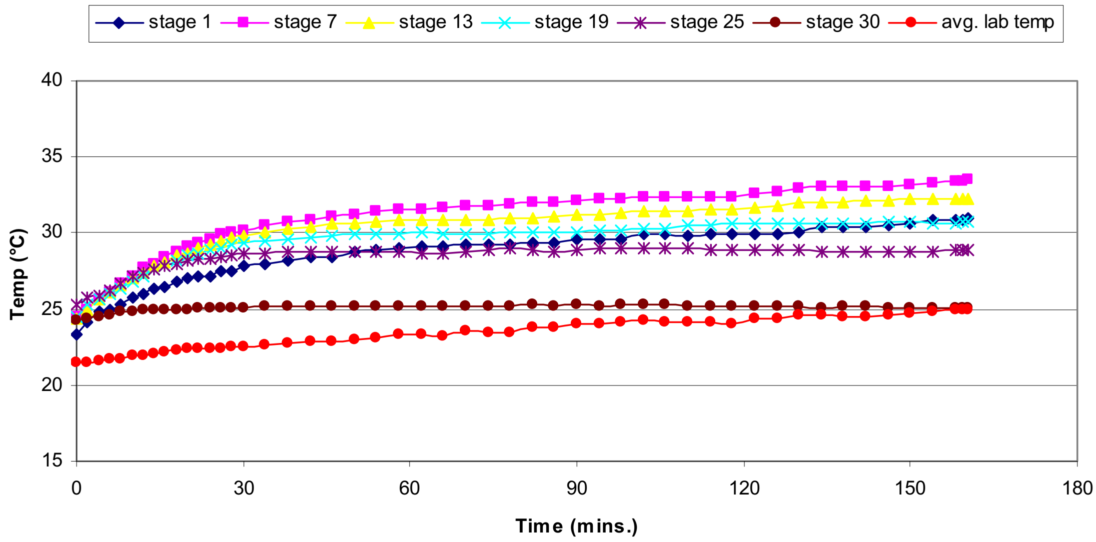

Figure 19. Contactor surface temperatures of selected stages and average lab temperature at F_T of 1.4 L/min and rotor speed of 4000 rpm at an O/A ratio of 1.

A comparison of the two-phase stage surface temperature profiles in Figure 19 with single-phase temperature profiles (Figure 7 and Figure 8) at ambient temperature shows little difference from single phase flow temperature trends.

All stage temperatures at shutdown for the aqueous and organic solutions are plotted in Figure 20 and Figure 21, respectively.

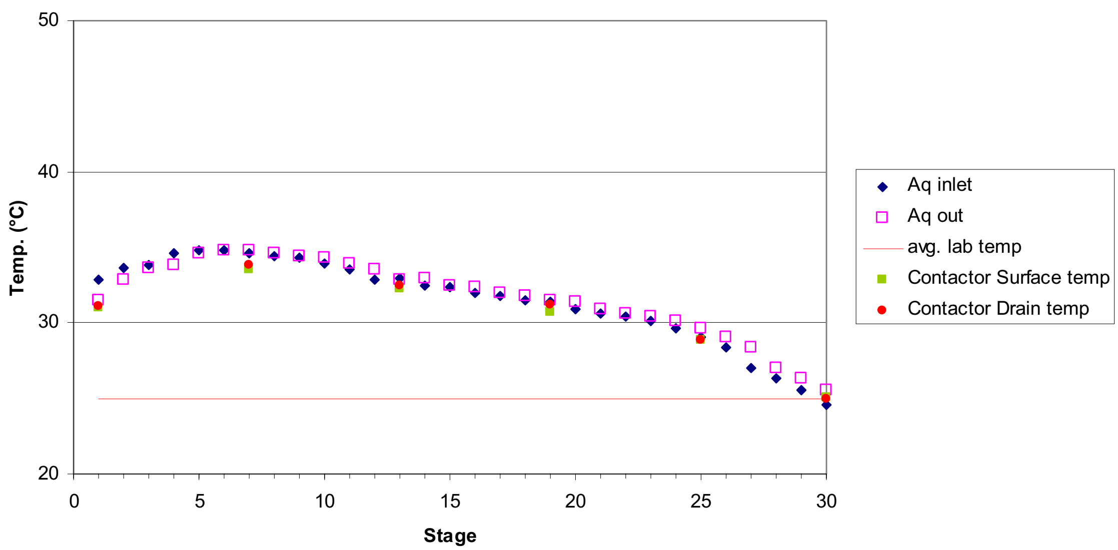

Figure 20. Aqueous phase solution temperatures with surface and drain temperatures for each stage at shutdown for ambient two-phase testing.

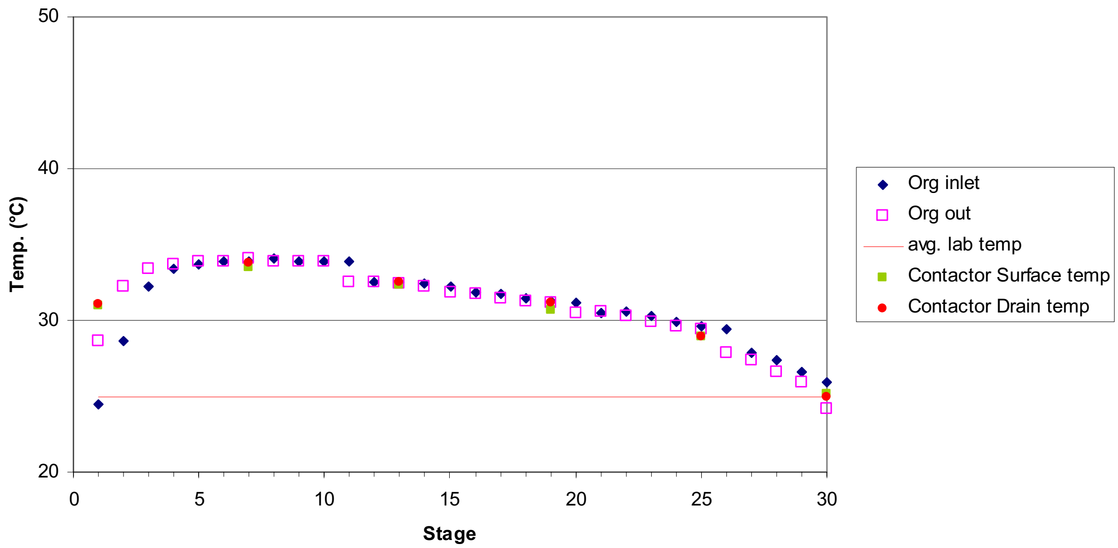

Figure 21. Organic phase solution temperatures with surface and drain temperatures for each stage at shutdown for ambient two-phase testing.

Solution temperature trends for both aqueous and organic stage solution temperatures at shutdown for the two-phase ambient temperature tests are in good agreement. The effects of mixing with the addition of lamp oil did not add additional heat to the system and in fact, actually contributed to an overall cooler temperature trend across the 30-stage cascade especially from stages 1-5 when compared to single-stage ambient temperature profiles.

2.3.2 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 inlet solution temperatures were set to 40° C and the second test inlet solution temperatures were set to 50° C. Both phases were continuously recycled for the duration of each test to maximize runtime. Total runtime for the 40° C test was 7.5 hours while the 50° C test ran for 8 hours. The F_T for each test was 1.3 L/min at contactor rotor speeds of 4000 rpm 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 Figure 22 and Figure 23.

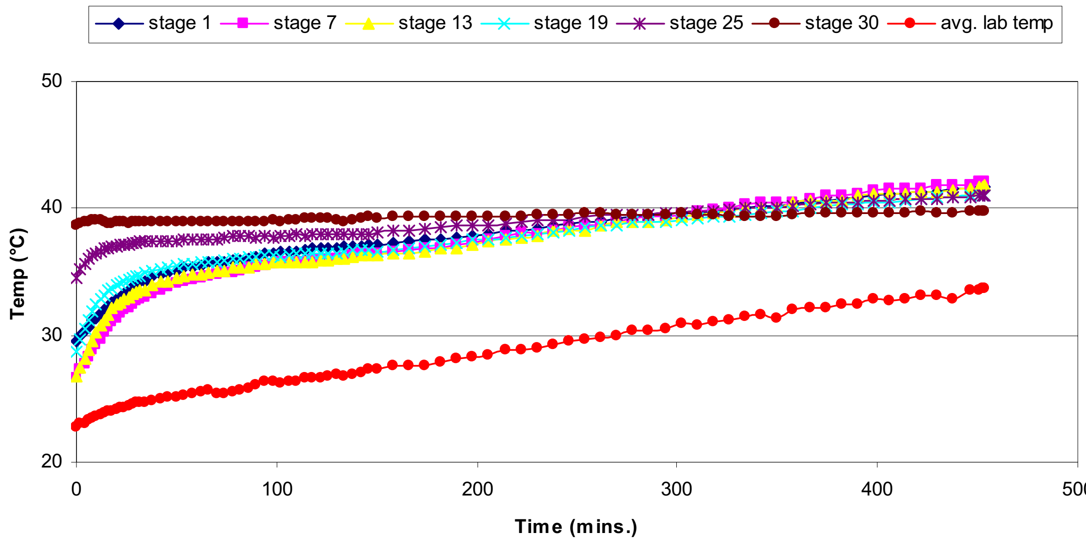

Figure 22. Contactor surface temperatures and average lab temperature plotted with time. Both phases heated to 40° C with F_T at 1.3 L/min at rotor speeds of 4000 rpm at an O/A of 0.8.

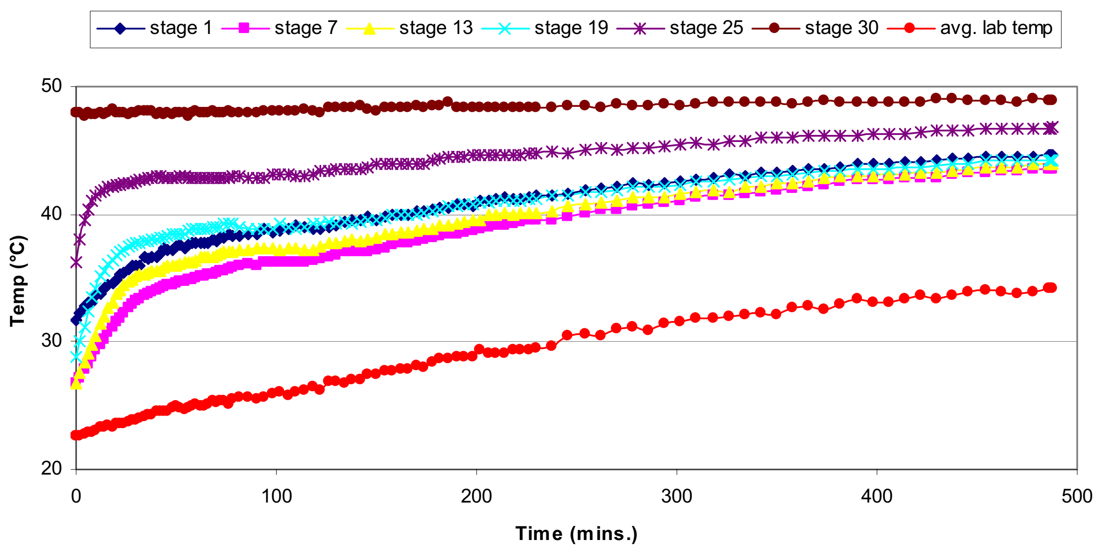

Figure 23. Contactor surface temperatures and average lab temperature plotted with time. Both phases heated to 50° C with F_T at 1.3 L/min at rotor speeds of 4000 rpm at an O/A of 0.8.

Stage surface temperature profiles for the 40° C two-phase heated solution test indicate an interesting trend after about 300 minutes of runtime. All stages below 30 actually show a temperature trend increase greater than the stage 30 surface temperature trend. These rates of increase appear to run in parallel with the average lab temperature trend increase. This indicates that the temperature increases of the stages appear to be directly related to the increasing lab temperature.

The stage surface temperature profile for the 50° C shown in Figure 23 describes a larger temperature spread for stages 1, 7, 13, and 25 than that of the previous test. However, the gradual increase over the test duration still somewhat mirrors the average lab temperature trend.

A final set of graphs is provided in Figure 24 through Figure 28 depicting individual stage inlet/outlet aqueous and organic solution temperatures and surface and drain temperatures along with the average lab temperature as a function of time at shutdown conditions for the heated two-phase tests.

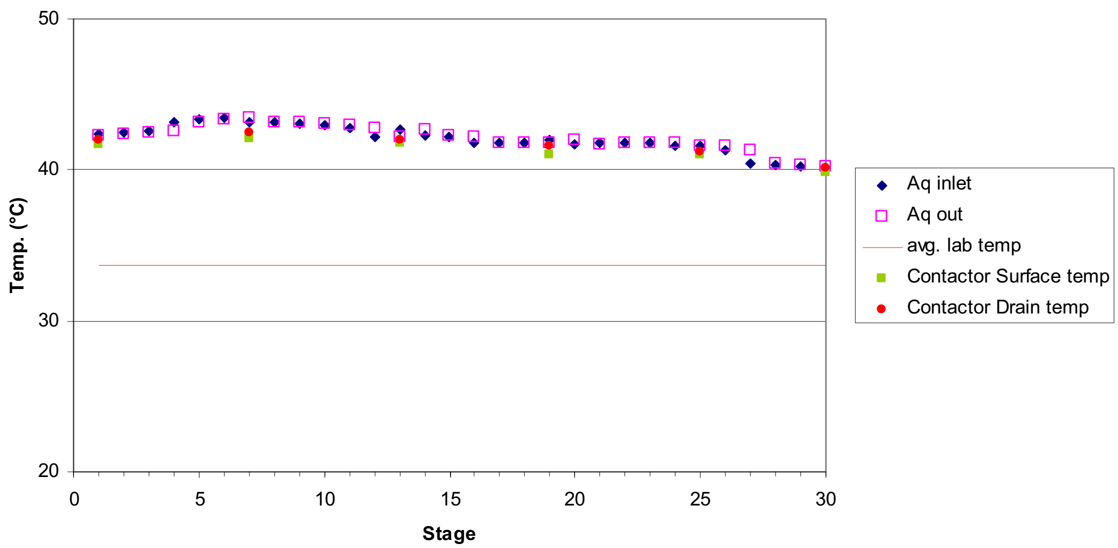

Figure 24. Aqueous solution inlet/outlet stage temperatures and stage surface and drain temperatures at shutdown. Both phases heated to 40° C with a F_T of 1.3 L/min at rotor speeds of 4000 rpm at an O/A ratio of 0.8 for 7.5 hours.

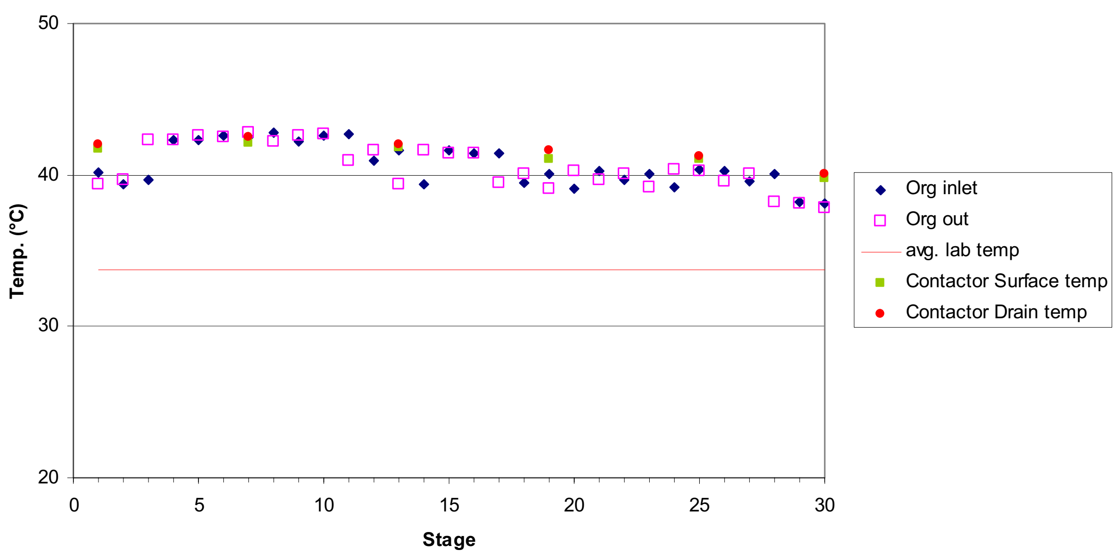

Figure 25. Organic solution inlet/outlet stage temperatures and stage surface and drain temperatures at shutdown. Both phases heated to 40° C with a F_T of 1.3 L/min at rotor speeds of 4000 rpm at an O/A ratio of 0.8 for 7.5 hours.

The 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 (as seen in Figure 24 at stages 6 and 7) when the ambient lab temperature reaches 34° C. The organic solution stage temperature profiles shown in Figure 25 are less uniform in appearance than the aqueous profiles. Organic effluent physical flow characteristics exiting stage 30 did show signs of pulsed flow which may have caused some of the erratic temperature readings. This organic temperature non-uniform appearance was routinely observed when processing heated solutions at elevated temperatures but the temperature deviations are still quite small.

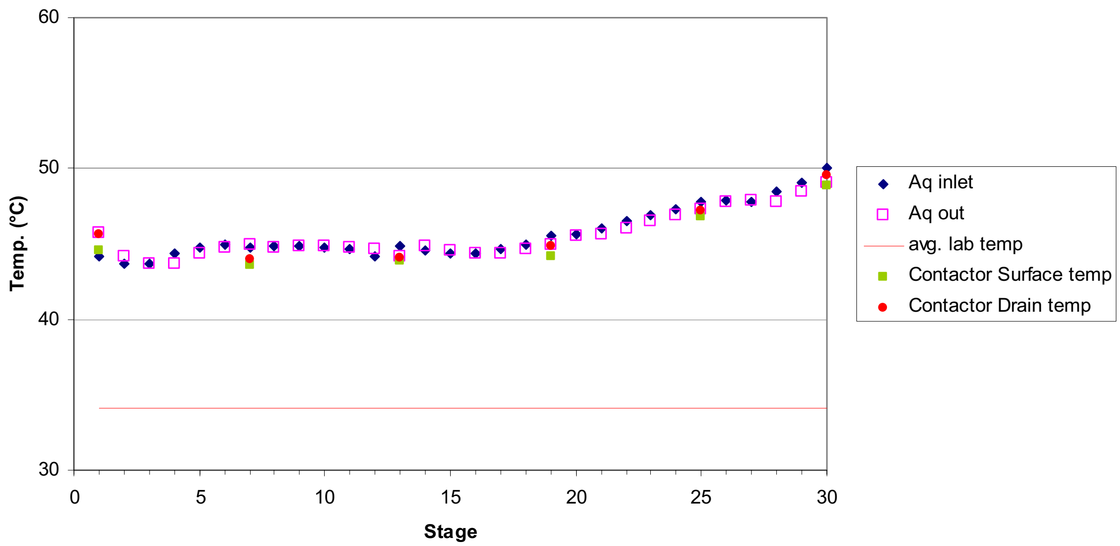

Figure 26. Aqueous solution inlet/outlet stage temperatures and stage surface and drain temperatures at shutdown. Both phases heated to 50° C with a F_T of 1.3 L/min at rotor speeds of 4000 rpm at an O/A ratio of 0.8 for 8 hours.

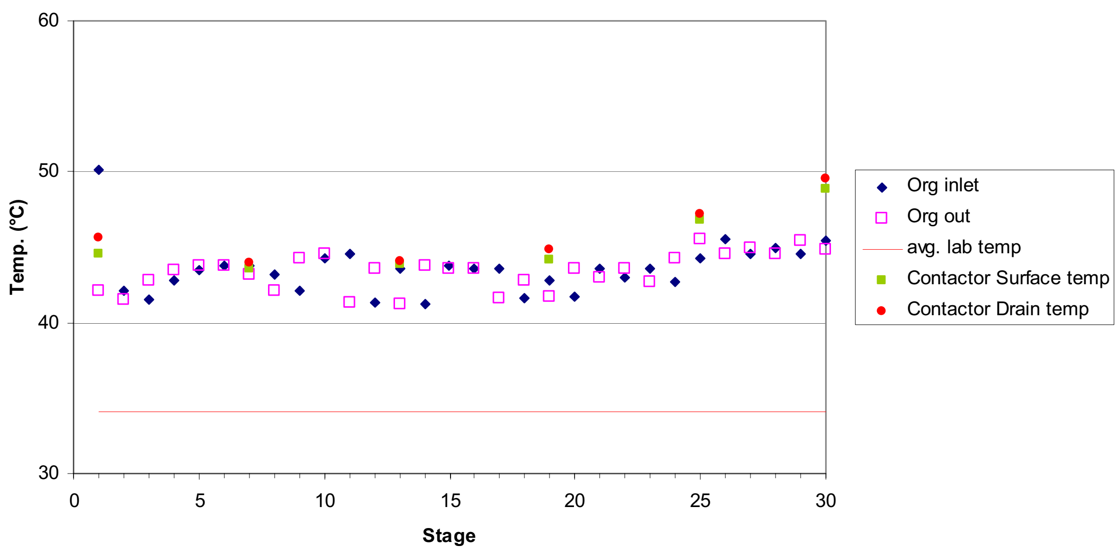

Figure 27. Organic solution inlet/outlet stage temperatures and stage surface and drain temperatures at shutdown. Both phases heated to 50° C with a F_T of 1.3 L/min at rotor speeds of 4000 rpm at an O/A ratio of 0.8 for 8 hours.

Aqueous stage solution temperatures measured for the 50° C heated tests show that overall stage temperatures do not reach the heated inlet solution temperatures after 8 hours of operation when average lab temperatures reach ~34° C. 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.

2.3.3 Extraction/Heated Strip Testing

This testing incorporated 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 solution heated to 50° C for the strip section entered stage 30 and exited from stage 16. The unheated organic solution entered stage 1 and exited at stage 30. All solutions were processed as once-through with no recycle. The F_T 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 28 and Figure 29 show selected stage surface temperatures with average lab temperature as a function of time for the extraction and heated strip sections, respectively.

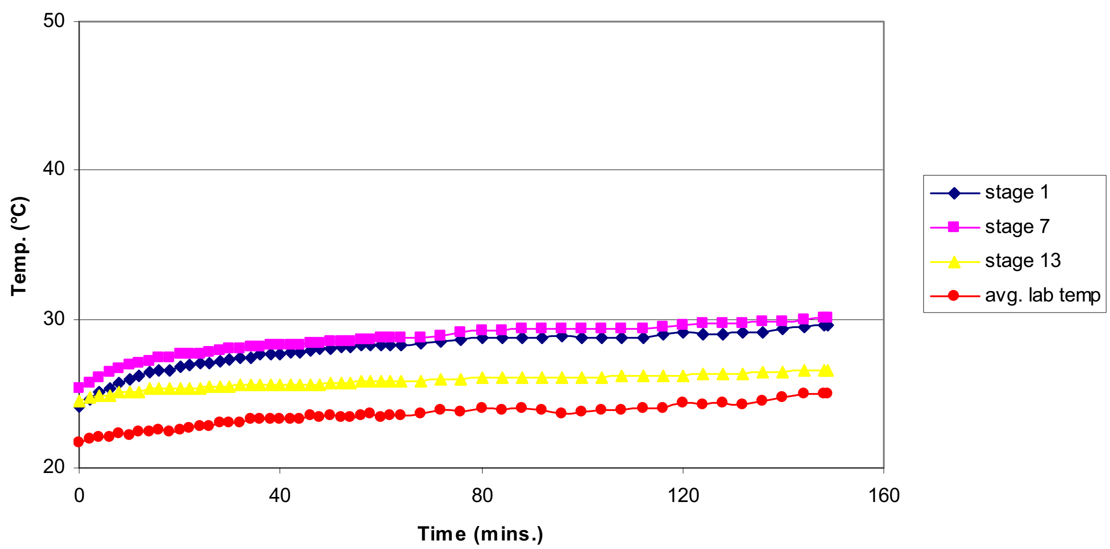

Figure 28. Selected stage surface temperatures for the extraction section with average lab temperature over time.

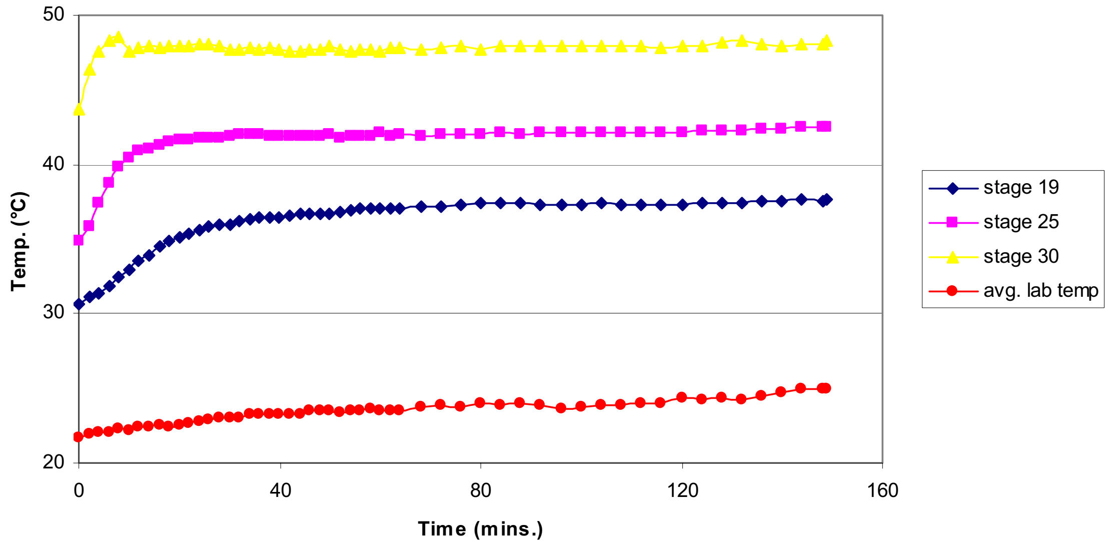

Figure 29. Selected stage surface temperatures for the strip section with average lab temperatures over time.

Stage surface temperature profiles representing the extraction section show slight temperature increases of the three selected stages over time. Stage surface temperature profiles representing the heated strip section indicate a cooling trend starting at the stage 30 heated aqueous inlet with gradual cooling at stage 19. Stage surface temperatures for the heated strip section also appear to reach steady-state after about one hour of operation.

Graphs for both aqueous and organic phase solution temperatures for each stage at shutdown for the heated strip testing are shown in Figure 30 and Figure 31. Each graph includes the extraction and strip section stage temperatures with surface and drain temperatures with the average lab temperature.

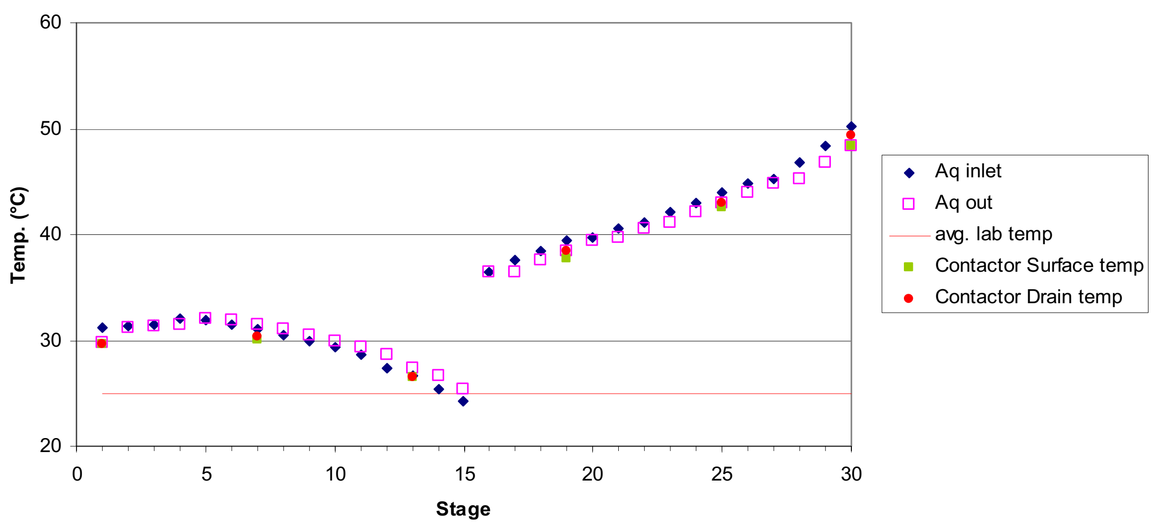

Figure 30. Aqueous phase solution temperatures for each stage for both the extraction and strip section with average lab temperature and surface and drain temperatures at shutdown.

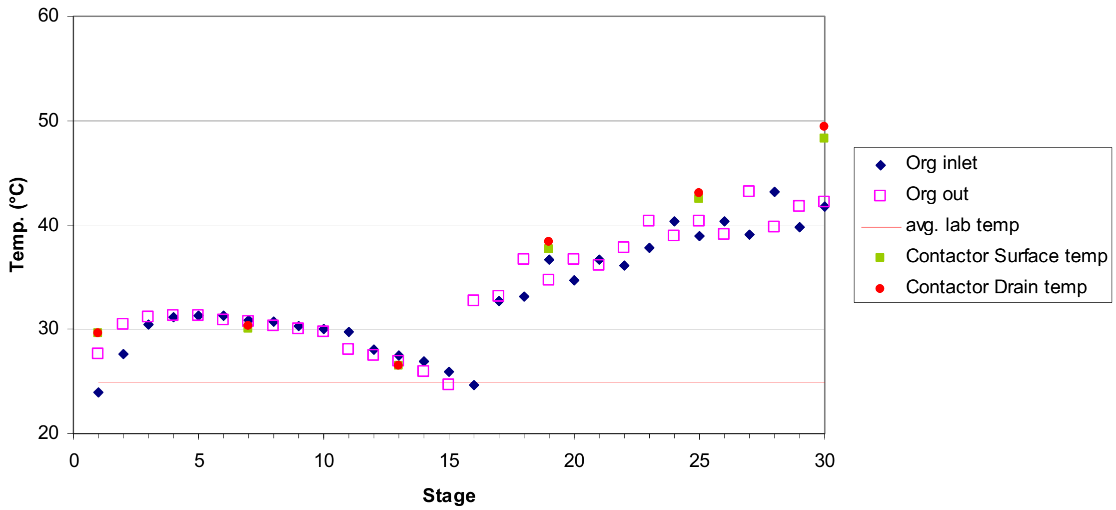

Figure 31. Organic phase solution temperatures for each stage for both the extraction and strip section with average lab temperature and surface and drain temperatures at shutdown.

The aqueous solution stage temperatures at shutdown provide an excellent representation of a temperature profile across a 15 stage heated strip section and a 15 stage extraction section using ambient inlet solution temperatures. The temperature trends indicate the 50° C heated aqueous strip solution does not significantly impact stage temperatures in the extraction section. However, the organic flow entering the strip section at stage 16, at ambient temperature, contributes to the decrease in temperature gradient across the strip section (50° C to 38° C) as seen in Figure 30.

3. SUMMARY AND CONCLUSIONS

Numerous temperature profiles were completed using a 30-stage centrifugal contactor pilot plant. The automated process control and data acquisition system worked very well throughout testing. Temperature data profiles for an array of total flowrates and contactor rpm values were collected. The F_T ranged from 0.5-1.4 L/min with rotor speeds tested from 3500-4000 rpm. Solution inlet temperatures from ambient up to 50° C were used.

During the single-phase testing, a leveling bar was installed to provide individual stage leveling ability to enhance flow characteristics, in particular the organic phase, across the cascade. The leveling bar did not entirely alleviate the organic effluent pulsing flow. Higher rotor speeds were required to optimize flow from stage to stage but there was no loss of contactor separation efficiency or measurable other phase carryover observed. The inter-connective tubing fittings could be optimized for enhance flow.

Single-phase testing demonstrated that water could be successfully processed through 30 stages at F_T = 0.5-1.0 L/min with rotor speeds from 3500-4000 rpm. Temperature profiles were completed for three inlet temperatures with changing conditions.

Tests with solutions processed as once-through were operated for a time sufficient to achieve steady-state conditions. Results from this initial testing did indicate that higher flowrates provide more rapid heat removal from the system even with increased contactor rotor speeds.

Extended runtime single-phase run data showed that the increase in stage temperatures tracks with ambient lab temperature increases after steady-state temperature conditions are reached.

Two-phase testing was performed at F_T = 1.3-1.4 L/min at a rotor speed of 4000 rpm and an O/A ratio of 1. Inlet solution temperatures of ambient, 40° C and 50° C were used. Organic solution entered the cascade at stage 1 and exited at stage 30, while the aqueous solution entered stage 30 and exited at stage 1. Organic and aqueous phase samples taken from streams exiting the cascade contained no measureable or observable carryover.

The ambient two-phase temperature profiles indicate that the inclusion of ambient temperature lamp oil pumped into stage 1 has little impact on temperature profiles when compared to the single-phase testing data. Therefore, heat addition attributed to two-phase mixing was negligible in these tests.

Heated two-phase testing at elevated inlet solution temperature tests were operated for ~ 8 hours each. Both tests used F_T = 1.3 L/min and rotor speeds of 4000 rpm at an O/A ratio of 0.8. Aqueous and organic solutions were recycled continuously during both tests. Individual stage temperature profiles indicated that after initial system steady-state conditions are realized, the rate of stage temperature increases relates quite well to the ambient lab temperature rate increase. Temperature profile results also indicate that maximum stage temperatures are expected to not reach values > 50° C for the conditions tested.

A final heated strip test was performed to study temperature trends in a process operated with two different inlet solution temperatures. The 30-stage cascade was divided equally into two 15-stage sections with the first section designated as the extraction and the second as the strip section. The unheated aqueous solution for the extraction section entered stage 15 and exited from stage 1. Aqueous solution heated to 50° C for the strip section entered stage 30 and exited from stage 16. The unheated organic solution entered stage 1 and exited at stage 30. All solutions were processed as once-through without recycle.

The temperature profile results for the heated strip testing indicate that after an operating time of > 2.5 hours, steady-state temperatures are reached for all stages in about 30 minutes. No impacts to stage temperature profiles in the extraction section were observed due to heating of the strip solution.

Using the CINC V-02 centrifugal contactors, small temperature increases to the process solutions were observed due to the mechanical contactor motors, ambient lab temperatures and solution mixing. At F_T = 1 L/min, the temperature of the cascade is primarily driven by changes in the aqueous feed temperature. Therefore, it is feasible that solvent extraction process sections requiring chilled or heated process temperature for increased efficiency can be operated using in-line feed solution heat exchangers for adequate temperature control. The addition of thermal jacketing to individual contactors would only be required for processes needing precise stage temperature control. Therefore, the added cost and complexity of thermal jackets and associated connections may not be necessary. Contactor surface thermocouples were in good agreement with process solution temperature measurements and could therefore provide reliable data for process control.

It should be noted that all testing was performed at higher than desired rotor speeds, due to contactor outlet flow path issues described within this report, affecting hydraulic performance. It is expected that these issues could be alleviated with design modifications which would allow the contactors to be operated at lower rotor speeds at the same total flowrates tested. At lower rotor speeds, the temperature increases across the cascade are expected to be lower. Testing in FY-09 is planned to verify this.

In conclusion, testing with larger CINC 12.5 cm (V-05) units is recommended as the temperature profiles could be different.

4. REFERENCES

1. D. H. Meikrantz, J. D. Law, R. S. Herbst, T. G. Garn, N. R. Mann, and T. A. Todd, "Hydraulic and Mass Transfer Testing of Commercial 5 cm Annular Centrifugal Contactors at INL," INL/EXT-05-00793, 2005.
2. T. G. Garn, N. R. Mann, D. H. Meikrantz, J. D. Law and T. A. Todd, "Hydraulic and Reliability Tests of the CINC V-05TA-CIP 12.5 cm Annular Centrifugal Contactor," INL/EXT-06-11958, 2006.
3. N. R. Mann, T. G. Garn, D. H. Meikrantz, J. D. Law, and T. A. Todd, "Clean in Place Testing of a Commercial 12.5 cm Annular Centrifugal Contactor at the INL," INL/EXT-06-11533, 2006.
4. T. G. Garn, D. H. Meikrantz, J. D. Law, N. R. Mann, and T. A Todd, "Mass Transfer Efficiency Testing of a Single Stage Commercial 12.5 cm Annular Centrifugal Contactor at the INL," INL/EXT-07-12739, 2007.