OPERATION AND PROCESS CONTROL DEVELOPMENT FOR A PILOT-SCALE LEACHING AND SOLVENT EXTRACTION CIRCUIT RECOVERING RARE EARTH ELEMENTS FROM COAL-BASED SOURCES

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ABSTRACT

The US Department of Energy in 2010 has identified several rare earth elements as critical materials to enable clean technologies. As part of ongoing research in REEs (rare earth elements) recovery from coal sources, the University of Kentucky has designed, developed and is demonstrating a ¼ ton/hour pilot-scale processing plant to produce high-grade REEs from coal sources. Due to the need to control critical variables (e.g. pH, tank level, etc.), process control is required. To ensure adequate process control, a study was conducted on leaching and solvent extraction control to evaluate the potential of achieving low-cost REE recovery in addition to developing a process control PLC system. The overall operational design and utilization of Six Sigma methodologies is discussed. Further, the application of the controls design, both procedural and electronic for the control of process variables such as pH is discussed. Variations in output parameters were quantified as a function of time. Data trends show that the mean process variable was maintained within prescribed limits. Future work for the utilization of data analysis and integration for data-based decision-making will be discussed.

INTRODUCTION

As part of their stewardship, Department of Energy (DOE) has evaluated various rare earth elements critical to the ongoing development and utilization of clean energy technologies. These elements are Yttrium (Y), Europium (Eu), Terbium (Tb), Neodymium (Nd), and Dysprosium (Dy) [1]. Due to changes in green energy technologies and shifting markets on a global scale, the future of the world's energy market is uncertain. To protect domestic critical materials supply in the United States, the U.S Department of Energy has funded numerous research initiatives in the area of alternate REE supply, recovery, and extraction, in the United States.
Although researchers have reported the occurrence of REEs in coal and coal byproducts, literature of recovery processes is limited for coal-based REEs. Zhang et al., 2015 [2] reported that the composition and complex distribution of REEs in coal and coal byproducts, as well as, the non-existent or lab scale REE recovery process for coal products is the reason for such limited literature. To compensate, researchers have been exploring the existing REEs enrichment processes and recovery techniques used on other REEs sources. Typical REE extraction and recovery methods include; roasting and leaching; ion exchange, precipitation, adsorption and solvent extraction (SX) [3-5]. The successful recovery of metals through acid leaching and solvent extraction has been noted in literature. Research has also presented evidence on the unique extraction and recovery of REEs and other metals (e.g. copper, zinc) through acid leaching and solvent extraction [2, 3, 6].

Regardless of the exact REEs recovery process, the flowsheet will involve a series of complex steps; hence, the development of effective process control is vital to the operation and recovery of critical rare earth elements. The implementation of process control in chemical and mineral-metallurgical recovery processes is critical to the safety, operation, performance, productivity, quality, and overall product recovery [7].

Process control is a way of achieving both simple and complex chemical industrial production goals using control systems in an economic and safe approach. Consequently, the benefits of process control implementation in the metallurgical process industry have been well researched.

Ray et al., [8] presented a study where instrumentation and automation system control was utilized for a coal leaching pilot plant. The study explored the use of automation (PLC) in increasing yields of chemical coal leaching processes.

Wu et al., [9] studied model-based expert control system which tracked the optimal pH of a continuous leaching process. Their research explored the use of a computer programmed expert control system that combines both mathematical models and rule-based models.

In 2014, Dash et al [10] described how a PLC-based control system was used to ensure the smooth operation of a coal leaching pilot plant producing low ash coal product. The study applied supervisory control and data acquisition (SCADA)-based system for a leaching pilot plant treating Indian coal. The strong control system implemented at the pilot plant efficiently regulated plant parameters, improved data acquisition, and optimized plant operation. To achieve high-level process control of the leaching process, the plant automation involved a Rockwell-based PLC and other industrial instruments. Overall research findings showed that the successful implementation of PLC-based automation and control system helped fulfill the objective of regulating process variables (e.g., chemical composition, temperature, and pressure, etc.) and optimization.

There are other applications of process control PLC-systems, some are implemented in energy research, manufacturing, education research, training, and wastewater management control where measurement and monitoring of pH and other process variables of a closed-loop system in real time are critical [11-12].

This paper will discuss the design and development of a PLC control system with the capability of controlling critical variables and effectively producing high-grade REEs. The pilot-scale leaching and solvent extraction rare earth element recovery plant is designed to operate using both Allen Bradley PLC-based automated control system and manual controls. The overall objective of this project is to design and develop a safe, efficient, and low-cost process control system with the capability of effectively operating a pilot plant producing high-grade rare earth elements from coal-based sources based on six sigma methodologies.

PLANT PROCESS DESCRIPTION

The schematic flowsheet diagram of the rare earth element recovery process utilized at the University of Kentucky (UK) pilot plant is shown in Figure 1. The operation and control primarily focused on the leaching circuit feeding solvent extraction. The plant feed preparation consisted of feed size reduction via crushing and milling. As part of the leach circuit, an intermediate acid make up tank is used to dilute and achieve the correct acid concentration for leaching feed. Acid leaching consists of with cascading overflows with the pH controlled in the first feed tank. Tanks arranged in series are used to achieve requisite residence time. Leaching occurs at 75℃ with pH controlled automatically by acid addition via PLC. The leached slurry is transferred into a thickener. Here, the underflow solid is sent to the filter press for solids recovery and the clarified overflow liquid together with the liquid recovered from the filter press is transferred to SX (solvent extraction) pre-treatment tanks. This process consists of a reduction tank and neutralization tank prior to SX. In the reduction and neutralization tanks, ORP and pH of the solution respectively are manipulated for optimum SX feed by the same PLC system.

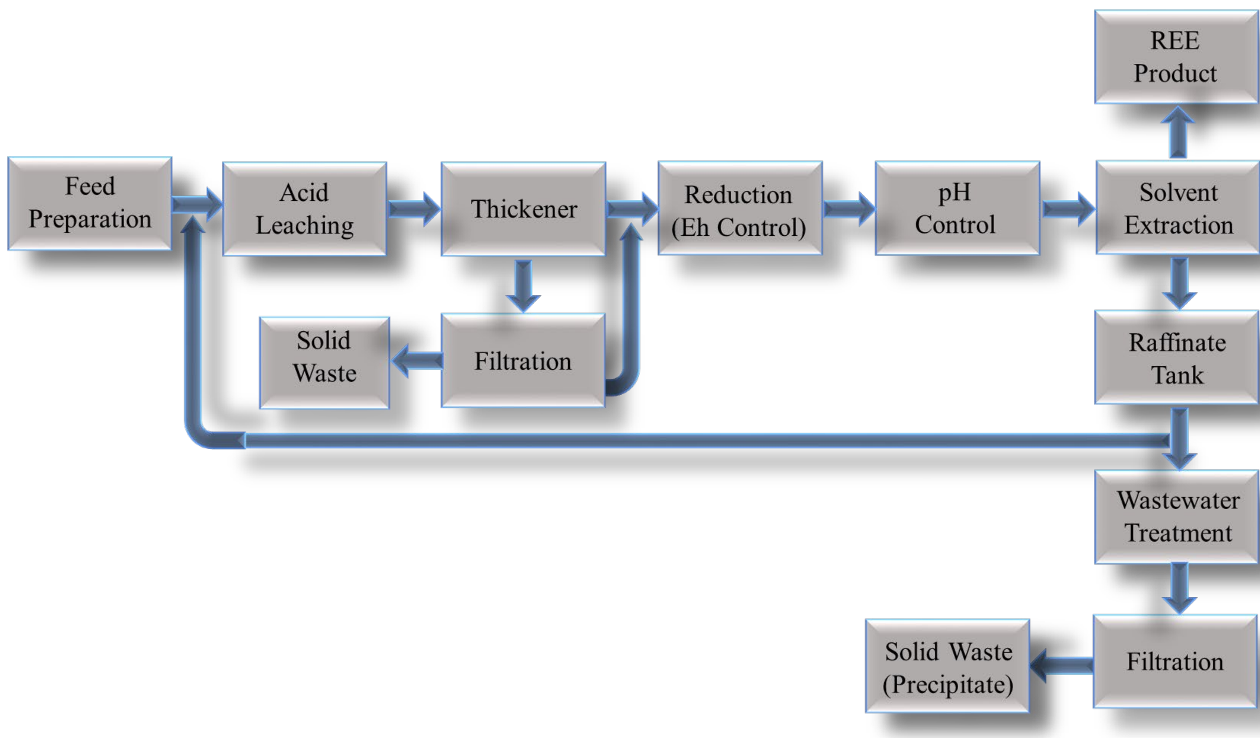

Figure 1. Process flow diagram of the pilot plant.

As part of wastewater treatment, the pH is adjusted and precipitate is removed by filter press. Also, the recovered leach solids are washed and neutralized before disposal. To assist in controls design, a piping and instrumentation diagram (P&ID) detailing the leaching circuit has been created detailing needed inputs and outputs (I/O) as shown in Figure 2 which shows the originally designed leaching circuit. Discussed below are leaching circuit control loops and safety interlocks.

Control Loops

- Feed flow control consists of thickener underflow, water makeup, raffinate recirculation, and acid flow as shown in Figure 2. Control is achieved by pump speed (rpm) adjustment using proportional-integral (PI) control. Tank level is controlled by gravity overflow.
- pH and/or ORP control in tanks 6, 3, 4, and 19 as shown in Figure 2 is achieved by reagent addition via proportional-integral-derivative (PID) control.
- The temperature control loop in Tanks 6 to 10 is implemented by a heater controller employing a standalone temperature control loop.

Safety Considerations

- The plant will have a Master ON/OFF switch. In the event of power failure; the plant must be restarted by the operator rather than automatically resuming operation.
- Agitators and pumps must be stopped whenever the tank liquid level is below impeller.
- Emergency stops (E-Stops) are strategically located throughout the plant and are assigned priority in the control system.
- Safety checks or inspections must be performed before starting pumps, variable frequency drives (VFD) or proportional valves.
- Whenever a set point in pH, ORP, levels or coal feed is reached, pumps and valves should stop.

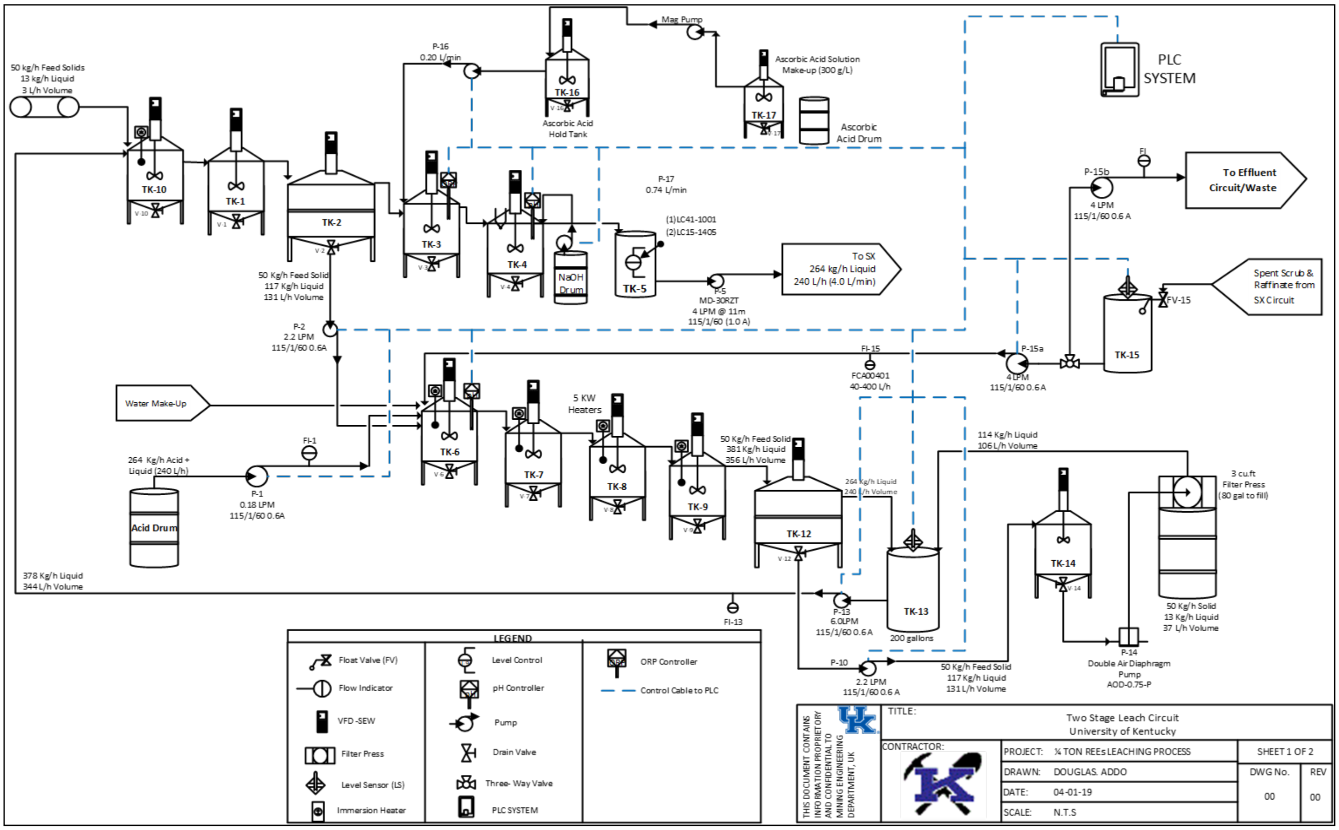

Figure 2. Pilot plant leaching circuit P&ID (unscaled).

DEVELOPMENT OF PROCESS CONTROL SYSTEM

Control Methods

This section involves the investigation and implementation of six-sigma methods to develop a PLC-based automated control system. Benchmarking activities and discussions were conducted to gather information on critical processes in the pilot plant and variables critical to operation while exploring control theories and plans to reinforce the development concept of the project.

In the development of any process control system, the control theory is vital. As part of the control theory, a select group of variables that are critical to the operation of the leaching circuit was identified as well as previously used control steps in literature.

- Percent solid concentration.
- pH value.
- Eh or ORP.
- Temperature.
- Level and residence time.

The overall controls design is based on four fundamental principles; create smooth controls, controls with simple interlocks, control the critical variables that matter, and have non-competing control loops. In addition, critical assumptions are made in the design of the control system including

- Feed – the filter cake feeding system will be composed of 20-25% water and 70-75% solids.
- Variability in feed material – feed rate will be controlled and monitored closely to maintain the required percent solids feeding the leaching tank. The system will be set to deliver 20 percent solids.
- pH Drift – the pH will drift up throughout the circuit. Initial feed rate and recirculation rate will be set constant with acid/base additions varying according to pH set point.

These assumptions provide guidance to the system design process and control implementation.

An important phase of the control system design is the development of a control plan. The control plan is a written description of how system control will be achieved. It is also used for documentation and communication. Subsequently, the control plan describes,

- What should be controlled?
- What should be measured and how often?
- What are the process specifications?
- How to ensure the process is performing well.
- What to do when it is not.

The control plan is intended to be as a living document updated as control methods and measurements tools are evaluated and improved. As part of a general rule to establish an effective control plan for the leaching control, the team utilized the basic understanding of the process and available information to develop an initial control plan. This included:

- Process flowsheets.
- Design/process failure mode and effects analysis (FMEA).
- Lessons learned from literature.
- Team expertise or experience with the process.

Benefits of a control plan include reducing waste and ensuring improvement in product quality, communicating changes and documenting improvement, and meeting downstream customer specifications.

For example, solvent extraction is the downstream customer of the leaching process. Figure 3 shows a sample leaching control plan for REE recovery. Similarly, a safety FMEA which evaluates the plant processes to identify potential areas of failure and relative impacts was developed in order to affect necessary change where needed (Figure 4). Finally, the total number of input and output (I/O) needed for the operation was determined as a result of the detailed control plan.

Control Plan

Control Plan Number:	Control Plan Owner / Phone:	Date Original:	Date Revised:
001-Leach Process Control Design	Douglas (859)684-1690	04/10/2019	NA
Process: REE 1/4 hr Pilot Plant	**Team:** Douglas Ado, Josh Werner, Bob Braton, Jacob Gill	**Revision Note**	
Description: Operation of Leach Circuit	**Approval Date:**		

Process Number	Process Name/ Description	Tank, Device, Equipment	Characteristics			CTQ	Methods					Reaction Plan
			No.	Inputs	Output		Product/Process Specification/Tolerance	Evaluation/ Measurement	Sample Size	Sample Freq.	Control Method	
1	Leaching	TK-10	10a	Filter Cake Solid Mass		N	70-75 %	VT?	?	?	?	?
			10b	Filter Cake Liquid Mass		N	25-30 %	VT?	?	?	?	?
			10c	Filter Cake Liquid pH		Y	?	VT?	?	?	?	?
			10d	Filter Cake Feed Rate		Y*	~100lb hr	VT?	?	?	?	?
			10e	Inlet Flow Rate (P-13)		Y	4 - 8 (Target 6 LPM)	RPM	N/A	Continuous	Set Point / PLC	Operator Adjust
			10f	Inlet Flow Temperature		Y	None	N/A	N/A	N/A	N/A	N/A
			10g		Leaching Temperature	Y	75 °C	Thermocouple	N/A	Continuous	Set Point / PLC	Operator Adjust
			10i		Residence Time	Y	See Sys-a	See Sys-a	See Sys-a	See Sys-a	See Sys-a	See Sys-a
			10j		pH	Y	TBD	pH Probe	N/A	N/A	See Set Point in TK-6	SOP
			10k		% Solids	Y*	1-20%	Weight of Known Volume	N/A	Hourly	P-13 Flow Rate	SOP

Figure 3. Leaching process control plan.

FMEA			
FMEA Number: 002-Hydromet Circuit Startup Safety Evaluation	**FMEA Plan Owner / Phone:** Josh Werner (509)995-6697	**Date Original:** 6/14/2018	**Date Revised:** 4/1/19
Process: Plant	**Team:** Rick Honaker, Josh Werner	**Revision Note:** Revised by Blanton Park on 7/31/18 and Douglas Addo to assign responsibility and target date to various FMEA actions. *Weekly JHA, where numerous operators will observe & analyze the current operating procedures with the goal of continuously working towards the safest work environments and mitigating hazards.	
Description: Operation of Pilot Plant	**Approval Date:** Rick Honaker 6/15/2017		

Process Number	Process Name/ Description	Tank, Device, Equipment	Characteristics			Potential Failure Mode	Potential Effect(s) of Failure	Severity	Potential Cause of Failures	Occurrence	Current Process Controls Prevention	Current Process Controls Detection	Detection	R.P.N.	Recommended actions	Responsibility & Target Date	Actions Taken & Completion Date	Severity	Occurrence	Detection	R.P.N.
			No.	Inputs	Output																
1	Leaching	Leach Tank	1a			Leaking Acid	Acid Burn	7	Loose Fittings	7	Water Test	Visual	5	245	Water Shake Down	Joshua Werner 6/19/2018	Water Test 6/19/18	7	1	5	35
			1b			Leaking Acid	Acid Burn	7	Leaking Pipes	3	No Acids Pumped Overhead	Design	5	105	Check Off Walk Through	Jacob Gill 8/1/18					
			1c			Leaking Acid	Acid Burn	7	Acid Exposure	3	Shower/Eye wash	Design	3	63	Test Shower Montly, Test Log	Kin Craig 8/3/18					
			1d			Leaking Acid	Acid Burn	7	Peristaltic Pump Wear	7	Pre and Post Shift Start Inspection Logs	SOPs	7	343	Maintenance and Replacement Schedule	Jacob Gill 8/6/18					
			1e			Contact Acid	Acid Burn	7	Inspecting Tank	7	Design	Operator	5	245	SOP and Procedure for Checking Tank	Alind Chandra 8/20/18					
			1f			Fire	Badness	10	Electric Heater	3	Low Solution Sensor	Shut Off	10	300	Test Shutoff each tank and record	Jacob Gill 6/15/18	Sensors Installed 6/19/18				0
			1g			Fire	Badness	10	Electric Induced	1	Fire Alarms	Auditory	5	50	Test Bi annually	Rick Honaker 1/1/19					
			1h			Fire	Badness	10	Electric Induced	1	Fire Extinguishers	Location	3	30	Inspect Bi annually	Rick Honaker 1/1/19					
			1i			Fire	Badness	10	Electric Induced	1	Fire Department Training	Fire Department Training	5	50	Host a Fire Department Field Trip	Joshua Werner 8/8/18					

Figure 4. Safety FMEA

PLC Control System Build

Several brands of PLCs were considered, but an Allen Bradley (AB) PLC was deemed the most appropriate choice for the pilot operation based on support infrastructure available to the team.

Figure 5 shows the complete assembly of the plant automation PLC systems with all I/O components. The plant process control system build was based on Allen Bradley 1769-L33ER CompactLogix PLC by Rockwell Automation. Further components consisted of:

- 24VDC power supply mounted at the bottom part of a waterproof enclosure.
- Ethernet switch miniature circuit breakers for the controller.
- Ethernet adapter.
- 15 inch display.
- DeviceNet scanner module.
- 16 point DC input module.
- 8 point relay output.
- Analog I/O modules.

DeviceNet module will be utilized to communicate with the various VFD drives. The main PLC control box wiring layout is shown in Figure 6.

Figure 3. Pilot plant PLC system.

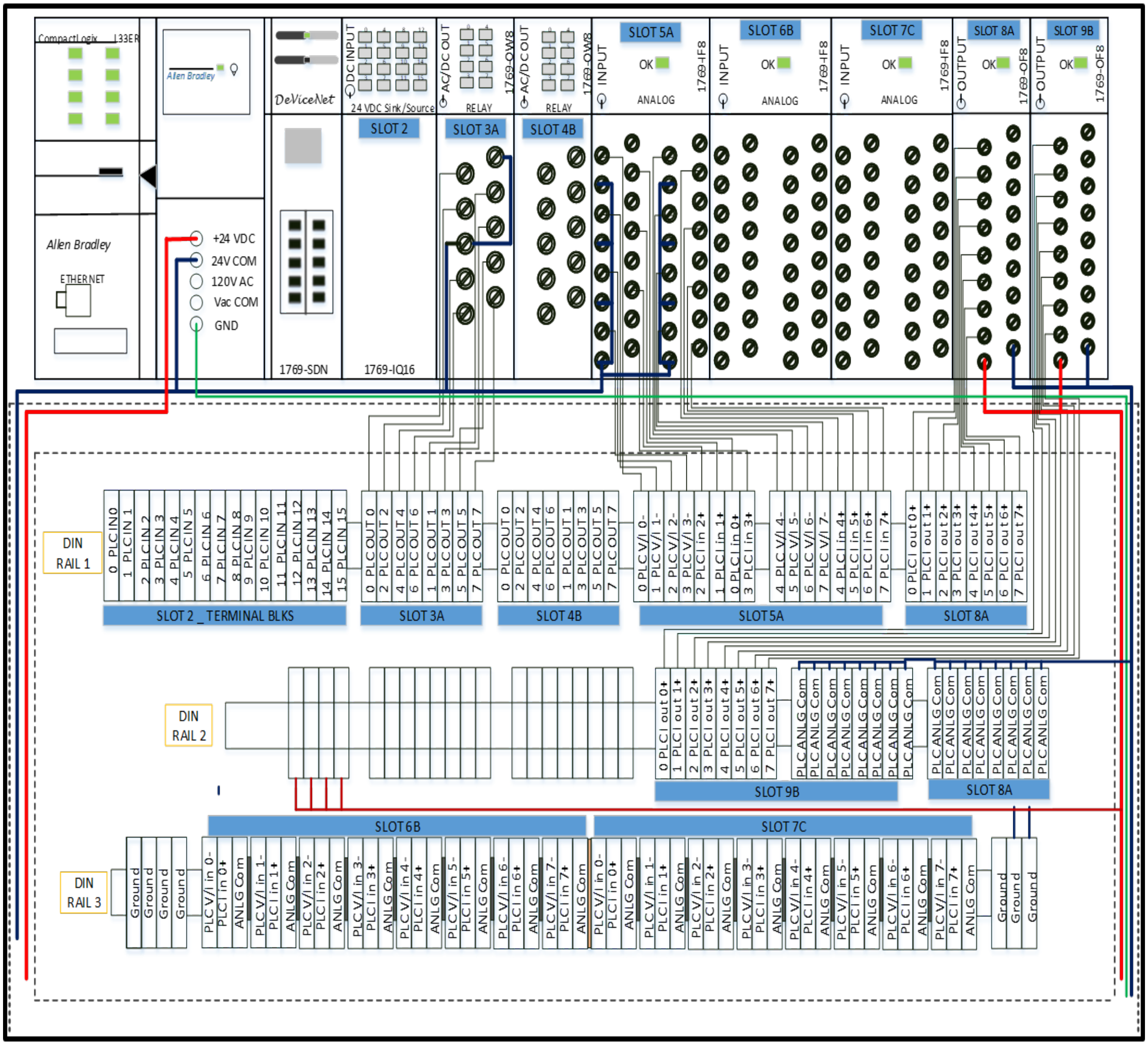

Figure 4. Plant PLC system wiring layout.

After the build of the PLC system, small junction boxes for connecting remotely located instrumentation and equipment was built as shown in Figure 7. This approach made wiring bundles and terminal connections much easier. Each junction box is built primarily for a pump control relay and to pass other sensor wiring to the main PLC unit. The wiring layout for the remote junction box is shown in Figure 8.

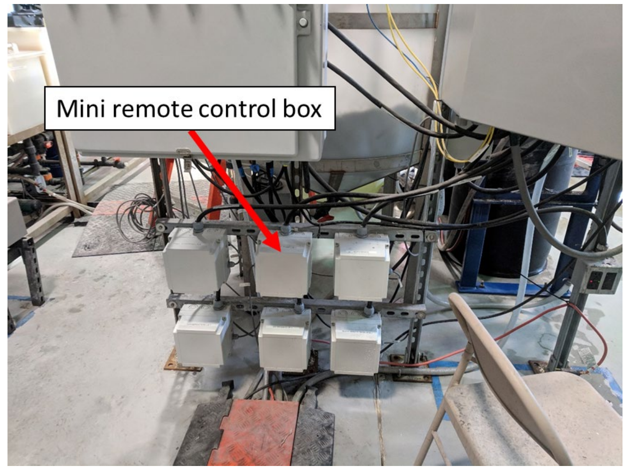

Figure 5. Remote junction boxes.

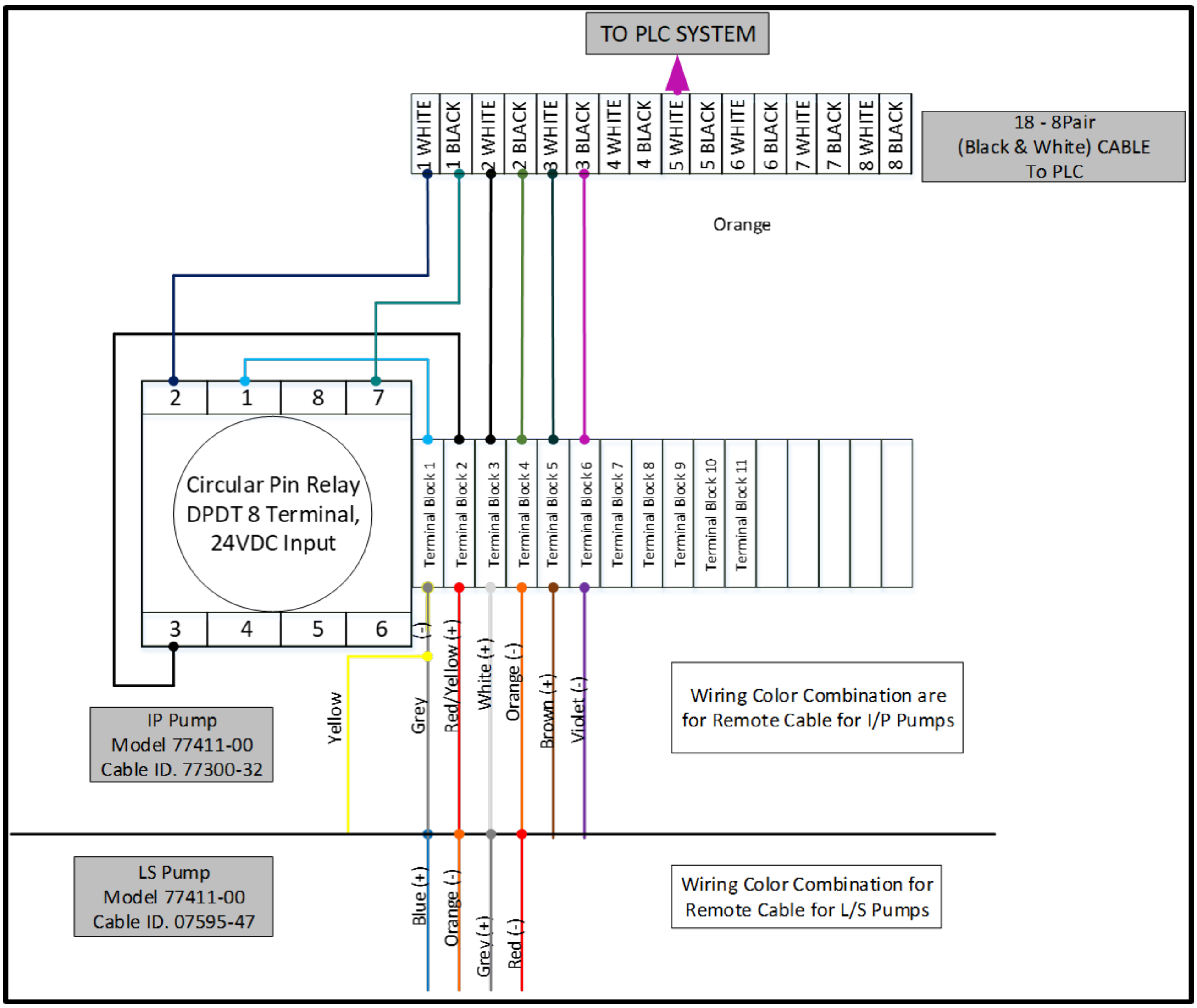

Figure 8. Remote junction box wiring schematic.

The plant automation process control is divided into four levels. Figure 9 shows the architecture of the overall plant automation strategy. The communication of the SCADA and supervisory system with the PLC controller is possible through an Ethernet connection. Here the SCADA system consists of a laptop computer that handles analog and digital I/O's through the PLC at the second level. SCADA in this control system is temporary and is as needed for testing purposes. In future work, the supervisory system level of control will incorporate OSIsoft PI for data management and advanced statistical process control. This data server will collect all field and plant data to the SCADA system for advanced control and optimization analysis.

The PLC receives information from field input instrumentation, and sensors, processes the signal using the logic program and sends an appropriate response signal to output devices.

The system has RSLogix 5000 and FactoryTalk View. The RSLogix 5000 is the platform for ladder logic programming whiles FactoryTalk View is a human-machine interface (HMI) software application dedicated for machine-level operator interface devices (e.g., Panelview Plus) by providing visualization of the system.

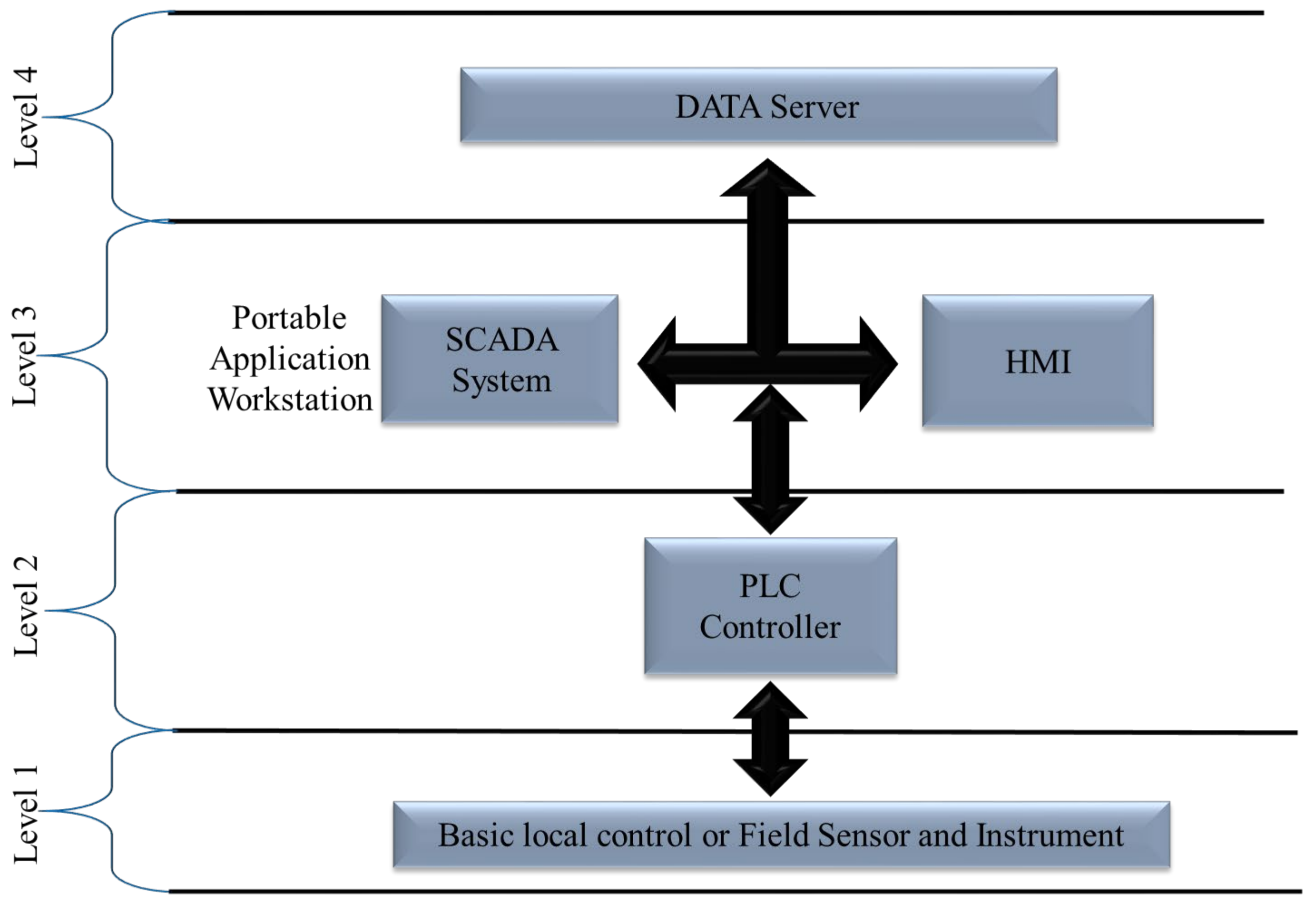

Figure 9. The plant automation system architecture.

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Instrumentation and Equipment

This section presents the different equipment and instrument sensors selected for use in the pilot plant. Selection criteria were based on low cost, suitability, and reliability for industrial chemical purposes. For heating, a 400-watt fluoropolymer heater (Process Control HX 4229-P2) is used in combination with a conductive liquid level probe (Process Control LC2H12) and thermocouple (top mounted). For tank level control an ultrasonic analog output sensor (AB 873P-D30AI-2500-D4) has been used for solution holding tank or recirculation tanks. Two wire submersion pH and ORP sensors (S8000CD & S8000CD-ORP) assembled with 4-20mA module transmitter and automatic temperature compensation (ATC) of Sensorex make has been used for proportional-integral-derivative (PID) based neutralization and reduction processes. Masterflex peristaltic pumps (IP 7741-00 or LS 07528-10) of external 4-20mA control capability using DB9 cable and variable area flow meters (FCA00151) proves to be a good choice for plant flow applications.

Logic and GUI-HMI Design

A ladder logic program has been designed to operate acid leaching tank pH controls in both manual and automatic PID control mode. The same PID logic approach is used for ORP control, wastewater pH treatment, and level control.

The graphic user interface (GUI) HMI display is available on a 15-inch color touch screen mounted on the plant-based PLC controller box. Different display screens are created to allow easy operator control of processes whilst in the plant. The main screen of the HMI (Figure 10) presents the overview of the pilot plant leaching circuit as laid out in the P&ID. The next panel (Figure 11) display presents the current date, time and the operational control mode status for the critical variables for monitoring purposes. The HMI also provides status information about the set points, current variable reading, pump speed (rpm), and liquid levels. All the important data and trends are noted and stored in the system at a regular time interval.

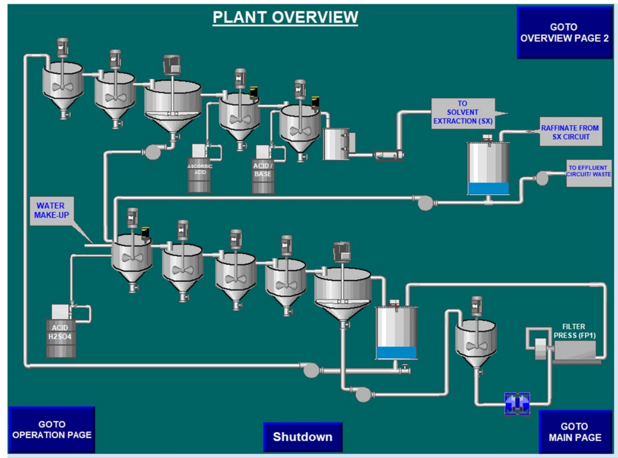

Figure 10. HMI display design.

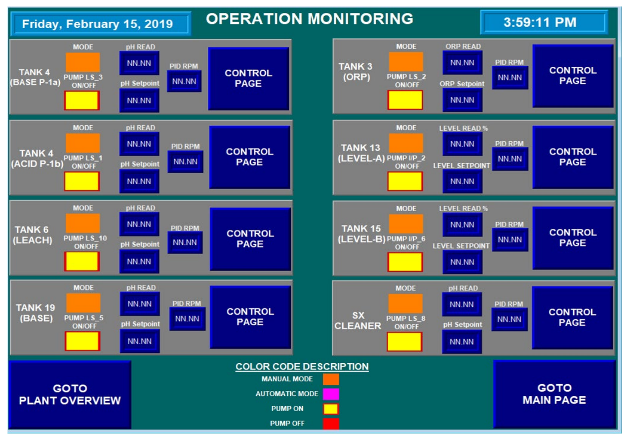

Figure 11. HMI operation monitoring display.

A control page push button (Figure 11) can be seen for each tank, which presents a new screen for controls. The control page screen as shown in Figure 12, for example, presents the graphical control panel for ORP and pH control system operation and each corresponding pumping system control buttons. When any pump images are pushed or clicked, the operator is able to access relevant trends corresponding to the individual loop control. The design of the control system allows operation in both manual control mode and automatic control mode.

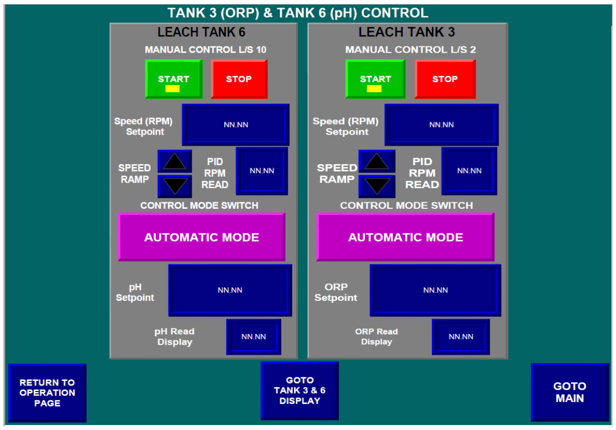

Figure 12. Variable PID control screen.

PLANT OPERATION

Installation and Shakedown

The leaching circuit for this project was assembled and installed at mine site located in Western KY. Leaching experimentation was achieved using Tanks 6, 7 and thickener (tank 12) (see Figure 2). Each leaching vessel was filled with water and tested under proposed control conditions. Issues such as temperature lose, and leakages at connecting joints and pump tubing were resolved. After successfully installing the system, exploratory no-load testing of the leaching system was performed with only water to examine system functionality and dependability before coal is fed into system.

Leaching experimentation

Initial leaching experiments were conducted following the shake down tests. It is important to note that the initial leach tests were performed in a circuit modified from that shown in Figure 2. The changes were to leach in one concurrent stream utilizing leach Tank 6 and 7. This arrangement bypassed the first leaching leg of tanks 10, 1, 2, 8, 9. The general circuit operational parameters are as follows:

- Leach solution: Fill both 100-gallon volume tank 6 and 7 with 350 liters of 1.2M sulfuric acid and heat to
- Temperature: Desired feed temperature of 75°C is achieved by manually adjusting heater.
- Feed: Prepare coal to the required size, then convey over the top of Tank 6 through a screw feeder system at a set feed rate of 50 lbs. per hour while simultaneously feeding water at a controlled flow rate of 1 gallon per minute using a rotameter. Agitators and pumps are kept in running condition for proper mixing and to preventing clogging.
- pH control: The operator enters pre-determined set point for the required acid leaching pH value. Set point of the pH and the exact pH reading of leaching material in Tank 6 are compared. The acid solution was automatically introduced into the leach tank 6 using a controlled peristaltic pump. The pH control was achieved by the manipulation of pH via proportional-integral (PI) method. To reduce signal disturbance in the readings by the pH electrode, a time average of 12 seconds was used to control the pH read.

After leaching, the slurry overflows into the thickener where the solid underflow goes into a holding tank for removal by filtration. Solid waste is treated and disposed of appropriately.

SX Pretreatment operation

The thickener overflow and clarified leachate from the filtration process described previously are transferred to tank 13 and tank 4 from the leaching process. In the reduction tank, ORP of the solution is measured in millivolts and pH value of the solution is adjusted in tank 4.

The valence state of iron in the SX feed is important to prevent SX contamination. The reductant solution was automatically introduced into the mixing tank 3 using a controlled peristaltic pump. ORP control was achieved by ORP manipulation via PID methodology. The pH of the neutralization vessel (tank 4) solution was maintained by the PLC controller using the solution pH as the controlling parameter. To reduce signal noise in the readings by the pH electrode, a rolling average of 10 seconds was used to control the pH value.

Solvent Extraction (SX) operation

The continuous SX circuit following leaching and pretreatment steps consists of loading, scrubbing, and stripping stages. The SX mixer-settler unit is setup to internally recirculate the aqueous stream. The loading stage is filled with leachate from the SX pretreatment step at a set constant pump speed (rpm) whiles stripping and scrubbing stages is filled with hydrochloric acid solution at different concentration respectively at startup. Recirculation is needed to build the concentration of desired REEs and reduce reagent consumption. Due to low concentration of REEs in the feed stream, the SX circuit was operated for extended period of time to reach equilibrium. Samples taken from solvent extraction process are analyzed to ascertain the REE concentration. Further treatment of stripped solution bleed was treated with acid to selectively precipitate REEs to produce the final rare earth oxide (REO) product.

The trial-and-error method of PID control tuning used for pH, ORP, level and flow control was implemented by RSLogix 5000 PID function block in the ladder logic programs. This function is used for level control, control of ORP and pH in the leaching circuit and also controlling the wastewater pH value. Tuning of the individual proportional, integral, and derivative term to any particular system can be achieved using the trial and error method for each process. In the PID function, the measured variable or process variable is compared to the reference set point to calculate an error value which is used to manipulate the controlled variable to maintain the desired reference.

PROCESS MONITORING TRENDS

The HMI is also used as a monitoring tool for present and past data of analog and digital types. These trend graphs are designed and programmed using the Factory Talk View SE software. Based on operators need; the trend displays control performance at different times. With different number of process variables being maintained within specified limits in order for the plant to operate smoothly, routine process monitoring will ensure that system performance satisfies the operating objectives.

Statistical process control was evaluated to ensure that the process operates efficiently. Figure 13 and Figure 14 both show control charts showing the performance of leaching experimentation pH for different case scenarios at specific set points. In each case, the leaching temperature condition was maintained at 75°C and constant feed rate.

Table 1 and Table 2 show the test process data for Case 1 and 2 at pH set points 2.0 and 2.3 respectively.

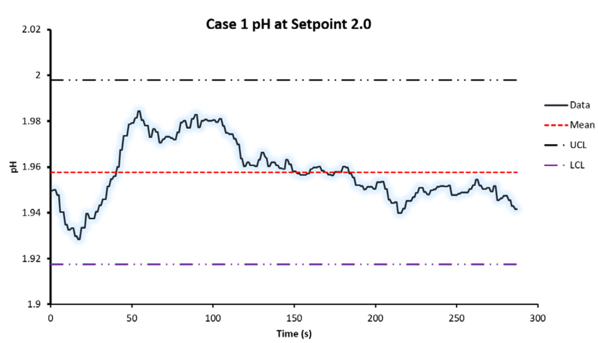

Figure 13. Leaching pH control chart.

Table 1. Average of pH value recorded.

pH Setpoint	Mean pH	Standard Deviation	Upper Control Limit (UCL)	Lower Control Limit (LCL)
2.00	1.96	0.0134	1.998	1.917

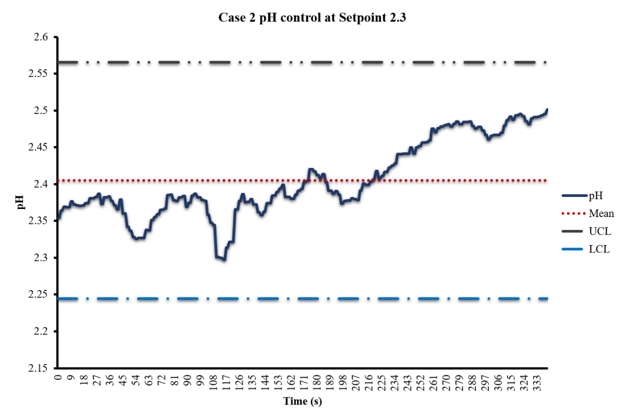

Figure 14. A pH control trend during the leaching test.

Table 2. Mean pH values for the leaching test.

pH Setpoint	Mean pH	Standard Deviation	Upper Control Limit (UCL)	Lower Control Limit (LCL)
2.30	2.40	0.054	2.565	2.245

By estimating the standard deviation (σ) of the sample data, the control limits were computed to determine the $\pm$ 3σ control limits to characterize the system. Based on this result, the typical process variation can be inferred to detect any anomalous or out of control conditions. The small variability of data about the mean value is attributed to common electric noise associated with adjacent motor drives. Tuning of the PID will consequently help improve small deviations about the mean.

CONCLUSION AND RECOMMENDATIONS

The main goal for the development of a PLC-based process control system is to operate the pilot plant leaching and solvent extraction circuit safely, accurately, and at the lowest cost for the recovery of coal-based rare earth elements (REEs).

Literature review on leaching, solvent extraction, and process control assisted in a more complete understanding of mineral recovery processes and the different implementation methods of process control. The implemented Six Sigma techniques were used successfully to scope the process and provided valuable knowledge and understanding towards safe design and development of process controls.

Based on the experimentation, testing logic programming, tuning and debugging the pilot-scale process control PLC system and logic described in this paper was able to operate and successfully control critical process variables (pH, ORP, level, and flow) in the recovery of REEs.

As part of ongoing research, further system tuning is essential to fully regulate process variables and fulfill the optimization objective. In addition, SCADA and supervisory level data will be used to perform advanced process control and statistical analysis

REFERENCES

1. Bauer, D., Diamond, D., Li, J., Sandalow, D., Telleen, P., & Wanner, B. (2010). US Department of Energy critical materials strategy
2. Zhang, W., Rezaee, M., Bhagavatula, A., Li, Y., Groppo, J., & Honaker, R. (2015). A review of the occurrence and promising recovery methods of rare earth elements from coal and coal by-products. *International Journal of Coal Preparation and Utilization*, *35*(6), 295-330
3. Honaker, R. Q., Zhang, W., Yang, X., & Rezaee, M. (2018). Conception of an integrated flowsheet for rare earth elements recovery from coal coarse refuse. *Minerals Engineering*, *122*, 233-240.
4. Xie, F., Zhang, T. A., Dreisinger, D., & Doyle, F. (2014). A critical review on solvent extraction of rare earths from aqueous solutions. *Minerals Engineering*, *56*, 10-28
5. Zhang, W., & Honaker, R. Q. (2018). Rare earth elements recovery using staged precipitation from a leachate generated from coarse coal refuse. *International Journal of Coal Geology*.
6. Kashiwakura, S., Kumagai, Y., Kubo, H., & Wagatsuma, K. (2013). Dissolution of rare earth elements from coal fly ash particles in a dilute H2SO4 solvent. *Open Journal of Physical Chemistry*, *3*(02), 69.
7. Bascur, O. (2019). Process Control and Operational Intelligence. SME Mineral Processing and Extractive Metallurgy Handbook, 277.
8. Ray, T., Suresh, A., Dash, P. S., & Banerjee, P. K. (2015). Instrumentation and automation system at coal leaching pilot plant. *International Journal of Automation and Control*, *9*(2), 158-171.
9. Wu, M., She, J. H., Nakano, M., & Gui, W. (2002). Expert control and fault diagnosis of the leaching process in a zinc hydrometallurgy plant. *Control Engineering Practice*, *10*(4), 433-442.
10. Dash, P. S., Adari, P. R. K., Suresh, A., & Banerjee, P. K. (2014). SCADA-based control system of pilot plant for chemical leaching of high ash Indian coals. *International Journal of Automation and Control*, *8*(4), 362-380.
11. Alphonsus, E. R., & Abdullah, M. O. (2016). A review on the applications of programmable logic controllers (PLCs). Renewable and Sustainable Energy Reviews, 60, 1185-1205.
12. He, J. Q., Chen, W., Gu, G. L., & Fu, X. R. (2013). Automated Processing System of Electrolytic Manganese Wastewater Treatment and Resource Utilization. In *Applied Mechanics and Materials* (Vol. 271, pp. 204-207). Trans Tech Publications.