Argonne National Laboratory
Energy Systems Division
Chemical Process Pilot Plant

The pilot plant pictured below was constructed at Argonne in 1999-2000 to evaluate the application of electrodialysis (ED) to the recovery of salts from concentrated brines. The pilot plant is equipped with a commercial scale ED stack served by computer-monitored-and-controlled diluate, concentrate and electrode rinse solution loops. Although designed initially for the salt recovery application, the facility can be used to determine the technical feasibility of electrodialysis for a variety of applications. All equipment, instrumentation and piping in contact with the process are made of non-corrosive materials that will accommodate a wide range of solutions. The piping is schedule 80 PVC and can be easily modified or rerouted depending on the requirements of the process to be evaluated.

Electrodialysis Pilot Plant

Plant Equipment

The heart of the pilot plant is the electrodialysis system. The ED stack was supplied by Eurodia. The stack, EUR-40-76-5, contains 76 active cell pairs and is equipped with membranes of 0.4 m^2 effective area. The cation membranes are Neosepta CMX and anion membranes are Neosepta AM1. The stack was supplied with three sets of rod electrodes: (i) nickel anodes and cathodes for operations with NaOH electrode rinse solution; (ii) DSA/iridium coated anodes and graphite cathodes for operations with $NaNO_3$ electrode rinse solution; and (iii) DSA/ruthenium coated anodes and graphite

cathodes for electrode rinse solutions containing HCl. Power is supplied to the ED stack by a Rapid Power DC rectifier specified to provide up to 575 amps and 100 volts (changeable to 210 volts). Platinum wire probes are installed in the ED stack near the cathode, anode and both sides of the mid electrode chamber, and at cells 14, 28, 52 and 66 to allow monitoring of voltages across the stack. The stack has a pressure rating of 75 psig.

The pilot-plant is designed to provide for operation of the stack at differential pressures up to 20 psi should differential pressure be expected to improve stack performance for a given application. A differential pressure controller (operating on the differential pressure between the concentrate and diluate return streams) is employed for testing at varying differential pressures. For testing from 0-20 psi differential pressure, a back pressure controller on the diluate return stream is used to compensate for the higher downstream pressure of the concentrate loop.

The ED diluate loop consists of a 1500-gallon HDPE feed tank, Vantom model CG-PY800 centrifugal feed pump, mass feed rate and back-pressure control, and instrumentation to monitor mass flow rates, densities, temperatures and pressures. The feed tank is mounted on a weigh scale and equipped with an ultrasonic level transmitter.

The ED concentrate loop consists of a 1250-gallon HDPE feed/salt settling tank, Vantom model CG-PY800 centrifugal feed pump, mass feed rate and differential pressure control, and instrumentation to monitor mass flow rates, densities, temperatures and pressures. The concentrate loop also is equipped with a plate and frame exchanger cooled by chilled water. The feed tank is mounted on a weigh scale and equipped with an ultrasonic level transmitter. The tank has an installed mixer and a bottom fed slurry pump for continuous operation with solids.

The plant is equipped with an in-line filtration system on both the concentrate and the diluate flow loops. This system uses Hayward bag filters model FLT4202, with pre-filtration Y-strainers. This allows for the continuous filtration of the concentrate and diluate flows, down to the micron level. The system can be bypassed to feed unfiltered solutions to the stack.

Coriolis meters that provide accurate measure of the mass flow, density and temperature of a process stream are installed in the diluate feed and return streams and in the concentrate feed stream. The diluate and concentrate tanks are mounted on load cells.

The electrode rinse loop contains a 500-gallon HDPE feed tank, March model TE-8K-MD feed pump, manual anolyte and catholyte flow rate adjustment, instrumentation to monitor flow rates, pressures and temperatures, and hydrogen purge equipment. A small amount of hydrogen gas (max 0.16 CFM) is generated at the cathode and oxygen gas (max 0.08 CFM) at the anode. To prevent the mixing of these gases, the hydrogen is removed from the catholyte by bubbling nitrogen through retained solution. The diluted hydrogen gas is vented through an FRP blower to atmosphere. The electrode rinse tank is equipped with an ultrasonic level indicator and pH probe.

Features included in the pilot plant for operations with solids include: clear piping runs for visual observation of solids; diluate and concentrate piping manifolded to allow upflow or downflow through the ED stack; cooler installed in the concentrate return loop to crystallize out solids from saturated solutions; and concentrate tank design w/ mixer to allow salts to settle or to be slurried.

Other systems in the pilot plant are dedicated to the storage and periodic preparation and handling of commercial, prepared, and waste brines. Pilot plant control alarms, shutdowns and safety interlocks are handled by a dual computer control and data acquisition system.

Data Acquisition System

The plant incorporates an integrated, dual stage supervisory control and data acquisition system (SCADA). The hardware interface consists of an Allen-Bradley small logic controller (SLC) 5/05 system running parallel to a National Instruments FieldPoint system. The SLC, interfaced with RSView32, provides robust system's control and safety interlock capabilities, while the FieldPoint system, when interfaced with BridgeView software, allows for advanced data acquisition. Seamless integration between systems is achieved utilizing Object Linking and Embedding for Process Control (OPC) Servers, linked over Ethernet. This design allows for the SLC to handle plant operations (pump control, PID loops, and safety interlocks) while BridgeView is dedicated to data acquisition.

Complete facility operations are controlled through the SLC. Flow, pressure, and temperature parameters for PID loops are entered through the HMI using RSView32. Facility interlocks are also monitored by the SLC. Multi-level system shutdowns are initiated upon upset conditions to insure operator safety and equipment protection. Using OPC servers, SLC data common to both operations and data acquisition can be accessed by BridgeView. All critical systems are monitored and recorded by BridgeView. Data from instrumentation surrounding the ED concentrate and diluate flow loops are particularly important for material balances to determine water transfer and current efficiency. Over 100 different operating parameters can be monitored and recorded. The following is a list variables typically recorded.

- Weight of concentrate tank
- Weight of diluate tank
- Level of concentrate tank
- Level of diluate tank
- Concentrate feed pressure at stack
- Diluate feed pressure at stack
- Differential pressure across stack
- Concentrate feed flowrate
- Concentrate return flowrate
- Diluate feed flowrate
- Concentrate feed density
- Concentrate return density

- Diluate feed density
- Concentrate feed temperature
- Concentrate return temperature
- Diluate feed temperature
- Voltage across stack
- Current across stack