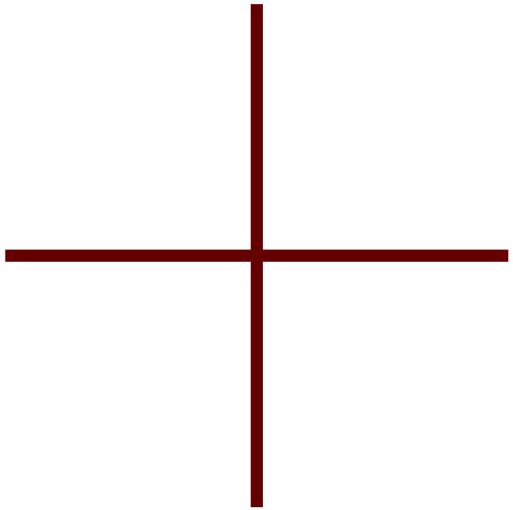
Lessons Learned from SRS Salt Processing Scale-Up: Filtration and Solvent Extraction



SRNL™
SAVANNAH RIVER NATIONAL LABORATORY

We Put Science To Work

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Filtration and Solvent Extraction Testing

- Pilot-Scale Filtration Testing
- Rotary Microfilter Development and Testing
- Solvent Extraction (MCU) Testing at Vendor
- Solvent Extraction (MCU) Testing at SRS prior to Startup

Pilot-Scale Filtration Testing

- Constructed pilot-scale filtration unit at the University of South Carolina in 1994-1995
  - DOE grant to USC
  - Commercial construction firm with USC oversight
  - Full-scale filter tubes
    - 0.625 in ID
    - 10 ft length
    - 7 tubes versus 144 in facility
  - 150 – 500 gallon feed
  - 225 gpm feed pump

Testing Scope and Objectives

- Completed ~ 30 test campaigns from 1995 – 2003
  - Tetraphenylborate slurries (1 – 10 wt %)
  - Sludge/MST slurries (0.06 – 12 wt %)
  - Sludge slurries (0.06 – 4.5 wt %)
  - 0.5 and 0.1 μm filter media
  - Lasentec particle size analyzer
- Prepared 600 gallons of simulated sludge for SRS Salt Processing
- Cleaned with oxalic acid and nitric acid

Operation

- Operated around the clock
- Student operators
  - 4 hour shifts, 2 per shift
- Limited SRNL presence at test site
  - Routine data review
  - Hold points established for critical items
  - All corrective actions and recovery paths approved by SRNL
- Data acquisition system
- Analytical laboratory
  - Particle sizing, IC, GC, AA, Turbidimeter, CEM solids oven
- SRNL controlled and monitored all test scope
- Frequent communications
- Data review by SRNL in near "real time"
- Bulk of data analysis and reports by SRNL

Pilot-Scale Filtration Testing Discussion

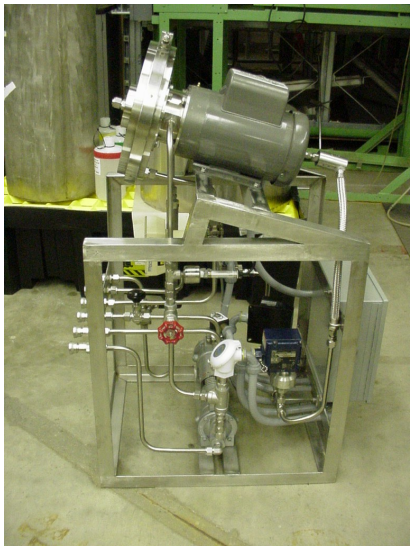
- Procedure development
  - USC personnel drafted procedures
  - SRNL personnel reviewed and approved
- Laboratory analyses conducted at USC lab
  - Delays in receiving results due to large number of samples and one chemist
  - Required cross-checking of methods by SRNL and learning curve for USC personnel
- Equipment problems
  - Filter leak
- Test results reviewed by USC and SRNL personnel
- Test changes approved by SRNL
- Pretest reviews
  - Formal Readiness Reviews with Lines of Inquiry performed for some tests by cross discipline team (Customer, Operations, SRNL)
  - Independent and periodic QA audits
  - Clean water flux evaluated prior to start of test

Pilot-Scale Filtration Summary

- What went well
  - Quantity and quality of data
  - Close location (1.5 hr drive) allowed rapid recovery actions
  - Strong teaming
  - Ran continuously, around the clock
  - USC personnel available around the clock
- What did not go well
  - Delay in analytical results
  - Heels and dead legs increased cleaning time and chemicals
  - Difficulty measuring pressure around filter accurately
  - Failed to develop DOE-independent funding for facility leading to higher operating costs
  - Limited staffing led to extended delays for maintenance issues
- What would you do differently if doing it over
  - More aggressive effort to establish long term business plans
  - Measure turbidity with meter rather than visually earlier (quantitatively rather than qualitatively)
- What would you recommend definitely be done in designing, constructing, operating a similar facility
  - Minimize heels and dead legs in facility to make cleaning easier
  - Broader vision of life cycle and converting to multipurpose testing facility
  - Involvement of other university departments (e.g., for instrumentation, data acquisition, chemical analysis support)

Rotary Microfilter Development and Testing

- 1 – disk commercial unit for feasibility testing at vendor
- 1 – disk commercial unit for actual waste testing at SRS
- 3 – disk commercial unit for pilot-scale reliability testing at USC
- 25 – disk prototype unit (full-scale) for demonstration of enhanced design



Rotary Microfilter Testing Scope and Objectives

- Conducted feasibility test at vendor site
  - Evaluate performance on simulated SRS waste
- Conducted 2 tests with actual waste
  - Evaluate performance on actual SRS waste
  - Evaluate impact of radiation on unit
- Conducted 4 tests with pilot-scale unit (> 4,000 hours)
  - Evaluate reliability of unit
  - Evaluate impact of radiation on filter disks
  - Evaluate alternative filter media
- Conducted 2 tests with full-scale prototype unit
  - Enhanced 25 – disk units for deployment
  - Evaluate performance of new equipment design
  - Evaluate remote installation of disk stack
  - Evaluate technology for SRS IX prefilter, SRS sludge washing, Hanford Supplemental Pretreatment

Rotary Microfilter Operation

- Vendor Feasibility Test
  - Operated days
  - SRNL approved test procedure
  - SRNL provided feed
  - SRNL representative present for test
  - Manual data collection
  - Vendor report reviewed by SRNL
- Actual waste test
  - SRNL Shielded Cells
  - Operated days
  - 1 technician + 1 researcher
  - Manual data collection
- Pilot-scale
  - University of South Carolina
    - Around the clock
    - Student operators
      - 2/shift, 4 hour shifts
    - Data acquisition system
    - SRNL reviewed data and wrote report
  - SRNL
    - Operated days
    - 2 personnel
    - Manual data collection
- Full-scale Prototype
  - 2 technicians + 1 researcher
  - Operated days
  - Manual data collection

Rotary Microfilter Discussion

- Procedure development
  - USC prepared procedures – SRNL reviewed and approved
  - SRNL prepared procedures
- Laboratory analyses limited to turbidity
  - Conducted at site
- Equipment problems
  - None during actual waste and full-scale tests
  - Rotor plugged during pilot-scale test – due to concentrated manganese feed slurry
  - Rotary union failure – due to manufacturer using wrong material
  - Ceramic membrane tore – final selection was SS membrane
  - Interlocks shutting system down due to low tank level, elevated temperature, and low feed pressure
- Test results reviewed by SRNL, LWO, and DOE-SR
- Pretest reviews
  - Readiness Review (with formal lines of inquiry) performed before some tests
  - Clean water flux evaluated prior to start of test

Rotary Microfilter Summary

- What went well
  - No problems with mechanical seal
  - Thorough review of prior test history and related designs (SRS, LANL, INEEL, DuPont, ASPECT)
  - Developed Intellectual Property working relationship with vendor and long term vision early
- What did not go well
  - Limited review of vendor electronics
  - Overaggressive procurement of 2 prototype units
  - Did not obtain end user commitment and project design owner early as desired
- What would you do differently if doing it over
  - Data acquisition system would improve quantity and quality of data
  - Incorporate more advanced vibration, wear, and heat measurements earlier
  - Order broader set of membranes for testing (instead of 2 units)
  - Expand effort to understand flow distribution in equipment
  - Pursue a commercial end user in parallel more aggressively
  - Earlier independent review of seal materials and options
- What would you recommend definitely be done in designing, constructing, operating a similar facility
  - Upgrade supply pump rather than use “best available”
  - Add automated data acquisition
  - Obtain operations engineer overview and involvement (attempted but unable to raise user interest high enough)
  - Upgrade system electronics

Solvent Extraction (MCU) Testing at Vendor

- Contract awarded for testing to Wright Industries (WII)
- 3 months from award of contract to start of testing
- Testing conducted over 16 months
- Scope increased as a result of testing (as expected)
- Cost ~ $5 – 6 million

Solvent Extraction Vendor Test Work Completed

- Individual V-10 & V-05 Testing
  - Hydraulics
  - Air Flow
  - Mass Transfer
  - Solvent Carry Over
- Decanter Testing
  - Solvent Carryover
  - Solvent Droplet Distribution Generated by Mix/Shear Pump
  - Pump Required for Coalescer Pressure Drop
- Integrated Testing
  - Durability Testing
  - Mass Transfer
  - Solvent Carryover
- Significant modification resulted from work completed

Solvent Extraction Vendor Test Operation

- Operation included around the clock and day only
- SRS-LWO personnel at test site
- PLC control and data acquisition system
- Offsite analytical lab
- Personnel
  - 1 operator
  - 1 SRS representative
  - 1 test engineer
  - 2 maintenance personnel available

Solvent Extraction Vendor Testing Summary

- What went well
  - Contactors/coalescers met performance requirements
  - Testing met aggressive schedule
  - Transfer of sample handling protocol to commercial lab
- What did not go well
  - Material compatibility - contamination of solvent from DEHP in plastic tubing
  - Vendor measured vibration by acceleration rather than vibration amplitude
  - Transfer of organic sample digestion protocol to commercial lab
  - Pressure drop across the coalescer higher than expected
    - May have been early indication of sodium aluminosilicate
- What would you do differently if doing it over
  - Select vendor with more expertise in process engineering
- What would you recommend definitely be done in designing, constructing, operating a similar facility
  - Ensure vendor control system is aligned with SRS/MCU control system

SRS Solvent Extraction (MCU) Testing Prior to Startup

- Moved unit from WII to SRS
- Installed and conducted mass transfer tests with simulant
- Full-scale
- Conducted tests to confirm unit performance following installation

SRS Solvent Extraction Testing Issues

- Sodium Aluminosilicate (NAS) Precipitation
  - Resulted in coalescer media pluggage
  - Required filtration unit installation
  - May have occurred during WII testing
  - Could have tested in advance
  - Did not control vendor sufficiently
- Iron Contamination in Scrub Feed Tank
  - Provided sodium aluminosilicate precipitation site
  - Existing tank re-used and not thoroughly cleaned
- Emulsion
  - Formed In wash contactors
  - Solvent density out of specification
  - Solvent density monitored with bubbler – went off scale – could have collected additional samples

SRS Solvent Extraction Testing Operation

- Around the clock
- 2 operators in control room
- 2 operators in field
- 1 engineer
- Samples sent to SRNL for analysis

SRS Solvent Extraction (MCU) Testing Summary

- What went well
  - Process performed as expected
  - Preplanning between MCU, SRNL, and Analytical personnel allowed rapid completion of sample analyses
  - Teaming between MCU and SRNL allowed rapid resolution of operational issues
- What did not go well
  - Sodium aluminosilicate precipitate formed
  - Scrub feed tank contained iron contaminant
  - Emulsion formed due to Isopar® L evaporation
- What would you do differently if doing it over
  - When density measurement went off scale, collect additional samples
  - Ensure all tanks are clean
  - Re-use of existing equipment requires thoroughly documented inspections/ testing – expect the unexpected
- What would you recommend definitely be done in designing, constructing, operating a similar facility
  - Consider Process Upsets for Instrumentation Design
  - Ensure adequate instrumentation in facility