Small-Scale Testing of Potential Small Column Ion Exchange (SCIX) Grinder Technologies

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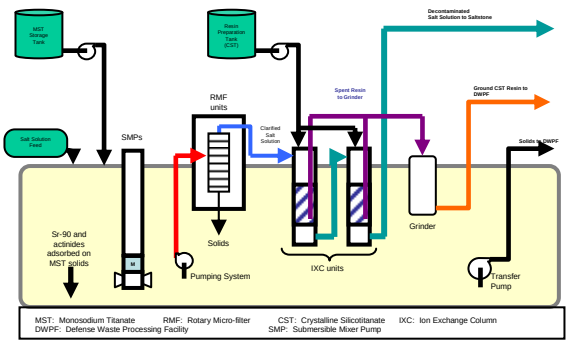
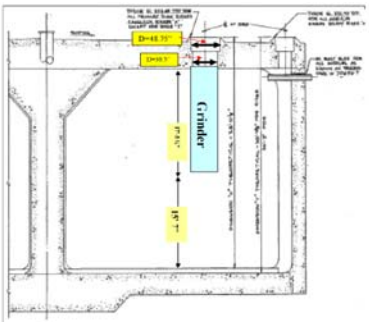
Purpose: Evaluate candidate technologies for grinding spent SCIX bed material, Crystalline Silicotitanate (CST)



HLW tank schematic with SCIX columns and grinder



Grinder module concept

Project Strategy

- Downselection to three candidate technologies
  - Suitable geometry
  - Suitable grinding capability (to be confirmed by testing)
  - Suitable ability to design/customize for remote environment
- Testing at benchtop or pilot-scale
  - Inline disperser: Ikaworks, Wilmington, NC
  - Immersion mill: Hockmeyer, Elizabeth City, NC
  - Sonication: Benchtop testing at the Vitreous State Laboratory, Catholic University
- Chabazite zeolite simulant and CST used for testing
- Test reporting and recommendations (in progress)
- Related SCIX sluicing and ground product pumping testing activities, in progress
- Decisions, design, etc. - Savannah River Remediation (SRR) activities, in progress

System Requirements

- Grinder module must fit inside an HLW tank riser
- Integrate with sluicing operation
  - Reasonable batch sizes for processing approximately 420 gallons of spent column bed material every two weeks
  - Reasonable flexibility to handle variable solids contents from sluicing
- Ground product will be transferred to a sludge batch for eventual vitrification at the Defense Waste Processing Facility (DWPF)
- Initial CST particle size: 250 – 600 microns
- Ground particle specifications:
  - Mean particle size: 5-20 microns
  - All particles (>98%) less than 38 microns
  - Minimize fines

Previous Savannah River National Laboratory (SRNL) Testing - Ikaworks

- WSRC-TR-2000-00350: Tested unit with CST and zeolite.
- WSRC-TR-2005-00282: Tested grinder in inverted configuration with recirculation, see Figure.
- Some clogging experienced in 2000 testing was alleviated by 2005 configuration; test objectives were met including testing of a full-scale grinder. Ground product specification was larger than current requirements.

View of inverted inline disperser (2005) during test setup. Red cap is the inlet to the unit; flow is recirculated using a pump.

Previous Savannah River Site (SRS) Deployment - Hockmeyer

- 2008: High concentrations of zeolite (IE-95) were ground during heel removal from Tanks 18 and 19, see Figure.
- Processing of about 21,000 gallons of zeolite to size specification required for CST.
- Hockmeyer HM-20 (16" diameter mill) in a 23" diameter vessel.
- Overflow configuration used as classification method for particle size. Material fed to grinding chamber at a controlled rate to match overflow rate.

View of the Hockmeyer immersion mill during setup at SRS. Motor is at the top; mill inside near the bottom of the tube.

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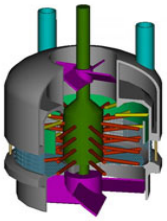
Hockmeyer Test Setup - August 2010

- Based (partly) on previous grinding of zeolite at SRS, in Tank 18/19 in 2008
- Batch processing tested with projected scaleup to approximately 210 gallon batch sizes
- Overflow method tested as a possible classification method; however, results inconclusive due to excessive fines in the chabazite simulant
- HM-2.5 (2.5 liter bead volume) mill (9.5" diameter) with grinding batch size of approximately 50 gallons of chabazite simulant slurry
- Rotating pegs within the mill basket loaded with 1.5 mm grinding beads provided the grinding energy
- Mixing of grinding tank volume provided by a propeller on the shaft
- Tested 10 – 40 wt% solids concentrations of chabazite simulant

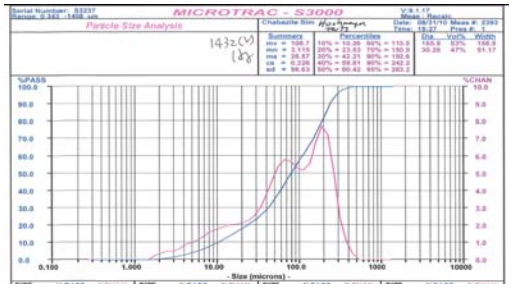
Hockmeyer test setup: Immersion Mill being lowered into the grinding tank

Hockmeyer test setup: Immersion Mill in grinding tank showing mixing from propeller rotation

Hockmeyer test setup: Immersion Mill (after test) showing beads and mixing pegs



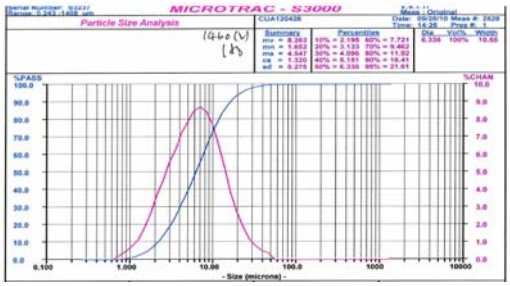
Immersion mill basket cutaway view showing pegs and propeller



Hockmeyer test results: Particle size distribution of chabazite before grinding (Material is smaller size than desired)

Hockmeyer Test Results

- Initial material had excessive fines, but with sufficient number of larger particles present to conduct testing
  - Mean diameter of volume distribution: ~109 microns
  - About 91% passes 250 micron
- Quick grinding (30 minutes) to approximately 8 micron mean diameter with less than 2% above 38 microns
- Overgrinding possible with potential for excessive fine production
- Successful grinding at 20 – 40 wt% concentrations; however, shear-thinning flow behavior observed at 40 wt%
- Vigorous mixing and recirculation provided by the propeller
- Minor wear measured for grinding beads and pegs
- Followup testing (analysis in progress) suggests CST grinds more quickly and more finely than simulant



Hockmeyer test results: Particle size distribution of chabazite after grinding 30 minutes (Concentration 28 wt%)

Ikaworks Test Setup – September 2010

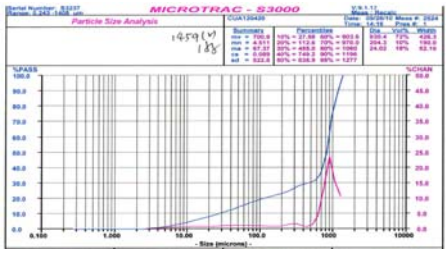
- Based (partly) on previous design and testing work at SRS in 2000 and 2005
- Recirculating line with a sidestream to the grinder
- Dispax reactor DR2000/5, 25 hp motor, three-stage grinder, recirculating in a batch size of approximately 10 gallons, using a recirculation flow rate of approximately 50-60 gpm with about 20% shunted through the grinder
- Tested 10 – 25 wt% solids concentrations of chabazite simulant
- Tested several rotor/stator sets with different grinding clearances

Ikaworks test setup: Inline disperser in foreground with mixing tank on the left

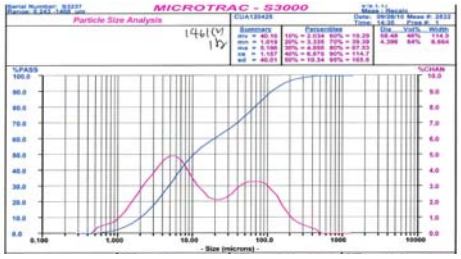
Inline disperser cutaway view

Ikaworks Test Results

- Quick grinding within a few minutes to approximately 15 micron mean diameter
- Bimodal size distribution with little change after initial grinding
- Ground product has greater than 2% by volume oversized (larger than 38 microns)
- Clogging of inlet experienced at 25 wt% concentration and finer rotor/stator sets
- Wear data suggest the coarser rotor/stator sets do little of the grinding work



Ikaworks test results: Particle size distribution of pre-ground chabazite



Ikaworks test results: Particle size distribution of chabazite after 2 hours (25 wt% solids)

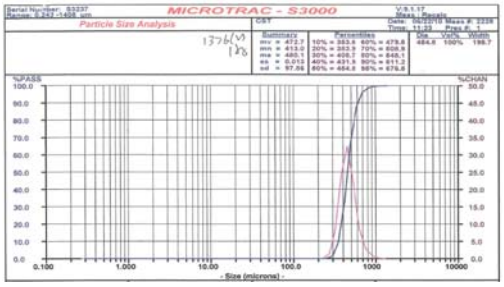
Sonication Test Setup – August/September 2010

- Benchtop scale: Sonicator probe in a beaker
- Heat Systems W-380 Sonicator operating at 20 kHz with a 475 watt power supply and ½" disruptor horn, and adjustable probe oscillation of approximately 0 to 120 micron
- Tested CST and chabazite

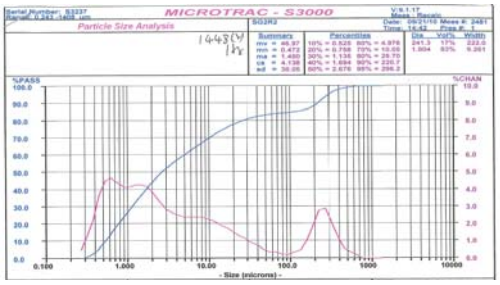
Sonication of chabazite

Sonication Test Results

- CST grinds to a bimodal distribution with apparent erosion of initial particle into micron-sized particles
- Chabazite is harder to break apart than CST



Sonication Results: Particle size distribution of pre-ground CST



Sonication Results: Particle size distribution of CST after 90 seconds

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