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**Transport and Storage
Properties of CST Slurries
for the Savannah River Site**

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T. D. Hylton
T. E. Kent

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Chemical Technology Division

**TRANSPORT AND STORAGE PROPERTIES OF CST SLURRIES
FOR THE SAVANNAH RIVER SITE**

P. A. Taylor
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T. D. Hylton
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TRANSPORT AND STORAGE PROPERTIES OF CST SLURRIES FOR THE SAVANNAH RIVER SITE

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ABSTRACT

The Oak Ridge National Laboratory (ORNL) is performing tests to address issues related to the handling and storage of crystalline silicotitanate (CST) for the Savannah River Site (SRS) Defense Waste Processing Facility (DWPF) recycle treatment program. The DWPF recycle treatment program and the SRS Salt Disposition Alternatives program share many common concerns related to CST slurry transport. Therefore, the DWPF recycle treatment program scope was modified to better address the salt disposition concerns. These tests evaluated the physical and chemical compatibility of CST with the operating environments that could be experienced during treatment of the SRS high-level tank waste or DWPF recycle stream, and subsequent handling, storage, and transport of the CST.

Pumping tests have shown that a 24 wt % slurry of CST in water can be transported at fluid velocities of 4.3 ft/s (45 gpm in a 2-in. pipe) with no visible settling of the CST particles, while a 5 wt % slurry will stay suspended at a velocity of 3.8 ft/s. The CST was easily mobilized after purposely plugging sections of pipe. The CST particles were rapidly broken up by a centrifugal pump into very small particles ($<150\text{ }\mu\text{m}$, with the majority being $<1\text{ }\mu\text{m}$). A progressing cavity (Moyno) pump caused less damage to the CST particles.

Slurries of CST in water showed low abrasivity to 304L stainless steel and moderate abrasivity to A106 mild steel. A slurry of CST in median supernate (2.9 *M* NaOH plus other salts) showed low abrasivity to A106 steel, so the higher abrasivity result for CST in water was probably caused by oxidative corrosion. Sludge simulant slurries showed low abrasivity, but mixtures of sludge and zeolite showed moderate abrasivity.

Storage of cesium-loaded CST in supernate simulants has shown possible leaching of the cesium from the CST into median and high-concentration (13.5 *M* NaOH) supernate simulants at 80°C. There was no measurable leaching after 105 days in any of the simulants at 25°C, or in low-concentration simulant (0.01 *M* NaOH) at 80°C.

Mixtures of CST in SRS sludge simulants have shown minimal tendency to cause caking or hard layers. Supernate simulants that cover the range of solutions in the SRS high-level waste storage tanks have shown minimal effect on the particle size of the CST sorbent.

1. INTRODUCTION

The Oak Ridge National Laboratory (ORNL) is performing tests to address issues related to the handling and storage of crystalline silicotitanate (CST) for the Savannah River Site (SRS) Defense Waste Processing Facility (DWPF) recycle treatment program. The DWPF recycle stream is a relatively dilute wastewater that requires pretreatment for radioactive cesium removal prior to discharge to the SRS Effluent Treatment Facility (ETF). CST is also being considered for treatment of the high-salt, high-pH supernate solutions in the high-level waste tanks at SRS, by the SRS Salt Disposition Alternatives Team. The DWPF recycle treatment program and the SRS Salt Disposition Alternatives program share many common concerns related to CST slurry transport. Therefore, the DWPF recycle treatment program scope was modified to better address the salt disposition concerns. A test program is in progress to evaluate the physical and chemical compatibility of CST with the operating environment that could be experienced during treatment of the SRS high-level tank waste or DWPF recycle stream, and subsequent handling, storage, and transport of the CST. This report describes the early results of these tests, to provide as much information as possible about CST properties to the Salt Disposition Alternatives Team at SRS. Many of the tests will continue through the next year, to determine the effect of long-term storage on the properties of the CST slurries. These tests provide information that will be used to determine the most appropriate design and mode of operation for a CST ion-exchange system.

The CST will be handled in two different ways following the ion-exchange loading cycle. For supernate treatment, the cesium-loaded CST will be sluiced from the ion-exchange vessels and transferred directly to the DWPF for subsequent blending with the melter feed. For the recycle stream treatment, the loaded CST will be removed from the ion-exchange vessel and added to the existing tank sludges to await transfer to the DWPF melter at a later date. In either case, there are handling issues that must be resolved to evaluate the feasibility of these approaches. If the loaded CST is to be transferred in a cross-site transfer pipeline to a holding area near the DWPF, physical properties such as abrasion, attrition, pressure drop, and viscosity of CST slurries must be investigated. If the loaded CST is to be added to the existing tank sludge following the loading cycle, information regarding the chemical and physical compatibility with the tank sludges must be obtained. This type of information is crucial for making flowsheet selections and equipment specifications for the CST applications. A combination of literature searches, bench-scale testing, and engineering-scale testing is in progress to gather the needed data.

Slurry transfer properties such as pressure drop, viscosity, and attrition during transfer were measured using a continuous-flow system with equipment similar to what is expected in field operations. Erosion or abrasion testing with CST will involve the use of a standard test method performed by a commercial laboratory to determine the need for using abrasion-resistant materials or modifying transfer conditions to reduce particle velocity and impact on internal surfaces of the equipment. Some chemical compatibility information is available from previous CST studies, and this information suggests that there could be some problems with cesium retention by the CST during long-term storage at high temperatures in a waste tank.¹ Bench-scale experiments using loaded CST and supernate simulants are being performed to measure cesium

loss during long-term storage conditions. Physical testing, such as friability in contact with tank waste and the tendency to form hard layers, is being performed using bench-scale equipment.

2. EXPERIMENTAL PROCEDURES AND RESULTS

2.1 TRANSPORT PROPERTIES OF CST SLURRIES

These tests followed the procedures outlined in "*Scoping Test Plan for CST Slurry Testing at Oak Ridge*," as modified by SRS personnel. Properties such as pressure drop, movement of the solids, and attrition were determined by pumping various concentrations of CST in water through a test loop designed for the evaluation of slurry properties. The CST used for all of these tests is the commercial, granular form of the sorbent, IONSIV® IE-911 (UOP Molecular Sieves, Mt. Laurel, New Jersey). The sorbent as received is slightly acidic, so sodium hydroxide was used to stabilize the CST at a pH of 13 before it was used. The CST was also backwashed to remove any fines generated during shipping. An in-line manual sampler (Isolock Model SAA-19-09-44, Bristol Equipment Company, Yorkville, Illinois) was used to verify the suspended solids concentration and density. The sampler is located in a vertical section of 2-in. pipe and uses a hollow tube that extends about one-third of the way into the pipe and then retracts to drain the collected sample into a container. Larger samples for particle size analysis were collected from a low-point drain valve. The test loop was operated using a computer running data acquisition and control software from Intellution, Inc. (Norwood, Massachusetts). Pressure sensors, thermocouples, and magnetic flowmeters were used to monitor the system operation. An M-Point Coriolis mass flowmeter (Endress + Hauser, Greenwood, Indiana) was used to continuously monitor the solids content and density of the slurry. The M-Point meter measures the mass flow and volumetric flow of the slurry and calculates the slurry density from these measurements. It also calculates the solids content using the slurry density determined by the instrument and liquid and particle densities input by the operator. The following equation is used by the instrument:

$$\text{wt \% solids} = D_p(D_s - D_L)/D_s(D_p - D_L) \cdot 100,$$

where D_s = slurry density, D_L = liquid density, and D_p = particle density. This equation becomes very sensitive to variations in the slurry density when the slurry density is close to the liquid density (i.e., at low solids content).

Figure 1 shows a flow diagram for the test loop. Visual observations of the slurries were made in several transparent sections of 2-in. PVC pipe. The first set of tests used a Moyno progressing cavity pump (Model 2L8SSB), with a 5-in.-ID housing and a 7.5-hp motor for circulation. Circulation rates were varied between 10 and 30 gal/min, corresponding to a velocity range of 1 to 3 ft/s for 2-in. pipe and 4 to 12 ft/s for 1-in. pipe. The pressure drop through a vertical section of 1-in. pipe, 10 ft high, was measured at each flow rate. For the second set of tests, a centrifugal pump was installed in the test loop, and all of the 1-in. pipe sections were eliminated (see Fig. 2). A differential pressure gauge was installed in a horizontal section of 2-in. pipe,

Fig. 1. Diagram of the test loop with the Moyno pump.

Fig. 2. Diagram of the test loop with the centrifugal pump.

with an equivalent length of 70 ft (including bends and flanges). The internal volume of the lines in the test loop was 25 gal, which corresponds to a total length of 143 ft.

The slurry test loop was also used to observe the effect of nonroutine activities such as an unplanned shutdown of the pumping system during slurry transfer, where the CST would settle in the pipeline. The ability to resuspend the CST after settling was evaluated by observing pressure drop data and by observing the CST via the transparent sections of pipe. The transparent pipe sections include several 90° elbows, horizontal and vertical spool pieces, and a removable horizontal section of clear pipe.

2.1.1 Moyno Pump Test Results

The test loop was operated with three concentrations of CST in water, nominally 5, 13, and 30 wt %. For each concentration, the test loop was run at flow rates of 30, 20, and 10 gpm, except for the 30 wt % slurry, which was only run at 30 and 20 gpm. The tests at each CST concentration were started at a flow rate of 30 gpm and operated for about 30 min. The pump was turned off for 15 min to allow the CST to settle in the piping and then restarted to determine how well the settled CST was resuspended. The 30 wt % slurry was also allowed to settle for 16 h before the pump was restarted, and then it was recirculated for 7.5 h to determine the CST particle size breakdown during extended recirculation.

With the 5 wt % CST slurry, no stagnant CST particles were visible in any of the clear sections during recirculation at 30 gpm. At 20 and 10 gpm there was a small amount of stagnant CST (~1/2 in. deep) in the outer radius of a 2-in. elbow where the pipe turned from horizontal to vertical. At 10 gpm there was also a small amount in a horizontal section just before the pipe size transition from 2 in. to 1 in. The particle size distribution of the CST after various recirculation times is shown in Fig. 3. The CST samples from the test loop always show an increase in the fraction of particles greater than 425 μm , compared with unused CST. The reason for this slight increase in particle size is not known. The M-Point Coriolis meter showed a solids content of 3–4 wt % for the slurry. The particle density setting in the instruments control system was set at 2.7 g/cm³ during this run but was later changed to 2.3 g/cm³ based on measurements using air-dried CST. Manual calculations show that changing the particle density from 2.7 to 2.3 g/cm³ only makes about a 10% difference in the calculated solids concentration. A graph of the flow rate, solids content, and slurry temperature during the run is shown in Fig. 4.

The solids concentration determined by the M-Point meter oscillated during the 30-gpm test and showed a drop at 20 and 10 gpm, as CST was being deposited in the test loop. These results show that a flow rate of 30 gpm (fluid velocity of 2.9 ft/s) is marginal, at best, for transporting a 5 wt % CST slurry.

Fresh CST was added to the 5 wt % slurry in the test loop to bring the total concentration to 13 wt %. At this concentration, and a flow rate of 30 gpm, there was a small amount of CST stagnant in the 2-in. elbow where the pipe turns from horizontal to vertical, but none visible in the horizontal sections. Following a 15-min shutdown, the settled CST remobilized, except for

Fig. 3. Breakdown of CST particles during recirculation of 5 wt % slurry in test loop using the Moyno pump.

Fig. 4. Test loop data for 5 wt % CST slurry using the Moyno pump.

the stagnant layer in the elbow, without any problems when the pump was started at 30 gpm. At flow rates of 20 and 10 gpm, the 2-in. elbow was about half full of stagnant CST, with small amounts present in the 2-in. horizontal section just before the elbow and in a separate horizontal section, just before a pipe size change from 2 in. to 1 in. The particle size distribution of the CST after various recirculation times is shown in Fig. 5. The water in the test loop was very opaque from the CST fines during this test, which indicated attrition of the CST and made it difficult to distinguish the settled CST heels. The density and solids content readings from the M-Point meter were 1.03–1.04 g/cm³ and 7.0–8.5 wt % solids, respectively. A graph of the flow rate, solids content, and slurry temperature during the run is shown in Fig. 6. At a flow rate of 30 gpm, the solids concentration oscillated, suggesting that the solids were moving through the system in waves, and at the lower flow rates, the solids concentration dropped. A flow rate of 30 gpm was not sufficient to prevent CST deposition in a 2-in. pipeline.

New water and unused CST was added to the test loop to prepare a 30 wt % concentration slurry. SRS personnel were present to observe this test. At a flow rate of 30 gpm, the 2-in. elbow was about one-quarter full of stagnant CST, and at 20 gpm, the elbow was about half full and stagnant CST was visible in several of the horizontal sections. The M-Point Coriolis meter was giving error messages periodically, and the readings were varying wildly. From visual observations, it appeared that the solids were moving through the piping in waves and were not uniformly distributed. The settled CST remobilized easily following a planned 15-min shutdown, except for the stagnant layers noted above, but after a 16-h shutdown it was difficult to get the pump started. The motor controller overloaded several times before the pump shaft started turning. Once the pump started turning, the settled CST in the piping remobilized without any problem. The elbow stayed about half full of stagnant CST during 6 h of running at 30 gpm, indicating that a higher flow rate would be required to keep the CST suspended. The particle size distribution of the CST, after various recirculation times, is shown in Fig. 7.

The frictional pressure drops in the 10-ft section of 1-in. vertical pipe are shown in Fig. 8 for all of the CST slurry concentrations. The frictional pressure drop is calculated by subtracting the static head (120 in. $\times$ density) from the pressure drop readings. For flow rates of 10 to 30 gpm, the nomograph for turbulent flow in Crane² predicts pressure drops ranging from 6 to 47 in. of water for the 5 wt % CST slurry ($\mu=5$ cp and $f=0.019$), while the measured values were 3 to 43 in. of water. We do not currently have a viscosity measurement for the higher concentration CST slurries, so pressure drop predictions for these slurries could not be made. The flow in the 1-in. pipe was turbulent for all of the flow rates tested.

During the following morning, a differential pressure gauge was installed in a horizontal section of 2-in. pipe. The total pipe length between the pressure taps is 64.5 ft, but the equivalent length including bends and flanges is 70 ft, based on correlations in Crane.² Following the recommendations of technical personnel from Endress + Hauser, the time constant in the M-Point meter was changed to one recommended for “dense or viscous” fluids. The test loop was run at a flow rate of 30 gpm for 30 min, and then we started removing CST from the system by draining solution through a 60-mesh screen to recover the CST. A graph of flow rate, solids concentration, pressure drop, and temperature for the period before any CST was removed is

Fig. 5. Breakdown of CST particles during recirculation of 13 wt % slurry in test loop using the Moyno pump.

Fig. 6. Test loop data for 13 wt % CST slurry using the Moyno Pump.

Fig. 7. Breakdown of CST particles during recirculation of 20 wt % slurry in test loop using the Moyno pump.

Fig. 8. Frictional pressure drop for 10-ft vertical section of 1-in. pipe in test loop.

shown in Fig. 9. The solids concentrations from the M-Point meter and from samples (see Table 1) show that the concentration of the slurry was 16–20 wt % solids, rather than the target concentration of 30 wt %. The measured concentrations of CST in the test loop have always been lower than what we thought we were preparing, but the reason for this difference is not known. At several different slurry concentrations, the test loop was run for 30 min, readings from the M-point meter and differential pressure meter were recorded, and a sample of the slurry was taken using the in-line sampler for density and suspended solids measurement. A summary of the results for this run is shown in Table 2. The suspended solids readings from the M-Point meter were generally higher than those for the sample measurements, but the density measurements compared very well. Based on the density and solids concentrations measured for the samples, the particle density of the CST should be about 2.7 g/cm³, rather than the 2.3 and 2.5 g/cm³ measured for air-dried and oven-dried CST, respectively (Sect 2.5.1).

Fig. 9. Test loop data for 20 wt % CST slurry using the Moyno pump.

Table 1. Density and solids concentration measurements

Target solids concentration (wt %)	M-Point Coriolis meter readings		Sample measurements	
	Density (g/cm ³)	Solids (wt %)	Density (g/cm ³)	Solids (wt %)
5	1.02	4	1.01	2.2
15	1.07	12	1.07	10
30	1.16	24	1.16	22

Table 2. Pressure drop, density, and suspended solids concentration measurements for slurries of CST and water in the test loop

Pressure drop at 30 gpm (in. of water)	M-Point Coriolis meter readings		Sample measurements	
	Density (g/cm ³)	Solids (wt %)	Density (g/cm ³)	Solids (wt %)
17–20	1.11–1.12	18–20	1.11	16.4
15–16	1.08	13–14	1.08	10.8
9–10	1.02	3–4	1.01	1.5

Following all of these tests, it was jointly decided by SRS and ORNL personnel that a larger pump was needed in the test loop to achieve higher fluid velocities. A flow rate of 30 gpm did not keep all of the CST moving through the test loop, allowing stagnant pockets in elbows and before pipe size transitions.

2.1.2 Centrifugal Pump Test Results

A centrifugal pump, Defco CYL-TEC (Decatur, Alabama) model D7600 was installed in the test loop to provide higher flow rates (see Fig. 2). The pump has an 8-in.-diam impeller, with ½-in. clearance between the rotor and casing, and a 15-hp motor. An existing variable-frequency motor controller was used to adjust the pump speed. At full speed (60 Hz), the motor is rated for 3450 rpm. Table 3 shows the results of pump tests using water at various motor controller frequencies, and the calculated motor/pump speed and rotor tip speed. The pump could not be run at full speed because the motor controller would shut down from a current overload.

Table 3. Test results for centrifugal pump using water in test loop

Drive frequency (Hz)	Pump speed (rpm)	Water flow rate (gpm)	Impeller tip speed (ft/s)
7	403	11	14
13	748	21	26
18	1035	30	36
24	1380	39	48
31	1783	51	62
37	2128	61	74
43	2473	71	86
48	2760	78	96

Tests of the current showed that one of the power phases was pulling much less than the other two, and the motor was pulling about 7 A at zero speed. These results suggest that the motor controller is going bad. We were able to complete the tests for this program with the existing motor controller, but it will be repaired or replaced in the future.

A 5 wt % CST slurry was prepared and recirculated at 30, 40, 50, and 60 gpm. There was a small amount of stagnant CST visible at 30 gpm, but not at the higher flow rates. At 30 gpm the pressure drop and the solids concentration measured by the M-point meter oscillated (see Fig. 10), indicating that the slurry was not uniformly suspended. At the higher flow rates the readings were steady. For flow rates of 30, 40, and 60 gpm, the fluid velocities in the 2-in. pipe are 2.9, 3.8, and 5.7 ft/s and the Reynolds numbers are 9500, 12,600 and 19,000, so the flow was turbulent for all of the flow rates. Michael Poirier of the Savannah River Site supplied information on predictions of the minimum fluid velocity required to transport slurries. For several different correlations,³⁻⁶ the minimum required velocities ranged from 4 to 5.6 ft/s, which agree fairly well with the 3.8-ft/s value determined experimentally. For flow rates of 30 to 60 gpm, the nomograph for turbulent flow in Crane² predicts pressure drops ranging from 13 to 46 in. of water for the 5 wt % CST slurry ($\mu=5$ cp and $f=0.019$), while the measured values were 15 to 41 in. of water.

Fig. 10. Test loop data for 5 wt % CST slurry using the centrifugal pump.

A 15 wt % CST slurry was prepared using fresh CST and recirculated at 55 and 60 gpm, with no visible stagnant CST. The test loop data is shown in Fig. 11. The M-Point meter gave a solids concentration of 12 wt %. After letting the slurry set stagnant for 11 h, the pump was restarted at 55 gpm with no problem, and all of the settled CST remobilized. The centrifugal pump was very aggressive in breaking up the CST particles, as shown in Fig. 12. After 1 h of recirculation (about 60 times through the pump), almost all of the particles had been reduced to a powder.

With a 30 wt % CST slurry (24 wt % reading on the M-Point meter), we had to increase the maximum allowable current from 16 A (the maximum motor rating) to 20 A to get the pump to run at 44 gpm. The pump was run at 40 gpm for a short time, and at this flow rate there was a small amount of stagnant CST in the elbow that varied with time. At 44 gpm (4.2 ft/s) no stagnant CST was visible. The data from the test loop at 44 gpm (see Fig. 13) shows a corresponding oscillation in the flow rate and in the pressure drop. It is possible that the problems with the motor controller caused periodic variations in the pump speed. There were oscillations in the solids concentration at the beginning and end of the run that also followed the flow rate changes. Figure 14 shows a comparison of CST breakup for the centrifugal pump and for the earlier tests using the Moyno pump. The Moyno pump causes much less damage to the CST particles. A sample of the fine particles ($<150\ \mu\text{m}$) generated using the centrifugal pump were sent the ORNL Analytical Lab for analysis using a particle size analyzer (Malvern Instruments Ltd., Malvern, United Kingdom). Most of the particles ranged from 0.1 to $1.0\ \mu\text{m}$, with an average size of $0.35\ \mu\text{m}$. Table 1 shows a comparison of the density and solids concentration measurement from the M-Point meter and from samples of the slurry.

Fig. 11. Test loop data for 12 wt % CST slurry using the centrifugal pump.

Fig. 12. Breakup of CST particles during recirculation of 12 wt % slurry in test loop using the centrifugal pump.

Fig. 13. Test loop data for 22 wt % CST slurry using the centrifugal pump.

Fig. 14. Comparison of CST breakup using the centrifugal pump at 44 gpm and the Moyno pump at 30 gpm.

The solids concentrations measured in samples and by the M-Point meter have consistently been lower than the target concentrations. The CST particle density calculated from the measured slurry density and solids concentration of the samples is also consistently higher ($\sim 2.7 \text{ g/cm}^3$) than the measured values (2.3 and 2.5 g/cm^3 for air-dried and oven-dried CST, respectively; see Sect 4.A). The reason for these differences is not known at this time.

2.1.3 Mobilization and Rinsing Tests

Several tests were performed to evaluate mobilizing settled heels and plugs of CST in the test loop. At the end of the test with 24 wt % CST slurry, the CST (mostly $<150\text{-}\mu\text{m}$ particle size) was allowed to settle in the test loop and the water was slowly drained off. The test loop was then configured so that water from the feed tank was pumped through the test loop and then into a separate tank (once-through flush). Water was added to the feed tank in 30-gal batches and then pumped through the test loop at 50 gpm. The first batch of water filled the test loop and discharged a few gallons of slurry into the receiving tank. The second flush did not achieve the desired flow rate because a valve at the bottom of the feed tank was left closed for a few seconds after the pump was started, but most of the visible CST was removed. There was not any CST visible in the clear sections of piping, which are fairly near the pump, by the middle of the third flush (two to three line volumes of water). One of the replaceable horizontal spool pieces was packed with used CST (a mixture of fines and full-size particles from the earlier runs using the Moyno pump). The spool piece was full of damp CST for a length of 24 in. When the valves that isolated the spool piece were opened, water from the lines started seeping into the CST plug, displacing some of the CST. The feed tank was filled with 50 gal of water, and the pump was turned on for about 30 s at a speed that would give a flow rate of about 50 gpm. The plugged line was cleared in a few seconds. Figure 15 shows the pump discharge pressure for this test, compared with readings for pumping water and 24 wt % CST slurry with no restrictions in the lines. These results show that the plugged line caused little if any pressure surge before the line cleared.

2.1.4 Once-Through Attrition Test

A test was performed to measure the change in CST particle size as the CST was pumped once through a centrifugal pump and to test mobilization of full-size CST that was deposited in a line. Water was recirculated through the test loop at 50 gpm, 12 kg of CST was added to the feed tank at one time, and a sample of the CST slurry was collected as it passed the clear PVC section of the test loop for the first time. The pump was then stopped, and the CST was allowed to settle in the test loop. The CST sample was dried, sieved, and weighed, and the results are shown in Fig. 16. There was a small amount of fines generated from the CST, but overall there was very little change in the CST particle size. After the CST left in the test loop had settled for 30 min, the system was reconfigured for once-through operation, and the lines were flushed with two 30-gal batches of water at a flow rate of 50 gpm. Most of the CST was removed during the first

Fig. 15. Comparison of pump output pressure for line plugged with CST vs normal start with water and 22 wt % CST slurry. Maximum flow rates were 45 gpm for plug and 22 wt % slurry, and 58 gpm for water.

Fig. 16. Breakup of CST sorbent pumped once through the centrifugal pump.

flush, and the water in the clear PVC sections of the loop was clean by the early part of the second flush (about two line volumes of water). These tests have shown that it is very easy to mobilize and remove settled CST particles or fines from pipelines and to remove plugs or blockages of CST.

2.2 SLUDGE AND SUPERNATE SIMULANT COMPOSITIONS

Nonradioactive, nonhazardous simulants of the sludges and supernates in the SRS waste tanks were developed for this test program. The formulations were reviewed and approved by SRS representatives. Three supernate compositions were prepared, representing high, median, and low concentrations of the main components (NaNO_3 , NaNO_2 , Na_2SO_4 , NaOH , and KNO_3). The simulant sludges were based on Sludge D (HM LAW) in the sludge compositions table provided to ORNL by SRS personnel. Trace hazardous metals, such as mercury and nickel, were not used, and lanthanum oxide was used as a simulant for uranyl hydroxide. The sludge formulations were prepared with and without chabazite zeolite, which is likely to have major effects on agglomeration and abrasivity measurements. Without the zeolite, the sludge has a composition similar to Sludge A (Purex HAW), so a wide range of SRS sludge compositions was covered by these simulants. The sludge and supernate compositions are shown in Tables 4 and 5, respectively.

Table 4. Supernate simulant compositions

Component	Low	Median	High
NaNO_3	0.065 <i>M</i>	1.2 <i>M</i>	
NaNO_2	0.12 <i>M</i>	0.71 <i>M</i>	
Na_2SO_4		0.17 <i>M</i>	
NaOH	0.01 <i>M</i>	2.9 <i>M</i>	13.5 <i>M</i>
KNO_3	52 mg/L	0.015 <i>M</i>	

Table 5. Sludge simulant compositions

Component	Sludge 1 (with zeolite)		Sludge 2 (with zeolite)	
	Amount		Amount	
	(g)	Wt %	(g)	Wt %
FeO(OH)	29.5	34.3	29.5	46.8
Al(OH) ₃	16.0	18.6	16.0	25.4
MnO ₂	9.0	10.5	9.0	14.3
La ₂ O ₃	4.0	4.6	4.0	6.3
CaCO ₃	3.5	4.1	3.5	5.6
Ce(OH) ₃	1.0	1.2	1.0	1.6
Zeolite	23.0	26.7	0	0
Totals	86.0	100	63.0	100

The CST used for all of these tests is the commercial, granular form of the sorbent IONSIV IE-911 (UOP Molecular Sieves, Mt. Laurel, New Jersey). The sorbent as received is slightly acidic, so sodium hydroxide was used to stabilize the CST at a pH of 13 before it was used. The CST was also backwashed to remove any fines generated during shipping.

The amorphous ferric oxyhydroxide and manganese dioxide needed for the sludge simulant was prepared by mixing solutions of ferrous sulfate and potassium permanganate, which react to form the desired sludge components following pH adjustment to above 7. The procedure for preparing a large batch of sludge is as follows.

1. Dissolve 6.5 kg of Fe(SO₄)·6H₂O in 50 L of water.
2. Dissolve 1.14 kg of KMnO₄ in 50 L of water.
3. Slowly add the KMnO₄ solution to the Fe(SO₄) solution, while stirring vigorously.

5. Add 1.21 kg of $\text{Al}(\text{OH})_3$, 0.28 kg of La_2O_3 , 0.25 kg of CaCO_3 and 0.16 kg of $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$. The $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ precipitates to form the desired $\text{Ce}(\text{OH})_3$.

This yields about 40 L of settled sludge. In order to adjust the supernate to the composition of the median supernate listed in Table 4, add 11.2 kg of NaNO_3 , 5.36 kg of NaNO_2 , and 20 L of 50 wt % NaOH solution. The potassium and sulfate needed for the supernate are already present from the $\text{Fe}(\text{SO}_4)_2$ and KMnO_4 used to prepare the sludge. After settling, the clear supernate is pumped off, and the settled sludge is ready to be used for the caking and abrasivity tests. Zeolite and/or CST are added separately as needed to individual containers of sludge.

2.3 EROSION/ABRASION TESTING OF SLUDGE/CST MIXTURES

Erosion or abrasion potential for the CST and other sludge components is being evaluated using method ASTM G75-95 "Determination of Slurry Abrasivity (Miller Number) and Slurry Abrasion Response of Materials (SAR Number)." Hydril Company (Humble, Texas) performed the SAR tests, which measure the loss of material from standard metal wear blocks at a specified time on the curve of cumulative material loss (abrasion plus corrosion) vs. time. ASTM G75-95 states that slurries with an SAR number of about 50 or lower can be pumped with only minor abrasive damage to the system. Six different slurry compositions were prepared at ORNL and shipped to Hydril for testing (CST in water, CST in median supernate, sludge 1, sludge 1 + CST, sludge 2, and sludge 2 + CST). Wear blocks of two different metals (304L stainless steel and ASTM A106 carbon steel) were purchased from Metal Samples Company (Munford, Alabama) for these tests, which were performed at 80°C. The test measures the weight loss from the metal specimens every 2 h, for a total of 6 h, as they are driven in a reciprocating motion along the neoprene bottom of a tray containing the test slurry with a standard force applied to the metal specimens.

The results of the tests are summarized in Table 6. Jim Miller of Hydril Company stated that slurries with SAR numbers below 10 are considered to have low abrasivity, while values of 10–50 are considered moderate. The higher value for A106 in water vs. supernate suggests that oxidative corrosion was contributing to the material loss in the CST/water slurry. The supernate has a high pH and contains sodium nitrite, both of which reduce the corrosion rate of mild steel. These results suggest that CST slurries will cause little if any damage to pumps and piping system, as long as chemical corrosion is controlled. The results for sludge 2 + CST were unexpectedly high, particularly for the stainless steel, and the weight loss for the metal specimens was inconsistent during the tests. This type of result can be caused by the wear blocks coming loose or being misaligned, or by the slurry drying out during the test. The reason for the inconsistent results in these two tests is not known. The tests were not repeated due to time constraints and the limited applicability of the results to the currently planned operating modes.

Table 6. SAR number for CST slurries in water and median supernate

Slurry	A106 carbon steel	304L stainless steel
CST in water	102	13
CST in supernate	2.1	2.5
Sludge 1	31	13
Sludge 1 + CST	26	18
Sludge 2	3.2	3.1
Sludge 2 + CST	28	31

2.4 CHEMICAL LEACHABILITY TESTS

CST has been used to remove cesium from a wide range of different solutions, including high-salt basic,^{1,7} high-salt acidic,^{2,8} and neutral groundwater and process wastewaters.⁹ These results illustrate the chemical stability and cesium affinity of CST; however, chemical stability tests have shown that CST partially converts to zeolite when stored at 95°C in high-salt, high-pH solutions for three months, but is stable when stored at 60°C.¹ This crystal change could result in previously loaded cesium being released back into solution. The ability of CST to retain loaded cesium while in contact with SRS supernates was determined by performing bench-scale simulant tests using CST samples pre-loaded with stable cesium and radioactive cesium tracer.

About 10 g of pretreated CST was added to each of 12 250-mL glass bottles. Samples of the three supernate simulants were spiked with 50 mg/L Cs and 6 $\mu\text{Ci/L}$ ^{137}Cs and then each simulant (200 mL) was added to four of the bottles. The 50-mg/L Cs concentration was chosen based on loading data, obtained from D. J. McCabe of Savannah River Technology Center, using an average SRS supernate. All of the bottles were placed on rockers for four days to contact the CST with the simulant solutions. A sample of solution from each bottle was filtered (0.2- μm pore size syringe filters) and then counted using a shielded germanium detector. Three background counts and two samples of the starting solution for each supernate concentration were counted each time a set of samples was counted. The samples were returned to the

appropriate bottle after counting. The bottles were stored without agitation for three days at room temperature, while an oven was installed in the hood, and then samples were counted again. Two bottles of each solution were then placed in an oven at 80°C, with the other two bottles stored at room temperature. Samples were counted after various storage times, ranging from 7 to 105 days, at the appropriate temperature.

The cesium concentrations left in the supernate, and the K_d (cesium on CST/Cs in supernate), are shown in Table 7 for the loading phase of the test. Table 8 shows the cesium concentrations in the supernates after storage for up to 105 days at room temperature or 80°C. The conditions of this experiment exceeded UOP's recommended maximum temperature (60°C) and represent a worst-case scenario for storage of cesium-loaded CST in the Savannah River Site tank farm.

Table 7. Cesium loading results for CST in supernate simulants

Bottle no.	Supernate conc.	CST weight (g)	<u>After 4 days rocking</u>		<u>After 3 days left stagnant</u>	
			[Cs] (mg/L)	K_d (L/kg)	[Cs] (mg/L)	K_d (L/kg)
1	Low	10.20	0.018	53100	0.044	22510
2	Low	10.57	0.023	40260	0.034	27810
3	Low	10.18	0.012	80730	0.030	32810
4	Low	10.20	0.029	34350	0.029	34310
5	Median	10.20	0.869	1109	0.556	1744
6	Median	10.25	1.068	894	0.599	1610
7	Median	10.31	1.247	758	0.745	1283
8	Median	10.16	0.479	2033	0.346	2824
9	High	10.01	1.069	915	1.16	839
10	High	10.30	7.182	116	6.294	135
11	High	10.10	1.055	918	0.989	981
12	High	10.05	0.962	1014	1.190	816

Table 8. Cesium leaching results for loaded CST in supernate simulants

Bottle no.	Supernate conc.	Temp. (°C)	Cesium concentration (mg/L) after storage for					
			7 days	14 days	21 days	28 days	47 days	105 days
1	Low	25	0.024	0.000	0.001	0.004	0.002	0.003
2	Low	25	0.095	0.015	0.006	0.001	0.005	0.000
3	Low	80	0.023	0.005	0.005	0.014	0.004	0.019
4	Low	80	0.032	0.000	0.010	0.020	0.007	0.014
5	Median	25	0.482	0.421	0.384	0.458	0.455	0.557
6	Median	25	0.429	0.476	0.394	0.422	0.412	0.506
7	Median	80	3.635	3.770	3.851	4.861	4.309	6.361
8	Median	80	3.711	3.521	4.342	4.869	3.667	5.921
9	High	25	1.023	1.024	0.808	0.775	0.671	0.681
10	High	25	4.860	4.556	3.628	2.913	0.732	0.710
11	High	80	3.902	5.436	6.865	8.934	8.989	15.356
12	High	80	5.171	5.299	7.247	7.512	4.675	11.372

The cesium concentrations in the supernate samples were calculated from the ratio of net counts of the sample divided by the average of the two starting solutions for that supernate composition. The samples were fairly difficult to filter, because of the CST fines in the solutions. Other tests have shown that these fines can be very small, so isolated cases of high ^{137}Cs concentrations could be caused by a few fines getting through the filter. It appears that bottle no. 10 was not agitated properly during the loading phase, since the cesium concentration in the liquid was much higher than in the comparable bottles. This concentration was slowly reduced during storage.

There was no leaching of cesium in any of the solutions stored at room temperature, but at 80°C the median supernate has leached 11% of the cesium originally loaded on the CST, and the high concentration supernate has leached 29% after 105 days. The cesium concentration in the samples kept at 80°C had appeared to level off, with concentrations of about 4 mg/L and 9 mg/L in the median- and high-concentration simulants, respectively, during 27 to 47 days of storage, but the concentrations increased significantly in the next samples, taken after 105 days of storage.

In order to determine if the leaching was caused by an equilibrium shift at high temperature or irreversible changes in the CST, one bottle each of the high- and median-concentration supernate was removed from the oven after 34 days at 80°C and rocked for four days at room temperature. The CST in each supernate reloaded part of the previously leached cesium but left about 1.6 mg/L cesium in solution. The experiment was repeated after 70 days storage, with the bottles being rocked for seven days at room temperature. The CST in the median supernate reloaded most, but not all, of the cesium, showing that an equilibrium shift at high temperature was responsible for most of the cesium leaching. The CST in the high concentration supernate did not reload any of the previously leached cesium, so it appears that a physical change is occurring in this CST.

There is a lot of scatter in the low concentration supernate samples, because of the very low concentration of ^{137}Cs in the solutions (the count rates are just barely above background), but there is no evidence of cesium leaching from the CST at either temperature in the low concentration supernate.

The CST samples were loaded with cesium under equilibrium conditions with the supernate simulants. If the CST is loaded with cesium under one set of conditions, and added to supernate that has a different composition, the tendency for cesium to elute or adsorb may change as the loaded CST and supernate mixture reaches a new equilibrium. Additional tests will be performed in FY 1999 (as part of the Salt Disposition Alternates Studies) to evaluate these scenarios and to further evaluate the effect of temperature and supernate composition.

2.5 IN-TANK SETTLING PROPERTIES, ATTRITION, AND AGGLOMERATION (CAKING) TESTS

These tests are being performed using bench-scale equipment, samples of the engineered form of CST (IONSIV7 IE-911 from UOP Molecular Sieves Corp.), and simulated SRS sludge and supernate.

2.5.1 Density

The density of dry as-received and pretreated and damp pretreated CST was measured by weighing settled (by tapping) volumes of the sorbent. The density of CST/water slurries was determined by

mixing various ratios of water and dry, pretreated CST, letting set overnight to wet the interior of the CST particles, removing the air bubbles from the sorbent bed, and measuring the volume of the slurry. The particle density was determined by plotting 1/density vs. wt % CST and extrapolating to 100 wt % CST. The highest concentration of CST in water that produced a slurry was 60 wt %.

The bulk density of air-dried CST was measured as 1.05–1.10 g/cm³ for both as-received and pretreated (backwashed and pH adjusted) CST. The density of damp CST, with all free water removed, was 1.53–1.59 g/cm³. The measured densities for slurries of air-dried CST and water are shown in Table 9, and the particle density derived from these measurements is 2.3 g/cm³.

Table 9. Measured densities for CST/water slurries

wt % CST	5	15	30	40	50	60
Density, g/mL	1.03	1.09	1.20	1.29	1.39	1.52

The particle density of the CST was also measured by using Hubbard Specific Gravity Bottles (also known as pycnometers). A volume of CST was weighed and placed in the bottles, and the amount of water displaced by the CST was measured after all the air in the CST had been displaced. These measurements gave a particle density of 2.26 g/cm³ for air-dried CST and 2.50 g/cm³ for CST dried at 80°C.

2.5.2 In-Tank Caking Tendency

The purpose of these tests is to determine if the CST sorbent will form hard layers, either by itself or through interaction with the zeolite or other components of the sludge. The initial shear strength and then viscosity vs. shear rate have been measured for samples of sludge, and CST that were stored for up to 90 days at either room temperature or 80°C. The initial-shear-strength test measures the tendency of the CST and sludge to cake, and the viscosity measurements provide data needed to estimate pumping requirements to transport the sludges.

A batch of settled sludge simulant was prepared as described above in Sect. 2.2 (sludge 2). The sludge was pumped into a series of 500-mL glass bottles, with each bottle containing about 400 mL of sludge (~70 g of dry sludge). Chabazite zeolite (20 g) was added to half the bottles to produce the sludge 1 composition (Table 5), and 40 g of pretreated CST was added to half of the sludge 1 and sludge 2 bottles. A total of 22 bottles of each of the four sludge types was prepared, and 11 of each were placed in an oven at 80°C, with the rest stored at room temperature. Samples of each sludge type, stored at both temperatures, were submitted to the ORNL Analytical Laboratory for analysis after storage times of 1, 14, 21, and 90 days. Newly prepared

samples of CST, zeolite, silica sand, and kaolin clay in median supernate were also submitted for comparison of initial shear strength.

The samples of CST, zeolite, sand, and clay were all too hard for measurement of initial shear strength using the Rotovisco RV30 rheometer (Haake, USA, Paramus, New Jersey) available at the ORNL Analytical Laboratory. The initial shear strength results for the sludge samples are shown below. For these measurements, the sludge completely covered the shear vane rotor, but the zeolite and/or CST, which settles to the bottom of the sludge layer, only covered a small portion of the rotor, varying with the amount added to the bottles. The bottles with CST plus zeolite would cover the largest part of the rotor, followed by CST only and then by zeolite only. The initial shear strength would increase as the amount of solids contacting the rotor increased, even if the actual shear strength of the solids was the same. The samples were then stirred to homogenize the sludge, and the apparent viscosity was measured.

There was a lot of scatter in the viscosity measurements, because of the large CST and zeolite particles, which were difficult to keep suspended. The viscosity numbers in Table 10 are an average of the values from the increasing and decreasing shear rate measurements.

The samples stored for 14, 21, and 90 days generally showed significantly higher values for the initial shear strength and viscosity than the 1-day samples. The sludge samples that contained CST usually showed the highest values for initial shear strength, especially those stored at 80°C. The 14-, 21-, and 90-day samples showed Bingham Plastic-type behavior, with the apparent viscosity increasing rapidly at the lower shear rates.

The Analytical Laboratory also attempted to measure the viscosity of 5, 15, and 30 wt % slurries of CST in water. They were unable to keep the 15 and 30 wt % slurries suspended in the viscometer, so the viscosities could not be measured. The 5 wt % slurry had a constant viscosity of 5 cp at shear rates from 50 to 250 s⁻¹. A sample of the 30 wt % CST slurry that was recirculated through the test loop with the centrifugal pump, and was therefore mostly fines, showed a constant viscosity of 5.5 cp.

2.5.3 In-Tank Attrition

UOP has performed some mechanical attrition tests by mixing CST/water slurries in a paint shaker for 1 h at room temperature. Attrition losses for this test are typically 2 to 5 wt %, which is similar to results for UOP's other inorganic sorbents. Previous chemical stability tests have shown that the CST powder is stable in most solutions, although strong nitric acid will dissolve some material, and milder acids and high-pH, high-salt solutions will change the crystal structure after extended time periods at elevated temperatures.¹ The extent of possible attrition of the granular CST due to chemical interactions with SRS supernates was determined by contacting CST samples with the three supernate simulant compositions at room temperature and at 80°C for specific time periods. The samples were then analyzed to determine the particle size distribution.

Table 10. Initial shear strength and viscosity measurements of sludge samples

Sample Description	Initial Shear	Viscosity (mPa · s = cp) at shear rate of		
	Strength (Pa)	50 s ⁻¹	150 s ⁻¹	250 s ⁻¹
Sludge, 25°C				
1 day	0.06	6	7	8
14 days	0.16	74	37	28
3 months	0.05	42	22	19
Sludge, 80°C				
1 day	0.14	4	6	8
14 days	0.22	101	44	33
3 months	>0.5	41	22	18
Sludge + CST, 25°C				
1 day	0.18	15	12	16
14 days	0.31	71	35	26
21 days	0.26	73	36	27
3 months	0.13	74	34	26
Sludge + CST, 80°C				
1 day	0.17	8	10	12
14 days	>0.5	106	46	34
21 days	0.48	115	52	36
3 months	>0.5	33	23	21
Sludge + zeolite, 25°C				
1 day	0.11	2	6	8
14 days	0.20	68	33	25
21 days	0.30	70	33	26
3 months	0.19	67	32	24
Sludge + zeolite, 80°C				
1 day	0.13	8	9	11
14 days	0.24	120	50	40
21 days	0.17	85	42	30
3 months	0.33	43	31	28
Sludge+zeolite+CST, 25°C				
1 day	0.34	3	6	8
14 days	0.44	170	80	60
21 days	>0.5	75	35	27
3 months	0.18	31	20	18
Sludge+zeolite+CST, 80°C				
1 day	0.23	10	9	12
14 days	0.25	88	43	33
21 days	0.49	93	44	24
3 months	>0.5	9	9	13

About 5 g of pretreated CST was added to each of 66 glass vials. Low-, median-, and high concentration supernate simulants (Table 4) were added to a third of the vials each. Half of the vials were stored in an oven at 80°C, with the other half stored at room temperature. After storage for 7, 14 and 21 days, one sample of each type was submitted to the ORNL Analytical Laboratory for particle size analysis using a Mastersizer Particle Size Analyzer (Malvern Instruments Ltd., Malvern, United Kingdom).

The results after 7 days show a small decrease in the particle size for all of the samples stored at 80°C (see Table 11 and Fig. 17). Unused CST showed an average size of 441 μm . There was not a significant difference caused by the three supernate compositions, except for the high concentration supernate at 80°C, which caused a slight increase in size compared with the other supernate compositions at the same temperature. After storage for 14 and 21 days, there was more scatter in the data and no clear trends (see Table 11 and Fig. 18). There does not appear to be a significant breakdown of the CST particles caused by any of the supernate simulants.

Table 11. Average particle size of CST sorbent stored in various supernate simulants

Supernate/temperature	Average (Mode 1) particle size (μm)		
	7 days	14 days	21 days
Low, 25°C	457	440	405
Low, 80°C	358	368	366
Median, 25°C	464	423	422
Median, 80°C	357	436	421
High, 25°C	486	465	439
High, 80°C	421	376	394

2.5.4 In-Column Physical Properties

The purpose of these tests is to compare the rheological properties of new and used CST and identify any potential problems with sluicing CST from columns. Samples of used CST slurry, which was recirculated in the test loop using the Moyno pump (see Section 2.1.1), were added to a series of 500-mL glass bottles. Similar bottles were prepared using new, pretreated CST. The particle size distribution of the used CST showed 21 wt % >250 μm , 17 wt % 150–250 μm , and 62 wt % <150 μm , while the new CST is 99.9 wt % > 250 μm . Each bottle contained about 100 g CST and 200 mL water. Half of the bottles were stored at room temperature, and the other half were stored at 80°C.

Fig. 17. Effect of storage for 7 days in low (L), median (M), or high (H) concentration supernate simulants at 25°C or 80°C on CST particle size.

Fig. 18. Effect of storage for 14 days in low (L), median (M), or high (H) concentration supernate simulants at 25°C or 80°C on CST particle size.

The bottles of CST, zeolite, and clay that were prepared for the In-Tank Caking Tendency Tests (Section 2.5.2) showed that the initial shear strength of all of these slurries was above the range that could be measured with the available viscometer. A penetrometer, which is normally used to measure the strength of wet grout samples, was used to measure the caking tendency of the CST slurries, after storage times of one and four months. Samples of zeolite and kaolin clay in water were also measured for comparison. The penetrometer measures the amount of force required to push a cylindrical probe into the settled solids. The results are shown in Table 12.

The power level required to stir the settled solids was measured using a LabMaster mixer (Lightnin, Rochester, New York) with an R-100, high-shear, 2.0-in.-OD impeller. The mixer measures the additional power required to stir the samples, compared with the power required to turn the impeller in air. These results are also shown in Table 12.

Table 12. Penetrometer and mixer test results for samples of CST, zeolite, and clay

Sample description	Storage time		
	One month	Four months	Four months
	penetration pressure (psi)	penetration pressure (psi)	penetration pressure (watts)
New CST, 80°C	<5	<5	0.3
Used CST, 25°C	<5	11	0.2
Used CST, 80°C	<5	11	0.4
Zeolite, 25°C	<5	56	0.7
Kaolin Clay, 25°C	<5	16	0.5

Two small columns (2-in I.D.) were filled with new or used CST, backwashed, and then stored stagnant for a month. Each column was then backwashed with water (30 mL/min), and the pressure drop was measured. There was no measurable difference between the two columns, with each showing a pressure drop of 2 psi, and each bed fluidized almost immediately. These results show that there should not be any problem sluicing CST from columns, even if the CST is left in the column for extended periods of time.

3. CONCLUSIONS

Pumping tests in a recirculating loop have shown that a 24 wt % slurry of CST in water can be transported at fluid velocities of 4.3 ft/s (45 gpm in a 2-in. pipe) with no visible settling of the CST particles, while a 5 wt % slurry will stay suspended at a velocity of 3.8 ft/s. The CST was easily mobilized after purposely plugging sections of pipe. The CST particles were rapidly broken up by a centrifugal pump into very small particles ($<150\ \mu\text{m}$, with the majority being $<1\ \mu\text{m}$). A progressing cavity (Moyno) pump caused less damage to the CST particles.

Samples of cesium-loaded CST stored in median- and high-concentration supernate simulants at 80°C have leached cesium into the supernate. There was no measurable leaching in any of the simulants at 25°C , or in the low-concentration simulant at either temperature.

Slurries of CST in water showed low abrasivity to 304L stainless steel and moderate abrasivity to A106 carbon steel. A slurry of CST in median supernate (2.9 M NaOH plus other salts) showed low abrasivity to A106 steel, so the higher abrasivity result for CST in water was caused by oxidative corrosion. Sludge simulant slurries showed low abrasivity, but mixtures of sludge and zeolite showed moderate abrasivity.

Mixtures of CST in SRS sludge simulants have shown minimal tendency to cause caking or hard layers. Supernate simulants that cover the range of solutions in the SRS high-level waste storage tanks have shown minimal effect on the particle size of the CST sorbent.

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