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Aluminosilicate Formation in High Level Waste Evaporators: A Mechanism for Uranium Accumulation

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ABSTRACT

High level waste Evaporators at the Savannah River Site (SRS) process radioactive waste to concentrate supernate and thus conserve tank space. In June of 1997, difficulty in evaporator operation was initially observed. This operational difficulty evidenced itself as a plugging of the evaporator's gravity drain line (GDL). The material blocking the GDL was determined to be a sodium aluminosilicate. Following a mechanical cleaning of the GDL, the evaporator was returned to service until October 1999. At this time massive deposits were discovered in the evaporator pot. These deposits were also determined to be sodium aluminosilicate. These aluminosilicate deposits were also found to include ~ 7 wt % uranium of which about 3 % was ^{235}U and absent of neutron poisons. Historically, across the tank farms, uranium solids are intermixed with iron and manganese in sufficient ratios in sludge to protect against nuclear criticality. The only aspect to evaporator operation was uranium was a source-term consideration. This new accumulation mechanism for uranium in the SRS High Level Waste system in the absence of sufficient neutron poisons is a new discovery.

As a result of the changes in evaporator chemistry and the resulting formation of aluminosilicate deposits in the evaporator, a comprehensive research and development program has been undertaken. This program is underway in order to assist in understanding the new evaporator chemistry and gain insight into the deposition phenomena. This effort involves three Department of Energy research institutions (SRTC, ORNL, and PNNL). This multi-site team has been examining the nucleation and growth kinetics of aluminosilicate formation, thermodynamic modeling, and evaluation of potential mitigation strategies such as feed aging prior to introduction to the evaporator, crystal inhibition additives, and mixing, among others.

Key results from testing in FY01 have demonstrated that the chemistry of the evaporator feed favors aluminosilicate formation. Both the reaction kinetics and particle growth of the aluminosilicate material under SRS evaporator conditions has been demonstrated to occur within the residence times utilized in the SRS evaporator operation. Batch and continuous-flow experiments at known levels of supersaturation have shown a significant correlation between the deposition of aluminosilicates and mixing intensity in the vessel. Advances in thermodynamic modeling of the evaporator chemistry have been accomplished. The resulting thermodynamic model has been related to the operational history of the evaporator, is currently assisting in feed selection, and could potentially assist in expanding the operating envelopes technical baselines for evaporator operation.

INTRODUCTION

Liquid high level waste is stored in underground storage tanks at the Savannah River Site. Currently, the Defense Waste Processing Facility (DWPF) is vitrifying the sludge portions of the wastes in borosilicate glass. Operation of the DWPF is crucial to meet regulatory agreements with the state of South Carolina and the federal Environmental Protection Agency.

During the operation of DWPF condensate water from the slurry receipt and adjustment tank (SRAT) and the slurry mix evaporator (SME) are collected along with laboratory wastes in the recycle condensate tank (RCT). The slightly acidic waste is neutralized to 1 M free hydroxide and discharged to the SRS H-area tank farm (Tank 43H). Silica-based frit is entrained in each of the streams feeding the RCT and is dissolved during the caustic adjustment.

The DWPF recycle water is discharged to Tank 43H, aggregated with typical canyon waste and evaporated using the 242-16H (2H) evaporator. Problems with chemical incompatibility between the silicon-containing DWPF recycle and the aluminum-containing canyon waste initially manifested as an aluminosilicate deposit in the exit line from the evaporator, i.e., gravity drain line, in July 1997 (1). These aluminosilicate deposits were removed with high-pressure water. Frequent video inspections showed improved evaporator operation occurred during 1998. However, significant aluminosilicate scale build-up in the evaporator pot was discovered in October 1999 (2). These deposits included a copious quantity of fuel-grade uranium. This uranium accumulation had not previously been identified nor analyzed for safety implications. Scale of this nature had previously been observed in the aluminum refining industry and in the pulp and paper manufacturing industry (3, 4, 5, and 6). The formation of aluminosilicates had previously been studied for Hanford high level waste (7).

Therefore, a multi-site team from the across the Department of Energy complex was assembled to investigate aluminosilicate scale formation under SRS evaporator chemistry conditions. This team consisting of personnel from the Savannah River Technology Center (SRTC), Oak Ridge National Laboratory (ORNL), and the Pacific Northwest National Laboratory (PNNL). A near-term Research and Technology (R&T) roadmap was defined, funded and executed during FY 01. Areas of specific research included reaction kinetics, use of inhibiting agents for deposition and particle growth, role of hydrodynamics in deposition location and rate of growth, and thermodynamic modeling for use as a predictive process control strategy for continued operation of the other SRS evaporators.

EXPERIMENTAL DETAILS

Testing was preformed at the various DOE facilities under a variety of experimental conditions. However, a base simulant was developed that was simple yet resembled the conditions in the feed tank (Tank 43H) of the 2H evaporator system. Wilmarth had analyzed (8) the samples from the feed tank and found the tank chemistry was stratified. However, normalizing the anions to a specific sodium ion concentration gave rise to set of standard concentrations. Therefore, work performed used a simulant that was 6 *M* in sodium, 4 *M* free hydroxide and 1*M* in both nitrate and nitrite.

Kinetics, Formation and Inhibitor Studies

In the kinetics tests performed at Oak Ridge, all tests were performed in a constant-temperature water bath for low temperature tests (40, 60, and 80 °C) and an oil bath for 100 °C testing. A Lightnin mixer was used to control the high-density polyethylene (HPDE)-coated stirrer. Reaction vessels were made of polished 304-L stainless steel. The formation and inhibitor tests at SRTC were performed in Teflon® bottles. The bottles were mounted in a temperature-controlled orbital shaker and agitated at a rotational speed of 150 rpm. Periodically samples were removed and filtered at elevated temperature. Silicon, sodium and aluminum concentrations were measured by inductively coupled plasma – emission spectroscopy. Researchers at PNNL, also, performed aluminosilicate formation testing in similar methods.

Thermodynamic Modeling and Confirmatory Testing

Two different computer models were applied to the solution chemistry of the 2H Evaporator. The first was SOLGASMIX (9), an Oak Ridge National Laboratory product. This program uses the Pitzer method to calculate activity coefficients. The code calculates phase equilibrium by minimizing total free energy. The second model is a commercially-available product, Geochemist Workbench (10). This application is particularly well suited to the thermodynamic calculations related to the SRS or Hanford evaporators. The GWB has the ability to estimate activity coefficients for high ionic strength solutions such as those in the evaporator. Additionally, GWB includes usage of Lawrence Livermore National Laboratory (LLNL) extensive database for minerals and aqueous species used to model the performance of waste forms in the High Level Waste (HLW) Repository and includes the ability to calculate the relative stability of multiple solid phases simultaneously.

Confirmatory testing is conducted by personnel at the Pacific Northwest National Laboratory. The goal of this work is to react solution of known composition, i.e., silicon and aluminum activities, in Savannah River waste simulants to validate the phase boundaries predicted by the various modeling programs. Additionally, the work established the movement of the phase boundaries as a function of temperature and reaction time. The work consists of bench-scale reaction vessels heated to temperatures as high as 130 °C.

Deposition Testing

A powerful technique to monitor the dynamics of solid phase formation in bulk solutions is light scattering. In these studies, a specially constructed, *real-time* dynamic laser light scattering (DLS) setup at ORNL was used to study the solid particle evolution and growth (typically from 10 to 1000 nm). A synthesis solution is placed in a thermally controlled cell, allowing particle growth to be observed in situ during heating of the precursor solutions. A 10-mW He-Ne laser (632.8 nm) generates a beam that is directed through the liquid sample. The scattered light provides information on the Brownian motion of the nanoparticles in the liquid, which allows a calculation of the average particle size by applying the Stokes-Einstein equation.

Batch aluminosilicate deposition tests were conducted using the above formulation for simulated evaporator feed. These tests were performed on a 200-mL scale at 80 °C. A water bath was used to provide a constant reaction temperature for the stainless steel beaker that served as the reaction/deposition chamber. The total time in the stainless steel beaker was 2 hours. A new beaker was used for each experiment and was pre-weighed prior to starting of the experiment. At the completion of the test the slurry was decanted, filtered, dried, and weighed to determine the fraction of solids that did not adhere to the beaker. The weight of solids adhering to the metal beaker was determined by rinsing the beaker and heating to dryness. Continuous aluminosilicate deposition tests were conducted using the above formulation for simulated evaporator feed. These tests were performed on a nominal 200mL scale at 80°C for three residence times given a fixed feed rate into the simulated evaporator. The reaction vessel included an overflow system to remove excess solution. The overflow consisted of a stainless steel "T" that was welded to the wall of the vessel to allow for effluent to be drawn from under the surface. For this phase of the work, the primary scenarios evaluated were the effects of mixing and seeding on the deposition with each condition being run in triplicate. Two response variables were measured. These were the amount of solids adhered to vessel as a deposit and amount of solids suspended in solution and passed through the vessel. Three mixing conditions were evaluated. These were the unmixed, low-mixing and high-mixing. The low-mixing condition utilized 90 RPM that corresponds to an energy dissipation rate of <0.2 W/kg of solution. The high-mixing condition was 250 RPM that corresponds to an energy dissipation rate of 0.4-0.8W/kg of solution.

RESULTS AND DISCUSSION

Kinetics, Formation and Inhibitor Testing

Mattus, et al. (11) studied the second order reaction kinetics for the disappearance of silicon and aluminum from solution during formation of aluminosilicate solid phases. Previously studies at SRTC using pseudo first order approximations had found the reaction to be fast at evaporator temperatures and measured an activation energy of 30 kJ/mol. Mattus started with equal molar Al: Si ratios and studies the kinetics at 40, 60, 80, and 100°C. The solution phase concentration data was regressed for each reaction temperature and fitted to normal second order kinetic expressions as shown in Figure 1 for the 100 °C test. Second order rate constants of 0.0014, 0.0022, and 0.0053 L mol^{-1} s^{-1} were obtained for 60, 80, and 100 °C. An Arrhenius fit of this data gave an activation energy of 34 kJ/mol.

Several variables have the potential to affect the amount and type of precipitate formed by the reaction of aluminate and silicate ions in highly caustic media. Therefore, we examined the effect of four primary variables in a reduced matrix statistical design. The design is a three level screening factorial design. The variables were sodium (reflective of ionic strength) aluminum, silicon, and hold time. All testing was at ambient temperature prior to heating at elevated temperature (80 °C). The results of the statistical modeling (12) from the formation tests show that four terms incorporating the experimental variables proved to be statistically significant, i.e., have the value of Prob > |t| less than 0.05. These variables were the aluminum concentration, the time the solution was held at ambient temperature, quadratic term of silicon concentration and the quadratic term of the hold time. Interestingly, the silicon concentration was not statistically significant other than the quadratic term. However, with the limited number of experimental observations, we may not be able to discriminate between the silicon terms. Another key observation is that the sodium ion concentration did not have an influence over the formation of aluminosilicate.

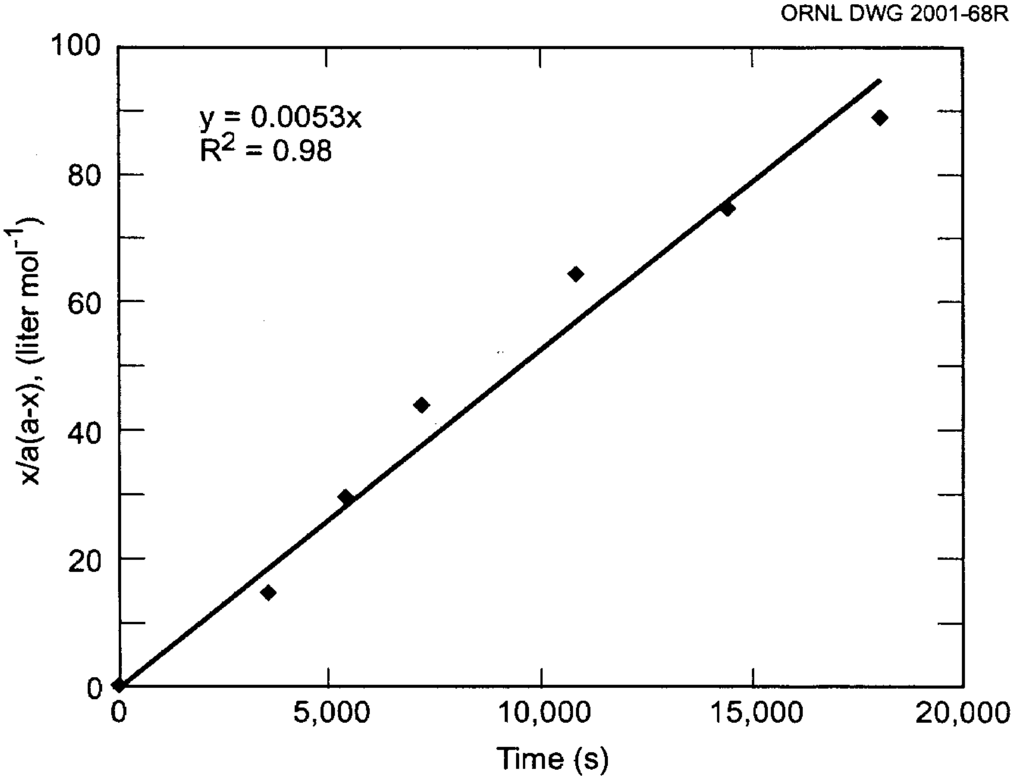

Figure 1. Second Order Reaction Test Plot for 100 °C Test

Tests (13) have been conducted to determine if chemical agents could prevent aluminosilicate formation or inhibit particle growth under conditions similar to Tank 43H. Chemical agents tested included fluoride ion and a number of organic agents such as EDTA, Geosperse C-44 (Crystal Growth Inhibitor), Trisbis, and LUDOX inhibitor in Dowisil. Results from SRTC indicate that tested materials were not effective at limiting the formation of the thermodynamically stable aluminosilicate. However, research at UNOCAL Corporation does show a reduction in the amount of aluminosilicate formed with three candidate materials. These materials were Geosperse C-44, Geogard, and Cridos 93P10. Although a reduction in the amount of solids formed was observed, the experience of the UNOCAL personnel indicate the inhibitors may not control scale formation in larger equipment, e.g., the evaporator pot.

Thermodynamic Modeling

Weber at ORNL (14) used the SOLGASMIX computer code to model the tank chemistry of the 2H Evaporator. A thermodynamic model was developed that simulates phase equilibria over the temperature range of 25 to 125 °C. Parameter values for most of the species of interest had been collected and incorporated into the SOLGASMIX model previously. Pitzer parameters were obtained from the literature for well known electrolytes such as NaOH and NaCl and the three-component systems like Na-Al-OH. However, the model had to be upgraded for several species such as $NaNO_3$, $NaNO_2$, and the corresponding potassium salts of these anions.

Shown in Figure 2 is a comparison of aluminum and silicon concentrations that were measured during an aluminosilicate formation test (15) conducted by Dr. Addai-Mensah at the University of South Australia in an evaporator simulant. The model predictions agree quite well with the experimental data for aluminum and silicon. However in both cases, the model over predicts the solubility. The solubility product constant, K_{sp}, simply the multiplication of the measured silicon and aluminum molar concentrations, for the aluminum and silicon concentrations is 3×10^{-4} M^2 for the SOLGASMIX model prediction. In the experiment the K_{sp} measured 7×10^{-5} M^2.

Using the Geochemist's Workbench computer program, activity diagrams were generated from solution measurements for the feed tank (Tank 43H) of the 2H Evaporator system. Solution measurement (16) were made from samples from two different time populations. Tank samples are routinely analyzed for a number of constituents; however, until the GDL pluggage in 1997 silicon concentration were not routinely measured. There were measurements, however, taken in 1992 and these results are shown in the activity diagram on the left in Figure 3. This silicon and aluminum measurement from 1992 (star in Figure 3) shows that composition was in the gibbsite stability field and not the sodium aluminosilicate (NAS-gel) region. In other words, that solution composition would make gibbsite. In contrast, the average solution composition (oval in Figure 3) from samples taken from 1997 through 1999 lies on the boundary of the sodium aluminosilicate stability field. Moreover, many of the individual samples from the 1997 – 1999 timeframe had composition well within the aluminosilicate stability field.

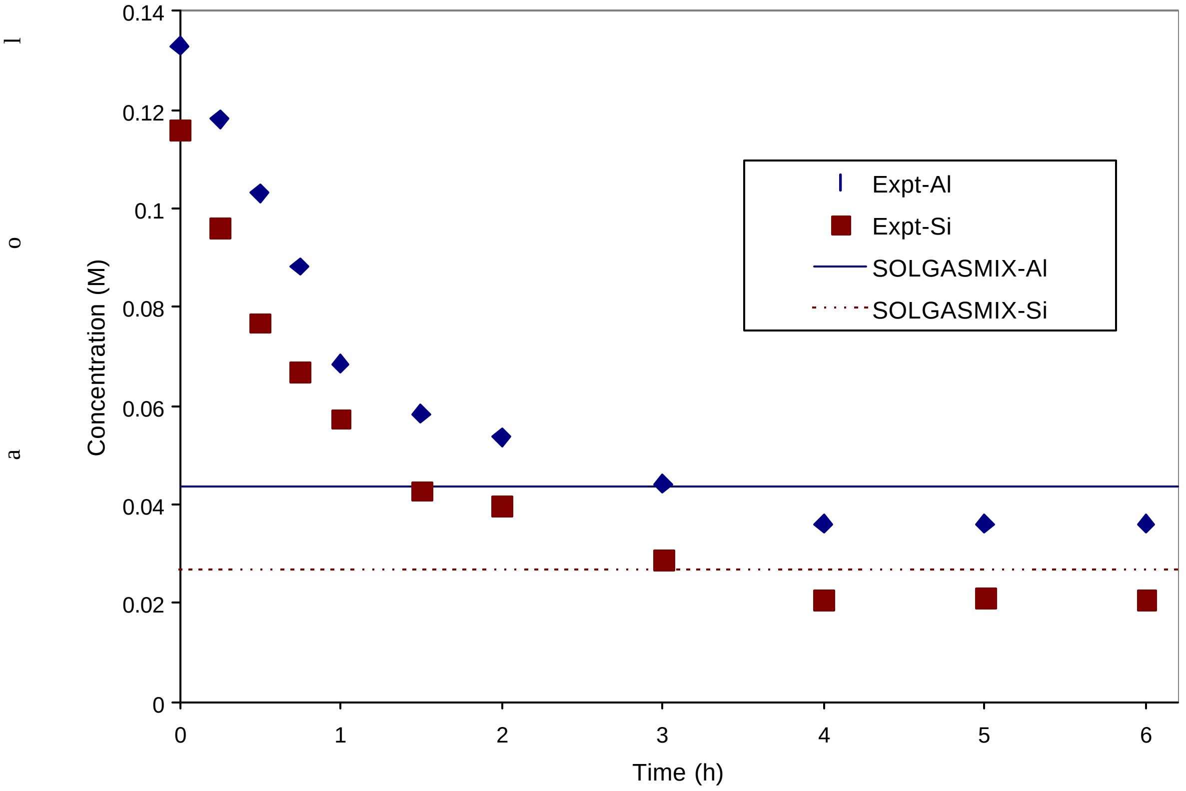

Figure 2. Model Fit of Al and Si Solubility Based on SOLGASMIX Prediction

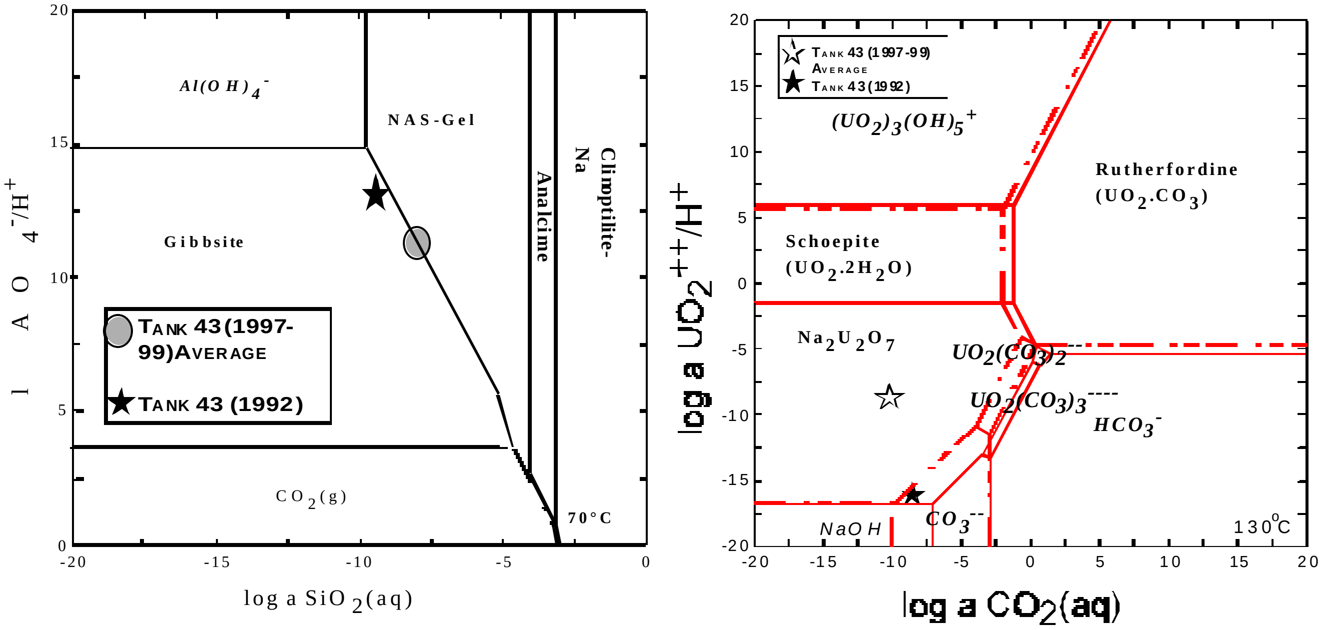

Figure 3. Activity Diagrams for Aluminosilicate and Diuranate Formation in 2H Evaporator

The activity diagram on the right in Figure 3 shows the uranium chemistry for the same samples. It was not unexpected that the solution composition for these samples supports uranium precipitation. In the case of the 1992 sample (darken star) a uranyl carbonate solid phase was predicted. For the 1997 – 1999 samples, sodium diuranate was the predicted solid phase and sodium diuranate was observed by X-ray diffraction in samples from the scale in the evaporator pot and the deposits in the gravity drain line.

The solution thermodynamic data in the Lawrence Livermore National Laboratory database had to be supplemented with aluminum and silicon measurements from a number of literature studies in matrices more akin to the high level waste. The stability boundary location changed accordingly. This model has been successfully applied to the operation of the other SRS evaporators (2F and 3H) (17).

Personnel at the Pacific Northwest National Laboratory were tasked (18) with three objectives regarding the aluminosilicate chemistry of the SRS evaporators. The first objective is to verify the stability regions depicted in the activity diagrams currently being developed at Savannah River. Secondly, PNNL personnel were to identify and characterize insoluble aluminosilicate phases. Lastly the kinetics of solid phase transformation under the alkaline hydrothermal conditions are to be measured.

Results to date indicate that with starting silicon concentrations of 0.01 M solid phases will form rapidly at 80 °C in agreement with SRTC and ORNL data. At low hydroxide levels (0.1 M) the predominant solid phase that forms is sodalite. There appears to be a slow conversion of the sodalite phase to cancrinite. The rate of conversion can be estimated from testing conducted with 0.2 M aluminum concentration. In this test, after ten hours at 80 °C, the solid phase was nearly 100 % sodalite based on X-ray diffraction analysis. After 300 h at 80 °C, 30 % of the sodalite had converted to cancrinite. However, at higher levels of hydroxide (1 M) cancrinite forms as a minor solid phase from the beginning of the heat treatment. Data from PNNL also shows that at lower temperature zeolite A is formed and converts to the denser phase of sodalite and further transforms to cancrinite.

Deposition Testing

Dynamic-light-scattering tests conducted at ORNL (19) indicated that the rate of particle-forming reactions varied over a wide range, depending on the temperature and reactant concentrations. With an increasing concentration of aluminum, the induction time (i.e., the time for detectable particle formation) decreases monotonically. A similar decrease in induction time was found for increasing silicon concentration; however, for the conditions tested; the induction time did not appear to significantly decrease as silicon concentration was increased above approximately 0.05 M. A linear relationship holds between log $(1/t_I)$ and $1/T$ (where t_I is the induction period and T is the temperature); the activation energy for aluminosilicate solid-phase formation was found to be ~65 kJ/mol. Extrapolating the results to the normal operating temperature of the evaporator, particle formation could be relatively rapid (less than 10 min) with significant silicon and aluminum concentrations. The deposition of solids onto stainless steel surfaces was studied; both the growth of solids directly on the surface and the deposition and fusion of particles formed in bulk solution could contribute to scaling. The surface solids grow and expand their coverage of substrate surface in a monolayer manner, and this surface-grown monolayer can be a dense, continuous layer between the substrate and the top deposited/fused particles (Figure 4). Fluid flow was found to have a significant effect on the position, amount, and form of the resulting solids.

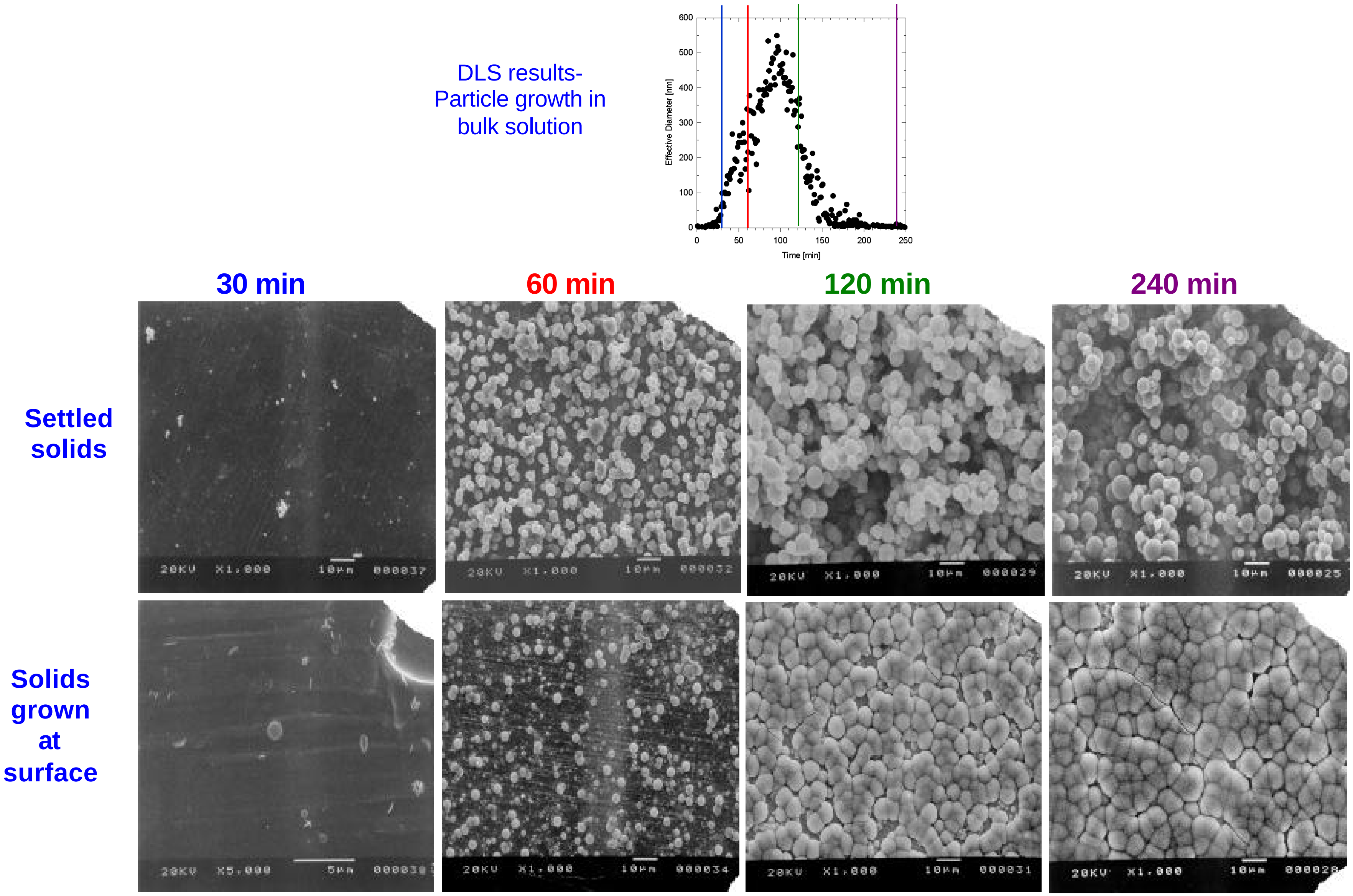

Figure 4. Effect of reaction time on surface-solid formation in solutions of $0.133\ M$ Si–$0.133\ M$ Al–$6\ M$ Na^+ at 80°C. Images labeled “Settled solids” and “Solids grown at surface” are of the upper and lower surfaces, respectively, of samples of stainless steel foil positioned horizontally in solutions. Sample times (see dynamic-light-scattering growth curve above) was selected to correspond with the following phases: nucleation time, middle of growth stage, aggregation, and fully settled.

SRTC performed tests aimed at determining the fraction of aluminosilicate that forms and depositions on the wall of a stainless steel test beaker. The first series of tests were perform in a batch mode and studied the effects of mixing. The results of these SRTC tests agreed well with work performed at ORNL. In both sets of tests, the alimnosilicate depositions showed clear effects of the mixing or hydrodynamic forces. Deposits with mixing showed swirl patterns at SRTC and ORNL in the direction of the propeller motion. Additionally, in the SRTC testing ,the mean deposition coefficients and precision confidence intervals for batch testing are summarized in Table 1. For the conditions evaluated, increased mixing reduces the deposition coefficient dramatically. As observed in the table, under the condition of no mixing, 81 % of the aluminosilicate formed during the tests adhered to the stainless steel beaker whereas, only 37 % adhered to the beaker under the high mixing condition.

The effects of hydrodynamic mixing are even more dramatically observed in the continuous flow test results. The mean deposition coefficients and confidence intervals for continuous feed testing of mixing effects are also summarized in Table 1. For the conditions evaluated, increased mixing reduces the deposition coefficient dramatically. In the case of no mixing in the continuous flow tests, 65 % of the aluminosilicate adhered to the metal beaker and did not flow through the mock evaporator pot. However, under the high mixing case, only 15 % of the aluminosilicate were deposited as a scale indicating that mixing could be utilized to reduce deposition in the evaporator pot.

Table 1. Summary of Mixing Effects on Deposition

Experiment Type	**Mean for Deposition**	**Confidence Interval$_{80}$**
Batch Testing		
No Mixing	81 %	± 10 %
Low Mixing (< 0.2 W/kg)	77 %	± 1 %
High Mixing (0.4 – 0.8 W/kg)	37 %	± 11 %
Continuous Flow		
No Mixing	65 %	± 9 %
Low Mixing (< 0.2 W/kg)	49 %	± 17 %
High Mixing (0.4 – 0.8 W/kg)	15 %	± 3 %

SUMMARY

The research and development program for examining the formation of a sodium aluminosilicate scale and associated accumulation of uranium in high level waste evaporators has made significant advances in determining the rate of formation of aluminosilicate deposits and the factors influencing scale growth. To date, the program has provided direct evidence of aluminosilicate formation, deposition and uranium precipitation in both batch and continuous flow testing. A key finding is the significant influence of mixing on the aluminosilicate deposition rate and location of deposit.

The support provided by the Tanks Focus Area (TFA) has been utilized not only to explain the past problems of evaporation of SRS high level waste, but is being used today to provide the safety basis for evaporation of waste not containing DWPF recycle in the other two operating evaporators. The pressing needs to maintain available space in the tank farms must rely on the research results provided by this effort to ensure that a previously unknown deposition mechanism is observed elsewhere. In particular, the newly operation 3H Evaporator operates at higher temperatures and involves the evaporation of wastes with a significantly higher uranium enrichment.

Efforts continue to address safety and operational issues for aluminosilicate deposition. The 2H evaporator pot has been cleaned using a dilute nitric acid flowsheet (20). However, this was an extensive and expensive activity. Therefore, research efforts are aimed at three aspects to the long term processing of supernates containing silicon. The first aspect supports the planned segregation of the silicon stream (DWPF recycle) and sending to the 2H evaporator and sending the aluminum containing stream (H-canyon) to the 3H evaporator. The work focuses on providing technical baseline to the 2H evaporator. The second avenue focuses on continued efforts to utilize thermodynamic solubilities and hydrodynamic forces to develop and operating window for preventing aluminosilicate scale deposition. Additionally, removal of silicon either within the DWPF or in the tank farm prior to evaporation is being studied. Lastly, a series of tests will examine the method/mechanism for uranium inclusion in the scale. Material balance calculations indicate only a small portion of the silicon that flowed through the evaporator deposited. However, a large fraction of the uranium remained in the scale.

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