

**SUPPLEMENTARY LEACHING TESTS
OF INSULATION AND CONCRETE
FOR GSI-191 CHEMICAL EFFECTS PROGRAM**

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QUALITY OF DATA, ANALYSES, AND CODE DEVELOPMENT

DATA: CNWRA-generated data that are presented in this report meet quality assurance requirements described in the Geosciences and Engineering Division Quality Assurance Manual of Southwest Research Institute®. Experimental data and details of modeling simulations have been recorded in scientific notebooks numbered 686 and 777. The sources of non-CNWRA data referenced in this report should be consulted separately for determining levels of quality assurance.

ANALYSES AND CODES: The publicly available geochemical modeling code PHREEQC Version 2.8 (U.S. Geological Survey, 2003) was used by CNWRA to perform aqueous speciation, saturation, and mass transfer calculations in this report. The referenced software package is controlled under the Technical Operating Procedure (TOP)-18, Development and Control of Scientific and Engineering Software (Revision 10, Change 0).

Reference:

U.S. Geological Survey. "PHREEQC." Version 2.8. Reston, Virginia: U.S. Geological Survey. 2003.

1 INTRODUCTION

In a loss-of-coolant accident (LOCA), insulation or other materials dislodged by the release of coolant water may be transported as debris to the containment sump. Concerns that the post-accident accumulation of entrained debris on the sump screens potentially could impede the recirculation of water in the emergency core cooling system or containment spray system of pressurized water reactors (PWRs) prompted the U.S. Nuclear Regulatory Commission (NRC) to open Generic Safety Issue (GSI)–191, Assessment of Debris Accumulation on PWR Sump Performance (NRC, 2003). In the subsequent Generic Letter 2004-02, Potential Impact of Debris Blockage on Emergency Recirculation During Design Basis Accidents at Pressurized-Water Reactors (NRC, 2004), the NRC requested licensees, on a plant-specific basis, to evaluate the potential for adverse effects of post-accident debris blockage or debris-laden fluids to impede the recirculation functions of the emergency core cooling system. Respondents to an industry survey conducted by the PWR Owners Group (formerly Westinghouse Owners Group) identified the plant-specific types and amounts of containment materials that potentially would be exposed to the spray solution or submerged in the containment sump after a LOCA.

1.1 Integrated Chemical Effects Test Project and Related Studies

Technical assessments conducted as part of GSI–191 have considered the sources, generation, and transport of debris in containment and have characterized the relationship between debris clogging and sump screen head loss. Other GSI–191 studies have investigated the possibility of post-LOCA chemical interactions between coolant water and exposed containment components such as metals, insulation materials, and concrete. Chemical effects could potentially impede pump performance by clogging screens with corrosion products of existing materials or by precipitation of secondary solid phases. The GSI–191 chemical effects research program, informed by an outside peer review panel (Torres, 2006), has included experimental and chemical modeling simulations of post-LOCA chemical environments, dissolution and leaching experiments, and head-loss tests using likely suspended solids. The most detailed experimental study was the Integrated Chemical Effects Test (ICET) Project, consisting of five large-scale experiments conducted at the University of New Mexico. To study post-LOCA chemical reactions, these experiments were conducted in a tank that included 949 L [250 gal] of a simulated neutral or borated containment water at 60 °C [140 °F] and sets of solid samples (metals, insulation materials, and concrete) scaled proportionately to represent post-LOCA submerged and unsubmerged debris components (Dallman, et al., 2006). The ICET experiments were supplemented by bench-scale experiments at the Center for Nuclear Waste Regulatory Analyses (CNWRA) to determine corrosion and leaching rates for many of the ICET sample materials (Jain, et al., 2005; McMurry, et al., 2006) and by bench-scale head-loss experiments at Argonne National Laboratory to simulate physical effects of precipitates as observed in the ICET experiments (Kasza, et al., 2006).

1.2 Westinghouse Supplementary Chemical Effects Tests

Although the ICET experiments tested many containment materials that might be involved in post-LOCA chemical interactions, the tests were not specific to a particular plant. Moreover, the test parameters were defined based on the plant information available at the time, so in some cases the amount of sample material included in the ICET experiments was overestimated. To provide licensees with additional data and methods to assess plant-specific potential for sump

screen blockage by chemical precipitates, the PWR Owners Group commissioned a supplementary chemical effects test program. The Westinghouse test program and results are described in the report WCAP-16530-NP (Westinghouse Electric Company, LLC, 2006).

Using the compiled survey responses, the Westinghouse test program divided the containment materials listed by plant operators into 15 material classes based on chemical compositions. Dissolution and precipitation experiments were conducted on a subset of 10 material classes (Table 1-1). The materials (many of which are identified in Table 1-1 by their brand names, as they were listed in the survey) were selected for testing based on results from the ICET program and known properties of other containment materials expected to generate secondary precipitates. Each material class was represented in the experiments by one sample material except for E-glass insulation, which was represented by two sample materials: NUKON (low-density fiberglass) and high-density fiberglass from unspecified manufacturers.

For the Westinghouse dissolution and precipitation experiments, bench-scale testing was performed on each representative sample material at starting pH values of 4.1, 8, or 12 and at a temperature of 129 or 87.8 °C [265 or 190 °F]. The material-to-solution ratio was scaled by volume based on the maximum ratios reported in the industry survey. The starting solution for all of the tests was a deionized water with 4,400 ppm of boron added as boric acid (H_3BO_3) to give a starting pH of 4.1. Nominal pH values of 8 and 12 were obtained by adding appropriate quantities of sodium hydroxide (NaOH) to the borated water. The leachate was sampled during the dissolution tests after 30, 60, and 90 minutes and was analyzed by induction-coupled plasmospectrometry (ICP) for aluminum, calcium, silicon, magnesium, phosphorus, sulfur, iron, zinc, and titanium. Aluminum, silicon, and calcium dominated the releases from the various materials tested.

At the end of the 90-minute dissolution tests, the Westinghouse leachate solutions were pumped into settling cones, cooled in a water bath controlled at 27 °C [80 °F] for a minimum of 8 hours, and observed for visible evidence of precipitation. Measurable precipitation occurred upon cooling in 10 of the dissolution tests (Table 1-2). The precipitated solids were tentatively identified on the basis of their chemical composition as estimated from scanning electron microscopy (SEM) analysis of filtered material.

In the Westinghouse tests, the leachates produced in the dissolution tests were also used in two other sets of precipitation experiments. In one set of experiments, a pH buffering agent such as trisodium phosphate was added to one of the existing leachates to change its chemistry and promote oversaturated conditions. In the other set of experiments, various combinations of two of the existing leachate solutions were mixed together. The additional experiments were intended to simulate chemical environments that involved multiple reactants, and in this respect they were similar to the ICET experiments. Precipitates were obtained in only three of these additional experiments. A calcium phosphate solid precipitated in two experiments where trisodium phosphate was added to an existing leachate. The third precipitate resulted from a mixed-leachate test where a high silicon concentration from a fiberglass leachate at pH 12 and a high calcium concentration from a CalSil leachate at pH 4.1 combined to form a secondary calcium silicate solid (Westinghouse Electric Company, LLC, 2006).

Table 1-1. Material Classes Tested in Westinghouse Experiments		
Material Class	Description	Products in Class (Bold = Sample Tested by Westinghouse)
Aluminum silicate insulation	Includes synthetic insulation materials and natural aluminum silicates such as kaolin clay and vermiculite	<ul style="list-style-type: none"> • Fiber Frax Durablanket • 3M M-20C insulation • 3M I-Series insulation • Cerablanket • Kaowool • Mat-Ceramic insulation • Mineral fiber • PAROC mineral wool
Calcium silicate insulation	Includes low-density mat insulation and high-density refractory materials	<ul style="list-style-type: none"> • Cal-Sil insulation • Asbestos (natural) • Kaylo • Marinite • Mudd • Transite • Unibestos
E-glass insulation	Includes fiberglass and cellular glass containing amorphous silicon dioxide, calcium oxide, aluminum oxide, and boric oxide	<ul style="list-style-type: none"> • NUKON • “High density” fiberglass (unspecified manufacturers) • Foamglas • Temp-Mat (Temp-Mat “A” and Temp-Mat (AlphaMat “B”) • Thermal Wrap
Mineral wool insulation	Produced from steel slag and from naturally occurring rocks such as basalt and dolomite	<ul style="list-style-type: none"> • Min-Wool • Rock wool (unspecified manufacturers)
Amorphous silica insulation	Microporous insulation containing amorphous silicon dioxide, with a small percentage of E-glass	<ul style="list-style-type: none"> • Min-K • Microtherm
Interam E-Class insulation	Fibrous hydrated alumina and an alumina silicate blanket, with aluminum alloy foil outer layer	<ul style="list-style-type: none"> • Interam E-Class insulation
Concrete	Building material made of cement, sand and gravel aggregate, and admixing agents	<ul style="list-style-type: none"> • Generic concrete (aged 28 days or more)
Aluminum metal	Includes structural members, coatings, small mechanical components (e.g., valves), and thin coatings on insulation	<ul style="list-style-type: none"> • Aluminum alloys (tested SA 1100)
Carbon and low alloy steels	Structural building material	<ul style="list-style-type: none"> • All uncoated or ungalvanized carbon and low alloy steels (tested SA508 Class 2)
Zinc coatings	Galvanized coatings on carbon steel, other zinc coatings	<ul style="list-style-type: none"> • Galvanized steel coatings • Other zinc coatings

Table 1-2. Positive Westinghouse Results for Precipitation Upon Cooling*													
Sample Material	Initial Conditions		Precipitate Formed in 100 mL of Solution†								Mass (mg)‡	Volume (cm ³)‡	Tentative Identification of Precipitate by EDSS§
	pH	T (°F)†	wt%‡										
			Na	Al	Si	Ca	Cu	Zn	Fe				
FiberFrax Durablanket	4.1	265	—	85.0	7.9	—	7.2	—	—	—	2.8	0.28	Hydrated AlOOH
FiberFrax Durablanket	12	265	25.3	29.1	38.8	—	6.8	—	—	—	42.7	9.94	NaAlSi ₃ O ₈
“High Density” Fiberglass	12	265	17.3	26.9	53.5	2.3	—	—	—	—	10.3	3.33	NaAlSi ₃ O ₈ with minor calcium aluminum silicate
Concrete (powdered)	4.1	265	0.5	74.4	16.1	4.9	4.1	—	—	—	3.4	0.36	Calcium aluminum silicate, Al-rich
Concrete (powdered)	8	265	0.8	41.6	27.0	24.1	5.6	—	—	0.8	37.5	9.10	Calcium aluminum silicate
Mineral Wool	4.1	265	0.5	79.6	12.7	0.1	7.1	—	—	—	trace	0.54	Hydrated AlOOH
Aluminum Metal	4.1	265	—	83.1	12.2	—	4.7	—	—	—	1.6	0.10	Hydrated AlOOH
Aluminum Metal	8	265	2.0	95.2	—	—	2.8	—	—	—	755	33	Hydrated AlOOH
Aluminum Metal	12	265	3.4	96.6	—	—	—	—	—	—	615	27	Hydrated AlOOH
Galvanized Steel	12	265	—	1.1	17.0	2.4	3.7	75.9	—	—	4.5	0.03	Zn ₂ SiO ₄ , Ca, and Al impurities

* Adapted from Westinghouse Electric Company, LLC. “Evaluation of Post-Accident Chemical Effects in Containment Sump Fluids to Support GSI-191.”

Tables 5.2-4 and 5.2-5. WCAP-16530-NP. Rev. 0. Pittsburgh, Pennsylvania: Westinghouse Electric Company, LLC. 2006.

†129 °C [265 °F], 100 mL [0.0264 gal], 1 mg [3.5 × 10⁻⁵ oz], 1 cm³ [0.06 in³]

‡no data

§Energy dispersive x-ray spectrometry

1.3 Purpose

The purpose of this study was to conduct supplementary and confirmatory dissolution and precipitation experiments for insulation materials and concrete using an approach similar to the Westinghouse experiments.

The Westinghouse study was designed to provide plant operators with a consistent modeling approach to determine the types and amounts of chemical precipitates that might form for a specific set of containment materials under expected post-LOCA conditions of pH and temperature. Dissolution rates for each material class were estimated from the experiment leachate compositions, which had been sampled at three different exposure times for this purpose. In applying the chemical model for a specific plant environment, the rates are used to calculate the concentration of a solution after reaction with the solid materials in a post-accident environment for a specified amount of time. Rather than calculate how much, if any, solid phases might precipitate from a given solution composition, the model assumes that all of the dissolved material reprecipitates in unidentified secondary solids. This assumption, which is based on the relatively low solubility of several key solid phases of aluminum, calcium, and silicon, provides a conservative estimate of the maximum mass of secondary precipitates that could form from a given solution composition.

The Westinghouse chemical model also assumes that the various insulation materials assigned to the same material class have similar dissolution characteristics, so that the dissolution rates obtained from one type of insulation are representative of all the materials in that class. The main purpose of the supplementary leaching tests performed at CNWRA was to examine the validity of this assumption by testing other sample materials from the same material classes and comparing results with those in the Westinghouse study. Using an approach developed for GSI-191 for a modeling study of post-LOCA chemical effects (McMurry, et al., 2006), the compositions of the leachates also were examined using a chemical modeling code and database of likely precipitates to determine if the leachates were oversaturated with respect to solid phases that had not precipitated for kinetic reasons.

For the CNWRA study, samples were chosen from five of the insulation material classes that had been designated in WCAP-16530-NP (Westinghouse Electric Company, LLC, 2006). A simplified test plan was developed, based on the chemical analyses of leachates in the Westinghouse study. The plan focused on the test conditions that previously had produced the most concentrated solution for each material class (Table 1-3). In addition to obtaining cooled leachate solutions for precipitation tests, the Westinghouse experiments had been designed to obtain short-term dissolution rate data by online sampling of the test solutions from pressurized reaction vessels at 15- to 30-minute intervals throughout each 90-minute test. As it was not practical or necessary to duplicate the Westinghouse testing apparatus or procedures exactly for the CNWRA tests, several of the previously tested sample materials (CalSil, Fiber Frax Durablanket, Temp-Mat, and "high density" fiberglass) also were included in the CNWRA test matrix for comparison. The duplicated sample materials included the insulation types for which precipitated silicates tentatively were identified in the Westinghouse experiments (Table 1-2).

The fiberglass, a high-density type, used in the CNWRA study was obtained from the same supplier as for the Westinghouse study. The other insulation samples were obtained directly from the manufacturers, except for CalSil, which was obtained from Argonne National Laboratory.

Table 1-3. Leachate Analyses From Westinghouse Tests

Westinghouse Sample Material	pH		T (°F)*		Solution Chemistry† (ppm)						Equivalent Sample Materials in CNWRA‡ Test Matrix
	Initial	Final	Initial	Final	Al	Ca	Mg	S	Si		
CaSil	4.1	6.6	190	80	2.7	137.2	0.1	7.9	108.7	CaSil, Marinite	
CaSil	8	8.2	190	80	1.5	38.6	0.1	42.1	377.9	CaSil, Marinite	
CaSil	12	11.5	190	80	0.7	9.5	0.0	24.9	242.4	CaSil, Marinite	
CaSil	4.1	6.8	265	80	0.8	49.3	0.1	7.3	94.4	CaSil, Marinite	
CaSil	8	8.1	265	80	1.2	11.4	0.0	7.2	129.6	CaSil, Marinite	
Durablanket	4.1	5.4	265	80	4.0	1.1	0.1	0.0	10.7	Durablanket, Kaowool	
Durablanket	12	12	265	80	38.2	0.4	0.0	0.3	71.3	Durablanket, Kaowool	
Fiberglass	4.1	6.2	265	80	1.7	19.8	0.4	0.4	110.0	Fiberglass, Temp-Mat A, B	
Fiberglass	12	12	265	80	8.6	7.1	0.0	3.7	222.7	Fiberglass, Temp-Mat A, B	
Nukon	4.1	5.9	265	80	1.0	8.3	0.1	0.0	30.8	Fiberglass, Temp-Mat A, B	
Nukon	12	12	265	80	8.1	8.5	0.0	1.5	191.9	Fiberglass, Temp-Mat A, B	
Min-K	4.1	4.9	190	80	1.2	1.7	0.1	0.4	10.1	Microtherm	
Min-K	8	8	190	80	2.7	6.2	0.1	1.0	217.8	Microtherm	
Min-K	12	11.9	190	80	1.0	0.0	0.0	0.9	141.9	Microtherm	
Min-K	4.1	5.3	265	80	0.9	1.7	0.0	0.0	52.2	Microtherm	
Min-K	12	12.1	265	80	16.6	1.8	0.0	0.6	99.8	Microtherm	
Concrete (powdered)	4.1	6.4	190	80	7.0	143.6	0.1	12.4	30.9	Concrete (solid)	
Concrete (powdered)	8	8.1	190	80	7.6	43.4	0.1	5.4	40.6	Concrete (solid)	

*190 °F = 87.8 °C, 265 °F = 129 °C

†Concentrations taken from Table A-1 of WCAP-16530-NP for samples at 90-minute exposures

‡CNWRA = Center for Nuclear Waste Regulatory Analyses

To supplement the Westinghouse data reported for powdered concrete samples, the CNWRA test matrix included experiments using solid aged concrete coupons, with the surface area {15.72 m²/m³ [4.79 ft²/ft³]} scaled to a high-end loading for uncoated concrete as reported in the industry survey.

2 EXPERIMENTAL METHODS AND TEST MATRIX

The CNWRA confirmatory leaching tests were conducted in borated waters containing 4,000 ppm boron as boric acid (H_3BO_3), at starting pH values of 4.1, 8, and 12 and at temperatures of 129 and 87.8 °C [265 and 190 °F]. The boric acid solution was used without modification for the tests in which the starting solution had a pH of 4.1. For the other tests, appropriate amounts of sodium hydroxide (NaOH) were added to the boric acid solution to obtain starting solutions with pH values of 8 and 12.

Nine sample materials were selected for the confirmatory leaching tests (Table 2-1). Except for concrete, all were insulation products belonging to one of the material classes shown in Table 1-1. All materials except the concrete were used as received from suppliers. Table 2-1 lists the base composition of all the products, their density, and the material-to-borated water volume ratio (or, for concrete, the surface area-to-volume ratio) used in the leaching tests.

The leaching tests were conducted in polytetrafluoroethylene vessels, using 50 mL [1.7 oz] of solution in each test. The vessels were cleaned in accordance with ASTM Standard C-1220 (ASTM International, 2006). The test matrix, including sample identification, temperature, and amount of material used, is provided in Table 2-2. The test matrix included two Temp-Mat insulation products, Temp-Mat and AlphaMat Plus, and one example of each of the other insulation products. Sample amounts were calculated in proportion to 50 mL [1.7 oz] of solution based on the density of materials and the material-to-water volume ratio listed in Table 2-1. The initial physical appearance and the relative quantities of sample materials are shown in Figure 2-1 prior to placing them in the test vessels.

For each leaching test, the dry sample material was placed on top of the solution in the test vessel and was allowed to sink to the bottom of the vessel, without stirring, as it absorbed water. All of the sample materials remained relatively intact upon wetting except for fiberglass and CalSil insulation. Some small particles of fiberglass separated from the bulk of the sample and floated in the test solution. The CalSil insulation expanded in the water and tended to disaggregate as it sank, causing the test solution to become cloudy.

After the sample material was added to each test vessel, the vessel was tightly closed. Sets of vessels were placed in an oven and were maintained at constant temperature for 90 minutes, which corresponded to the maximum exposure time in the Westinghouse tests (Westinghouse Electric Company, LLC, 2006). Then the oven was turned off and the door was opened to allow the oven to cool to room temperature. The entire cooling process took approximately 2 hours. The test vessels were opened to observe precipitate formation. A 1-mL [0.034-oz] sample of leachate was withdrawn from each test cell using a syringe filtered through a 0.45- μ m [1.8×10^{-5} mil] filter and was submitted for chemical analysis using the inductively coupled plasma method. The pH of the leachate was measured, and the remaining leachate was decanted from the vessel into capped glass vials. Leachates were examined for precipitation after 1 day and again after 85 days. The sample materials were dried in air, and 10 of the 25 samples were selected for surface analysis by scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDS).

Table 2-1. Base Composition of Tested Materials and Material-to-Water Volume Ratio			
Material	Chemical Composition	Density {g/cm³ [lb/ft³]}	Material-to-Water Volume Ratio {cm³/cm³ [ft³/ft³]}
Fiberglass	>95% E-glass + <5% binders (Table 3.2-1, WCAP-16530-NP*)	0.064 [4] (Table 5.1-4, WCAP-16530-NP*)	0.14 (Table 5.1-4, WCAP-16530-NP*)
Durablanket	Vitreous aluminosilicate fiber (MSDS)	0.19 [12] (Table 5.1-4, WCAP-16530-NP*)	0.0213 (Table 5.1-4, WCAP-16530-NP*)
Kaowool	80% aluminum silicate + 20% kaolin clay (hydrated aluminum silicate) (Table 3.2-1, WCAP-16530-NP*)	0.13 [8] (sample label)	0.02 (Table 3.1-1, WCAP-16530-NP*)
Marinite	70% calcium silicate + 22% calcium metasilicate + organic fiber + fiberglass (Table 3.2-1, WCAP-16530-NP*)	0.96 [60] (data sheet)	0.0012 (Table 3.1-1, WCAP-16530-NP*)
Temp-Mat A	100% E-glass fiberglass (Table 3.2-1, WCAP-16530-NP*)	0.18 [11] (data sheet)	0.0051 (Table 3.1-1, WCAP-16530-NP*)
AlphaMat (Temp-Mat B)	Similar in composition to Temp-Mat A but more heat resistant	0.18 [11] (data sheet)	0.0051 (Table 3.1-1, WCAP-16530-NP*)
Microtherm	90% (amorphous silica + silicon carbide) + 10% (E-glass + aluminum oxide) (Table 3.2-1, WCAP-16530-NP*)	0.50 [31.2] (data sheet)	0.00055 (Table 3.1-1, WCAP-16530-NP*)
CalSil	Calcium silicate (tobermorite)	0.232 [14.5] (Table 5.1-4, WCAP-16530-NP*)	0.18 (Table 5.1-4, WCAP-16530-NP*)
Concrete	>80% silicon dioxide, 13% cement (Table 3.2-1, WCAP-16530-NP*)	—	4.79 ft ² /ft ³ (Table 3.1-1, WCAP-16530-NP*)
*Westinghouse Electric Company, LLC. "Evaluation of Post-Accident Chemical Effects in Containment Sump Fluids to Support GSI-191." WCAP-16530-NP. Rev. 0. Pittsburgh, Pennsylvania: Westinghouse Electric Company, LLC. 2006.			

Table 2-2. Leaching Test Matrix				
Material	Test ID*	T {°C [°F]}	Start pH	Material Added for 50 mL [1.7 oz] of Solution (g)†
AlphaMat Plus (Temp-Mat B)	AMHTPH4	129 [265]	4.1	0.045
AlphaMat Plus (Temp-Mat B)	AMHTPH12	129 [265]	12	0.045
Temp-Mat A	TMHTPH4	129 [265]	4.1	0.045
Temp-Mat A	TMHTPH12	129 [265]	12	0.045
Fiberglass	FGHTPH4	129 [265]	4.1	0.450
Fiberglass	FGHTPH12	129 [265]	12	0.450
Microtherm	MHTPH4	129 [265]	4.1	0.014
Microtherm	MHTPH12	129 [265]	12	0.014
Microtherm	MTLTPH4	87.8 [190]	4.1	0.014
Microtherm	MTLTPH8	87.8 [190]	8	0.014
Microtherm	MTLTPH12	87.8 [190]	12	0.014
Kaowool	KWHTPH4	129 [265]	4.1	0.128
Kaowool	KWHTPH12	129 [265]	12	0.128
Durablanket	DBHTPH4	129 [265]	4.1	0.205
Durablanket	DBHTPH12	129 [265]	12	0.205
Marinite	MAHTPH4	129 [265]	4.1	0.058
Marinite	MAHTPH12	129 [265]	12	0.058
Marinite	MALTPH4	87.8 [190]	4.1	0.058
Marinite	MALTPH8	87.8 [190]	8	0.058
Marinite	MALTPH12	87.8 [190]	12	0.058
CalSil	CSLTPH4	87.8 [190]	4.1	2.090
CalSil	CSLTPH8	87.8 [190]	8	2.090
CalSil	CSLTPH12	87.8 [190]	12	2.090
Concrete (solid)	COLTPH4	87.8 [190]	4.1	786 mm ² (surface area)
Concrete (solid)	COLTPH8	87.8 [190]	8	786 mm ² (surface area)
*ID = identification †1 g = 0.0022 lb				

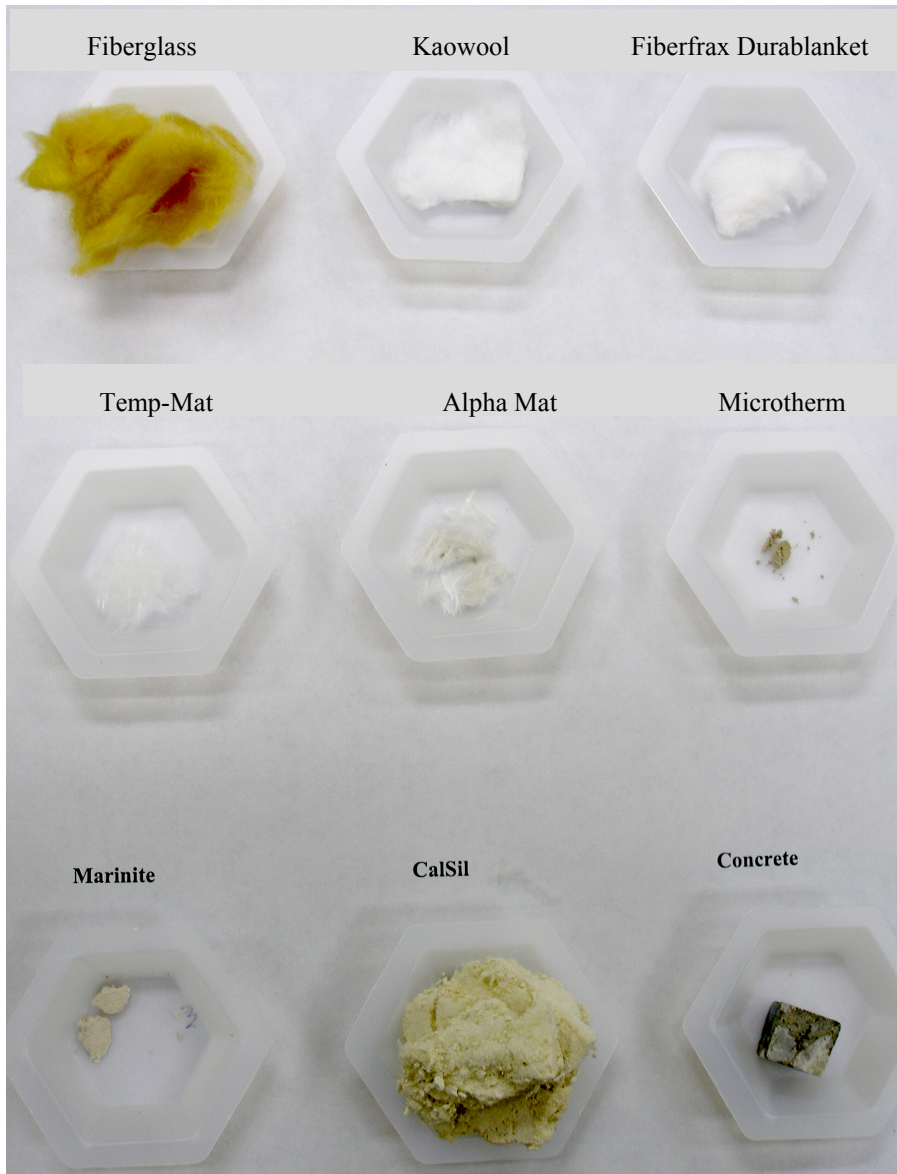


Figure 2-1. Photograph of Weighed Quantities of Starting Materials

3 RESULTS

3.1 Precipitate Formation and Solubility Calculations

At the end of the two-hour cooling period in the leaching tests, the test vessels were opened and examined for precipitation. No precipitates were observed in any of the test vessels. The three CalSil test solutions remained cloudy, with no evidence of new solid phases. In the two fiberglass test solutions, the small fiberglass particles were suspended near the top of the solution, giving it a slightly cloudy, layered appearance. Examples of the cloudy CalSil and fiberglass solutions are shown in Figure 3-1. For comparison, Figure 3-1 includes an example of a clear post-test solution, in a test vessel with Temp-Mat insulation, which was representative of the appearance of all the other leachates.

The leachate solutions were decanted without filtration to capped glass vials. The fiberglass leachates were extracted with a sterilized syringe inserted beneath the layer of suspended particles. The separated fiberglass leachates were clear instead of cloudy.

The vials of leachate were examined again the next day for evidence of precipitation or other changes in appearance. No precipitates were observed, and all of the leachates were clear except for the three cloudy CalSil samples. In contrast, visible precipitates had settled out within 8 hours in the Westinghouse tests.

The CNWRA leachate samples were examined a third time after 85 days, but no precipitates were observed to have settled out from in any of the solutions. The three CalSil leachates remained cloudy. Some material had settled from solution onto the bottom of the vial, but it closely resembled the color and texture of the original disaggregated CalSil particles.

To evaluate the possibility that some of the leachates were oversaturated with respect to secondary solid phases but precipitation was hindered for kinetic reasons, the geochemical modeling code PHREEQC Version 2.8 (U.S. Geological Survey, 2003) was used to calculate the solubility (saturation index) of potential solid phases in the leachates. The input solution compositions were the leachate chemical analyses, as reported below, at the specified final pH for each test and at an assumed final temperature of 27 °C [80 °F]. All of the modeled solution compositions were modified slightly to allow for partial equilibration with atmospheric carbon dioxide equivalent to a log p_{CO_2} value of -9.5. This was considered to be a limiting maximum value because the test vials were capped, restricting the amount of air available, and larger concentrations of dissolved carbon dioxide lowered the calculated pH of the alkaline test solutions below a value of 12.0, contrary to measured results.

The PHREEQC thermodynamic database file on which the solubility calculations were based was adapted from the database file "thermo.com.V8.R6.full" (Wolery and Daveler, 1992) developed at Lawrence Livermore National Laboratory (LLNL). In addition to thermodynamic data for aqueous chemical species, the LLNL database contains solubility constants for more than a thousand potential solids, few of which would be expected to form under low-temperature short-term conditions. To facilitate the PHREEQC calculations, the LLNL database was edited (McMurry, et al., 2006) to shorten the list of solid phases to about twenty solids of interest (Table 3-1). These included phases that are known to crystallize from low-temperature aqueous solutions of appropriate composition, phases tentatively identified as precipitates in the Westinghouse tests (e.g., sodium aluminum silicate minerals), and tobermorite and xonotlite,

Table 3-1. Solid Phases Used in Solubility Calculations*	
Solid	Chemical Formula
Albite	$\text{NaAlSi}_3\text{O}_8$
Amorphous silica	SiO_2
Artinite	$\text{Mg}_2\text{CO}_3(\text{OH})_2 \cdot 3\text{H}_2\text{O}$
B_2O_3	B_2O_3
Boehmite	AlOOH
Borax	$\text{Na}_2(\text{B}_4\text{O}_5(\text{OH})_4) \cdot 8\text{H}_2\text{O}$
Boric acid	$\text{B}(\text{OH})_3$
Brucite	$\text{Mg}(\text{OH})_2$
Calcite	CaCO_3
Diaspore	AlOOH
Gibbsite	$\text{Al}(\text{OH})_3$
Huntite	$\text{CaMg}_3(\text{CO}_3)_4$
Hydroboracite	$\text{MgCaB}_6\text{O}_{11} \cdot 6\text{H}_2\text{O}$
Lansfordite	$\text{MgCO}_3 \cdot 5\text{H}_2\text{O}$
Magnesite	MgCO_3
Monohydrocalcite	$\text{CaCO}_3 \cdot \text{H}_2\text{O}$
Nesquehonite	$\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$
Tobermorite-14A	$\text{Ca}_5\text{Si}_6\text{H}_{21}\text{O}_{27.5}$
Xonotlite	$\text{Ca}_6\text{Si}_6\text{O}_{17}(\text{OH})_2$
*Based on PHREEQC thermodynamic database file <i>lml.dat</i> (U.S. Geological Survey. "PHREEQC." Version 2.8. Reston, Virginia: U.S. Geological Survey. 2003.)	

which are found in calcium silicate insulation products. The list of solids also included carbonate minerals, assuming that carbonate phases might form because the leachate solutions would have acquired some dissolved carbon dioxide by exposure to air.

The results of the solubility calculations are discussed next in the context of the leachate chemistry for each class of sample materials.

3.2 Chemical Analysis of Leachates

3.2.1 E-Glass Insulation Materials

Test results for AlphaMat Plus, Temp-Mat, and fiberglass leachates in this study are presented in Table 3-2. The samples were exposed to borated water at a temperature of 129 °C [265 °F] and at starting pH values of approximately 4.1 or 12, as indicated in the table. For comparison, WCAP-16530-NP (Westinghouse Electric Company, LLC, 2006). All results are for experiment times of 90 minutes.

In the CNWRA tests, AlphaMat Plus and Temp-Mat samples were very similar in terms of their leaching characteristics. At pH 12, about 50 ppm silicon dissolved from both samples. The fiberglass sample was more reactive, with 120 ppm silicon released to solution at pH 12, but all 3 materials released similar minor amounts of aluminum, calcium, and potassium. Less dissolution overall was noted for all three sample materials at pH 4.1. In particular, silicon concentrations were less than 10 ppm, and aluminum and potassium were below detection limits. The starting pH of 4 increased in all three tests to a final value of about 5.5.

The fiberglass sample tested by Westinghouse was from the same supplier as for the CNWRA tests, but the Westinghouse test at pH 12 produced a dissolved silicon concentration almost twice as large as in the CNWRA test (Table 3-2). In the Westinghouse test, a precipitate was tentatively identified by EDS as sodium aluminum silicate ($\text{NaAlSi}_3\text{O}_8$), although this mineral, albite, is more commonly associated with high-temperature magmatic and metamorphic processes. No precipitated solids were observed in the CNWRA test for the same sample material, but the chemical modeling calculations predicted that the leachate from the fiberglass sample at pH 12 would be at or near equilibrium with calcite (i.e., log saturation index near zero). An increase in calcium concentration would cause the solution to be oversaturated with respect to calcite. In contrast, the solubility calculations indicated that the CNWRA leachate at pH 12 would be undersaturated with respect to albite. The chemical analysis for the corresponding Westinghouse leachate did not report the sodium concentration (Table 3-2), but its value for solubility calculations can be estimated from the CNWRA leaching test at equivalent conditions. The calculations indicate that the Westinghouse leachate, also, was likely to be distinctly undersaturated with respect to albite (log saturation index less than -0.8). It is unlikely that the precipitate observed in the Westinghouse test was $\text{NaAlSi}_3\text{O}_8$. However, the solubility calculations also indicated that the fiberglass leachate in the Westinghouse test would have been at or near equilibrium with respect to the aluminum oxyhydroxide mineral diaspore (AlOOH) and slightly oversaturated (log saturation index greater than 0.30) with respect to another sodium aluminum silicate mineral, analcime ($\text{NaAlSi}_2\text{O}_6 \cdot \text{H}_2\text{O}$), that is commonly found in hydrothermal volcanic settings. The Nukon sample tested by Westinghouse released a high concentration of silicon similar to that of the fiberglass solution (Table 3-2), but no precipitate was observed or predicted in the Nukon test.

The dissolution chemistry of the fiberglass tested by CNWRA at pH 4.1 was markedly different from that in the equivalent Westinghouse test. About 3 ppm of silicon was released in the CNWRA test, compared with 100 ppm in the Westinghouse test. A small amount of precipitate, tentatively identified as hydrated AlOOH , was noted in the Westinghouse test. In contrast, no precipitate was observed in the CNWRA leachate.

Table 3-2. E-Glass Insulation Leachate Compositions

Source of Data*	Sample	T (°F)†	pH Initial	pH Final	Concentration (ppm)											Comments
					B	Na	Al	Ca	Mg	K	Si	S				
CNWRAT	AlphaMat Plus	265	12	12.0	3,830	8,670	6.1	9.3	—§	15.0	—	50.5	—			
CNWR	Temp-Mat	265	12	12.0	3,690	8,480	5.2	7.4	—	15.2	—	50.9	—			
CNWR	Fiberglass	265	12	12.0	3,760	8,570	4.3	17.2	—	15.8	—	120	4.5	Predicted equilibrium with CaCO ₃		
Westinghouse	Fiberglass	265	12	12.0	n.r.¶	n.r.	8.6	7.1	—	n.r.	—	222.7	3.7	Precipitate observed (NaAlSi ₃ O ₈ and minor calcium aluminum silicate?)		
Westinghouse	Nukon	265	12	12.0	n.r.	n.r.	8.1	8.5	—	n.r.	—	191.9	1.5			
CNWR	AlphaMat Plus	265	4.1	5.5	3,900	8.2	—	8.0	2.5	—	—	6.4	—			
CNWR	Temp-Mat	265	4.1	5.5	3,860	7.1	—	7.8	2.7	—	—	6.5	—			
CNWR	Fiberglass	265	4.1	5.4	3,800	21.1	—	1.7	—	—	—	3.2	3.9	"Cloudy" solution observed		
Westinghouse	Fiberglass	265	4.1	6.2	n.r.	n.r.	1.7	19.8	0.4	n.r.	—	110.0	0.4			
Westinghouse	Nukon	265	4.1	5.9	n.r.	n.r.	1.0	8.3	0.1	n.r.	—	30.8	—			

*CNWR = present study; Westinghouse = WCAP-16530-NP (Westinghouse Electric Company, LLC. "Evaluation of Post-Accident Chemical Effects in Containment Sump Fluids to Support GSI-191." WCAP-16530-NP. Rev. 0. Pittsburgh, Pennsylvania: Westinghouse Electric Company, LLC. In review. 2006.)

†129 °C [265 °F]; 87.8 °C [190 °F]

‡CNWR = Center for Nuclear Waste Regulatory Analyses

§—below detection limit

¶n.r. = not reported

Table 3-2 also includes leachate analyses from similar tests by Westinghouse for fiberglass and Nukon, as reported in

3.2.2 Amorphous Silica Insulation Materials

Only two insulation products, Min-K and Microtherm, were included in this material class in WCAP-16530-NP (Westinghouse Electric Company, LLC, 2006). Results of various CNWRA leaching tests for Microtherm are contrasted with equivalent Westinghouse tests for Min-K in Table 3-3. At 129 °C [265 °F], tests were conducted at starting pH values of 4.1 and 12. At 87.8 °C [190 °F], tests were conducted at starting pH values of 4.1, 8, and 12. All results in Table 3-3 are for the maximum exposure time of 90 minutes.

Overall, Microtherm was more resistant to leaching than the equivalent proportion of Min-K. At pH values of 8 and 12, silicon was the main element released to solution, with silicon concentrations ranging from 17 to 86 ppm for Microtherm and 100 to 218 ppm for Min-K. In contrast, almost no silicon was released from either sample material at pH 4.1 at 87.8 °C [190 °F]. At 129 °C [265 °F] and pH 4.1, less than 3 ppm Si was released from Microtherm, and 52 ppm was released from Min-K.

No precipitates were observed in any of the Microtherm leachate solutions, but microscopic examination of the samples indicated that they had undergone physical degradation (see Section 3.3).

3.2.3 Aluminum Silicate Insulation Materials

Test results for Kaowool and Durablanket leachates in this study are presented in Table 3-4. The samples were exposed to borated water at a temperature of 129 °C [265 °F] and at starting pH values of approximately 4.1 or 12, as indicated in the table. For comparison, Table 3-4 also includes Durablanket leachate analyses from similar tests by Westinghouse, as reported in WCAP-16530-NP (Westinghouse Electric Company, LLC, 2006). All results are for experiment times of 90 minutes.

In the CNWRA tests, the Kaowool and Durablanket samples exhibited almost identical leaching behaviors. Slightly higher concentrations of dissolved elements were observed in the Westinghouse tests for the Durablanket samples at the same conditions, but overall the leachate compositions were similar.

A precipitate tentatively identified as $\text{NaAlSi}_3\text{O}_8$ was observed in the Westinghouse test of Durablanket at pH 12 (Table 3-4), and a precipitate tentatively identified as a polymorph of AlOOH was observed in the Westinghouse test of Durablanket at pH 4.1. No precipitates were observed in the equivalent CNWRA tests, but solubility calculations indicated that the Durablanket leachate in the CNWRA test at pH 12 was oversaturated with respect to diaspore and boehmite, both of which are polymorphs of AlOOH . The aluminum concentrations in the Durablanket leachate and the Kaowool leachate at pH 4.1 were below detection limits (Table 3-4), so no oversaturated aluminum-bearing phases were predicted in the solubility modeling. However, the PHREEQC (U.S. Geological Survey, 2003) solubility calculations for the Westinghouse leachates (assuming that the unreported sodium and boron concentrations were similar to the equivalent CNWRA tests) indicated that diaspore and boehmite were strongly oversaturated in the Westinghouse leachate at pH 4.1. Given that one of these phases was identified as a precipitate in the experiment, the resulting leachate should have been in equilibrium with the precipitate instead of strongly oversaturated.

Table 3-3. Leachate Compositions for Amorphous Silica Insulation Samples

Source of Data*	Sample	T (°F)†	pH Initial	pH Final	Concentration (ppm)										Comments
					B	Na	Al	Ca	Mg	K	Si	S			
CNWR‡	Microtherm	265	12	12.0	3,810	8,690	2.7	—§	—	15.1	83.3	—	—	—	Physical degradation of sample
Westinghouse	Min-K	265	12	12.1	n.r.¶	n.r.	16.6	1.8	—	n.r.	99.8	0.6	—	—	
CNWR‡	Microtherm	265	4.1	4.5	3,830	8.7	—	—	—	—	2.4	—	—	—	
Westinghouse	Min-K	265	4.1	5.3	n.r.	n.r.	0.9	1.7	—	n.r.	52.2	—	—	—	
CNWR‡	Microtherm	190	8	8.0	3,820	1,470	—	—	1.6	—	16.8	—	—	—	
Westinghouse	Min-K	190	8	8.0	n.r.	n.r.	2.7	6.2	0.1	n.r.	217.8	1.0	—	—	
CNWR‡	Microtherm	190	12	12.0	3,780	8,580	2.5	1.1	—	14.5	86.2	—	—	—	
Westinghouse	Min-K	190	12	11.9	n.r.	n.r.	1.0	—	—	n.r.	141.9	0.9	—	—	
CNWR‡	Microtherm	190	4.1	4.5	3,800	8.3	—	—	—	—	2.9	—	—	—	
Westinghouse	Min-K	190	4.1	4.9	n.r.	n.r.	1.2	1.7	0.1	n.r.	10.1	0.4	—	—	

*CNWR‡ = present study; Westinghouse = WCAP-16530-NP (Westinghouse Electric Company, LLC. "Evaluation of Post-Accident Chemical Effects in Containment Sump Fluids to Support GSI-191." WCAP-16530-NP. Rev. 0. Pittsburgh, Pennsylvania: Westinghouse Electric Company, LLC. 2006.)

†129 °C [265 °F]; 87.8 °C [190 °F]

‡CNWR‡ = Center for Nuclear Regulatory Analyses

§—below detection limit

¶n.r. = not reported

Table 3-4. Leachate Compositions for Aluminum Silicate Insulation Samples

Source of Data*	Sample	T (°F)†	pH Initial	pH Final	Concentration (ppm)								Comments
					B	Na	Al	Ca	Mg	K	Si	S	
CNWR‡	Kaowool	265	12	12.0	3,770	8,660	4.7	—§	—	15.2	46.0	—	
CNWR‡	Durablanket	265	12	12.0	3,750	8,250	15.6	—	—	13.4	44.4	—	Precipitate predicted
Westinghouse	Durablanket	265	12	12.0	n.r.¶	n.r.	38.2	0.4	—	n.r.	71.3	0.3	Precipitate observed
CNWR‡	Kaowool	265	4.1	4.5	3,890	6.5	—	—	—	—	1.8	—	
CNWR‡	Durablanket	265	4.1	4.5	3,870	7.7	—	—	—	—	1.8	—	
Westinghouse	Durablanket	265	4.1	5.4	n.r.	n.r.	4.0	1.1	0.1	n.r.	10.7	—	Precipitate observed

*CNWRA = present study; Westinghouse = WCAP-16530-NP

†129 °C [265 °F]; 87.8 °C [190 °F]

‡CNWRA = Center for Nuclear Waste Regulatory Analyses

¶n.r. = not reported

§—below detection limit

In terms of predicted saturation and observed precipitation, the modeled results are more ambiguous for the Durablanket leachate at pH 12 in the Westinghouse test. The leachate was calculated to be strongly undersaturated with respect to albite, $\text{NaAlSi}_3\text{O}_8$, but it was in equilibrium with analcime, another sodium aluminum silicate mineral. Although analcime is a hydrothermal mineral unlikely to form under the experiment conditions, its precipitation would account for the observed solution composition as well as the presence of a solid that was tentatively identified by EDS as a sodium aluminum silicate phase. However, analcime is a hydrothermal mineral commonly associated with volcanic rocks, and it is unlikely that it would form under the experiment conditions.

3.2.4 Calcium Silicate Insulation Materials

The CNWRA leaching test results for Marinite and CalSil are presented in Table 3-5. The CalSil samples tested in the Westinghouse study were observed to be more soluble at lower temperature than at higher temperature, so with the exception of two Marinite leaching tests at 129 °C [265 °F], the CNWRA tests of calcium silicate insulation materials focused primarily on experiments at 87.8 °C [190 °F] and at pH values of 4.1, 8, and 12. For comparison, Table 3-5 also includes leachate analyses from similar tests by Westinghouse for CalSil samples, as reported in WCAP-16530-NP (Westinghouse Electric Company, LLC, 2006). All results are for experiment times of 90 minutes.

Although cloudy solutions were produced by the physical disaggregation of CalSil in all three leaching tests, no precipitates were observed and no oversaturated solid phases were indicated by chemical modeling for any of the leachate compositions.

At the experiment temperatures of 129 and 87.8 °C [190 and 265 °F], the leaching behavior of the Marinite samples did not appear to be affected by temperature. Marinite leachate compositions at both temperatures at pH 12 were very similar to each other, as were the leachates at pH 4.1 (Table 3-5). In the experiments at 87.8 °C [190 °F], more silicon and potassium were leached from the Marinite at pH 12 than at pH values of 8 or 4. At each pH value, the CalSil insulation was more readily leached than the Marinite. In the CNWRA tests, relatively large amounts of silicon (117 ppm), potassium (82 ppm), sulfur (56 ppm), and calcium (26 ppm) were leached from the CalSil at pH 12. At pH 4.1, a high concentration of calcium (252 ppm) was released from the CalSil, in addition to 383 ppm of sodium and 62 ppm of silicon. Except for dissolved calcium, the CalSil sample tested by CNWRA was less reactive at pH 8 than it was at pH 12 or 4. In contrast, the highest concentration of silicon (378 ppm) was released at pH 8 from the CalSil sample tested by Westinghouse. Overall, significantly larger releases of silicon and smaller releases of calcium at each pH value were obtained from CalSil in the Westinghouse tests compared to the CNWRA tests.

3.2.5 Concrete

Leaching test results for concrete in this study are presented in Table 3-6, where they are contrasted with leaching results for concrete under similar conditions in the Westinghouse study. The concrete samples tested in the Westinghouse matrix were the most soluble at 87.8 °C [190 °F] and at pH values of 4.1 and 8, so the CNWRA tests focused on the same conditions. All results are for experiment times of 90 minutes.

Table 3-5. Leachate Compositions for Calcium Silicate Insulation Samples

Source of Data*	Sample	T (°F)†	pH Initial	pH Final	Concentration (ppm)										Comments	
					B	Na	Al	Ca	Mg	K	Si	S				
CNWRA‡	Marinite	265	12	12.0	3,750	8,430	3.5	6.3	—§	17.3	58.2	—	—	—	—	—
CNWRA	Marinite	265	4.1	5.9	3,870	7.7	—	30.2	—	—	17.3	—	—	—	—	—
CNWRA	Marinite	190	12	12.0	3,640	8,140	2.9	4.0	—	16.2	53.7	—	—	—	—	—
CNWRA	CalSil	190	12	11.3	3,710	8,300	—	26.0	—	82.3	117	56.0	—	—	—	—
Westinghouse	CalSil	190	12	11.5	n.r.¶	n.r.	0.7	9.5	—	n.r.	242	24.9	—	—	—	—
CNWRA	Marinite	190	4.1	6.1	3,840	8.2	—	31.7	—	—	17.3	—	—	—	—	—
CNWRA	CalSil	190	4.1	7.4	3,750	383	—	252	1.5	13.6	62.1	43.8	—	—	—	—
Westinghouse	CalSil	190	4.1	6.6	n.r.	n.r.	2.7	137	0.1	n.r.	109	7.9	—	—	—	—
CNWRA	Marinite	190	8	8.0	3,850	1,490	—	9.8	—	3.9	13.4	—	—	—	—	—
CNWRA	CalSil	190	8	8.2	3,830	1,630	—	109	—	26.3	37.3	25.8	—	—	—	“Very cloudy” leachate
Westinghouse	CalSil	190	8	8.2	n.r.	n.r.	1.5	38.6	0.1	n.r.	378	42.1	—	—	—	—

*CNWRA = present study; Westinghouse = WCAP-16530-NP

†129 °C [265 °F]; 87.8 °C [190 °F]

‡CNWRA = Center for Nuclear Waste Regulatory Analyses

¶n.r. = not reported

§—below detection limit

Table 3-6. Leachate Compositions for Concrete Samples

Source of Data*	Sample	T (°F)†	pH Initial	pH Final	Concentration (ppm)										Comments
					B	Na	Al	Ca	Mg	K	Si	S			
CNWRA‡	Solid Concrete	190	4.1	6.6	3,890	7.3	—§	91.0	—	3.4	13.3	4.9			
Westinghouse	Powdered Concrete	190	4.1	6.4	n.r.¶	n.r.	7.0	143.6	0.1	n.r.	30.9	12.4	• Precipitate observed (aluminum- rich calcium aluminum silicate?) • Precipitate predicted (AIOOH)		
CNWRA	Solid Concrete	190	8	8.1	3,860	1,470	—	38.3	—	6.3	10.1	4.8			
Westinghouse	Powdered Concrete	190	8	8.1	n.r.	n.r.	7.6	43.4	0.1	n.r.	40.6	5.4	• Precipitate observed (calcium aluminum silicate?) • Precipitate predicted (AIOOH)		

*CNWRA = present study; Westinghouse = WCAP-16530-NP

†129 °C [265 °F]; 87.8 °C [190 °F]

‡CNWRA = Center for Nuclear Waste Regulatory Analyses

¶n.r. = not reported

§—below detection limit

The Westinghouse dissolution tests used crushed concrete samples. The tests used an arbitrary sample amount of 4.0 g/100 mL [5.3 oz/gal] because the surface area of the crushed material had not been measured when the tests began (Westinghouse Electric Company, LLC, 2006). This was a very conservative estimate because it was later determined that only 0.0002 g/100 mL [0.0003 oz/gal] of the crushed concrete sample was needed to obtain the desired material-to-coolant ratio of 15.72 m²/m³ [4.79 ft²/ft³]. The main purpose of the CNWRA testing was to compare the Westinghouse leachate results with results for a more realistic amount of solid concrete sample, using the ratio of 15.72 m²/m³ [4.79 ft²/ft³] as determined from the industry survey. This sample amount also is conservative because it represents the high end of the industry survey estimates for the amount of uncoated concrete that would be available to react with water under post-LOCA conditions. A smaller material-to-coolant ratio for the concrete would result in a more dilute leachate and less precipitation.

As expected, the leachate concentrations for the solid concrete coupon were consistently less than observed for crushed concrete in the Westinghouse tests. However, the amount of calcium released to solution was still relatively high for the solid concrete sample, particularly at low pH, compared to some of the insulation materials that were tested in this study.

Precipitates were observed in the Westinghouse tests and were tentatively identified as calcium aluminum silicate phases. Chemical modeling, based on the solution compositions presented in WCAP-16530-NP (Westinghouse Electric Company, LLC, 2006), indicated that both leachates (from the pH 4.1 test and the pH 8 test) were strongly oversaturated with respect to various polymorphs of AlOOH. Otherwise, the leachates were not at or near equilibrium with any of the solids in the database file that represented likely precipitates. The database file did not include any calcium aluminum silicate phases because these solids were not considered likely to form in aqueous solutions at the temperatures or timeframes represented in the experiments.

No oversaturated phases were indicated by modeling in either of the CNWRA leachates, and no precipitates were observed in the test solutions.

3.3 Surface Analysis of Sample Materials

After the leaching tests concluded, air-dried test samples were analyzed by EDS and by SEM. These samples were compared for surface changes with unleached, as-received samples of the same materials. The EDS analyses supplemented the leachate compositions by providing data about the starting composition of the solids and indicated what elements were leached from or added to the sample surface. The SEM images provided a visual check for textural changes in the sample materials, including small amounts of precipitates that may have adhered to the sample surface, where they would not have been detected in solution.

The Appendix contains the EDS spectra data for as-received and post-leaching surfaces of sample materials, and Table 1 summarizes the composition data derived from the EDS spectra. The post-test samples that were selected for surface analysis were from the most reactive test cases in the test matrix for each sample material, based on ICP analyses of the leachates. The contrast between as-received and post-test spectra in the Appendix consistently indicated that the surface concentration of sodium increased in every test that was conducted at pH values of 8 or 12. The surface increase in sodium is attributed to the relatively large amount of sodium hydroxide that was added to the starting solutions to obtain the alkaline pH values. It is likely

that evaporation of the solution when the post-test samples were air-dried caused sodium hydroxide to be deposited on the solid surfaces.

The SEM images of sample materials are presented (Figures 3-2 through 3-10) as a set of composite figures of each sample material tested. The images in each figure are at two magnifications, with views of the as-received material at the top (with the most enlarged view at right) of the composite figure and post-leaching samples shown at the bottom of each figure (with the most enlarged view at right). A 100-micrometer scale bar is shown on each photograph for reference. The figure caption indicates the specific test from which the post-leaching sample was taken in each case.

Overall, the SEM images show little change in the appearance of the sample materials before and after leaching. The fine glass fibers of the E-glass sample materials, for example, retained a smooth texture that does not indicate any significant dissolution of the sample. Enlarged views of post-test AlphaMat (Figure 3-2), Temp-Mat (Figure 3-3), and fiberglass ("high-density") (Figure 3-4) have a weblike coating on some small patches of fibers that was not apparent in the as-received material, but the thin and smooth appearance of the coating suggests that it was not precipitated during the test but instead formed as a surface tension effect when the residual leachate solution evaporated. A similar texture was observed on clean (as received) fiberglass samples dipped in the ICET #1 solution and allowed to dry (Klasky, et al., 2006).

The aluminum silicate sample materials were even more finely fibrous than the E-glass samples, but the post-leaching Kaowool (Figure 3-5) and Durablanket (Figure 3-6) remained almost identical texturally to the as-received samples, except that a small amount of the weblike coating and a few very small white crystals were noted in the post-test Durablanket sample.

The as-received texture of the calcium silicate sample materials, Marinite and CalSil, was more heterogeneous than that of the E-glass and aluminum silicate samples, as indicated by the SEM photographs of Marinite (Figure 3-7) and CalSil (Figure 3-8). Both materials were a composite of granular and fibrous components, and the appearance of the as-received and post-test surfaces varied with location on the sample. Although the chemical analyses of the leachate solutions indicated that Marinite and CalSil were generally reactive compared to most of the other tested sample materials, the photographed surfaces did not show any clear indications of dissolution or precipitation reactions.

The concrete coupons also were texturally heterogeneous, with a bimodal particle distribution of fine-grained cement and coarser aggregate. The cement phase, which is the more reactive part of the concrete mixture, showed some signs of surface etching at pH 4.1 (Figure 3-9). This corresponds to the increased leaching of calcium and some silicon from the coupon at this pH (Table 3-6).

The most conspicuous changes in sample appearance were for Microtherm, which was the only amorphous silica insulation material tested by CNWRA. Texturally, the Microtherm product tested was a molded pipe insulation that is a dense composite of granular and fibrous material. Compared to the other sample materials tested, very small quantities of Microtherm were used in the leaching experiments (Table 2-1), but the elevated dissolved silicon concentrations that were measured in the leaching tests at pH 12 indicated that the Microtherm reacted significantly (Table 3-3). The extent of the reaction is suggested by changes in the appearance and surface composition of the samples. The SEM photographs in Figure 3-10 show the as-received

appearance of the Microtherm at the top, contrasted with post-test views of sample surfaces from two experiments at pH 12. The middle pair of images in Figure 3-10 are from a test at 129 °C [265 °F] and the bottom pair of images are from a test at 87.8 °C [190 °F]. In both pairs of post-test images, the surface roughness of the original material is diminished or has been covered by a secondary coating. The coating is particularly well developed in the image at the lower right of Figure 3-10.

The EDS spectra for the as-received and post-test Microtherm samples (Figure 3-11) also indicate significant changes in the surface composition of the Microtherm samples in the leaching tests at pH 12. The EDS analyses are based on small and perhaps heterogeneous surface locations, but the post-test spectra for both of the leaching tests at pH 12 clearly indicate that the silicon concentration at the sample surface is greatly diminished compared to the as-received sample. As silicon was lost from the sample surface, the relative concentration of titanium (likely present as a refractory oxide in the as-received sample material) increased in the post-test samples. The EDS spectrum for the post-test sample at 190 °F [87.8 °C], shown at the bottom of Figure 3-11, also shows a spike in sodium concentration on the sample surface.

The Microtherm sample from the 87.8 °C [190 °F] leaching test at pH 12 also included a transparent white solid about 3 mm [0.12 in] across that was attached to a small tan fragment of more typical Microtherm (Figure 3-12). The solid was not noted until after the sample was air dried, and its time of formation is unknown. The transparent white solid was tentatively identified by XRD analysis (Figure 3-13) as thermonatrite, a relatively soluble sodium carbonate mineral ($\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$) that occurs in evaporite deposits and in volcanic hydrothermal deposits. The XRD analysis is supported by EDS analysis of the sample surface, which indicates that it is composed primarily of sodium, carbon, and oxygen.



**CalSil, 87.8 °C
[190 °F], pH 8**



**Fiberglass, 129 °C
[265 °F], pH 4**



**Temp-Mat, 129 °C
[265 °F], pH 12**

Figure 3-1. Photos of Cloudy Post-Test Leachates (CalSil and Fiberglass) and Example of Clear Post-Test Leachate (Temp-Mat)

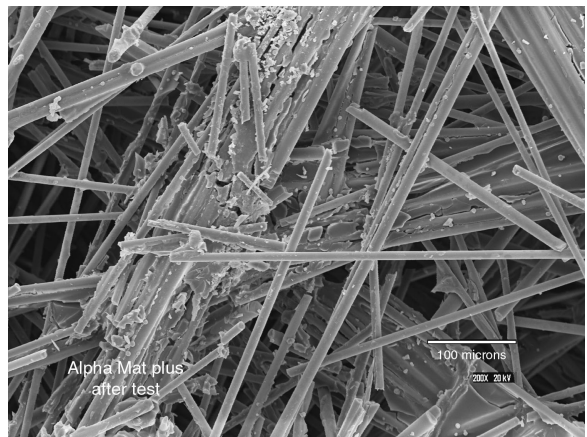
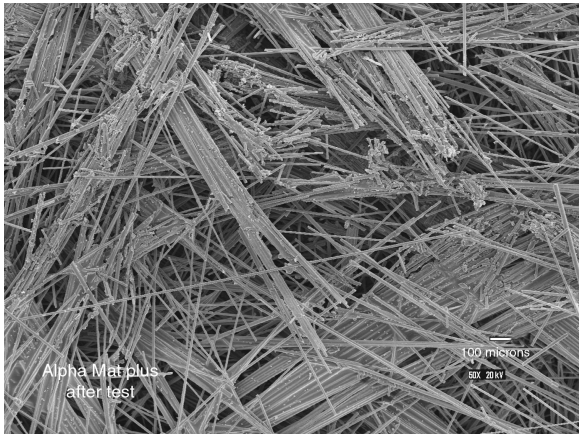
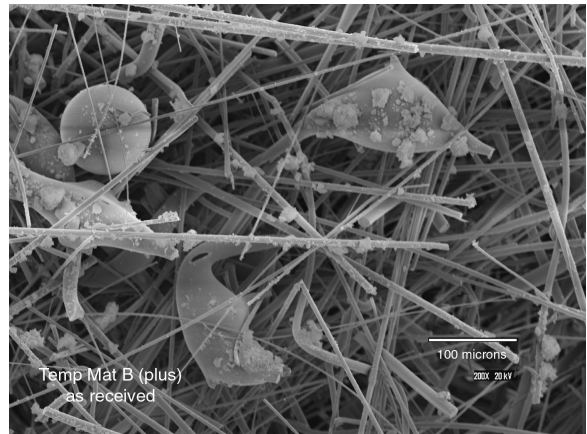
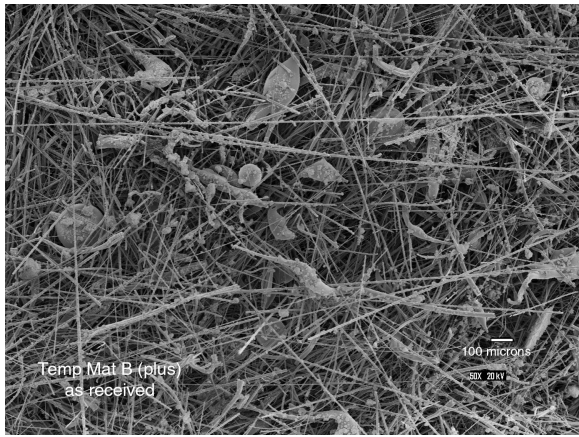


Figure 3-2. Images of AlphaMat Plus [“Temp-Mat B (Plus)”] Sample Material, as Received and After Leaching for 90 Minutes in pH 12 Borated Water at 129 °C [265 °F]

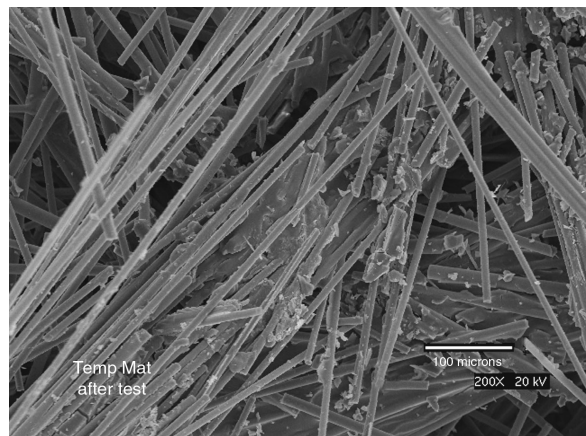
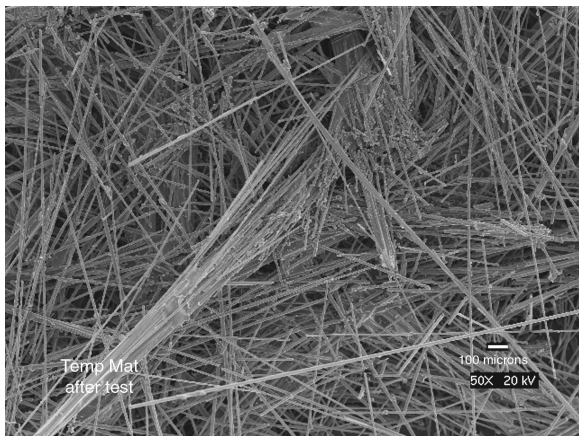
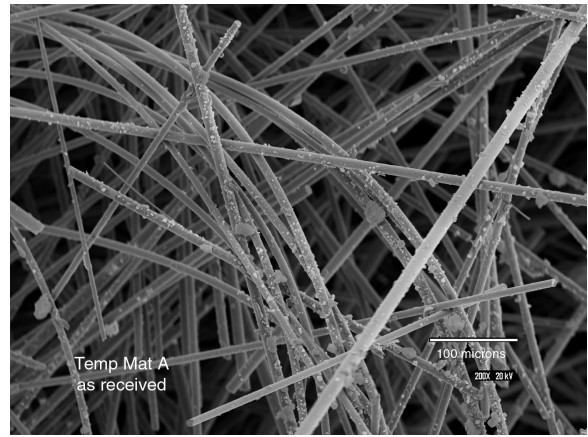
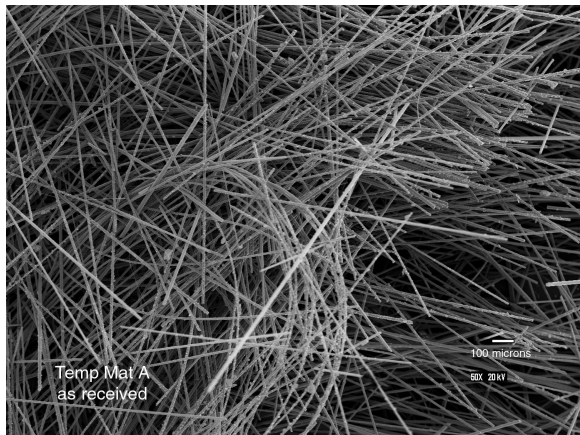


Figure 3-3. Images of Temp-Mat Sample Material, as Received and After Leaching for 90 Minutes in pH 12 Borated Water at 129 °C [265 °F]

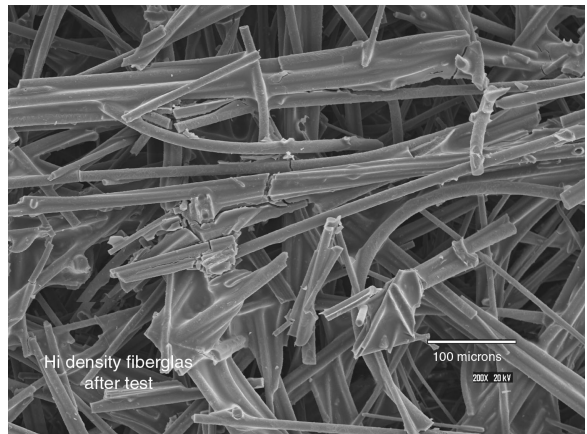
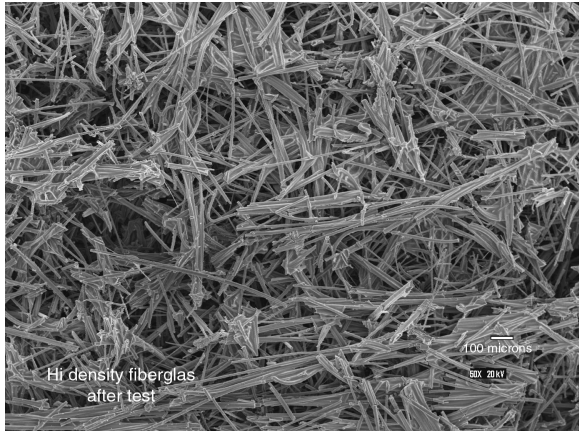
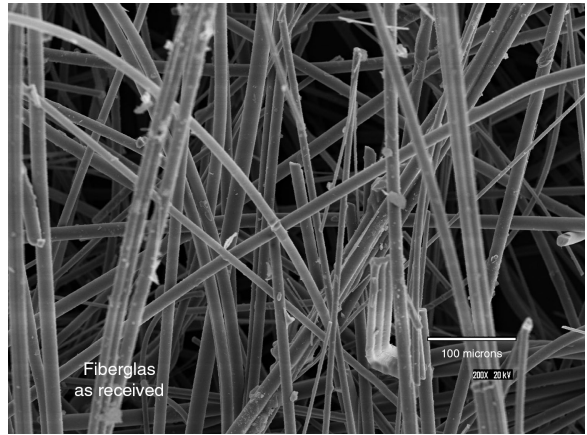
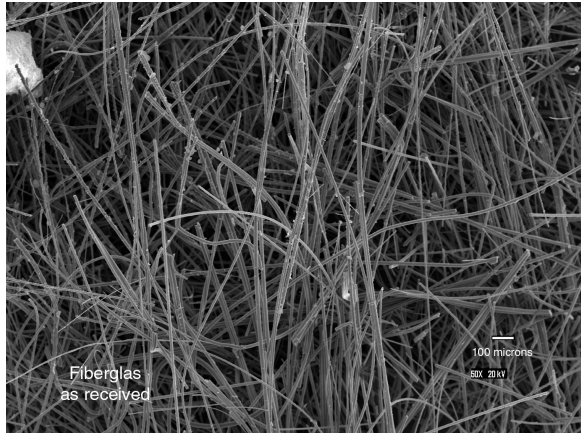


Figure 3-4. Images of Fiberglass (“High Density”), as Received and After Leaching for 90 Minutes in pH 12 Borated Water at 129 °C [265 °F]

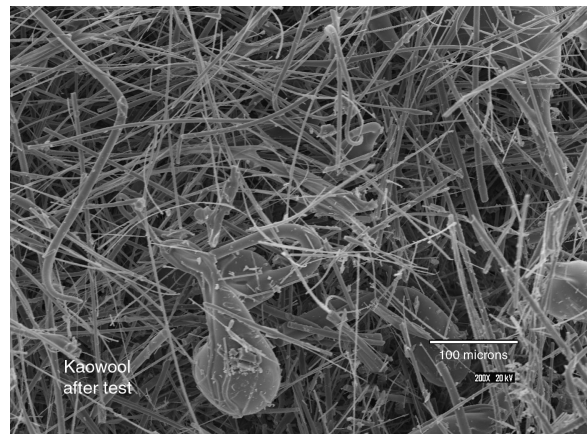
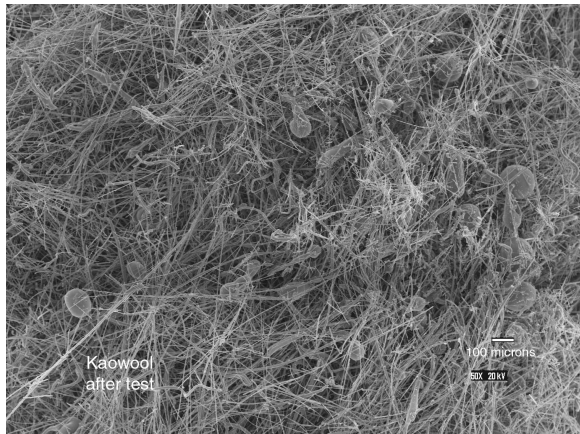
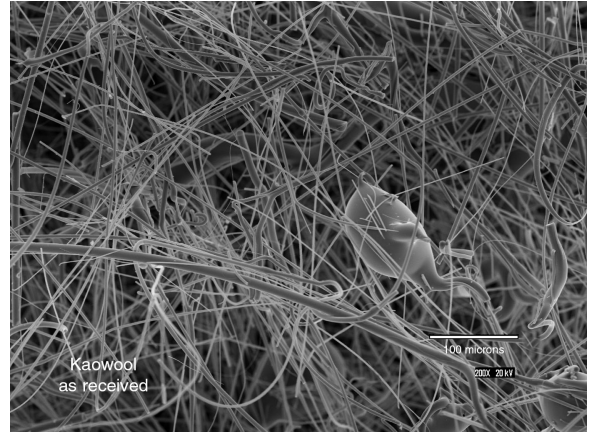
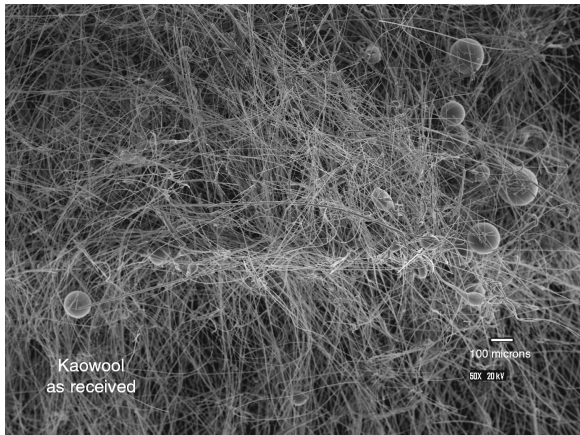


Figure 3-5. Images of Kaowool Sample Material, as Received and After Leaching for 90 Minutes in pH 12 Borated Water at 129 °C [265 °F]

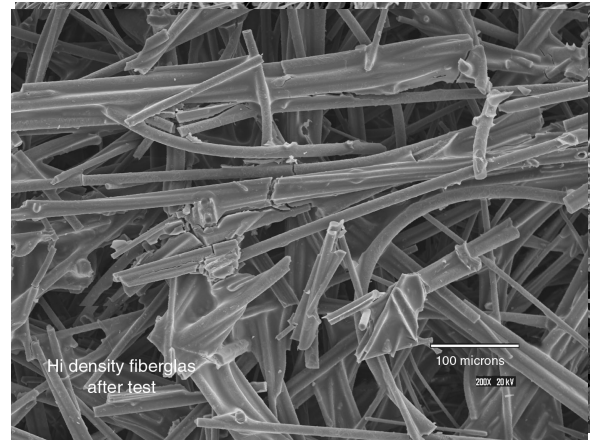
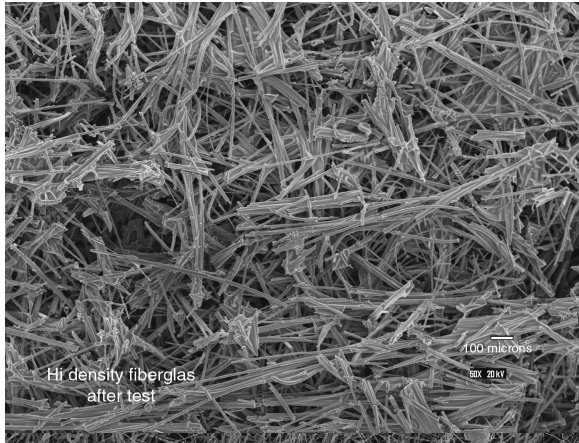
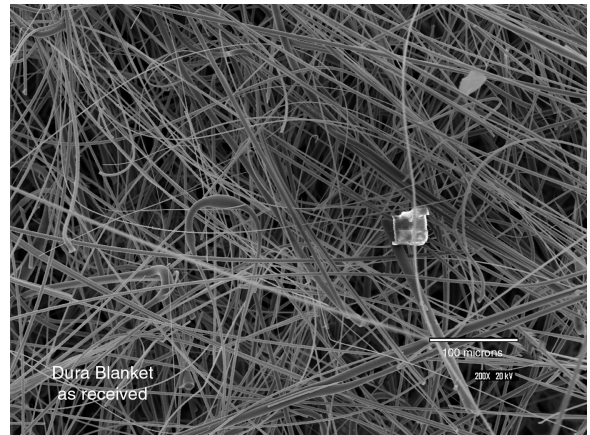
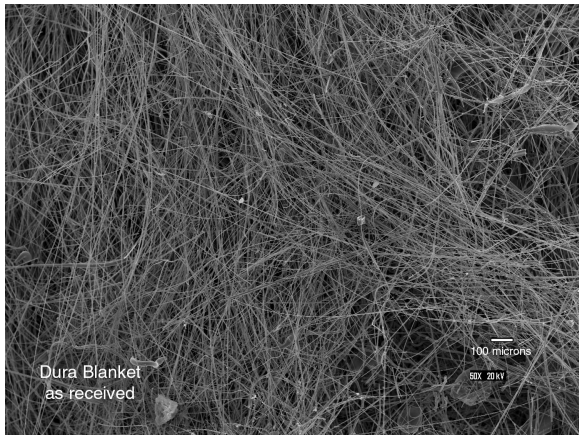


Figure 3-6. Images of Durablanket Sample Material, as Received and After Leaching for 90 Minutes in pH 12 Borated Water at 129 °C [265 °F]

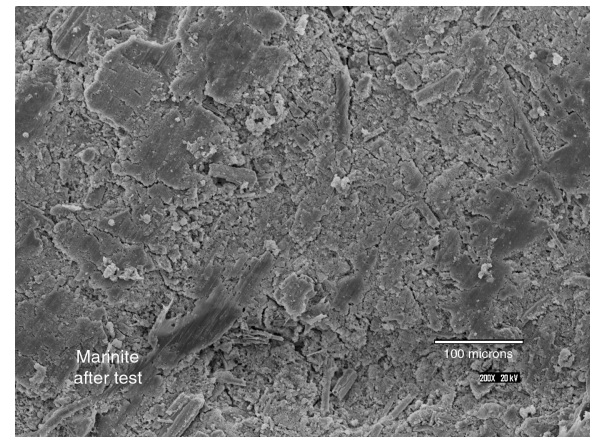
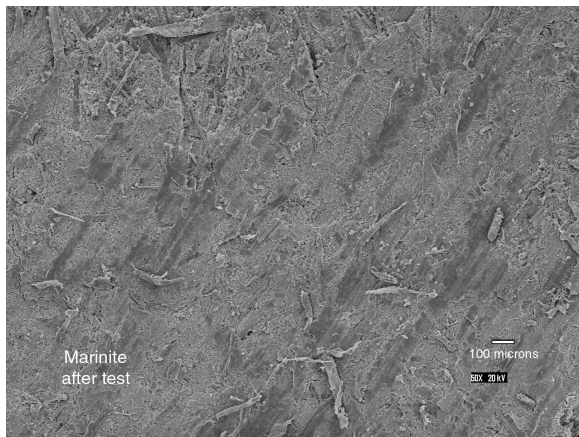
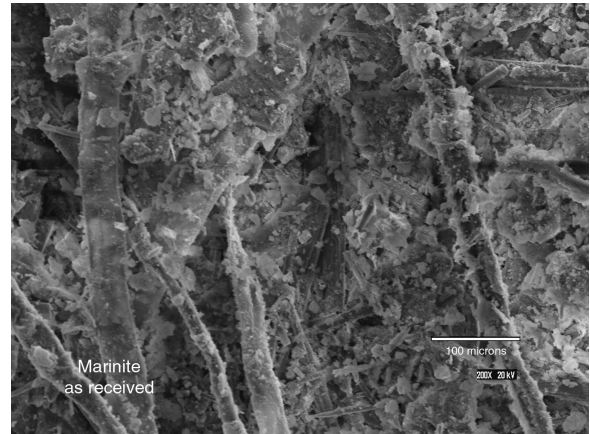
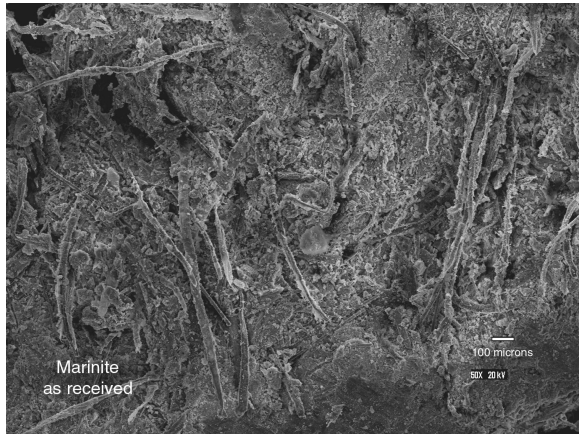


Figure 3-7. Images of Marinite, as Received and After Leaching for 90 Minutes in pH 8 Borated Water at 87.8 °C [190 °F]

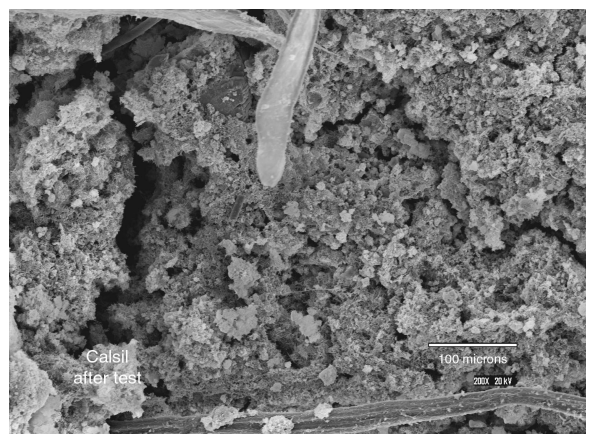
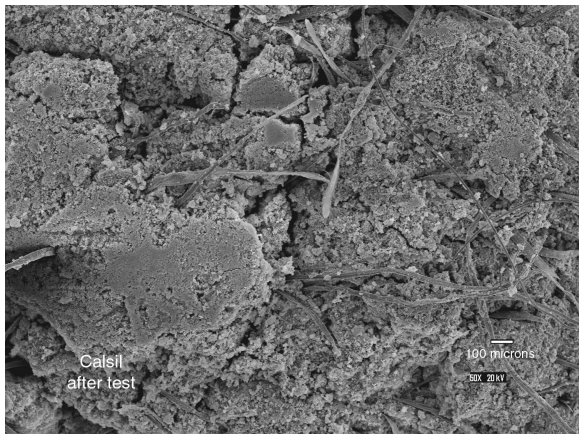
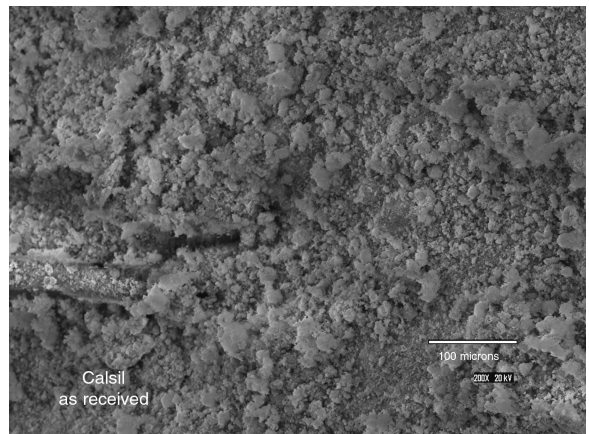
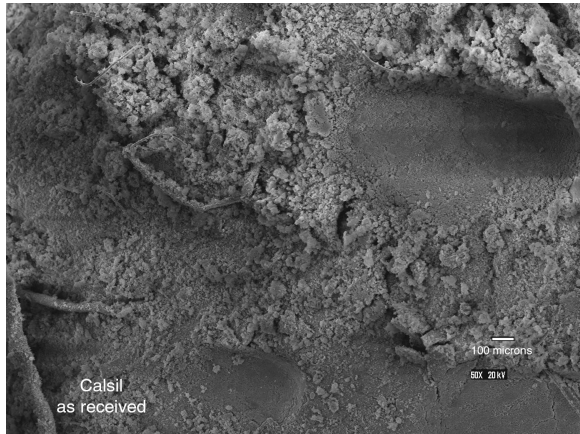


Figure 3-8. Images of CalSil Sample Material, as Received and After Leaching for 90 Minutes in pH 8 Borated Water at 87.8 °C [190 °F]

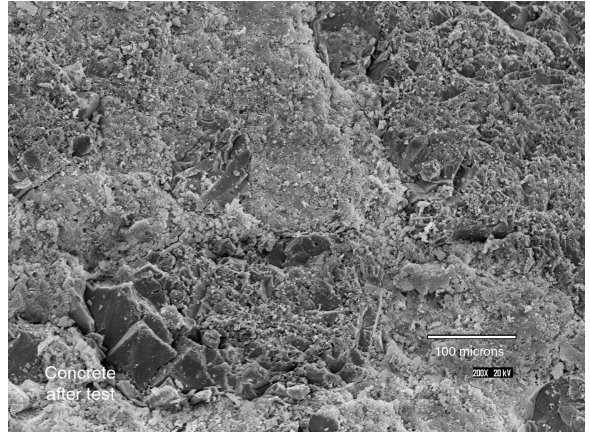
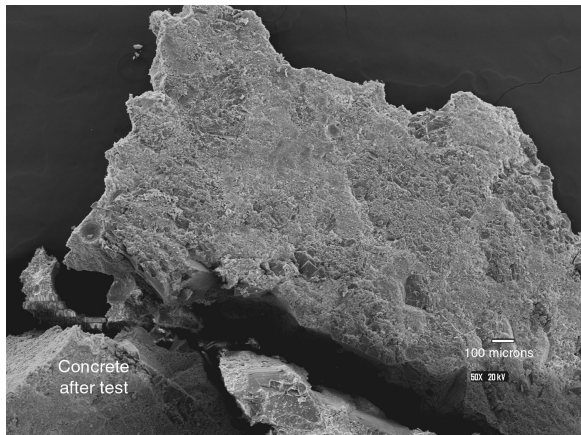
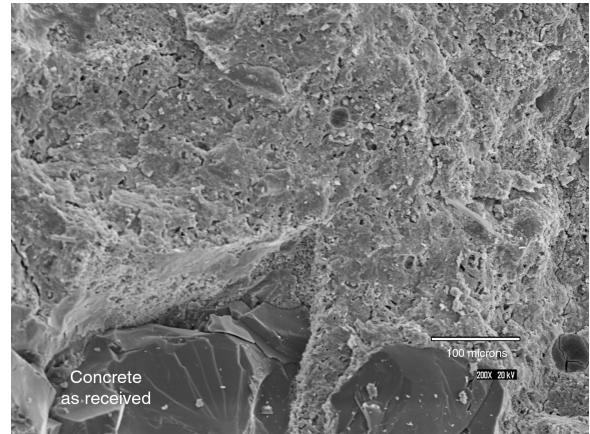
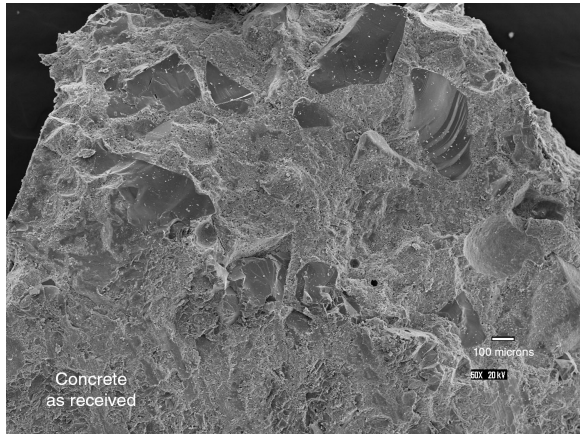


Figure 3-9. Images of Concrete Sample Material, as Received and After Leaching for 90 Minutes in pH 4.1 Borated Water at 87.8 °C [190 °F]

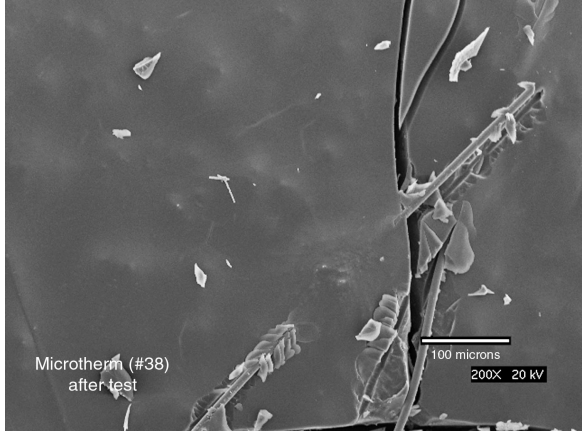
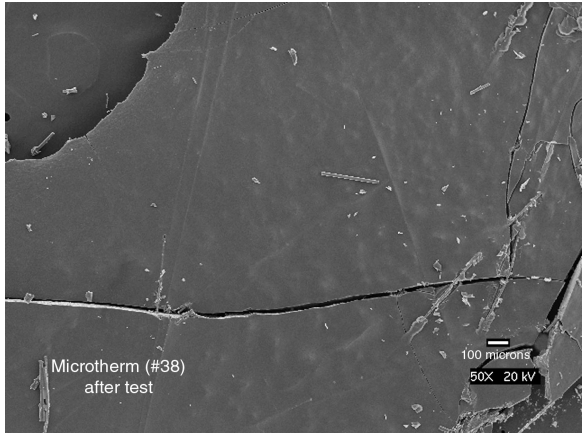
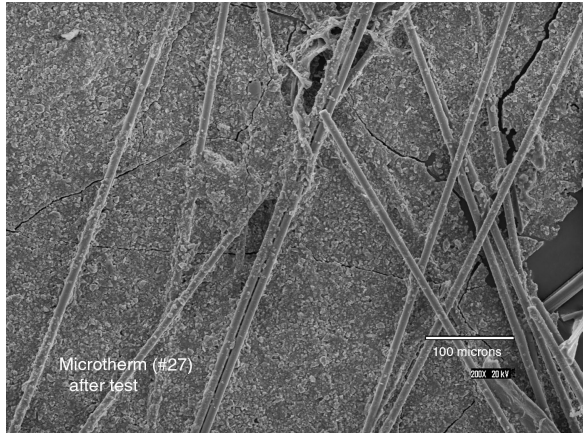
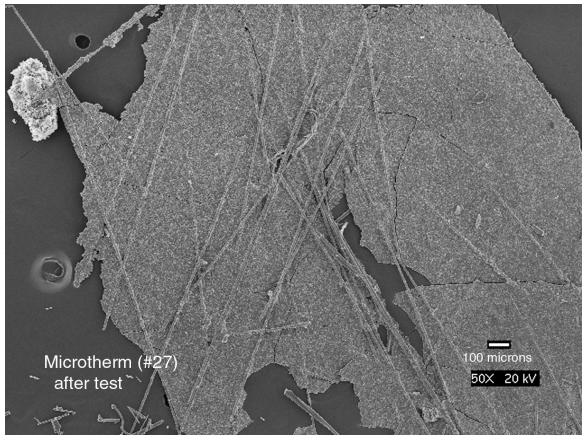
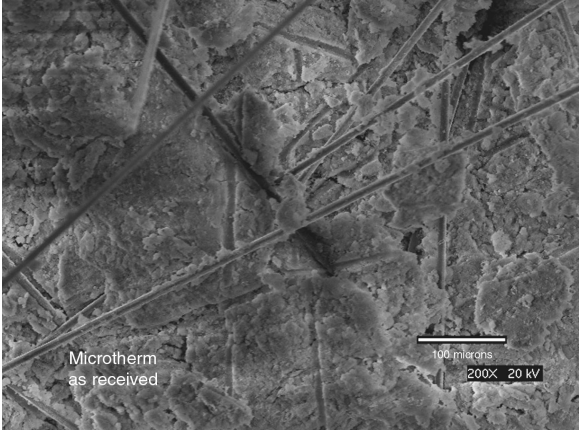
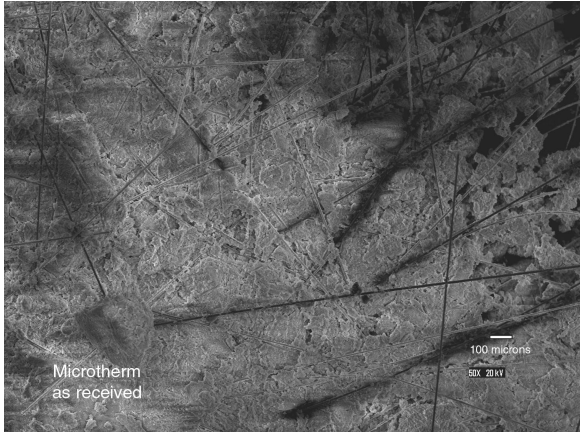


Figure 3-10. Images of Microtherm Sample Material, as Received (Top Row) and After Leaching for 90 Minutes in pH 12 Borated Water at 129 °C [265 °F] (Center Row) and at 87.8 °C [190 °F] (Bottom Row)

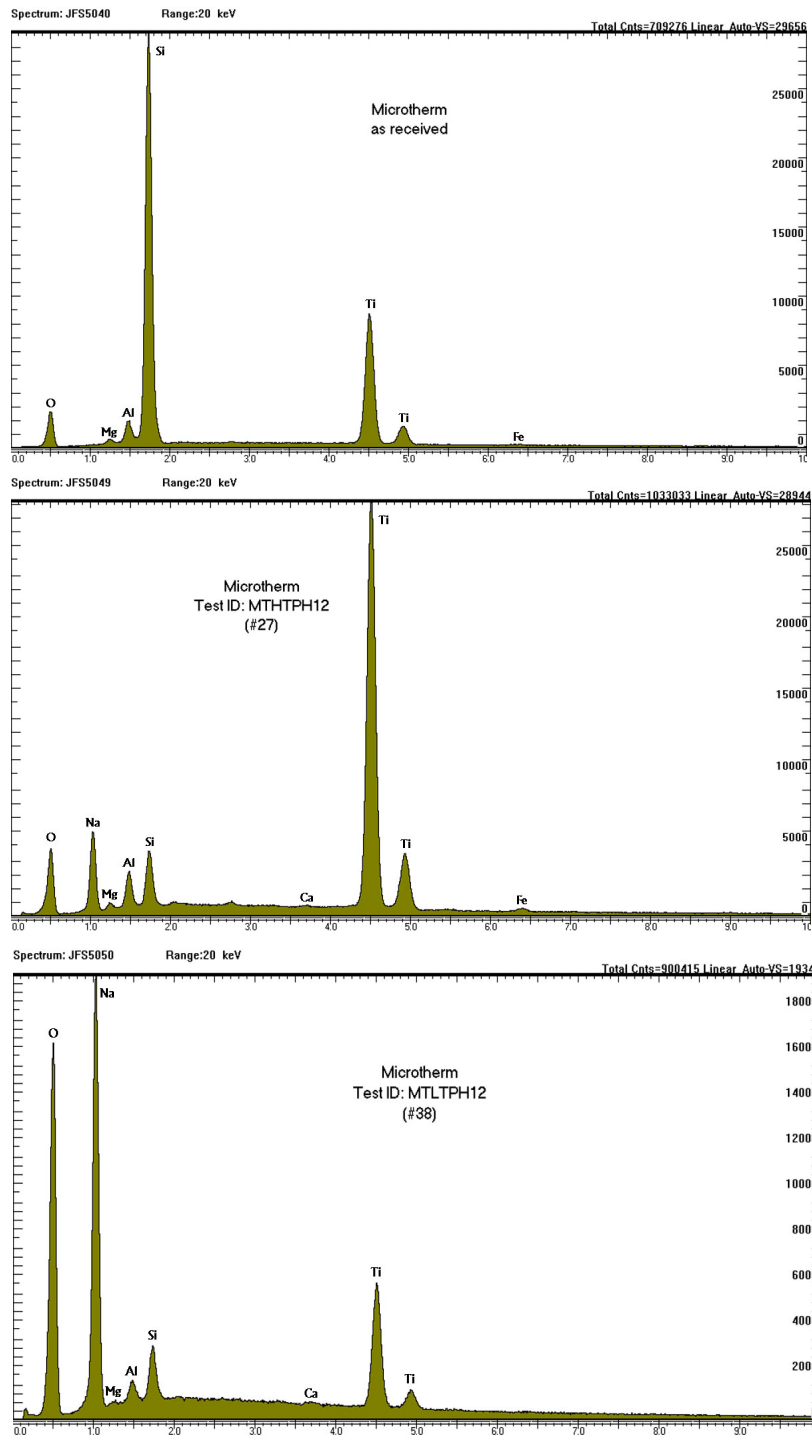


Figure 3-11. EDS Spectra for Microtherm Sample, as Received and After Leaching for 90 Minutes in pH 12 Borated Water at 129 °C [265 °F] (MTHTPH12) and at 87.8 °C [190 °F] (MLTPH12)

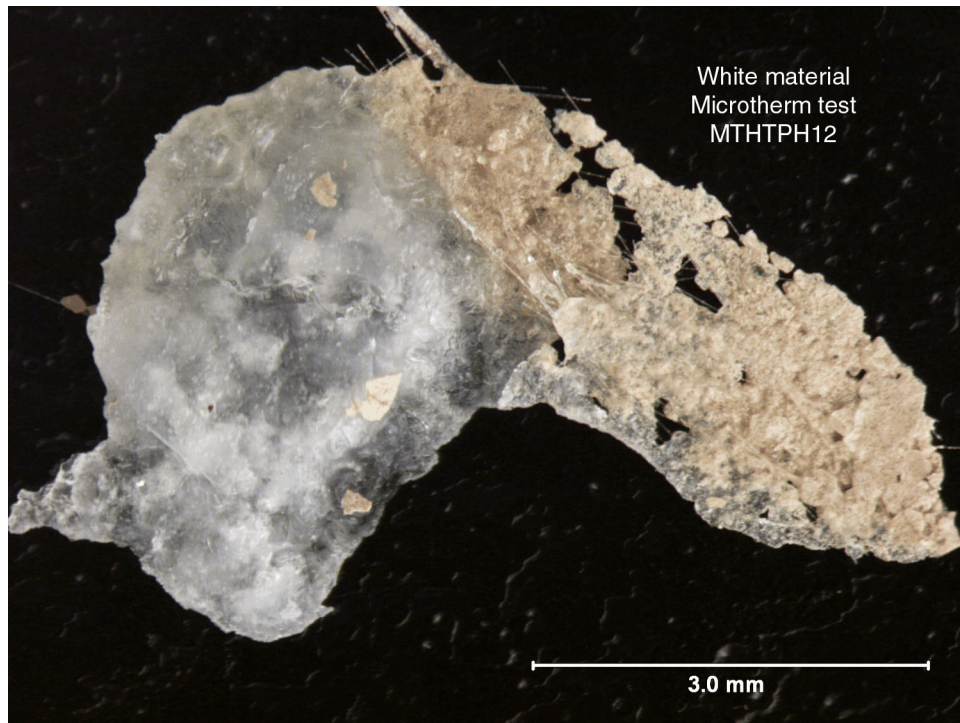


Figure 3-12. Transparent White Solid Adhering to Microtherm Sample After Leaching for 90 Minutes in pH 12 Borated Water at 129 °C [265 °F]

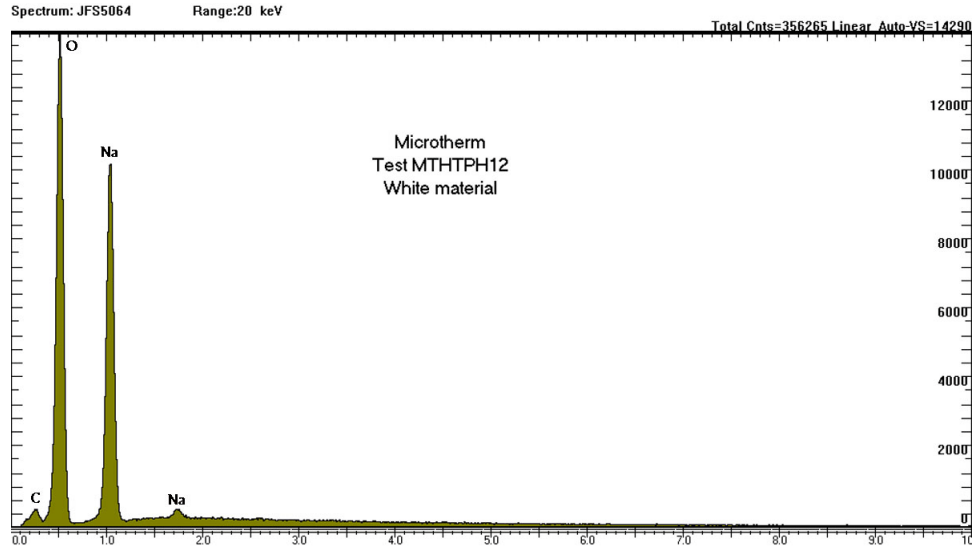
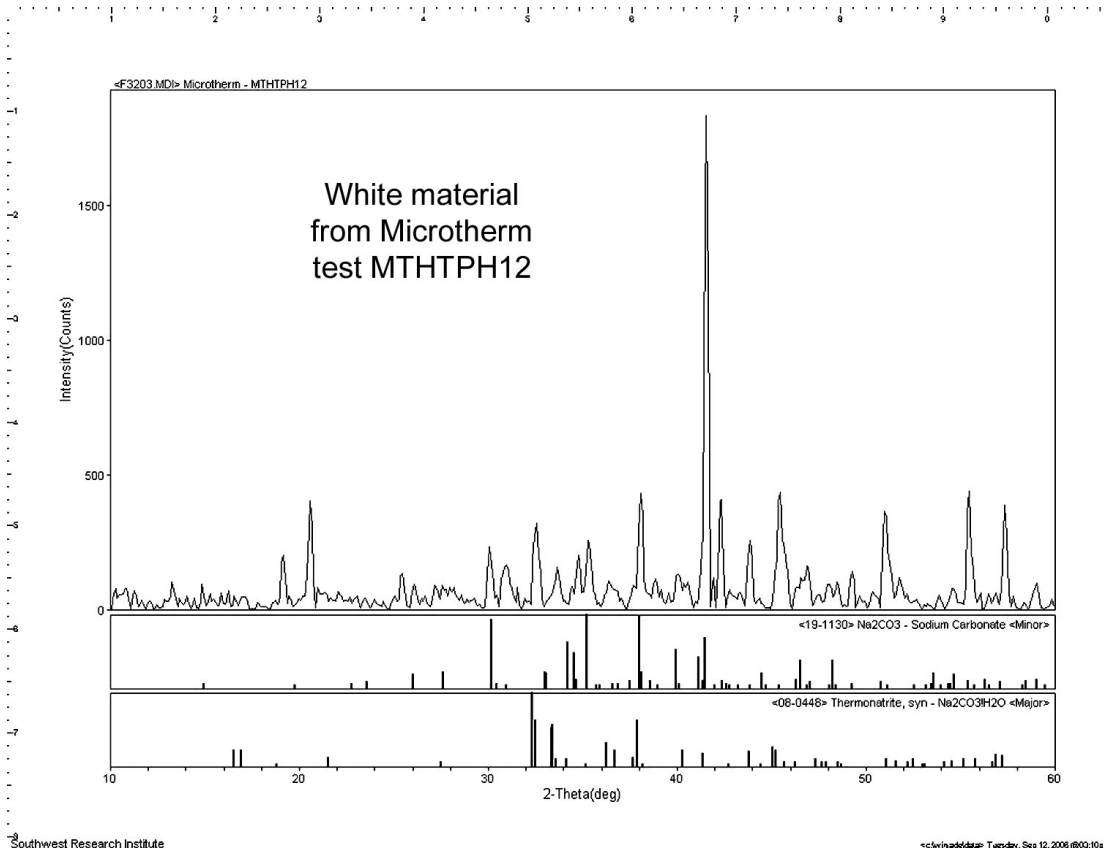


Figure 3-13. XRD (Top) and EDS (Bottom) Spectra for White Transparent Solid (See Figure 3-12) in Microtherm Leaching Test at pH 12 and 129 °C [265 °F]

4 DISCUSSION AND CONCLUSIONS

The Westinghouse dissolution and precipitation experiments (Westinghouse Electric Company, LLC, 2006) provided data for estimates of the type and amounts of chemical precipitates which may form under a post-LOCA conditions. For testing purposes, the Westinghouse study grouped various insulation products into several material classes on the basis of their common physical and chemical properties and selected one or two representative products from each class for dissolution and precipitation experiments.

The main objective of the CNWRA leaching tests was to examine the assumption that sample materials selected in the previous tests were sufficiently representative that their dissolution characteristics could be generalized from one product to the others in the same materials class. The CNWRA tests used different products from the material classes and exposed them to experiment conditions that were similar to those in the Westinghouse tests. Some of the CNWRA tests used the same materials that had been tested by Westinghouse, in order to compare results more directly. This approach provided an opportunity to attempt to reproduce specific Westinghouse tests that had resulted in the formation of precipitates. The precipitated phases in the Westinghouse tests had been tentatively identified on the basis of EDS analysis. A second objective of the CNWRA tests was to characterize any observed precipitates in more detail using techniques such as XRD analysis. Chemical modeling, as applied in a previous GSI-191 study (McMurry, et al., 2006), also was used to obtain a better understanding of the leachate solutions and their saturation state with respect to solid phases that would be reasonably expected to form under the test conditions. A third objective was to obtain leaching test data for concrete with a sample-to-solution ratio that was more representative of actual plant conditions than the ratio that had been used in the Westinghouse tests.

Overall, the results of the CNWRA leaching tests did not contradict the assumption that the chemical properties of insulation products in a given materials class were similar enough that the leaching behavior of a single product was representative of all products in the class. The concentrations of elements in the CNWRA test leachates were similar to or less than the concentrations in the Westinghouse test leachates. When the CNWRA tests and the Westinghouse tests were based on the same insulation product, the CNWRA leachate concentrations tended to be lower. The CalSil leaching test at 87.8 °C [190 °F] and pH 4.1 was the exception; the sample tested by CNWRA appeared to be significantly more reactive than the CalSil sample tested by Westinghouse (Table 3-5). A large disparity in dissolved silicon and calcium concentrations also was observed for the CalSil leaching tests at pH 8, but in this case, the Westinghouse sample was more reactive in terms of silicon loss, and the CNWRA sample was more reactive in terms of calcium loss.

No precipitates were observed to have settled out in any of the CNWRA tests, even using the same materials and test conditions in which precipitates formed in the Westinghouse tests. In general, the leachates produced in the CNWRA tests were more dilute than those in the Westinghouse tests, and so it was less likely that any secondary solids approached saturation with respect to the leachate composition. Although the test conditions, sample materials, and procedures were similar in both sets of experiments, several differences may have affected the results. For example, in the Westinghouse experiments the test apparatus was more elaborate and included a shaker mechanism inside the oven, whereas the CNWRA test vessels were kept stationary in the oven to minimize disaggregation of the insulation samples. The agitation in the Westinghouse tests may have promoted more dissolution by stirring or by hindering the

formation of passive coatings on the insulation material. In addition, some of the variability in leachate compositions may have resulted from initial differences between the insulation samples as obtained from suppliers. Industrial insulation is typically developed to meet physical, not chemical, performance criteria. Some variation in chemical composition or additives is possible between batches of the same product. Other differences in dissolution properties may have resulted from undocumented differences in the age or storage conditions (e.g., thermal exposure) prior to testing.

Cooling rates also may have affected the formation of precipitates. However, all other factors being equal, the CNWRA test procedures should have promoted precipitation of oversaturated phases instead of hindering it. Compared to the Westinghouse leachates, which were transferred immediately from the test oven to a room temperature {27 °F [80 °C]} water bath as soon as the dissolution experiment ended, the CNWRA leachates were cooled to ambient temperatures inside the oven for approximately two hours. The slightly slower cooling rate would favor the formation of fewer but larger particles that settled from solution. Moreover, the CNWRA leachates remained in their test vessels throughout the two-hour cooling period, which increased the total amount of time that the water was exposed to the insulation sample. To the extent that an insulation sample was reactive at the lower temperatures, this extended exposure time would tend to increase the concentration of the leachate. Even so, the CNWRA solutions typically were still more dilute than those observed for the equivalent Westinghouse tests (see Tables 3-2 through 3-5).

Relative to the amount of sample tested, Microtherm at pH 12 was the most reactive insulation material, and its surface texture was visibly affected by leaching. A transparent sodium carbonate phase was associated with the degraded post-test Microtherm sample in one case. The origin of this material and its potential effect on sump screen performance under post-LOCA conditions are undetermined.

The leachate concentrations for the solid concrete coupons were consistently more dilute than the leachate concentrations measured in the Westinghouse tests. This result was expected because an unrealistically large material-to-coolant ratio had been assumed for the crushed concrete samples in the Westinghouse tests. Based on responses to the PWR Owners Group survey for PWR operators (NRC, 2004), the material-to-coolant ratio of 15.72 m²/m³ [4.79 ft²/ft³] for the solid concrete coupons represents a conservative upper limit of the amount of concrete that would be exposed to water under post-LOCA conditions. No oversaturated solid phases were observed to precipitate or were indicated by modeling to be oversaturated in the leachates from the CNWRA tests. However, the amount of calcium released to solution was still relatively high, particularly in the test at low pH conditions, compared to some of the insulation materials that were tested at similar pH in this study. Combined with other materials in a post-LOCA environment, concrete dissolution could still contribute to the formation of secondary precipitates in some circumstances.

Although precipitation tests involving trisodium phosphate as a pH buffer were not included in the present study, the evaluation of calcium leaching from containment materials such as CalSil, Marinite, and concrete is particularly important to plants using trisodium phosphate because calcium phosphate precipitates can form under these conditions (e.g., Dallman, et al., 2006).

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APPENDIX

ENERGY-DISPERSIVE X-RAY SPECTROGRAPHS OF SAMPLE SURFACE COMPOSITIONS BEFORE AND AFTER LEACHING TESTS

The spectra on the following pages are a comparison of as-received sample materials and post-leaching test samples. The test identification (ID) numbers on each graph correspond to the ID numbers in the test matrix in Table 2-2 of this report. Leaching conditions for each post-test sample are indicated in the figure caption.

Note:

EDS = energy-dispersive x-ray spectroscopy

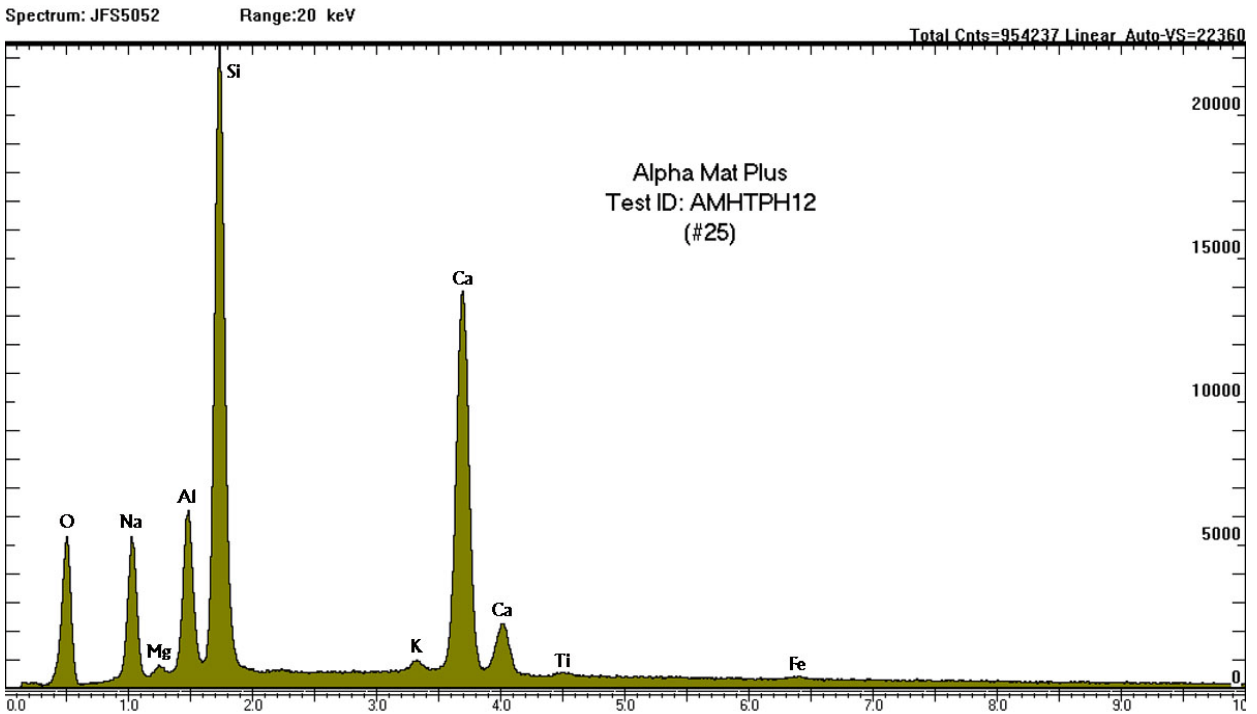
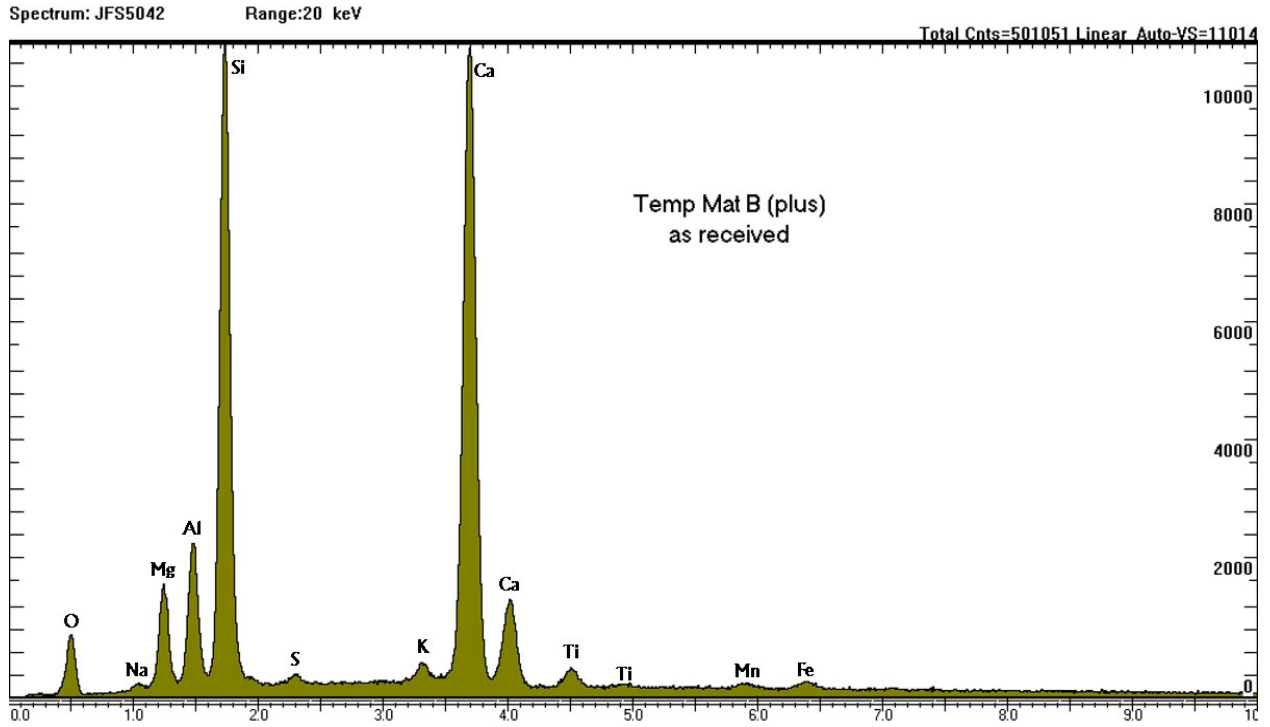


Figure 1. EDS Spectra for AlphaMat (“Plus”) [“Temp-Mat B (Plus)”] Sample Before and After Leaching in pH 12 Borated Water at 129 °C [265 °F] for 90 Minutes

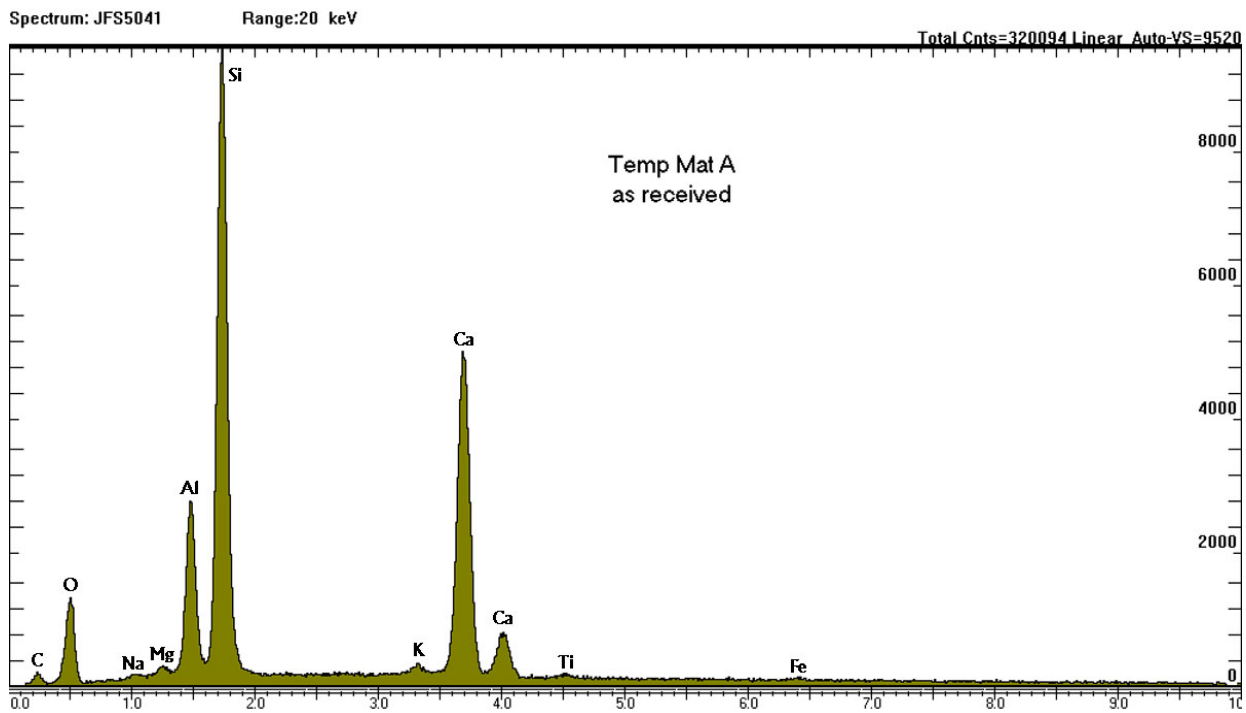


Figure 2. EDS Spectra for Temp-Mat Sample Before and After Leaching in pH 12 Borated Water at 129 °C [265 °F] for 90 Minutes

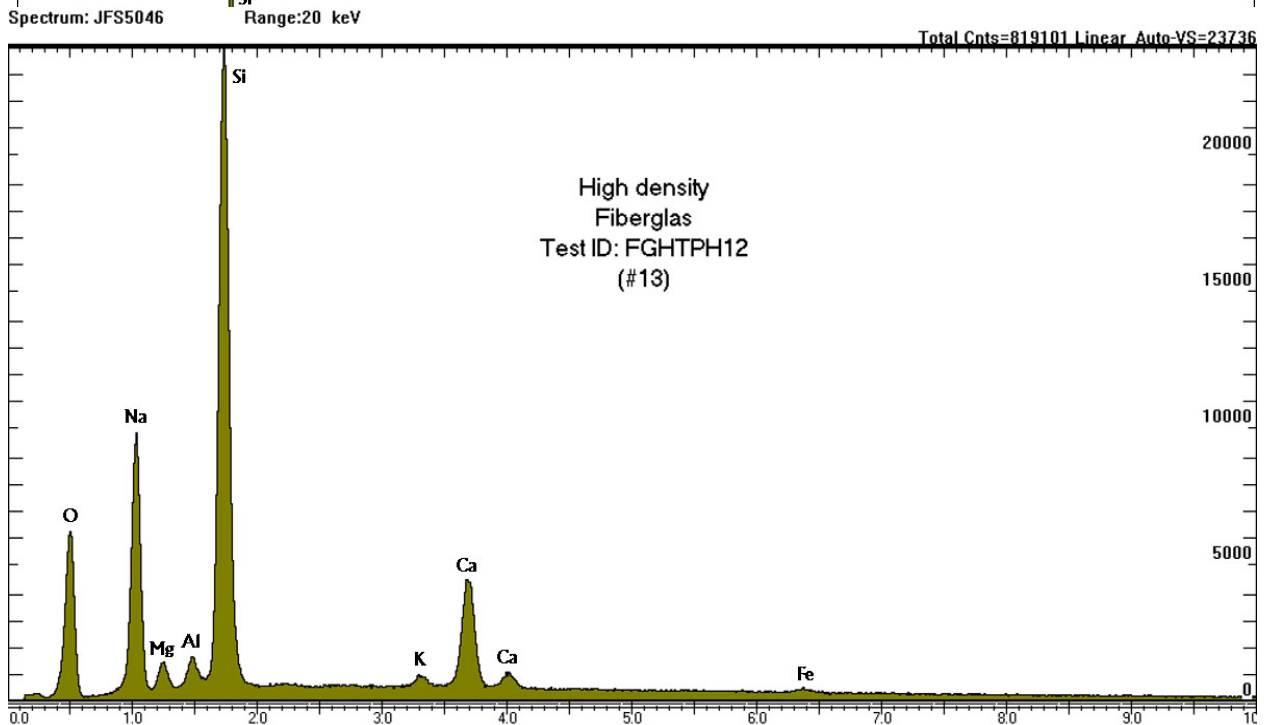
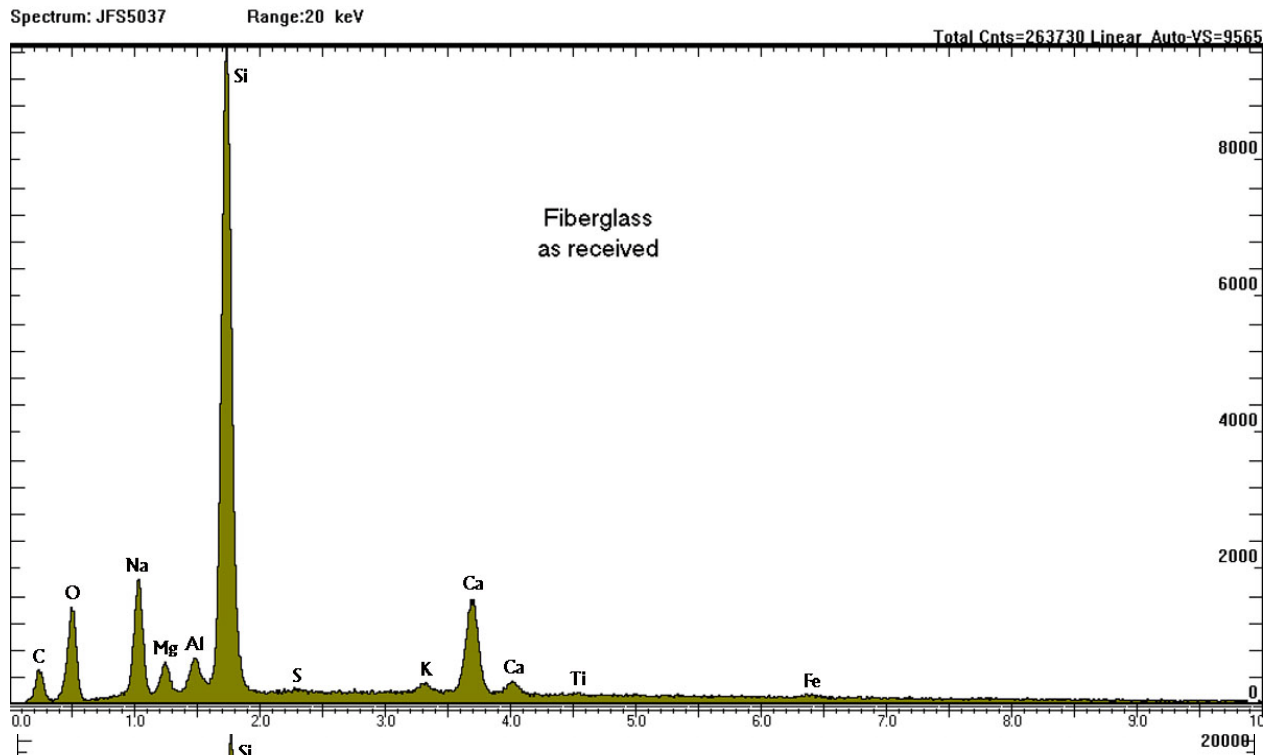


Figure 3. EDS Spectra for Fiberglass Sample (“High Density”) Before and After Leaching in pH 12 Borated Water at 129 °C [265 °F] for 90 Minutes

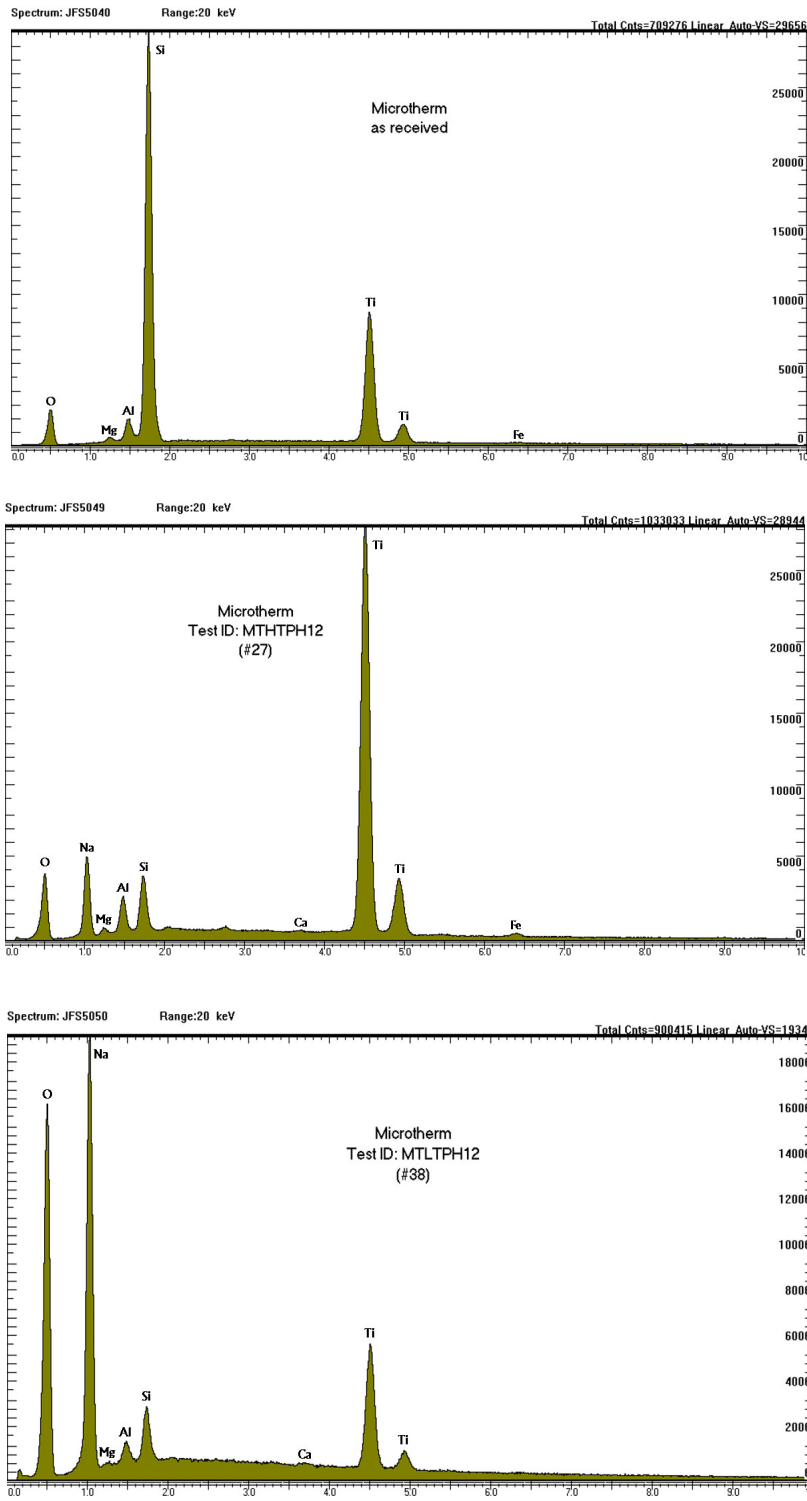


Figure 4. EDS Spectra for Microtherm Sample, as Received and After Leaching in pH 12 Borated Water at 129 °C [265 °F] (MTHTPH12) and at 87.8 °C [190 °F] (MTLTPH12) for 90 Minutes

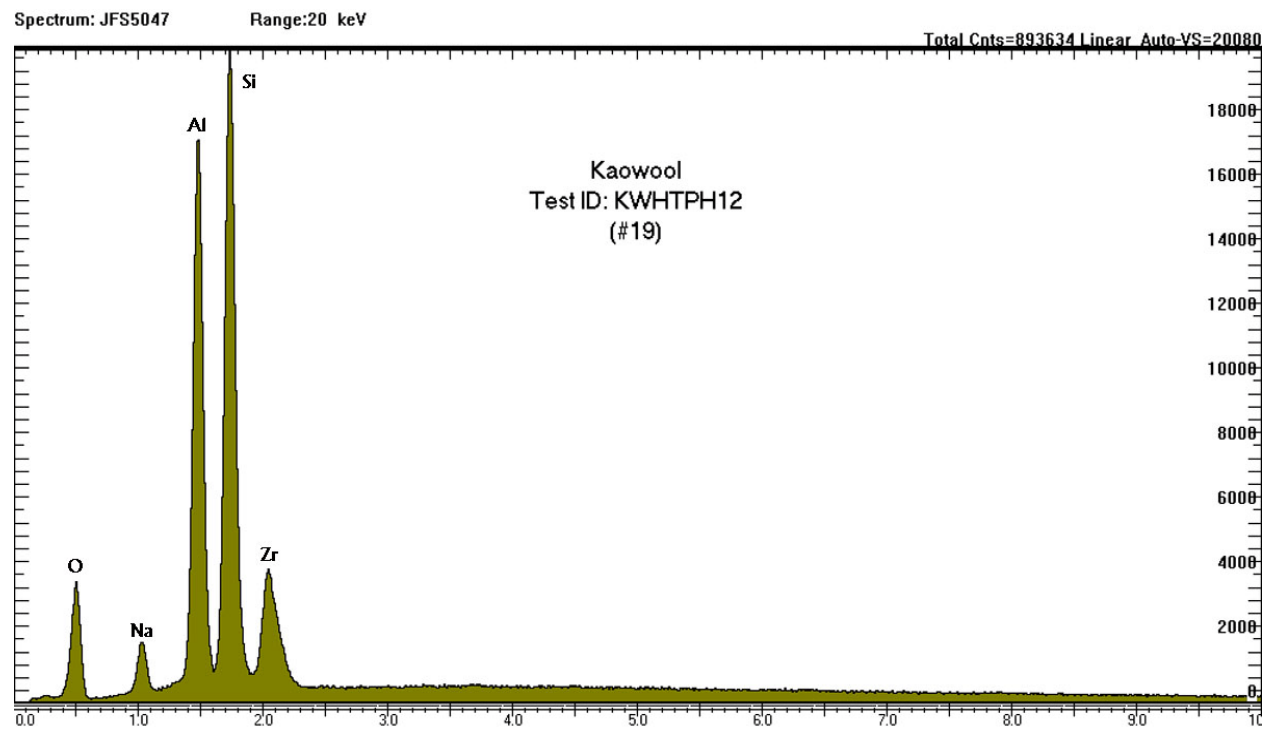
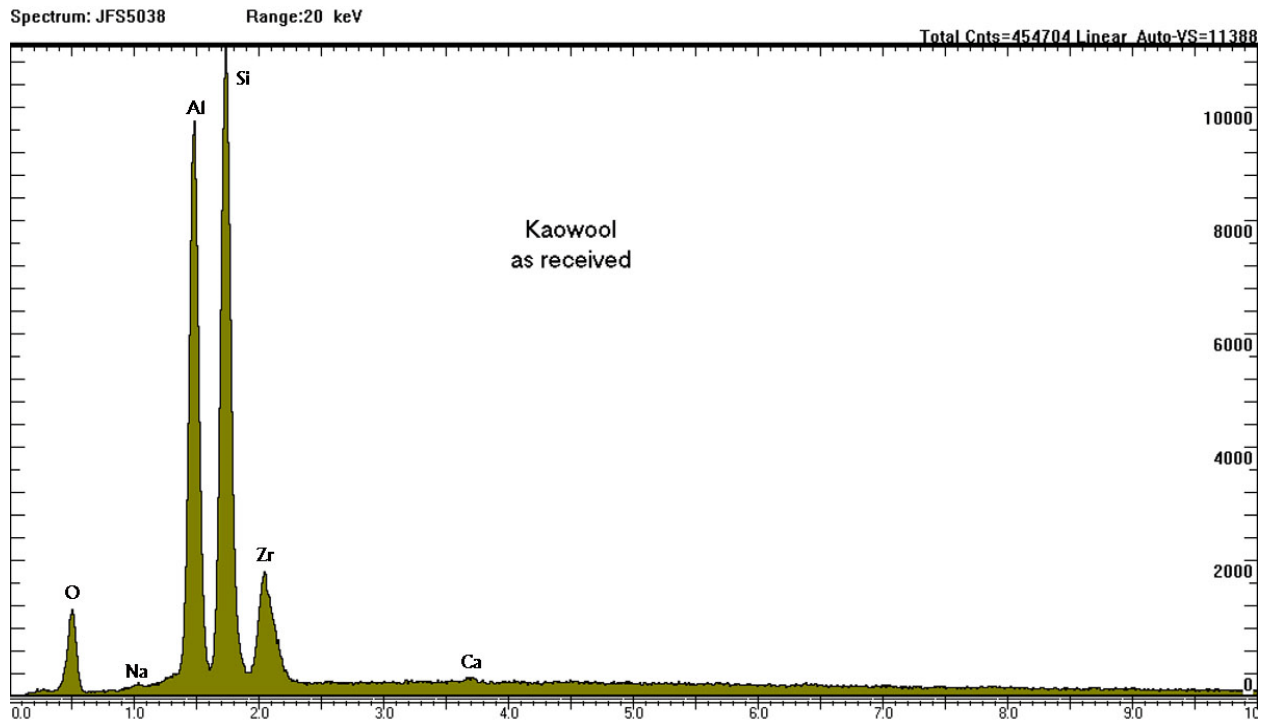


Figure 5. EDS Spectra for Kaowool Sample Before and After Leaching in pH 12 Borated Water at 129 °C [265 °F] for 90 Minutes

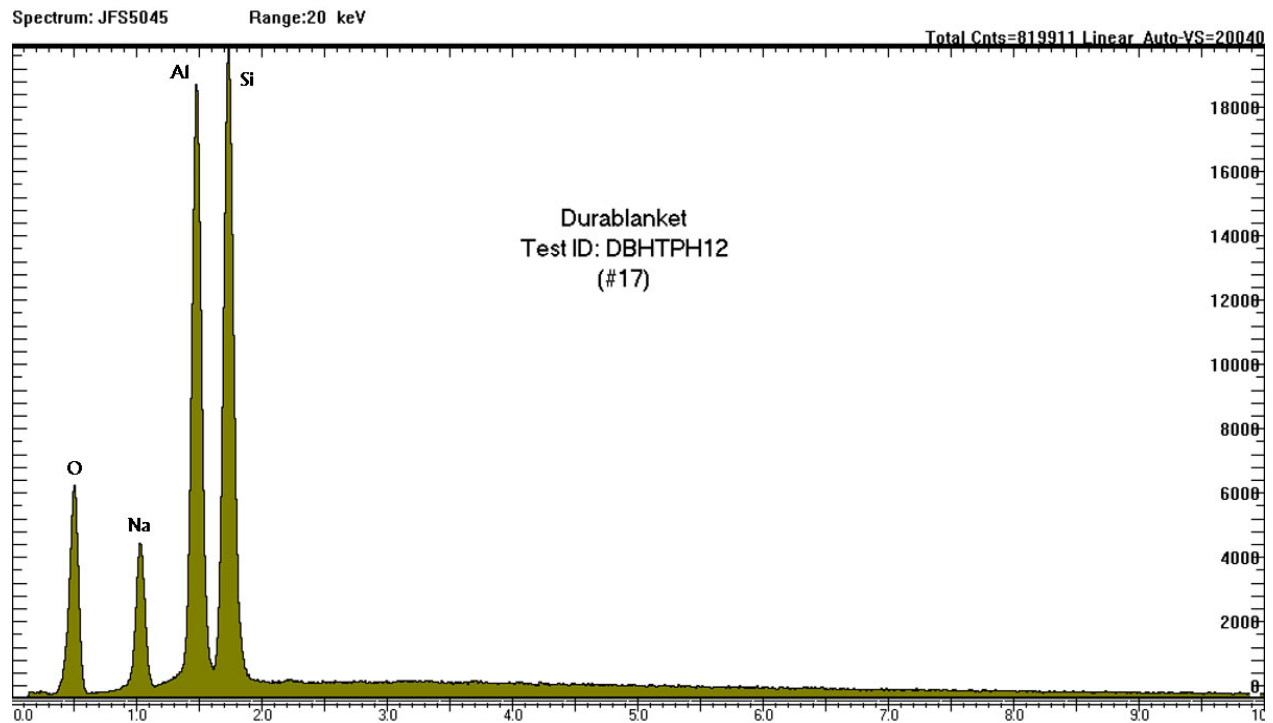
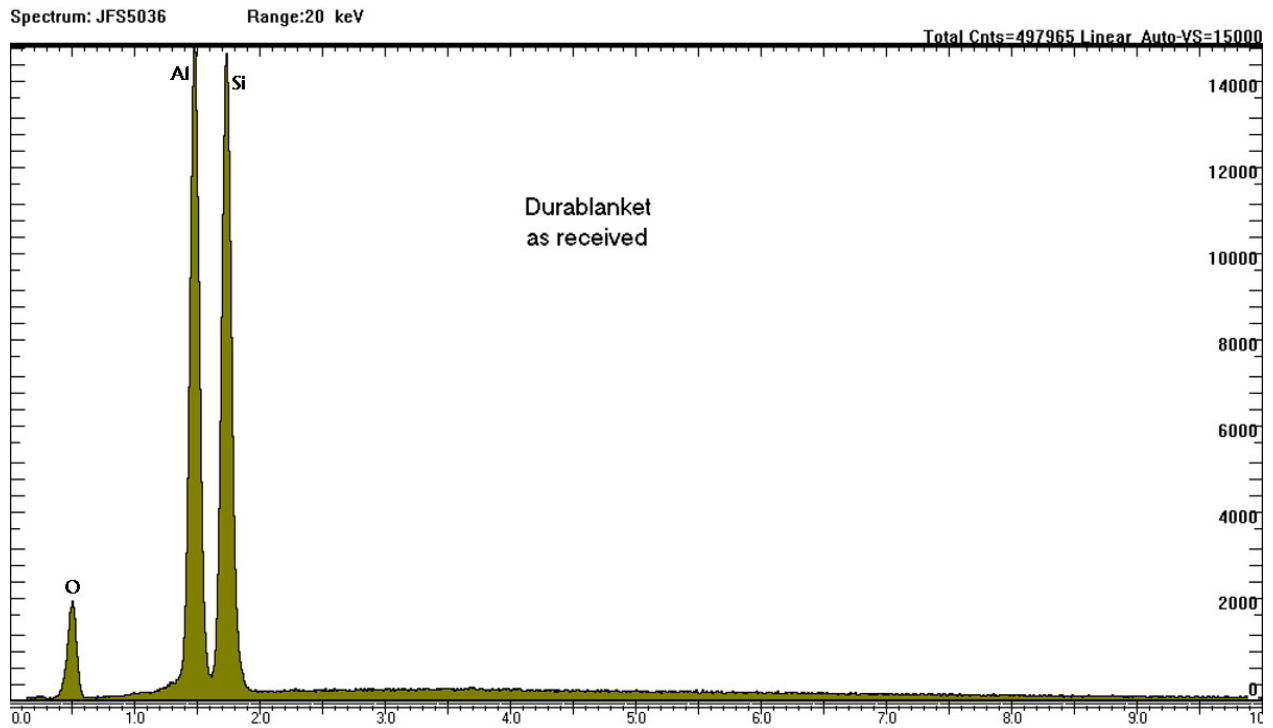
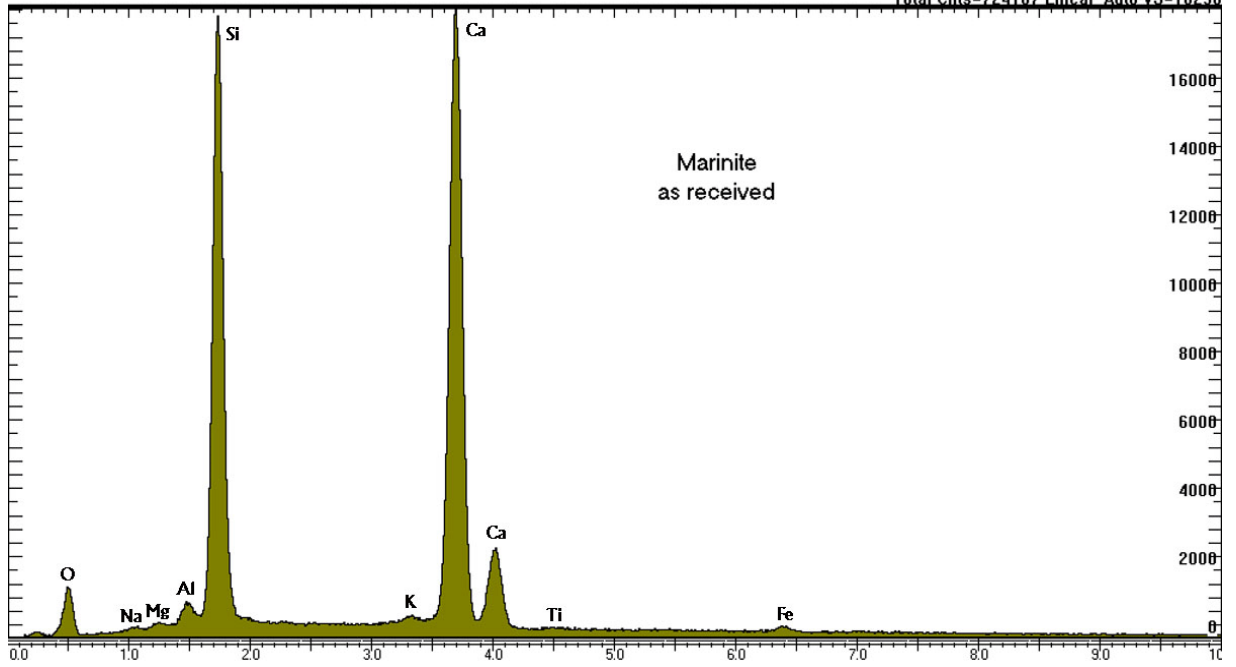


Figure 6. EDS Spectra for High-Density Durablanket Sample Before and After Leaching in pH 12 Borated Water at 129 °C [265 °F] for 90 Minutes

Spectrum: JFS5039

Range:20 keV

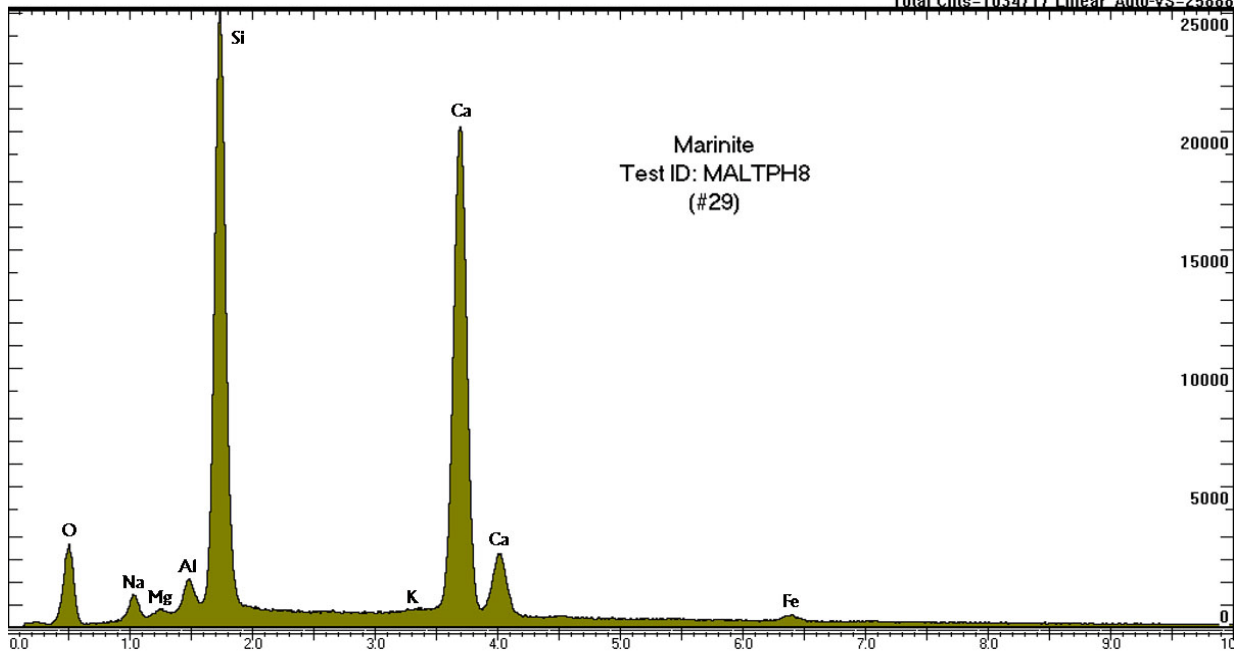
Total Cnts=724167 Linear Auto-VS=18296



Spectrum: JFS5048

Range:20 keV

Total Cnts=1034717 Linear Auto-VS=25888



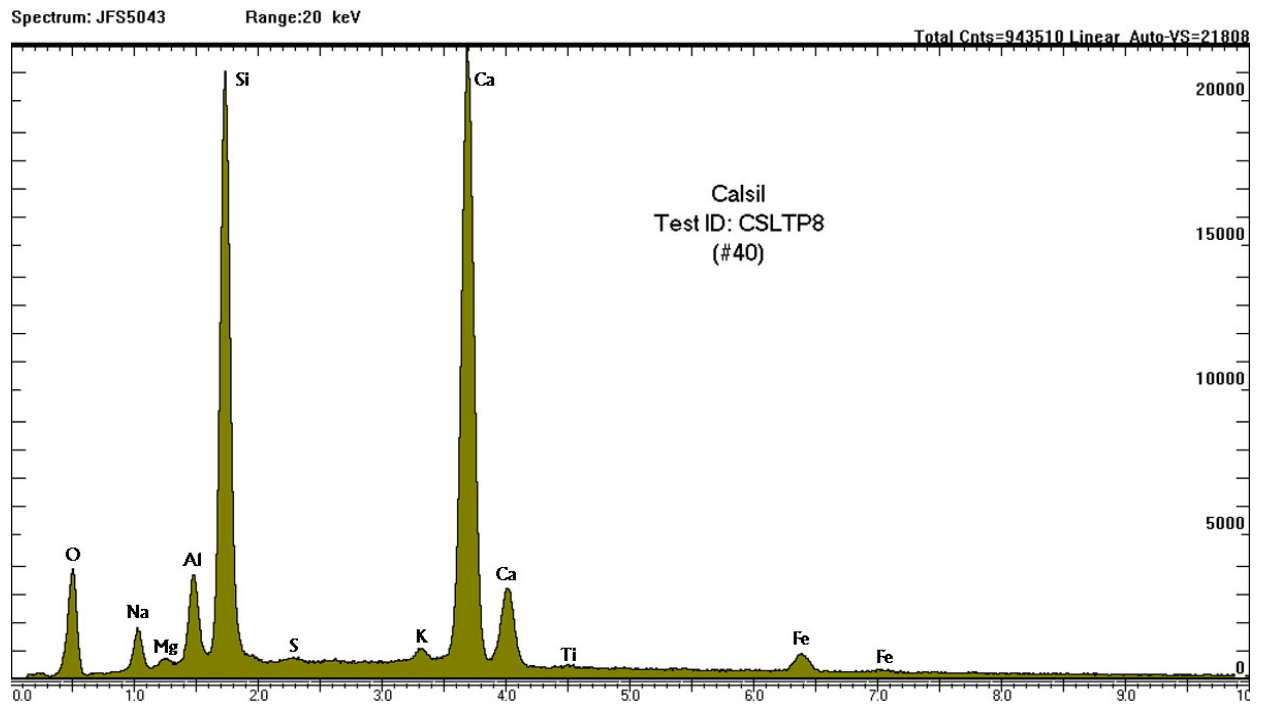
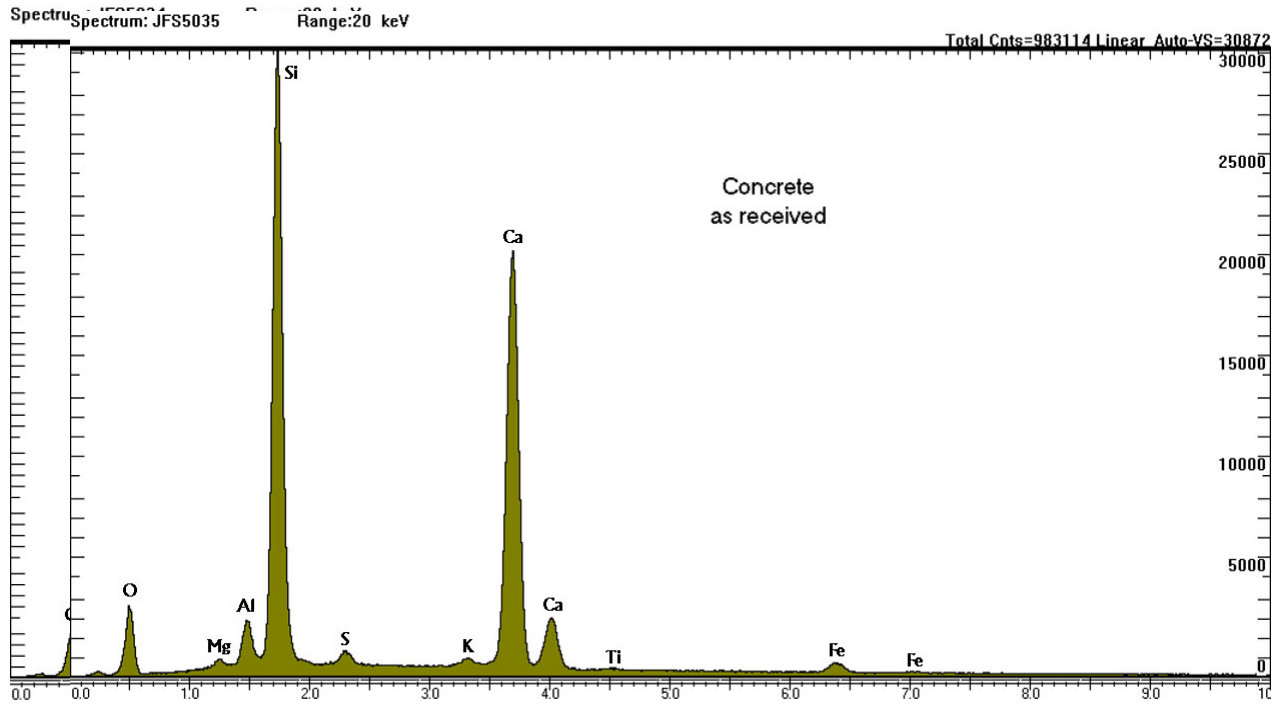


Figure 8. EDS Spectra for Calsil Sample Before and After Leaching in pH 8 Borated Water at 87.8 °C [190 °F] for 90 Minutes

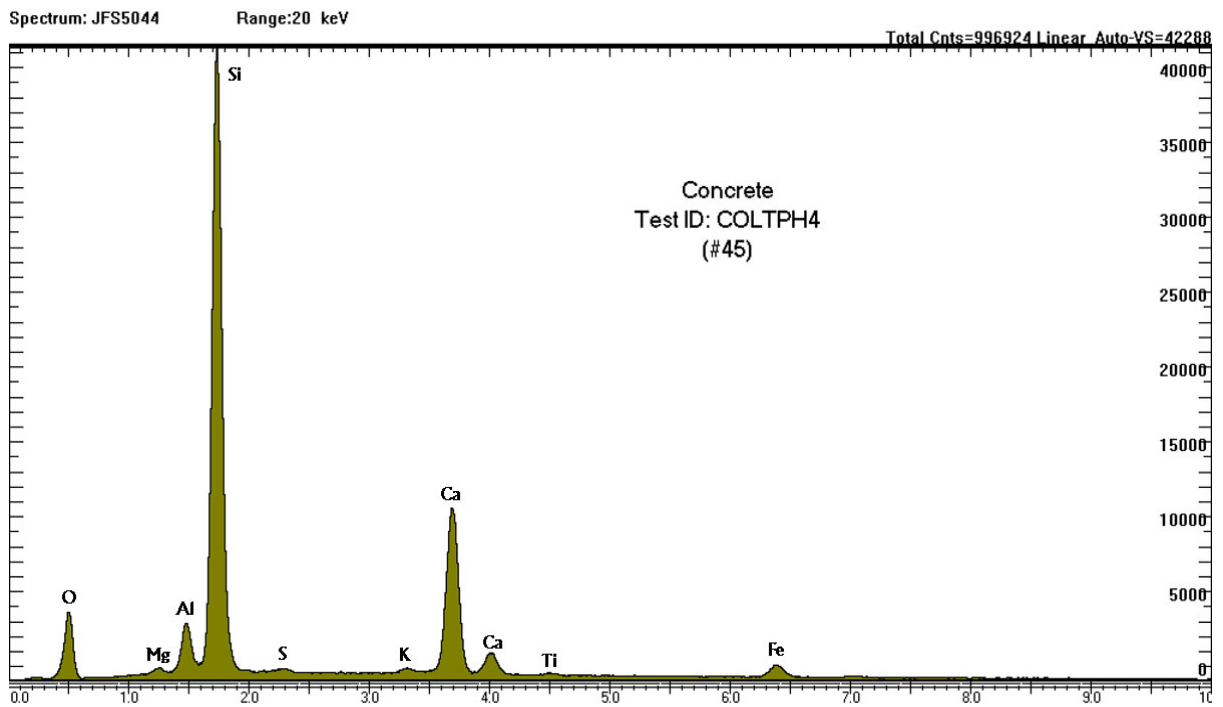


Figure 9. EDS Spectra for Concrete Sample Before and After Leaching in pH 4.1 Borated Water at 87.8 °C [190 °F] for 90 Minutes

Table 1. Surface Composition of Materials Before and After Leaching Tests											
Sample Material and Test ID†	Test Conditions		Relative Concentration at Surface (Atomic Percent)*								
	T (°C) [°F]	pH	Na	Mg	Al	Si	S	K	Ca	Ti	Zr
AlphaMat (As received)	—	—	1.1	8.6	9.9	38.5	0.6	1.2	37.5	—	—
AlphaMat AMHTPH12	129 [265]	12	16.4	1.6	12.2	42.5	—	0.8	25.8	—	—
Temp-Mat (As received)	—	—	1.2	1.7	14.8	52.1	—	0.9	28.7	—	—
Temp-Mat TMHTPH12	129 [265]	12	15.3	1.3	12.3	42.7	—	0.8	27.0	—	—
Fiberglass (As received)	—	—	17.2	4.6	4.4	61.2	0.6	1.1	10.2	—	—
Fiberglass FGHTPH12	129 [265]	12	29.3	3.8	3.3	52.6	—	1.0	9.5	—	—
Microtherm (As received)	—	—	—	1.7	4.8	67.2	—	—	—	26.1	—
Microtherm MTHTPH12	129 [265]	12	27.1	1.8	6.6	8.4	—	—	0.2	55.2	—
Microtherm MTLTPH12	87.8 [190]	12	69.2	2.4	5.2	8.9	—	—	0.3	13.9	—
Kaowool (As received)	—	—	0.9	—	35.9	52.0	—	—	0.4	—	10.7
Kaowool KWHTPH12	129 [265]	12	5.6	—	34.2	49.5	—	—	—	—	10.7
Durablanket (As received)	—	—	—	—	39.0	61.0	—	—	—	—	—
Durablanket DBHTPH12	129 [265]	12	11.9	—	34.9	53.2	—	—	—	—	—
Marinite (As received)	—	—	1.0	1.2	2.6	44.8	—	0.8	48.7	—	—
Marinite MALTPH8	87.8 [190]	8	4.9	1.5	3.9	47.7	—	0.5	40.6	—	—
CaSil (As received)	—	—	3.5	1.1	6.4	35.8	0.4	1.3	48.4	—	—
CaSil CSLTPH8	87.8 [190]	8	6.2	1.3	7.3	39.7	0.4	0.9	41.9	—	—
Concrete (As received)	—	—	—	1.5	4.9	50.9	1.6	0.8	38.6	—	—
Concrete COLTPH4	87.8 [190]	4.1	—	1.3	6.3	66.8	0.6	0.7	21.6	—	—

*measured by Electron Dispersive X-Ray Spectroscopy (EDS)
†ID = identification