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Integrated Chemical Effects Test Project: Test #5 Data Report

Los Alamos National Laboratory

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INTEGRATED CHEMICAL EFFECTS TEST PROJECT: TEST #5 DATA REPORT

ABSTRACT

A 30-day test was conducted in the Integrated Chemical Effects Test (ICET) project test apparatus. The test simulated the chemical environment present inside a pressurized water reactor containment water pool after a loss-of-coolant accident. The initial chemical environment contained 6.48 kg of boric acid, 10.0 kg of sodium tetraborate, and 0.284 g of lithium hydroxide. 90.8 mL of hydrochloric acid was added during the last two hours of the four-hour spray phase. The test was conducted for 30 days at a constant temperature of 60°C (140°F). The materials tested within this environment included representative amounts of submerged and unsubmerged aluminum, copper, concrete, zinc, carbon steel, and insulation samples (100% fiberglass). Representative amounts of concrete dust and latent debris were also added to the test solution. Water was circulated through the bottom portion of the test chamber during the entire test to achieve representative flow rates over the submerged specimens. The test solution pH varied from 8.2 to 8.4 for the duration of the test. The test solution turbidity decreased to approximately 2 NTU after 7 days. The turbidity at 60°C decreased to approximately 1 NTU the following day and remained near 1 NTU for the duration of the test. However, when the solution was cooled to 23°C, the turbidity increased to 5 NTU at Day 19 and remained near that value for the duration of the test. After the water samples had cooled to room temperature for several days, precipitates were visible in the water. These formed wispy patterns when the sample bottles were turned upside down and took 2–3 days to settle again. The submerged metallic coupons all developed thin particulate deposits that dulled their color and roughened their surface. Post-test examinations showed that the submerged aluminum coupons lost approximately 3% of their weight, but there were very little weight changes on the other coupons. The unsubmerged coupons exhibited some streaking, but little or no weight changes. The bottom of the tank contained very little sediment at the end of the test. The test solution at 60°C remained Newtonian for the entire test. When cooled to 25°C, the solution exhibited shear thinning, and the viscosity generally increased at all shear rates as the test progressed. Aluminum concentration rose to over 50 mg/L by Day 11 and fluctuated between 33 and 55 mg/L for the duration of the test.

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EXECUTIVE SUMMARY

The U.S. Nuclear Regulatory Commission (NRC) Office of Nuclear Regulatory Research has developed a comprehensive research program to support resolution of Generic Safety Issue (GSI)-191. GSI-191 addresses the potential for debris accumulation on pressurized water reactor (PWR) sump screens, with the consequent loss of net-positive-suction-head margin in the emergency core-cooling system (ECCS) pump. Among the GSI-191 research program tasks is the experimental investigation of chemical effects that may exacerbate sump-screen clogging.

The Integrated Chemical Effects Test (ICET) project represents a joint effort by the U.S. NRC and the nuclear utility industry, undertaken through the Memorandum of Understanding on Cooperative Nuclear Safety between NRC and Electric Power Research Institute (EPRI), Addendum on Integral Chemical Effects Testing for PWR ECCS Recirculation. The ICET project simulates the chemical environment present inside a containment water pool after a loss-of-coolant accident (LOCA) and monitors the chemical system for an extended time to identify the presence, composition, and physical characteristics of chemical products that form during the test. The ICET test series is being conducted by Los Alamos National Laboratory at the University of New Mexico, with the assistance of professors and students in the civil engineering department.

This report describes the ICET experimental apparatus and surveys the principal findings of Test #5. This interim data report summarizes both primary and representative findings that were available at the time the report was prepared. The NRC and the nuclear power industry may conduct additional analyses to enhance the understandings obtained from this test.

All of the ICET tests were conducted in environments that simulate expected containment pool conditions during recirculation. The tests are conducted for 30 days at a constant temperature of 60°C (140°F). The materials tested within each environment include representative amounts of submerged and unsubmerged aluminum, copper, concrete, zinc, carbon steel, and insulation samples. Representative amounts of concrete dust and latent debris are also added to the test solution. Tests consist of an initial 4-hour spray phase to simulate containment spray interaction with the unsubmerged samples. Water is circulated through the bottom portion of the test chamber during the entire test to achieve representative flow rates over the submerged specimens. Test #5 had a different initial boron concentration and a buffering agent that was different from the other 4 tests. Boric acid (6.48 kg), sodium tetraborate (10.0 kg), and lithium hydroxide (0.284 g) were added and dissolved in the ICET tank solution. That resulted in the initial test solution having a boron concentration of 2400 mg/L. Also, 90.8 mL of hydrochloric acid was added during the last two hours of the spray phase.

ICET Test #5 was conducted using sodium tetraborate as a buffering agent, with a target pH of 8 to 8.5. Insulation samples consisted of scaled amounts of NUKON™ fiberglass. In addition, the test apparatus contained 373 metal coupon samples and 1 concrete sample. Process control

consisted of monitoring online measurements of recirculation flow rate, test solution temperature, and pH. Flow rate and temperature were controlled to maintain the desired values of 25 gpm and 140°F. Daily water samples were obtained for measurements of pH, turbidity, total suspended solids, kinematic viscosity, and shear-dependent viscosity and for analytical laboratory evaluations of the chemical elements present. In addition, microscopic evaluations were conducted on water sample filtrates, fiberglass, coupons, and sediment.

The test ran for 30 days, and all conditions were maintained within the accepted flow and temperature ranges, with one exception. On Day 5, the addition of cold makeup water caused the test solution temperature to drop to 57.7ºC, which is 0.3ºC below the target minimum. The minimum temperature was below 58.0ºC for less than 10 minutes. At the start of the test, the measured pH was 8.4. During the addition of hydrochloric acid, the pH dropped slightly to 8.3, and it remained between 8.2 and 8.4 for the duration of the test.

Daily measurements of the constant-shear kinematic viscosity of the test solution revealed an approximately constant value at both test temperature and room temperature. Measurements of the shear-dependent viscosity indicated that at 60°C the test solution remained Newtonian for the entire test. At 25°C, the test solution exhibited shear thinning, and the viscosity generally increased at all shear rates as the test progressed. Light, wispy precipitates were visible after the test solution sat at room temperature for several days.

Analyses of the test solution showed that aluminum in the solution rose above 50 mg/L on Day 11 and fluctuated between 33 and 55 mg/L for the duration of the test. Calcium, silica, and sodium were prevalent in the solution.

Examinations of fiberglass taken from the test apparatus revealed chemical byproducts and web-like deposits that spanned individual fibers. Flocculent deposits were also observed. The amounts of these deposits did not increase significantly over the duration of the test, and the web-like deposits were absent in the Day 30 samples. The deposits were likely formed by chemical precipitation. In addition to flocculent deposits, some samples had significant amounts of particulate deposits on their exteriors that were likely physically attached.

The submerged metallic coupons all developed thin particulate deposits that dulled their color and roughened their surface. Post-test examinations showed that the submerged aluminum coupons lost approximately 3% of their weight, but there were very little weight changes on the other coupons. The unsubmerged coupons exhibited some streaking, but little or no weight changes.

The ICET series is being conducted under an approved quality assurance (QA) program, and QA procedures and project instructions were reviewed and approved by the project sponsors. Analytical laboratory results are generated under a quality control program approved by the Environmental Protection Agency, and other laboratory analyses were performed using standard practices.

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ACRONYMS AND ABBREVIATIONS

Al	Aluminum
Au	Gold
CPVC	Chlorinated Polyvinyl Chloride
CS	Coated steel
Cu	Copper
DAS	Data Acquisition System
DHR	Decay Heat Removal
ECCS	Emergency Core-Cooling System
EDS	Energy-Dispersive Spectroscopy
EPA	Environmental Protection Agency
ESEM	Environmental Scanning Electron Microscopy
F	Filtered
GS	Galvanized Steel
GSI	Generic Safety Issue
HCl	Hydrochloric Acid
HI	High-Flow Interior Fiberglass Sample
HX	High-Flow Exterior Fiberglass Sample
ICET	Integrated Chemical Effects Tests
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
IOZ	Inorganic Zinc
LANL	Los Alamos National Laboratory
LI	Low-Flow Interior Fiberglass Sample
LiOH	Lithium Hydroxide
LOCA	Loss-of-Coolant Accident
LX	Low-flow Exterior Fiberglass Sample
NIST	National Institute of Standards and Technology
NRC	Nuclear Regulatory Commission
NTU	Nephelometric Turbidity Unit
P	Phosphorous
Pd	Palladium
PI	Project Instruction
PVC	Polyvinyl Chloride

PWR	Pressurized Water Reactor
QA	Quality Assurance
RO	Reverse Osmosis
SEM	Scanning Electron Microscopy
SS	Stainless Steel
TEM	Transmission Electron Microscopy
TOC	Total Organic Carbon
TSP	Trisodium Phosphate
TSS	Total Suspended Solids
U	Unfiltered
UNM	University of New Mexico
US	Uncoated steel
WD	Working Distance
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence

1 INTRODUCTION

The Integrated Chemical Effects Test (ICET) project represents a joint effort by the United States Nuclear Regulatory Commission (NRC) and the nuclear utility industry to simulate the chemical environment present inside a containment water pool after a loss-of-coolant accident (LOCA) and to monitor the chemical system for an extended time to identify the presence, composition, and physical characteristics of chemical products that may form. The ICET series is being conducted by Los Alamos National Laboratory (LANL) at the University of New Mexico (UNM), with the assistance of professors and students in the civil engineering department.

1.1. Objective and Test Conditions

Containment buildings of pressurized water reactors (PWRs) are designed to accommodate the energy release following a postulated accident. They also permit recirculation of reactor coolant and emergency-core-cooling-system (ECCS) water to the decay heat removal (DHR) heat exchangers. The water collected in the sump from the reactor coolant system, the safety injection system, and the containment spray system is recirculated to the reactor core to remove residual heat. The sump contains a screen to protect system structures and components in the containment spray and ECCS flow paths from the effects of debris that could be transported to the sump. Concerns have been raised that fibrous insulation material could form a mat on the screen, obstructing flow, and that chemical reaction products such as gelatinous or crystalline precipitants could migrate to the screen, causing further blockage and increased head losses across the debris bed. Another potential adverse chemical effect includes increased bulk fluid viscosity that could also increase head losses through a debris bed.

The primary objectives for the ICET series are (1) to determine, characterize, and quantify chemical reaction products that may develop in the containment sump under a representative post-LOCA environment and (2) to determine and quantify any gelatinous material that could be produced during the post-LOCA recirculation phase.

The ICET series was conceived as a limited-scope suite of five different 30-day tests with different constituents. The conditions selected for each test are shown in Table 1-1. Test #5 had a different initial boron concentration and a buffering that was different from the other 4 tests. A 107-gallon solution containing 2800 mg/L of boron, and 0.7 mg/L of lithium hydroxide was mixed with a 143-gallon solution containing 18.5 g/L of sodium tetraborate (Borax). That resulted in the initial test solution having a boron concentration of 2400 mg/L. Also, 90.8 mL of hydrochloric acid was added during the last two hours of the spray phase. The resulting pH was an intermediate value of 8-8.5. All tests in the series included metal coupons whose surface areas were scaled to those in representative PWR containment and sump systems. A complete rationale for the selection of these test conditions is provided in Reference 1.

Table 1-1. Test Series Parameters

Run	Temp	TSP	NaOH	Sodium Tetraborate	pH	Boron	Notes
	(°C)					(mg/L)	
1	60	N/A	Yes	N/A	10	2800	100% fiberglass insulation test. High pH, NaOH concentration as required by pH
2	60	Yes	N/A	N/A	7	2800	100% fiberglass insulation test. Low pH, TSP concentration as required by pH.
3	60	Yes	N/A	N/A	7	2800	80% calcium silicate/20% fiberglass insulation test. Low pH, TSP concentration as required by pH
4	60	N/A	Yes	N/A	10	2800	80% calcium silicate/20% fiberglass insulation test. High pH, NaOH concentration, as required by pH.
5	60	N/A	N/A	Yes	8 to 8.5	2400	100% fiberglass insulation test. Intermediate pH, sodium tetraborate (Borax) buffer.

The ICET apparatus consists of a large stainless-steel (SS) tank with heating elements, spray nozzles, and associated recirculation pump and piping to simulate the post-LOCA chemical environment. Samples of structural metals, concrete, and insulation debris are scaled in proportion to their relative surface areas found in containment and in proportion to a maximum test dilution volume of 250 gal. of circulating fluid. Representative chemical additives, temperature, and material combinations are established in each test; the system is then monitored while corrosion and fluid circulation occur for a duration comparable to the ECCS recirculation mission time.

1.2. Information Presented in this Report

This report surveys the principal findings of ICET Test #5. As an interim data report, this exposition summarizes both primary and representative findings, but it cannot be considered comprehensive. For example, only a small selection of photographs out of several hundred is presented here. In addition, this report presents observations and data without in-depth analyses or interpretations. However, trends and typical behaviors are noted where appropriate. Section 2 of this report reviews the test procedures followed for Test #5. Analytical techniques used in evaluating test results are also briefly reviewed in Section 2. Section 3 presents key test results for Test #5, including representative and noteworthy results of water sampling, fiberglass insulation samples, metallic and concrete coupon samples, tank sediment, deposition products, and water property analyses. The results for Test #5 are presented in both graphical and narrative form. Section 4 presents a summary of key observations for Test #5. This report also includes several appendices that capture additional Test #5 images and information. The data presented in the appendices are largely qualitative, consisting primarily of environmental scanning

electron microscopy (ESEM), scanning electron microscopy (SEM), transmission electron microscopy (TEM) micrographs, and energy-dispersive spectroscopy (EDS) spectra.

2 TEST PROCEDURES

The functional description and physical attributes of the ICET apparatus were presented in detail in the ICET Test #1 report (Ref. 2). The experimental apparatus is briefly described below, followed by a review of the test operation and analytical techniques used to evaluate the test results.

2.1 Chemical Test Apparatus Functional Description

The test apparatus was designed to meet the functional requirements of the Project Test Plan (Ref. 1). Functional aspects of the test apparatus are as follows:

1. The central component of the system is a test tank. The test apparatus was designed to prevent solids from settling in the test piping.

2. The test tank can maintain both a liquid and vapor environment, as would be expected in post-LOCA containment.

3. The test loop controls the liquid temperature at 140°F (±5°F).

4. The system circulates water at flow rates that simulate spray flow rates per unit area of containment cross section.

5. The test tank provides for water flow over submerged test coupons that is representative of containment pool fluid velocities expected at plants.

6. Piping and related isolation valves are provided such that a section of piping can be isolated without interrupting the test.

7. The pump discharge line is split in two, one branch directing the spray header in the tank's vapor space and the other returning to the liquid side of the tank. Each branch is provided with an isolation valve, and the spray line includes a flow meter.

8. The recirculation piping includes a flow meter.

9. The pump circulation flow rate is controlled at the pump discharge to be within ±5% of the flow required to simulate fluid velocities in the tank. Flow is controlled manually.

10. The tank accommodates a rack of immersed sample coupons, including the potential reaction constituents identified in the test plan.

11. The tank also accommodates six racks of sample coupons that are exposed to a spray of liquid that simulates the chemistry of a containment spray system. Provision is made for these racks to be visually inspected.

12. The coupon racks provide sufficient space between the test coupons to preclude galvanic interactions among the coupons. The different metallic test coupons are also electrically isolated from each other and the test stand to prevent galvanic effects resulting from metal-to-metal contact between specimens or between the test tank and the specimens.

13. The fluid volumes and sample surface areas are based on scaling considerations that relate the test conditions to actual plant conditions.

14. All components of the test loop are made of corrosion-resistant material (for example, SS for metallic components).

The as-built test loop consists of a test tank, a recirculation pump, 2 flow meters, 10 isolation valves, and pipes that connect the major components, as shown schematically in Figure 2-1. P, T, and pH represent pressure, temperature, and pH probes, respectively.

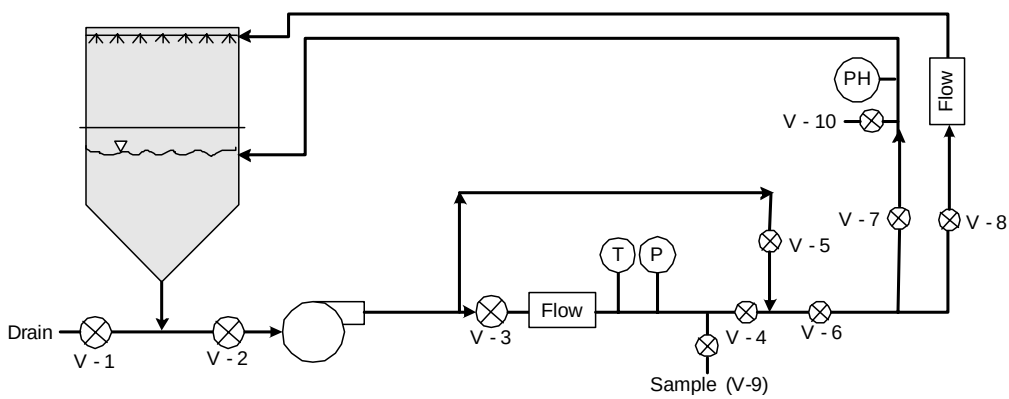

Figure 2-1. Test loop process flow diagram.

2.2 Pre-Test Preparation

2.2.1. Test Loop Cleaning

In preparation for Test #5, the experiment test loop was thoroughly cleaned to remove all Test #4 deposits and residues. In addition to visual inspections, the test apparatus was flushed and cleaned per the written direction given in the pre-test operations project instruction (PI) (Ref. 3). The system was flushed with ammonium hydroxide, followed by ethanol, and then nitric acid until it was visually clean and the water conductivity was <50 μS/cm.

2.2.2. Test Coupons and Samples

Each ICET experiment exposes metallic and concrete coupons to anticipated post-LOCA environments. Each coupon is approximately 12 in. square. The metallic coupons are approximately 1/16 in. thick, except for the inorganic zinc-coated steel coupons, which are approximately 3/32 in. thick. The concrete coupons (one per test) are approximately 1-1/2 in. thick. Insulation materials are also exposed. For Test #5, NUKON™ fiberglass insulation samples were included in the test. As with previous tests, Test #5 subjected seven racks of coupons to the specified environment, with one being submerged in the test tank and the remaining six being held in the tank's gas/vapor space. The Test #5 coupons of each type were as shown in Table 2-1.

Table 2-1. Quantity of Each Coupon Type in Test #5

Material	No. of Coupons
Coated Steel (CS)	77
Aluminum (Al)	59
Galvanized Steel (GS)	134
Copper (Cu)	100
Uncoated Steel (US)	3
Concrete	1

Note: Inorganic zinc (IOZ) coated steel and CS are the same coupon type.

The arrangement of the coupon racks in the test tank is schematically illustrated in Figure 2-2. The figure shows a side view of the ICET tank, with the ends of the seven CPVC racks illustrated. The normal water level is indicated by the blue line in the figure. Rack #1 is the only submerged rack, and it sits on angle iron. It is centered in the tank so that flow from the two headers reaches it equally. Racks #2–#4 are positioned above the water line, supported by angle iron in the tank. Racks #5–#7 are positioned at a higher level, also supported by angle iron. Racks #2–#7 are exposed to spray. In the figure, north is to the right, and south is to the left. Directions are used only to identify such things as rack locations and sediment locations.

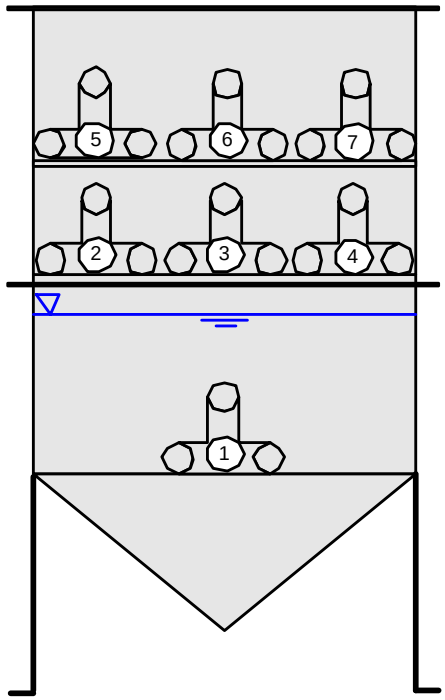

Figure 2-2. Coupon rack configuration in the ICET tank. The blue line represents the surface of the test solution.

Figure 2-3 shows the configuration of a typical unsubmerged coupon rack loaded with metal coupons in the ICET tank. The loading pattern of the racks was nearly identical, varying by only one or two coupons. Shown in the figure from left to right, the coupons are arranged as follows: 4 Cu, 4 Al, 4 inorganic zinc (IOZ), 7 GS, 4 Cu, 3 Al, 4 IOZ, 7 GS, 4 Cu, 3 Al, 4 IOZ, and 7 GS.

Figure 2-3. A typical loaded coupon rack in the ICET tank.

Several fiberglass samples were placed in the ICET tank. Samples were either submerged or held above the water level. The unsubmerged fiberglass samples were positioned so they would be exposed to sprays. The fiberglass samples were contained in SS wire mesh that allowed water flow while confining the fiberglass material. Both loosely packed and more tightly packed samples were used. In addition, some submerged fiberglass samples were located where they would be exposed to relatively high-flow conditions, and others were located in quiescent regions of the tank. Figure 2-4 shows the so-called “sacrificial” fiberglass samples in wire mesh pouches attached to the submerged coupon rack (Rack 1 in Figure 2-2). Each pouch contains approximately 5 g of fiberglass. Those samples were attached with SS wire; removed from the tank on Days 4, 15, and 30; and examined. As shown in the figure, bigger insulation bags were wrapped around the sacrificial specimens during the test. In addition, small, sacrificial samples called fiber pucks were added to the solution. The fiber pucks are described in Reference 4. Subsection 2.4.1.1 contains descriptions of other fiberglass samples.

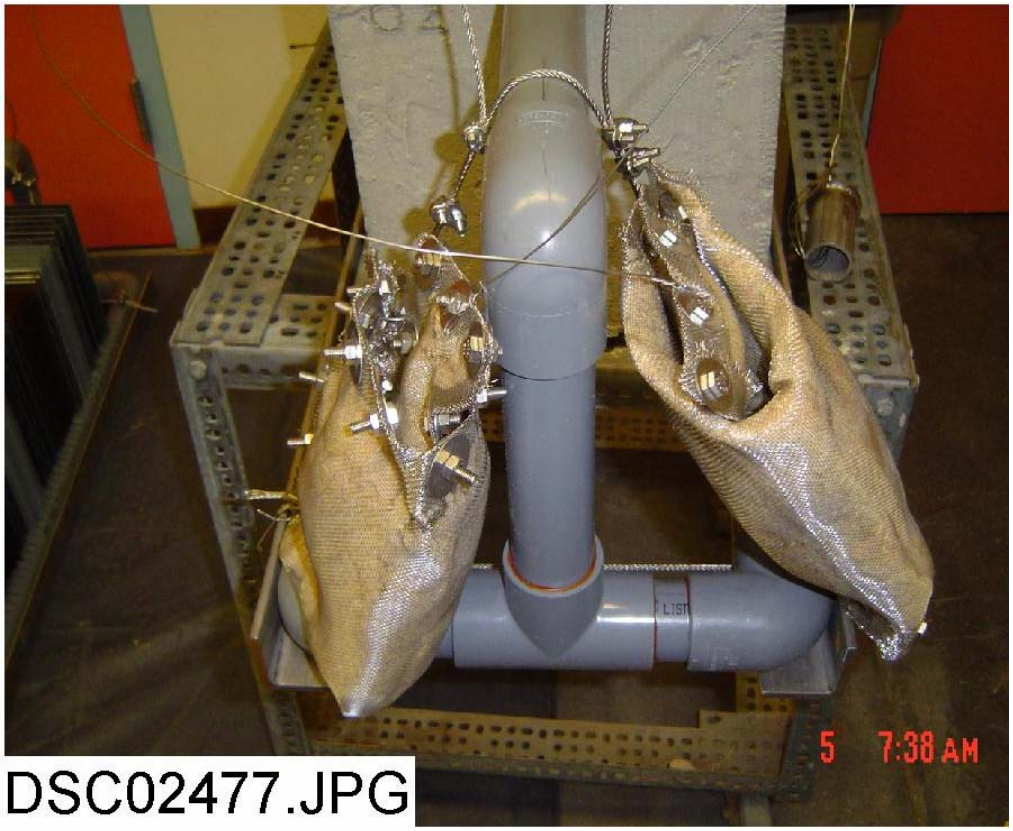

Figure 2-4. Fiberglass samples attached to a coupon rack.

2.2.3. Quality Assurance Program

A project QA manual was developed to satisfy the contractual requirements that apply to the ICET project. Specifically, those requirements were to provide credible results by maintaining an appropriate level of QA in the areas of test loop design, sampling, chemicals, operation, and analysis. These requirements were summarized in the contract requirement that QA was to be consistent with the intent of the appropriate sections of 10CFR50, Appendix B.

The 18 criteria of 10CFR50, Appendix B, were addressed separately in the QA manual, and the extents to which they apply to the ICET project were delineated. A resultant set of QA procedures was developed. In addition, test-specific project instructions (PIs) were written to address specific operational topics that required detailed step-by-step guidance. PIs generally applicable to all tests were written for the following topics and were followed for Test #5:

- Data Acquisition System (DAS)
- Coupon Receipt, Preparation, Inspection, and Storage
- DAS Alarm Response
- Chemical Sampling and Analysis

- TEM Examination of Test Samples
- SEM Characterization of Test Samples
- Viscosity Measurements
- Post-Test Operations

Project instructions specific to Test #5 were written for the following:

- Pre-Test Operations, Test #5
- Test Operations, Test #5 (fiberglass and sodium tetraborate at pH 8)

The pre-test, test, and post-test operations PIs that were used in Test #5 are included in Appendix I.

2.2.4. Test Parameters

ICET test parameters were selected based on literature surveys and the results of surveys of United States nuclear power plants. Quantities of test materials were selected to preserve the scaling of representative ratios between material surface areas and total cooling-water volumes. Chemical additives also simulate the post-LOCA sump environment. The Project Test Plan (Ref. 1) is the basis for the following information in this section.

The materials included in the tests are zinc, aluminum, copper, carbon steel, concrete, and insulation materials such as fiberglass and calcium silicate. The amounts of each material are given in Table 2-2 in the form of ratios (material surface area to water volume), with three exceptions: concrete dust, which is presented as a ratio of mass to water volume, and fiberglass and calcium silicate, which are presented as a ratio of insulation volume to water volume. Also shown in the table are the percentages of material that are submerged and unsubmerged in the test chamber.

Table 2-2. Material Quantity/Sump Water Volume Ratios for the ICET Tests

Material	Value of Ratio for the Test (ratio units)	Percentage of Submerged Material (%)	Percentage of Unsubmerged Material (%)
Zinc in Galvanized Steel	8.0 (ft^2/ft^3)	5	95
Inorganic Zinc Primer Coating (non–top coated)	4.6 (ft^2/ft^3)	4	96
Inorganic Zinc Primer Coating (top coated)	0.0 (ft^2/ft^3)	–	–
Aluminum	3.5 (ft^2/ft^3)	5	95
Copper (including Cu-Ni alloys)	6.0 (ft^2/ft^3)	25	75
Carbon Steel	0.15 (ft^2/ft^3)	34	66
Concrete (surface)	0.045 (ft^2/ft^3)	34	66
Concrete (particulate)	0.0014 (lbm/ft^3)	100	0
Insulation Material (fiberglass or calcium silicate)	0.137 (ft^3/ft^3)	75	25

The physical and chemical parameters that are critical for defining the tank environment and that have a significant effect on sump-flow blockage potential and gel formation have been identified in Reference 1. These physical and chemical parameters are summarized as follows:

Physical Parameters

• Water volume in the tank	949 L	250 gal.
• Circulation flow	0–200 L/min	0–50 gpm
• Spray flow	0–20 L/min	0–5 gpm
• Sump temperature	60°C	140°F

Chemistry Parameters

• H_3BO_3 concentration	2800 mg/L as boron
• $Na_3PO_4 \cdot 12H_2O$ concentration	As required to reach pH 7 in the simulated sump fluid
• NaOH concentration	As required to reach pH 10 in the simulated sump fluid
• Sodium tetraborate (Borax)	As required to reach boron concentration of 2400 mg/L
• HCl concentration	100 mg/L
• LiOH concentration	0.7 mg/L as Li

The parameters planned for each ICET test run are described in Table 1-1.

2.3 Test Operation

2.3.1. Description

Preparation of ICET Test #5 (Run 5 in Table 1-1) began with 248 gal. of reverse osmosis (RO) water heated to 65°C. (Adding the metal coupons and insulation samples reduces the water temperature by approximately 5°C, so the water was heated initially to 65°C.) With 25 gpm circulating through the loop, the predetermined quantities of boric acid (6.48 kg), sodium tetraborate (10.0 kg), and lithium hydroxide (0.284 g) were added and dissolved in the ICET tank solution. After the chemicals were added and observed to be well mixed, a baseline grab sample and measurements of the test solution were taken. Then the pre-measured latent debris and concrete dust were added to the tank solution. After the solution circulated for 10 minutes, the pump was stopped and the coupon racks and insulation samples were put into the tank (see Section 2.2.2).

The test commenced with initiation of the tank sprays (3.5 gpm). After two hours, 2 gallons of RO water containing 90.8 mL of hydrochloric acid were metered into the spray. The sprays were terminated after 4 hours. The test ran uninterrupted for 30 days.

The experiment commenced at 11:00 a.m. on Tuesday, July 26, 2005, and it ended on August 25, 2005. During the test, grab samples were taken daily for wet chemistry and inductively coupled plasma – atomic emission spectroscopy (ICP-AES) analyses. Water loss due to water sample removals and evaporation was made up with RO water. Water samples, insulation, and metal coupons were analyzed after the test. Sampling and analyses were conducted in accordance with approved project instructions (Refs. 3, 4, and 5).

2.3.2. Process Control

During the test, critical process control parameters were monitored to ensure that the test conditions met the functional test requirements. Recirculation flow rate and temperature were controlled throughout the test. The solution pH was expected to reach a value of approximately 8 to 8.5 after the spray phase ended. The predetermined amounts of chemicals were added to achieve 2400 mg/L of boron, and pH was not controlled.

Recirculation flow in the test loop was controlled by adjusting the pump speed. Fine tuning was achieved by manually adjusting a valve located downstream of the recirculation pump. In-line flow meters were used to measure the flow rate in the recirculation line and the spray line.

Titanium-jacketed immersion electric heaters controlled the water temperature. The heaters were thermostatically controlled to automatically maintain the desired temperature.

2.4 Analytical Methods

Data collected during Test #5 included the in-line measurements of temperature, pH, and loop flow rate. During the daily water grab sample analysis, bench-top measurements were obtained for temperature, pH, turbidity, total suspended solids (TSS), and kinematic viscosity. The concentration of hydrogen in the tank atmosphere was also measured and could be used as an indicator of chemical reactions taking place. Water, fiberglass, and metal coupon samples were taken to other laboratory locations for additional analyses. These analyses included shear-rate viscosity, ESEM, SEM, EDS, TEM, ICP-AES, x-ray fluorescence (XRF), and x-ray diffraction (XRD). EDS provided a semi-quantitative elemental analysis after calibration of the instrument's x-ray signal using an internal element standard. Descriptions of the principles of operation and limitations of these analytical methods were provided in the Test #1 report (Ref. 2).

2.4.1. Data Compilation and Nomenclature

This section provides a brief guide to assist the reader in interpreting the ICET Test #5 information and data presented in the following sections and in the appendices. Standardized nomenclature is defined first to clarify the origin of samples that are described in the data sets. The appendices are listed, and a description is provided of how they were compiled.

2.4.1.1. Nomenclature

Many spatially unique but physically similar sample types were collected in ICET Test #5. To ensure that consistent interpretations and comparisons of data sets are made, it is imperative that a standardized nomenclature be adopted when referring to each sample type. Many different qualitative descriptions of these samples might be equally suitable, but different adjectives convey different connotations to each observer. Therefore, the following definitions establish the convention used in this report when making generic references to sample type.

White Precipitate — The behavior of the solution at test temperature and upon cooling is observed during testing. Precipitates and their prominence indicate chemical interactions occurring in the solution. White precipitate formed in Test #1 water solution samples drawn from the test loop. Upon cooling below the test temperature, Test #1 daily water samples extracted from the tank formed a visible white material that is referred to as a precipitate. While less prominent from Test #1, there was a precipitate that formed in Test #5. After the test solution sat at room temperature for several days, a light, wispy precipitate was visible after the sample bottle was agitated. The precipitate could not be seen again until the sample sat for several days. The precipitate was not concentrated enough to allow samples to be obtained for analysis.

Latent Debris	Commercial power plant containments gradually accumulate dust, dirt, and fibrous lint that are generically referred to as latent debris. This classification distinguishes resident material from debris generated during an accident scenario. At the beginning of Test #5, measured quantities of crushed concrete and soil (sand and clay) were added to simulate the latent debris present in containment. These materials were examined via SEM/EDS to establish a baseline composition for comparison with sediment samples (see "Sediment" below).
Sediment	Surrogate latent debris particulates and fugitive fiberglass fragments that were initially suspended in water at the beginning of Test #5 gradually settled to the bottom of the tank. At the conclusion of the test, only a small amount of sediment remained on the tank bottom. It was recovered as completely as possible.
Powder	At the conclusion of Test #5, fine, yellow particulate deposits were found on the submerged CPVC coupon rack. They were referred to as powder and examined by SEM/EDS. These deposits are also referred to as deposition products (see Appendix D).
Fiberglass	The principal debris type introduced in Test #5 was shredded fiberglass insulation. This debris was bundled in 3-in.-thick bags (or blankets) of fiberglass confined in SS mesh to prevent ingestion through the pump and to better control the placement of debris in various flow regimes. Fiberglass samples are designated by their placement in high-flow and low-flow areas of the tank. Fiberglass in the "big envelope" sat on the tank bottom in a low-flow area of the tank. Additional 4-in.-square envelopes of fiberglass were also prepared for extraction during the course of the test. These samples are referred to as "sacrificial" samples. The "birdcage" sample was constructed so that the fiberglass within was loose and not compacted. The birdcage fiberglass sat on the tank bottom and was removed on Day 30. Some amount of fiber, especially short-fiber fragments, escaped the mesh bags and was deposited in other locations within the tank. This material is referred to as "fugitive" fiberglass. Two additional fiberglass samples were added after the test began and the water clarity improved, to investigate what deposited after the tank solution stablized. A sacrificial sample was placed directly in front of one of the flow headers (high-flow area) on Day 6. A sacrificial sample enclosed in nylon mesh was placed in a low-flow area also on Day 6.
Drain Screen	A 12-in.-tall screen made of coarse SS mesh (1/8-in. holes) wrapped into a 2-in.-diam cylinder was inserted into the outlet drain at the bottom of the tank to protect the pump from ingesting

	large debris items. Two inches of the screen were inserted into the tank outlet to provide a solid base and stability. A 6-in.-tall drain collar was installed around the drain screen. This drain collar was a cylinder of fiberglass held in SS mesh. The drain collar was exposed to higher-velocity water flow than other samples in the tank. The drain collar fiberglass was examined as a separate debris location to identify any apparent differences with other sample locations.
Gelatinous Material	This term generically refers to any observed sample constituent with amorphous, hydrated, or noncrystalline physical characteristics. When Test #5 was shut down, there was no evidence of gel-like precipitates in the tank or piping.
Water Sample	Daily water samples were extracted from the ICET tank for elemental concentration analyses. After the sample line was properly flushed, some of this water was extracted directly from the tap. An equal amount of water was also generally collected through a micropore filter. Thus, daily water samples were designated as filtered (F) and unfiltered (U), and a corresponding filter paper exists in the sample archive for each daily sample that was collected.
High-Volume Filter	If white precipitates are observed in the tests, larger quantities of test solution are periodically extracted for filtration to determine whether suspended chemical products are present in the test liquid under in situ conditions. The intent of this exercise is to maintain the liquid temperature while forcing the liquid through a micropore filter under vacuum. Because the precipitates were not present in Test #5, these high-volume filter samples were not obtained.
Filter Paper	Many different samples of tank solution were fractionated by micropore filtration into a liquid supernate and a solid filtrate that existed at the time and temperature conditions of the filtering process. These samples included (1) daily water samples filtered during extraction, (2) daily water samples filtered after cooling to room temperature, and (3) high-volume water samples.
Chemical Deposits	Sacrificial fiberglass samples that were extracted at Day 4, Day 15, and Day 30 showed evidence of chemical products forming on and between fiber strands. These products are referred to as “deposits,” although the exact physical mechanism of formation is not well understood. The physical appearance suggests growth, agglomeration, or crystallization on and around the fiber strands over time rather than capture or impaction of particles from the bulk solution. This observation is supported by the fact that the

	small sacrificial fiberglass samples were located in a region of lower-velocity water flow (i.e., in the interior of larger blankets).
Concrete Sample	Several chips of concrete (1/4–3/4 in. diam) were broken from the primary slab of submerged concrete and introduced to the tank in a small SS envelope at the start of the test. Examinations of these chips were conducted to determine if concrete surfaces provide a preferential site for gel formation.

Although these terms have been defined, the reader may note minor inconsistencies in the caption labels used in this document. The caption labels use the same descriptions that were applied in laboratory notebooks to improve traceability of the data.

2.4.1.2. Usage

The 9 appendices listed below are provided to present data collected for the sample types and analysis methods listed below. In addition, an appendix is provided with pertinent Test #5 project instructions.

Appendix A	ESEM/EDS Data for Test #5 Day-4 Fiberglass in a Low-Flow Zone
Appendix B	ESEM Day-15 Fiberglass
Appendix C	ESEM Day-30 Fiberglass
Appendix D	SEM/EDS Data for Test #5 Day-30 Deposition Products
Appendix E	SEM Day-30 Coupons
Appendix F	SEM/EDS Data for Test #5 Day-30 Sediment
Appendix G	TEM Data for Test #5 Solution Samples
Appendix H	UV Absorbance Spectrum – Day-30 Solution Samples
Appendix I	ICET Test #5: Pre-Test, Test, and Post-Test Project Instructions

These data are largely qualitative in nature, consisting primarily of ESEM, SEM, TEM micrographs, and EDS spectra. Each appendix subsection represents a separate session of laboratory work that can be traced to a batch of samples that were processed in chronological order. This organizational scheme preserves the connection with laboratory notebooks and timelines that naturally developed during operation; however, in a few cases, results for a given sample type may be mixed across two or more appendices because of the order in which the individual samples were analyzed.

ESEM analyses were added to the ICET diagnostic suite for the first time during Test #2 as a means of examining hydrated chemical products. This equipment operates as an electron microscope, but it does not require a high-vacuum condition in the sample chamber. Thus, a sample need only be thoroughly drained of free water content before examination rather than fully desiccated, making the ESEM ideal for examinations of

biological and environmental specimens. The complementary EDS capability that is often found with equipment of this type is not presently functional at UNM, so duplicate examinations are often performed on the same ICET sample first using ESEM to obtain images of hydrated structural details and then using SEM/EDS to obtain representative elemental compositions. Throughout the report, ESEM analyses are also indicated by the descriptions of “hydrated” and “low-vacuum” findings.

Transcriptions of the logbooks are provided for each appendix to better document commonalities that existed among the samples at the time of analysis. Interpretation and understanding of the images and their accompanying EDS spectra will be greatly improved by frequent reference to the logbook sample descriptions and sequences. Typically, a relatively large quantity of a test sample was delivered for SEM or TEM analysis, and then several small sub-samples of each item were examined. Note that each sub-sample was assigned a sequential reference number during the laboratory session. These reference numbers have been cited in the figure captions whenever possible to preserve the connection between the micrographs and the notebook descriptions. Electronic file names have also been stamped on the images to permit retrieval of the original data files that are archived elsewhere. Individual data sets for a given sample item have been collated into a typical sequence of (1) visual image, (2) EDS spectra, and (3) semi-quantitative mass composition.

For most of the EDS spectra, semi-quantitative mass compositions are also presented. These results are obtained from a commercial algorithm that decomposes the spectra into the separate contributions of each element. Several caveats, as enumerated below, should be considered when interpreting the numeric compositions thus obtained; however, despite these caveats, semi-quantitative EDS analysis offers a natural complement to micrographic examination as a survey technique for identifying trends in composition.

1. The spectral deconvolution algorithm is based on a library of unique signatures of each element that were obtained for pure samples using a standard beam setting that may not identically match the conditions applied for the test item.
2. The operator must select a limited number of elements to be used in the proportional mass balance. These candidates are chosen from among the peaks that are observed in the spectrum; however, the composition percentages can vary, depending on which elements are included in the list. In a few cases, two or more alternative compositions have been generated by selecting a different set of elements from the same spectrum to illustrate the sensitivity of this technique to operator input.
3. The spectral unfolding algorithm is a statistical technique having a precision that depends on the relative quality of the data in each peak. Compositions with high R^2 correlation coefficients and total-mass normalization factors closer to unity represent the more-reliable estimates. The precision obtained in the fit depends on the duration of the scan and the number of counts received in each energy bin.

4. All sub-samples examined in the SEM microprobe facility are coated with a thin layer of either carbon or gold/palladium alloy to prevent the sub-samples from accumulating a charge from the impinging electron beam. Spectral peaks visible for gold (Au) and palladium (Pd) are not indigenous to the samples.
5. The EDS spectral analysis software contains a peak-recognition algorithm and an automated cursor that scans across the spectrum to locate each peak. An accompanying library of elemental energy signatures is also provided to suggest what constituents might be contributing to a given energy bin, but the operator must judge what label to assign to the spectral image. It is possible that some peaks near closely neighboring elements have been mislabeled in these images. However, every effort was made to choose from candidate elements that were most likely to be present in the test material. In a few cases, the spectral peaks were not labeled by the SEM operator. These spectra should be viewed as corroborating evidence for similar samples that are definitively labeled. Careful comparisons of the energy scales in combination with a library of electron-scattering energies can also be used to infer the origin of the more-prominent peaks that are present in unlabeled spectra.
6. Unless an obvious spatial heterogeneity is being examined, the exact location of an EDS spectrum is not always relevant because the operator chooses arbitrary sites that are visually judged to be representative. It is not possible to sample a surface comprehensively on a microscopic basis and compute average compositions. In many cases, two or three replicate spectra are provided for this purpose, but SEM/EDS is most effective as a survey diagnostic.
7. EDS analysis is not particularly sensitive to the presence of boron for several reasons: (a) boron has a low atomic mass that does not interact well with electrons in the beam, (b) the emission lines are very close to those of carbon, and (c) the beam-port material has a high absorption cross section for these emission energies. Therefore, the correction factors used in the semi-quantitative composition analysis are quite large, as are the uncertainties in the estimated percentage of total composition for this element. There may be spectra presented in the appendices in which the lowest energy peaks are labeled as either B (boron) and/or C (carbon).

EDS locations were chosen manually at regions of specific interest. In many cases, multiple spectra were collected from a single sample and an annotated image is provided to identify the specific location. These annotated images are not generally noted in the laboratory logbook entries, but they are provided in proper sequence within the appendices.

Appendix G presents TEM data for water samples extracted from the ICET solution at Day 4, Day 15, and Day 30. The purpose of this examination was to determine whether the physical structure of any suspended products exhibits crystalline or amorphous characteristics. These data are also qualitative in nature, consisting generally of a set of high-resolution micrographs followed by companion electron diffraction images. The TEM sample holder consists of a carbon grid that is “lacey,” or filamentary, in nature.

This grid is visible as a relatively large-scale structure in the background of most images. Surface tension in a droplet of liquid suspends the particulates of interest across the grid so that the electron beam can illuminate the sample through the holes without interference from a substrate. Crystalline material will exhibit diffraction patterns unique to the molecular arrangement. Amorphous material that is diffuse or disorganized in structure will not exhibit regular diffraction patterns that can be identified.

Water samples submitted for TEM analysis are not temperature controlled because the temperature cannot be maintained during the examination. A tiny drop of the test solution was transferred to a copper mesh and dried in air for TEM analysis.

In a few cases, data file names that were noted by the operator in the laboratory log were not successfully saved in electronic form. These cases are noted in the transcribed log sheets, but the corresponding images are unavailable and therefore are not presented in the data sequence.

3 TEST RESULTS

This section describes the results obtained from Test #5**.** Some visual observations are first presented as an overview. This overview is followed by more-detailed information organized by the type of samples/data collected. Data and photographs are provided here for the (1) water samples, (2) NUKON^{TM} fiberglass samples, (3) metallic and concrete coupons, (4) sediment, and (5) deposition products. Then, TEM images, UV absorbance spectrum and shear-dependent viscosity measurements of water samples are also discussed.

3.1 General Observations

These observations are taken from the project daily log book. They were meant to capture visual observations of the test solution and/or test samples during the daily sampling activities.

Shortly after the latent debris and crushed concrete were introduced into the tank, the inline flow meter quit working. The unit was removed from the piping, rinsed with RO water, and returned to the piping. The flow meter then operated normally. Four hours after the sprays were activated, the tank solution was very turbid. It was difficult to observe the submerged coupons from the lower view window.

On Day 1, after approximately 24 hours of testing, the water clarity was still poor. The tank solution was a yellow-brown color.

On Day 5, 5 gallons of RO make-up water were added to the tank through the top using a recycle funnel.

On Day 6, the water clarity had improved enough that the opposite side of the tank could be seen through the submerged view window. The eight remaining fiber-pucks (see ICET-PI-018, Rev. 0 in Appendix I), the small fiberglass sample encased in the nylon mesh bag, and the small high-flow fiberglass header sample were placed into the tank at that time.

On Day 10, very small particles were observed on all of the submerged galvanized steel coupons. All other submerged samples appeared to be free of deposits.

On Day 13, 5 gallons of RO make-up water were added to the tank through the top using a recycle funnel.

On Day 17, it was observed that a slight amount of precipitate had settled in the UNM-archived test solution bottle labeled “ICET5-0803-0900-U” (Day 8 sample). In addition, 3 gallons of RO make-up water were added to the tank through the top using a recycle funnel.

On Day 21, four fiber-pucks that had been placed in the tank in the low-flow area on Day 6 were removed. They were placed in a re-closable plastic bag with a small volume of test solution. The bag was enclosed in a 5-gallon bucket and put into an oven set at 60°C.

On Day 22, it was observed that the submerged aluminum coupons had a light coating. The submerged aluminum coupons had a rough, dull surface similar to the inorganic zinc-coated steel coupons.

On Day 22, 5 gallons of RO make-up water were added to the tank through the top using a recycle funnel.

On Day 28, 5 gallons of RO make-up water were added to the tank through the top using a recycle funnel.

After the test was completed, observations of the sample bottles were made. Beginning with the Day 2 water sample, white precipitates were observed after the samples had been at room temperature for several days. These precipitates settled to the bottom of the sample bottles. When the bottles were gently turned upside down, the precipitates formed wispy patterns in the solution. They were re-suspended when the bottles were shaken and could not be seen. It takes 2–3 days for the precipitates to settle again in the sample bottles.

3.1.1. Control of Test Parameters

Recirculation flow rate: Excluding the spray phase, the average recirculation flow rate was 94.6 L/min (25.0 gpm). Recorded recirculation flow rate had a standard deviation of 0.72 L/min, with a range of 94.3 to 99.1 L/min (24.9 to 26.1 gpm).

Temperature: Temperature is recorded at three submerged locations in the ICET tank. The average recorded temperature at these locations was 60.6°C, 60.8°C, and 60.8°C (141.1°F, 141.4°F, and 141.4°F). The standard deviation in temperature recorded by all three thermocouples was within ±0.4°C (± 0.7°F), with a maximum range of 57.7°C to 61.7°C (135.8°F to 143.0°F). The temperature went below 58°C for less than 10 minutes, when make-up water was added quickly to the tank on Day 5. Make-up water was subsequently added at a slower rate to ensure that the temperature did not drop below 58°C.

pH: Before time zero, 6.48 kg of boric acid, 10.0 kg of borax, and 0.284 g of lithium hydroxide were dissolved into the ICET tank. The in-line pH probe, which produced the data in Figure 3-1, provides only an estimated pH measurement. The measured bench-top probe pH was 8.4 at 60°C. During the addition of the HCl, the pH of the system dropped slightly to a value of 8.3. The pH remained in the range of 8.2–8.4 for the duration of the test. This can be seen in Figure 3-2.

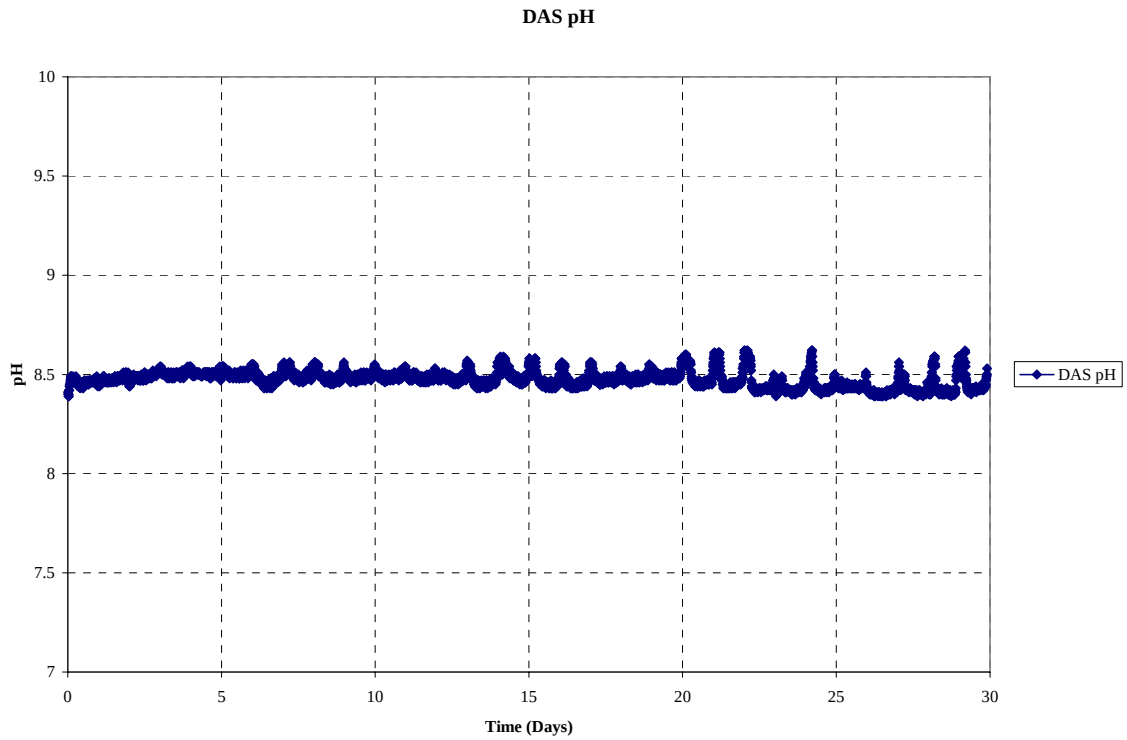

Figure 3-1. In-line pH measurements.

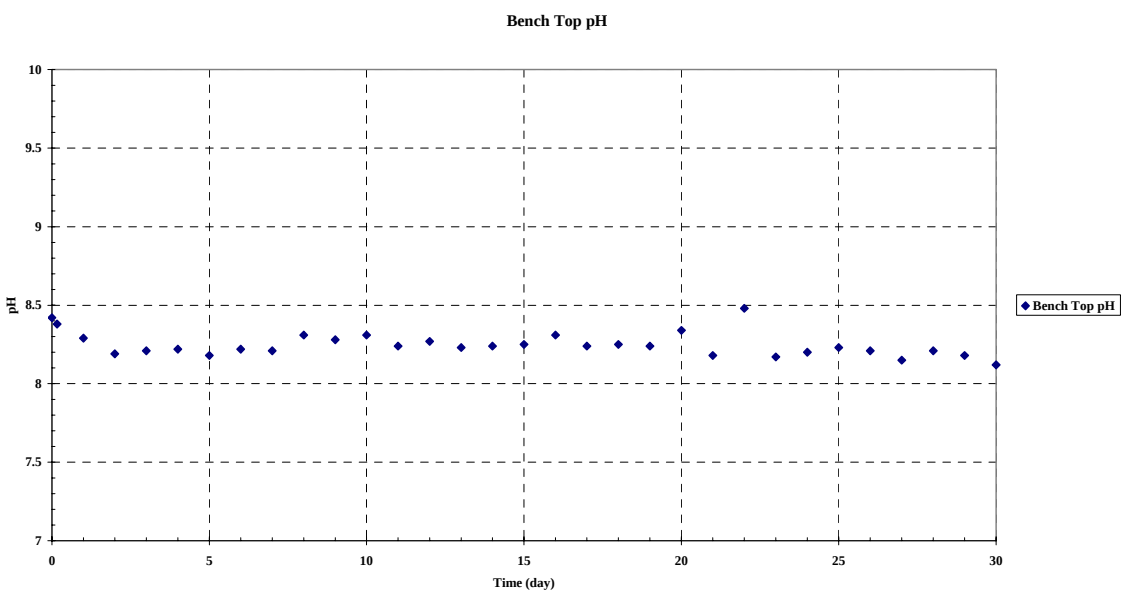

Figure 3-2. Bench-top pH meter results.

3.1.2. Hydrogen Generation

Hydrogen remained at or below 0.15% of the daily samples' air volume (from the tank atmosphere) for the duration of the test as shown in Figure 3-3. All of the measured values were well below the hydrogen safety action threshold of 0.4%.

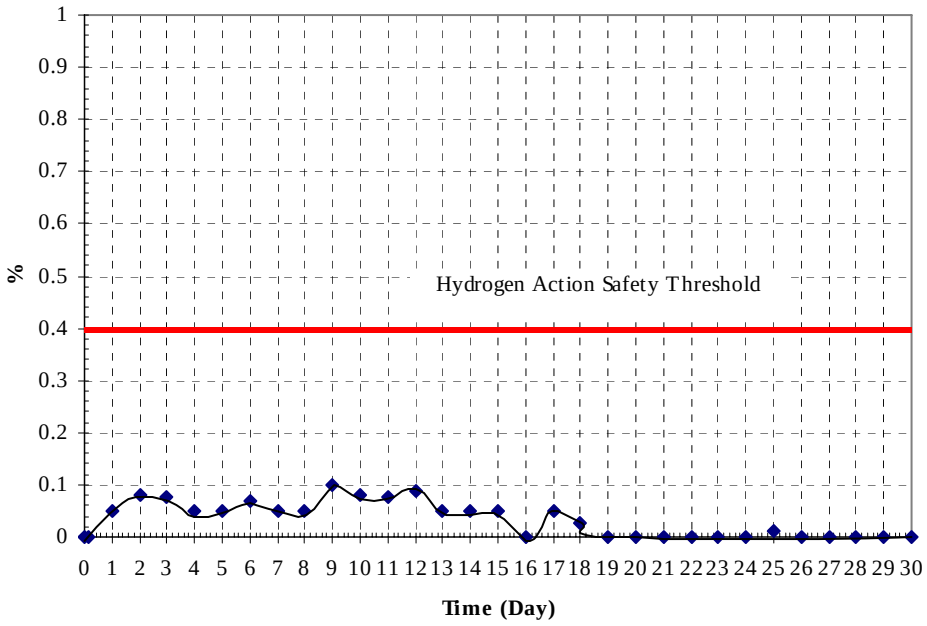

Figure 3-3. Hydrogen generation.

3.2 Water Samples

3.2.1. Wet Chemistry

Wet chemistry analyses included turbidity, TSS, and kinematic viscosity.

Turbidity: The baseline turbidity values, which were taken before the latent debris and concrete dust were added, for the 23°C and 60°C water samples were 0.81 NTU and 0.77 NTU. After the addition of latent debris and concrete dust, the tank solution turbidity was 14.1 NTU. The daily turbidity values are shown in Figure 3-5.

Due to the cloudy nature of the water in the tank after the recirculation pump was turned on, turbidity values were measured at 60°C over the initial 4-hour spray phase, in addition to regular daily monitoring. Figure 3-4 shows the turbidity during this time period. The x-axis on the graph represents the time in hours after the spray nozzles were turned on. As can be seen, a slight decrease in turbidity occurred from the time zero value to 12.4 NTU at 4 hours.

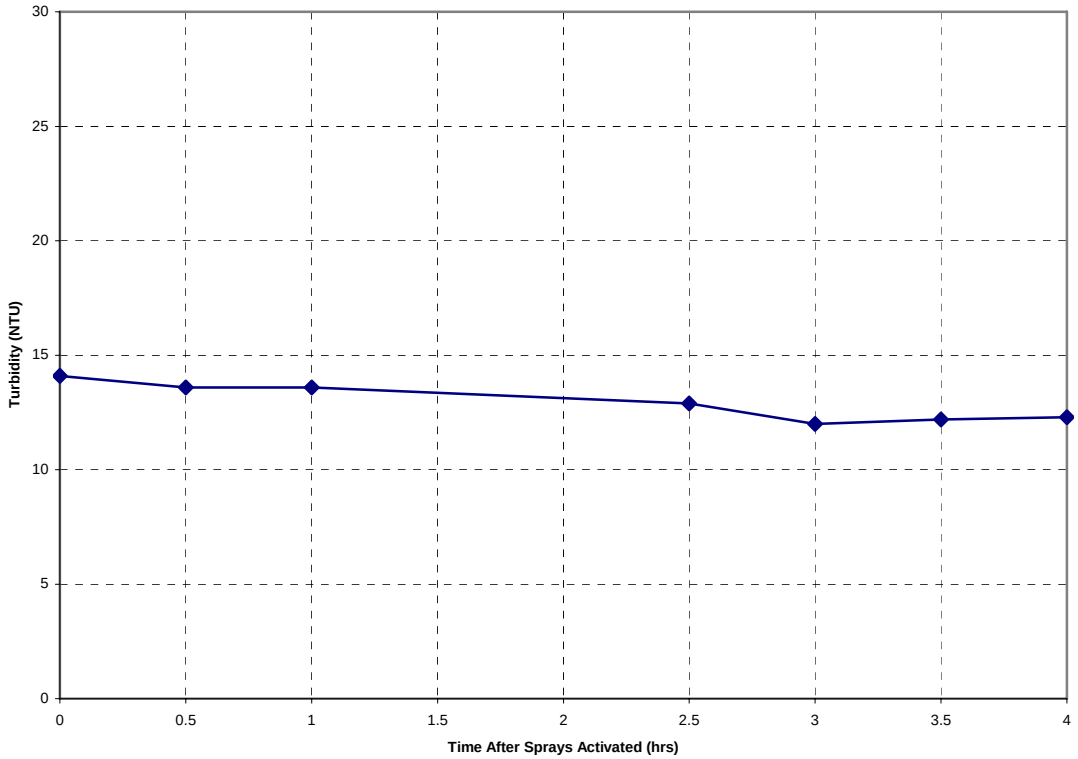

Figure 3-4. Turbidity results during spray phase.

Figure 3-5 shows the daily turbidity values at 23°C and 60°C throughout the test. The 60°C curve exhibited a steady decline over that time period. The 23°C turbidity curve exhibited a similar trend through the first seven days of the test. However, beginning on Day 8, the 23°C turbidity values began to deviate slightly, rising higher than the 60°C readings. From Day 8 through Day 30, the 23°C values were, on average, 2.95 NTU greater than the 60°C values. From Day 21 to Day 30, the mean turbidity measurements for the 60°C and 23°C samples were 0.7 NTU and 4.7 NTU, respectively.

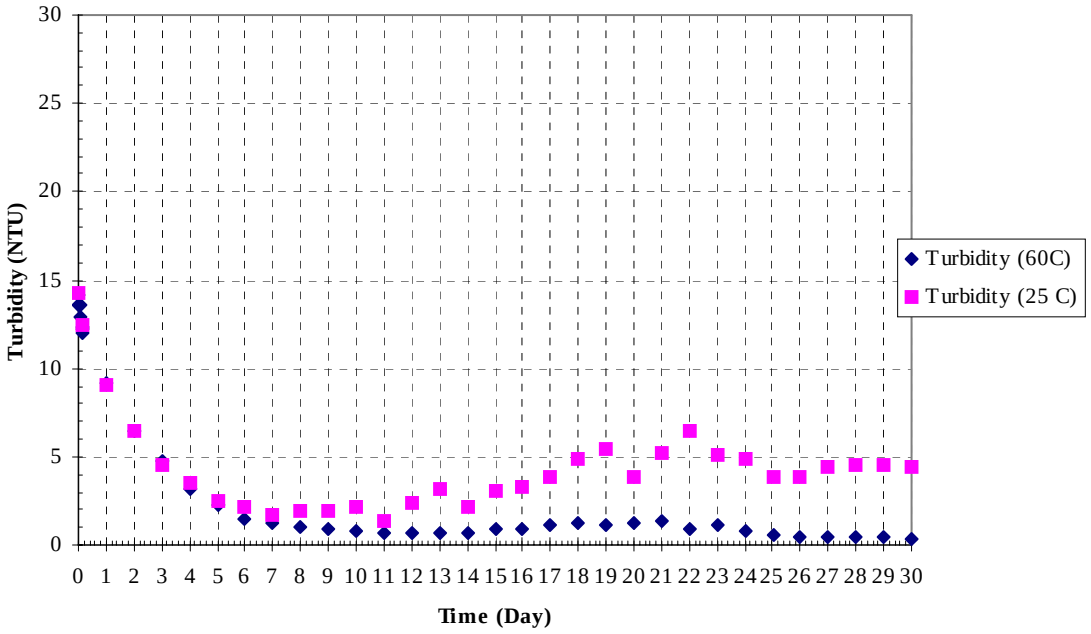

Figure 3-5. Daily turbidity results.

Total Suspended Solids: Total suspended solids (TSS) are measured by running a volume of approximately 500 mL through an in-line filter directly at the sample tap and measuring the dried mass added to the filter. The selected equipment assures that TSS measurements are not affected by temperature-dependent or time-dependent precipitation reactions that may occur once the process solution is removed from the tank. Figure 3-6 presents Test #5 TSS data as the experiment progressed. The baseline TSS measurement, taken prior to time zero, was 16.2 mg/L. At the end of the four-hour spray phase, the TSS value rose to 26.5 mg/L. Following the spray cycle and beginning at 24 hours, the TSS measurements were performed daily. TSS measurements gradually declined over the first seven days, with a Day 7 value of 17.6 mg/L, which is close to the baseline measurement. The TSS measurement remained constant through Days 7 through 9 but began increasing on Day 10 and continued increasing to Day 13. The measurements after Day 13 were somewhat erratic. This unexplained behavior in TSS occurred through Day 21. From Day 22 through the end of the test, the TSS measurements remained similar to the baseline measurement taken at time zero.

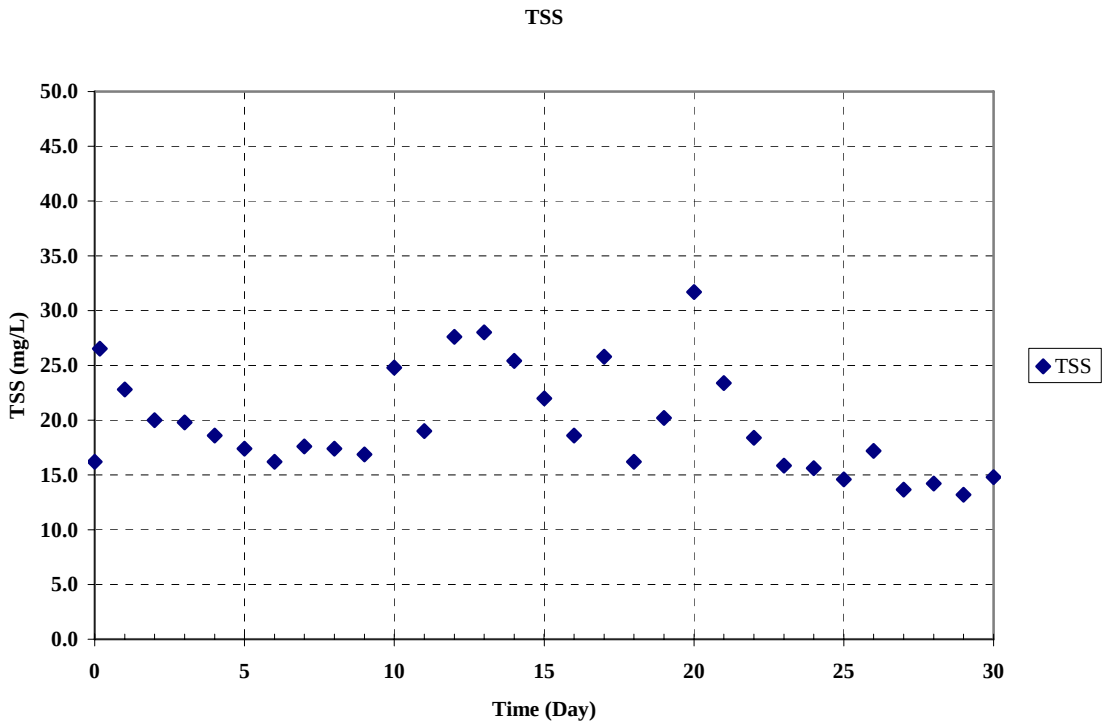

Figure 3-6. Test #5 TSS results.

Kinematic Viscosity: Kinematic viscosity was measured with a Cannon-Fenske capillary viscometer. Viscosity was measured on unfiltered samples, each at a temperature of 60°C ±1.0°C (140°F ±1.8°F) and 23°C ±2.0°C (73.4°F ±3.6°F). Water's viscosity is highly sensitive to temperature, and the allowed temperature range results in a variation of viscosity of 2.9% between 59°C (138.2°F) and 61°C (141.8°F), and a 9.3% variation between 21 C (69.8°F) and 25°C (77.0°F). For this reason, temperature was measured to 0.1°C accuracy with a NIST-traceable thermometer for all viscosity measurements, and the measured viscosity values were corrected to a common temperature to facilitate comparisons. The corrected temperatures were 60.0°C (140°F) and 23.0°C (73.4°F). Throughout Test #5, the viscosity measurements remained relatively constant. The average viscosity for the 23°C measurement was 0.96 mm^2/s, and for the 60°C measurement, it was 0.50 mm^2/s. The viscosity values are shown in Figure 3-7.

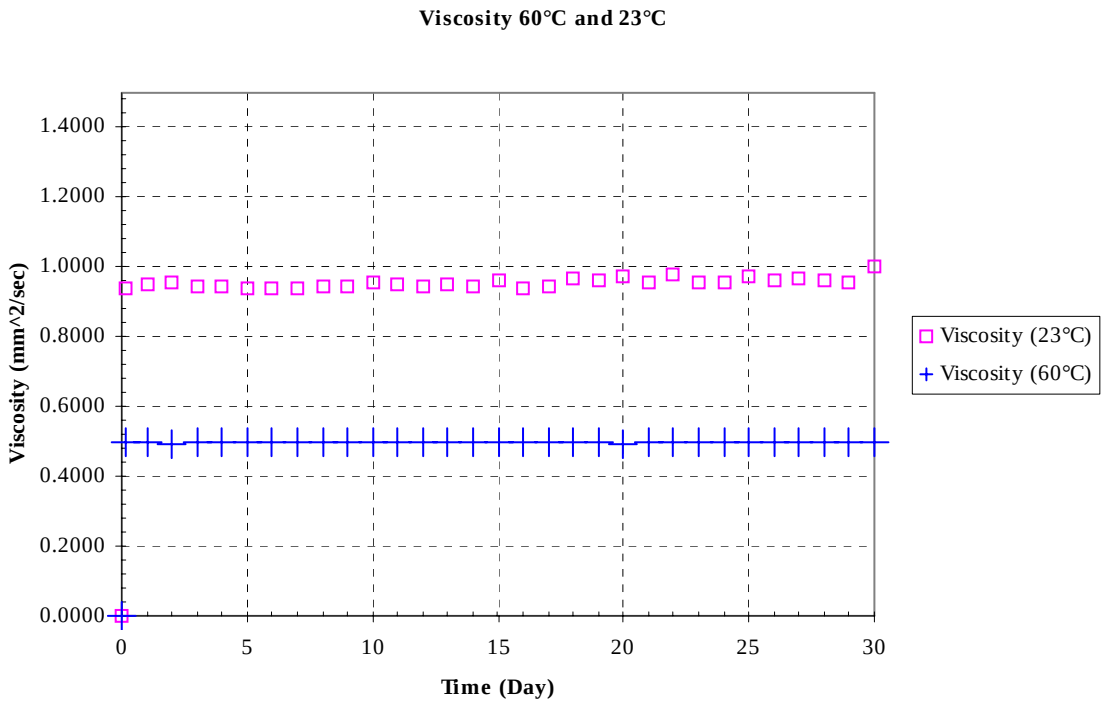

Figure 3-7. Viscosity at 60°C and 23°C.

3.2.2. Metal Ion Concentration

ICP results for daily water samples from Test #5 are displayed in Figures 3-8 through 3-15. Table 3-1 contains ICP results for elements that were analyzed at time zero and 4 hours and on Days 15 and 30. Table 3-1 shows the chloride, boron, lead, lithium, and potassium concentrations. An examination of the figures reveals that copper, iron, magnesium, and zinc were present in trace amounts, below 1 mg/L. It also can be seen that aluminum, calcium, silica, and sodium were present in higher concentrations. The concentrations of aluminum and calcium were verified by re-testing, but the reason for their variations was not determined.

Table 3-1. ICP Results for Selected Elements

Unfiltered Samples					
	Chloride	**Boron**	**Lead**	**Lithium**	**Potassium**
Sample Time	mg/L				
Baseline	1.5	2580	0.02	0.10	3.0
4 Hours	40.0	2860	0.03	0.11	4.3
Day 15	41.6	1920	0.02	0.11	5.2
Day 30	36.6	2320	0.02	0.10	8.4

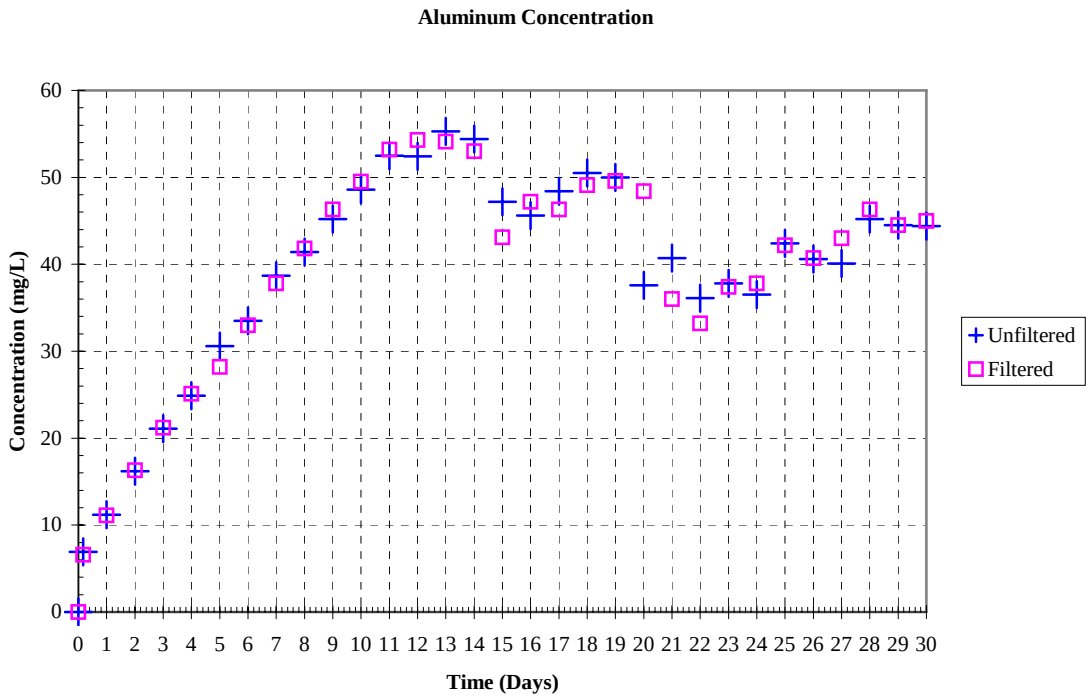

Figure 3-8. Aluminum concentration.

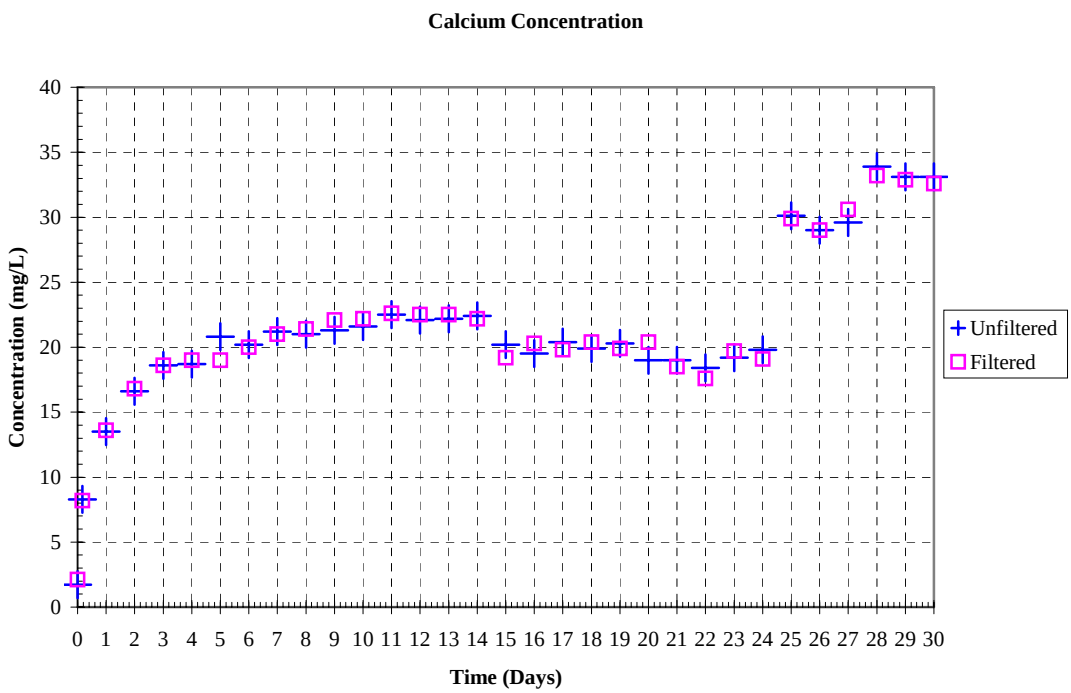

Figure 3-9. Calcium concentration.

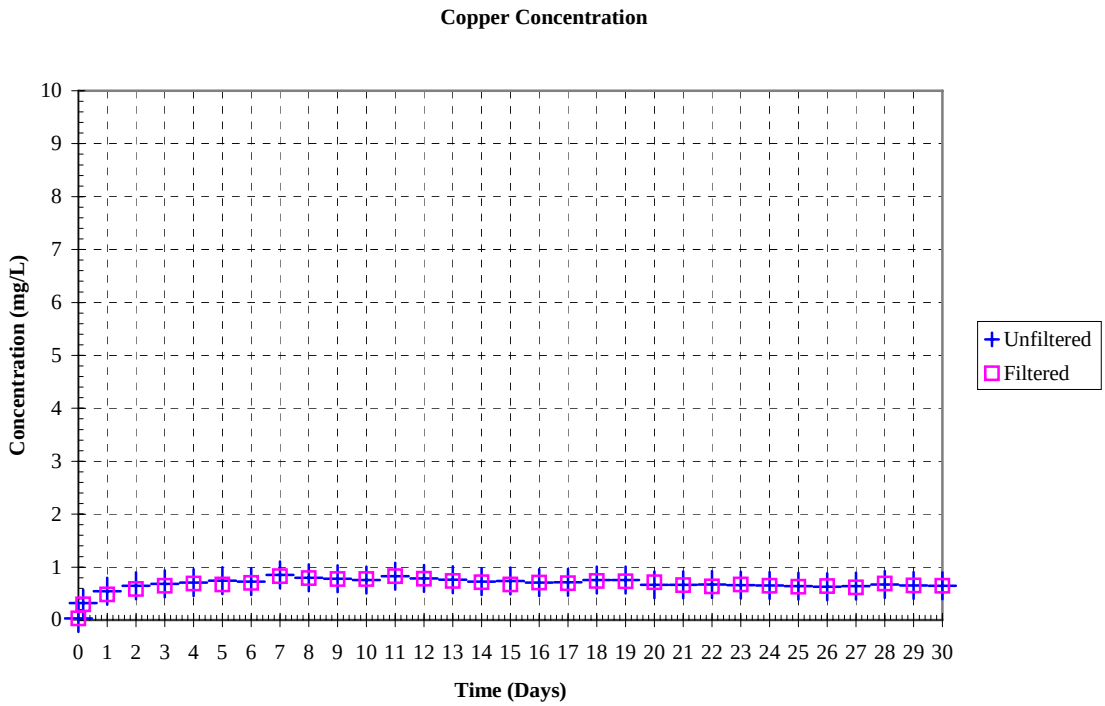

Figure 3-10. Copper concentration.

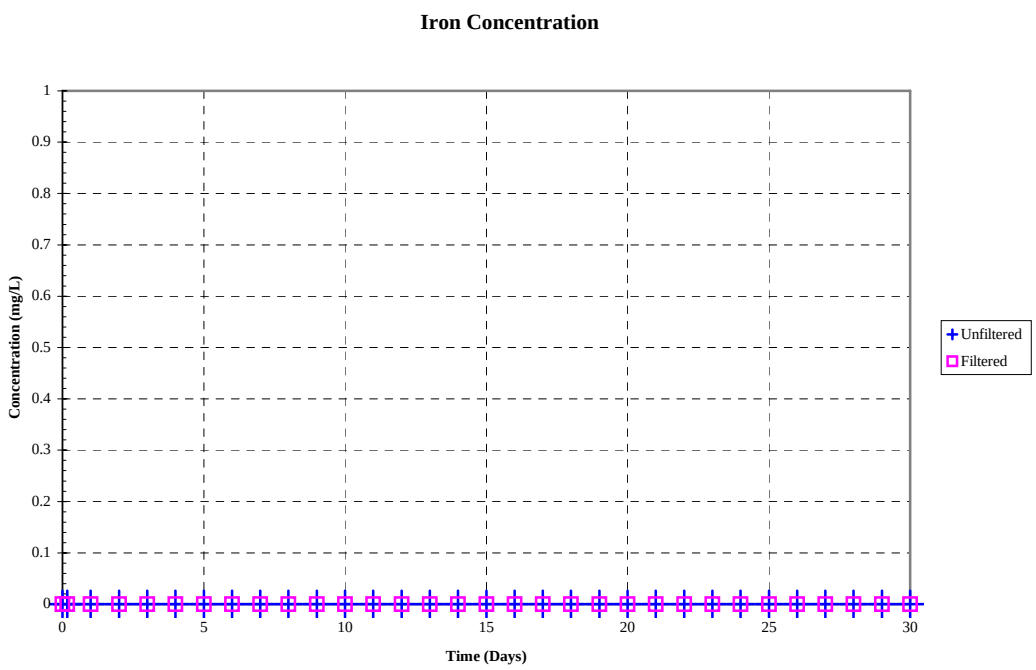

Figure 3-11. Iron concentration.

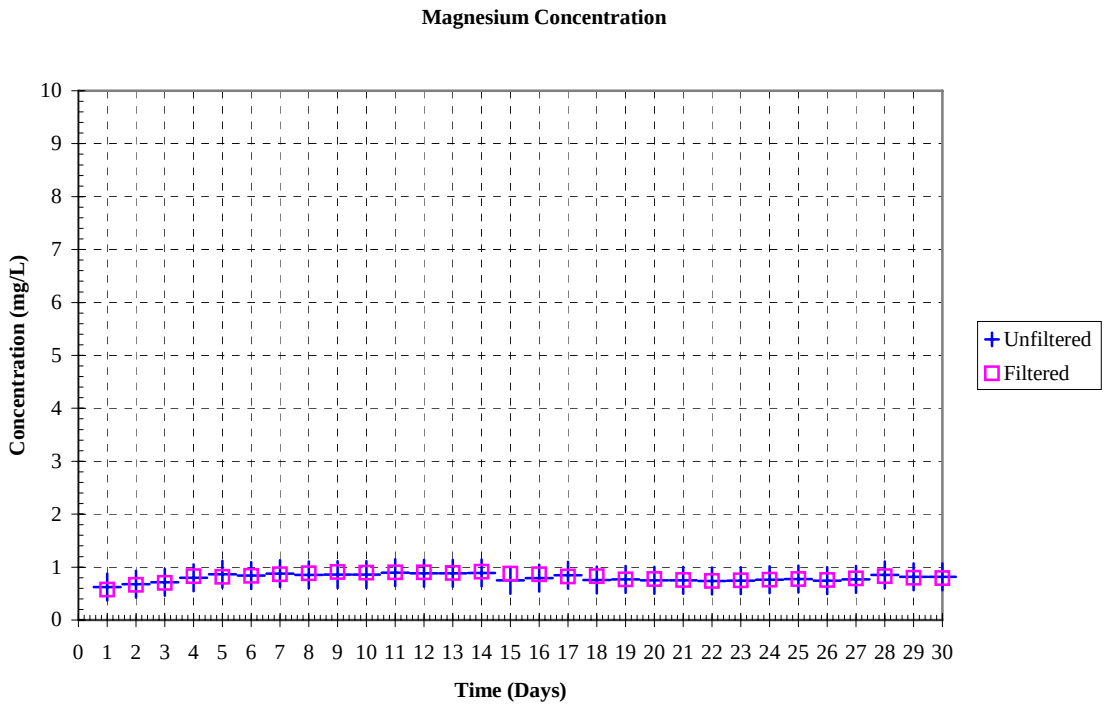

Figure 3-12. Magnesium concentration.

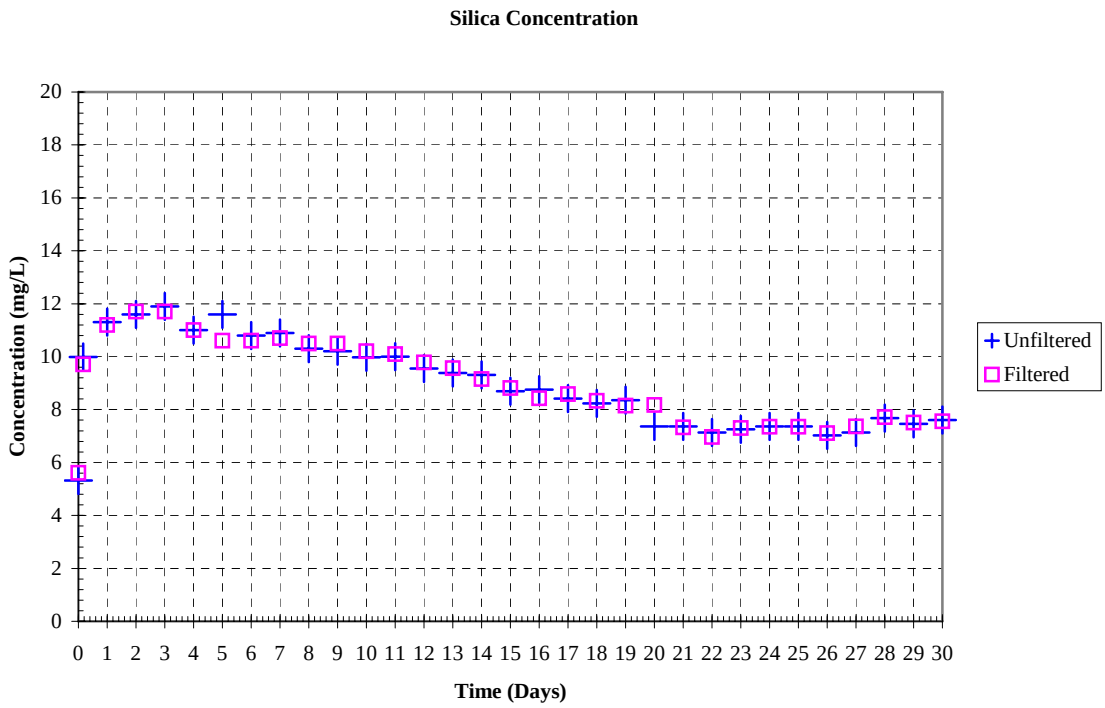

Figure 3-13. Silica concentration.

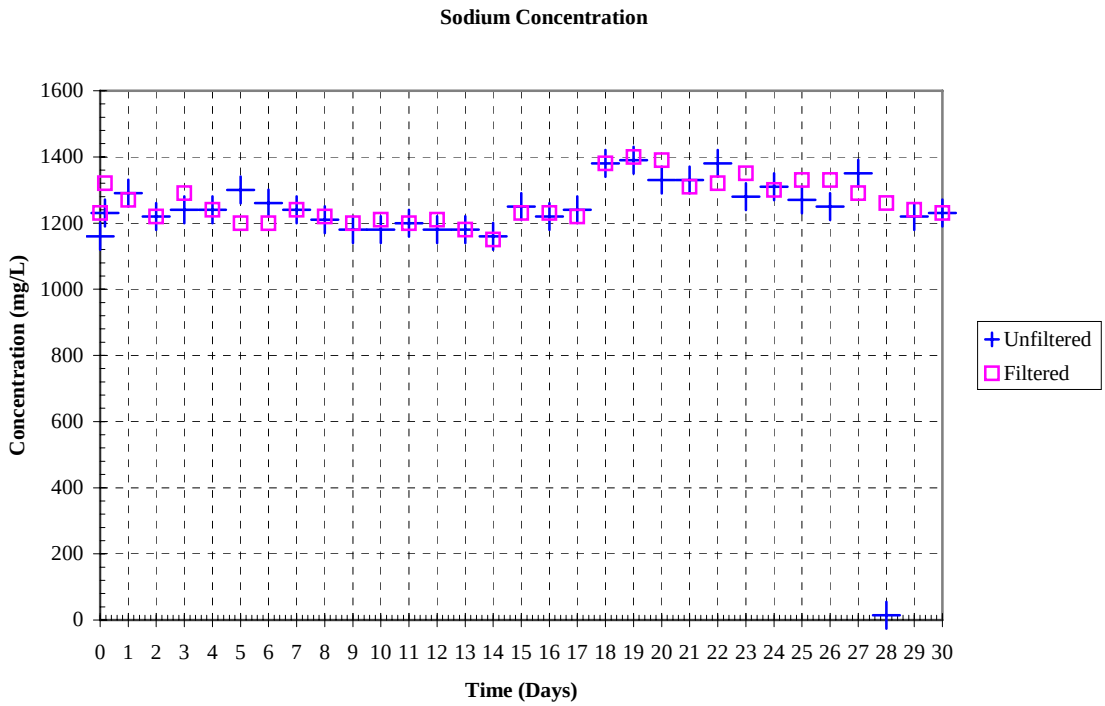

Figure 3-14. Sodium concentration.

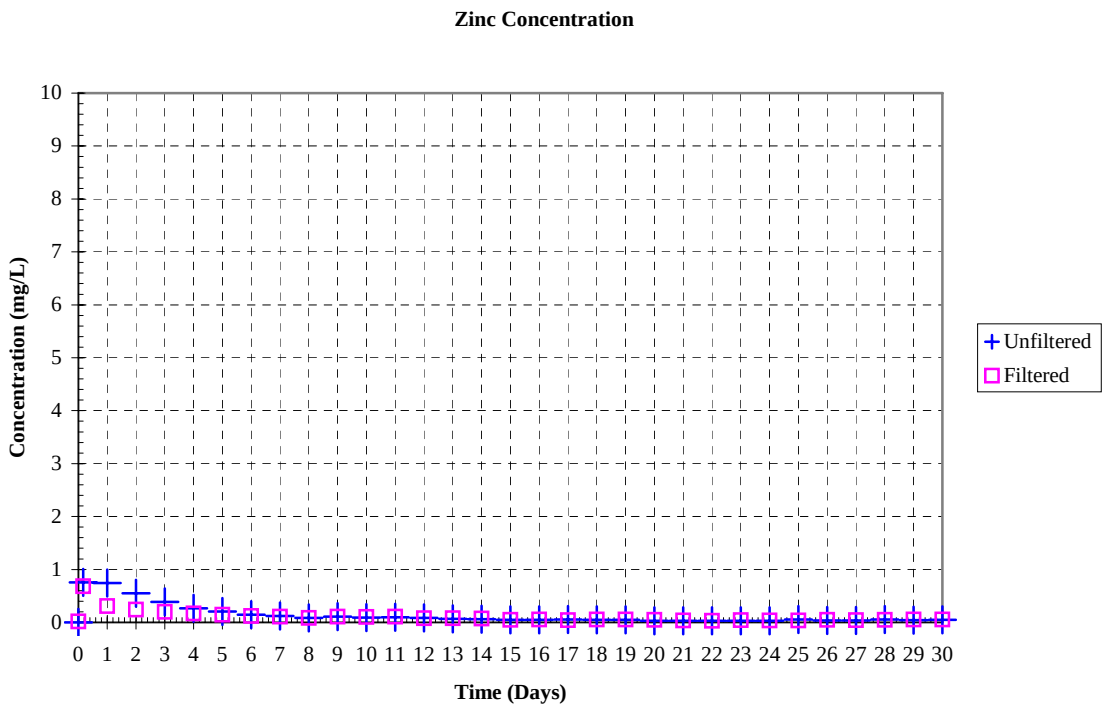

Figure 3-15. Zinc concentration.

3.3 Insulation

Test #5 contained NUKON™ fiberglass samples. The fiberglass samples were thoroughly investigated, with samples being removed from the tank on Days 4, 15, and 30.

3.3.1 Deposits in Fiberglass Samples

The fiberglass samples were contained in SS mesh bags to minimize migration of the fiberglass throughout the tank and piping. Small mesh envelopes, approximately 4 in. square, containing approximately 5 g of fiber, were pulled out of the tank periodically for SEM examination. These sample envelopes were placed in a range of water flow conditions, but none experienced direct water flow through the fiberglass. All were thoroughly immersed in the test solution until they were recovered from the tank.

There were four fiberglass locations in the tank that were examined in this test, including the low-flow areas, the high-flow areas, the drain collar, and the birdcage. (See Subsection 2.4.1.1 for descriptions of the fiberglass samples.) Both the exterior and the interior of the fiberglass samples from each location were examined. Subsections 3.3.1.1 through 3.3.1.10 give the ESEM/SEM/EDS results according to the location of the fiberglass samples in the tank and when the sample was removed from the tank. The different samples include Day 4 low-flow, Day 15 low-flow, Day 15 high-flow, Day 30 low-flow, Day 30 low-flow in a big envelope, Day 30 high-flow, Day 30 high-flow in front of a header, Day 30 in nylon mesh, Day 30 drain collar, and Day 30 birdcage. The corresponding figures are Figures 3-16 through 3-70. Additional micrographs of fiberglass samples are presented in Appendices A, B, and C.

In general, particulate deposits were found on only the exterior of the fiberglass. This result suggests that almost all of the particulate deposits were physically retained at the fiberglass exterior. Because there was no significant water flow directly through the fiber, the migration of particulate deposits into the fiberglass interior was insignificant. Comparing the amount of particulate deposits, the greatest amount was found on the drain collar exterior, especially the exterior farthest from the drain screen. Small amounts of particulate deposits were found on the fiberglass exterior within the birdcage and within the big envelope in a low-flow zone. All other fiberglass exteriors were relatively clean, and no significant particulate deposits were found. EDS shows that the particulate deposits on the drain collar exterior were mainly composed of O, Al, Na, Ca, Mg, C, and possibly Si. Unlike the exterior, the interior of the fiberglass samples at each location was relatively clean. Only flocculent and web-like deposits were observed. The web-like deposits were not present on Day 30 samples, which may be due to a change of the solution chemistry. There was no significant trend of the flocculent deposits with respect to either the location or time. The flocculent and web-like deposits were primarily composed of O, Na, Ca, Mg, Al, B, C, and possibly Si. These deposits were likely formed by chemical precipitation during dehydration of the samples after the samples were removed from the tank.

Results also show that the mesh material, i.e., stainless steel or nylon, did not significantly affect the deposits on the fiberglass. In addition, because of suspended particles settling out of the test solution, the Day 30 high-flow fiberglass (placed in front of a header on Day 6) sample exterior had much less particulate deposits attached/retained than did the high-flow samples put in the tank on Day 0.

3.3.1.1 Day 4 Low-Flow Fiberglass Samples

Since there was no significant water flowing through the fiberglass samples during the test, the migration of particulate deposits from the solution into the fiberglass interior was insignificant. ESEM results revealed some deposits on both the exterior and the interior of the low-flow fiberglass samples after 4 days of the test. These deposits were either formed like a webbing among glass fibers (see Figures 3-17 and 3-21) or flocculent (see Figure 3-19). The deposits are likely of chemical origin instead of being physically attached/retained. They may be formed during the drying process (semi-dehydrated) of the samples during ESEM analysis. EDS results indicated that both types of deposits were commonly composed of O, Na, Ca, Mg, Al, B, C, and possibly Si. Comparing the amount of the deposits on fiberglass revealed no significant difference between the interior and exterior samples. Again, this fact may be explained by the likely chemical origin of the deposits, since chemical precipitation would occur to a similar degree on both the exterior and the interior fiberglass samples if the concentrations were similar. Figures 3-16 through 3-22 show the Day 4 low-flow fiberglass results.

Figure 3-16. Environmental SEM image magnified 100 times for a Test #5 Day 4 low-flow exterior fiberglass sample.

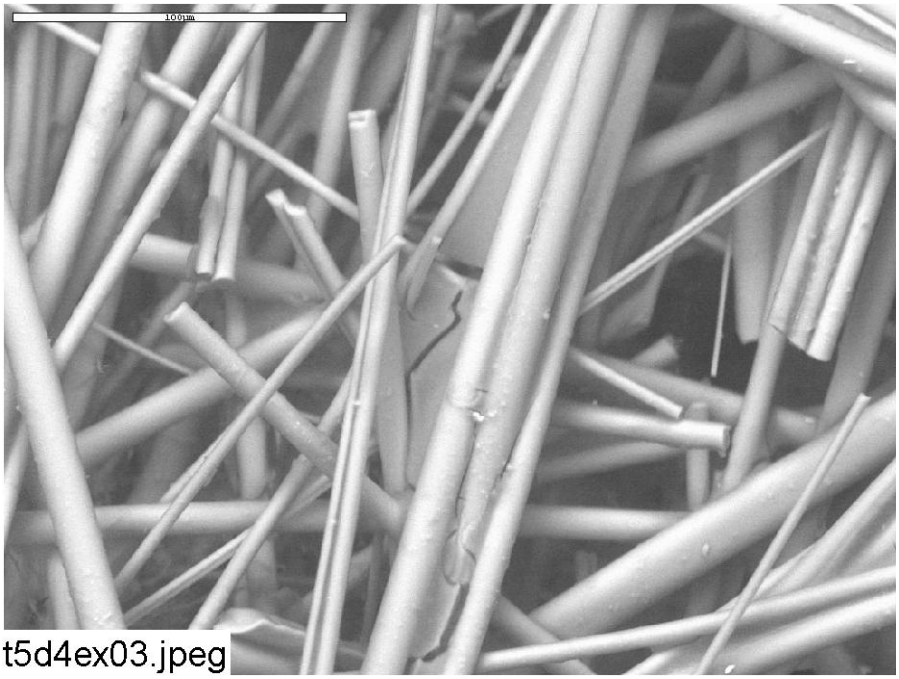

Figure 3-17. Environmental SEM image magnified 500 times for a Test #5 Day 4 low-flow exterior fiberglass sample.

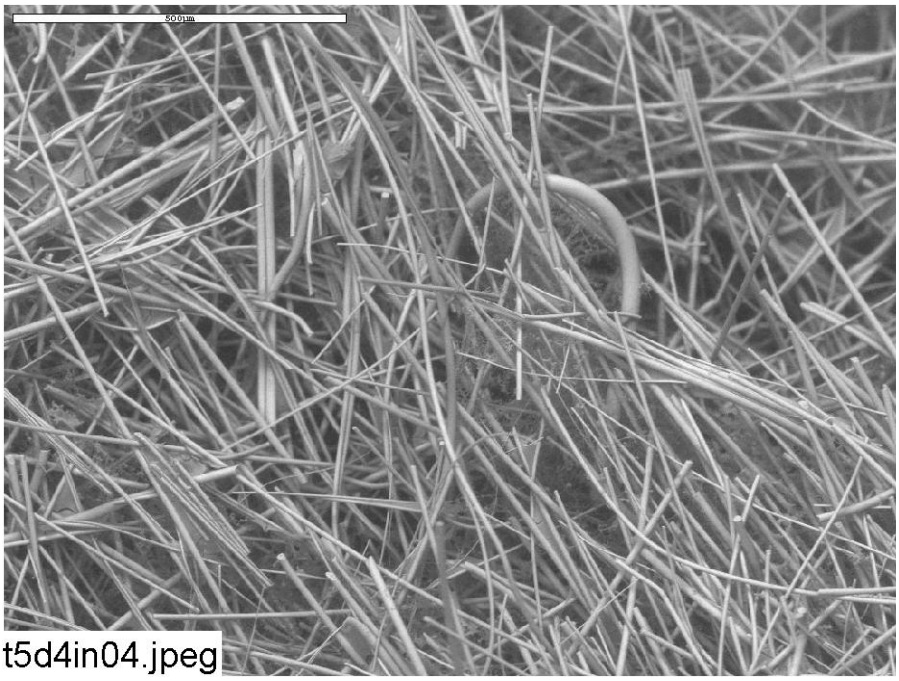

Figure 3-18. Environmental SEM image magnified 100 times for a Test #5 Day 4 low-flow interior fiberglass sample.

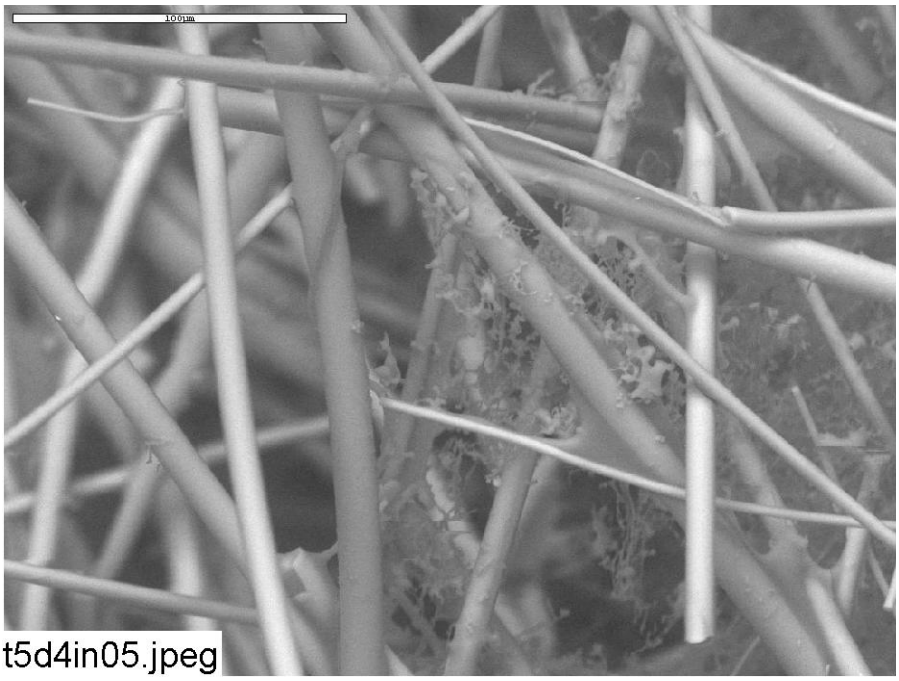

Figure 3-19. Environmental SEM image magnified 500 times for a Test #5 Day 4 low-flow interior fiberglass sample.

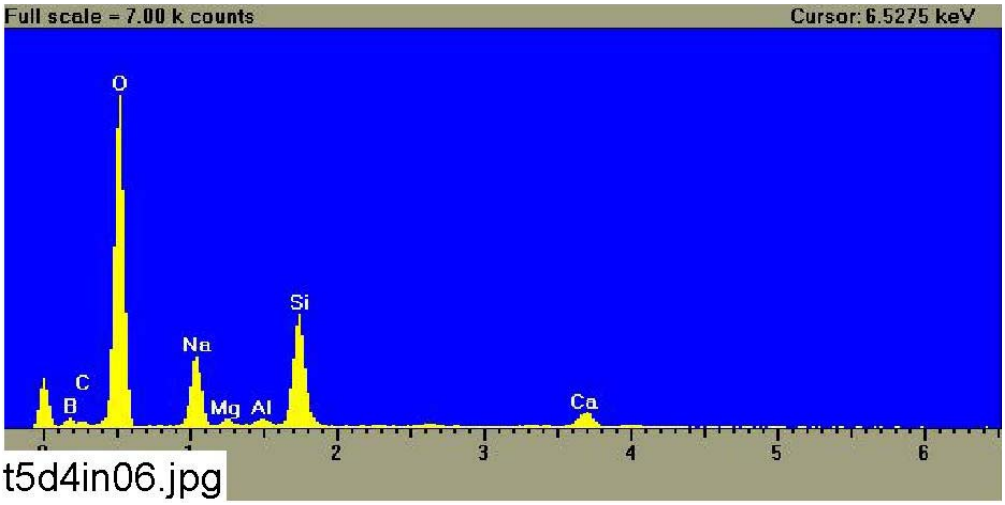

Figure 3-20. EDS counting spectrum for the flocculence between the fibers shown in Figure 3-19.

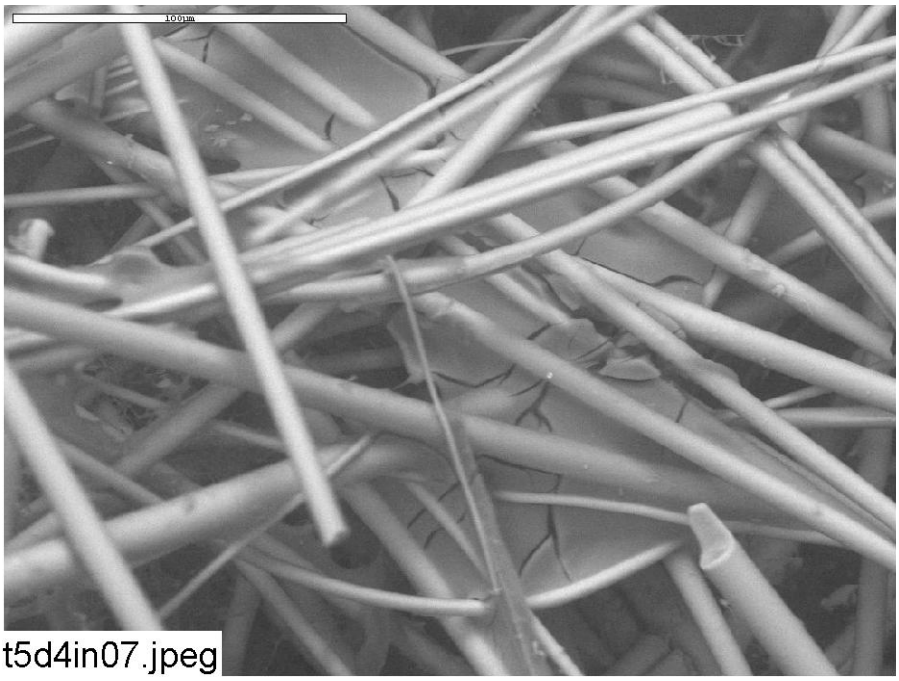

Figure 3-21. Environmental SEM image magnified 500 times for a Test #5 Day 4 low-flow interior fiberglass sample.

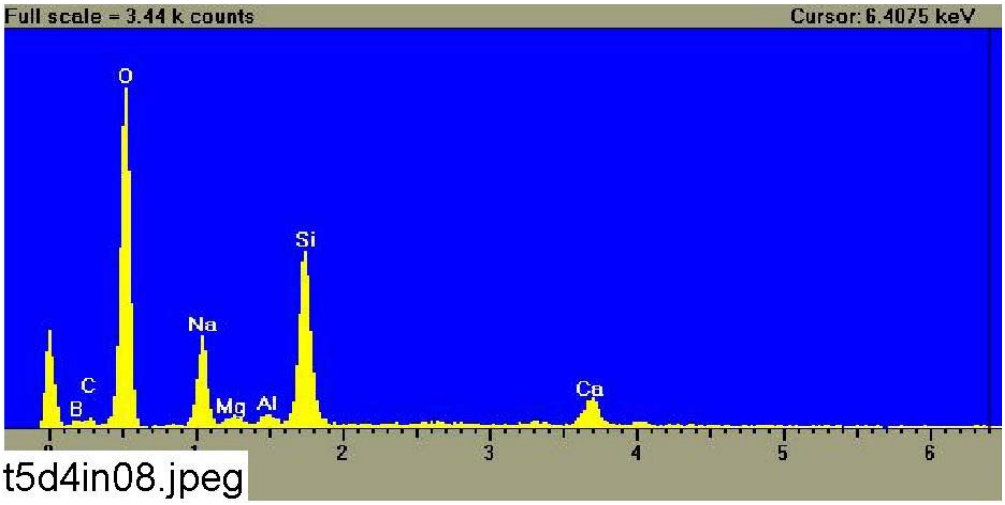

Figure 3-22. EDS counting spectrum for the web-like deposits between the fibers in Figure 3-21.

3.3.1.2 Day 15 Low-Flow Fiberglass Samples

Similar to the Day 4 samples, some flocculent and web-like deposits were found on the fiberglass exterior and interior of the Day 15 samples. EDS analyses showed that the deposits were mainly composed of O, Na, Ca, Mg, Al, B, C, and possibly Si. There was no significant increase in the amount of deposits on Day 15 samples compared with Day 4 samples. In addition, the difference in the amount of deposits on the exterior and the interior Day 15 low-flow fiberglass samples was insignificant. Figures 3-23 through 3-28 show the Day 15 low-flow fiberglass results.

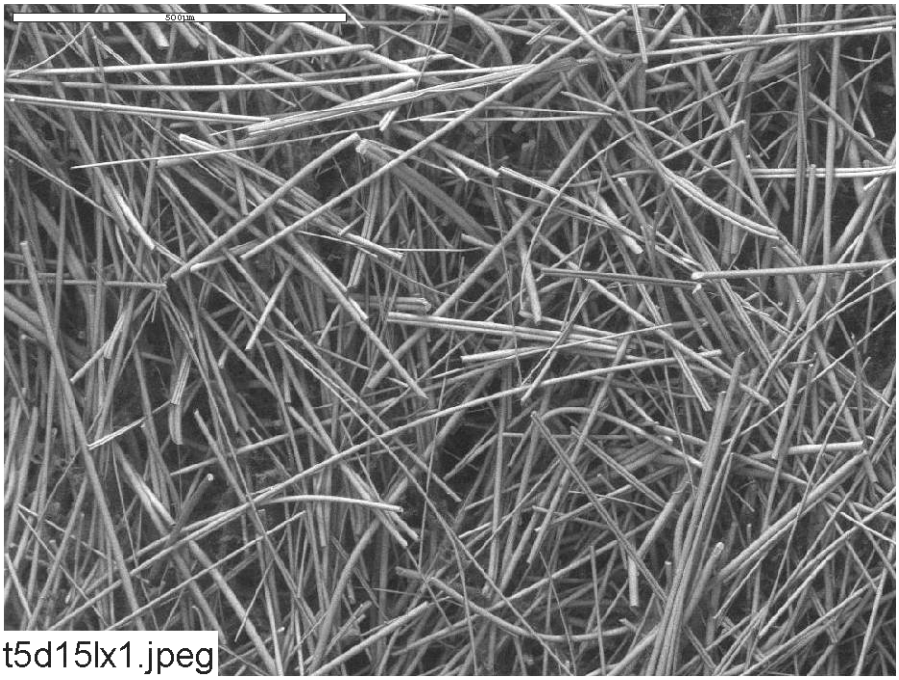

Figure 3-23. Environmental SEM image magnified 100 times for a Test #5 Day 15 low-flow exterior fiberglass sample.

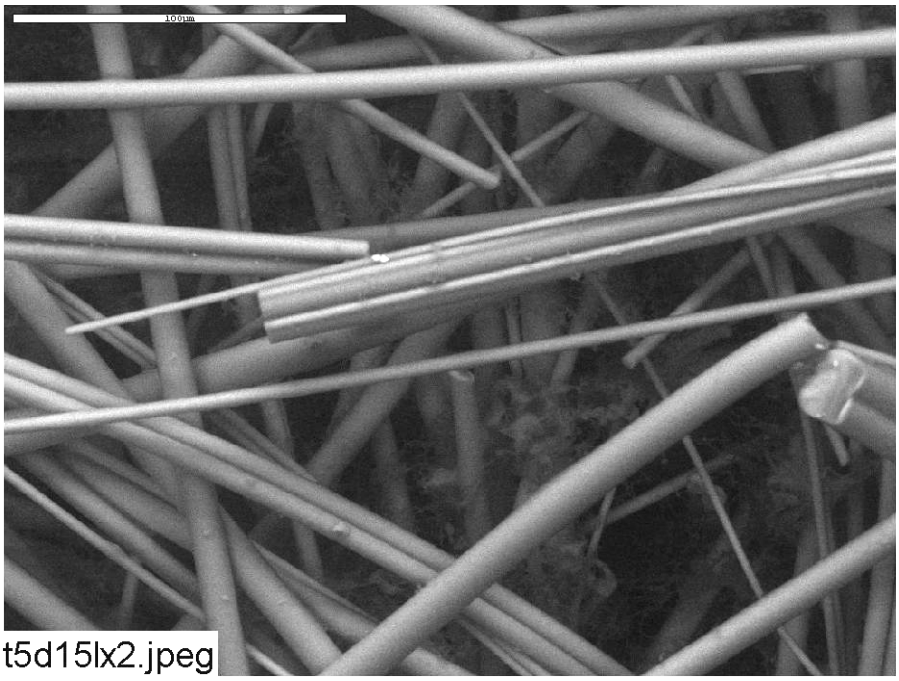

Figure 3-24. Environmental SEM image magnified 500 times for a Test #5 Day 15 low-flow exterior fiberglass sample.

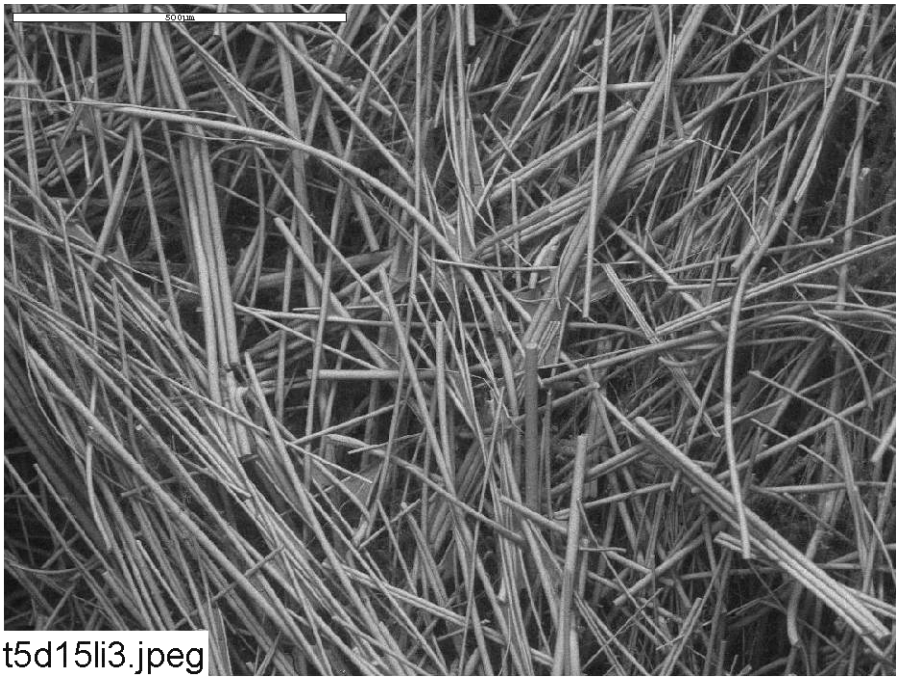

Figure 3-25. Environmental SEM image magnified 100 times for a Test #5 Day 15 low-flow interior fiberglass sample.

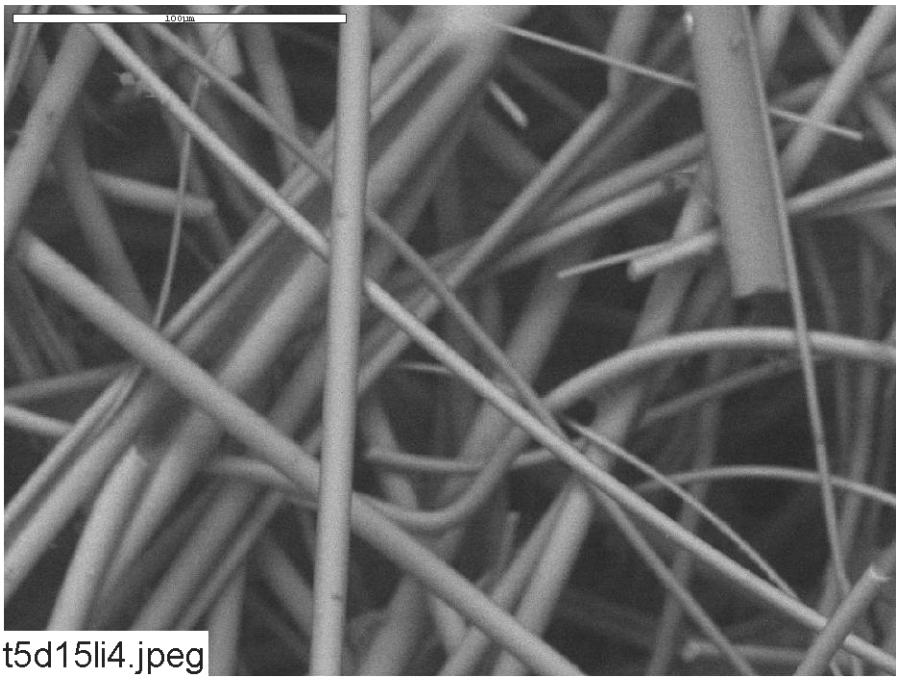

Figure 3-26. Environmental SEM image magnified 500 times for a Test #5 Day 15 low-flow interior fiberglass sample.

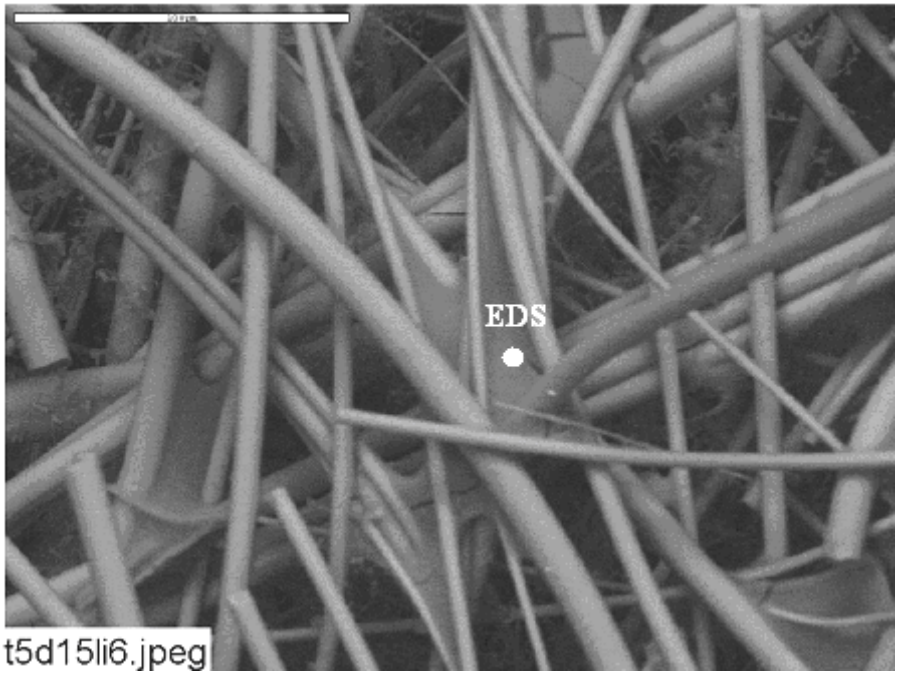

Figure 3-27. Annotated environmental SEM image magnified 500 times for a Test #5 Day 15 low-flow interior fiberglass sample.

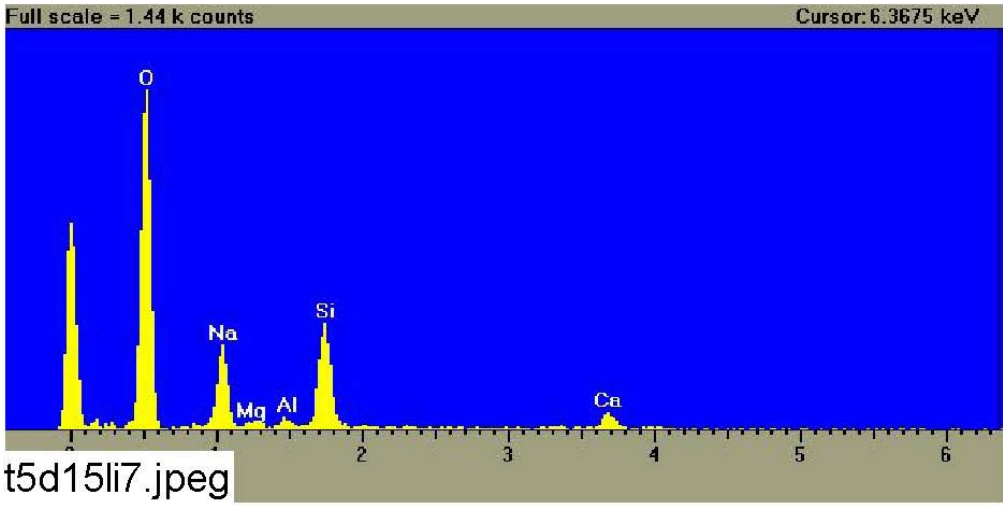

Figure 3-28. EDS counting spectrum for the web-like deposits between the fibers in Figure 3-27.

3.3.1.3 Day 15 High-Flow Fiberglass Samples

In contrast to the Day 15 low-flow samples, no web-like deposits were found in the Day 15 high-flow fiberglass samples. However, flocculent deposits were found on both the exterior and interior Day 15 high-flow fiberglass samples. There was no significant difference regarding the amount of the flocculent deposits between the exterior and the interior fiberglass samples, suggesting the deposits' likely chemical origin. The visual

appearance of the flocculent deposits was similar to other fiberglass samples, so no additional EDS was performed. In addition, no particulate deposits were observed on either the exterior or interior fiberglass samples. Figures 3-29 through 3-32 show the Day 15 high-flow fiberglass results.

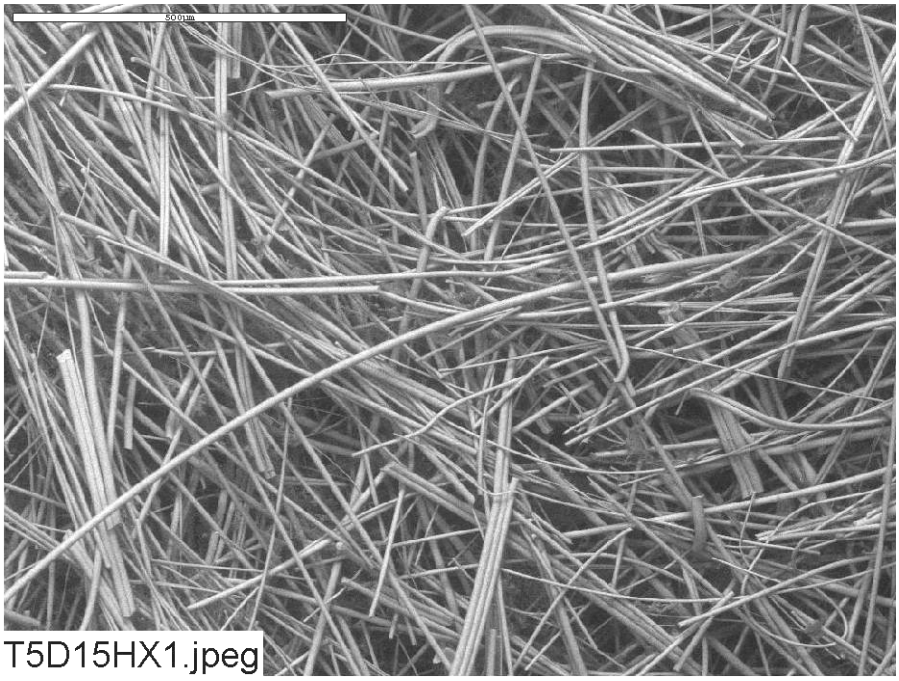

Figure 3-29. Environmental SEM image magnified 100 times for a Test #5 Day 15 high-flow exterior fiberglass sample.

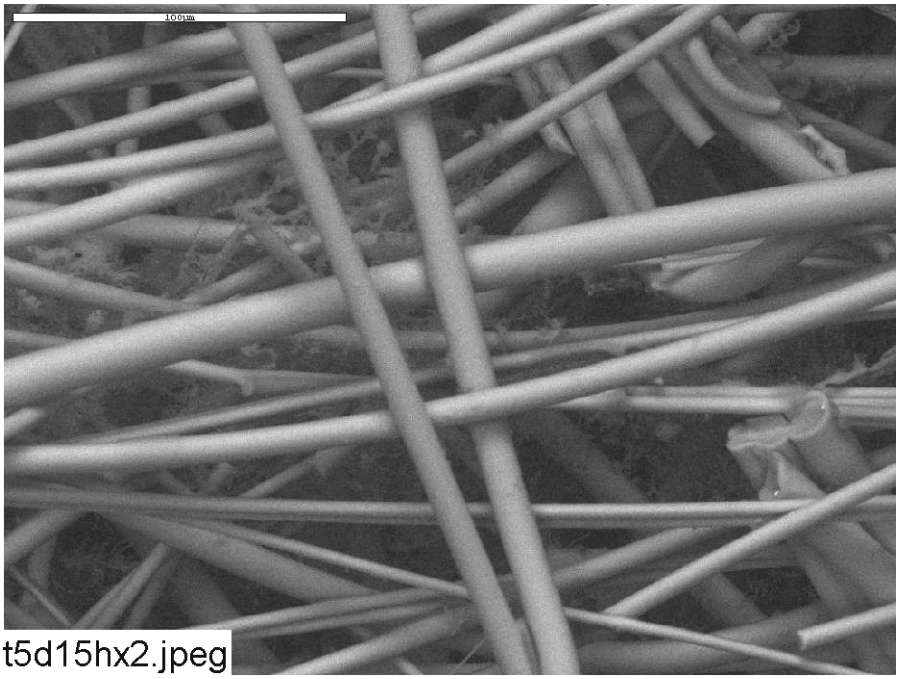

Figure 3-30. Environmental SEM image magnified 500 times for a Test #5 Day 15 high-flow exterior fiberglass sample.

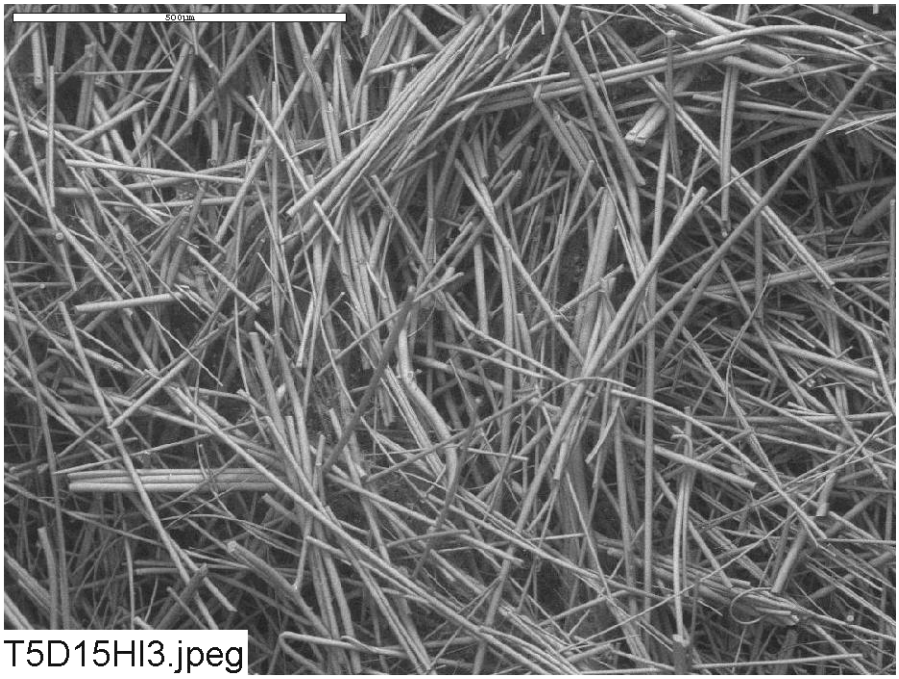

Figure 3-31. Environmental SEM image magnified 100 times for a Test #5 Day 15 high-flow interior fiberglass sample.

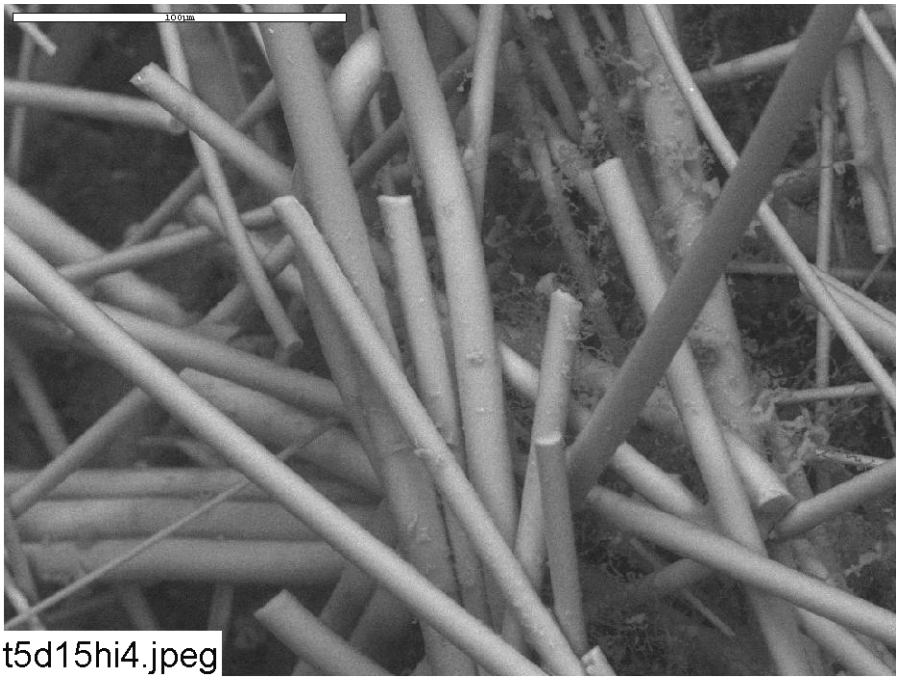

Figure 3-32. Environmental SEM image magnified 500 times for a Test #5 Day 15 high-flow interior fiberglass sample.

3.3.1.4 Day 30 Low-Flow Fiberglass Samples

No observable increase in the flocculent deposits was found with the Day 30 low-flow fiberglass samples compared with Day 4 and Day 15 low-flow fiberglass samples. No significant difference was found regarding the amount of the flocculent deposits between the exterior and the interior of the Day 30 low-flow fiberglass samples. No web-like deposits were found on either the exterior or interior of the Day 30 low-flow fiberglass samples. Furthermore, no particulate deposits were observed on either the exterior or interior fiberglass samples. Figures 3-33 through 3-36 show the Day 30 low-flow fiberglass results.

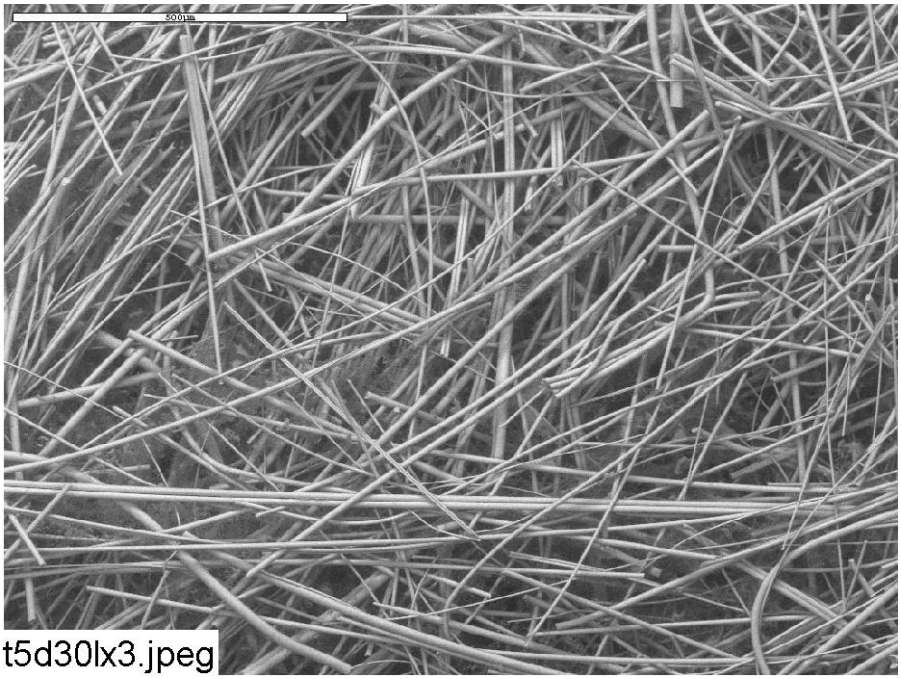

Figure 3-33. Environmental SEM image magnified 100 times for a Test #5 Day 30 low-flow exterior fiberglass sample.

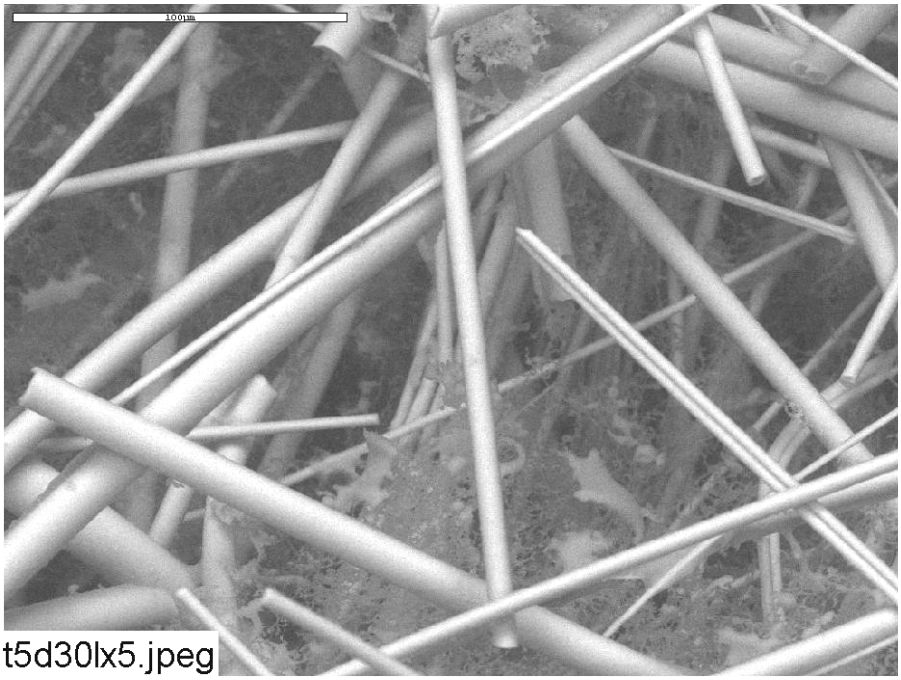

Figure 3-34. Environmental SEM image magnified 500 times for a Test #5 Day 30 low-flow exterior fiberglass sample.

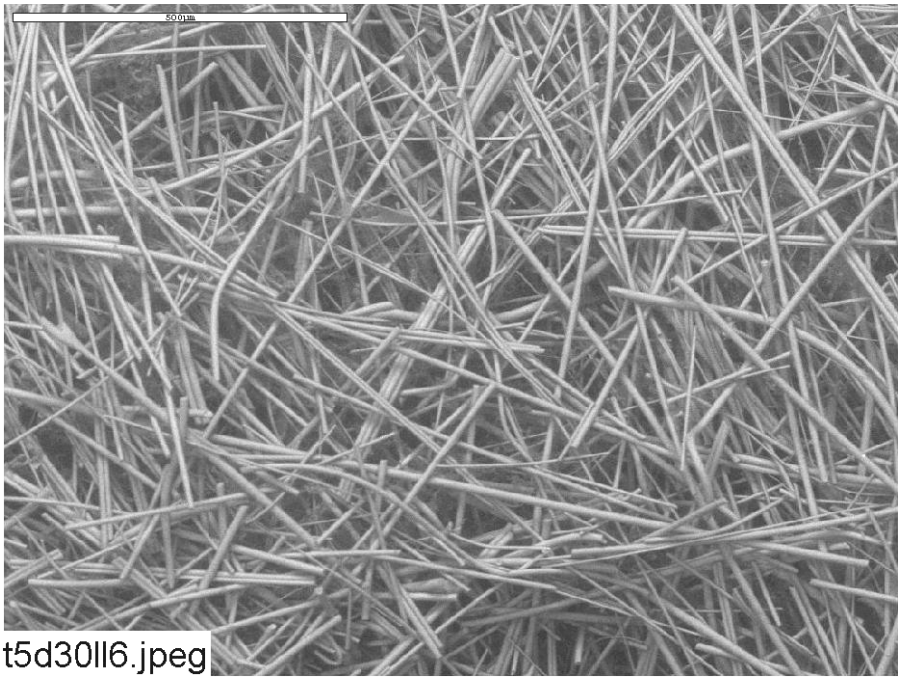

Figure 3-35. Environmental SEM image magnified 100 times for a Test #5 Day 30 low-flow interior fiberglass sample.

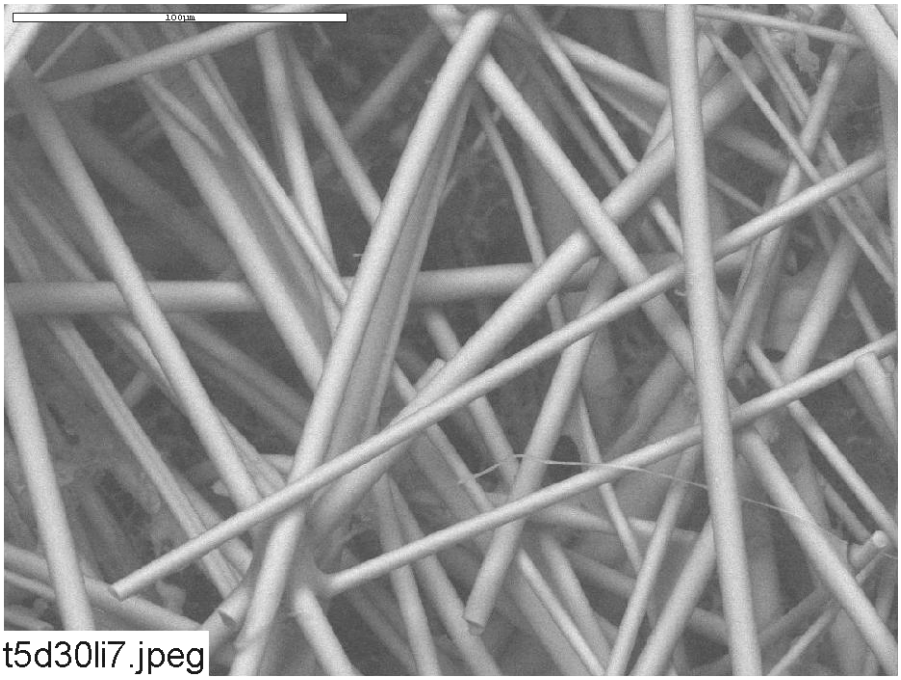

Figure 3-36. Environmental SEM image magnified 500 times for a Test #5 Day 30 low-flow interior fiberglass sample.

3.3.1.5 Day 30 Low-Flow Fiberglass Samples in the Big Envelope

The big envelope was located on the tank bottom, contacting the test sediment on the bottom of the tank. In contrast to Day 4, Day 15, and other Day 30 low-flow fiberglass samples, a small amount of particulate deposits were observed on the fiberglass exterior of the Day 30 low-flow samples in the big envelope. EDS results show that the particulate deposits (see Figure 3-39) were composed of O, Na, Ca, Mg, Al, and possibly Si. However, no particulate deposits were observed on the fiberglass interior. Instead, flocculent deposits were found on the fiberglass interior, as they were with other interior fiberglass samples. Figures 3-37 through 3-41 illustrate these deposits for the Day 30 low-flow fiberglass in the big envelope.

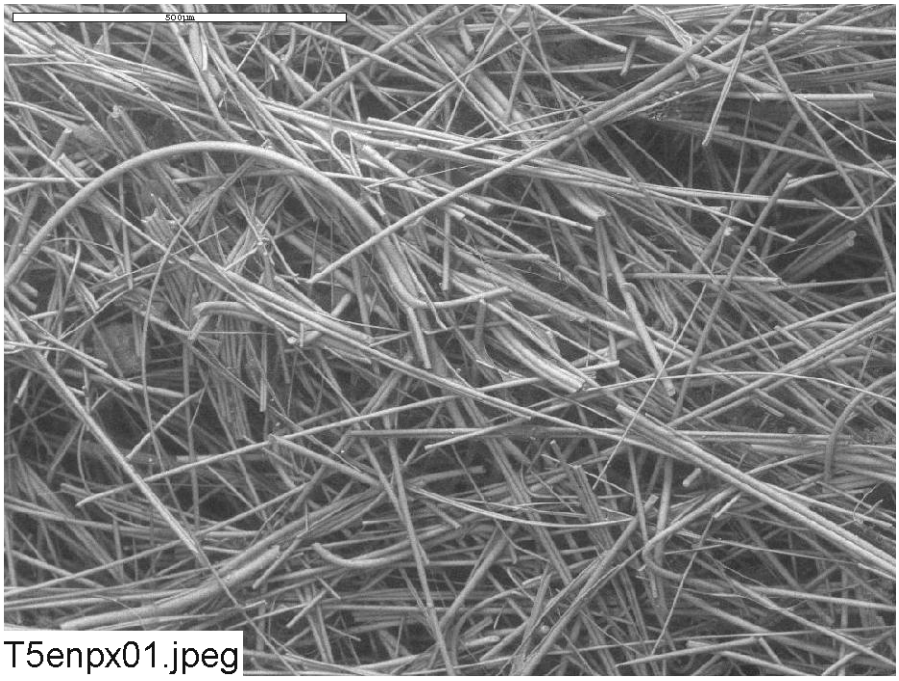

Figure 3-37. Environmental SEM image magnified 100 times for a Test #5 Day 30 low-flow exterior fiberglass sample in a big envelope.

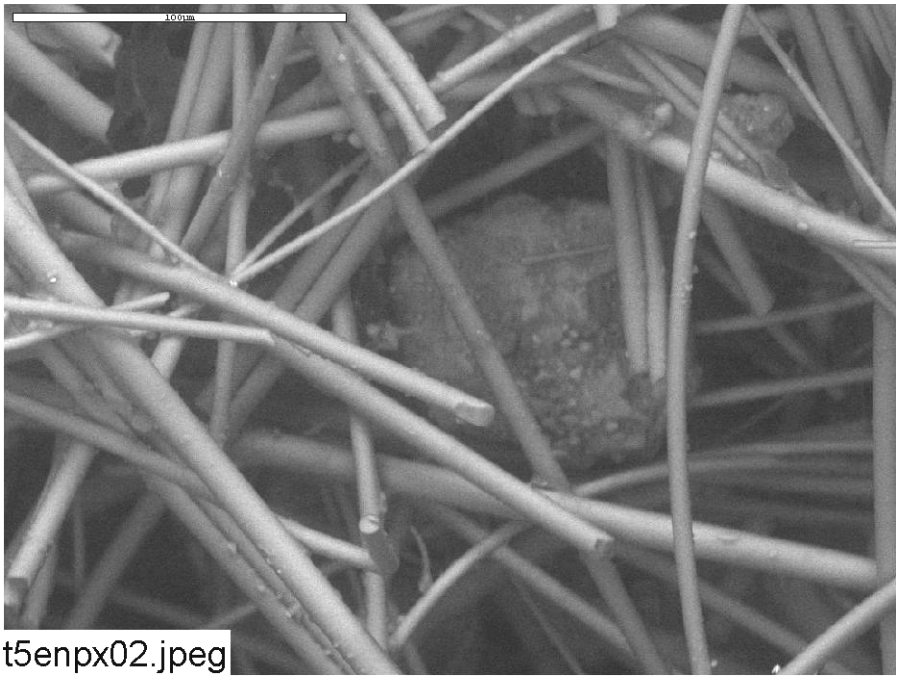

Figure 3-38. Environmental SEM image magnified 500 times for a Test #5 Day 30 low-flow exterior fiberglass sample in a big envelope.

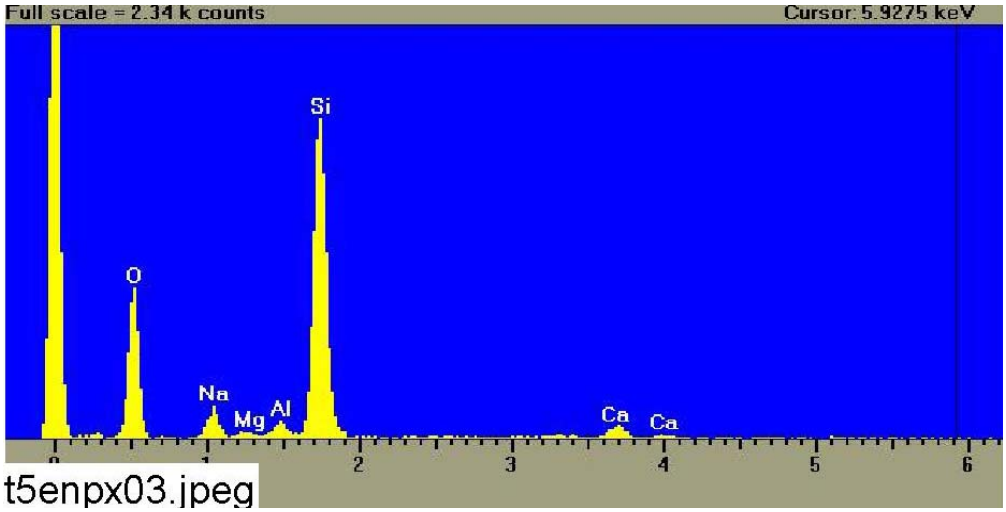

Figure 3-39. EDS counting spectrum for the particulate deposit between the fibers in Figure 3-38.

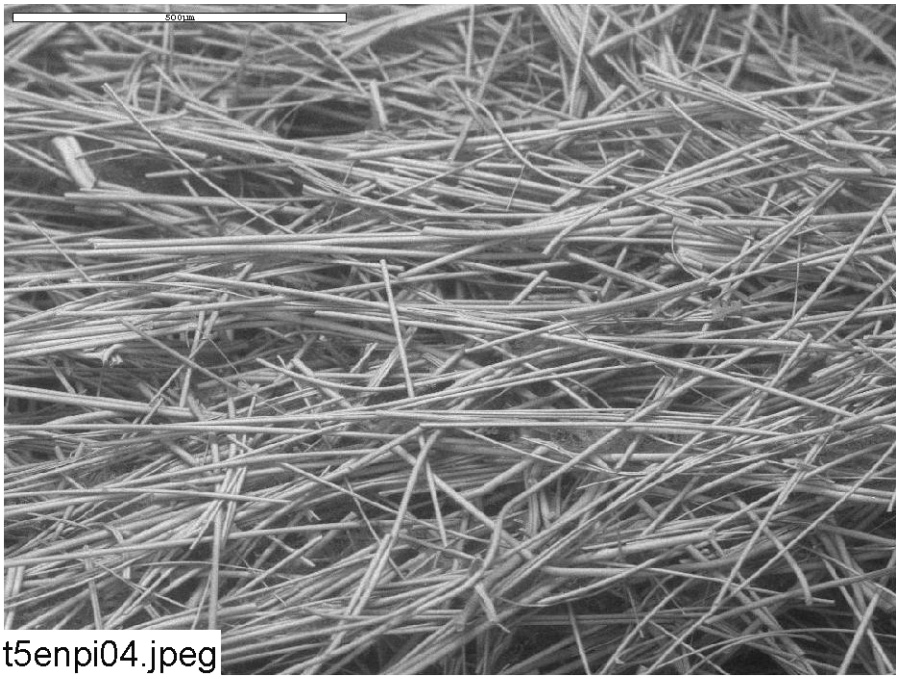

Figure 3-40. Environmental SEM image magnified 100 times for a Test #5 Day 30 low-flow interior fiberglass sample in a big envelope.

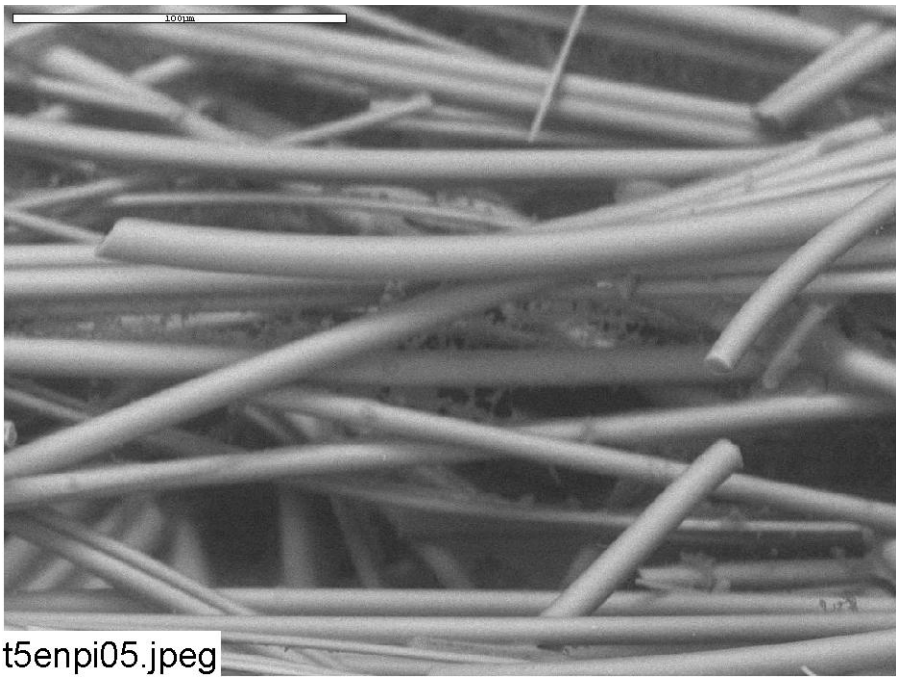

Figure 3-41. Environmental SEM image magnified 500 times for a Test #5 Day 30 low-flow interior fiberglass sample in a big envelope.

3.3.1.6 Day 30 High-Flow Fiberglass Samples

Compared with the Day 30 low-flow fiberglass samples, no significant amount of particulate deposits was found on high-flow exterior samples. In addition, consistent with the findings for other fiberglass interior samples, no particulate deposits were found on the interior. However, similar flocculent deposits were found on both the fiberglass exterior and interior. Figures 3-42 through 3-45 show the Day 30 high-flow fiberglass results.

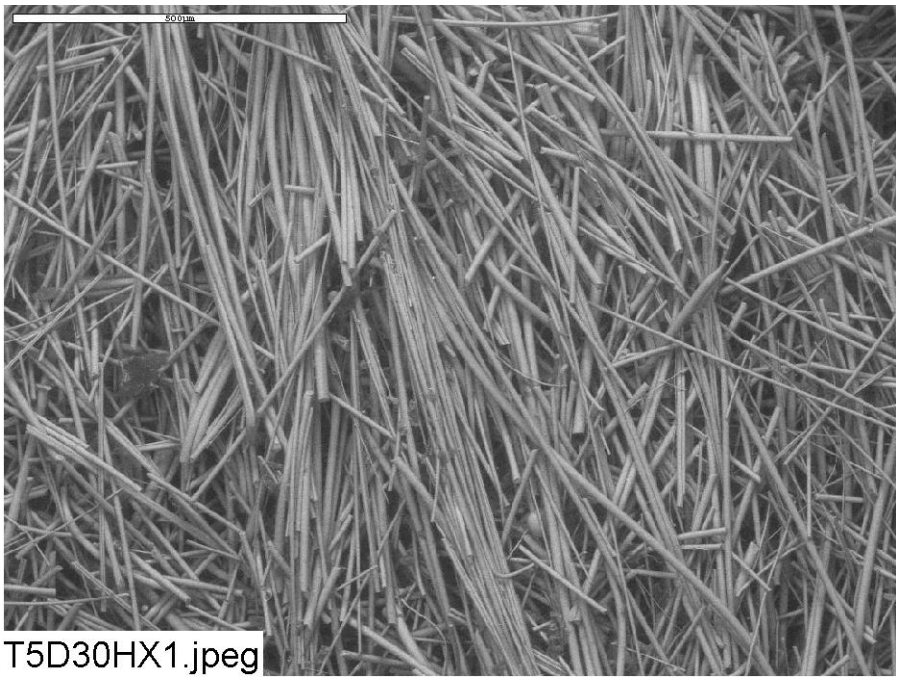

Figure 3-42. Environmental SEM image magnified 100 times for a Test #5 Day 30 high-flow exterior fiberglass sample.

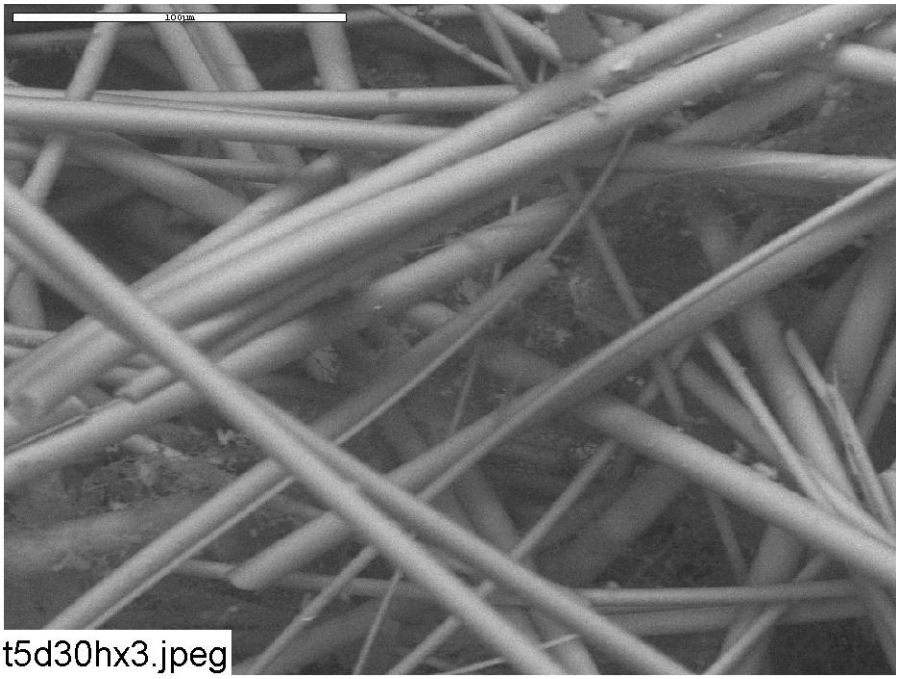

Figure 3-43. Environmental SEM image magnified 500 times for a Test #5 Day 30 high-flow exterior fiberglass sample.

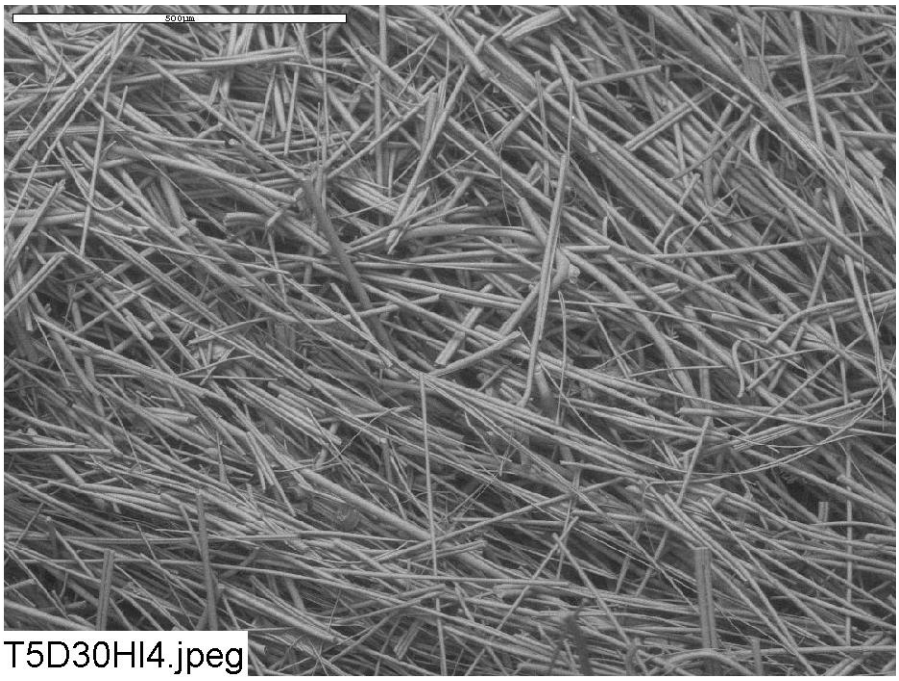

Figure 3-44. Environmental SEM image magnified 100 times for a Test #5 Day 30 high-flow interior fiberglass sample.

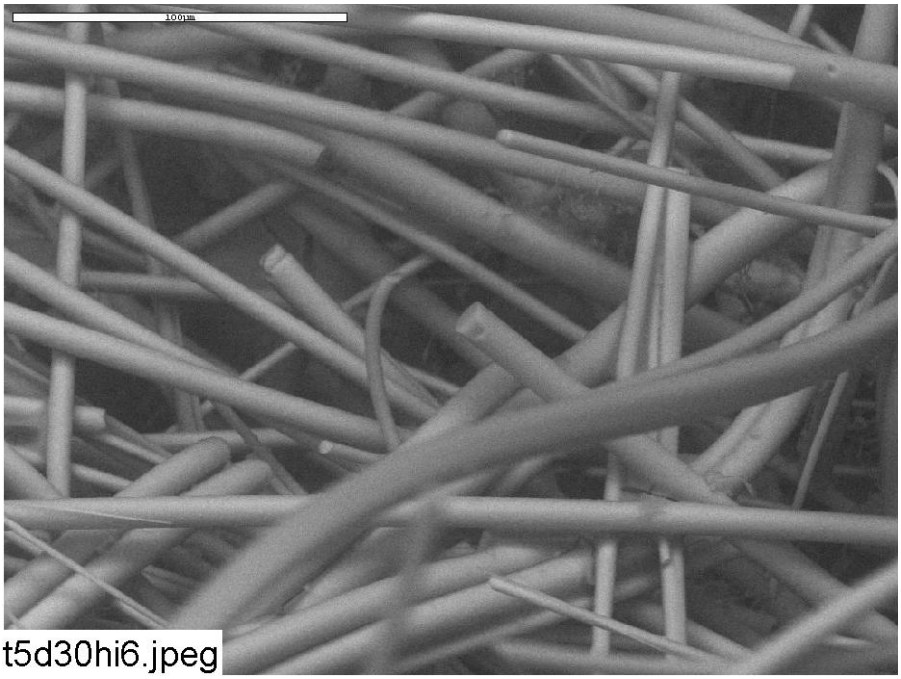

Figure 3-45. Environmental SEM image magnified 500 times for a Test #5 Day 30 high-flow interior fiberglass sample.

3.3.1.7 Day 30 High-Flow Fiberglass Samples in Front of a Header

The fiberglass sample in front of a header was different from the conventional high-flow fiberglass samples discussed in Subsections 3.3.1.3 and 3.3.1.6; the header sample was put in the tank on Day 6. By placing the sample in the tank on Day 6, turbidity and TSS would be much less than on Day 0, and deposits on the sample would be from the tank solution over the last 3 weeks of the test. Due to the settling of suspended particles and the decrease in turbidity during the first several days of the test, no significant particulate deposits were found on the header fiberglass exterior, as shown by ESEM images. However, flocculent deposits were found on both the exterior and the interior of the header samples. This result suggests again that the flocculent deposits were likely caused by chemical precipitation and may have formed when the samples were partially dehydrated. Figures 3-46 through 3-49 show the results from the Day 30 high-flow fiberglass in front of a header.

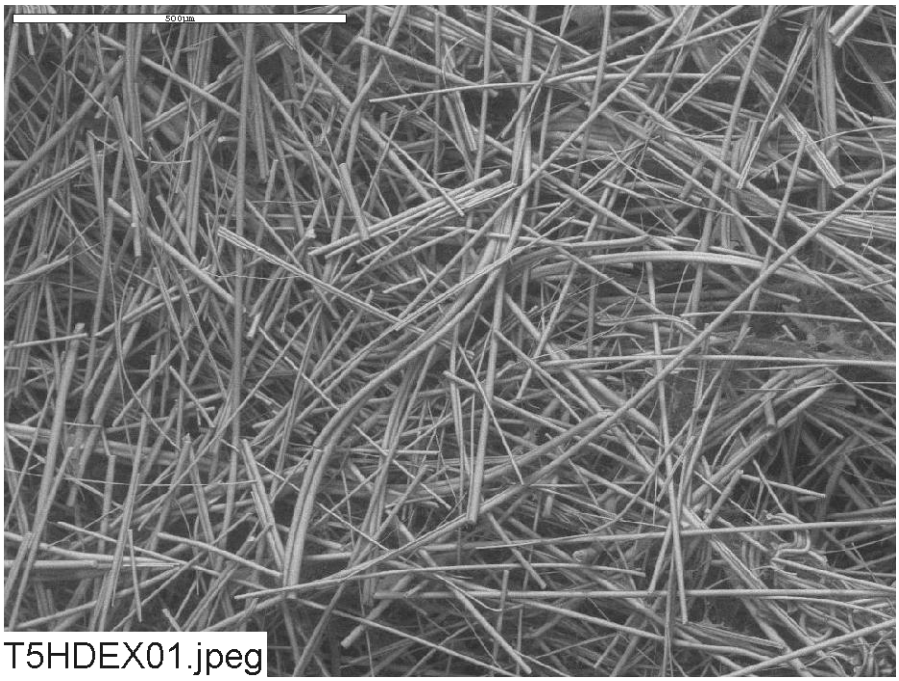

Figure 3-46. Environmental SEM image magnified 100 times for a Test #5 Day 30 high-flow exterior fiberglass sample in front of the header.

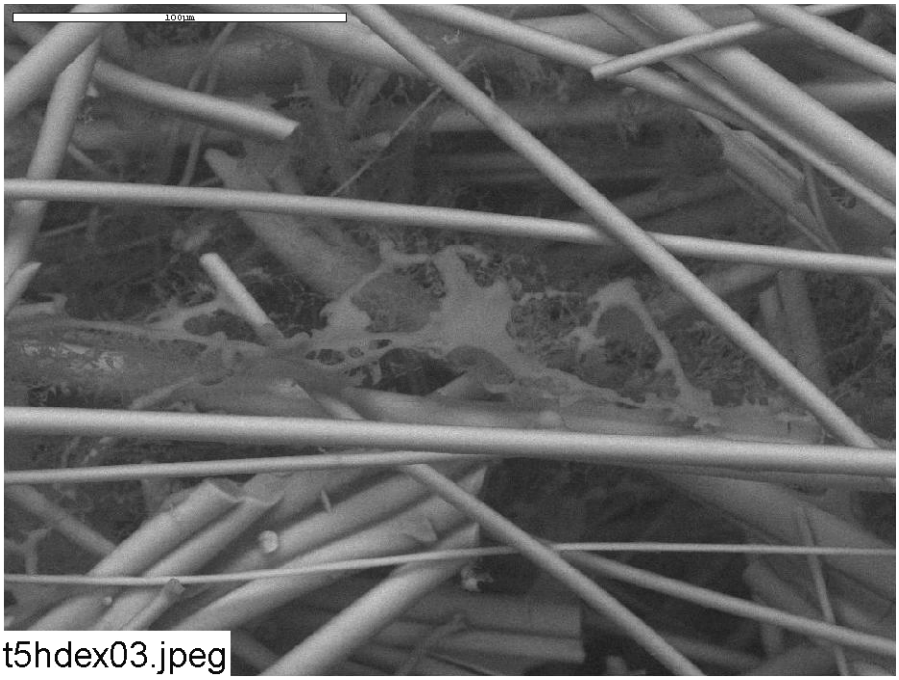

Figure 3-47. Environmental SEM image magnified 500 times for a Test #5 Day 30 high-flow exterior fiberglass sample in front of the header.

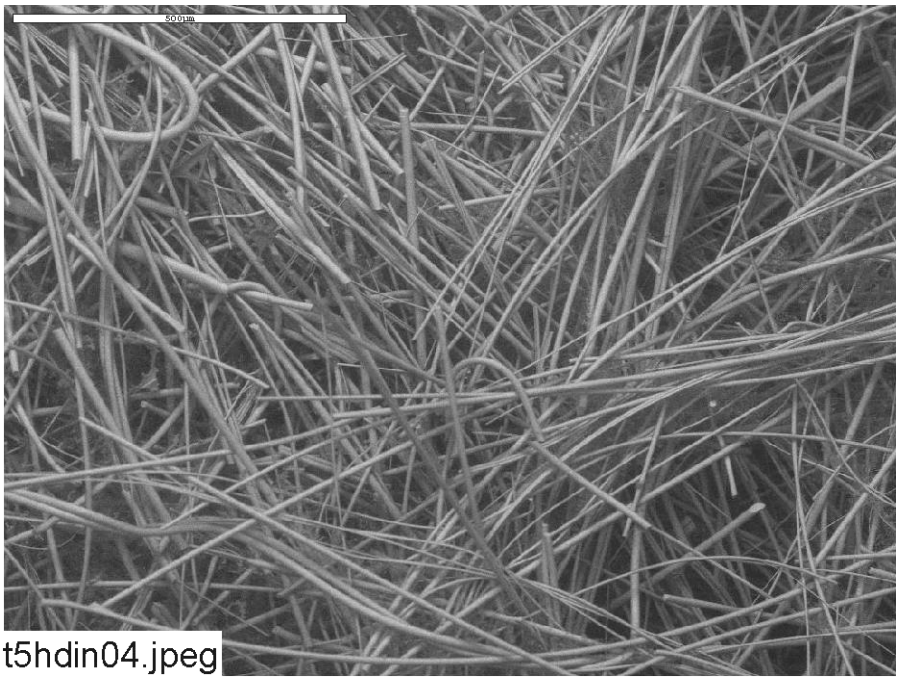

Figure 3-48. Environmental SEM image magnified 100 times for a Test #5 Day 30 high-flow interior fiberglass sample in front of the header.

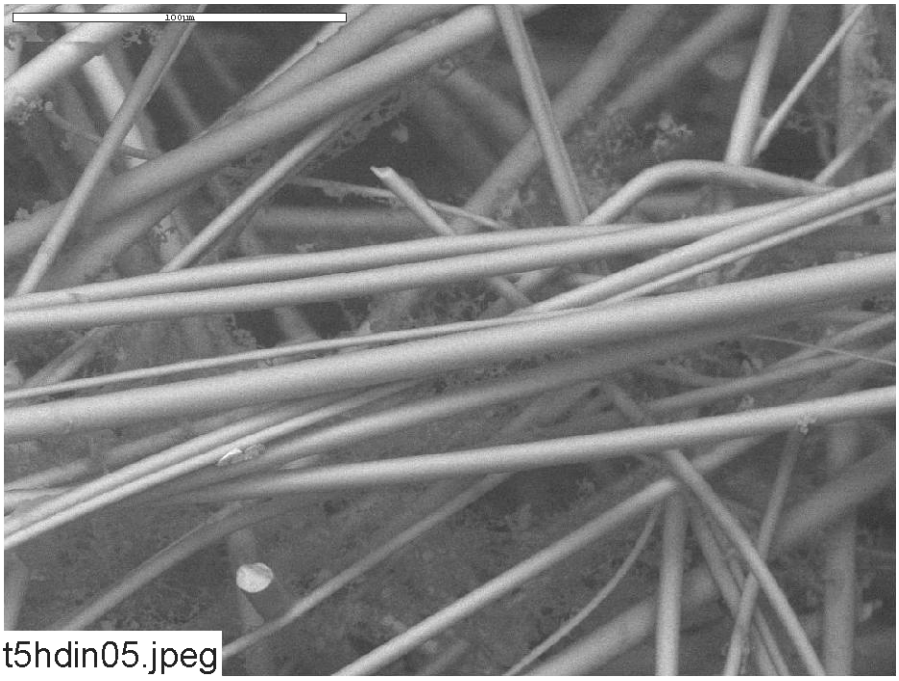

Figure 3-49. Environmental SEM image magnified 500 times for a Test #5 Day 30 high-flow interior fiberglass sample in front of the header.

3.3.1.8 Day 30 Low-Flow Fiberglass Samples in Nylon Mesh

A 5-g fiberglass sample was enclosed in nylon mesh and submerged in a low-flow zone of the tank on Day 6. This sample provided a comparison with all other fiberglass samples, which were enclosed in stainless steel mesh. The purpose of using a nylon mesh was to see if the mesh material (i.e., stainless steel or nylon) affects the deposits on the fiberglass samples. Comparing the sample in nylon mesh with Day 30 low-flow fiberglass samples contained in stainless steel mesh revealed no significant difference. Flocculent deposits were still the dominant deposit on both the exterior and interior samples. No particulate deposits were found on fiberglass. This result suggests that the mesh material did not significantly affect the deposits on fiberglass. Even though the nylon mesh sample was put in the tank on Day 6, no significant difference was observed between these samples and the low-flow fiberglass samples in the stainless steel mesh put into the tank on Day 0. This result is likely due to the low turbidity and low debris concentration in the test solution. Figures 3-50 through 3-54 show the Day 30 low-flow fiberglass in nylon mesh results.

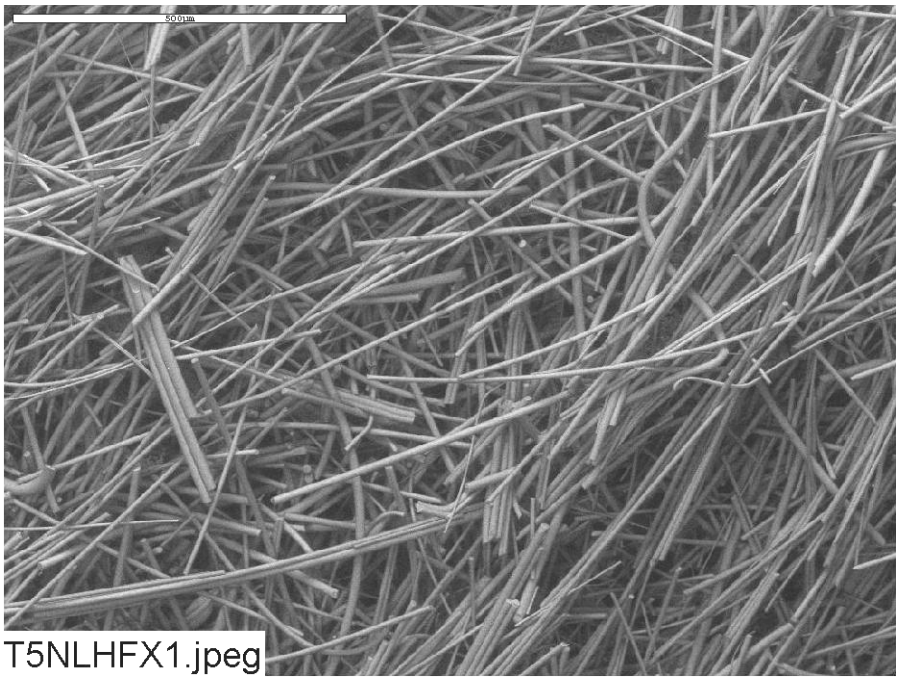

Figure 3-50: Environmental SEM image magnified 100 times for a Test #5 Day 30 low-flow exterior fiberglass sample in a nylon mesh.

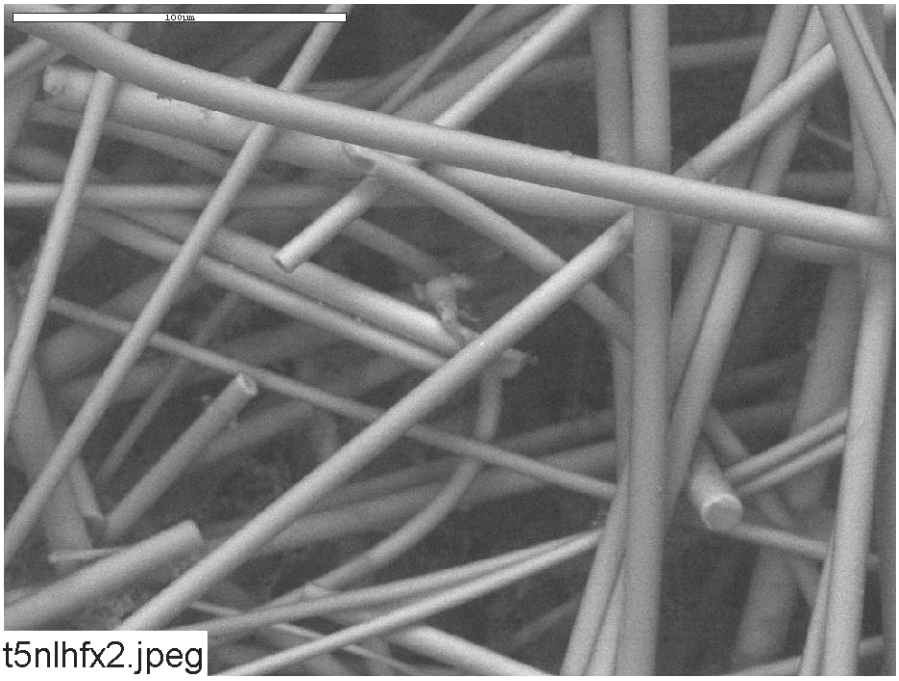

Figure 3-51: Environmental SEM image magnified 500 times for a Test #5 Day 30 low-flow exterior fiberglass sample in a nylon mesh.

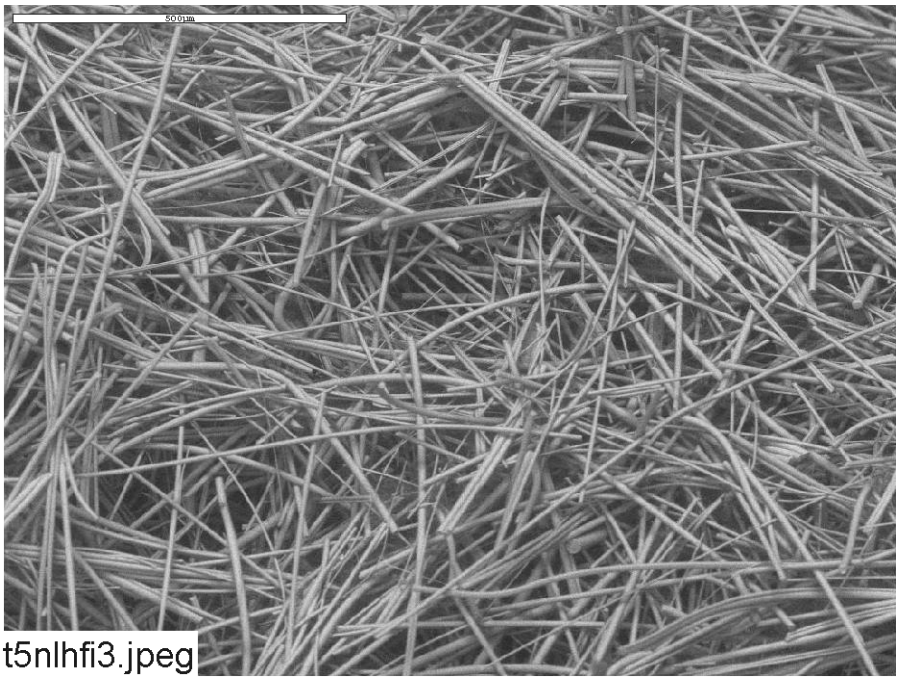

Figure 3-52: Environmental SEM image magnified 100 times for a Test #5 Day 30 low-flow interior fiberglass sample in a nylon mesh.

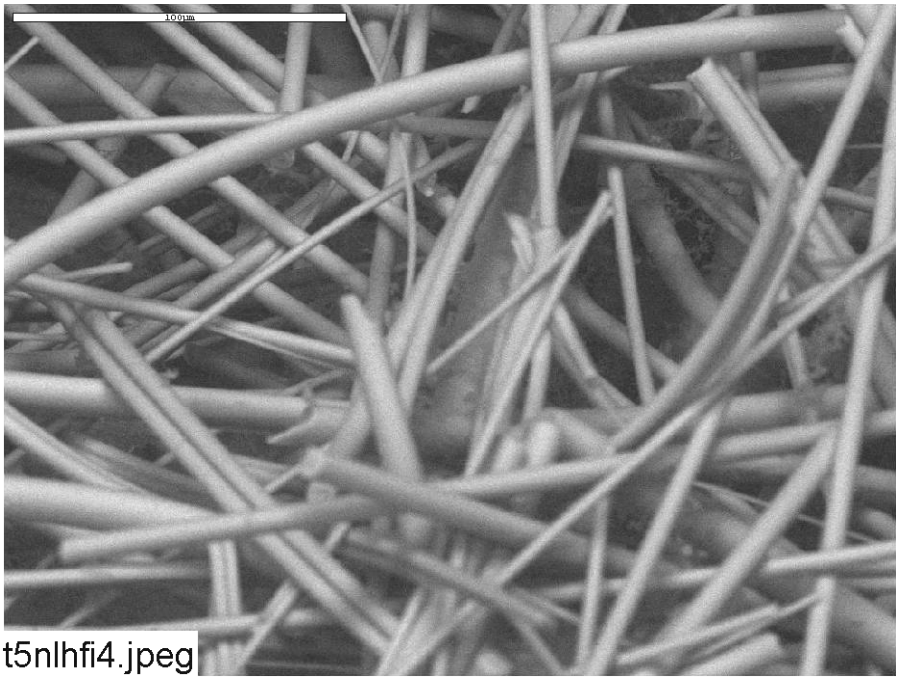

Figure 3-53: Environmental SEM image magnified 500 times for a Test #5 Day 30 low-flow interior fiberglass sample in a nylon mesh.

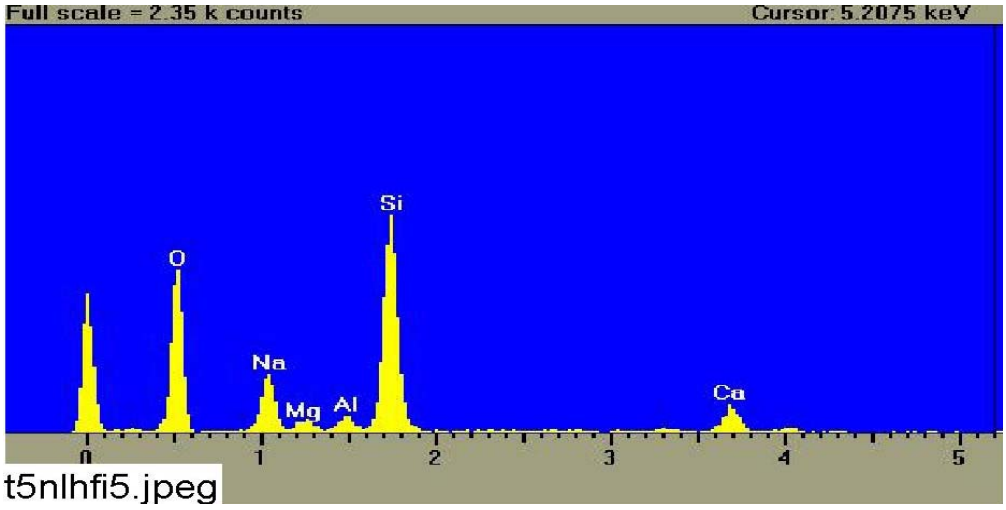

Figure 3-54: EDS counting spectrum for the deposits between the fibers shown in Figure 3-53.

3.3.1.9 Day 30 Drain Collar Fiberglass Samples

Figure 3-94 shows the drain collar after it was removed from the tank. Both the exterior fiberglass sample that was farthest from the drain screen and the exterior sample that was next to the drain screen have significant amounts of particulate deposits. The amount of deposits on the drain collar exterior was much greater than on the high- and low-flow fiberglass samples. However, the exterior farthest from the drain screen had the most

particulate deposits. ESEM results show that the development of a continuous coating on the drain collar exterior farthest from the drain screen, which includes particulate deposits that were likely physically retained or attached. EDS results indicate that the particulate deposits were composed mainly of O, Al, Na, Ca, Mg, C, and possibly Si, for both the drain collar exterior farthest from the drain screen and the exterior next to the drain screen. In addition to the particulate deposits, deposits rich in carbon were also found on the exterior farthest from the drain screen (see Figure 3-57). In contrast to the exterior, no significant particulate deposits were found in the drain collar interior sample, and only flocculent deposits were found (see image Figure 3-64). The drain collar interior is as clean as other high- or low-flow fiberglass interior samples. This result suggests that almost all of the particulate deposits were physically retained at the fiberglass exterior. Figures 3-55 through 3-64 show the drain collar fiberglass results.

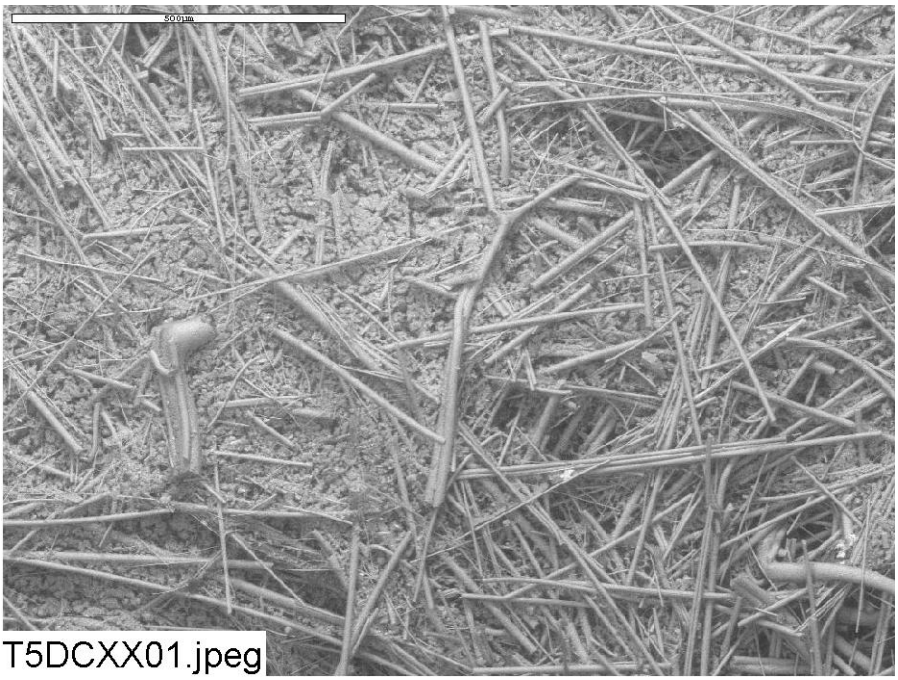

Figure 3-55: Environmental SEM image magnified 100 times for a Test #5 Day 30 exterior drain collar fiberglass sample farthest from the drain screen.

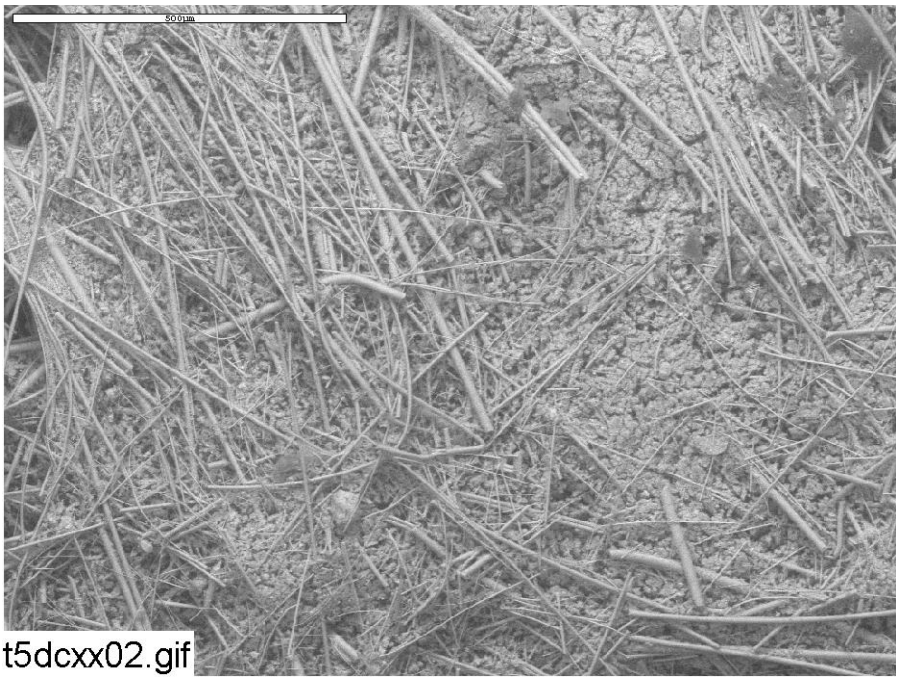

Figure 3-56: Environmental SEM image magnified 100 times for a Test #5 Day 30 exterior drain collar fiberglass sample farthest from the drain screen.

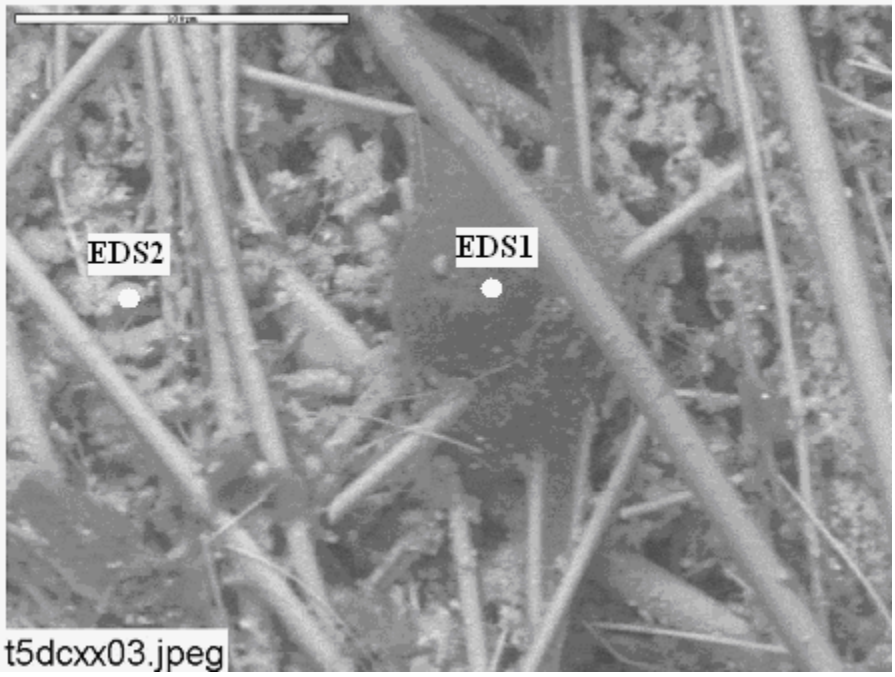

Figure 3-57: Annotated environmental SEM image magnified 500 times for a Test #5 Day 30 exterior drain collar fiberglass sample farthest from the drain screen.

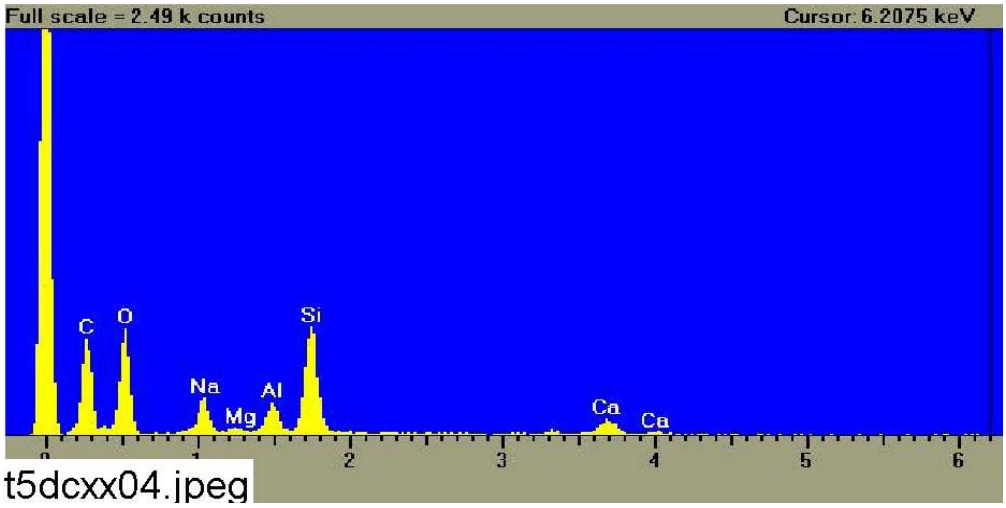

Figure 3-58: EDS counting spectrum for the large mass of particulate deposits (EDS1) on fiberglass shown in Figure 3-57.

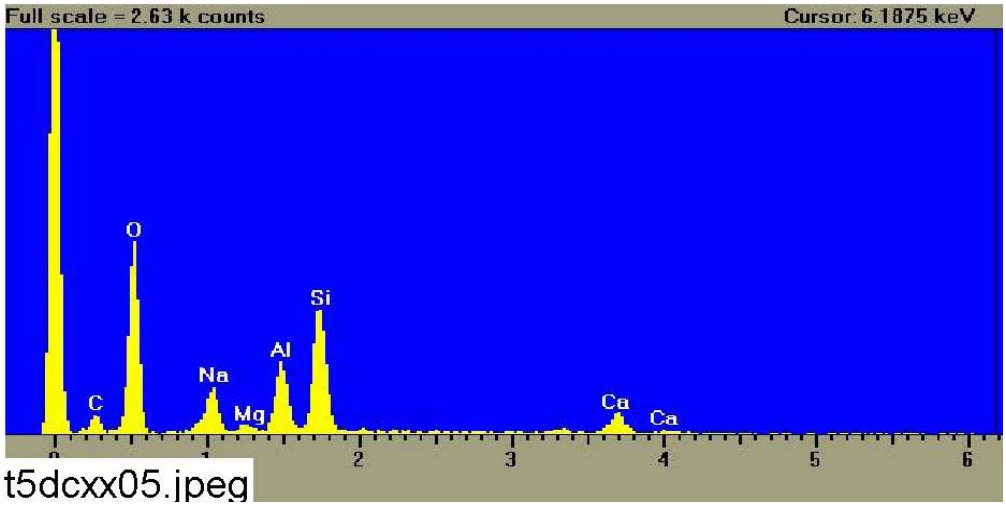

Figure 3-59: EDS counting spectrum for the small particulate deposits (EDS2) between fibers shown in Figure 3-57.

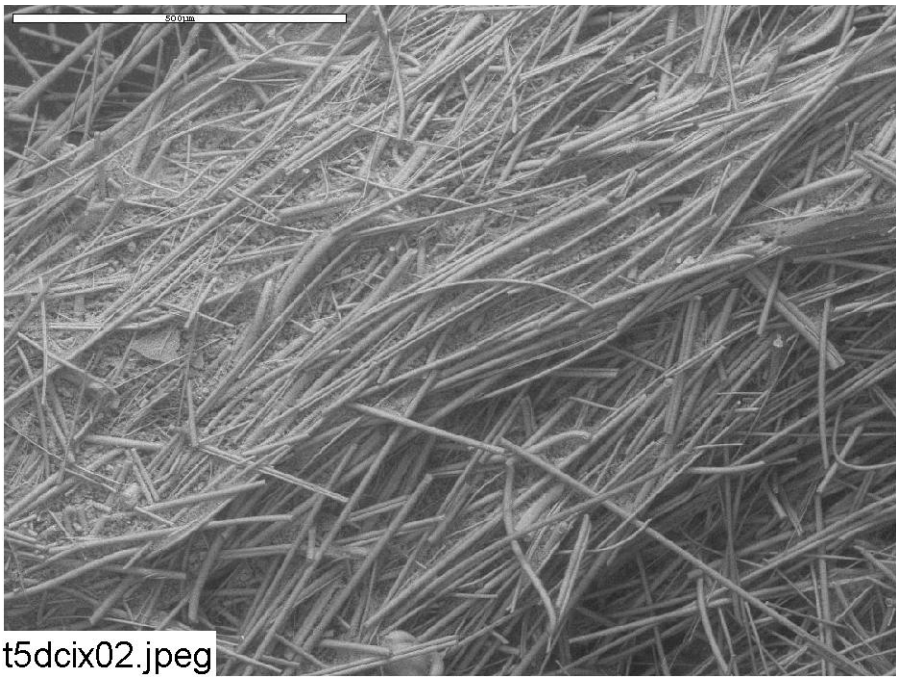

Figure 3-60: Environmental SEM image magnified 100 times for a Test #5 Day 30 exterior drain collar fiberglass sample next to the drain screen.

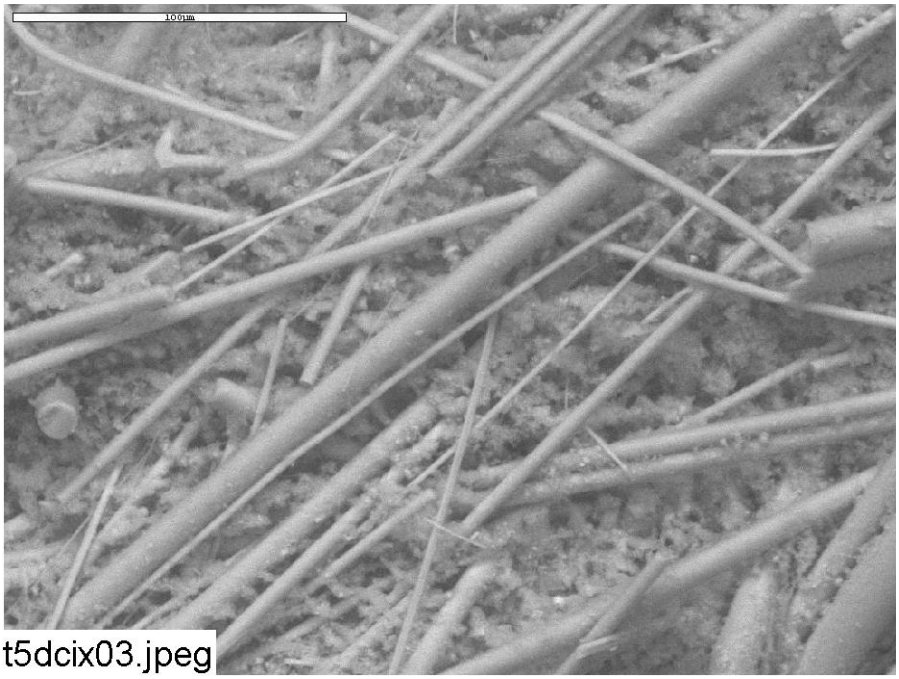

Figure 3-61: Environmental SEM image magnified 500 times for a Test #5 Day 30 exterior drain collar fiberglass sample next to the drain screen.

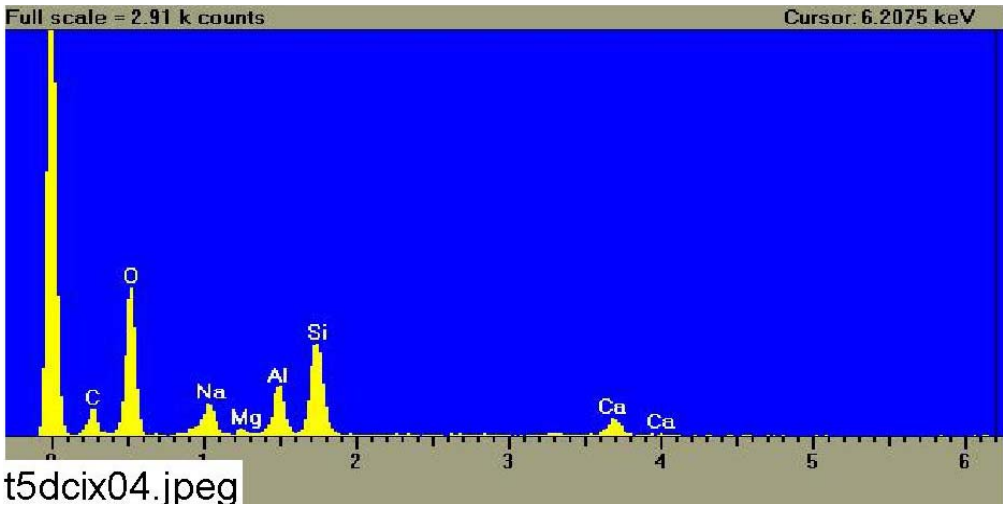

Figure 3-62: EDS counting spectrum for the particulate deposits between fibers in Figure 3-61.

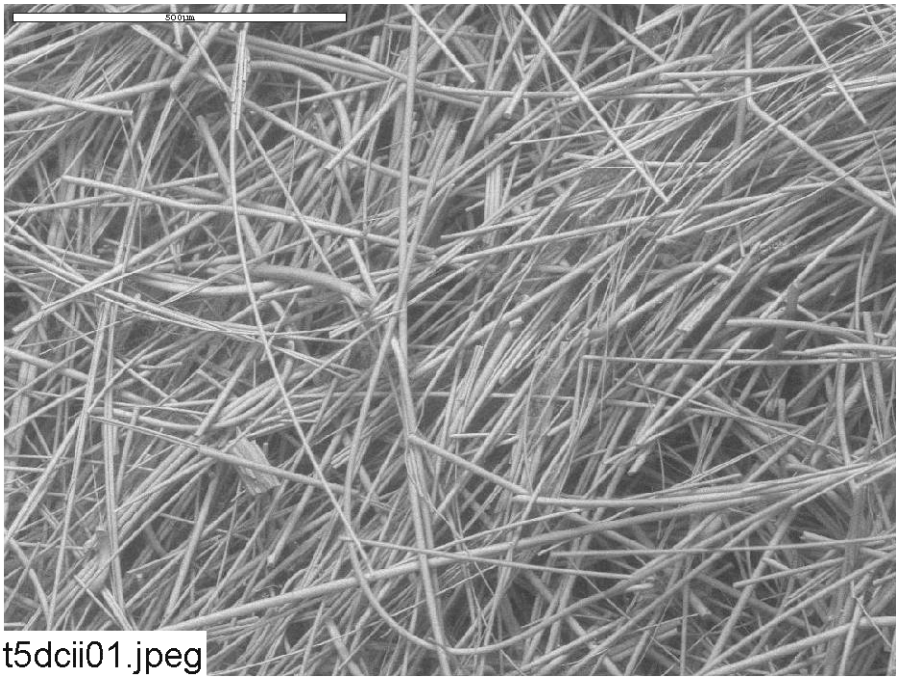

Figure 3-63: Environmental SEM image magnified 100 times for a Test #5 Day 30 interior drain collar fiberglass sample.

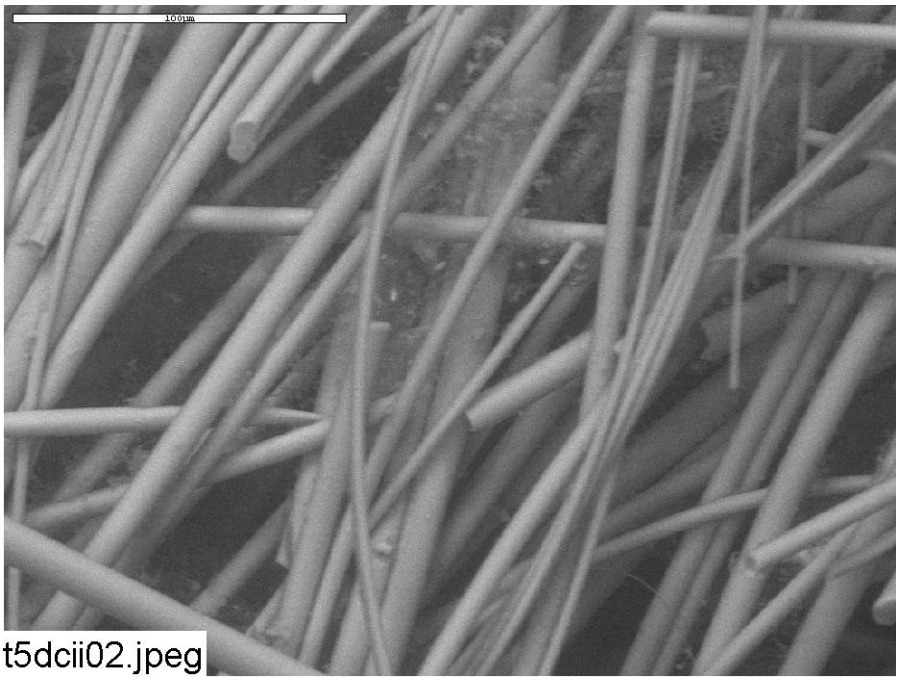

Figure 3-64: Environmental SEM image magnified 500 times for a Test #5 Day 30 interior drain collar fiberglass sample.

3.3.1.10 Day 30 Fiberglass Sample within the Birdcage

For the Day 30 fiberglass sample within the birdcage, the SEM images indicate that a small amount of particulate deposit (see Figure 3-65) was on the exterior of the fiberglass. The amount of particulate deposits was slightly greater than on high- and low-flow fiberglass samples but much less than on the drain collar exterior. The EDS result shows that the particulate deposits were composed of O, Na, Ca, Zn, Al, Mg, and possibly Si. The presence of Zn is inconsistent with the drain collar exterior. Compared with other fiberglass interior samples, the interior birdcage sample was relatively clean. Only flocculent deposits were found. These flocculent deposits were similar to those observed on the high- and low-flow fiberglass samples, which was likely caused by chemical precipitation during the drying process. Again, this result suggests that almost all of the particulate deposits were physically retained at the fiberglass exterior, consistent with findings for the Day 30 high-flow and drain collar fiberglass samples. Figures 3-65 through 3-70 show the birdcage fiberglass results.

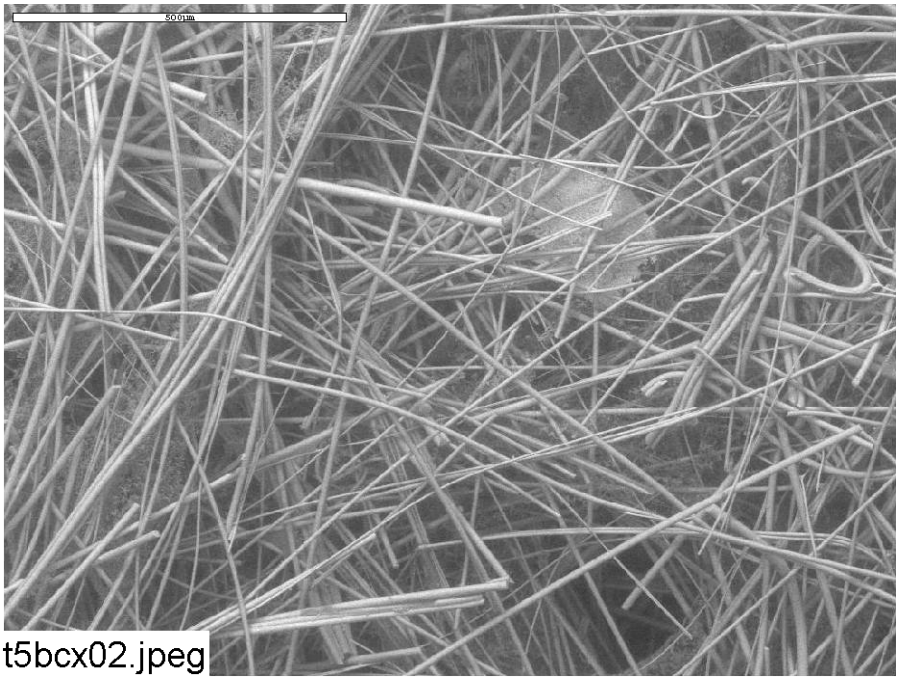

Figure 3-65: Environmental SEM image magnified 100 times for a Test #5 Day 30 exterior fiberglass sample in the bird cage.

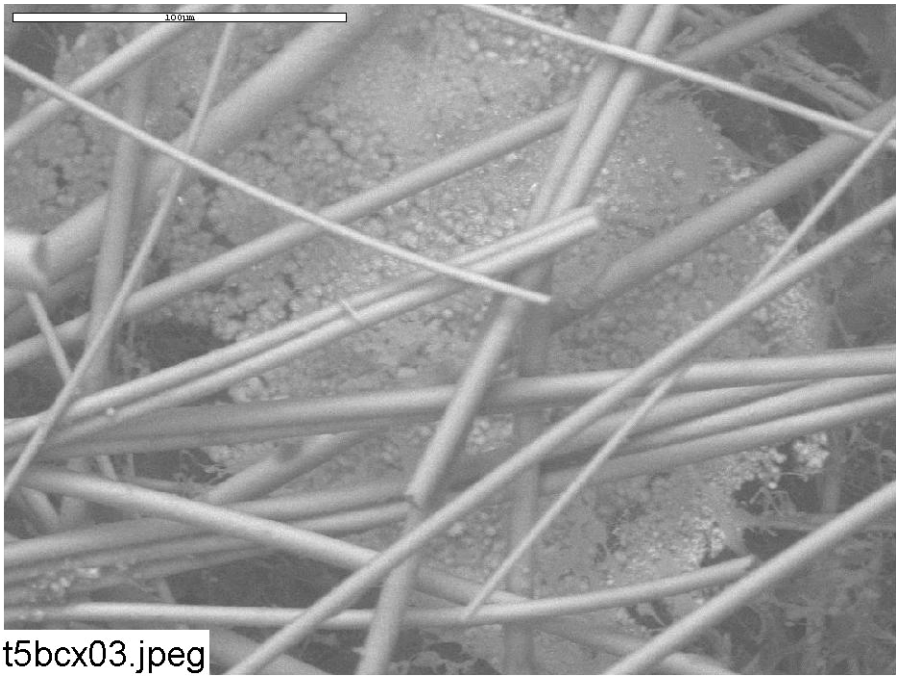

Figure 3-66: Environmental SEM image magnified 500 times for a Test #5 Day 30 exterior fiberglass sample in the birdcage.

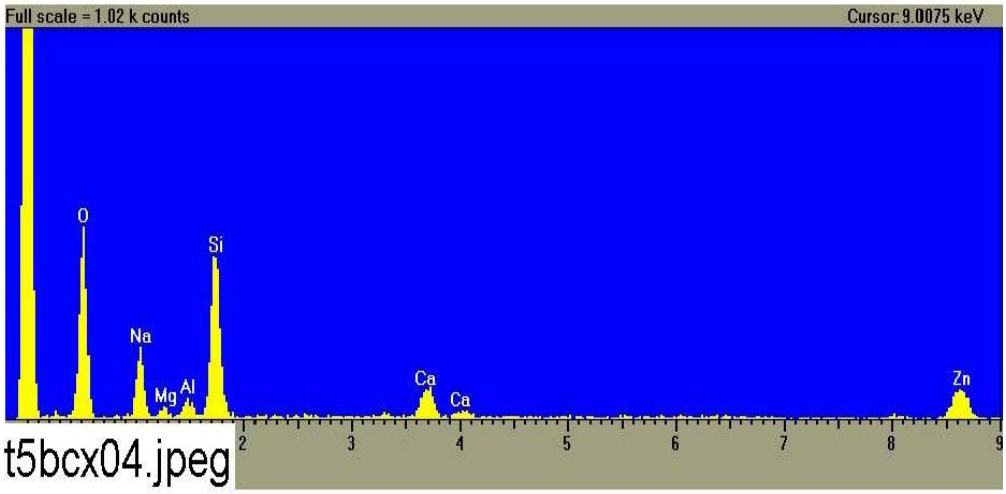

Figure 3-67: EDS counting spectrum for the deposits between fibers shown in Figure 3-66.

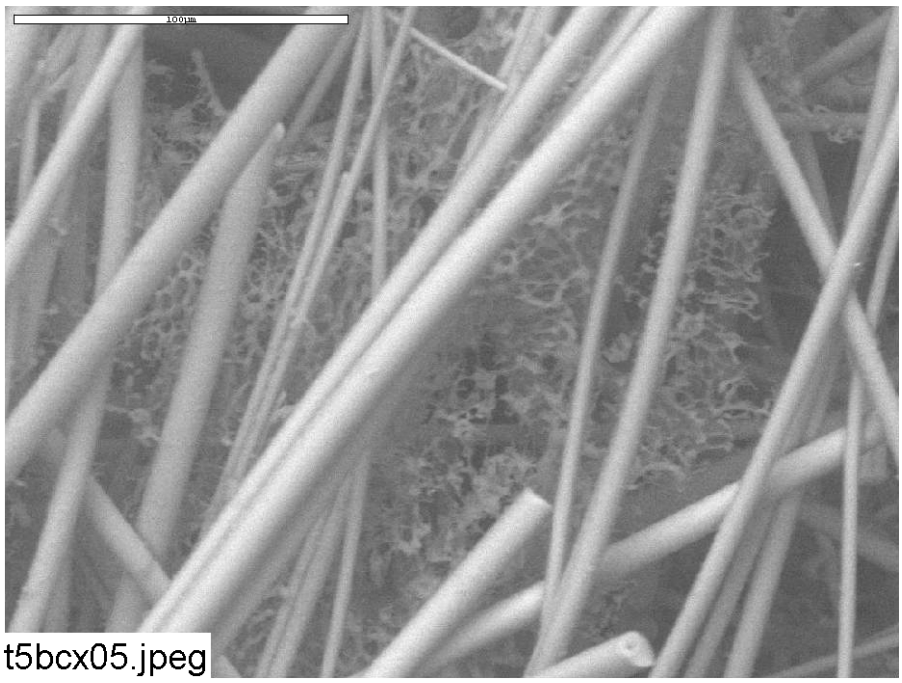

Figure 3-68: Environmental SEM image magnified 500 times for a Test #5 Day 30 exterior fiberglass sample in the birdcage.

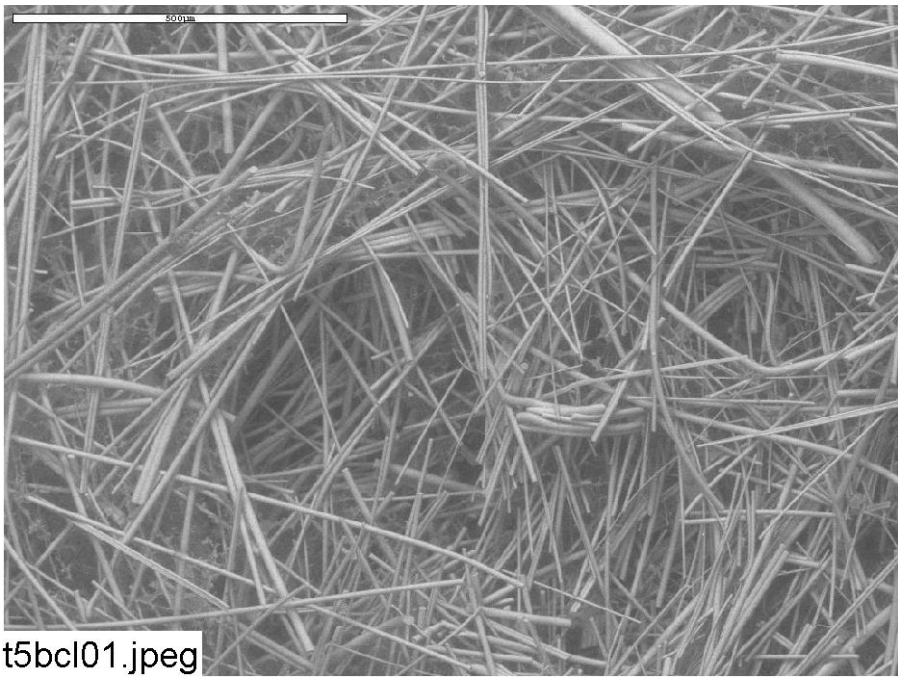

Figure 3-69: Environmental SEM image magnified 100 times for a Test #5 Day 30 interior fiberglass sample in the birdcage.

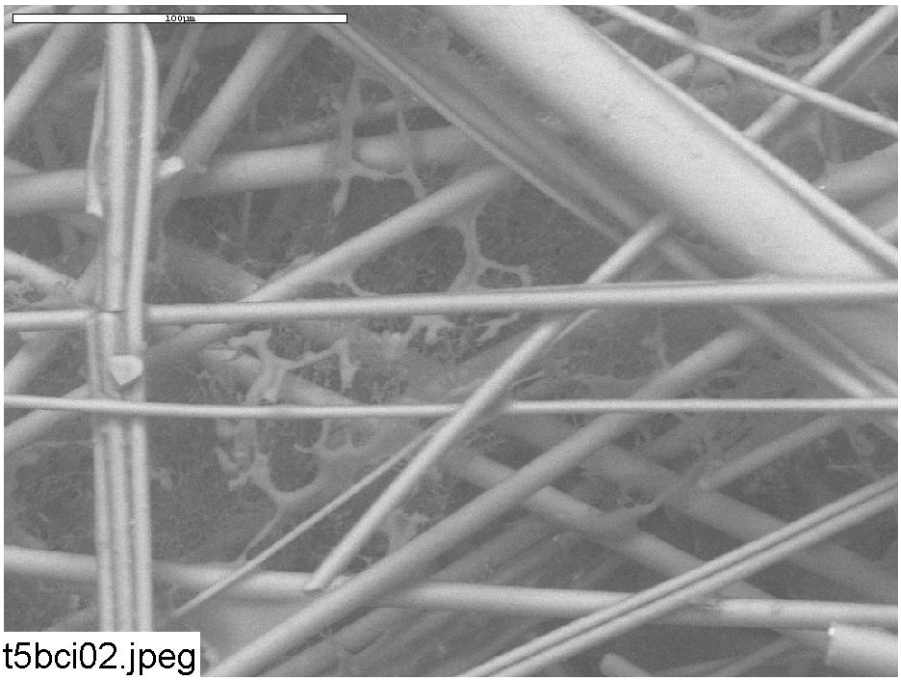

Figure 3-70: Environmental SEM image magnified 500 times for a Test #5 Day 30 interior fiberglass sample in the birdcage.

3.4 Metallic and Concrete Coupons

3.4.1 Weights and Visual Descriptions

3.4.1.1 Submerged Coupons

Examination of the 40 submerged coupons provides insights into the nature of the chemical kinetics that occurred during this 30-day test. The physical change that these coupons experienced is determined through both visual evidence and weight measurement of each coupon before and after the test. Pre-test pictures were taken of the coupons when they were received and before they were inserted into the racks. Post-test pictures were taken several days after the racks had been removed from the tank. All racks with coupons still inserted were staged to allow the coupons to dry completely before the post-test pictures were taken. The coupons were placed in a low-humidity room and allowed to air dry. All coupons were also weighed before they were inserted into the tank and after the 30-day test was completed.

There are three submerged aluminum coupons in each test. Figures 3-71 through 3-73 are the pre- and post-test pictures of the Test #5 coupons. The aluminum coupons A1-240, A1-241, and A1-242 (see Figures 3-71 through 3.73) were located from east to west, respectively, in the tank. The submerged aluminum coupons turned brown, and they developed a brown, powdery film on the surface. The film resulted in the surface of the coupon becoming rough to the touch.

Figure 3-71: Al-240 submerged, pre-test (left) and post-test (right)

Figure 3-72: Al-241 submerged, pre-test (left) and post-test (right)

Figure 3-73: Al-242 submerged, pre-test (left) and post-test (right)

Figures 3-74 through 3-76 present the pre- and post-test pictures of three submerged galvanized steel coupons. The galvanized steel coupons developed a white precipitate on the surface of the coupons, which caused the surfaces to have a coarse feel. The surfaces of the coupons had horizontal lines composed of this same white precipitate. The horizontal deposits may have been left during the slow draining of the tank solution.

Figure 3-74: GS-137 submerged, pre-test (left) and post-test (right).

Figure 3-75: GS-140 submerged, pre-test (left) and post-test (right).

Figure 3-76: GS-141 submerged, pre-test (left) and post-test (right).

Figures 3-77 and 3-78 present the pre- and post-test pictures of two submerged inorganic zinc (IOZ) coated steel coupons. Both submerged IOZ coated steel coupons have similar light particulate deposits. These coupons were covered with a brown coating over their entire surfaces.

Figure 3-77: IOZ-310 submerged, pre-test (left) and post-test (right).

Figure 3-78: IOZ-312 submerged, pre-test (left) and post-test (right).

Figures 3-79 and 3-80 present the pre- and post-test pictures of two submerged copper coupons. The submerged copper coupons developed very light horizontal white deposits, which may be due to the slow tank draining process.

Figure 3-79: CU-512 submerged, pre-test (left) and post-test (right).

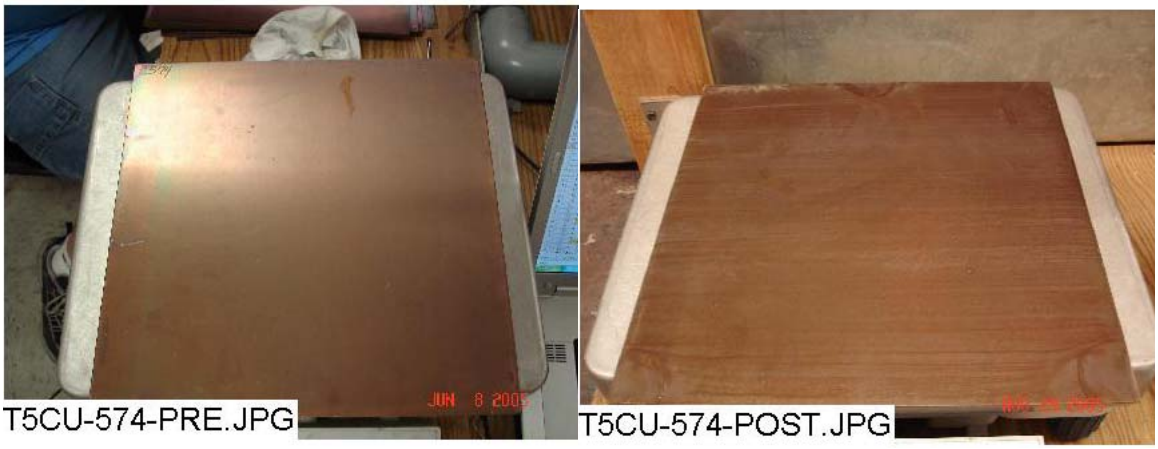

Figure 3-80: CU-574 submerged, pre-test (left) and post-test (right).

Figure 3-81 presents the pre- and post-test pictures of the submerged carbon steel coupon. The surface of the coupon was roughened by the deposition of white precipitate. There were also areas of rust on the coupon.

Figure 3-81: US-14 submerged, pre-test (left) and post-test (right).

Figure 3-82 presents the pre- and post-test pictures of the submerged concrete coupon. The post-test concrete coupon exhibits a brownish color.

Figure 3-82: Conc-003 submerged, pre-test (left) and post-test (right).

Table 3-2 presents the pre- and post-test weight data for each representative submerged coupon.

Table 3-2: Weight Data for Submerged Coupons

Type	Coupon No.	Pre-Test Wt. (g)	Post-Test Wt. (g)	Net Gain/Loss
Al	240	391.7	378.6	-13.1
Al	241	391.7	379.6	-12.1
Al	242	392.1	383.6	-8.5
GS	137	1043.2	1043.1	-0.1
GS	140	1067.5	1067.7	0.2
GS	141	1069.9	1070.0	0.1
IOZ	310	1652.9	1654.5	1.6
IOZ	312	1610.3	1611.6	1.3
CU	512	1322.6	1322.2	-0.4
CU	574	1306.0	1305.4	-0.6
US	14	1022.2	1022.2	0.0
Conc	003	7627.8	7853.7	225.9

The aluminum coupons' average weight differential was -11.2 g. The galvanized steel coupons' average weight gain was less than 0.1 g, and the coated steel coupons gained an average of 1.5 g. The representative copper coupons lost an average of 0.5 g, and the carbon steel coupon had no weight change. The concrete coupon gained 225.9 g, a gain of 3% of its original weight, which was likely due to post-test water retention.

3.4.1.2 Unsubmerged Coupons

Figures 3-83 and 3-84 show the pre- and post-test pictures of two unsubmerged aluminum coupons. Each unsubmerged aluminum coupon accumulated a white particle deposition along with some brownish areas. Also, the texture of each coupon is coarser, and the surface quality of each coupon is less lustrous than before the test. The reddish-brown color that was observed on the submerged coupons is absent on the unsubmerged coupons. The Al-247 coupon was loaded in Rack 2, which was located in the southern position of the middle tier of the tank. The Al-294 coupon was loaded in Rack 7, which was located in the northern position of the top tier of the tank.

Figure 3-83: Al-247 unsubmerged, pre-test (left) and post-test (right).

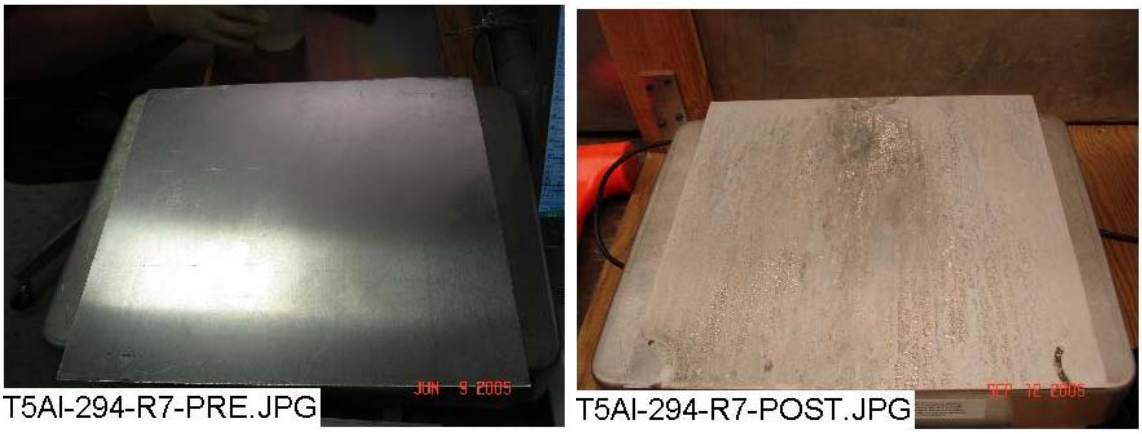

Figure 3-84: Al-294 unsubmerged, pre-test (left) and post-test (right).

Figures 3-85 and 3-86 show the pre- and post-test pictures of two unsubmerged galvanized steel coupons. There was some white deposition on the surface of these coupons, although it was in small amounts. This deposition is visibly different and less concentrated than that of the submerged coupons. The GS-167 coupon was loaded in Rack 3, which was located in the center position of the middle tier of the tank. The GS-638 coupon was loaded in Rack 6, which was located in the middle position of the top tier of the tank.

Figure 3-85: GS-167 unsubmerged, pre-test (left) and post-test (right).

Figure 3-86: GS-638 unsubmerged, pre-test (left) and post-test (right).

Figures 3-87 and 3-88 present the pre- and post-test pictures of two unsubmerged copper coupons. All of the copper coupons had vertical water marks that were likely caused by water flowing downward on the coupon surface during the spray portion of the test. The copper coupons did not appear to accumulate any particle deposition. The CU-430 coupon was loaded in Rack 2, which was located in the southern position of the middle tier of the tank. The CU-587 coupon was loaded in Rack 7, which was located in the northern position of the top tier of the tank.

Figure 3-87: CU-430 unsubmerged, pre-test (left) and post-test (right).

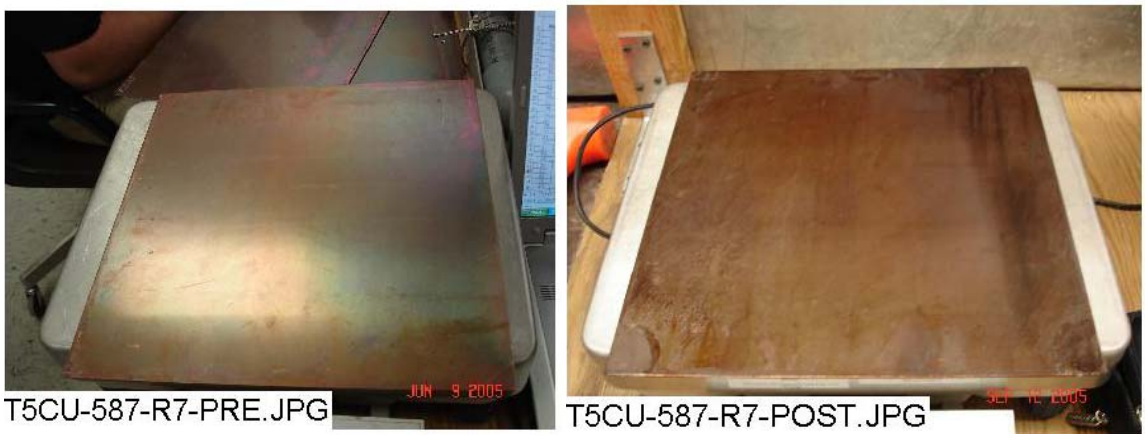

Figure 3-88: CU-587 unsubmerged, pre-test (left) and post-test (right).

Figures 3-89 and 3-90 present the pre- and post-test pictures of two unsubmerged inorganic zinc coated steel coupons. Each post-test coated steel coupon exhibits a similar pattern of deposition, which is less concentrated than that of the submerged coupons. The IOZ-342 coupon was loaded in Rack 4, which was located in the northern position of the middle tier of the tank. The IOZ-356 coupon was loaded in Rack 5, which was located in the southern position of the top tier of the tank.

Figure 3-89: IOZ-342 unsubmerged, pre-test (left) and post-test (right).

Figure 3-90: IOZ-356 unsubmerged, pre-test (left) and post-test (right).

Figure 3-91 presents the pre- and post-test pictures of one unsubmerged carbon steel coupon. The post-test carbon steel coupon exhibits rust along its center line and around its bottom edge. The deposition concentration is greater than that of the submerged carbon steel coupon. The US-18 coupon was loaded in Rack 6, which was located in the center position of the top tier of the tank.

Table 3-3 presents the pre- and post-test weight data for each representative unsubmerged coupon.

Figure 3-91: US-18 unsubmerged, pre-test (left) and post-test (right).

Table 3-3: Weight Data for Unsubmerged Coupons

Type	Coupon No.	Pre-Test Wt. (g)	Post-Test Wt. (g)	Net Gain/Loss
Al	247	390.2	391.6	1.4
Al	294	390.6	391.8	1.2
GS	167	1051.6	1051.5	-0.1
GS	638	1067.5	1067.3	-0.2
IOZ	342	1656.0	1658.3	2.3
IOZ	356	1621.5	1622.5	1.0
CU	430	1319.8	1320.6	0.8
CU	587	1328.0	1328.2	0.2
US	18	1032.1	1032.3	0.2

The aluminum coupons gained an average of 1.3 g, and the galvanized steel coupons lost an average of 0.2 g. The coated steel coupons' average weight gain was 1.6 g, and the copper coupons' average weight gain was 0.5 g. The carbon steel coupon lost 0.2 g.

Table 3-4 displays the mean weight gain/loss summary in grams for all of the submerged coupons. Table 3-5 displays the mean weight gain/loss summary in grams for all of the unsubmerged coupons by rack.

Table 3-4: Mean Gain/Loss Data for All Submerged Coupons (g)

Coupon Type	AL	GS	CU	IOZ	US	Concrete
Mean Gain - Loss (g)	-11.2	0.1	-0.2	1.6	0.0	225.9

Table 3-5: Mean Gain/Loss Data for All Unsubmerged Coupons (g)

	Mean Gain-Loss Per Coupon Type (g)				
Rack No.	**AL**	**GS**	**CU**	**IOZ**	**US**
2	0.8	0.6	0.4	1.4	n/a
3	0.1	<0.1	0.1	0.2	n/a
4	0.4	0.1	<0.1	1.1	0.2
5	0.9	<0.1	<0.1	1.1	n/a
6	0.4	0.1	0.1	1.5	0.2
7	1.2	0.3	0.2	1.8	n/a
Overall	0.4	0.2	0.2	1.2	0.2

3.4.2 SEM Analyses of Coupons

3.4.2.1 Submerged Coupons

During the ICET tests, trace metal cations may be released from the submerged metal coupon surfaces due to corrosion effects. Subsequently, the released metal cations may form complexes in solution through electrostatic interactions with anions such as OH^-, $B_4O_7^{2-}$, $H_2BO_3^-$, SiO_3^{2-}, and CO_3^{2-}. In addition, the complexed anions may attract other cations from the solution, such as Ca^{2+}, Mg^{2+}, Al^{3+}, Cu^{2+}, Zn^{2+}, and H^+. As a result, corrosion products (deposits) are formed and may continuously grow on the metal coupon surfaces. The thickness of the deposits was observed to be in the range of millimeters. The adherence between the metal coupons and the deposits is through chemical bonds, which are much stronger than van der Waals forces. Due to the vertical orientation of the metal coupons in the tank (with a small horizontal cross-sectional area), the deposits on the metal coupon surface are likely of chemical origin, rather than being the result of particulate deposits settling on the surface.

According to the SEM/EDS results, the dominant corrosion products on the submerged Al coupons are likely aluminum hydroxide with other substances containing Si, Ca, O, C, Fe, and Cu. For submerged Cu coupons, the possible corrosion products include CuO, $Cu_2(CO_3)(OH)_2$, and substances containing Cu, Ca, Si, Al, O, and C. For the submerged galvanized steel coupons, the possible corrosion products are oxides, hydroxides, silicate, and carbonate compounds of Zn, Ca, and Al. For the submerged steel coupon, the possible corrosion products include oxide, hydroxide, silicate, and carbonate compounds of Fe and Ca and compounds composed of Fe, Si, Ca, O, and Al.

3.4.2.2 Unsubmerged Coupons

The physical and chemical changes that the unsubmerged coupons experienced during Test #5 are less significant than the changes experienced by the submerged coupons. The unsubmerged coupons were affected by the test solution only during the initial 4-hour spray phase. They were also exposed to moisture vapor throughout the test.

According to SEM/EDS results, the dominant corrosion products on the unsubmerged Al coupons are likely aluminum hydroxide and/or aluminum oxide. For unsubmerged Cu, the corrosion products on the coupon surface are likely CuO; $Cu_2(CO_3)(OH)_2$; and the corrosion products containing Al, Si, O, Ca, Cu, Na, Fe, and C. The corrosion products on the unsubmerged galvanized steel coupon surface are composed of C, O, and Zn. They are likely ZnO and $ZnCO_3$. For the unsubmerged steel coupon, the likely corrosion products are Fe_2O_3, $Fe(OH)_3$, $Fe_2(CO_3)_3$, and compounds containing small amounts of Si.

The complete listing of Day 30 coupon analysis is shown in Appendix E.

3.5 Sedimentation

Sediment was collected from the tank bottom after the test solution was drained. The entire amount recovered was 89 g, wet, as shown in Figure 3-92. Figure 3-93 shows the tank bottom with the sediment, after the test solution was drained and the samples were removed. Figure 3-94 shows the top of the drain screen with the drain collar still attached.

Figures 3-95 through 3-99 and Table 3-6 provide SEM/EDS and XRD/XRF analysis results. The SEM/EDS and XRD/XRF analyses provided information on the morphology and composition of Test #5 sediment. SEM images show that the sediments were mainly composed of fiberglass debris, which mixed with some particulate deposits. Consistently, the XRF result shows that SiO_2 was about 64% of the total mass of the dried sediments. In addition, the XRD result indicates the presence of quartz crystal in the sediments. The quartz is likely from a crystalline form of fiberglass debris. The particulate deposits in the sediments possibly originated from the corrosion products, chemical precipitates, concrete debris, and dust.

It should be noted that the XRD result also shows the possible crystalline match of cobalt and uranium compounds in the sediments. However, cobalt and uranium were unlikely present in the testing material. Their match patterns are likely due to the variety of species contained within the sediment. As a result, these compounds may be excluded from the sediment compositions.

The complete Day 30 sediment analyses are given in Appendix F.

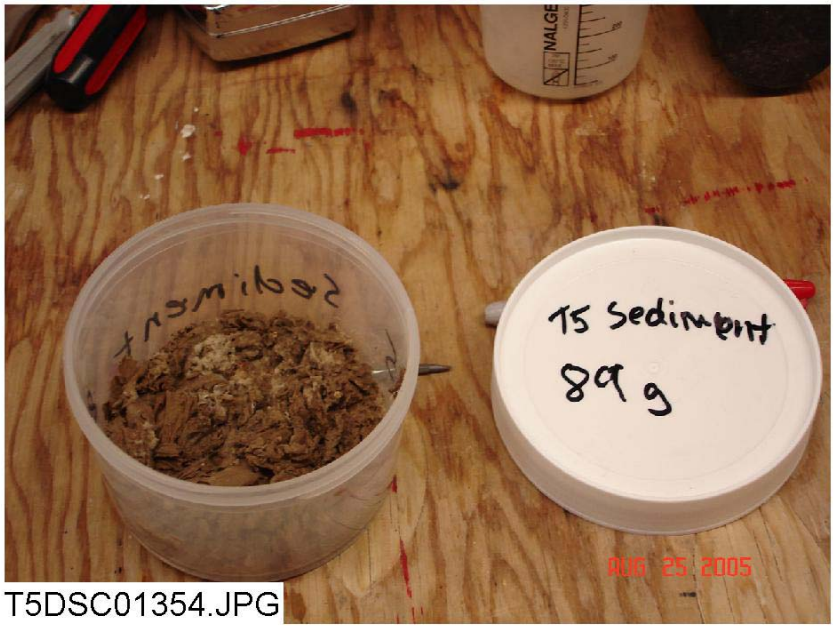

Figure 3-92. Sediment removed from the tank.

Figure 3-93. Tank bottom after draining.

Figure 3-94. Drain collar removed from the tank bottom.

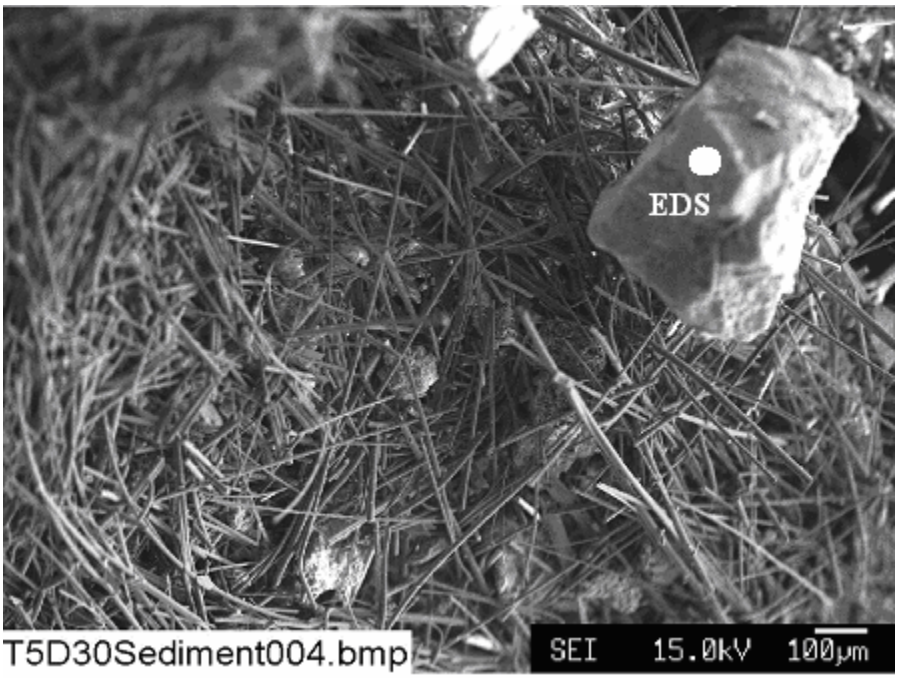

Figure 3-95: SEM image magnified 70 times for the Test #5 Day 30 sediment at the bottom of the tank.

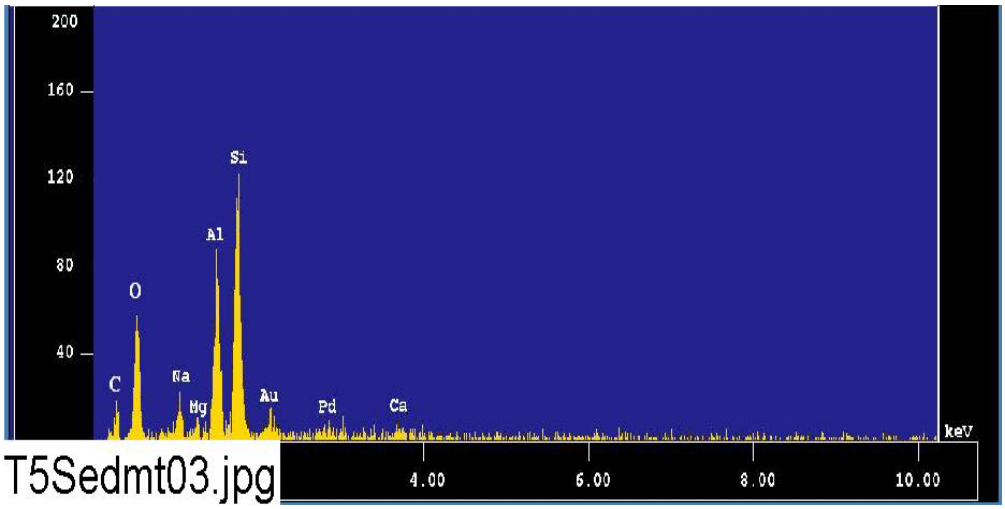

Figure 3-96: EDS counting spectrum for the big particulate deposit shown in Figure 3-95.

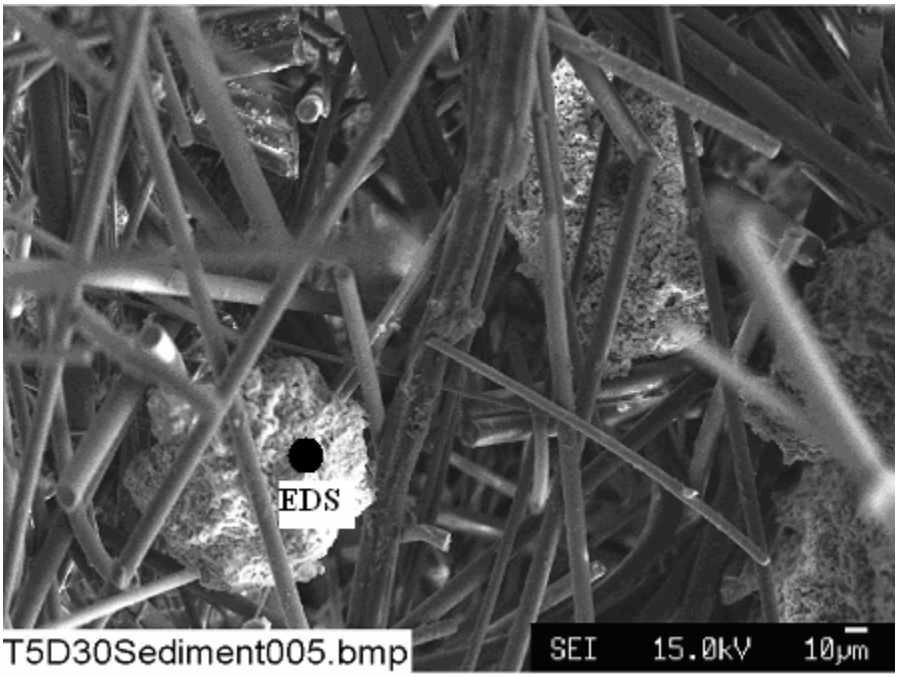

Figure 3-97: SEM image magnified 300 times for the Test #5 Day 30 sediment at the bottom of the tank.

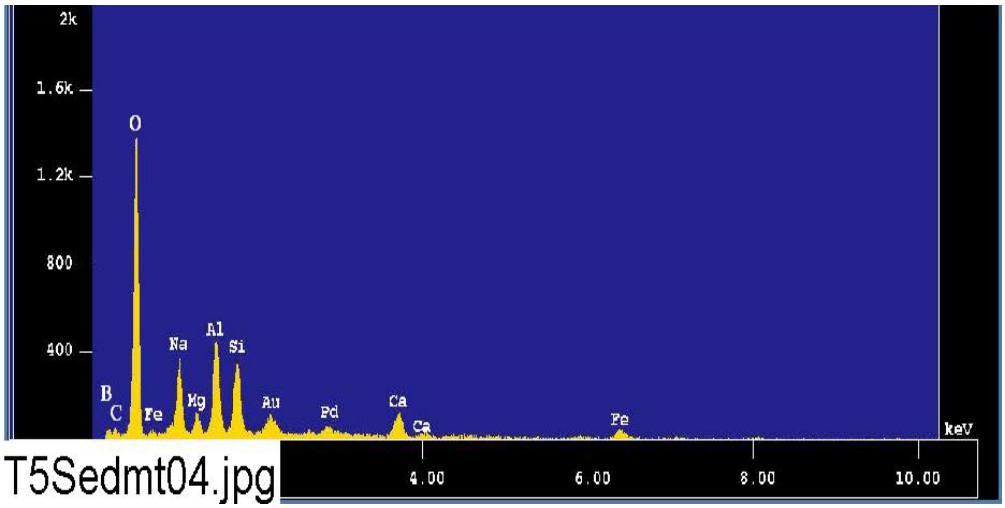

Figure 3-98: EDS counting spectrum for the particulate deposit shown in Figure 3-97.

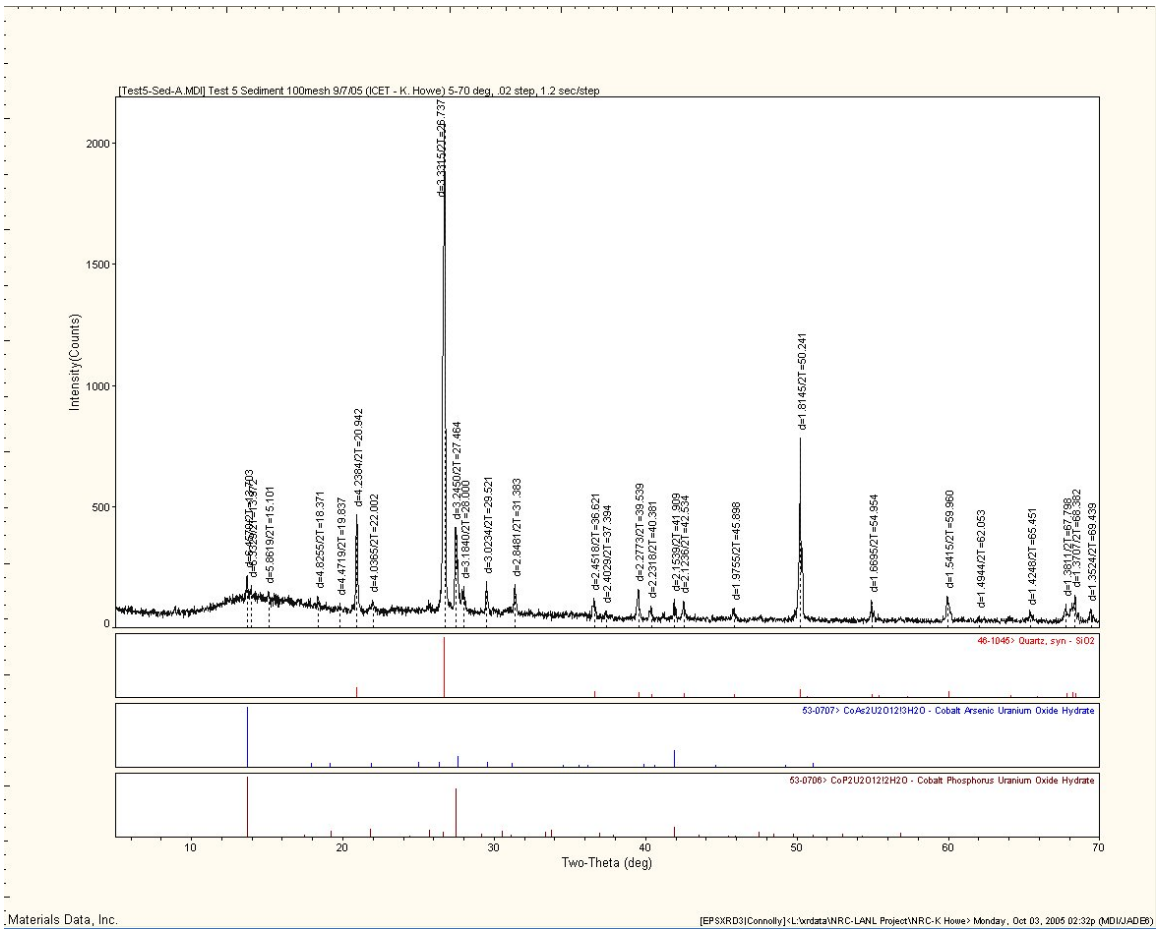

Figure 3-99: XRD result of the possible matching crystalline substances in Test #5 Day 30 sediment.

Table 3-6. Dry Mass Composition of Test #5 Day 30 Sediment by XRF Analysis

First row is chemical component; second row is mass composition (%).

SiO_2	TiO_2	Al_2O_3	Fe_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	$H_20(-)$	$H_2O(+)CO_2$	P_2O_5	Total
63.76	0.18	6.00	1.34	0.00	0.07	1.78	5.49	9.28	1.09	0.69	3.27	0.07	93.02

3.6 Deposition Products

For the ICET tests, one concern is the deposition of debris/corrosion products in the tank. To help understand this problem, the corrosion/deposition products were collected after completion of the test. These products are fine yellow powders removed from the submerged CPVC rack. SEM/ EDS results shown in Figures 3-100 through 3-102 indicate that the fine yellow powders were mainly composed of fiberglass debris and particulate deposits containing O, Na, Al, C, Ca, Mg, and possibly Si.

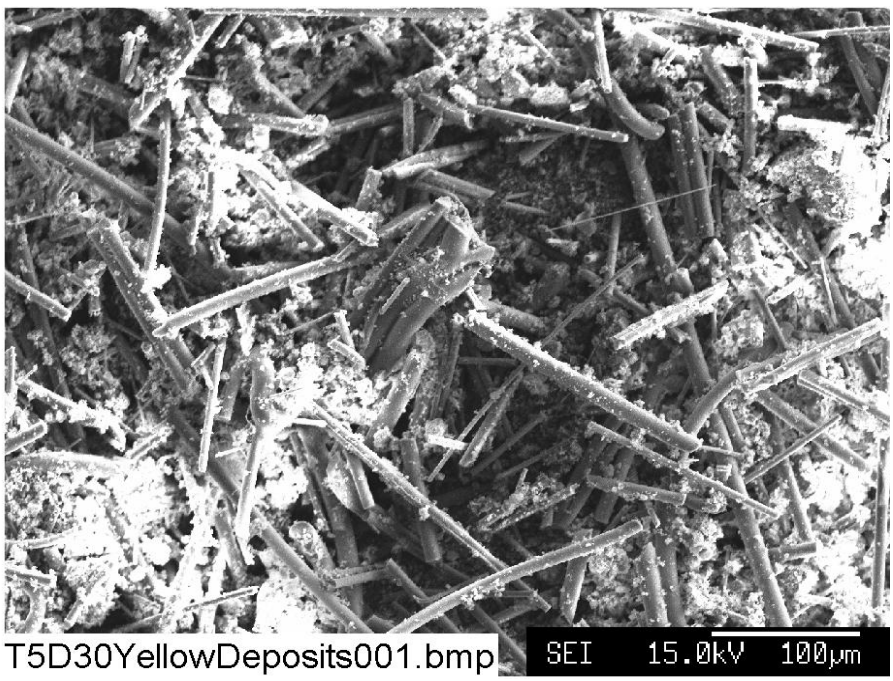

Figure 3-100: SEM image magnified 200 times for the Test #5 Day 30 fine yellow powder on the submerged rack.

Figure 3-101: Annotated SEM image magnified 1000 times for the Test #5 Day 30 fine yellow powder on the submerged rack.

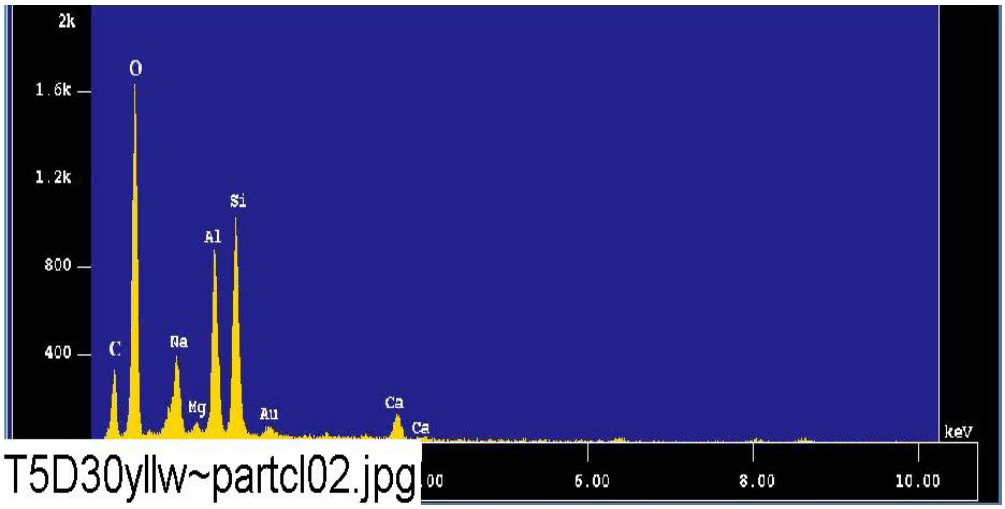

Figure 3-102: EDS counting spectrum for the particulate deposit shown in Figure 3-101.

3.7 Optical/TEM Images

The TEM images and EDS results for Test #5 Day 4, Day 15, and Day 30 unfiltered solution samples are shown in Appendix G. The unfiltered solution samples were extracted from the tank directly. A tiny drop of the test solution was transferred to a copper mesh and dried in air for TEM analysis.

TEM results show the presence of submicron-size colloidal particles in the samples (see Figures 3-103 through 3-105). However, it is unclear if the colloidal particles originally existed in the test solution or were formed during the drying process before TEM examination.

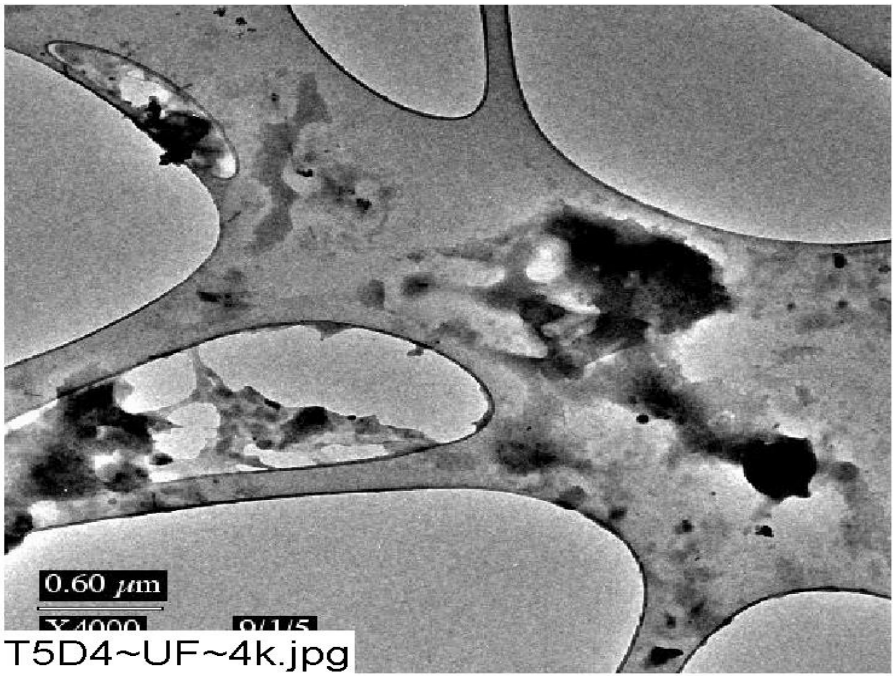

Figure 3-103: TEM magnified 4,000 times for one Test #5 Day 4 unfiltered sample location.

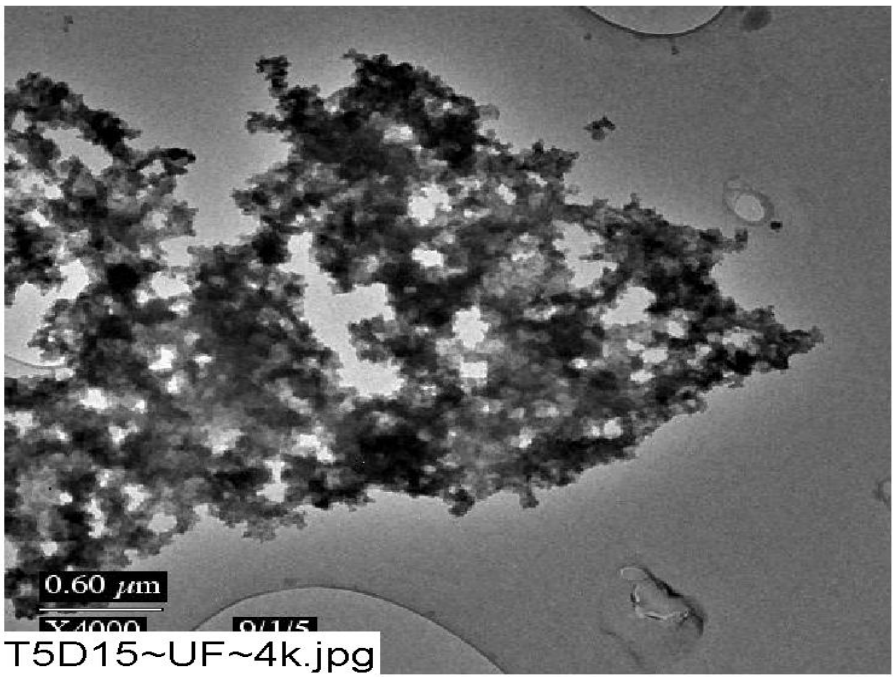

Figure 3-104: TEM magnified 4,000 times for one Test #5 Day 15 unfiltered sample location.

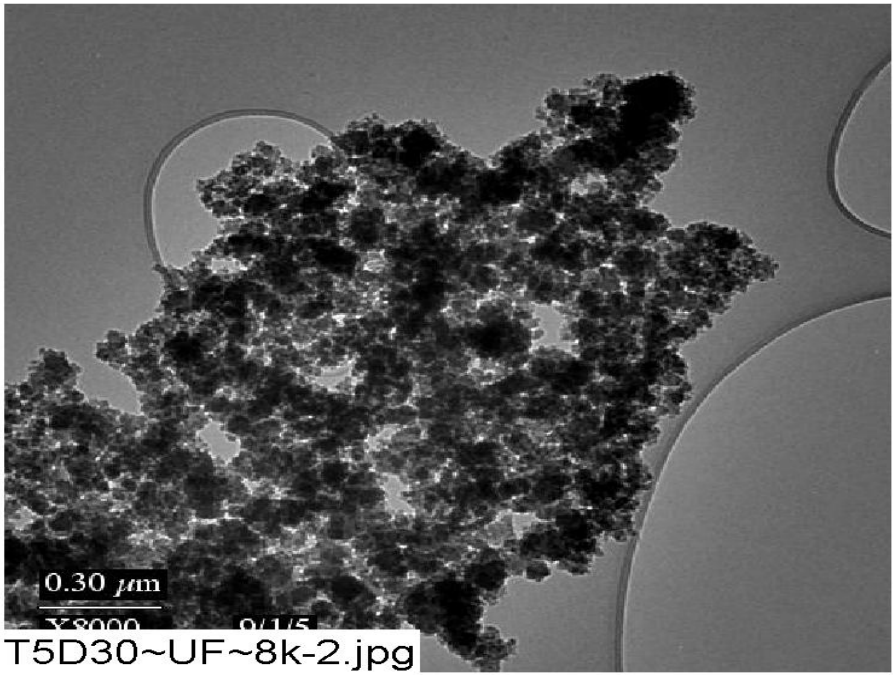

Figure 3-105: TEM magnified 8,000 times for one Test #5 Day 30 unfiltered sample location.

3.8 UV Absorbance Spectrum

UV absorbance was measured for the Day 30 filtered solution sample and is shown in Appendix H. The purpose of this analysis was to identify the presence of any distinguished absorbance peaks that may identify organics present in the solution. However, based on the result, no distinguished absorbance peaks were found due to the heterogeneous nature of the test solution.

3.9 Shear-Dependent Viscosity

As explained in the Test #1 data report (Ref. 2), shear-rate viscosity measurements were taken weekly on test solution samples. The Test #1 solution at 25ºC exhibited shear-thinning behavior, indicative of a non-Newtonian fluid. The solutions for Tests #2, #3, and #4 were all Newtonian fluids at 25ºC.

As with Test #1, the Test #5 solution at 25ºC exhibited shear-thinning behavior, as shown in Figure 3-106. The legend for the symbols gives the date the samples were remove from the solution. For example, “072705” is Day 2, and “082505” is Day 30. Shear thinning is indicated by the decrease in viscosity as the shear rate is increased to 20 1/s. Additionally, the solution’s viscosity increases at all shear rates with time over the test duration, with one exception. The last sample taken on Day 30 (082505-0900-U) decreased to a value similar to that of the mid-test sample. While there is no definitive explanation for the decrease, it may be related to the late-test behavior of the turbidity, TSS, and aluminum and calcium concentrations.

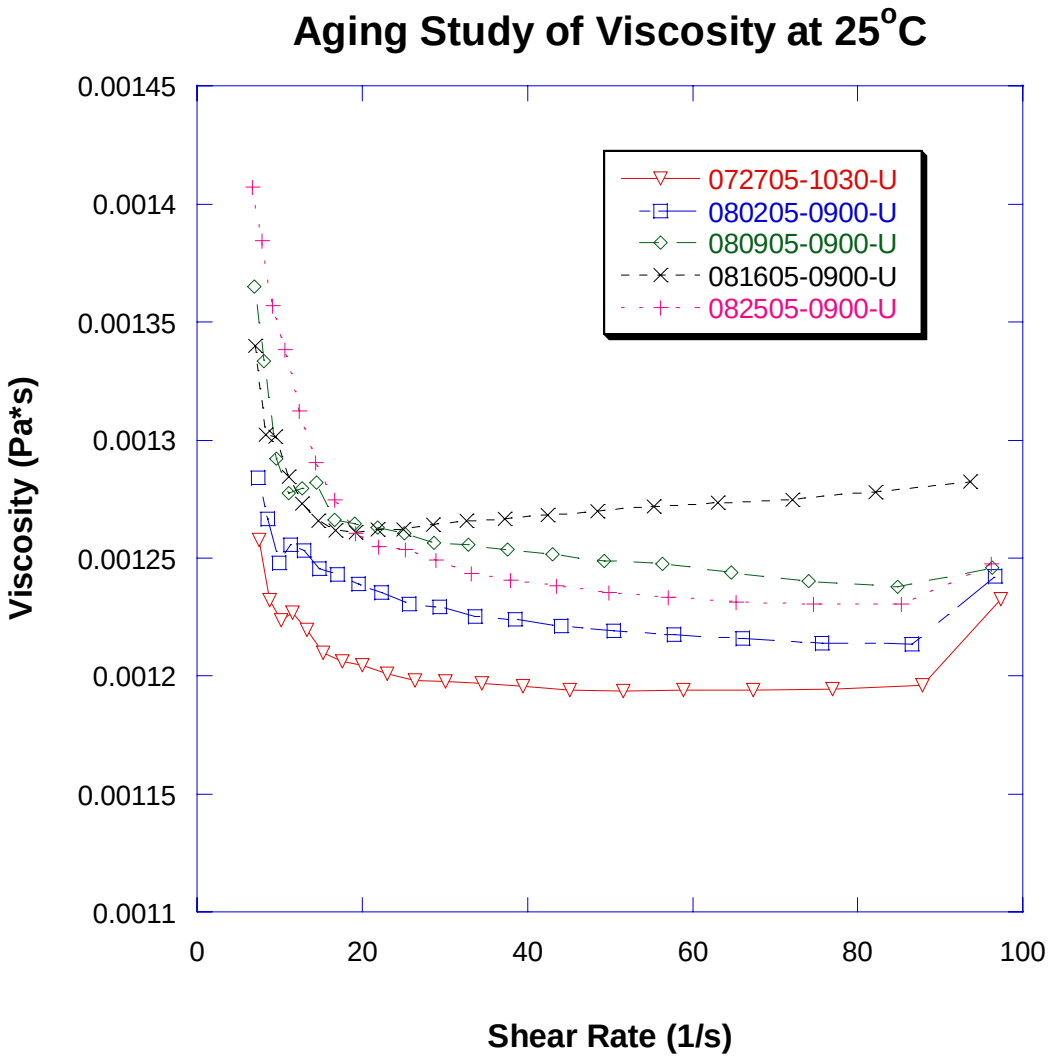

Figure 3-106: Shear-rate viscosity measurements at 25ºC.

4 SUMMARY OF KEY OBSERVATIONS

ICET Test #5 ran for 30 days, and all conditions were maintained within the accepted flow and temperature ranges, with one exception. On Day 5, the addition of cold makeup water caused the test solution temperature to drop to 57.7ºC, which is 0.3ºC below the target minimum. The minimum temperature was below 58.0ºC for less than 10 minutes. At the start of the test, the measured pH was 8.4. During the addition of hydrochloric acid, the pH dropped slightly to 8.3, and it remained between 8.2 and 8.4 for the duration of the test. The test solution turbidity decreased to approximately 2 NTU after 7 days. The turbidity at 60ºC decreased to approximately 1 NTU the following day and remained near 1 NTU for the duration of the test. However, when the solution was cooled to 23ºC, the turbidity increased to 5 NTU at Day 19 and remained near that value for the duration of the test.

Samples of the solution were taken daily. Analyses of the test solution showed that aluminum in the solution rose above 50 mg/L on Day 11 and fluctuated between 33 and 55 mg/L for the duration of the test. Calcium, silica, sodium, chloride, and boron were also prevalent in the solution.

Daily measurements of the constant-shear kinematic viscosity of the test solution revealed an approximately constant value at both test temperature and room temperature. Measurements of the shear-dependent viscosity indicated that at 60°C the test solution remained Newtonian for the entire test. At 25°C, the test solution exhibited shear-thinning behavior, and the viscosity generally increased at all shear rates as the test progressed. Light, wispy precipitates were visible after the test solution sat at room temperature for several days.

Examinations of fiberglass taken from the test apparatus revealed chemical byproducts and web-like deposits that spanned individual fibers. Flocculent deposits were also observed. The amounts of these deposits did not appear to increase significantly over the duration of the test, and the web-like deposits were absent in the Day 30 samples. The deposits were likely formed by chemical precipitation. In addition to these deposits, some samples had significant amounts of particulate deposits on their exteriors that were likely physically attached.

The submerged metallic coupons all developed thin particulate deposits that dulled their color and roughened their surface. Post-test examinations showed that the submerged aluminum coupons lost approximately 3% of their weight, but there were very little weight changes on the other coupons. The unsubmerged coupons exhibited some streaking but little or no weight change.

After the tank was drained, there was very little sediment on the tank bottom, but powdery deposition products were present on the submerged objects.

REFERENCES

1. "Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Rev. 13," July 20, 2005.

2. J. Dallman, J. Garcia, M. Klasky, B. Letellier, and K. Howe, "Integrated Chemical Effects Test Project: Test #1 Data Report," LA-UR-05-0124, June 2005.

3. "Pre-Test Operations, Test #5," ICET-PI-017, Rev. 0, July 22, 2005.

4. "Test Operations, Test #5 (fiberglass and sodium tetraborate at pH 8)," ICET-PI-018, Rev. 0, July 22, 2005.

5. "Post-Test Operations, Test #3," ICET-PI-008, Rev. 3, May 2, 2005.

Preface to Appendices

Eight separate appendices were developed to capture more of the images and information obtained for Test #5. Several appendices are further divided into subappendices to better segregate the information according to the time point in the test when the samples were extracted from the test apparatus, the location of the samples in the tank, the type of samples being evaluated, and the type of examinations performed. With the exception of Appendix I, each appendix represents a separate session of laboratory work that can be traced to a batch of samples that were typically processed in chronological order. Appendix I provides the detailed project instructions that were used to initiate Test #5, to conduct routine operations during the test, and to terminate the test with sample recovery and cleaning procedures.

Section 2.4.1.1 of this report reviewed the nomenclature adopted for reporting ICET results. This nomenclature is used in the caption labels for most of the figures presented in the appendices.

As noted in Section 2.4.1.1, the data presented in the appendices are largely qualitative in nature, consisting primarily of SEM and TEM micrographs and EDS spectra. The SEM data are further subdivided into environmental (or low-vacuum) SEM of hydrated samples and microprobe SEM of fully desiccated samples. Microprobe images were generated using secondary electrons, which are sensitive to attenuation, to reveal fine structural details in a sample.

Transcriptions of the laboratory logbooks are provided for each appendix to document better commonalities that existed among the samples at the time of analysis. Logbook information was developed for most, but not all, of the images presented in the appendices. Interpretation and understanding of the images and their accompanying EDS spectra can be improved by referring frequently to the logbook sample descriptions and sequences.

Typically, a relatively large quantity of a test sample was delivered for SEM or TEM analysis, and then several small subsamples of each item were examined. Note that each subsample was assigned a sequential reference number during the laboratory session. These reference numbers have been cited in the figure captions whenever possible to preserve the connection between the micrographs and the notebook descriptions. Electronic filenames have also been stamped on the images to permit retrieval of the original data files, which are archived elsewhere. Individual data sets for a given sample item have been collated into a typical sequence of (1) visual image, (2) EDS spectra, and (3) semiquantitative mass composition.

Semiquantitative mass compositions are also presented for many of the EDS spectra. These results are obtained from a commercial algorithm that decomposes the spectra into the separate contributions of each element. Composition estimates should be interpreted with the caveats stated in Section 2.4.1.1 fully in mind.

Appendix titles are listed below for reference.

Appendix A ESEM/EDS Data for Test #5 Day-4 Fiberglass in Low-Flow Zone

Appendix B ESEM Day-15 Fiberglass

B1. ESEM/EDS Data for Test #5 Day-15 Fiberglass in Low-Flow Zones
B2. ESEM Data for Test #5 Day-15 Fiberglass in High-Flow Zones

Appendix C ESEM Day-30 Fiberglass

C1. ESEM Data for Test #5 Day-30 Fiberglass in Low-Flow Zones
C2. ESEM/EDS Data for Test #5 Day-30 Fiberglass Samples in a Big Envelope in Low-Flow Zones
C3. ESEM Data for Test #5 Day-30 Fiberglass in High-Flow Zones
C4. ESEM Data for Test #5 Day-30 Fiberglass Inserted in Front of Header in High-Flow Zones
C5. ESEM/EDS Data for Test #5 Day-30 Fiberglass Inserted in Nylon Mesh in High-Flow Zones
C6. ESEM/EDS and SEM Data for Test #5 Day-30 Drain Collar Fiberglass
C7. ESEM/EDS Data for Test #5 Day-30 Birdcage Fiberglass

Appendix D SEM/EDS Data for Test #5 Day-30 Deposition Products

Appendix E SEM Day-30 Coupons

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E2. SEM/EDS Data for Test #5 Day-30 Copper Coupons
E3. SEM/EDS Data for Test #5 Day-30 Galvanized Steel Coupons
E4. SEM/EDS Data for Test #5 Day-30 Steel Coupons

Appendix F SEM/EDS Data for Test #5 Day-30 Sediment

Appendix G TEM Data for Test #5 Solution Samples

Appendix H UV Absorbance Spectrum – Day-30 Solution Samples

Appendix I ICET Test #5: Pre-Test, Test, and Post-Test Project Instructions

Appendix A

ESEM/EDS Data for Test #5 Day-4 Fiberglass in Low-Flow Zone

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During ICET Test #5, work continued for the purpose of identifying the origin and chemical composition of the products that were formed during the test. One objective of ICET tests is to identify the composition of debris that collects on fiberglass and the particulate substances in the test solution. To address this question partially, low-flow fiberglass samples on Test #5 Day 4 were examined by Environmental SEM (ESEM)/EDS, including both the exterior and the interior location of the fiberglass samples.

ESEM was employed to analyze the hydrated fiberglass samples. These samples were examined under a low vacuum condition (i.e., 80 Pa) and without any coating, to minimize the modification of the fiberglass samples through drying process. EDS results provide a semi-quantitative elemental analysis of the debris attached on fiberglass.

Test #5 Day-4 low-flow fiberglass samples were obtained on July 30, 2005 (Day 4 for Test #5). SEM/EDS data presented here were obtained on August 3, 2005.

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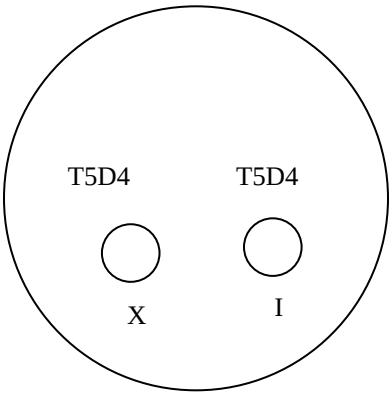
Transcribed Laboratory Log

Laboratory session from August 3, 2005.

ESEM/EDS Test #5 Day-4 Fiberglass in low-flow zone



ESEM Exterior Low-flow Fiberglass Samples

Image:	T5D4Ex01	100 ×	ESEM image	Figure A-1
	t5d4ex02	100 ×		Figure A-2
	t5d4ex03	500 ×	ESEM at higher magnification	Figure A-3

ESEM/EDS Interior Low-flow Fiberglass Samples.

Image:	t5d4lfin04	100 ×	ESEM image	Figure A-4
	t5d4lfin05	500 ×	ESEM of debris	Figure A-5
EDS:	t5d4lfin06		EDS on flocculence deposits t5drlfin05	Figure A-6
Image:	t5d4lfin07	500 ×	ESEM image	Figure A-7
EDS:	t5d4lfin08		EDS on deposits on t5d4lfin07	Figure A-8

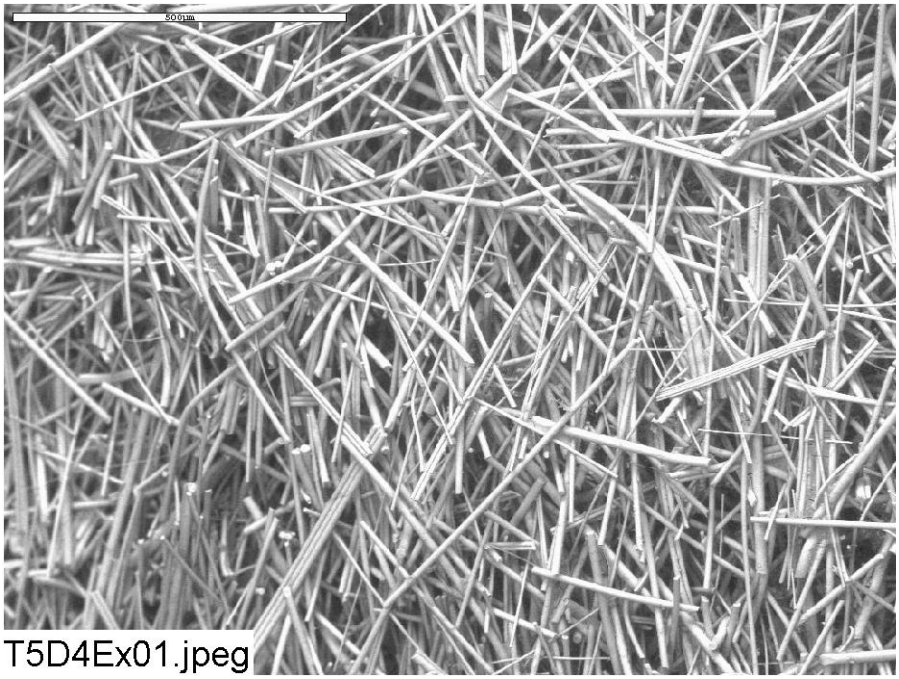


Figure A-1: Environmental SEM image magnified 100 times for a Test #5 Day-4 low-flow exterior fiberglass sample. (T5D4Ex01.jpg)

Figure A-2: Environmental SEM image magnified 100 times for a Test #5 Day-4 low-flow exterior fiberglass sample. (t5d4ex02.jpg)

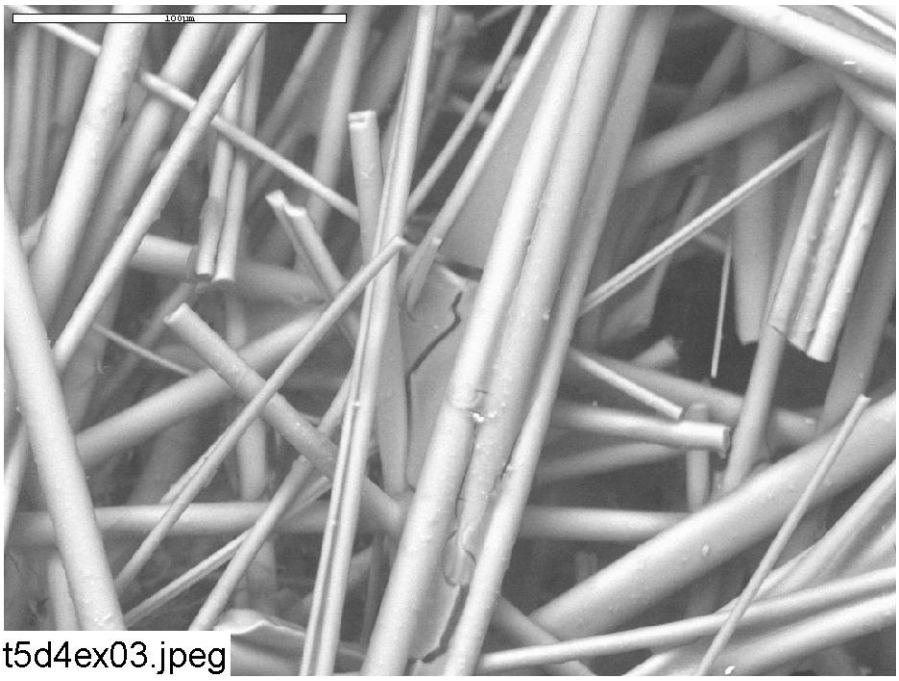


Figure A-3: Environmental SEM image magnified 500 times for a Test #5 Day-4 low-flow exterior fiberglass sample. (t5d4ex03.jpg)

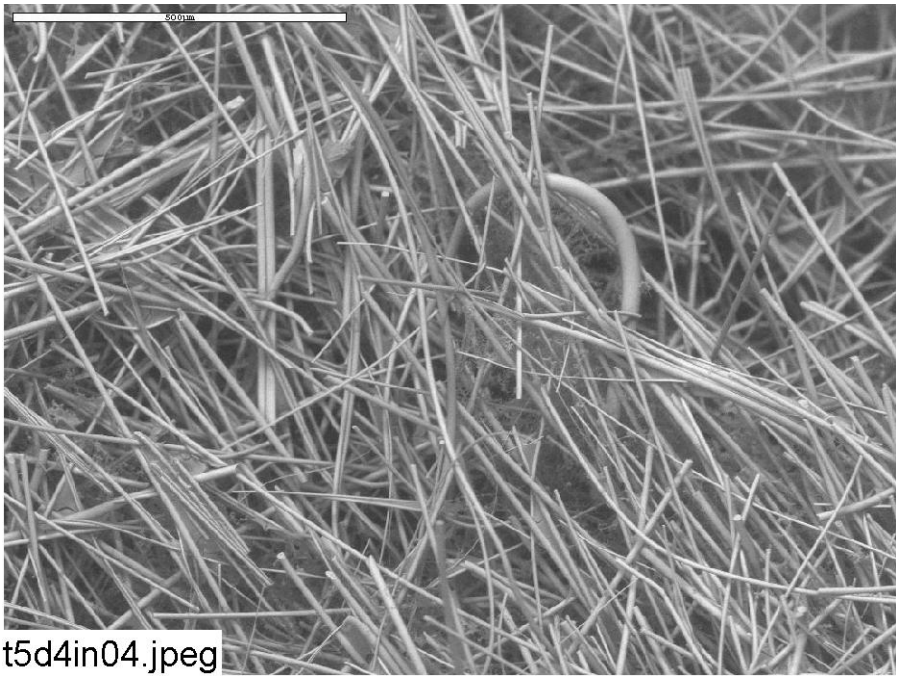


Figure A-4: Environmental SEM image magnified 100 times for a Test #5 Day-4 low-flow interior fiberglass sample. (t5d4in04.jpg)

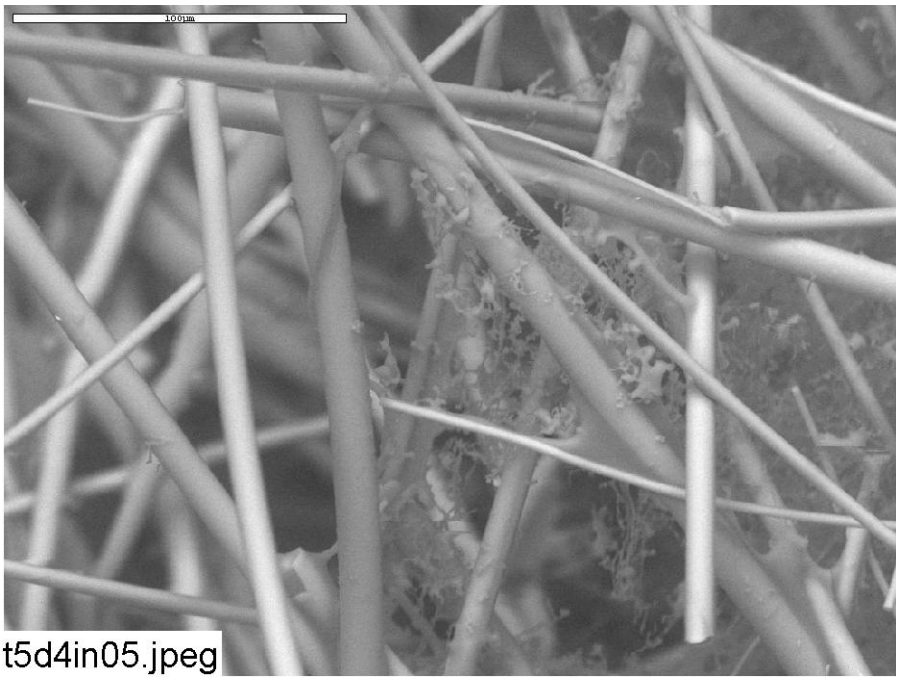


Figure A-5: Environmental SEM image magnified 500 times for a Test #5 Day-4 low-flow interior fiberglass sample. (t5d4in05.jpg)

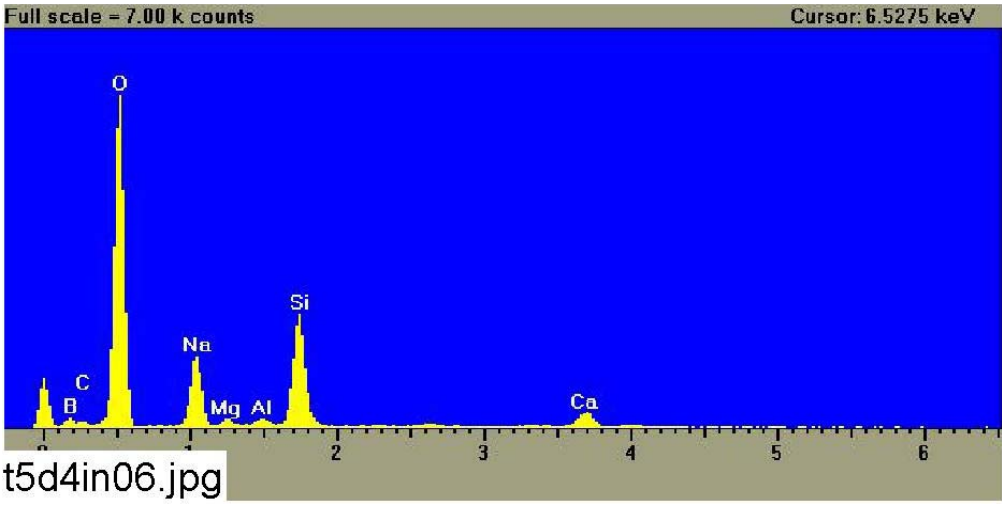


Figure A-6: EDS counting spectrum for the flocculence deposits between fibers shown in Figure A-5. (t5d4in06.jpg)

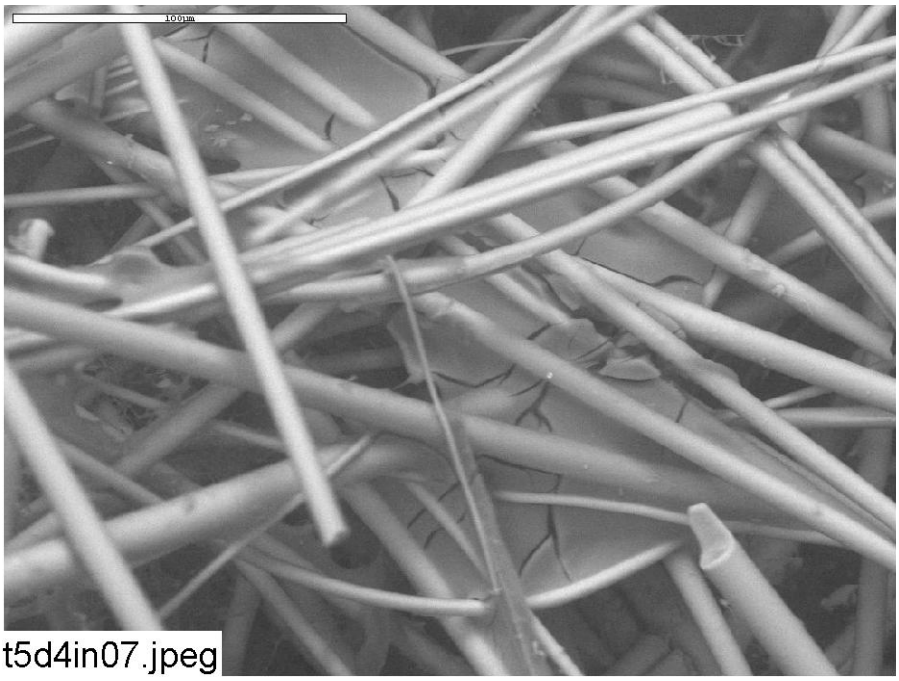


Figure A-7: Environmental SEM image magnified 500 times for a Test #5 Day-4 low-flow interior fiberglass sample. (t5d4in07.jpg)

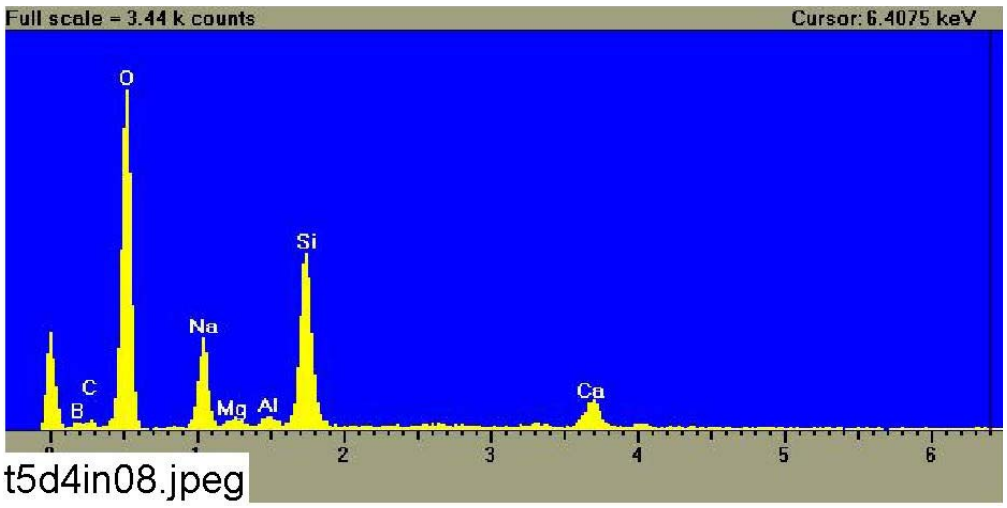


Figure A-8: EDS counting spectrum for the deposits between fibers shown in Figure A-7. (t5d4in08.jpg)

Appendix B1

ESEM/EDS Data for Test #5 Day-15 Fiberglass in Low-Flow Zones

List of Figures

Figure B1-1: Environmental SEM image magnified 100 times for a Test #5 Day-15 low-flow exterior fiberglass sample. (T5D15lx1.jpg) B1-4

Figure B1-2: Environmental SEM image magnified 500 times for a Test #5 Day-15 low-flow exterior fiberglass sample. (t5d15lx2.jpg) B1-4

Figure B1-3: Environmental SEM image magnified 100 times for a Test #5 Day-15 low-flow interior fiberglass sample. (t5d15li3.jpg) B1-5

Figure B1-4: Environmental SEM image magnified 500 times for a Test #5 Day-15 low-flow interior fiberglass sample. (t5d15li4.jpg) B1-5

Figure B1-5: Environmental SEM image magnified 500 times for a Test #5 Day-15 low-flow interior fiberglass sample. (t5d15li5.jpg) B1-6

Figure B1-6: Annotated environmental SEM image magnified 500 times for a Test #5 Day-15 low-flow interior fiberglass sample. (t5d15li6.jpg) B1-6

Figure B1-7: EDS counting spectrum for the deposits between fibers shown in Figure B1-6. (t5d15li7.jpg) B1-7

This appendix shows the ESEM/EDS results on ICET Test #5 Day-15 low-flow zone fiberglass samples. The samples were obtained on August 10, 2005 (Day 15 for Test #5). Both exterior and interior locations of the fiberglass samples were examined. ESEM/EDS data presented here were obtained on August 25, 2005. The hydrated fiberglass samples without any coating were examined by ESEM under a low vacuum condition (i.e., 80 Pa). EDS results provide a semi-quantitative elemental analysis of the debris attached on fiberglass.

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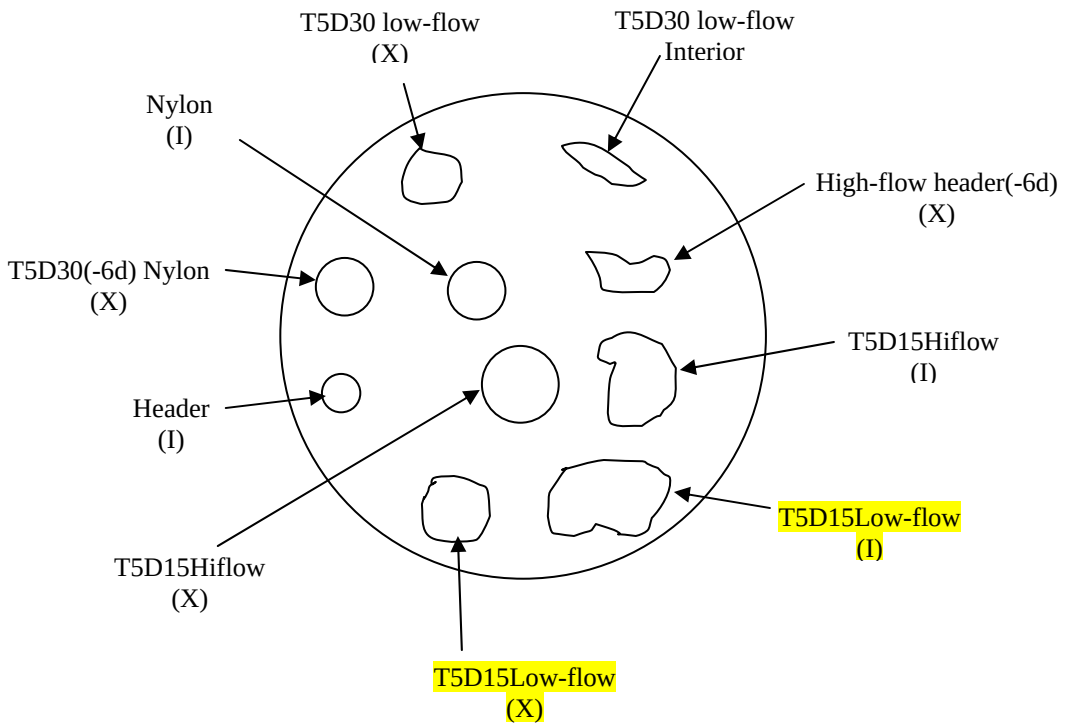
Transcribed Laboratory Log

Laboratory session from August 25, 2005.
Test #5 Day-15 fiberglass in low-flow zones

ESEM



ESEM Fiberglass Low-Flow Exterior

Image:	T5D15lx1	100 ×	ESEM image of fiberglass	Figure B1-1
	t5d15lx2	500 ×	ESEM image of fiberglass	Figure B1-2

ESEM Fiberglass Low-Flow Interior

Image:	T5d15li3	100 ×	ESEM image of fiberglass	Figure B1-3
	t5d15li4	500 ×	ESEM image at higher magnification	Figure B1-4
	t5d15li5	500 ×	ESEM image	Figure B1-5
	t5d15li6	500 ×	Annotated ESEM image	Figure B1-6
EDS:	t5d15li7		EDS on deposits between fibers for t5d15li6	Figure B1-7

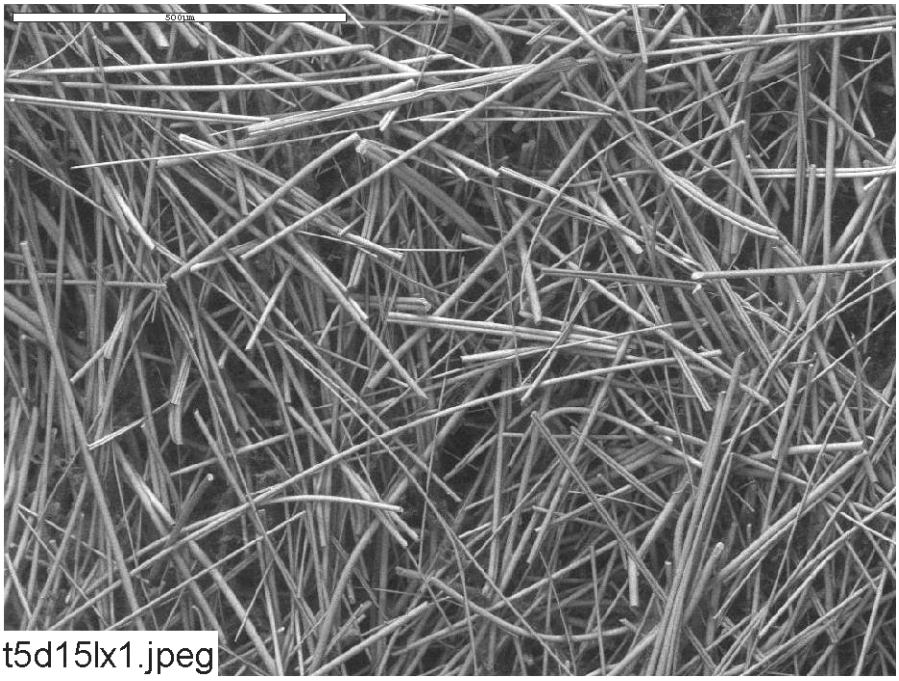


Figure B1-1: Environmental SEM image magnified 100 times for a Test #5 Day-15 low-flow exterior fiberglass sample. (T5D15lx1.jpg)

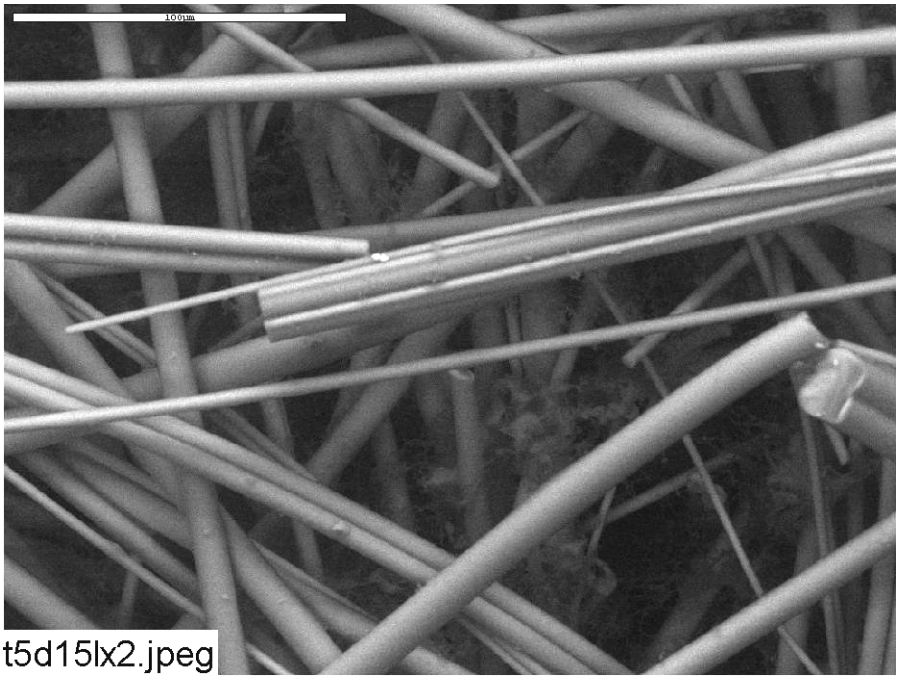


Figure B1-2: Environmental SEM image magnified 500 times for a Test #5 Day-15 low-flow exterior fiberglass sample. (t5d15lx2.jpg)

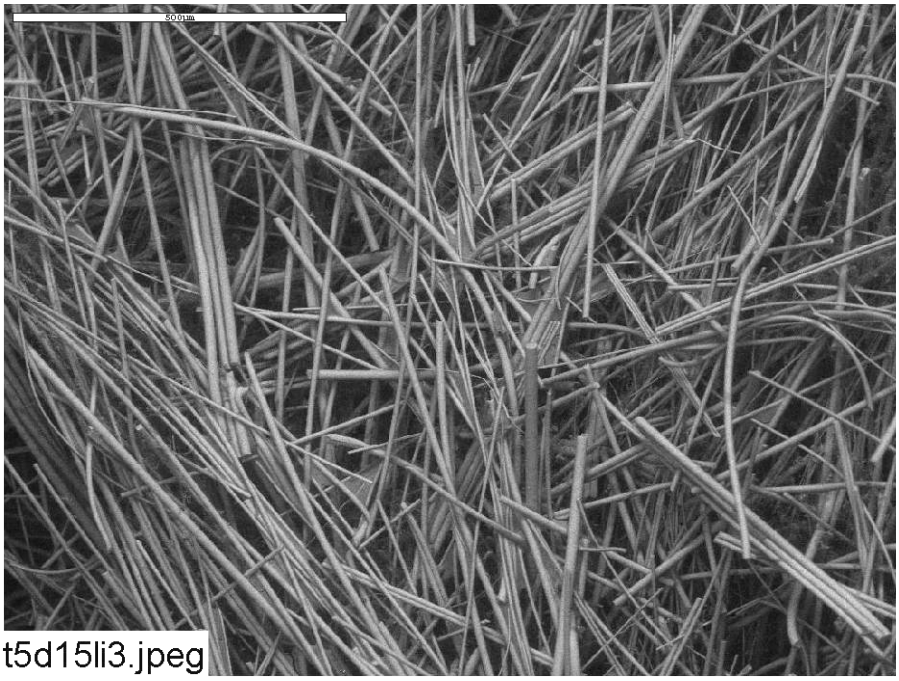


Figure B1-3: Environmental SEM image magnified 100 times for a Test #5 Day-15 low-flow interior fiberglass sample. (t5d15li3.jpg)

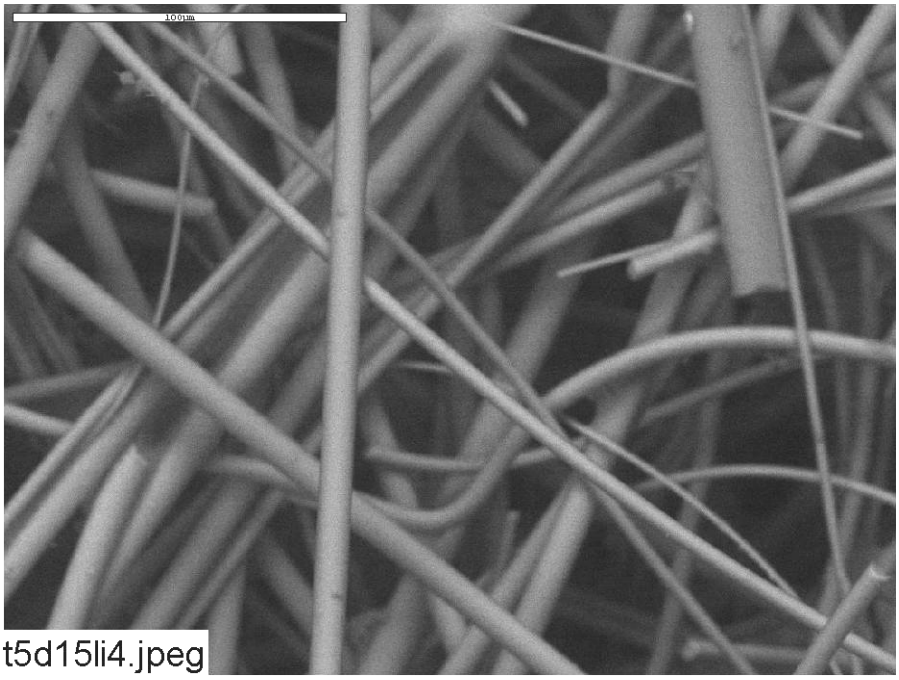


Figure B1-4: Environmental SEM image magnified 500 times for a Test #5 Day-15 low-flow interior fiberglass sample. (t5d15li4.jpg)

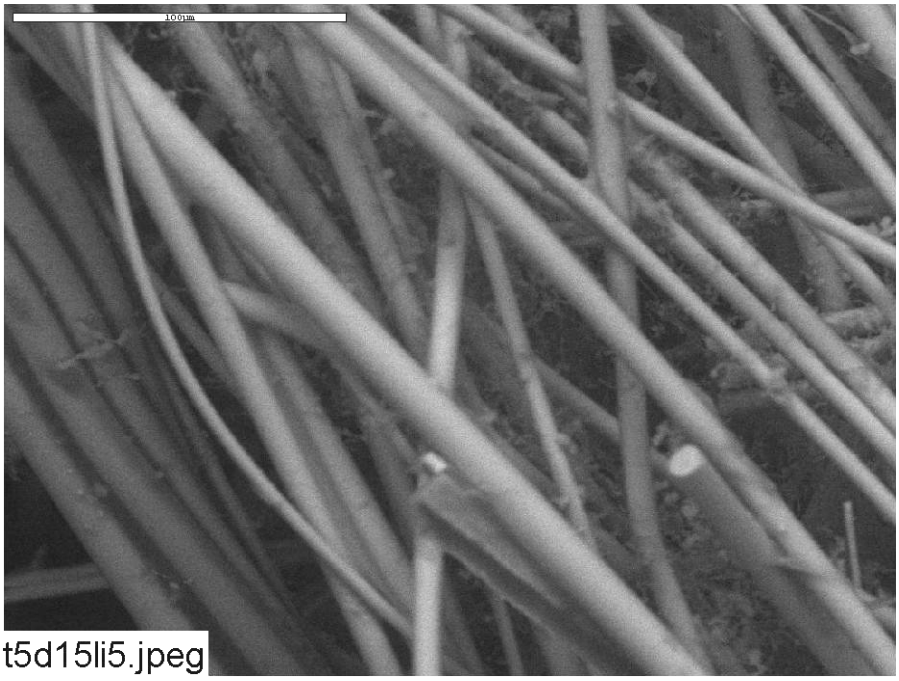


Figure B1-5: Environmental SEM image magnified 500 times for a Test #5 Day-15 low-flow interior fiberglass sample. (t5d15li5.jpg)

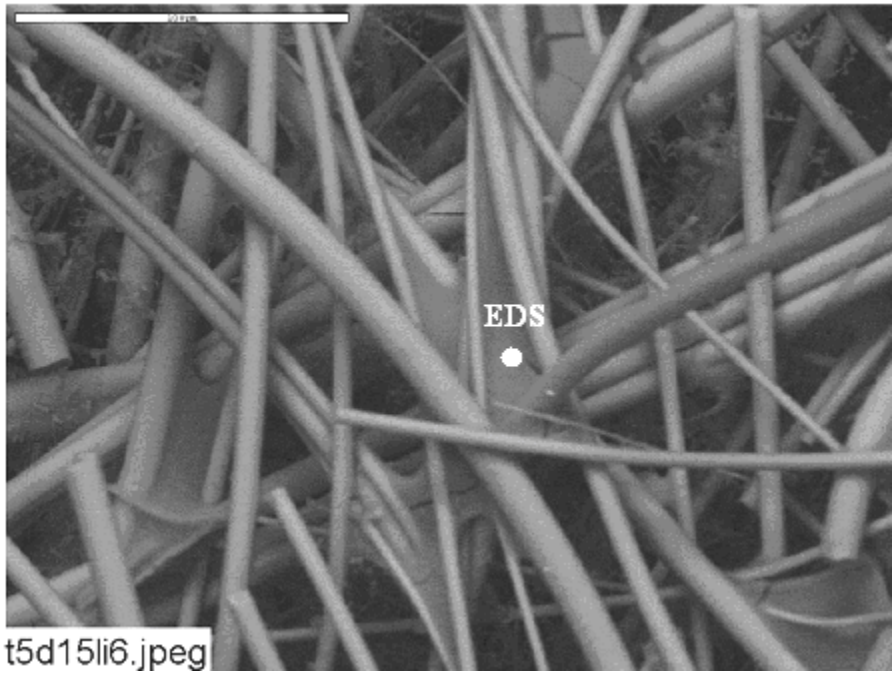


Figure B1-6: Annotated environmental SEM image magnified 500 times for a Test #5 Day-15 low-flow interior fiberglass sample. (t5d15li6.jpg)

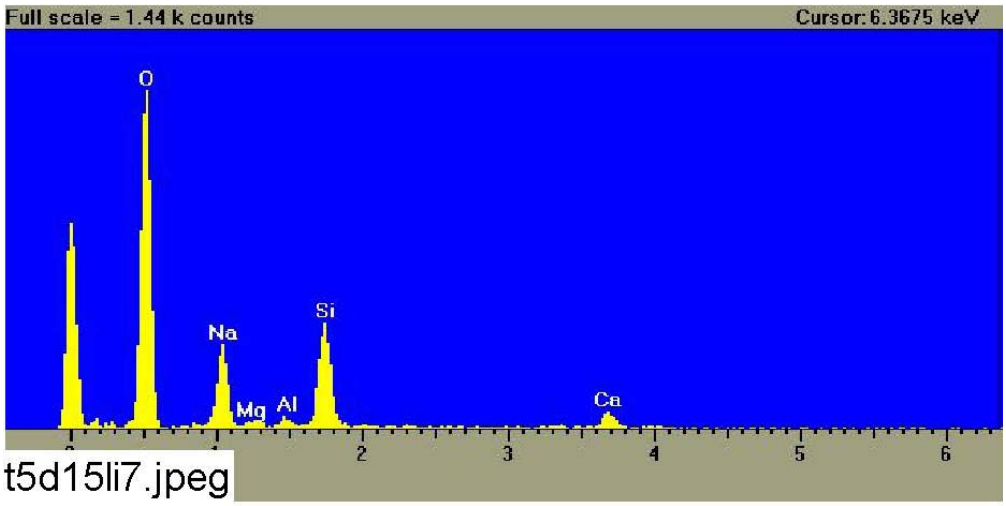


Figure B1-7: EDS counting spectrum for the deposits between fibers shown in Figure B1-6. (t5d15li7.jpg)

Appendix B2

ESEM Data for Test #5 Day-15 Fiberglass in High-Flow Zones

List of Figures

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Figure B2-2: Environmental SEM image magnified 500 times for a Test #5 Day-15 high-flow exterior fiberglass sample. (t5d15hx2.jpg) B2-4

Figure B2-3: Environmental SEM image magnified 100 times for a Test #5 Day-15 high-flow interior fiberglass sample. (T5D15HI3.jpg) B2-5

Figure B2-4: Environmental SEM image magnified 500 times for a Test #5 Day-15 high-flow interior fiberglass sample. (t5d15hi4.jpg)..... B2-5

This appendix lists the ESEM/EDS results on ICET Test #5 Day-15 high-flow zone fiberglass samples. The samples were obtained on August 10, 2005 (Day 15 for Test #5). Both exterior and interior locations of the fiberglass samples were examined. ESEM/EDS data presented here were obtained on August 25, 2005. The hydrated fiberglass samples without any coating were examined by ESEM under a low vacuum condition (i.e., 80 Pa).

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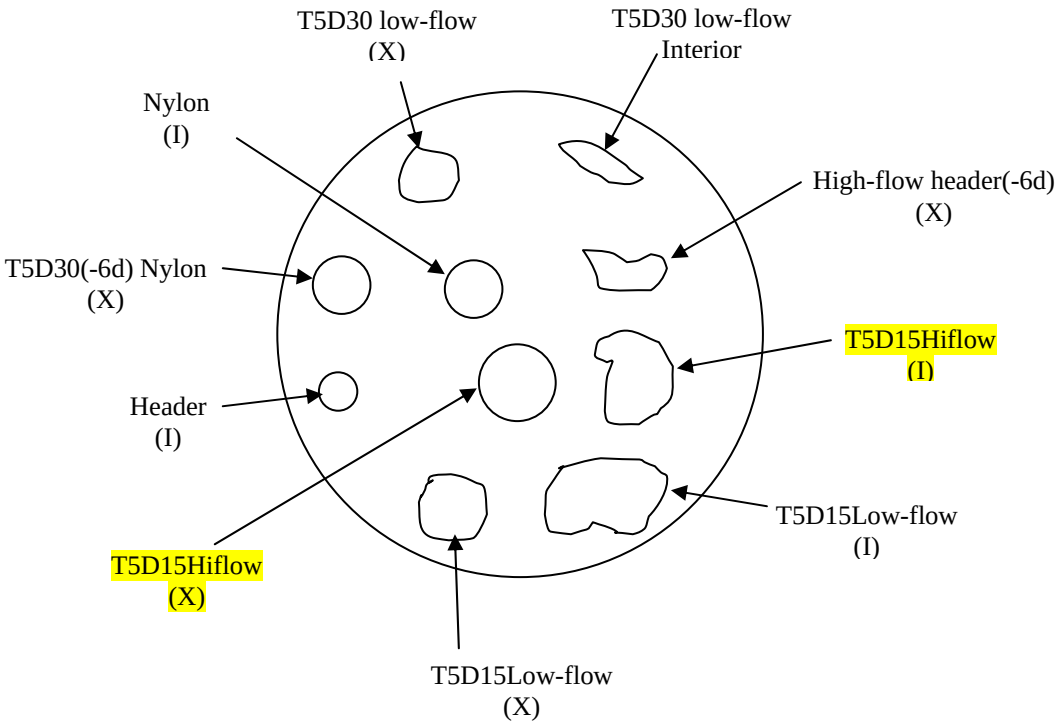
Transcribed Laboratory Log

Laboratory session from August 25, 2005.
Test #5 Day-15 fiberglass in high-flow zones

ESEM



ESEM Fiberglass High-flow Exterior

Image:	T5D15HX1	100 ×	ESEM image	Figure B2-1
	t5d15hx2	500 ×	ESEM image at higher magnification	Figure B2-2

ESEM Fiberglass High-flow Interior

Image:	T5D15HI3	100 ×	ESEM image	Figure B2-3
	t5d15hi4	500 ×	ESEM image at higher magnification	Figure B2-4

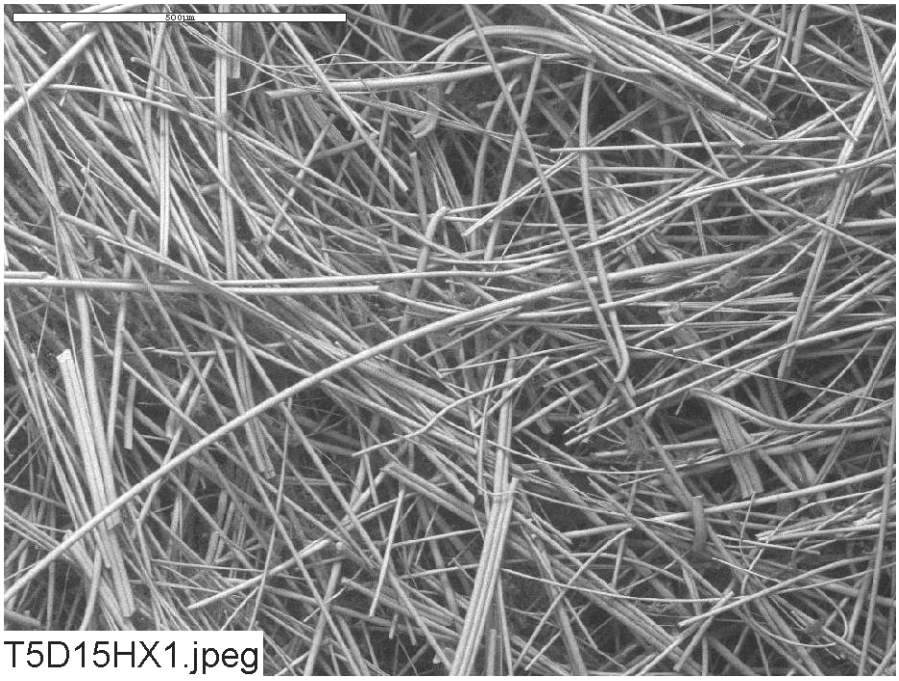


Figure B2-1: Environmental SEM image magnified 100 times for a Test #5 Day-15 high-flow exterior fiberglass sample. (T5D15HX1.jpg)

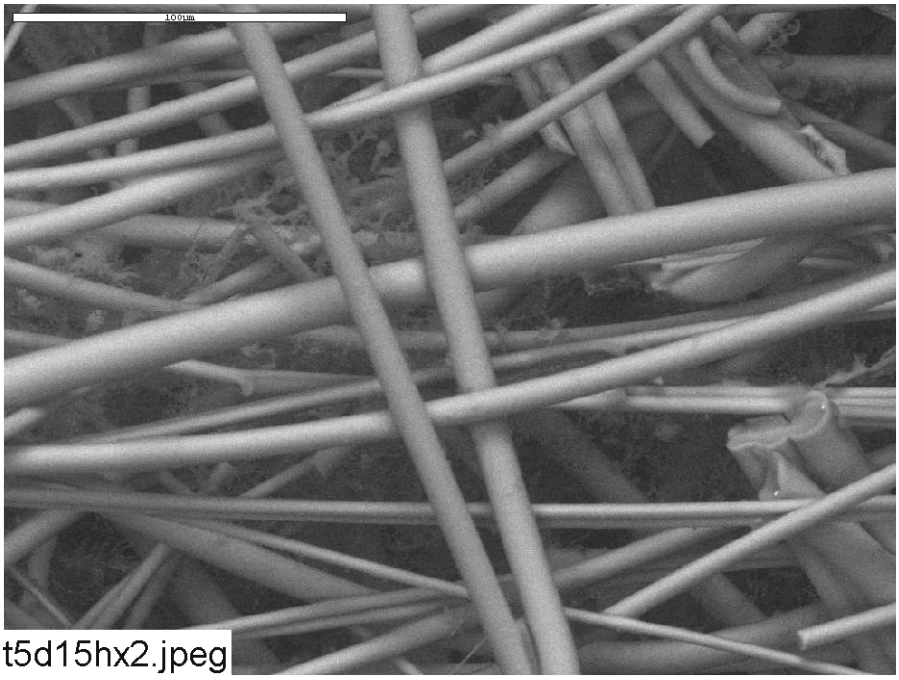


Figure B2-2: Environmental SEM image magnified 500 times for a Test #5 Day-15 high-flow exterior fiberglass sample. (t5d15hx2.jpg)

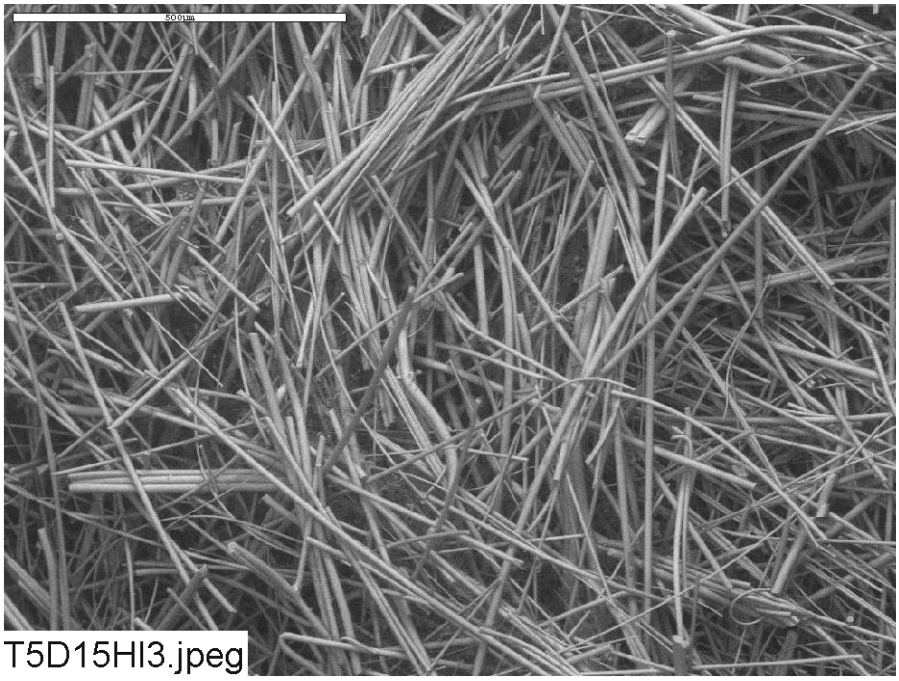


Figure B2-3: Environmental SEM image magnified 100 times for a Test #5 Day-15 high-flow interior fiberglass sample. (T5D15HI3.jpg)

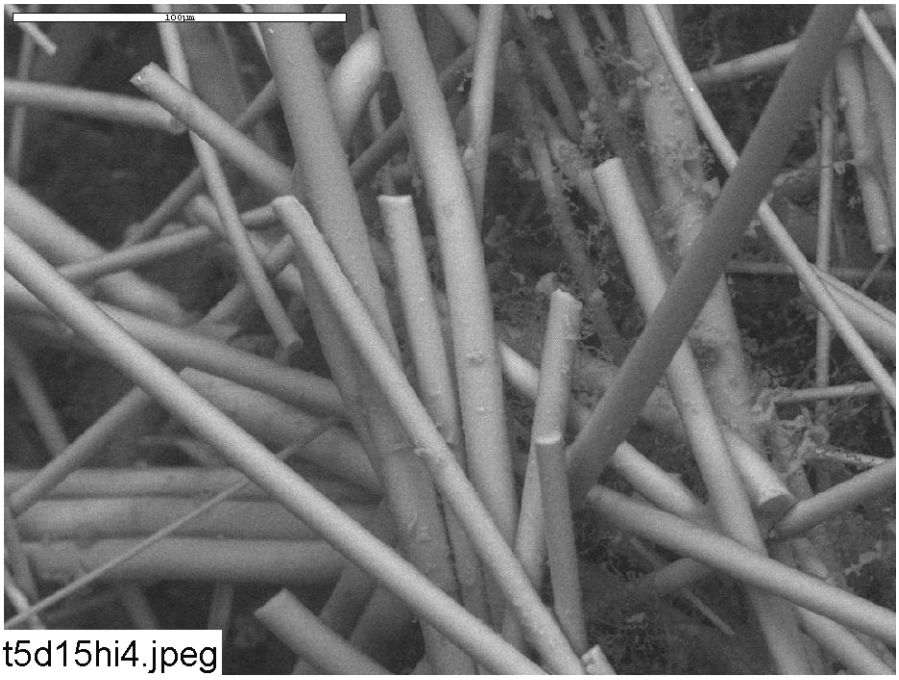


Figure B2-4: Environmental SEM image magnified 500 times for a Test #5 Day-15 high-flow interior fiberglass sample. (t5d15hi4.jpg)

Appendix C1

ESEM Data for Test #5 Day-30 Fiberglass in Low-Flow Zones

List of Figures

Figure C1-1: Environmental SEM image magnified 100 times for a Test #5 Day-30 low-flow exterior fiberglass sample. (t5d30lx3.jpg) C1-4

Figure C1-2: Environmental SEM image magnified 500 times for a Test #5 Day-30 low-flow exterior fiberglass sample. (t5d30lx4.jpg) C1-4

Figure C1-3: Environmental SEM image magnified 500 times for a Test #5 Day-30 low-flow exterior fiberglass sample. (t5d30lx5.jpg) C1-5

Figure C1-4: Environmental SEM image magnified 100 times for a Test #5 Day-30 low-flow interior fiberglass sample. (t5d30lI6.jpg)..... C1-5

Figure C1-5: Environmental SEM image magnified 500 times for a Test #5 Day-30 low-flow interior fiberglass sample. (t5d30li7.jpg)..... C1-6

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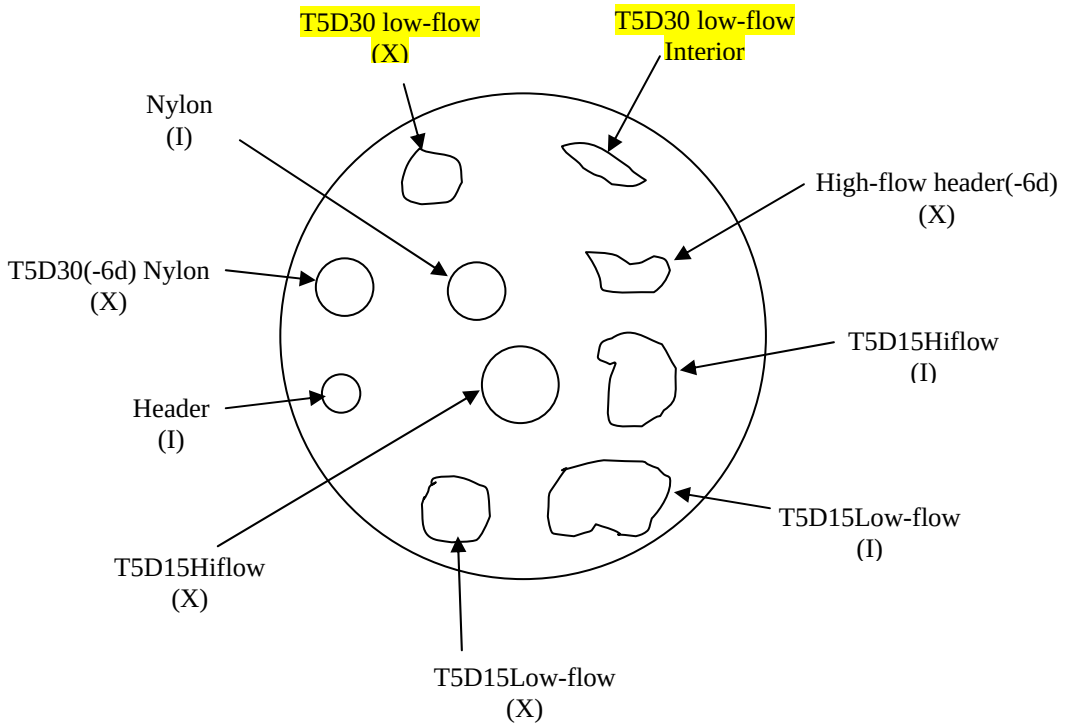
In this appendix, the fiberglass samples were extracted from the test tank on August 25, 2005, the date Test #5 was shut down. Both exterior and interior locations of the fiberglass samples were examined. Environmental SEM (ESEM) was employed to analyze the hydrated fiberglass samples under a low vacuum condition (i.e., 80 Pa), and without any coating. This examination approach minimizes the modification of the fiberglass samples that can occur if samples are dried. The results of Test #5 Day-30 low-flow fiberglass samples were obtained on August 25, 2005. EDS results provide a semi-quantitative elemental analysis of the debris attached on fiberglass.

Transcribed Laboratory Log

Laboratory session from August 25, 2005.
Test #5 Day-30 Low-Flow Fiberglass.

ESEM



ESEM Low-Flow Exterior

Image:	t5d30lx3	100 ×	ESEM image	Figure C1-1
	t5d30lx4	500 ×	ESEM image higher magnification	Figure C1-2
	t5d30lx5	500 ×	ESEM image	Figure C1-3

ESEM Low-Flow Interior

Image:	t5d30lI6	100 ×	ESEM image of fiberglass	Figure C1-4
	t5d30li7	500 ×	ESEM image higher magnification	Figure C1-5

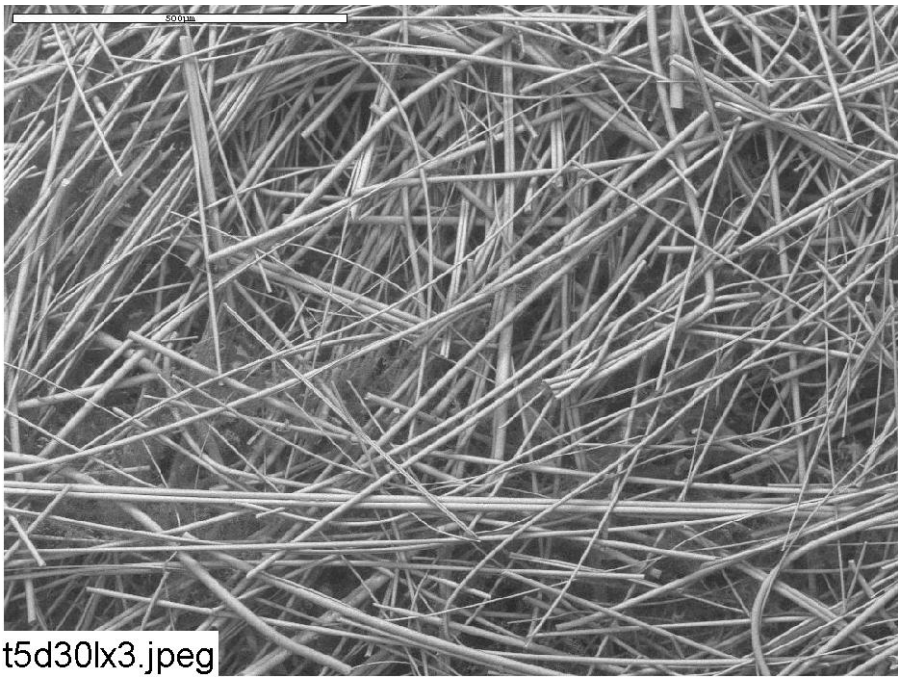


Figure C1-1: Environmental SEM image magnified 100 times for a Test #5 Day-30 low-flow exterior fiberglass sample. (t5d30lx3.jpg)

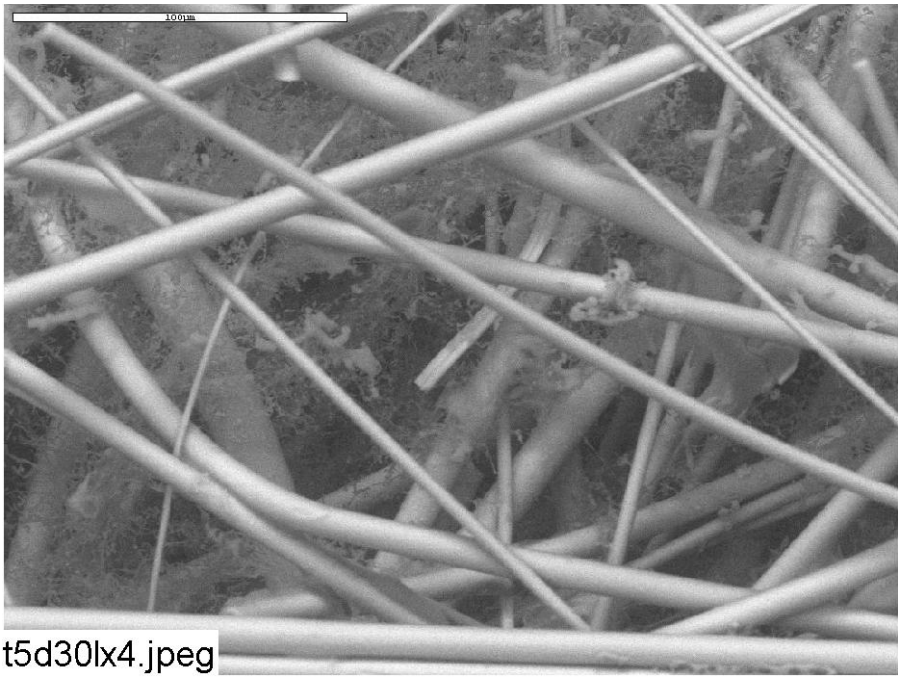


Figure C1-2: Environmental SEM image magnified 500 times for a Test #5 Day-30 low-flow exterior fiberglass sample. (t5d30lx4.jpg)

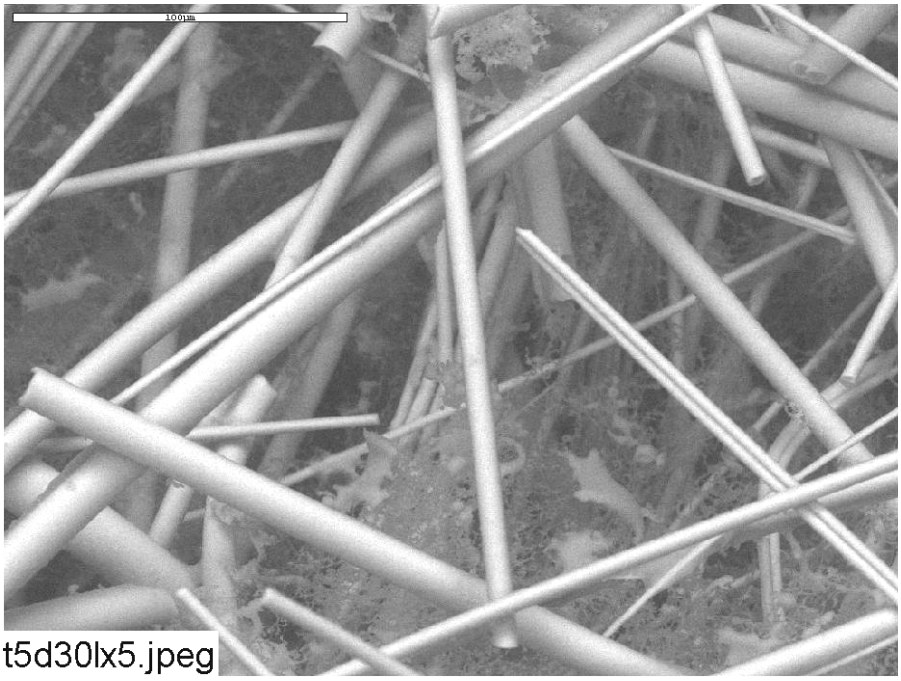


Figure C1-3: Environmental SEM image magnified 500 times for a Test #5 Day-30 low-flow exterior fiberglass sample. (t5d30lx5.jpg)

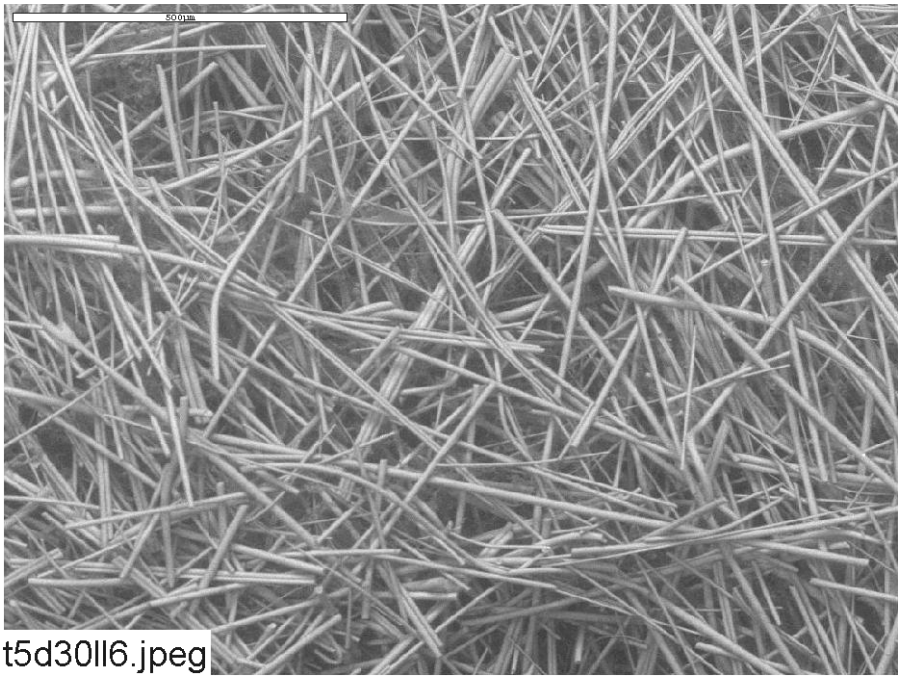


Figure C1-4: Environmental SEM image magnified 100 times for a Test #5 Day-30 low-flow interior fiberglass sample. (t5d30lI6.jpg)

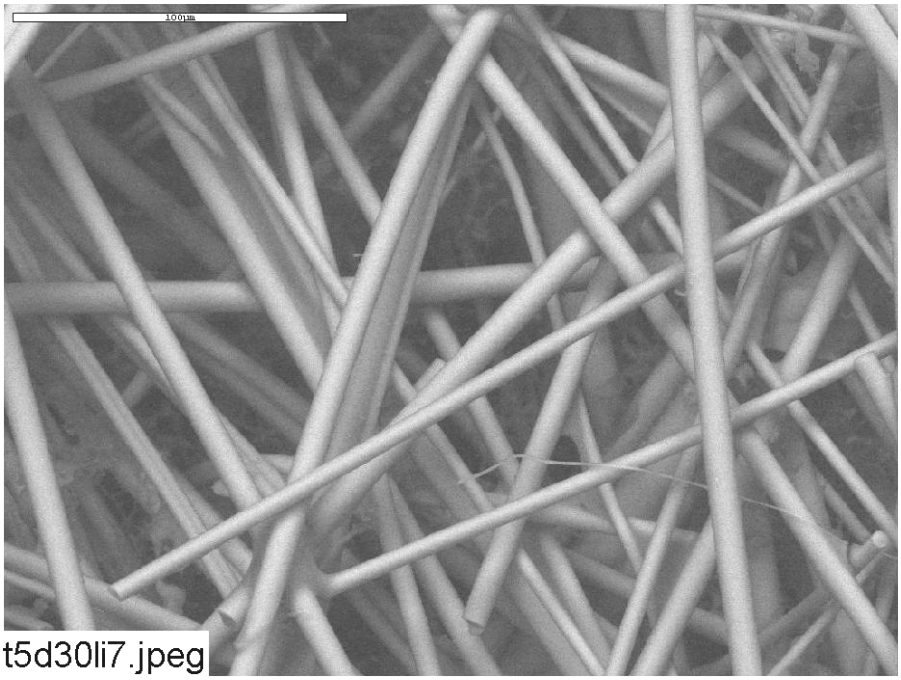


Figure C1-5: Environmental SEM image magnified 500 times for a Test #5 Day-30 low-flow interior fiberglass sample. (t5d30li7.jpg)

Appendix C2

ESEM/EDS Data for Test #5 Day-30 Fiberglass Samples in a Big Envelope in Low-Flow Zones

List of Figures

Figure C2-1: Environmental SEM image magnified 100 times for a Test #5 Day-30 low-flow exterior fiberglass sample in a big envelope. (T5enpx01.jpg) C2-4

Figure C2-2: Environmental SEM image magnified 500 times for a Test #5 Day-30 low-flow exterior fiberglass sample in a big envelope. (t5enpx02.jpg) C2-4

Figure C2-3: EDS counting spectrum for the particulate deposits between the fibers shown in Figure C2-2. (t5enpx03.jpg) C2-5

Figure C2-4: Environmental SEM image magnified 100 times for a Test #5 Day-30 low-flow interior fiberglass sample in a big envelope. (T5enpi04.jpg)..... C2-5

Figure C2-5: Environmental SEM image magnified 500 times for a Test #5 Day-30 low-flow interior fiberglass sample in a big envelope. (T5enpi05.jpg)..... C2-6

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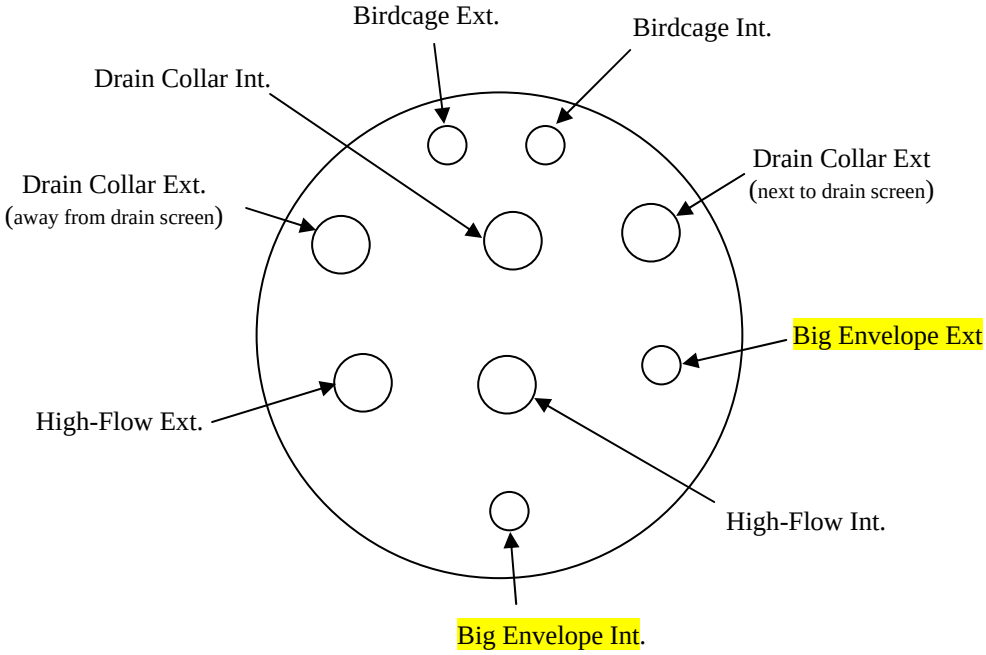
In this appendix, the fiberglass samples submerged in a big envelope in a low-flow zone of the test tank were extracted on the date Test #5 was shut down (August 25, 2005). Both exterior and interior locations of the fiberglass samples were examined. Environmental SEM (ESEM) was employed to analyze the hydrated fiberglass samples under a low vacuum condition (i.e., 80 Pa), and without any coating. This examination approach minimizes the modification of the fiberglass samples that can occur if samples are dried. The results of Test #5 Day-30 low-flow fiberglass samples in a big envelope were obtained on August 26, 2005. EDS results provide a semi-quantitative elemental analysis of the debris attached on fiberglass.

Transcribed Laboratory Log

Laboratory session from August 26, 2005.
Test #5 Day-30 Low-Flow Fiberglass Samples in a Big Envelope

ESEM



Big Envelope Exterior Low-Flow

Image:	T5enpx01	100 ×	ESEM image	Figure C2-1
	t5enpx02	500 ×	ESEM image higher magnification	Figure C2-2
EDS:	t5enpx03		EDS on particle in image t5enpx02	Figure C2-3

Big Envelope Interior Low-Flow

Image:	T5empi04	100 ×	ESEM image	Figure C2-4
	T5enpi05	500 ×	ESEM image higher magnification	Figure C2-5

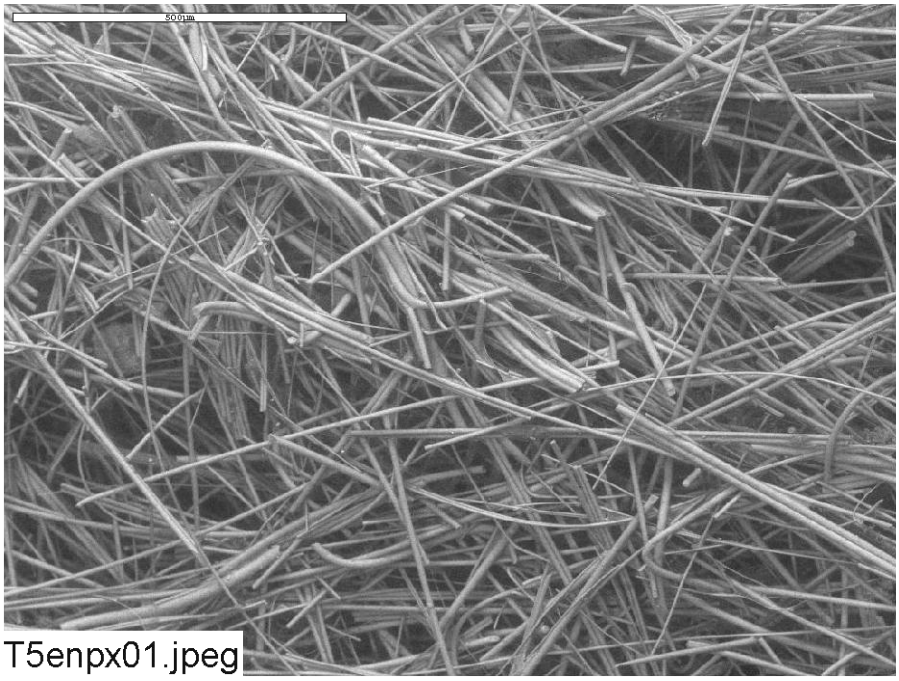


Figure C2-1: Environmental SEM image magnified 100 times for a Test #5 Day-30 low-flow exterior fiberglass sample in a big envelope. (T5enpx01.jpg)

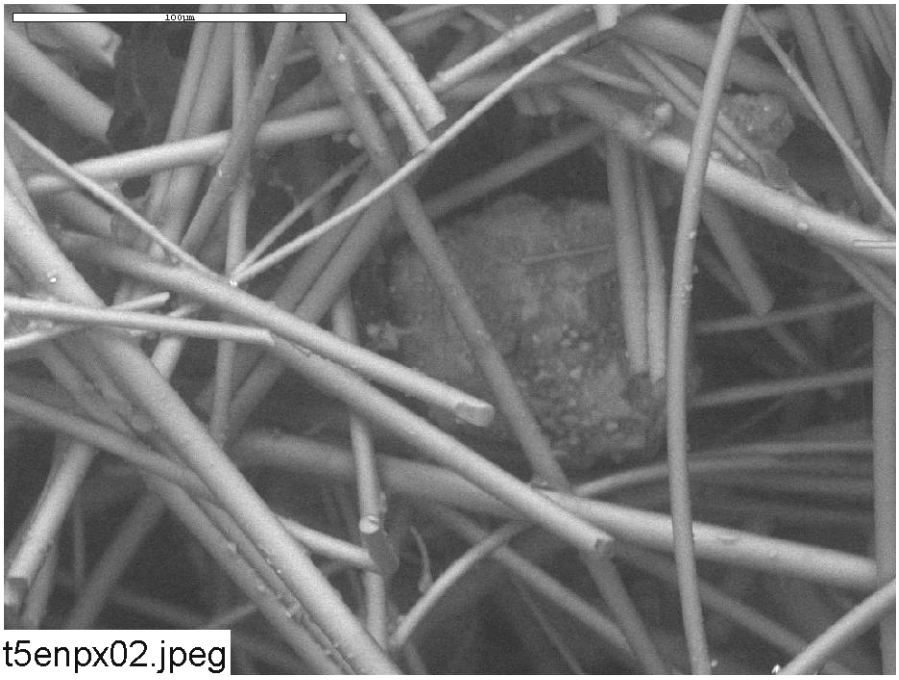


Figure C2-2: Environmental SEM image magnified 500 times for a Test #5 Day-30 low-flow exterior fiberglass sample in a big envelope. (t5enpx02.jpg)

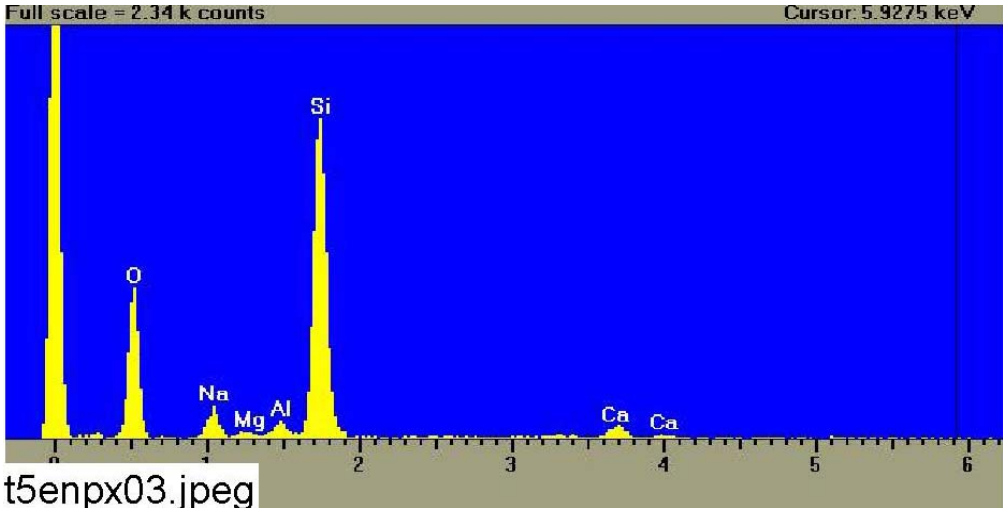


Figure C2-3: EDS counting spectrum for the particulate deposits between the fibers shown in Figure C2-2. (t5enpx03.jpg)

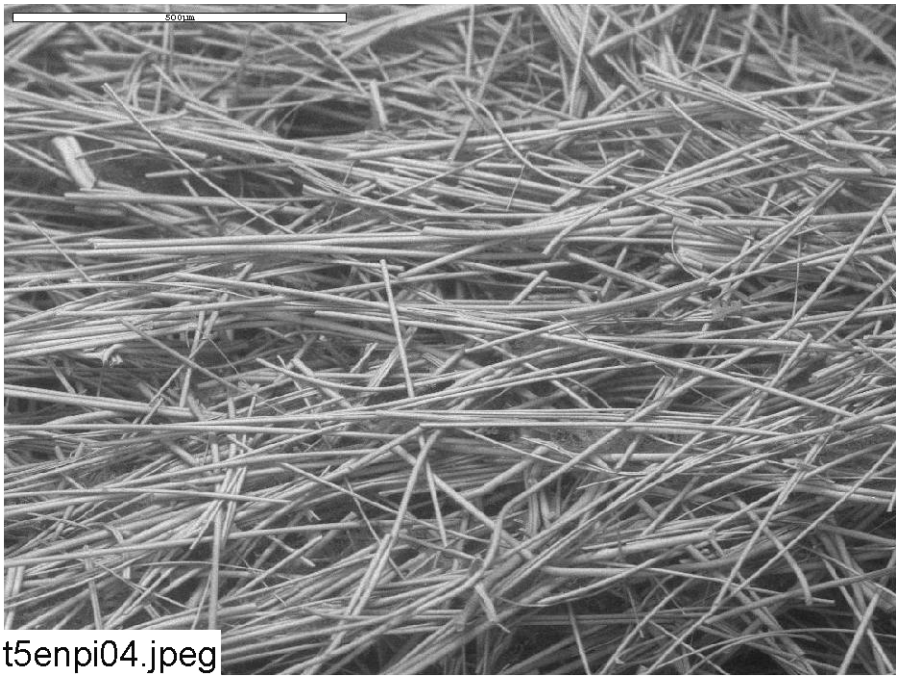


Figure C2-4: Environmental SEM image magnified 100 times for a Test #5 Day-30 low-flow interior fiberglass sample in a big envelope. (T5enpi04.jpg)

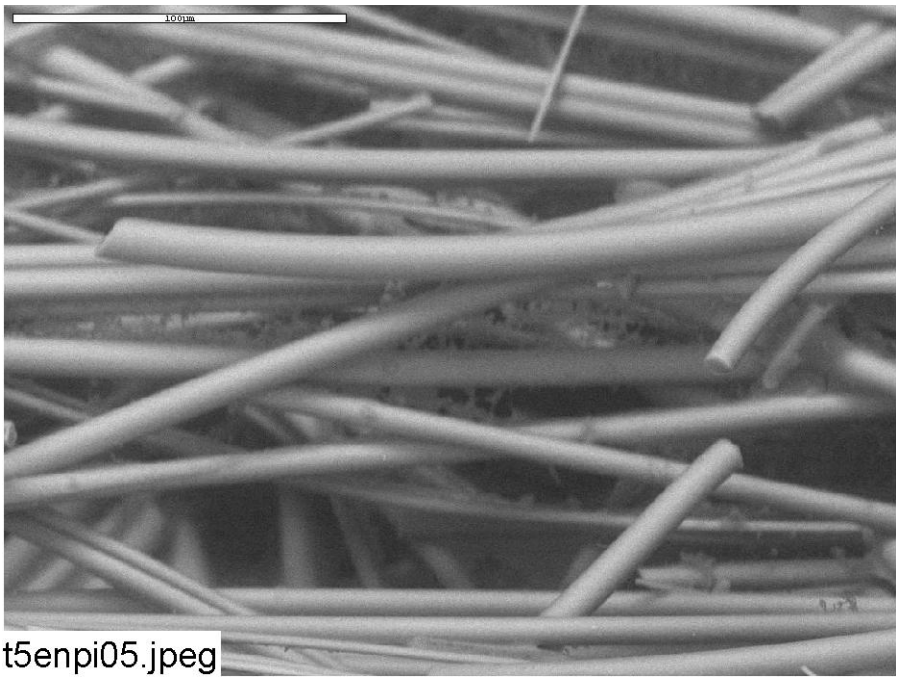


Figure C2-5: Environmental SEM image magnified 500 times for a Test #5 Day-30 low-flow interior fiberglass sample in a big envelope. (T5enpi05.jpg)

Appendix C3

ESEM Data for Test #5 Day-30 Fiberglass in High-Flow Zones

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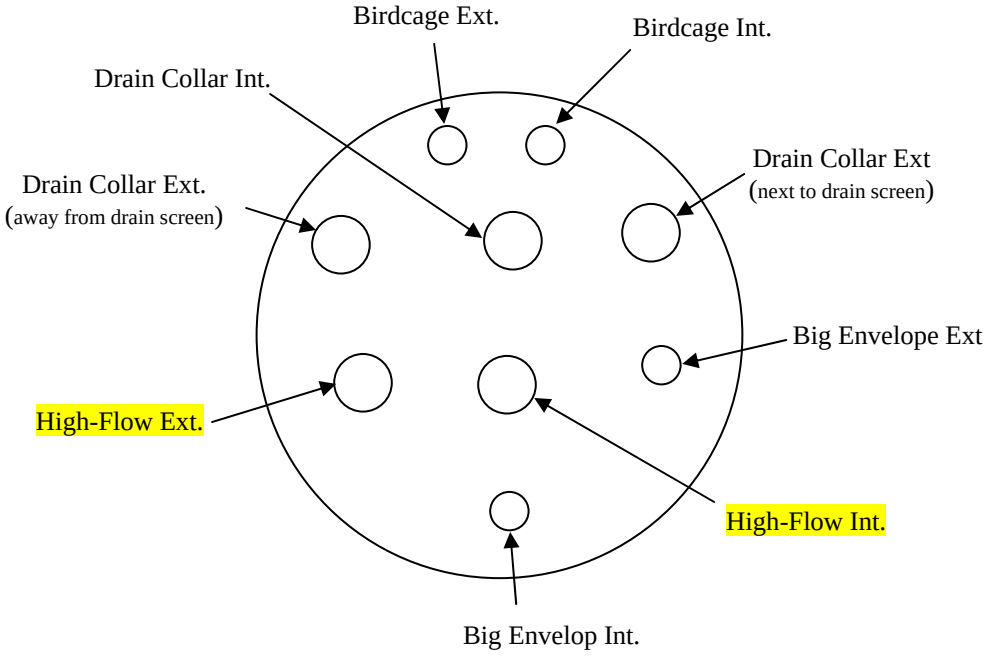
This appendix presents the fiberglass samples submerged in a high-flow zone, which were extracted on the date Test #5 was shut down (August 25, 2005). Both exterior and interior locations of the fiberglass samples were examined. Environmental SEM (ESEM) was employed to analyze the hydrated fiberglass samples under a low vacuum condition (i.e., 80 Pa), and without any coating. This examination approach minimizes the modification of the fiberglass samples that can occur if samples are dried. The results of Test #5 Day-30 high-flow fiberglass samples were obtained on August 26, 2005.

Transcribed Laboratory Log

Laboratory session from August 26, 2005.
Test #5 Day-30 High-Flow Fiberglass.

ESEM



Exterior High-Flow Fiberglass

Image:	T5D30HX1	100 ×	ESEM image	Figure C3-1
	t5d30hx2	100 ×	ESEM image	Figure C3-2
	t5d30hx3	500 ×	ESEM image higher magnification	Figure C3-3

Interior High-Flow Fiberglass

Image:	T5D30HI4	100 ×	ESEM image	Figure C3-4
	t5d30hi5	100 ×	ESEM image	Figure C3-5
	t5d30hi6	500 ×	ESEM image higher magnification	Figure C3-6

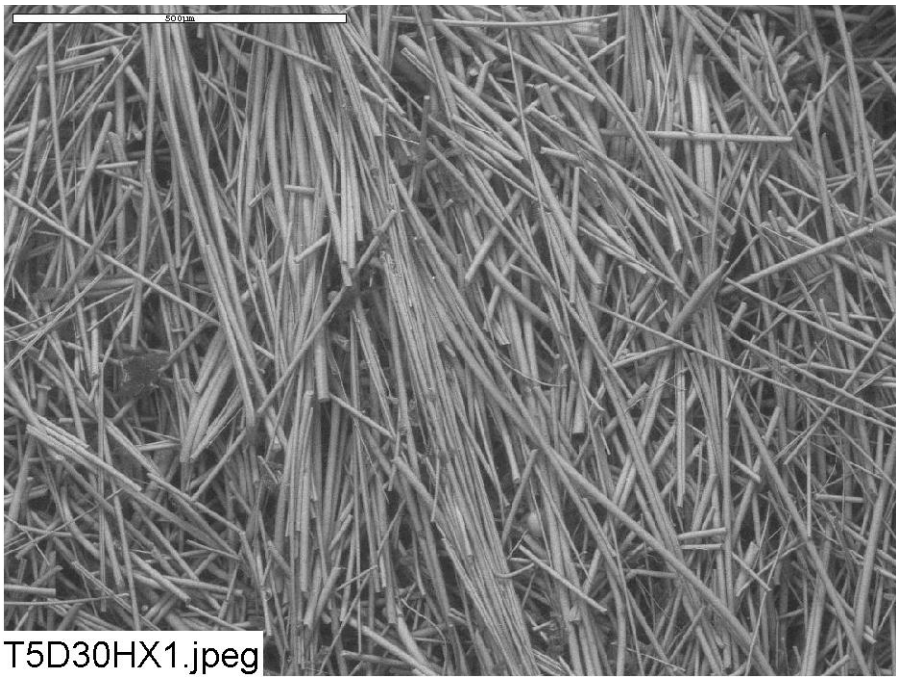


Figure C3-1: Environmental SEM image magnified 100 times for a Test #5 Day-30 high-flow exterior fiberglass sample. (T5D30HX1.jpg)

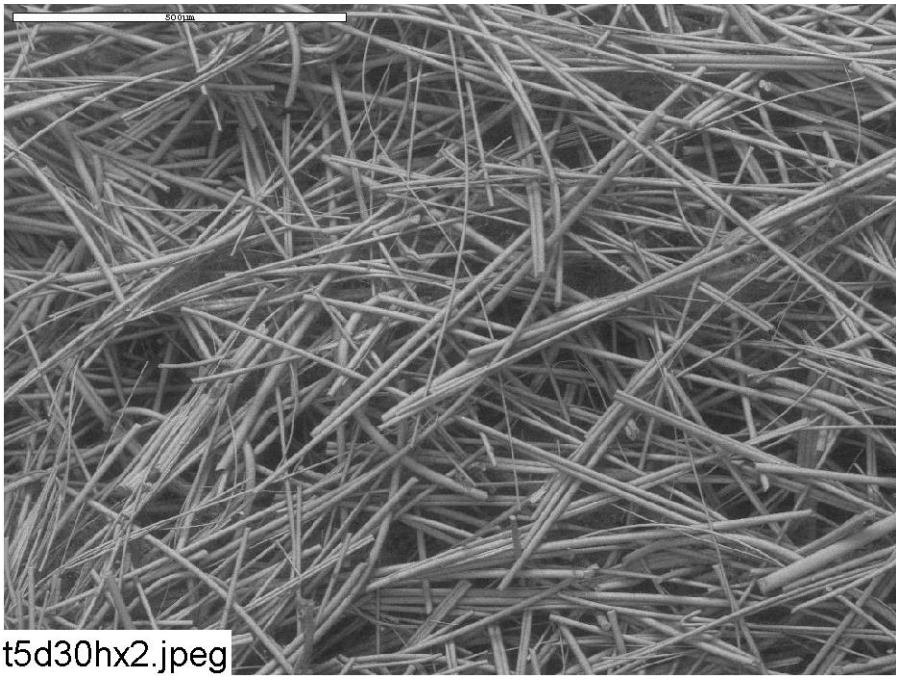


Figure C3-2: Environmental SEM image magnified 100 times for a Test #5 Day-30 high-flow exterior fiberglass sample. (t5d30hx2.jpg)

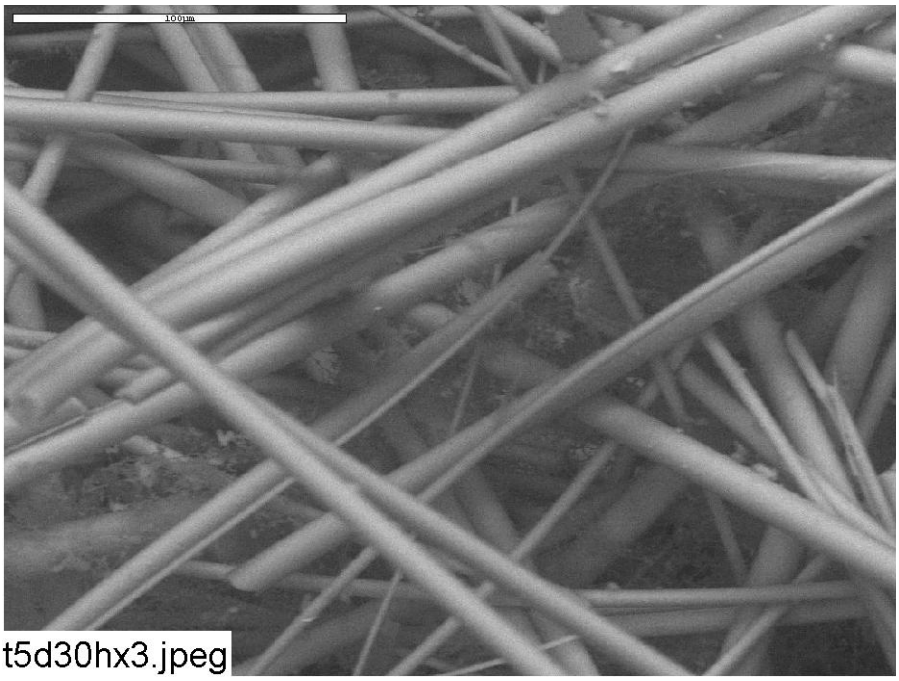


Figure C3-3: Environmental SEM image magnified 500 times for a Test #5 Day-30 high-flow exterior fiberglass sample. (t5d30hx3.jpg)

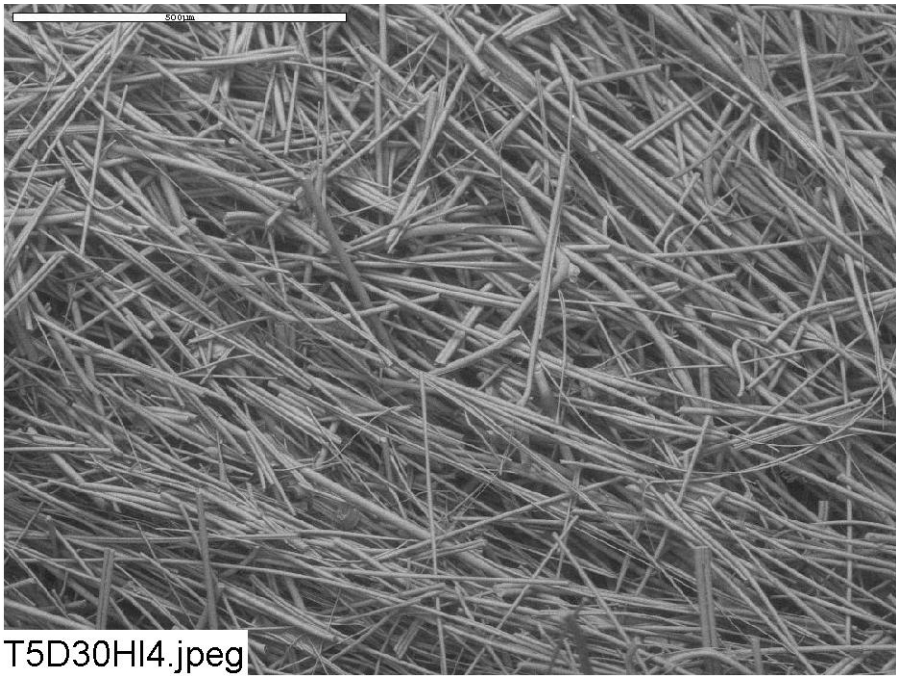


Figure C3-4: Environmental SEM image magnified 100 times for a Test #5 Day-30 high-flow interior fiberglass sample. (T5D30HI4.jpg)

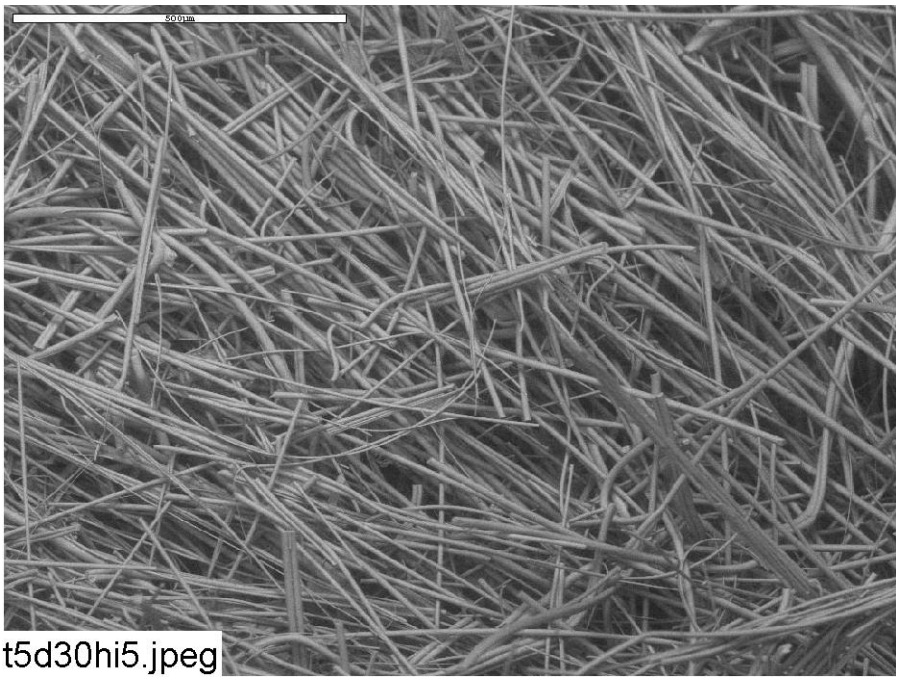


Figure C3-5: Environmental SEM image magnified 100 times for a Test #5 Day-30 high-flow interior fiberglass sample. (t5d30hi5.jpg)

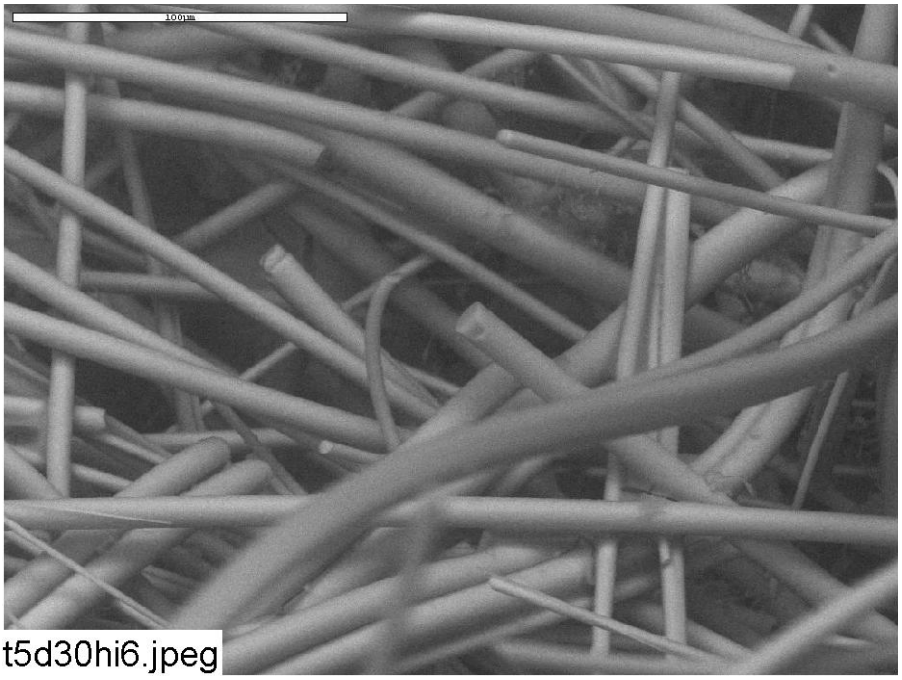


Figure C3-6: Environmental SEM image magnified 500 times for a Test #5 Day-30 high-flow interior fiberglass sample. (t5d30hi6.jpg)

Appendix C4

ESEM Data for Test #5 Day-30 Fiberglass Inserted in Front of a Header in a High-Flow Zone

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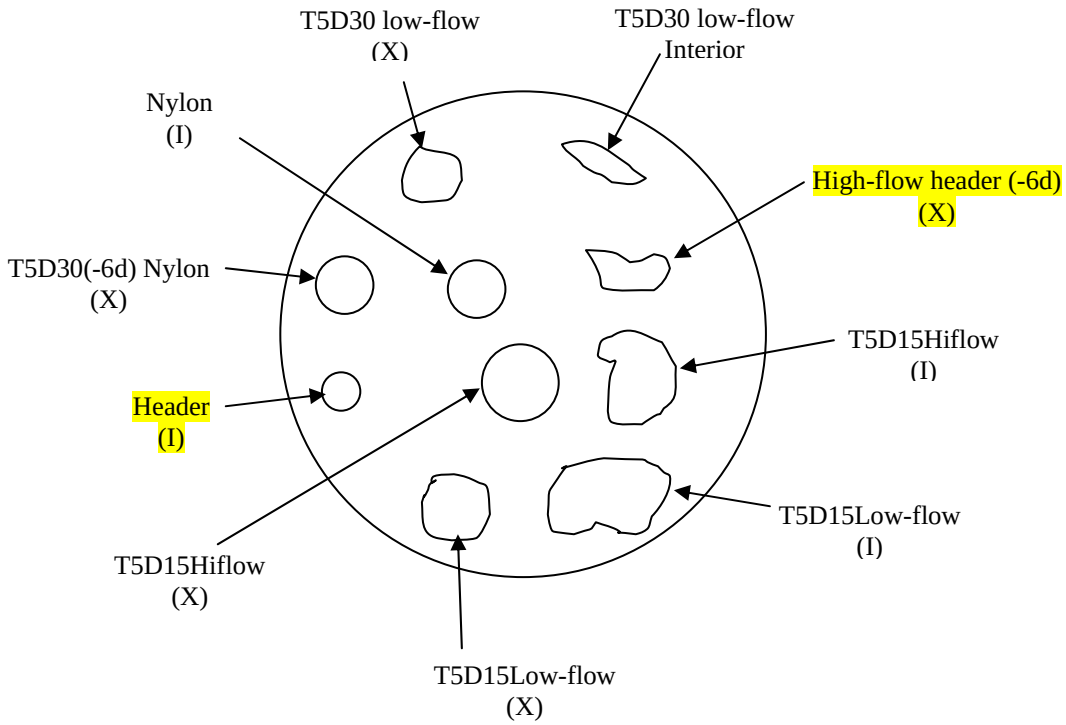
This appendix presents the results of the examinations performed on the high-flow fiberglass samples located in front of a test tank flow header. These samples were extracted on the date Test #5 was shut down (August 25, 2005). The samples were put in the tank on Day 6 of Test #5 (August 1, 2005). Both exterior and interior locations of the fiberglass samples were examined. Environmental SEM (ESEM) was employed to analyze the hydrated fiberglass samples under a low vacuum condition (i.e., 80 Pa), and without any coating. This examination approach minimizes the modification of the fiberglass samples that can occur if samples are dried. The results of Test #5 Day-30 high-flow fiberglass samples were obtained on August 25, 2005.

Transcribed Laboratory Log

Laboratory session from August 25, 2005.
Test #5 Day-30 Fiberglass in Front of Header in High-Flow.

ESEM



High-Flow Header (-6day) Exterior

Image:	T5HDEX01	100 ×	ESEM image	Figure C4-1
	t5hdex02	100 ×	ESEM image	Figure C4-2
	t5hdex03	500 ×	ESEM image higher magnification	Figure C4-3

High-Flow Header (-6day) Interior

Image:	t5hdin04	100 ×	ESEM image of fiberglass	Figure C4-4
	t5hdin05	500 ×	ESEM image higher magnification	Figure C4-5

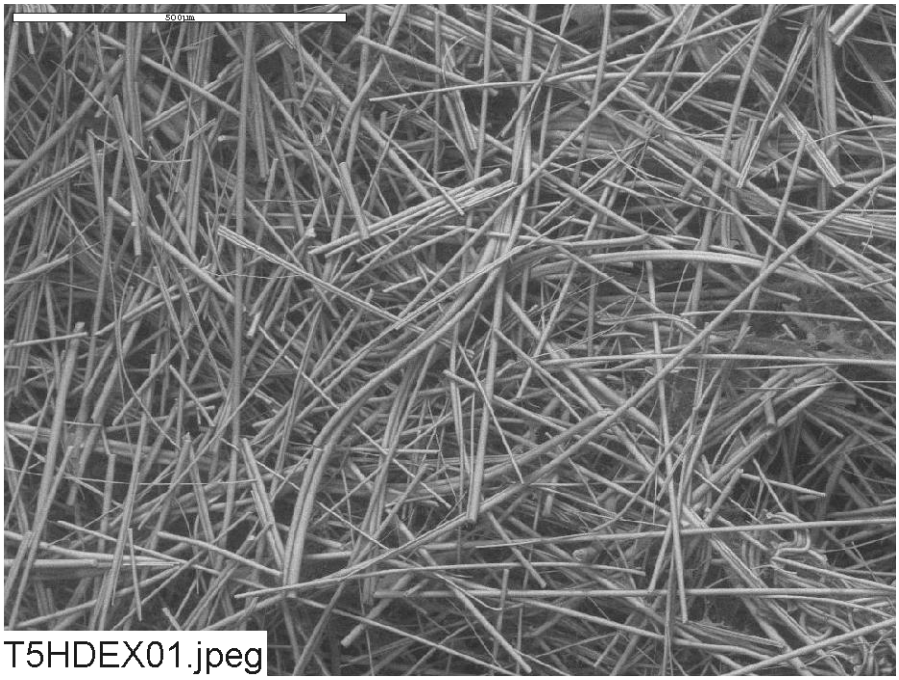


Figure C4-1: Environmental SEM image magnified 100 times for a Test #5 Day-30 high-flow exterior fiberglass sample in front of the header. The sample was put in the tank on Day 6. (T5HDEX01.jpg)

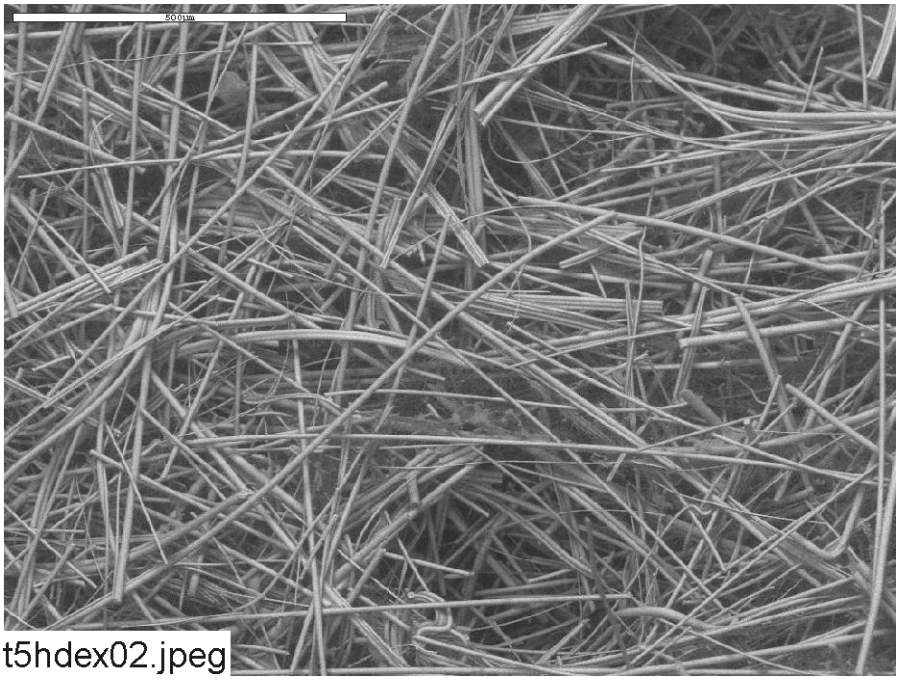


Figure C4-2: Environmental SEM image magnified 100 times for a Test #5 Day-30 high-flow exterior fiberglass sample in front of the header. The sample was put in the tank on Day 6. (t5hdex02.jpg)

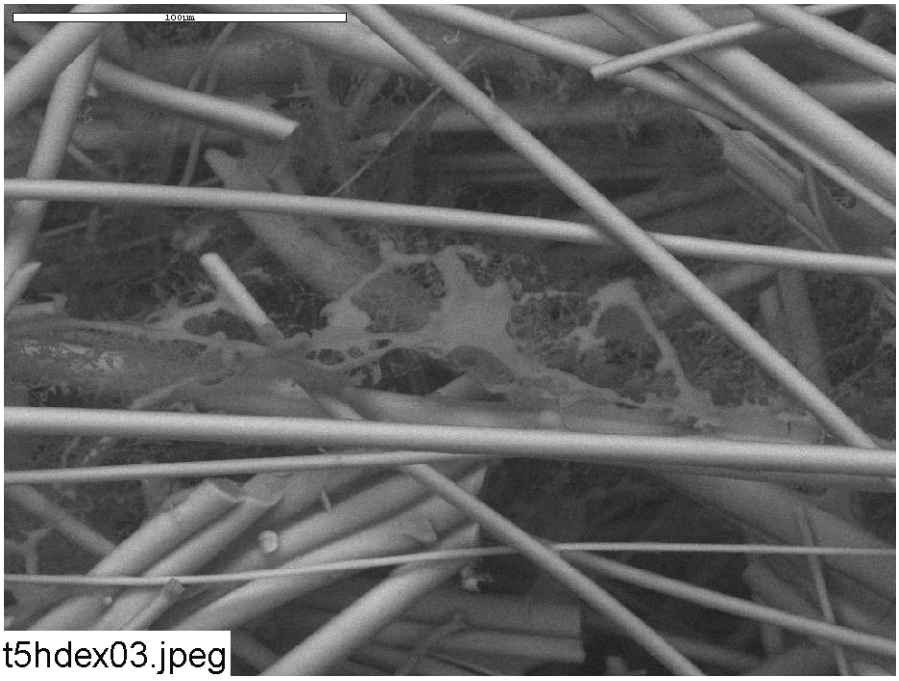


Figure C4-3: Environmental SEM image magnified 500 times for a Test #5 Day-30 high-flow exterior fiberglass sample in front of the header. The sample was put in the tank on Day 6. (t5hdex03.jpg)

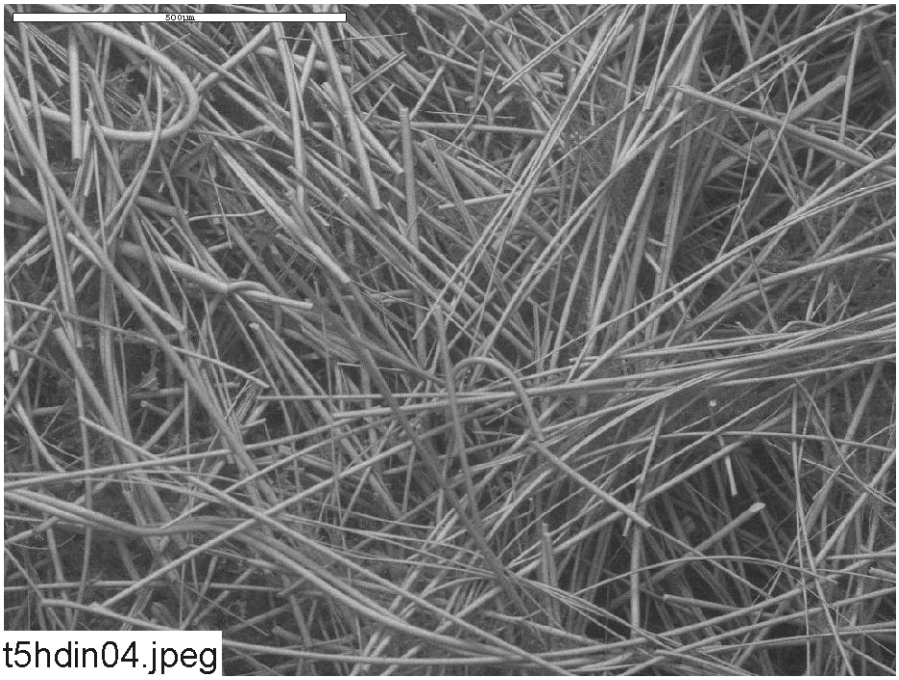


Figure C4-4: Environmental SEM image magnified 100 times for a Test #5 Day-30 high-flow interior fiberglass sample in front of the header. The sample was put in the tank on Day 6. (t5hdin04.jpg)

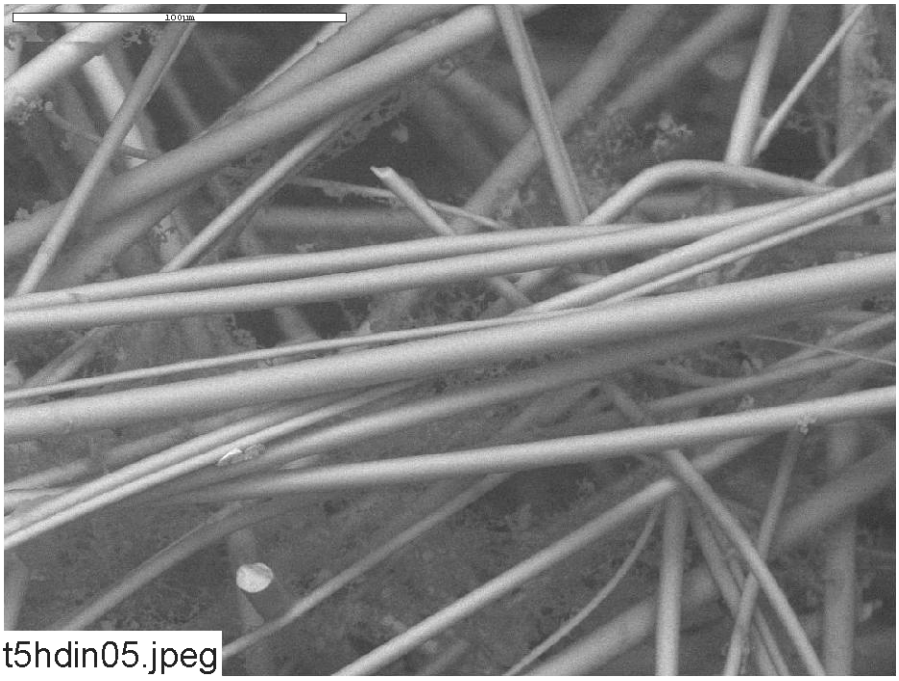


Figure C4-5: Environmental SEM image magnified 500 times for a Test #5 Day-30 high-flow interior fiberglass sample in front of the header. The sample was put in the tank on Day 6. (t5hdin05.jpg)

Appendix C5

ESEM/EDS Data for Test #5 Day-30 Fiberglass Inserted in Nylon Mesh in Low-Flow Zones

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Figure C5-5: EDS counting spectrum for the deposits between the fibers shown in Figure C5-4. (t5nlhfi5.jpg) C5-6

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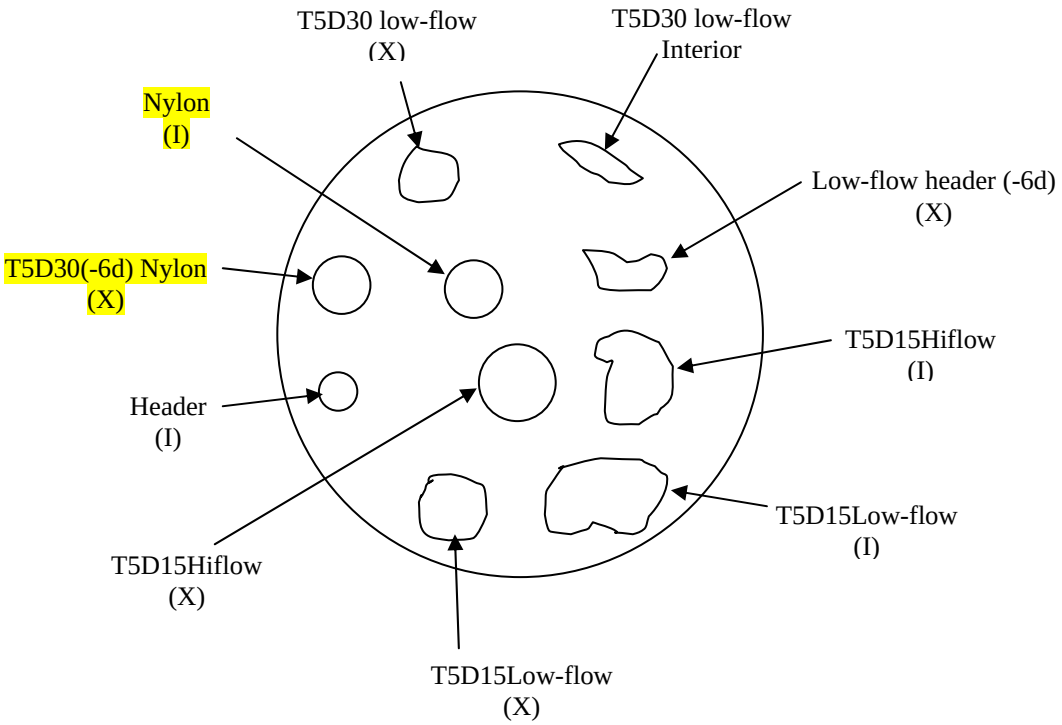
This appendix presents the ESEM results on fiberglass samples inserted in a nylon mesh submerged in a low-flow zone in the test tank. The purpose of using a nylon mesh is to see if the mesh material (i.e., stainless steel or nylon) affects the deposits on fiberglass samples. The samples were put in the tank on Day 6 (August 1, 2005). The fiberglass samples were extracted on August 25, 2005, the date Test #5 was shut down. Both exterior and interior locations of the fiberglass samples were examined. ESEM was employed to analyze the wet fiberglass samples without any coating and under a low vacuum condition (i.e., 80 Pa). This examination approach minimizes the modification of the fiberglass samples that can occur if samples are dried. The results of Test #5 Day-30 low-flow fiberglass samples in a nylon mesh were obtained on August 25, 2005.

Transcribed Laboratory Log

Laboratory session from August 25, 2005.
Test #5 Day-30 Nylon Mesh in Low-flow Zones

ESEM



Nylon Low-flow Exterior (-6days)

Image:	T5NLHFX1	100 ×	ESEM image	Figure C5-1
	t5nlhfx1	500 ×	ESEM image	Figure C5-2

Nylon Low-flow Interior (-6days)

Image:	t5nlhfi3	100 ×	ESEM image of fiberglass	Figure C5-3
	t5nlhfi4	500 ×	ESEM image higher magnification	Figure C5-4
EDS	t5nlhfi5		EDS on floc in image t5nlhfi4	Figure C5-5

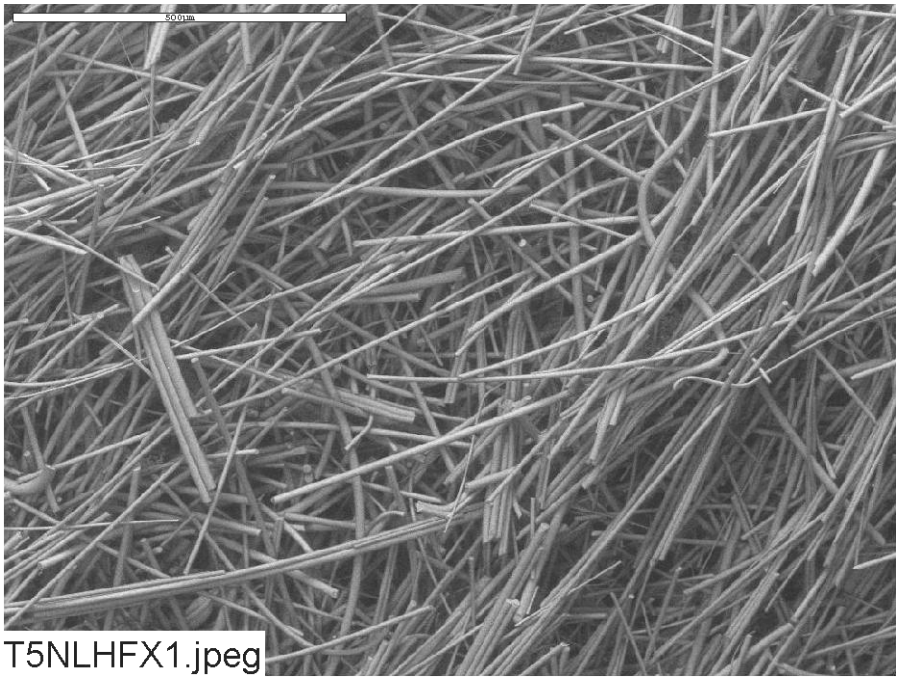


Figure C5-1: Environmental SEM image magnified 100 times for a Test #5 Day-30 low-flow exterior fiberglass sample in a nylon mesh. The sample was put in the tank on Day 6. (T5NLHFX1.jpg)

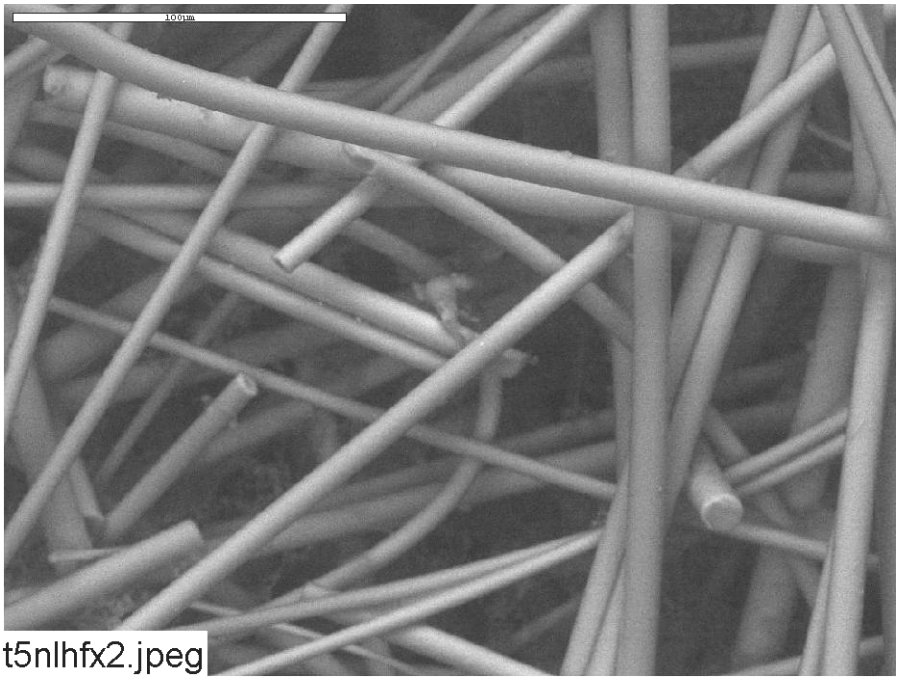


Figure C5-2: Environmental SEM image magnified 500 times for a Test #5 Day-30 low-flow exterior fiberglass sample in a nylon mesh. The sample was put in the tank on Day 6. (t5nlhfx2.jpeg)

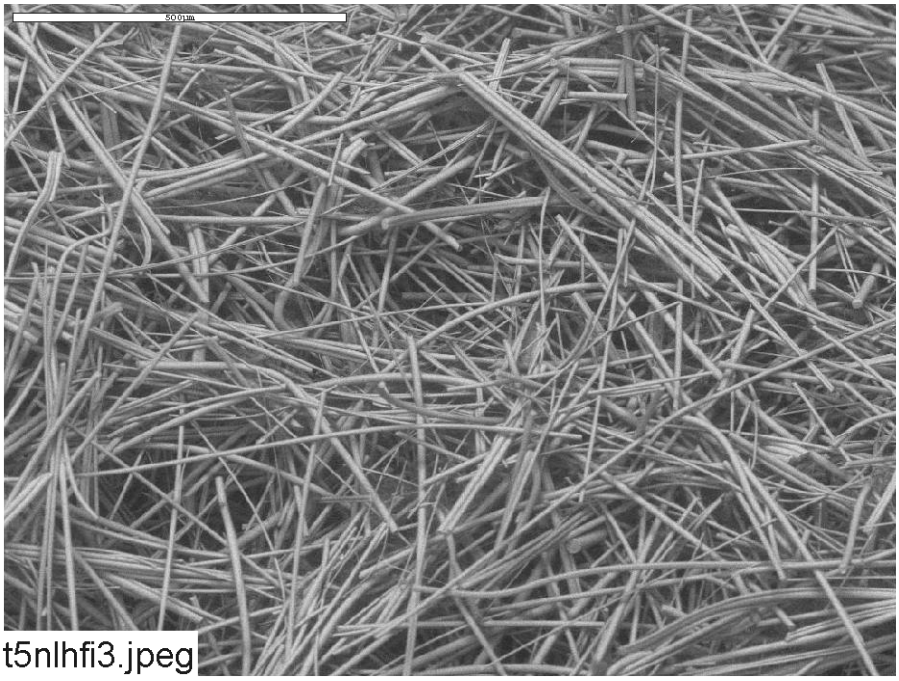


Figure C5-3: Environmental SEM image magnified 100 times for a Test #5 Day-30 low-flow interior fiberglass sample in a nylon mesh. The sample was put in the tank on Day 6. (t5nlhfi3.jpg)

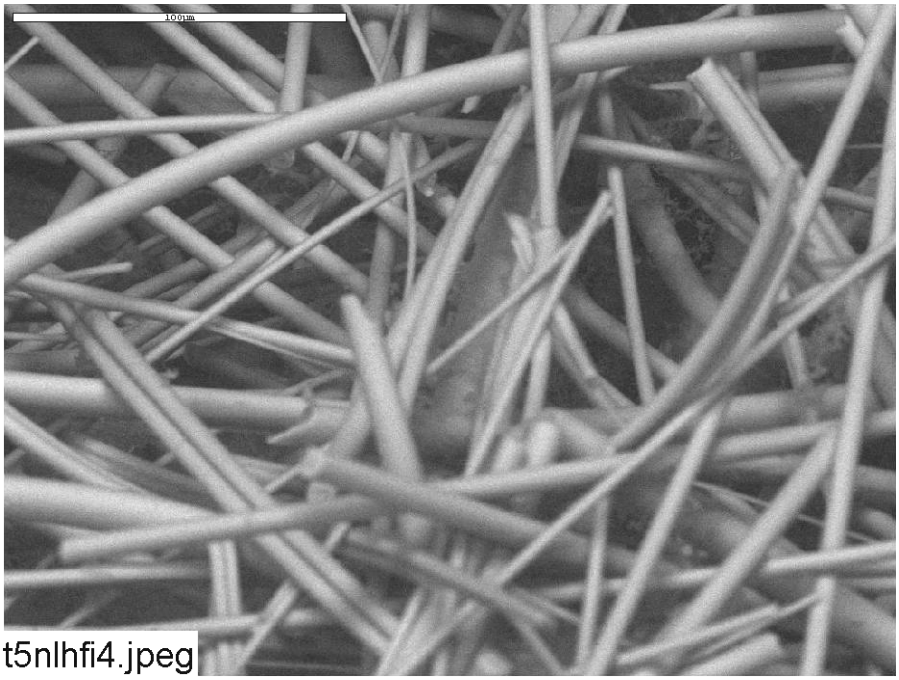


Figure C5-4: Environmental SEM image magnified 500 times for a Test #5 Day-30 low-flow interior fiberglass sample in a nylon mesh. The sample was put in the tank on Day 6. (t5nlhfi4.jpg)

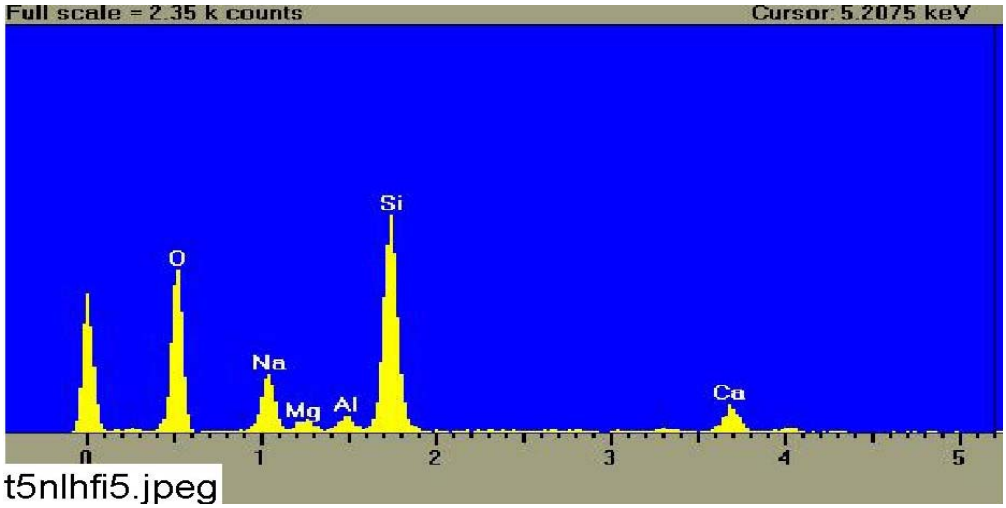


Figure C5-5: EDS counting spectrum for the deposits between the fibers shown in Figure C5-4. (t5nlhfi5.jpg)

Appendix C6

ESEM/EDS and SEM Data for Test #5 Day-30 Drain Collar Fiberglass

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Figure C6-9: Environmental SEM image magnified 100 times for a Test #5 Day-30 exterior drain collar fiberglass sample next to the drain screen. (t5dcix02.jpg)..... C6-10

Figure C6-10: Environmental SEM image magnified 500 times for a Test #5 Day-30 exterior drain collar fiberglass sample next to the drain screen. (t5dcix03.jpg)..... C6-10

Figure C6-11: EDS counting spectrum for the particulate deposits between fibers shown in Figure C6-10. (t5dcix04.jpg) C6-11

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Figure C6-13: Probe SEM image magnified 200 times for a Test #5 Day-30 interior drain collar fiberglass sample. (T5D30DraincollarInt027.bmp) C6-12
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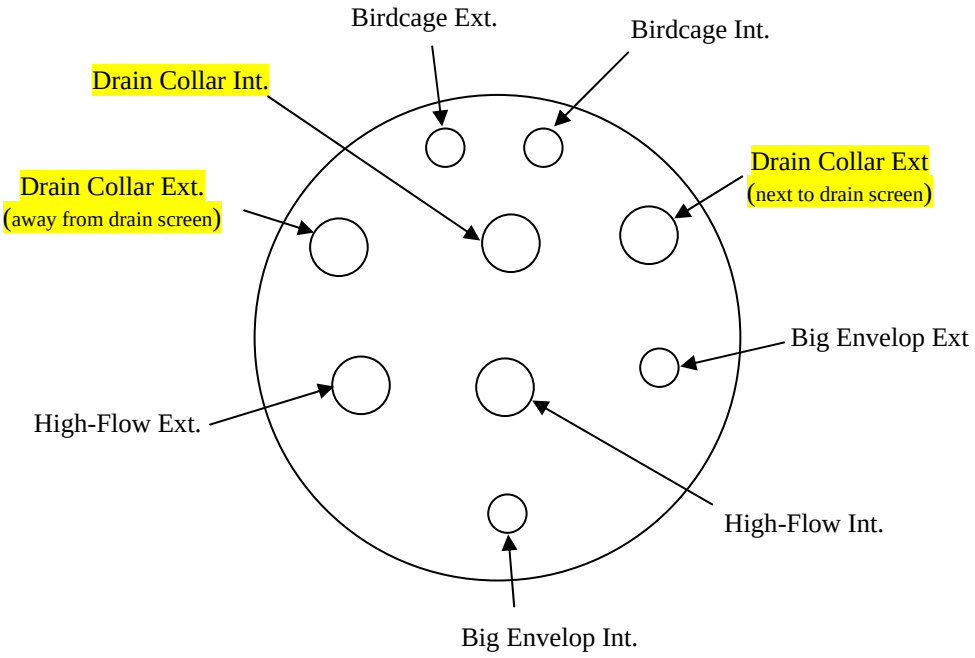
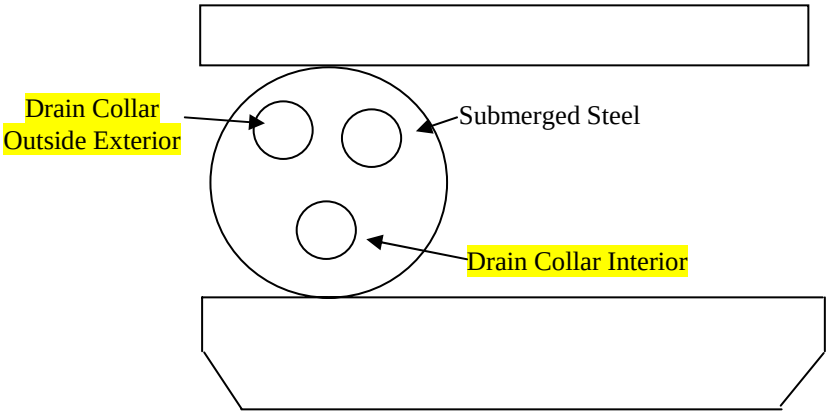
In this appendix, the fiberglass samples within the drain collar were extracted on the date Test #5 was shut down (August 25, 2005). The fiberglass samples located at the exterior farthest from the drain screen, the exterior next to the drain screen, and the interior were examined. Environmental SEM (ESEM) was employed to analyze the wet fiberglass samples under a low vacuum condition (i.e., 80 Pa) and without any coating. In addition to ESEM analysis, the samples were totally dried and coated with Au/Pd for probe SEM examination. ESEM/EDS results of the Test #5 Day-30 drain collar fiberglass samples were obtained on August 26, 2005, and probe SEM/EDS results were obtained on August 30, 2005 and September 6, 2005. EDS results provide a semi-quantitative elemental composition analysis of the debris attached on fiberglass.

Transcribed Laboratory Log

Laboratory session from August 26, 2005.
Test #5 Day-30 Drain Collar Fiberglass.

ESEM





Drain Collar Exterior (away from the drain screen)

Image:	T5DCXX01	100 ×	ESEM image	Figure C6-1
	t5dcxx02	100 ×	ESEM image	Figure C6-2
	t5dcxx03	500 ×	Annotated ESEM image	Figure C6-3
EDS:	t5dcxx04		EDS on particles in t5dcxx03	Figure C6-4
	t5dcxx05		EDS on particles in t5dcxx03	Figure C6-5
	T5D30Draincollar Particle16		EDS on particles on exterior of drain collar	Figure C6-6

Drain Collar Exterior (next to the drain screen)

Image:	t5dcix01	100 ×	ESEM image	Figure C6-7
	t5dcix05	100 ×	ESEM image	Figure C6-8
	t5dcix02	100 ×	ESEM image higher magnification	Figure C6-9
	t5dcix03	500 ×	ESEM image high magnification	Figure C6-10
EDS:	t5dcix04		EDS on particles image t5dcix03	Figure C6-11

Drain Collar Interior

Image:	t5dcii01	100 ×	ESEM image	Figure C6-12
	T5D30DraincollarInt027	200 ×	Probe SEM image	Figure C6-13
	t5dcii02	500 ×	ESEM image	Figure C6-14
	T5D30DraincollarInt026	1000 ×	Probe SEM image higher	Figure C6-15
EDS:	T5D30fiber floc17		EDS on particles in t4dcix08	Figure C6-16

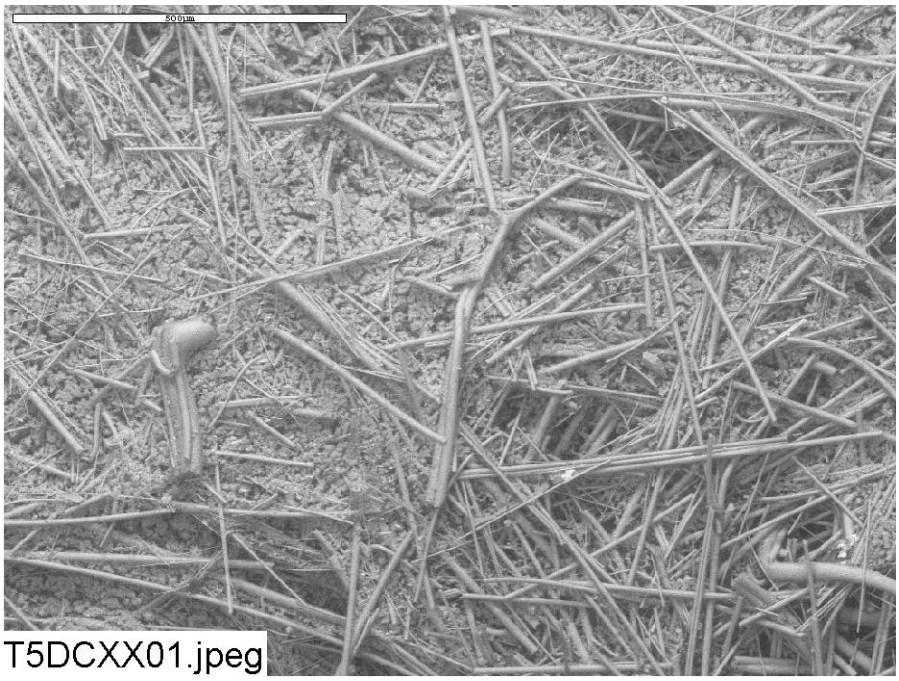


Figure C6-1: Environmental SEM image magnified 100 times for a Test #5 Day-30 exterior drain collar fiberglass sample farthest from the drain screen. (T5DCXX01.jpg)

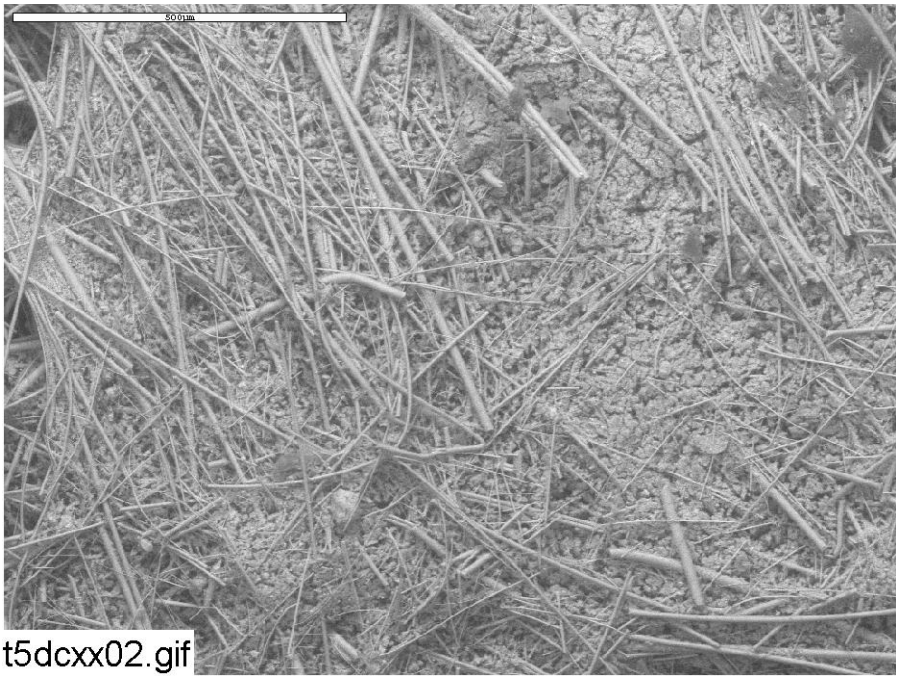


Figure C6-2: Environmental SEM image magnified 100 times for a Test #5 Day-30 exterior drain collar fiberglass sample farthest from the drain screen. (t5dcxx02.jpg)

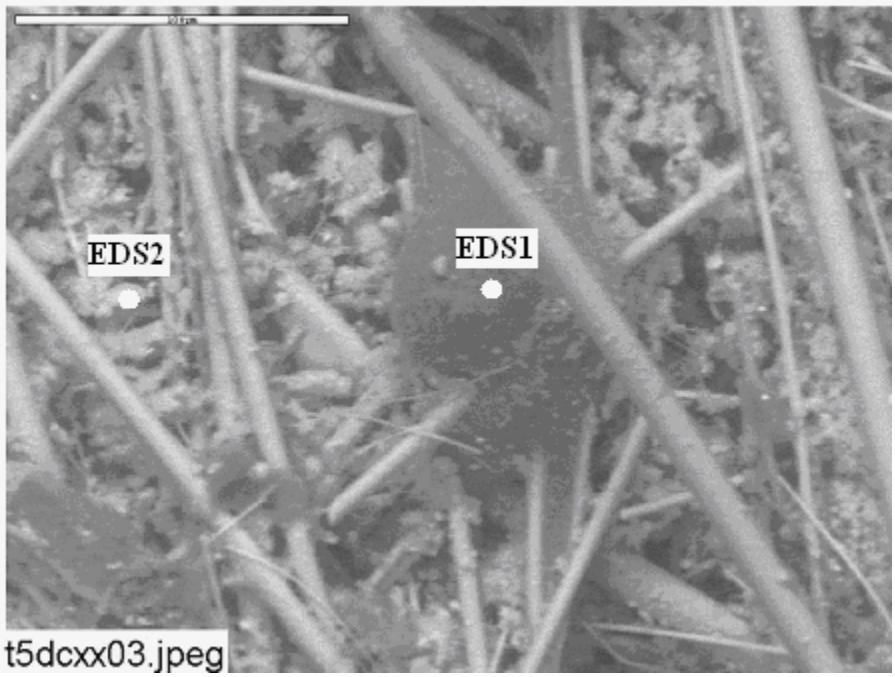


Figure C6-3: Annotated environmental SEM image magnified 500 times for a Test #5 Day-30 exterior drain collar fiberglass sample farthest from the drain screen. (t5dcxx03.jpg)

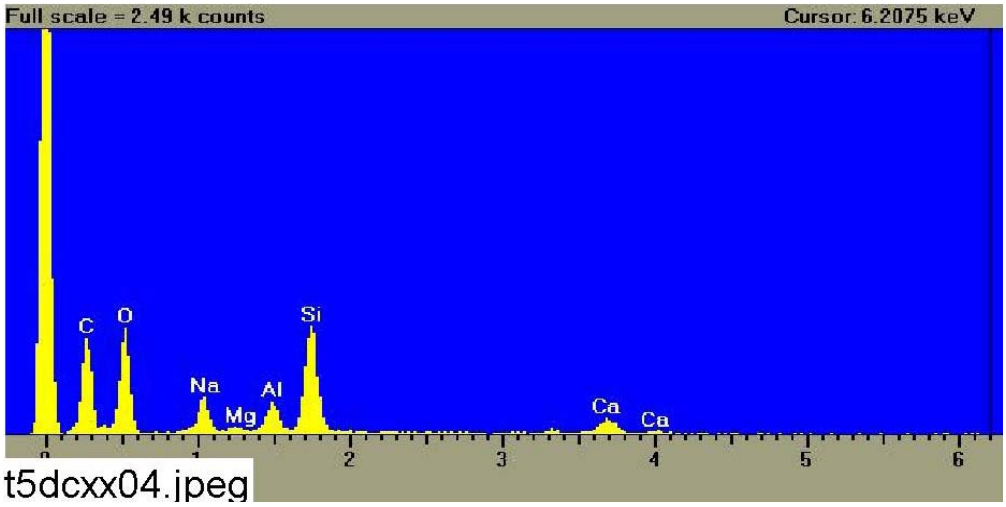


Figure C6-4: EDS counting spectrum for the dark deposits (EDS1) between fibers shown in Figure C6-3. (t5dcxx04.jpg)

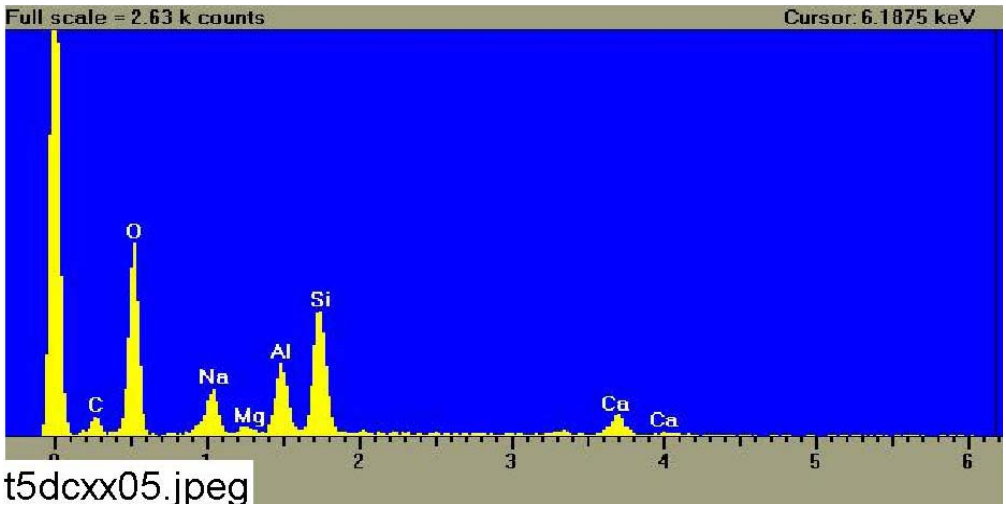


Figure C6-5: EDS counting spectrum for the small particulate deposits (EDS2) between fibers shown in Figure C6-3. (t5dcxx05.jpg)

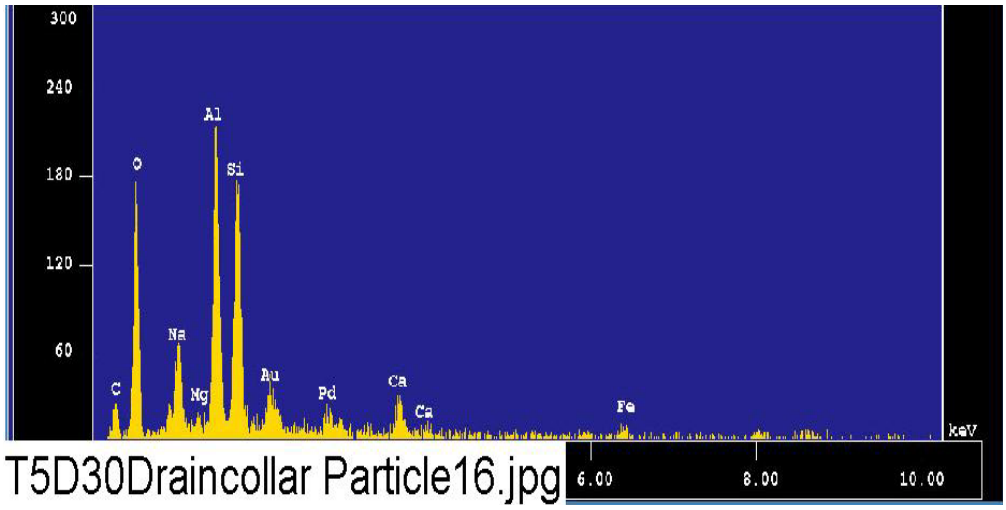


Figure C6-6: EDS counting spectrum by probe SEM for the small particulate deposits (EDS2) between fibers shown in Figure C6-3 (T5D30Draincollar Particle16.jpg)

The results from the chemical composition analysis for T5D30Draincollar Particle16.jpg are given in Table C6-1

Table C6-1. Chemical Compositions for T5D30Draincollar Particle16.jpg, Figure C6-6

```
Group     : NRC
Sample    : T5D30  ID# : 22
Comment   : Particles on Drain collar out exterior
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  72.190 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.003E-09 A
            Stage Point : X=76.455 Y=54.980 Z=11.000
            Acq. Date   : Tue Aug 30 18:08:20 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Na K      Normal   0.83- 1.28    0.3947   0.0041      351 /      16
 Al K      Normal   1.26- 1.78    1.2712   0.0008     1702 /      55
 Si K      Normal   1.50- 2.07    1.0873   0.0004     1598 /     110
 Ca K      Normal   3.40- 4.30    0.4688   0.0034      332 /       4
 Mg K      Normal   1.00- 1.53    0.1133   0.0002       85 /      66
  O K      Normal   0.31- 0.74    4.4301   0.0055     5013 /       7
  C K      Normal   0.11- 0.47   14.5489   0.0068      176 /      25
 Fe K      Normal   6.04- 7.40    0.4946   0.0012      158 /       2
-----------------------------------------------------------------
                            Chi_square =  2.8684

Element Mass%    Atomic%   ZAF      Z      A      F
    Na    2.761   1.9859 1.0916 0.9815 1.1113 1.0007
    Al    8.876   5.4401 1.0897 0.9899 1.1034 0.9977
    Si    8.579   5.0513 1.2314 0.9822 1.2538 0.9999
    Ca    2.914   1.2022 0.9700 0.9929 0.9778 0.9991
    Mg    1.039   0.7068 1.4318 0.9712 1.4778 0.9976
     O   42.666  44.1002 1.5030 0.9888 1.5200 1.0000
     C   29.326  40.3765 0.3146 0.9920 0.3171 1.0000
    Fe    3.840   1.1369 1.2115 1.2176 0.9950 1.0000
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  6.4077
```

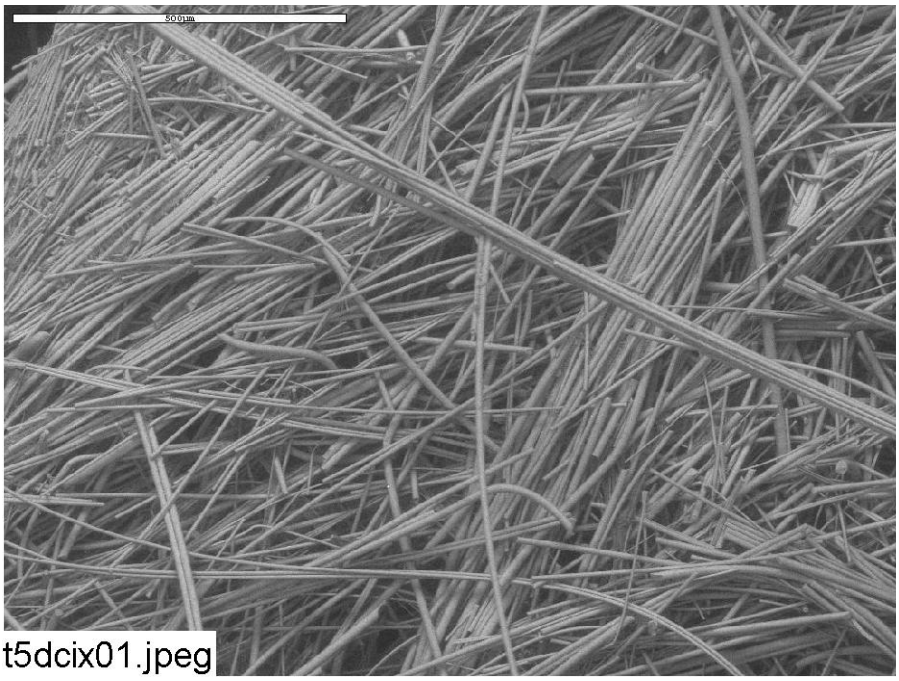


Figure C6-7: Environmental SEM image magnified 100 times for a Test #5 Day-30 exterior drain collar fiberglass sample next to the drain screen. (t5dcix01.jpg)

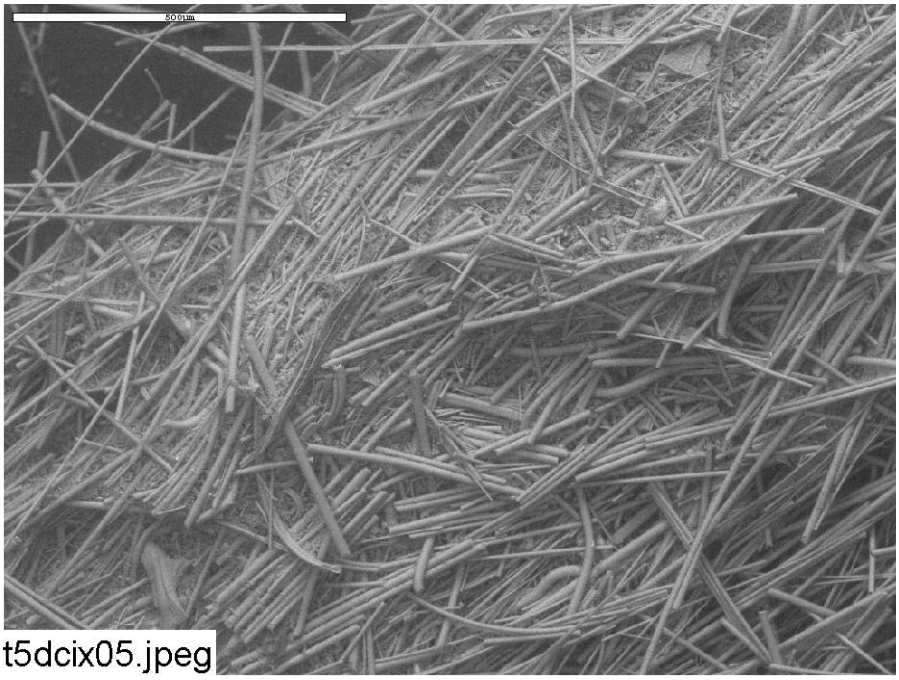


Figure C6-8: Environmental SEM image magnified 100 times for a Test #5 Day-30 exterior drain collar fiberglass sample next to the drain screen. (t5dcix05.jpg)

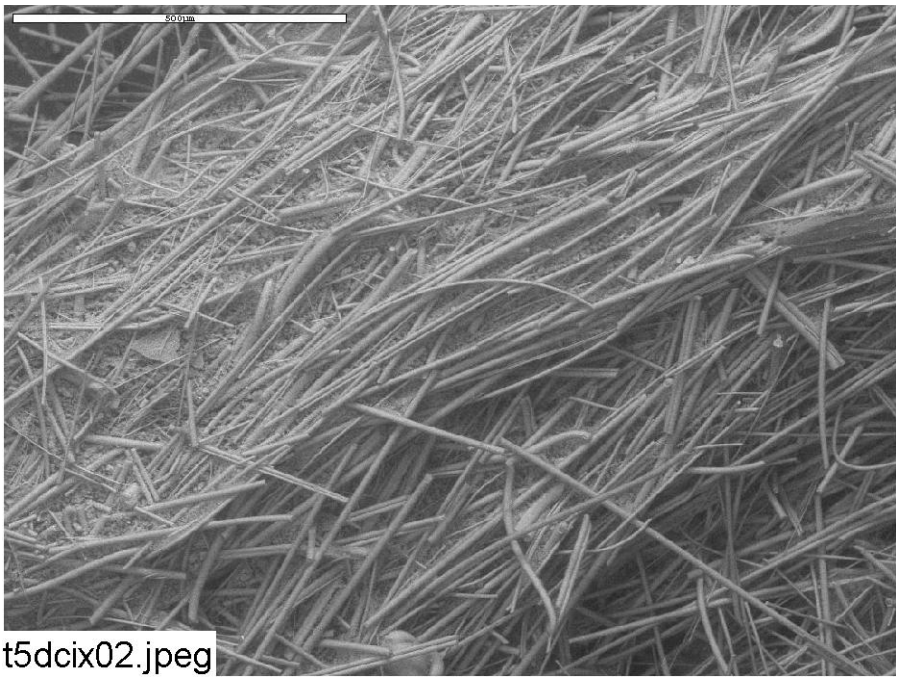


Figure C6-9: Environmental SEM image magnified 100 times for a Test #5 Day-30 exterior drain collar fiberglass sample next to the drain screen. (t5dcix02.jpg)

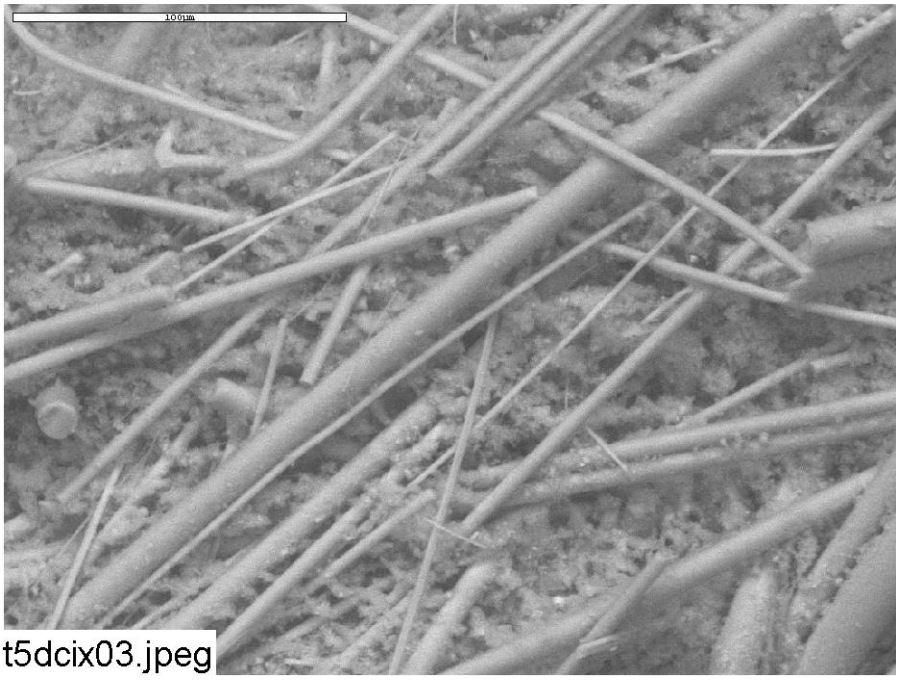


Figure C6-10: Environmental SEM image magnified 500 times for a Test #5 Day-30 exterior drain collar fiberglass sample next to the drain screen. (t5dcix03.jpg)

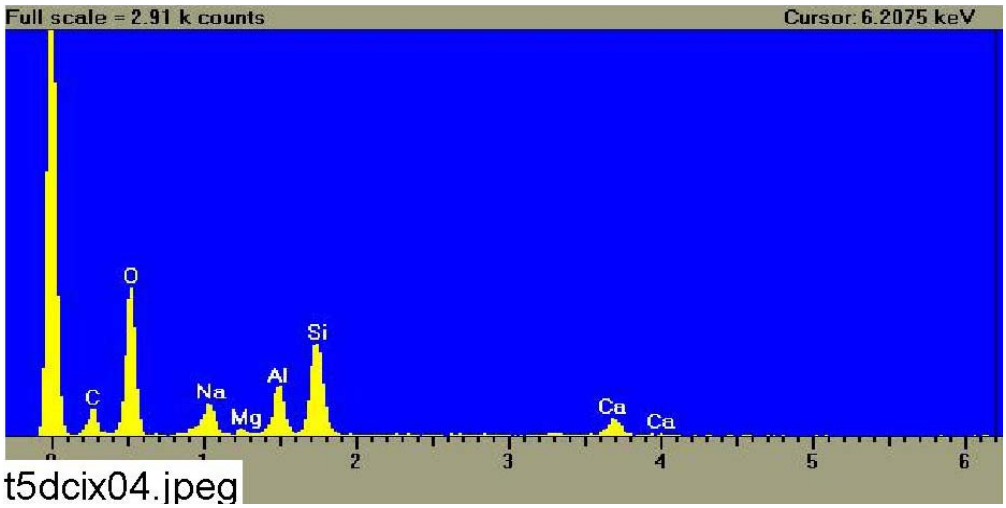


Figure C6-11: EDS counting spectrum for the particulate deposits between fibers shown in Figure C6-10. (t5dcix04.jpg)

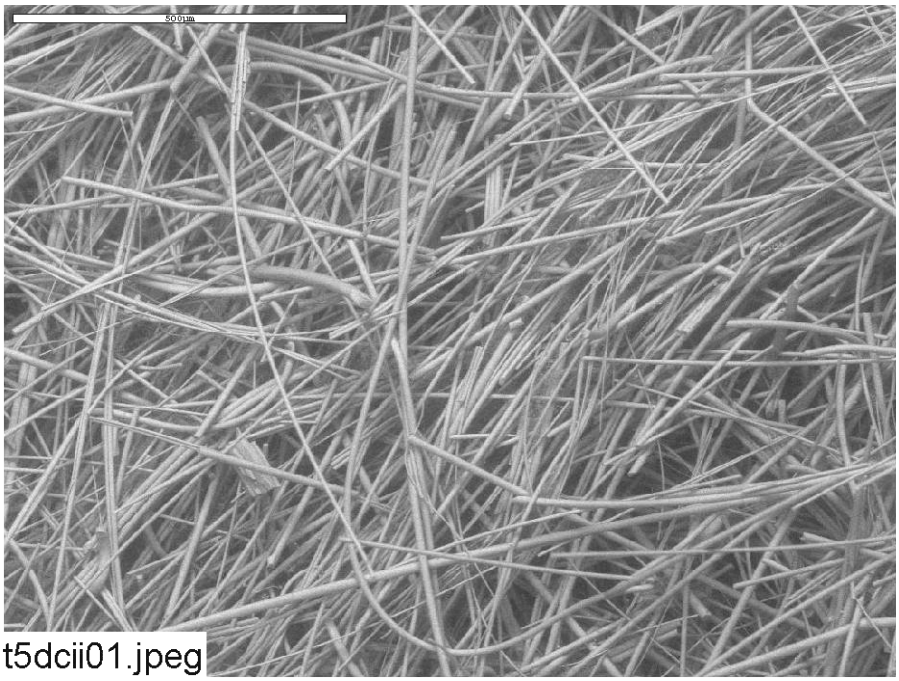


Figure C6-12: Environmental SEM image magnified 100 times for a Test #5 Day-30 interior drain collar fiberglass sample. (t5dcii01.jpg)

Figure C6-13: Probe SEM image magnified 200 times for a Test #5 Day-30 interior drain collar fiberglass sample. (T5D30DraincollarInt027.bmp)

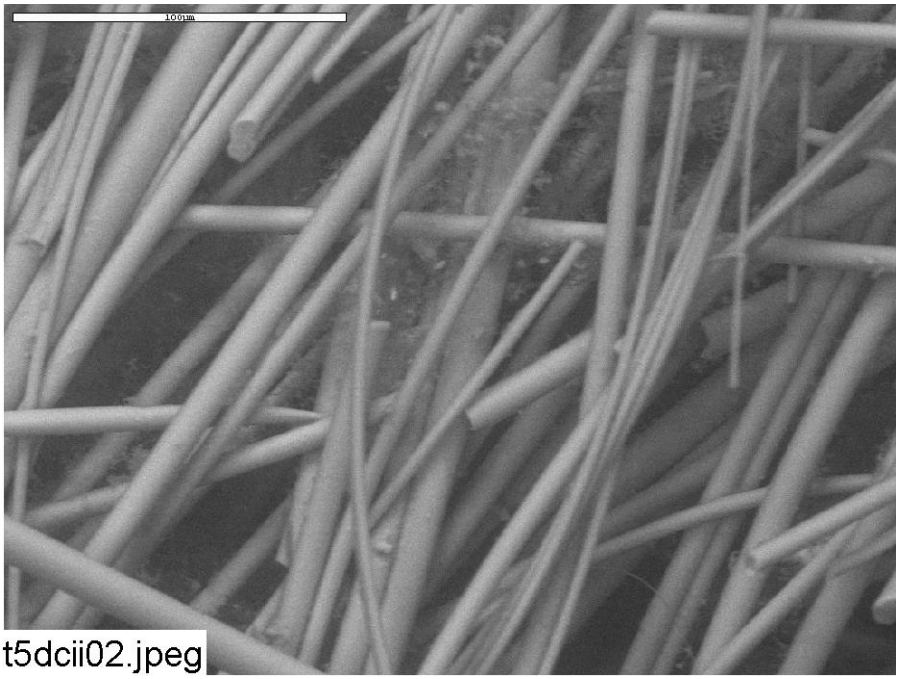


Figure C6-14: Environmental SEM image magnified 500 times for a Test #5 Day-30 interior drain collar fiberglass sample. (t5dcii02.jpg)

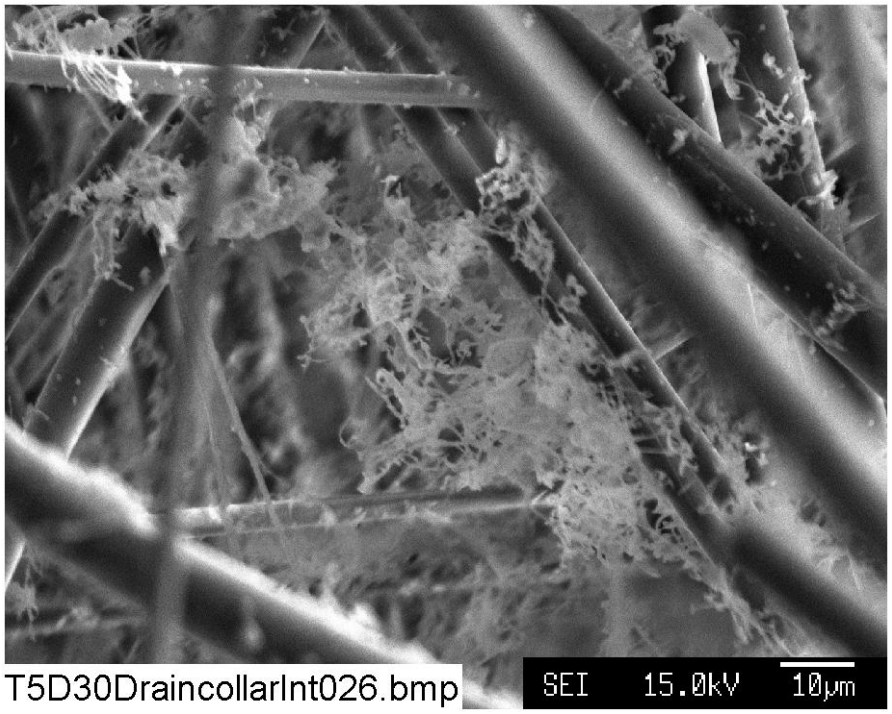


Figure C6-15: Probe SEM image magnified 1000 times for a Test #5 Day-30 interior drain collar fiberglass sample. (T5D30DraincollarInt026.bmp)

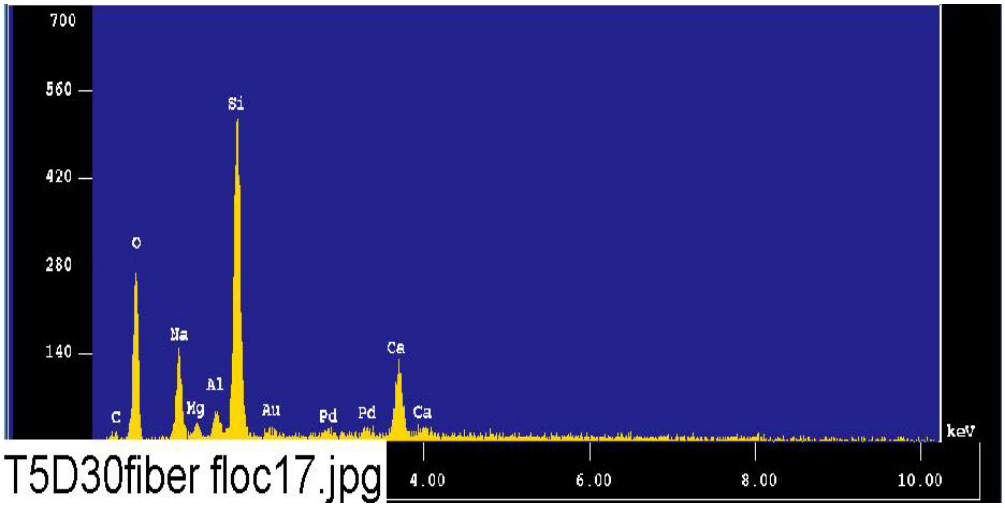


Figure C6-16: EDS counting spectrum by probe SEM for the flocculence deposits between fibers shown in Figure C6-15. (T5D30fiber floc17.jpg)

The results from the chemical composition analysis for T5D30fiber floc17.jpg are given in Figure C6-16

Table C6-2. Chemical Compositions for T5D30fiber floc17.jpg, Figure C6-16

```
Group     : NRC
Sample    : T5D30  ID# : 23
Comment   : Floculence on draincollar interior
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  96.780 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.003E-09 A
            Stage Point : X=76.455 Y=54.980 Z=11.000
            Acq. Date   : Tue Aug 30 18:19:56 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Na K      Normal   0.83- 1.28    0.9051   0.0053     1078 /      18
 Mg K      Normal   1.00- 1.53    0.1687   0.0003      171 /      53
 Al K      Normal   1.26- 1.78    0.1798   0.0004      323 /     152
 Si K      Normal   1.50- 2.07    2.4443   0.0007     4815 /      36
 Ca K      Normal   3.40- 4.30    1.4547   0.0064     1380 /      14
  O K      Normal   0.31- 0.74    5.4142   0.0068     8214 /       4
  C K      Normal   0.11- 0.47    3.3646   0.0061       55 /      46
-------------------------------------------------------------------
                            Chi_square =  3.5353

Element Mass%    Atomic%   ZAF      Z      A      F
    Na    6.733   5.4856 1.0942 0.9931 1.1011 1.0007
    Mg    1.737   1.3379 1.5143 0.9825 1.5445 0.9979
    Al    1.396   0.9688 1.1419 1.0012 1.1470 0.9944
    Si   19.786  13.1947 1.1906 0.9932 1.1990 0.9998
    Ca    9.803   4.5812 0.9912 1.0027 0.9884 1.0001
     O   51.409  60.1836 1.3966 1.0008 1.3954 1.0000
     C    9.137  14.2483 0.3994 1.0042 0.3978 0.9999
-------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  6.7987
```

Appendix C7

ESEM/EDS Data for Test #5 Day-30 Birdcage Fiberglass

List of Figures

Figure C7-1: Environmental SEM image magnified 100 times for a Test #5 Day-30 exterior fiberglass sample within the birdcage. (T5BCX01.jpg)..... C7-4

Figure C7-2: Environmental SEM image magnified 100 times for a Test #5 Day-30 exterior fiberglass sample within the birdcage. (t5bcx02.jpg) C7-4

Figure C7-3: Environmental SEM image magnified 500 times for a Test #5 Day-30 exterior fiberglass sample within the birdcage. (t5bcx03.jpg) C7-5

Figure C7-4: EDS counting spectrum for the deposits between fibers shown in Figure C7-3. (t5bcx04.jpg)..... C7-5

Figure C7-5: Environmental SEM image magnified 500 times for a Test #5 Day-30 exterior fiberglass sample within the birdcage. (t5bcx05.jpg) C7-6

Figure C7-6: Environmental SEM image magnified 100 times for a Test #5 Day-30 interior fiberglass sample within the birdcage. (t5bcI01.jpg)..... C7-6

Figure C7-7: Environmental SEM image magnified 100 times for a Test #5 Day-30 interior fiberglass sample within the birdcage. (t5bci03.jpg) C7-7

Figure C7-8: Environmental SEM image magnified 500 times for a Test #5 Day-30 interior fiberglass sample within the birdcage. (t5bci02.jpg) C7-7

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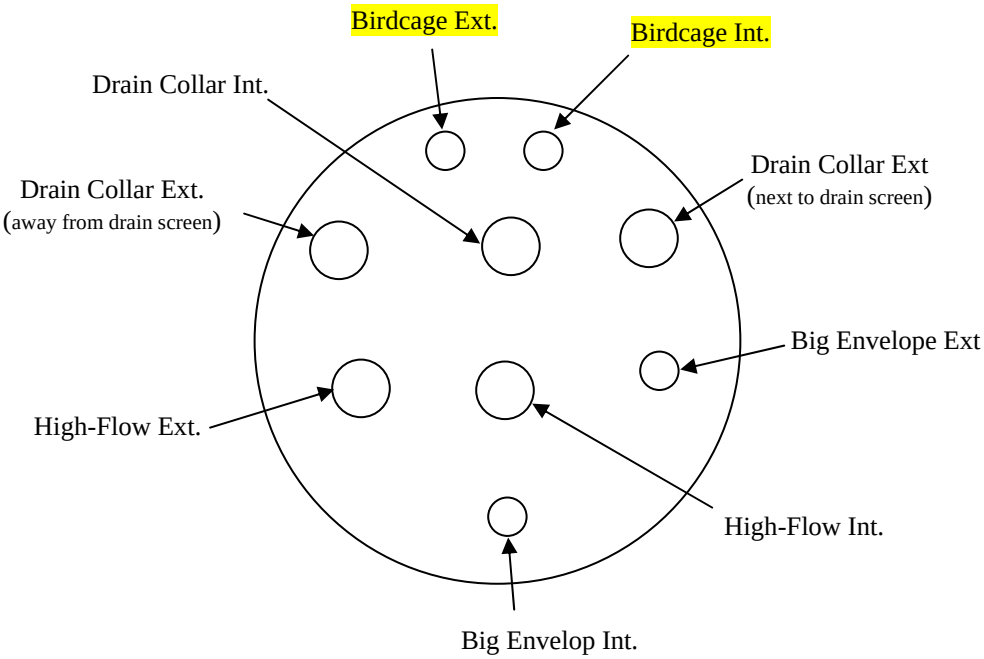
This appendix lists the ESEM/EDS results for the fiberglass samples within the birdcage submerged in the testing solution. The purpose of this analysis was to determine the degree and the extent to which particulate debris migrates and attaches to fiberglass. In this appendix, the fiberglass samples within the birdcage were extracted on the date Test #5 was shut down (August 25, 2005). Both exterior and interior fiberglass samples were examined. Environmental SEM (ESEM) was employed to analyze the hydrated fiberglass samples without any coating under a low vacuum condition (i.e., 80 Pa). ESEM/EDS results of the Test #5 Day-30 birdcage fiberglass samples were obtained on August 26, 2005.

Transcribed Laboratory Log

Laboratory session from August 26, 2005.
Test #5 Day-30 Birdcage Fiberglass.

ESEM



Birdcage Exterior

Image:	T5BCX01	100 ×	ESEM image	Figure C7-1
	t5bcx02	100 ×	ESEM image	Figure C7-2
	t5bcx03	500 ×	ESEM image higher magnification	Figure C7-3
EDS:	t5bcx04		EDS on particles on t5bcx03	Figure C7-4
Image:	t5bcx05	500 ×	ESEM image	Figure C7-5

Birdcage Interior

Image:	t5bcI01	100 ×	ESEM image of fiberglass	Figure C7-6
	t5bci03	100 ×	ESEM image	Figure C7-7
	t5bci02	500 ×	ESEM image higher magnification	Figure C7-8

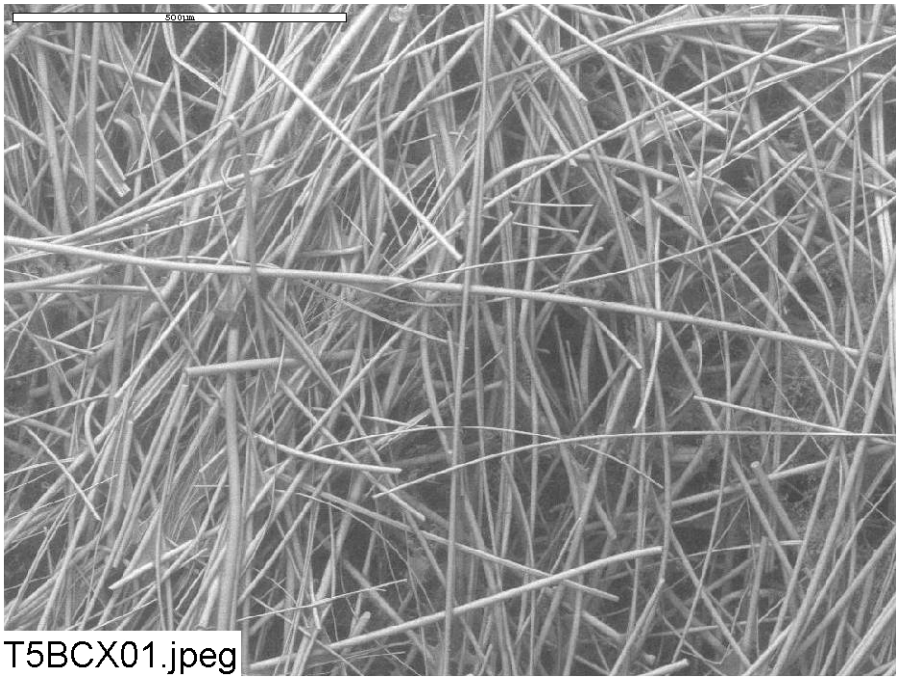


Figure C7-1: Environmental SEM image magnified 100 times for a Test #5 Day-30 exterior fiberglass sample within the birdcage. (T5BCX01.jpg)

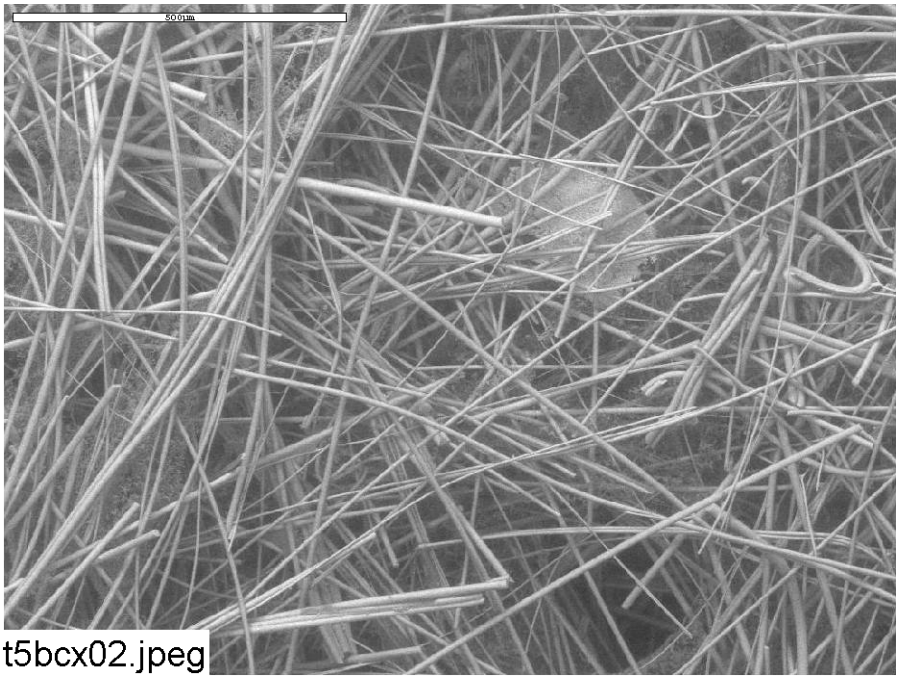


Figure C7-2: Environmental SEM image magnified 100 times for a Test #5 Day-30 exterior fiberglass sample within the birdcage. (t5bcx02.jpg)

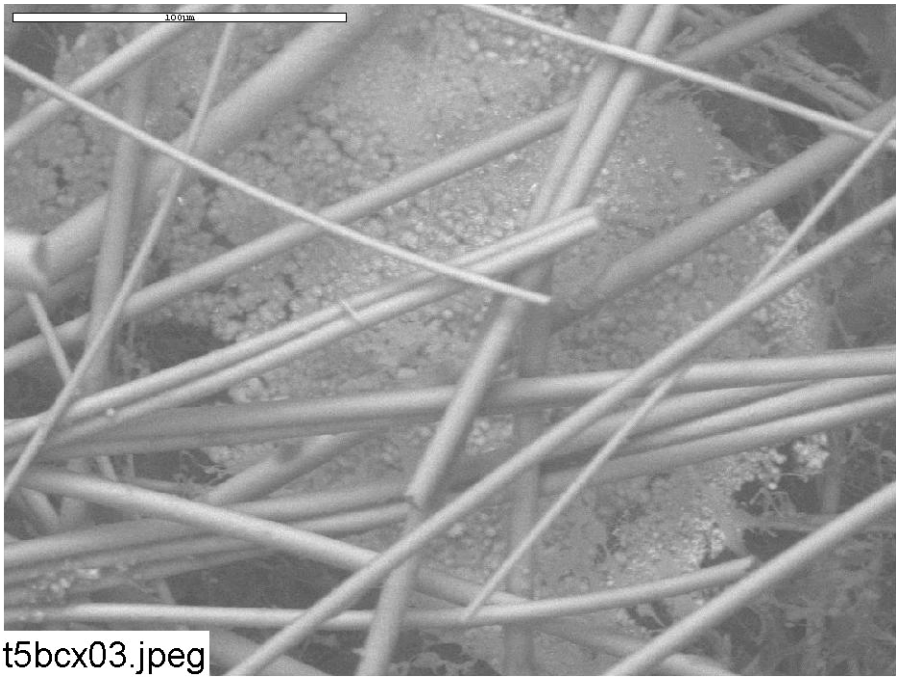


Figure C7-3: Environmental SEM image magnified 500 times for a Test #5 Day-30 exterior fiberglass sample within the birdcage. (t5bcx03.jpg)

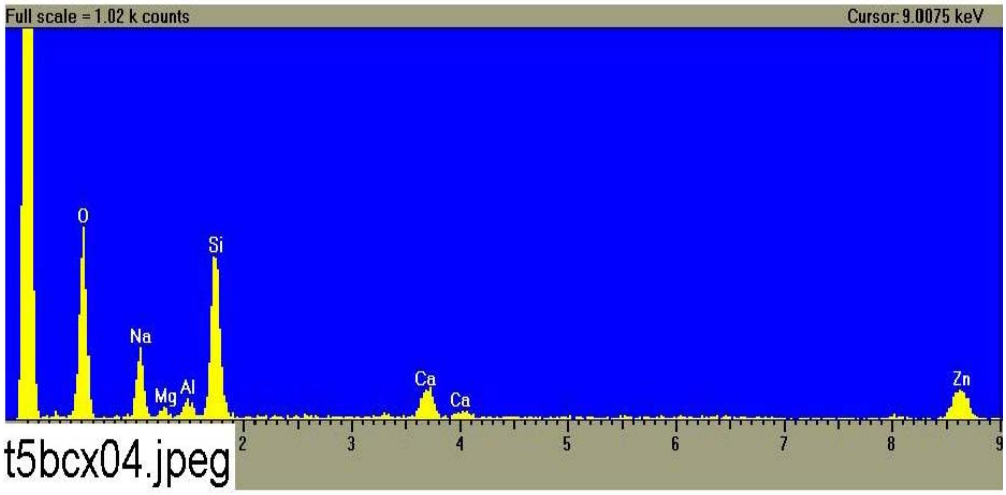


Figure C7-4: EDS counting spectrum for the deposits between fibers shown in Figure C7-3. (t5bcx04.jpg)

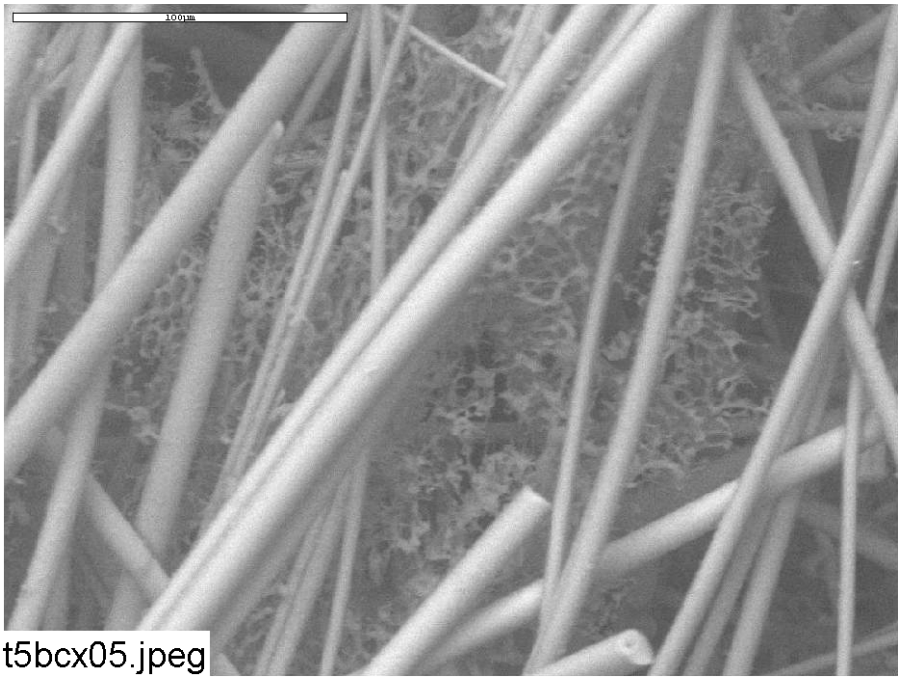


Figure C7-5: Environmental SEM image magnified 500 times for a Test #5 Day-30 exterior fiberglass sample within the birdcage. (t5bcx05.jpg)

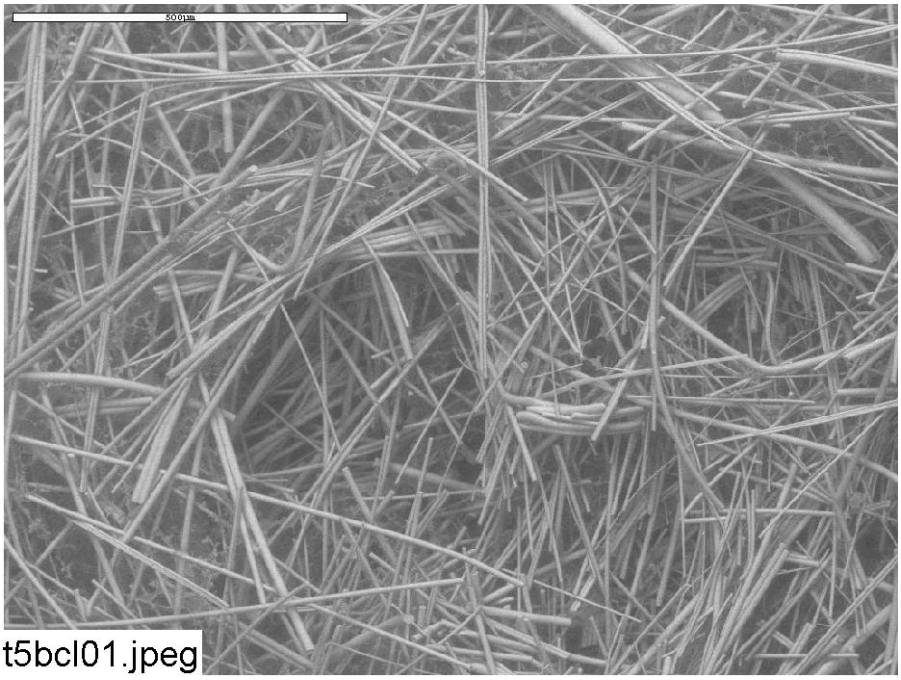


Figure C7-6: Environmental SEM image magnified 100 times for a Test #5 Day-30 interior fiberglass sample within the birdcage. (t5bcI01.jpg)

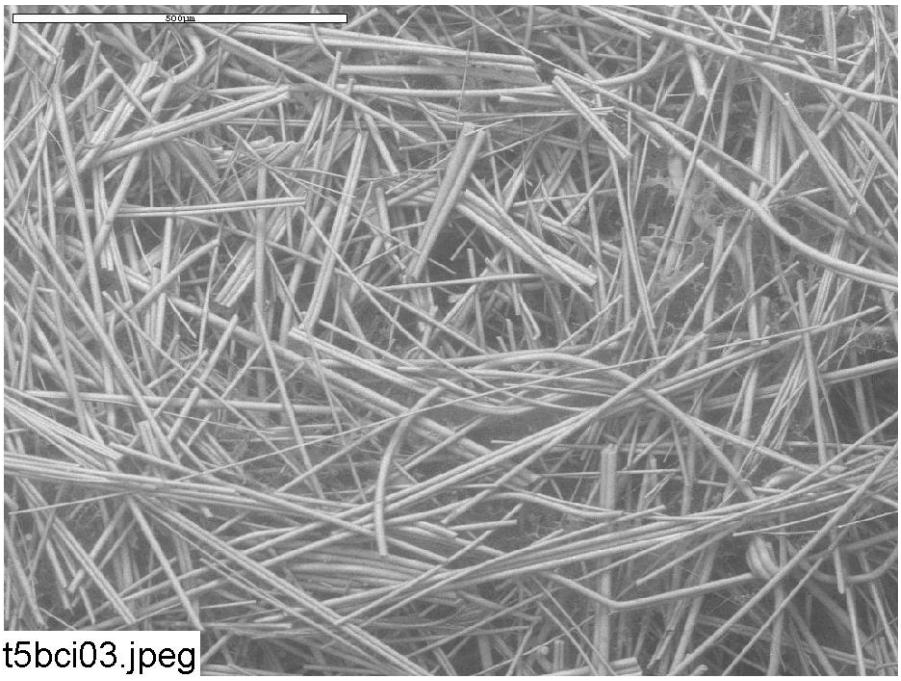


Figure C7-7: Environmental SEM image magnified 100 times for a Test #5 Day-30 interior fiberglass sample within the birdcage. (t5bci03.jpg)

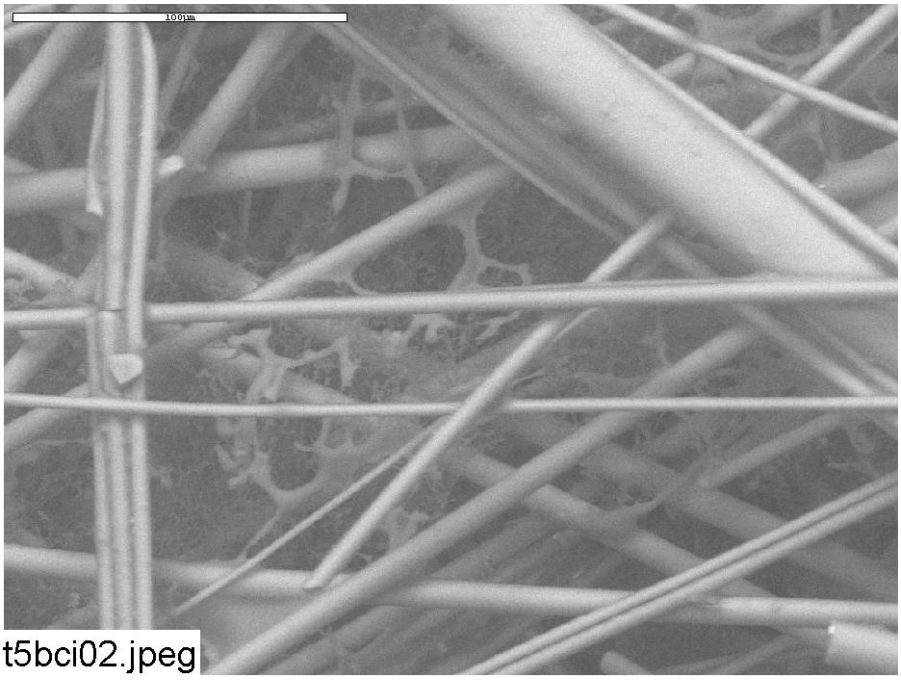


Figure C7-8: Environmental SEM image magnified 500 times for a Test #5 Day-30 interior fiberglass sample within the birdcage. (t5bci02.jpg)

Appendix D

SEM/EDS Data for Test #5 Day-30 Deposition Products

List of Figures

Figure D-1: SEM image magnified 100 times for the Test #5 Day-30 fine yellow powder on the submerged rack. (T5D30YellowDeposits001.bmp)..... D-4

Figure D-2: SEM image magnified 200 times for the Test #5 Day-30 fine yellow powder on the submerged rack. (T5D30YellowDeposits002.bmp)..... D-4

Figure D-3: Annotated SEM image magnified 1000 times for the Test #5 Day-30 fine yellow powder on the submerged rack. (T5D30YellowDeposits003.bmp)..... D-5

Figure D-4: EDS counting spectrum for the particulate deposit shown in Figure D-3. (T5D30yllw~partcl02.jpg) D-5

List of Tables

Table D-1. Chemical Compositions for T5D30yllw~partcl02.jpg, Figure D-4 ..... D-6

Deposition products were collected on the date Test #5 was shut down (August 25, 2005). The products examined were fine yellow powders that had deposited on a horizontal piece of the submerged CPVC rack.

These products were collected by directly adhering onto a double-sided carbon tape for probe SEM/EDS examination. After the samples were dried in air, Au/Pd coating was applied to enhance the surface conductivity of the samples and to prevent possible charging problems during SEM examination. Based on EDS results, a semi-quantitative elemental analysis was performed after calibration. This appendix presents the SEM/EDS data that were obtained on September 6, 2005.

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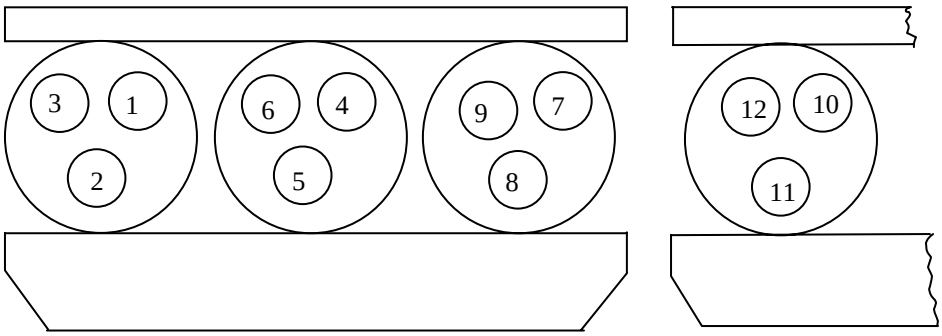
Transcribed Laboratory Log

Laboratory session from September 6, 2005.

Test #5 Day 30 Deposition Products



Conditions: e=15.0kV, WD=11mm

1--Yellow Deposits on Submerged Rack	2--Sediment (T5D30)	3--Al-Suspended
4--Al-Submerged	5--Gal-Steel Suspended	6--Gal-Steel Submerged
7--Cu Suspended	8--Cu-Submerged	9--Steel-Suspended
10--Steel-Submerged	11--Drain Collar Interior	12--Drain Collar Outside Ext.

Yellow Deposits on Submerged Rack

Image:	T5D30YellowDeposits001	100 ×	SEM image	Figure D-1
	T5D30YellowDeposits002	500 ×	SEM image	Figure D-2
	T5D30YellowDeposits003	1000 ×	Annotated SEM image	Figure D-3
EDS:	T5D30yllw~partcl02		EDS on particulate deposits	Figure D-4

Figure D-1: SEM image magnified 100 times for the Test #5 Day-30 fine yellow powder on the submerged rack. (T5D30YellowDeposits001.bmp)

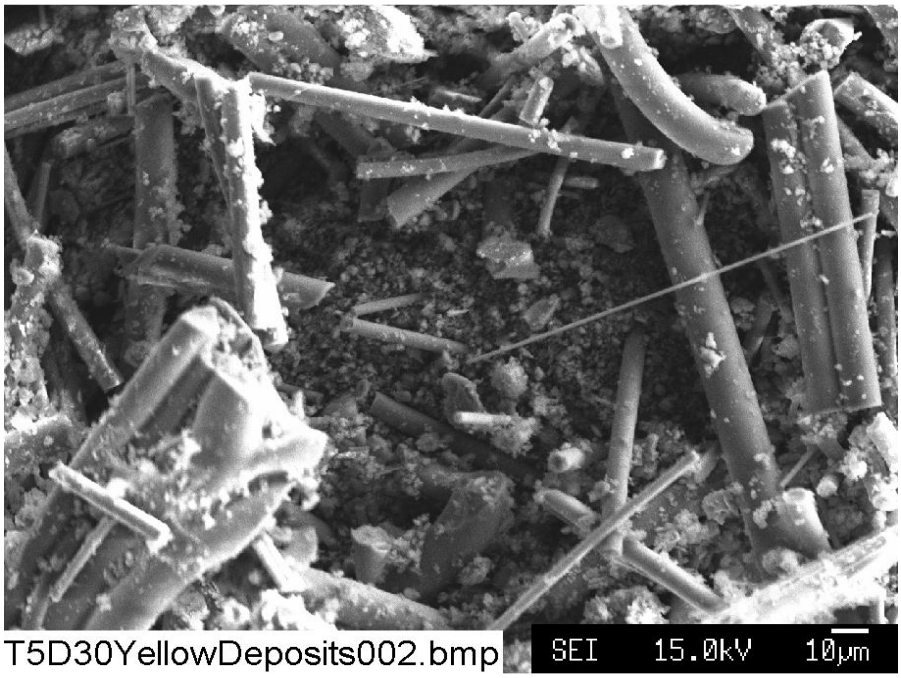


Figure D-2: SEM image magnified 200 times for the Test #5 Day-30 fine yellow powder on the submerged rack. (T5D30YellowDeposits002.bmp)

Figure D-3: Annotated SEM image magnified 1000 times for the Test #5 Day-30 fine yellow powder on the submerged rack. (T5D30YellowDeposits003.bmp)

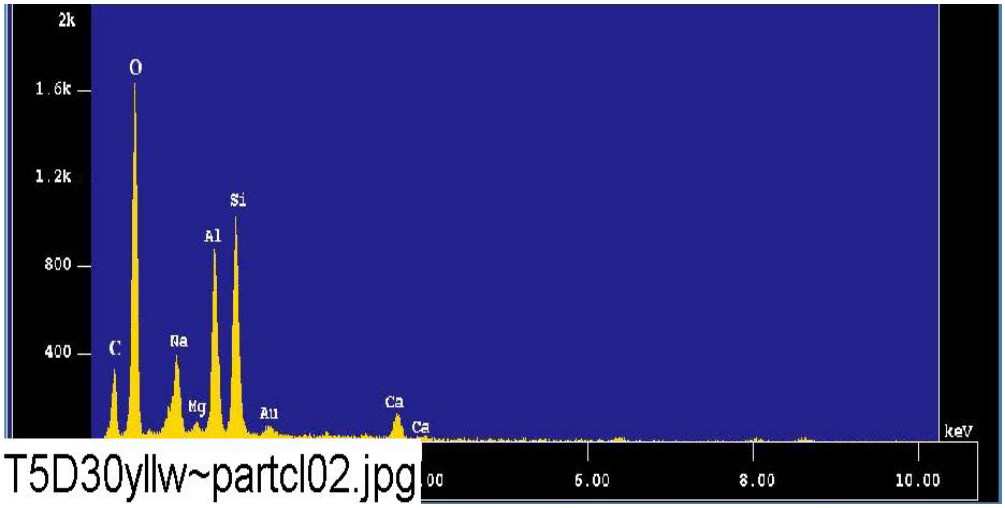


Figure D-4: EDS counting spectrum for the particulate deposit shown in Figure D-3. (T5D30yllw~partcl02.jpg)

The results from the chemical composition analysis for T5D30yllw~partcl02.jpg are given in Table D-1

Table D-1. Chemical Compositions for T5D30yllw~partcl02.jpg, Figure D-4

```
Group     : NRC
Sample    : T5D30  ID# : 30
Comment   : Yellow deposits on submerged rack
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  76.130 sec    Aperture #    : 5
            Acc. Volt   : 15.0 KV        Probe Current : 1.996E-08 A
            Stage Point : X=45.414 Y=58.152 Z=11.027
            Acq. Date   : Tue Sep  6 10:38:29 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Na K      Normal   0.83- 1.28  133.9949   0.0097     1973 /      65
 Mg K      Normal   1.00- 1.53   28.4267   0.0005      356 /     338
 Si K      Normal   1.50- 2.07  366.8570   0.0009     8937 /     400
 Ca K      Normal   3.40- 4.30  130.0604   0.0070     1526 /      18
 Al K      Normal   1.26- 1.78  325.7976   0.0016     7232 /     315
  O K      Normal   0.31- 0.74  2565.1008   0.0170    48124 /      66
  C K      Normal   0.11- 0.47  11427.6719   0.0238     2293 /     258
-------------------------------------------------------------------
                            Chi_square = 18.5321

Element Mass%    Atomic%   ZAF      Z      A      F
    Na    1.894   1.2481 1.0715 0.9872 1.0843 1.0010
    Mg    0.525   0.3269 1.3990 0.9770 1.4341 0.9985
    Si    5.653   3.0482 1.1678 0.9883 1.1817 0.9999
    Ca    1.657   0.6263 0.9658 1.0002 0.9655 1.0001
    Al    4.586   2.5745 1.0669 0.9959 1.0732 0.9982
     O   50.493  47.7996 1.4919 0.9942 1.5005 1.0000
     C   35.192  44.3764 0.2334 0.9972 0.2341 1.0000
-------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  0.0132
```

Appendix E1

SEM/EDS Data for Test #5 Day-30 Aluminum Coupons

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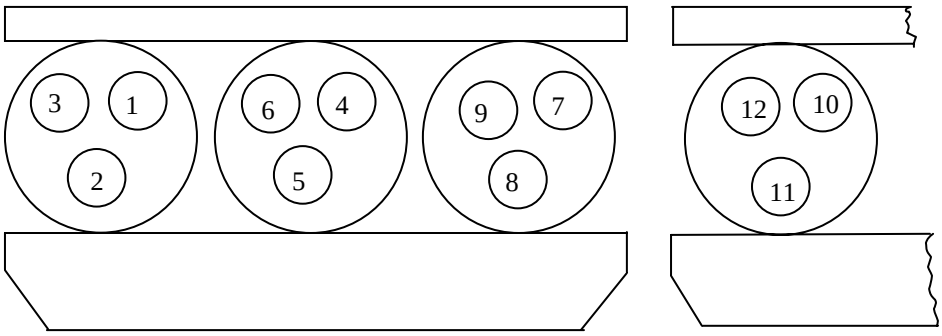
This appendix shows the SEM/EDS results for the metal aluminum coupons under two categories: (1) unsubmerged; (2) submerged. Unsubmerged refers to coupons located above the water level of the test tank during ICET tests. Unsubmerged coupons were only contacted with the solution during the initial 4-hour spray phase. In addition, the surface of the unsubmerged coupons may be affected during the test by the moist air in the tank gas space. Submerged refers to the coupons submerged in the solution during the test.

The coupon samples were collected on August 25, 2005 (the date Test #5 was shut down) and examined by SEM/EDS on August 30 and September 6, 2005. The aluminum coupon samples were dried in air before coating with Au/Pd for SEM examination. SEM results present the surface condition of the aluminum coupons. In addition, EDS results provide a semi-quantitative elemental analysis of the coupon surface and the corrosion products.

Transcribed Laboratory Log

Laboratory session from September 6, 2005.
SEM Test #5 Day-30 Aluminum Coupons.

Conditions: e=15.0kV, WD=11mm



1--Yellow Deposits on Submerged Rack	2--Sediment (T5D30)	3--Al-Unsubmerged
4--Al-Submerged	5--Gal-Steel Unsubmerged	6--Gal-Steel Submerged
7--Cu Unsubmerged	8--Cu-Submerged	9--Steel-Unsubmerged
10--Steel-Submerged	11--Drain Collar Interior	12--Drain Collar Outside Ext.

Unsubmerged Aluminum Coupons.

Image:	T5D30SuspAl007	100 ×	SEM image	Figure E1-1
	T5D30SuspAl008	500 ×	SEM image	Figure E1-2
EDS:	T5D30SuspAl_flat		EDS on flat surface in 008	Figure E1-3
	T5D30SuspAl_Granul		EDS on particle in 008	Figure E1-4
Image:	T5D30SuspAl009	1000 ×	SEM image higher magnification	Figure E1-5

Submerged Al Coupon.

Image:	T5D30SubmAl010	250 ×	SEM image	Figure E1-6
	T5D30SubmAl011	500 ×	SEM image higher magnification	Figure E1-7
	T5D30SubmAl012	1200 ×	SEM annotated	Figure E1-8
EDS:	T5D30SubmAl_dark01		EDS of dark structure in 012	Figure E1-9
	T5D30SubmAl_light02		EDS of light particle in 012	Figure E1-10

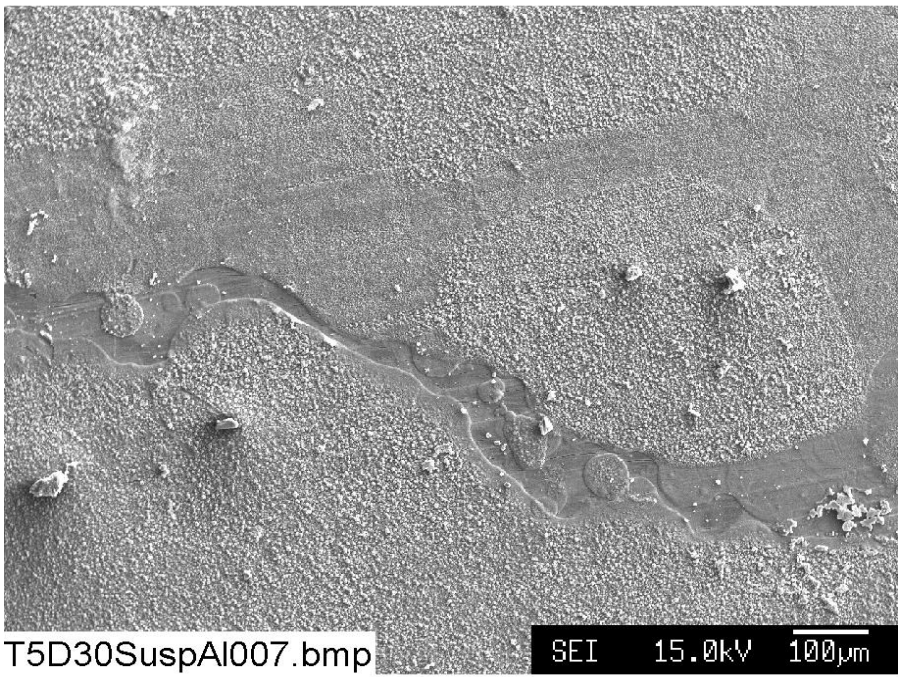


Figure E1-1: SEM image magnified 100 times for a Test #5 Day-30 unsubmerged aluminum coupon sample. (T5D30SuspAl007.bmp)

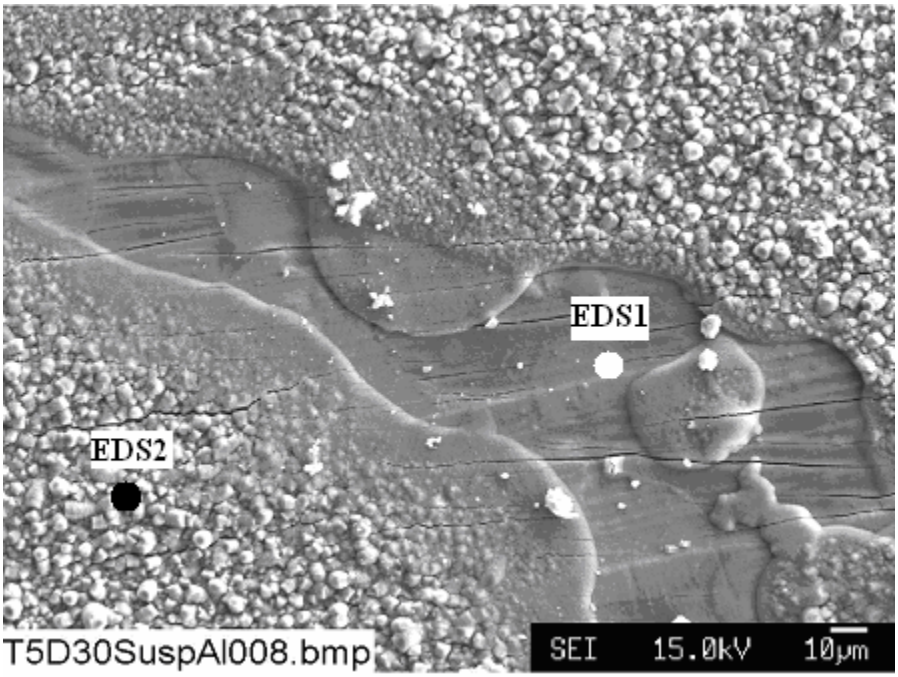


Figure E1-2: SEM image magnified 500 times for a Test #5 Day-30 unsubmerged aluminum coupon sample. (T5D30SuspAl008.bmp)

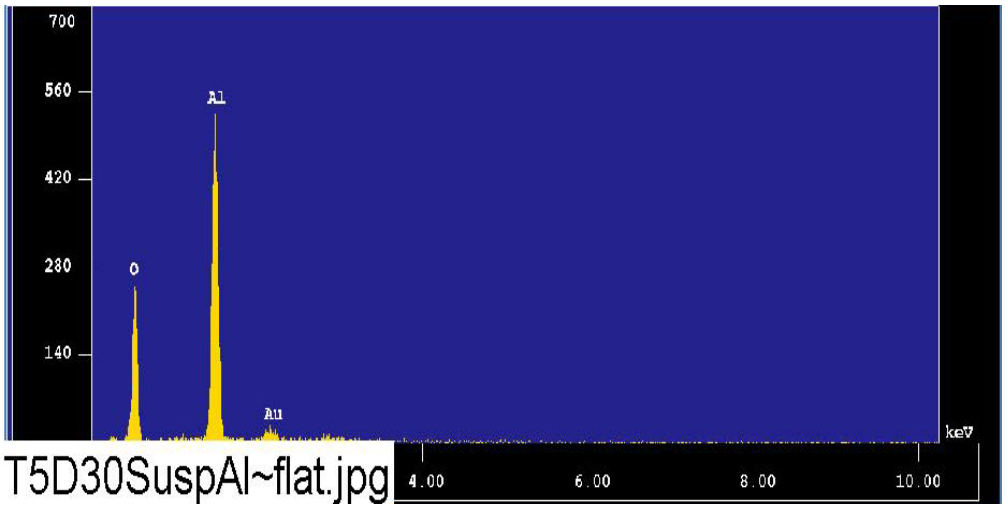


Figure E1-3: EDS counting spectrum for the smooth surface (EDS1) of the aluminum coupon shown in Figure E1-2. (T5D30SuspAl_flat.jpg)

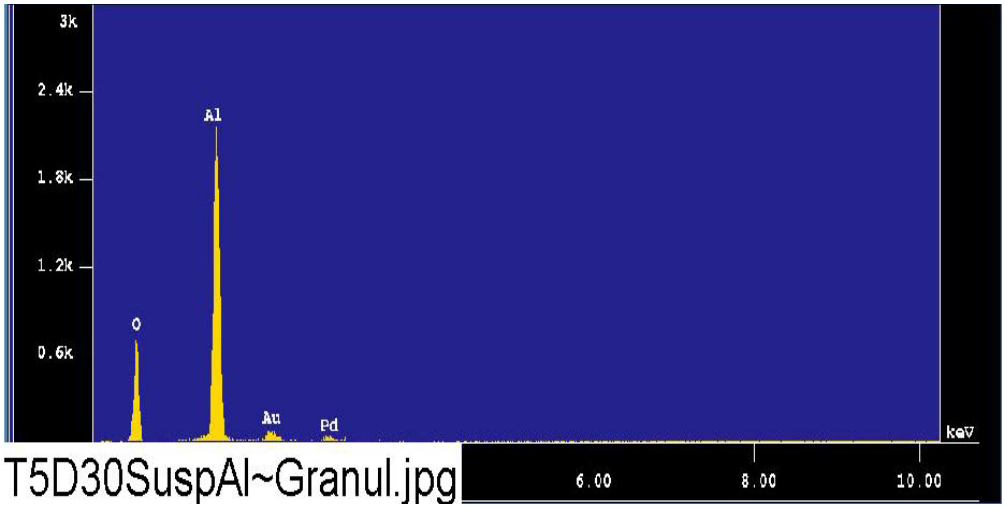


Figure E1-4: EDS counting spectrum for the granular particles (EDS2) on the aluminum coupon surface shown in Figure E1-2. (T5D30SuspAl_Granul.jpg)

The results from the chemical composition analysis for T5D30SuspAl_Granul.jpg are given in Table E1-1

Table E1-1. Chemical Compositions for T5D30SuspAl_Granul.jpg, Figure E1-4

```
Group     : NRC
Sample    : T5D30  ID# : 5
Comment   : Granular particles on suspended Al
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  60.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.006E-09 A
            Stage Point : X=88.063 Y=64.383 Z=11.848
            Acq. Date   : Tue Aug 30 13:44:34 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Al K      Normal   1.26- 1.78   16.5329   0.0023    18452 /      29
  O K      Normal   0.31- 0.74   20.9428   0.0107    19756 /      12
-------------------------------------------------------------------
                            Chi_square = 45.7092

Element Mass%    Atomic%   ZAF      Z      A      F
    Al   46.210  33.7485 1.0224 1.0020 1.0203 1.0000
     O   53.790  66.2515 0.9395 1.0018 0.9377 1.0000
-------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  2.7339
```

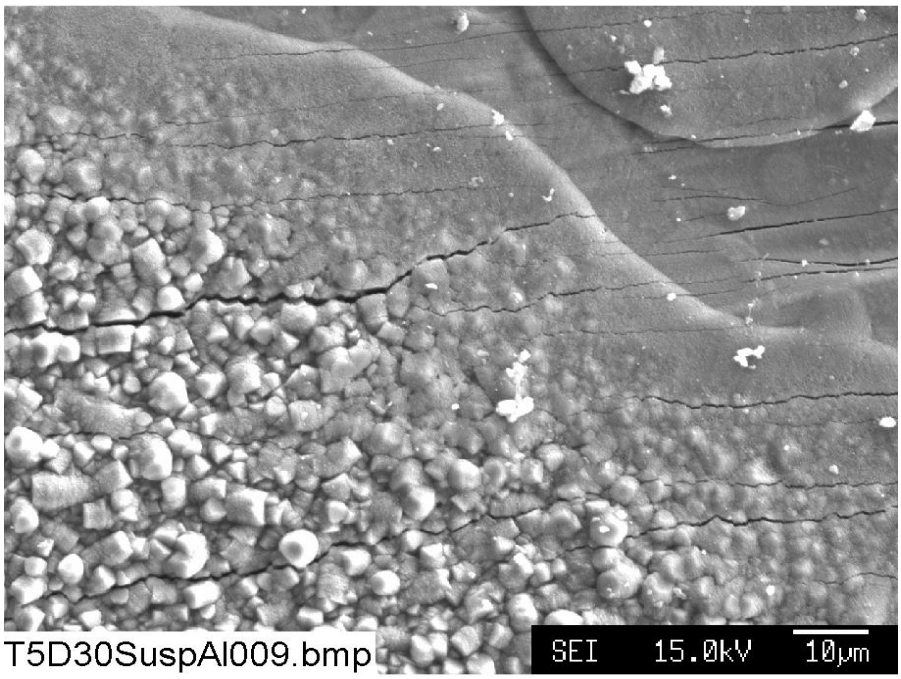


Figure E1-5: SEM image magnified 1000 times for a Test #5 Day-30 unsubmerged aluminum coupon sample. (T5D30SuspAl009.bmp)

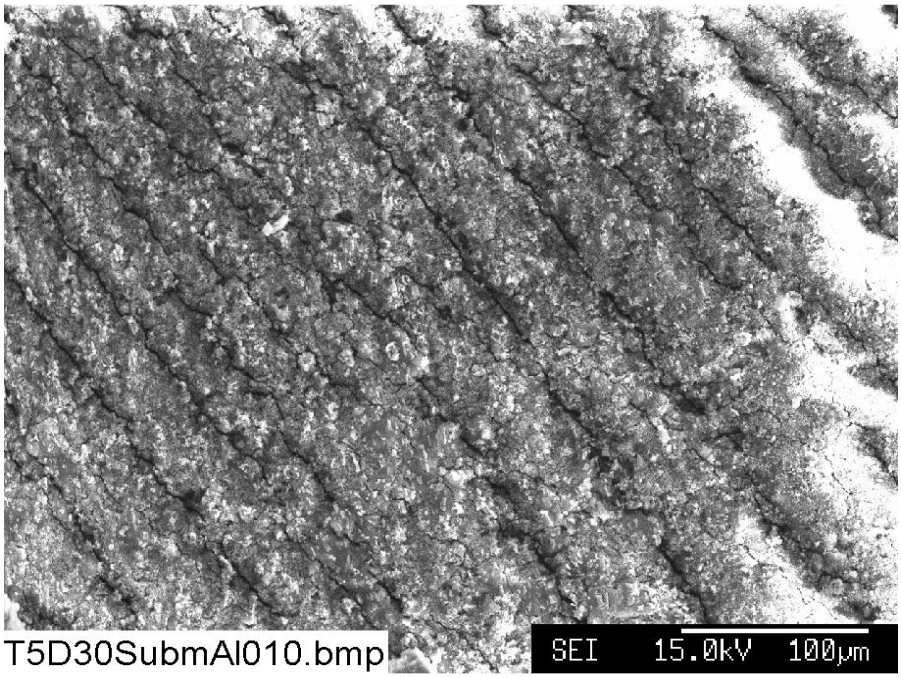


Figure E1-6: SEM image magnified 250 times for a Test #5 Day-30 submerged aluminum coupon sample. (T5D30SubmAl010.bmp)

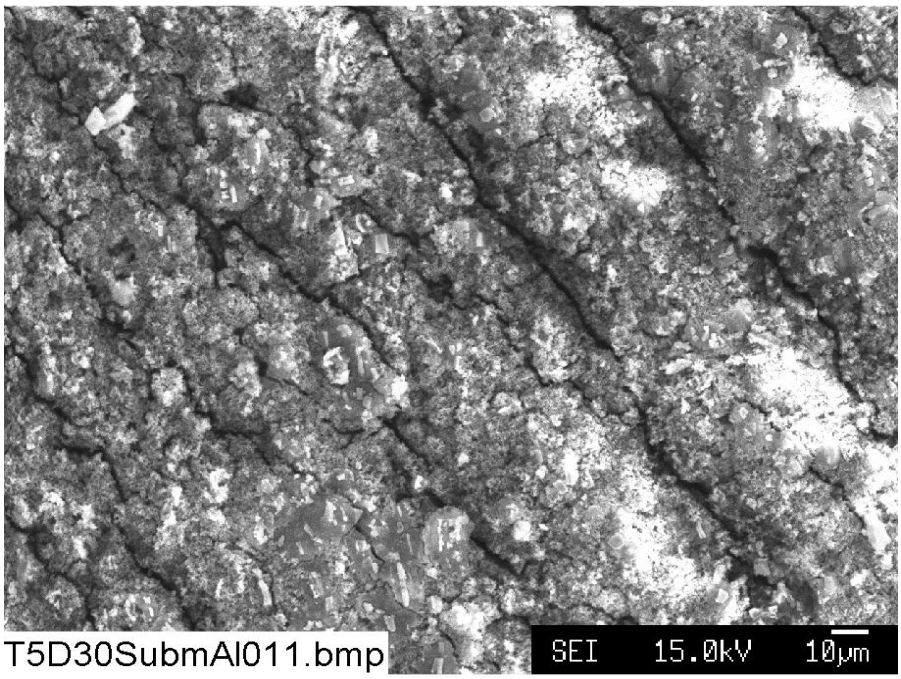


Figure E1-7: SEM image magnified 500 times for a Test #5 Day-30 submerged aluminum coupon sample. (T5D30SubmAl011.bmp)

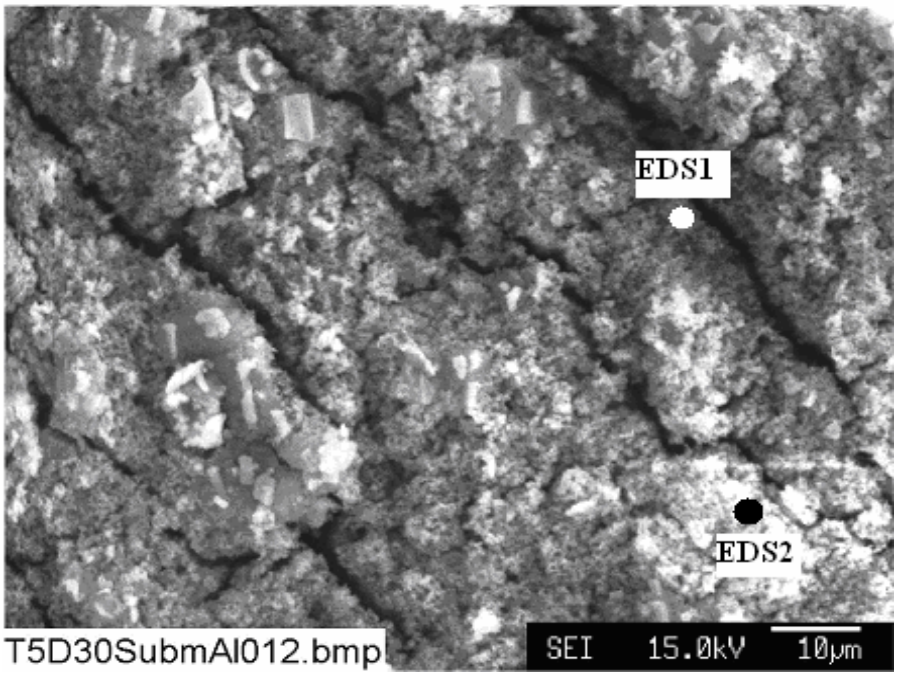


Figure E1-8: Annotated SEM image magnified 1200 times for a Test #5 Day-30 submerged aluminum coupon sample. (T5D30SubmAl012.bmp)

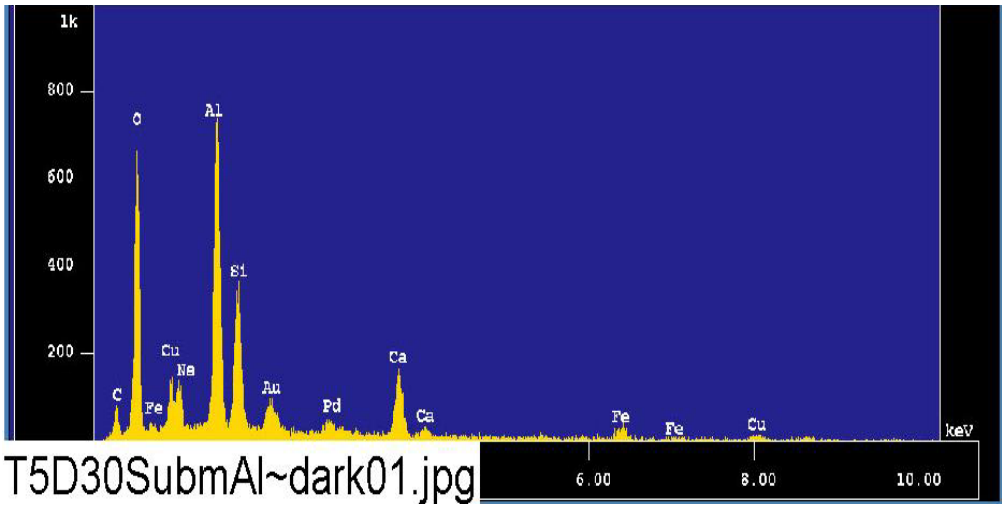


Figure E1-9: EDS counting spectrum for the dark porous surface (EDS1) of the aluminum coupon shown in Figure E1-8. (T5D30SubmAl_dark01.jpg)

The results from the chemical composition analysis for T5D30SubmAl_dark01.jpg are given in Table E1-2

Table E1-2. Chemical Compositions for T5D30SubmAl_dark01.jpg, Figure E1-9

```
Group     : NRC
Sample    : T5D30  ID# : 7
Comment   : Dark porous layer on submerged Al
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  60.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.006E-09 A
            Stage Point : X=50.175 Y=52.147 Z=11.848
            Acq. Date   : Tue Aug 30 14:03:41 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Na K      Normal   0.83- 1.28    0.9587   0.0065      710 /      37
 Al K      Normal   1.26- 1.78    5.5632   0.0015     6209 /     133
 Si K      Normal   1.50- 2.07    2.4170   0.0006     2961 /     364
 Ca K      Normal   3.40- 4.30    3.2624   0.0073     1925 /      13
 Fe K      Normal   6.04- 7.40    1.5120   0.0023      402 /       9
  C K      Normal   0.11- 0.47   50.6476   0.0123      511 /      98
  O K      Normal   0.31- 0.74   21.2659   0.0113    20061 /      35
 Cu K      Normal   7.61- 9.28    0.9478   0.0014      138 /       3
-----------------------------------------------------------------
                            Chi_square =  4.7269

Element Mass%    Atomic%   ZAF      Z      A      F
    Na    1.838   1.3545 1.1743 0.9804 1.1967 1.0009
    Al   10.097   6.3412 1.1119 0.9887 1.1261 0.9987
    Si    4.972   2.9996 1.2602 0.9810 1.2848 0.9999
    Ca    5.147   2.1761 0.9665 0.9913 0.9757 0.9992
    Fe    2.971   0.9016 1.2039 1.2151 0.9962 0.9946
     C   23.844  33.6407 0.2884 0.9910 0.2911 1.0000
     O   49.149  52.0575 1.4159 0.9878 1.4334 1.0000
    Cu    1.982   0.5287 1.2814 1.2850 0.9972 1.0000
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.6323
```

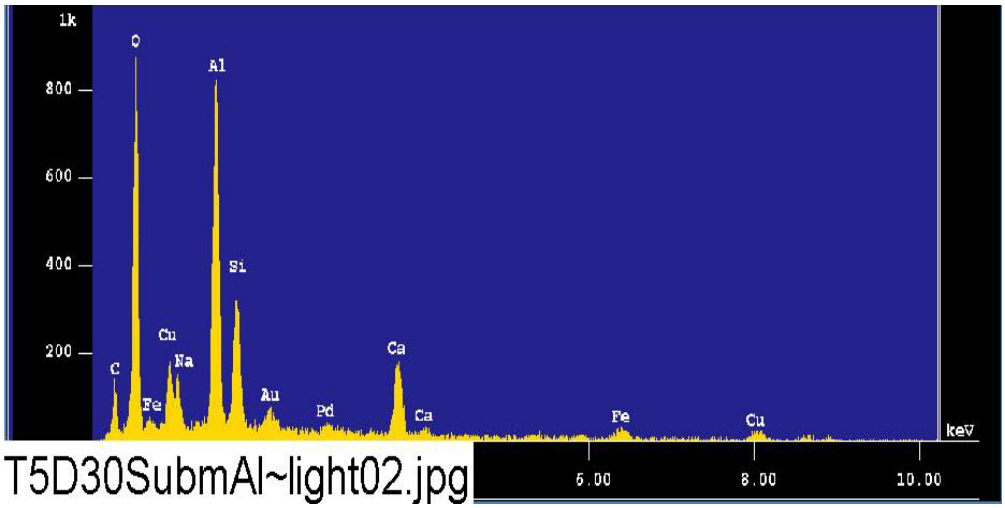


Figure E1-10: EDS counting spectrum for the light powder (EDS2) on the aluminum coupon surface shown in Figure E1-8. (T5D30SubmAl_light02.jpg)

The results from the chemical composition analysis for T5D30SubmAl_light02.jpg are given in Table E1-2

Table E1-3. Chemical Compositions for T5D30SubmAl_light02.jpg, Figure E1-9

```
Group     : NRC
Sample    : T5D30  ID# : 8
Comment   : Bright layer on submerged Al
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  60.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.006E-09 A
            Stage Point : X=46.894 Y=50.390 Z=11.848
            Acq. Date   : Tue Aug 30 14:12:44 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Na K      Normal   0.83- 1.28    0.9222   0.0067      683 /      43
 Al K      Normal   1.26- 1.78    6.2283   0.0015     6951 /     132
 Si K      Normal   1.50- 2.07    2.3139   0.0006     2834 /     415
 Ca K      Normal   3.40- 4.30    3.6587   0.0078     2159 /      22
  O K      Normal   0.31- 0.74   27.0711   0.0128    25537 /      34
  C K      Normal   0.11- 0.47   74.5273   0.0149      752 /     144
 Fe K      Normal   6.04- 7.40    1.1269   0.0022      299 /       4
 Cu K      Normal   7.61- 9.28    2.3661   0.0016      345 /       3
-------------------------------------------------------------------
                            Chi_square =  5.7109

Element Mass%    Atomic%   ZAF      Z      A      F
    Na    1.431   1.0376 1.2008 0.9788 1.2256 1.0010
    Al    9.046   5.5894 1.1241 0.9871 1.1399 0.9990
    Si    3.757   2.2298 1.2564 0.9794 1.2830 0.9999
    Ca    4.554   1.8942 0.9633 0.9899 0.9737 0.9994
     O   49.656  51.7418 1.4196 0.9861 1.4395 1.0000
     C   25.910  35.9628 0.2691 0.9893 0.2720 1.0000
    Fe    1.738   0.5188 1.1936 1.2136 0.9954 0.9881
    Cu    3.909   1.0256 1.2785 1.2835 0.9962 1.0000
-------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.2921
```

Appendix E2

SEM/EDS Data for Test #5 Day-30 Copper Coupons

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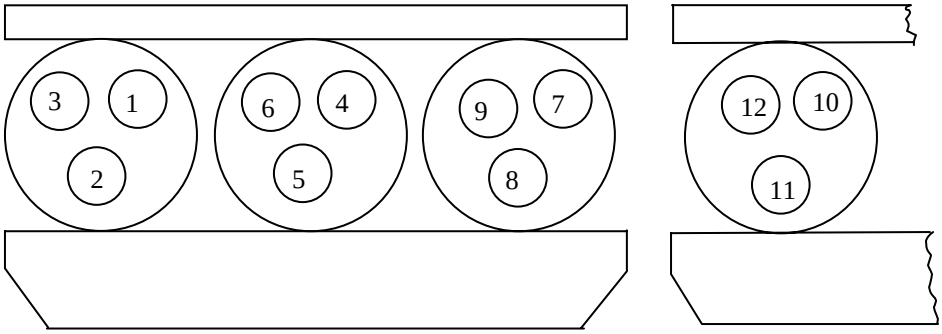
This appendix shows the SEM/EDS results for the metal copper coupons under two categories: (1) unsubmerged; (2) submerged. Unsubmerged coupons were only contacted with the solution during the initial 4-hour spray phase. In addition, the surface of the unsubmerged coupons may be affected during the test by the moist air in the tank gas space. Submerged refers to the coupons submerged in the solution during the test.

The coupon samples were collected on August 25, 2005 (the date Test #5 was shut down) and examined by SEM/EDS on August 30 and September 6, 2005. The copper coupon samples were dried in air before coating with Au/Pd for SEM examination. SEM results present the surface condition of the copper coupons. In addition, EDS results provide a semi-quantitative elemental analysis of the coupon surface and the corrosion products.

Transcribed Laboratory Log

Laboratory session from September 6, 2005.
SEM Test #5 Day-30 Copper Coupons.

Conditions: e=15.0kV, WD=11mm



1--Yellow Deposits on Submerged Rack	2--Sediment (T5D30)	3--Al-Unsubmerged
4--Al-Submerged	5--Gal-Steel Unsubmerged	6--Gal-Steel Submerged
7--Cu Unsubmerged	8--Cu-Submerged	9--Steel-Unsubmerged
10--Steel-Submerged	11--Drain Collar Interior	12--Drain Collar Outside Ext.

Unsubmerged Copper Coupons.

Image:	T5D30SuspCu017	100 ×	SEM image	Figure E2-1
	T5D30SuspCu018	1000 ×	SEM Annotated image higher magnification	Figure E2-2
EDS:	T5D30SuspCu particle07		On light particles shown in 018	Figure E2-3
	T5D30SuspCu Surface08		On coupon surface shown in 018	Figure E2-4

Submerged Copper Coupon.

Image:	T5D30SubmCu019	100 ×	SEM image of fiberglass	Figure E2-5
	T5D30SubmCu020	1000 ×	SEM image higher magnification	Figure E2-6
EDS:	T5D30Subm Cu surface09		EDS of smooth particles in 020	Figure E2-7
	T5D30Subm Cu particle10		EDS of white particles in 020	Figure E2-8

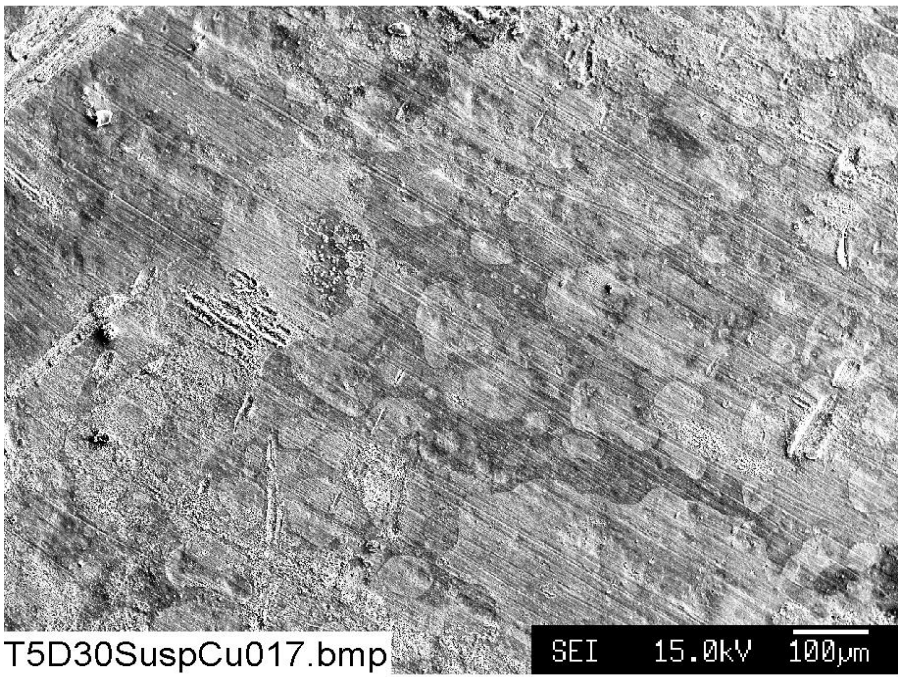


Figure E2-1: SEM image magnified 100 times for a Test #5 Day-30 unsubmerged copper coupon sample. (T5D30SuspCu017.bmp)

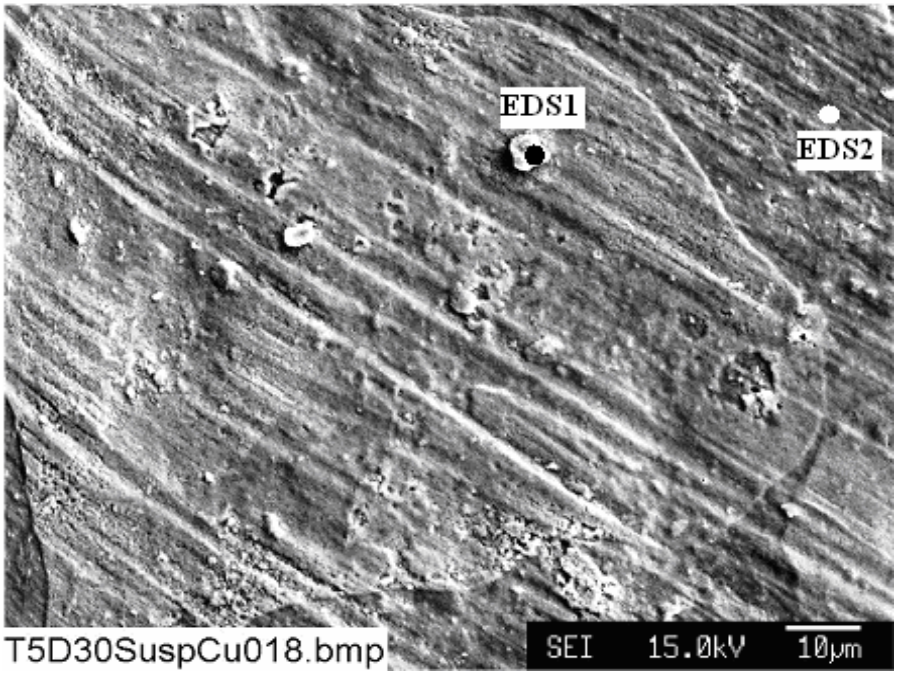


Figure E2-2: Annotated SEM image magnified 1000 times for a Test #5 Day-30 unsubmerged copper coupon sample. (T5D30SuspCu018.bmp)

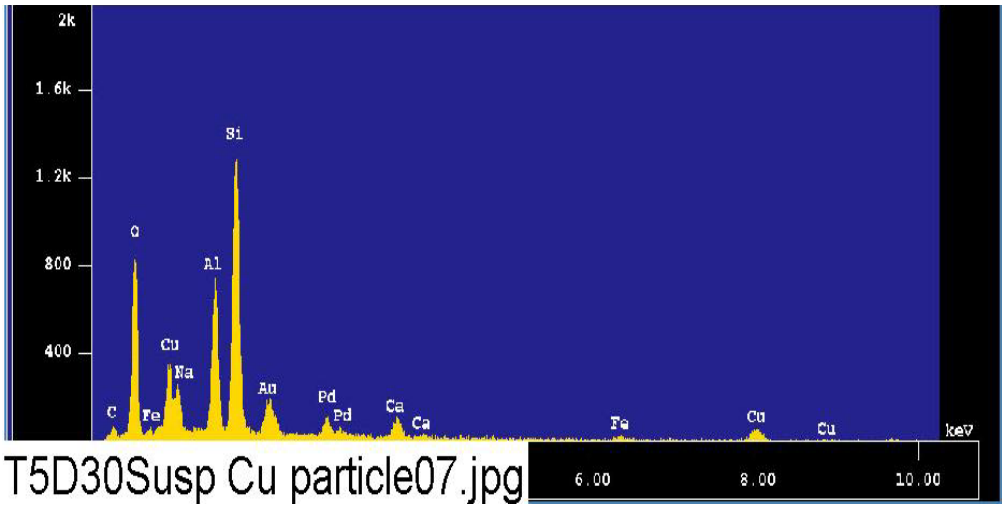


Figure E2-3: EDS counting spectrum for the light particle (EDS1) on the copper coupon surface shown in Figure E2-2. (T5D30Susp Cu particle07.jpg)

The results from the chemical composition analysis for T5D30Susp Cu particle07.jpg are given in Table E2-1

Table E2-1. Chemical Compositions for T5D30Susp Cu particle07.jpg, Figure E2-3

```
Group     : NRC
Sample    : T5D30  ID# : 13
Comment   : Particles on suspended Cu
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  60.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.005E-09 A
            Stage Point : X=16.143 Y=53.149 Z=11.848
            Acq. Date   : Tue Aug 30 15:04:14 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Na K      Normal   0.83- 1.28    2.3307   0.0092     1724 /      64
 Al K      Normal   1.26- 1.78    5.5249   0.0015     6160 /     395
 Si K      Normal   1.50- 2.07    9.9056   0.0011    12122 /     391
 Ca K      Normal   3.40- 4.30    1.8982   0.0067     1119 /      16
 Fe K      Normal   6.04- 7.40    0.8441   0.0021      224 /       9
 Cu K      Normal   7.61- 9.28    5.2252   0.0023      760 /       7
  O K      Normal   0.31- 0.74   28.9157   0.0133    27250 /      37
  C K      Normal   0.11- 0.47   22.6341   0.0128      228 /     155
-----------------------------------------------------------------
                            Chi_square =  4.3581

Element Mass%    Atomic%   ZAF      Z      A      F
    Na    3.761   3.1269 1.2445 0.9775 1.2722 1.0008
    Al    8.435   5.9747 1.1773 0.9854 1.1993 0.9962
    Si   16.555  11.2653 1.2888 0.9775 1.3185 0.9999
    Ca    2.419   1.1534 0.9827 0.9864 0.9968 0.9994
    Fe    1.290   0.4413 1.1781 1.2064 0.9981 0.9783
    Cu    8.607   2.5887 1.2701 1.2739 0.9971 1.0000
     O   46.196  55.1835 1.2320 0.9852 1.2504 1.0000
     C   12.737  20.2662 0.4339 0.9886 0.4390 1.0000
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.2968
```

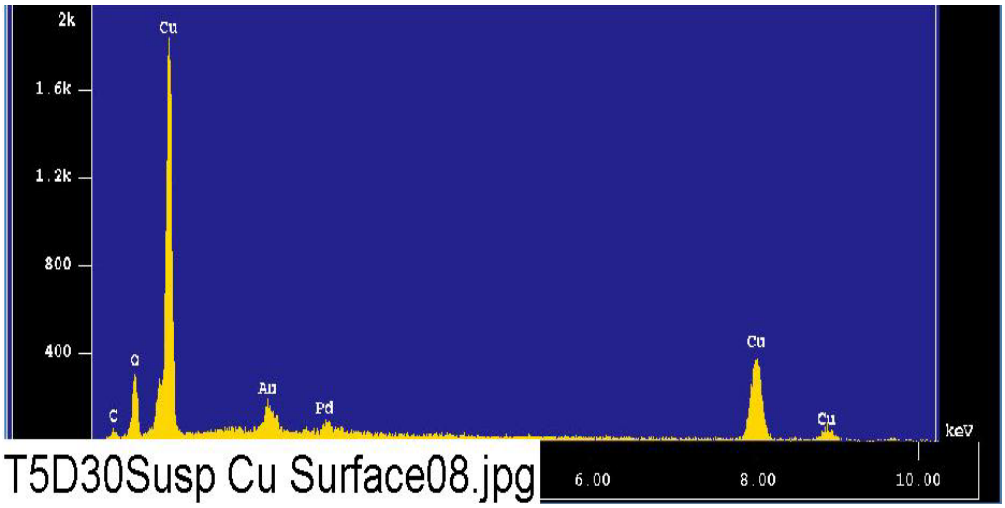


Figure E2-4: EDS counting spectrum for the copper coupon surface (EDS2) shown in Figure E2-2. (T5D30Susp Cu Surface08.jpg)

The results from the chemical composition analysis for T5D30Susp Cu Surface08.jpg are given in Table E2-2

Table E2-2. Chemical Compositions for T5D30Susp Cu Surface08.jpg, Figure E2-4

```
Group     : NRC
Sample    : T5D30  ID# : 14
Comment   : Surface of suspended Cu
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  60.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.005E-09 A
            Stage Point : X=16.143 Y=53.149 Z=11.848
            Acq. Date   : Tue Aug 30 15:09:59 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Cu K      Normal   7.61- 9.28   37.9341   0.0051     5518 /       9
  O K      Normal   0.31- 0.74    9.8150   0.0087     9250 /      52
  C K      Normal   0.11- 0.47   26.8105   0.0098      270 /      60
-------------------------------------------------------------------
                              Chi_square =  2.8337

Element Mass%    Atomic%   ZAF      Z      A      F
    Cu   67.862  31.6586 1.1006 1.1031 0.9978 1.0000
     O   17.836  33.0455 1.1180 0.8760 1.2764 0.9999
     C   14.302  35.2958 0.3282 0.8796 0.3731 1.0000
-------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.6254
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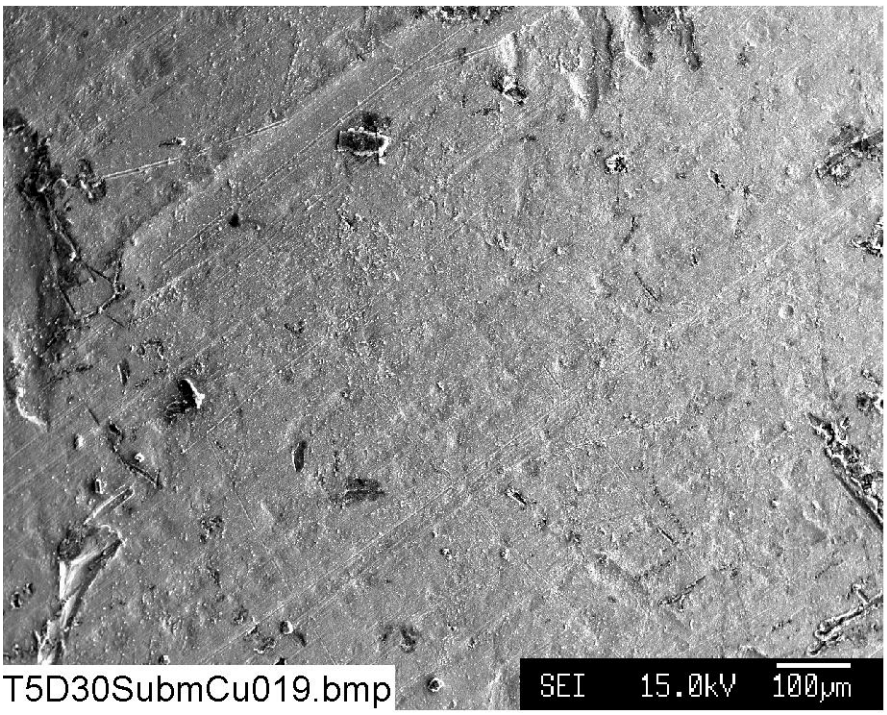

Figure E2-5: SEM image magnified 100 times for a Test #5 Day-30 submerged copper coupon sample. (T5D30SubmCu019.bmp)

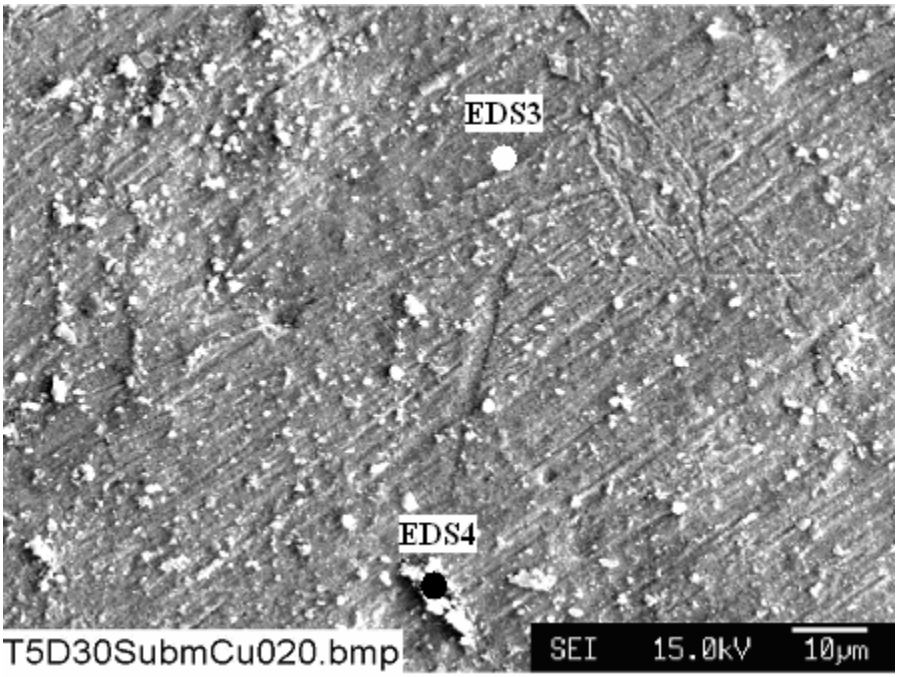

Figure E2-6: SEM image magnified 1000 times for a Test #5 Day-30 submerged copper coupon sample. (T5D30SubmCu020.bmp)

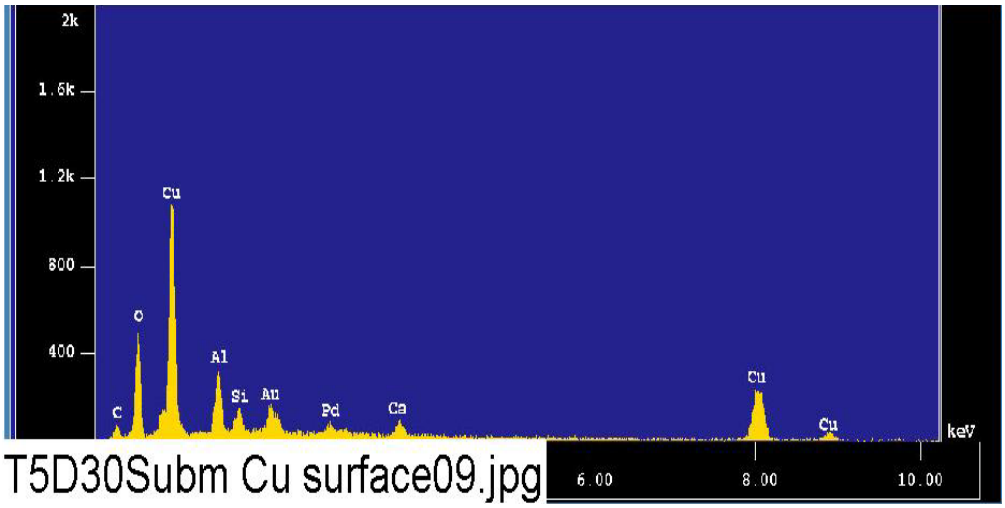

Figure E2-7: EDS counting spectrum for the grey copper coupon surface (EDS3) shown in Figure E2-6. (T5D30Subm Cu surface09.jpg)

The results from the chemical composition analysis for T5D30Subm Cu surface09.jpg are given in Table E2-3

Table E2-3. Chemical Compositions for T5D30Subm Cu surface09.jpg, Figure E2-7

```
Group     : NRC
Sample    : T5D30  ID# : 15
Comment   : Surface of submerged Cu
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  60.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.005E-09 A
            Stage Point : X=11.640 Y=63.166 Z=11.848
            Acq. Date   : Tue Aug 30 15:20:19 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Al K      Normal   1.26- 1.78    2.1463   0.0010     2393 /      79
 Si K      Normal   1.50- 2.07    0.7437   0.0005      910 /     189
 Ca K      Normal   3.40- 4.30    1.2241   0.0066      722 /      24
 Cu K      Normal   7.61- 9.28   24.4152   0.0042     3551 /       9
  O K      Normal   0.31- 0.74   15.7405   0.0101    14834 /      41
  C K      Normal   0.11- 0.47   37.1593   0.0132      375 /      85
-------------------------------------------------------------------
                            Chi_square =  3.4035

Element Mass%    Atomic%   ZAF      Z      A      F
    Al    4.915   4.1748 1.5037 0.9203 1.6344 0.9997
    Si    1.647   1.3440 1.4543 0.9126 1.5937 1.0000
    Ca    1.717   0.9819 0.9211 0.9183 1.0049 0.9982
    Cu   43.542  15.7063 1.1711 1.1747 0.9969 1.0000
     O   29.739  42.6032 1.2406 0.9212 1.3468 1.0000
     C   18.441  35.1897 0.3259 0.9247 0.3524 1.0000
-------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.5228
```

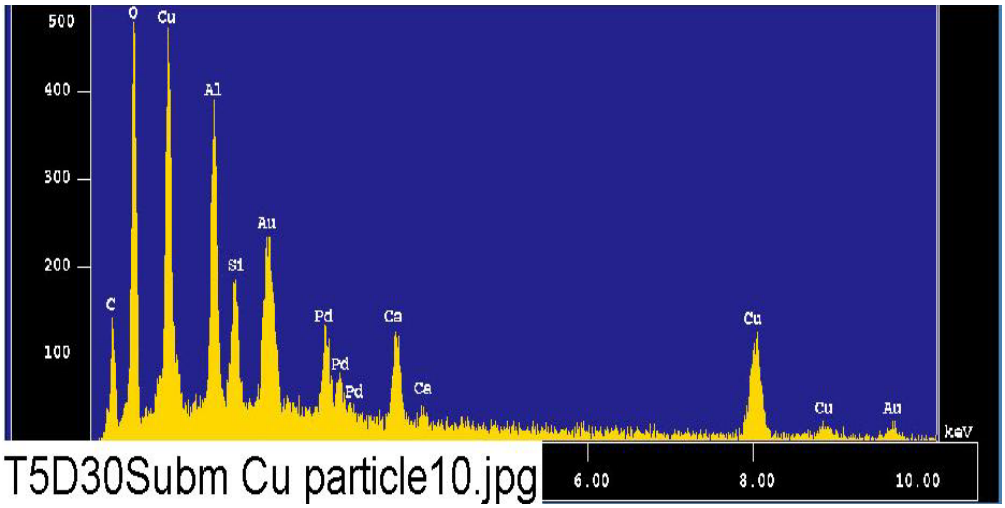

Figure E2-8: EDS counting spectrum for the light particles (EDS4) on the coupon surface shown in Figure E2-6. (T5D30Subm Cu particle10.jpg)

The results from the chemical composition analysis for T5D30Subm Cu particle10.jpg are given in Table E2-4

Table E2-4. Chemical Compositions for T5D30Subm Cu particle10.jpg, Figure E2-8

```
Group     : NRC
Sample    : T5D30  ID# : 16
Comment   : Particles on submerged Cu
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  60.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.005E-09 A
            Stage Point : X=11.640 Y=63.166 Z=11.848
            Acq. Date   : Tue Aug 30 15:24:43 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Al K      Normal   1.26- 1.78    2.6588   0.0011     2964 /      85
 Si K      Normal   1.50- 2.07    1.0195   0.0005     1248 /     247
 Ca K      Normal   3.40- 4.30    2.2751   0.0071     1341 /      27
 Cu K      Normal   7.61- 9.28   11.1316   0.0031     1619 /       7
  C K      Normal   0.11- 0.47   69.2823   0.0160      698 /      88
  O K      Normal   0.31- 0.74   15.1166   0.0099    14246 /      38
-----------------------------------------------------------------
                            Chi_square =  4.8904

Element Mass%    Atomic%   ZAF      Z      A      F
    Al    5.525   3.7131 1.2874 0.9559 1.3474 0.9995
    Si    2.175   1.4045 1.3219 0.9482 1.3942 0.9999
    Ca    3.453   1.5625 0.9405 0.9570 0.9839 0.9988
    Cu   22.087   6.3036 1.2293 1.2346 0.9957 1.0000
     C   30.146  45.5152 0.2696 0.9588 0.2812 1.0000
     O   36.614  41.5011 1.5007 0.9555 1.5705 1.0000
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.6140
```

Appendix E3

SEM/EDS Data for Test #5 Day-30 Galvanized Steel Coupons

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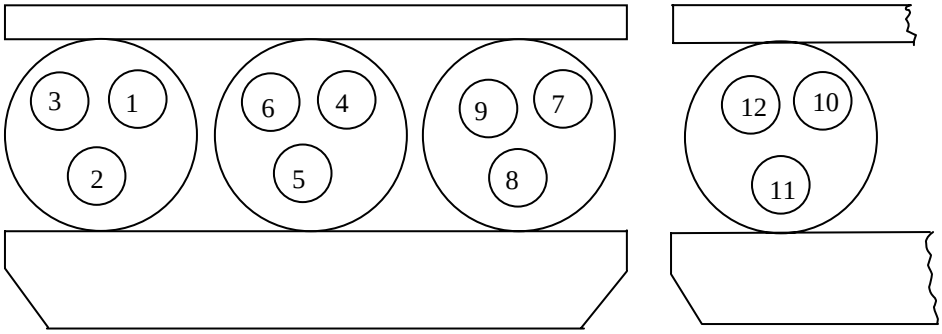
This appendix shows the SEM/EDS results for the metal galvanized steel coupons under two categories: (1) unsubmerged; (2) submerged. Unsubmerged coupons were only contacted with the solution during the initial 4-hour spray phase. In addition, the surface of the unsubmerged coupons may be affected during the test by the moist air in the tank gas space. Submerged refers to the coupons submerged in the solution during the test.

The coupon samples were collected on August 25, 2005 (the date Test #5 was shut down) and examined by SEM/EDS on August 30 and September 6, 2005. The galvanized steel coupon samples were dried in air before coating with Au/Pd for SEM examination. SEM results present the surface condition of the galvanized steel coupons. In addition, EDS results provide a semi-quantitative elemental analysis of the coupon surface and the corrosion products.

Transcribed Laboratory Log

Laboratory session from September 6, 2005.
SEM Test #5 Day-30 Galvanized Steel Coupons.

Conditions: e=15.0kV, WD=11mm



1--Yellow Deposits on Submerged Rack	2--Sediment (T5D30)	3--Al-Unsubmerged
4--Al-Submerged	5--Gal-Steel Unsubmerged	6--Gal-Steel Submerged
7--Cu Unsubmerged	8--Cu-Submerged	9--Steel-Unsubmerged
10--Steel-Submerged	11--Drain Collar Interior	12--Drain Collar Outside Ext.

Unsubmerged Galvanized Steel Coupon.

Image:	T5D30SuspGalsteel013	100 ×	SEM image	Figure E3-1
	T5D30SuspGalsteel014	1000 ×	SEM Annotated image	Figure E3-2
EDS:	T5D30Susp GS needle03		On needle like crystal shown in image 014	Figure E3-3
	T5D30 Susp GS layer04		On cracked skin layer shown in image 014	Figure E3-4

Submerged Galvanized Steel Coupon.

Image:	T5D30SubmGalsteel015	100 ×	SEM image of fiberglass	Figure E3-5
	T5D30SubmGalsteel016	1000 ×	SEM image higher magnification	Figure E3-6
EDS:	T5D30Subm GS Particle05		On particles shown in image 016	Figure E3-7
	T5D30Subm GS Particle05		On particles shown in image 016	Figure E3-8

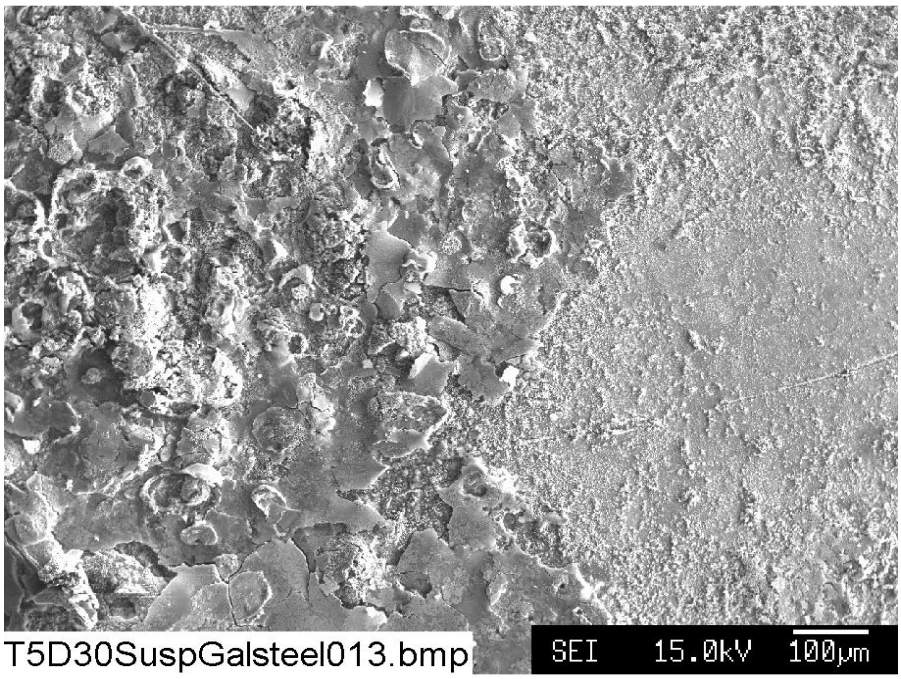


Figure E3-1: SEM image magnified 100 times for a Test #5 Day-30 unsubmerged galvanized steel coupon sample. (T5D30SuspGalsteel013.bmp)

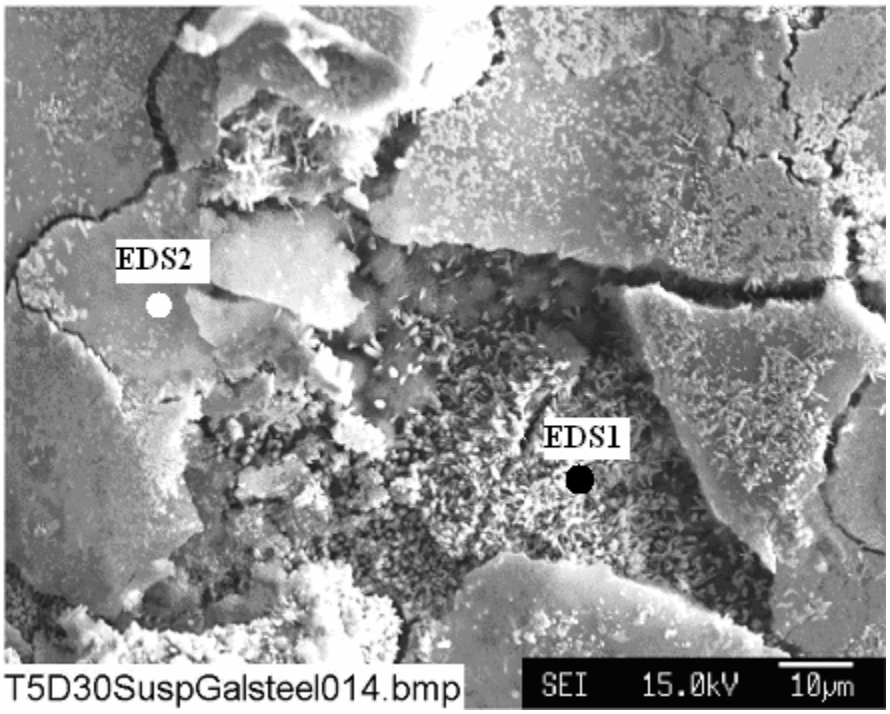


Figure E3-2: Annotated SEM image magnified 1000 times for a Test #5 Day-30 unsubmerged galvanized steel coupon sample. (T5D30SuspGalsteel014.bmp)

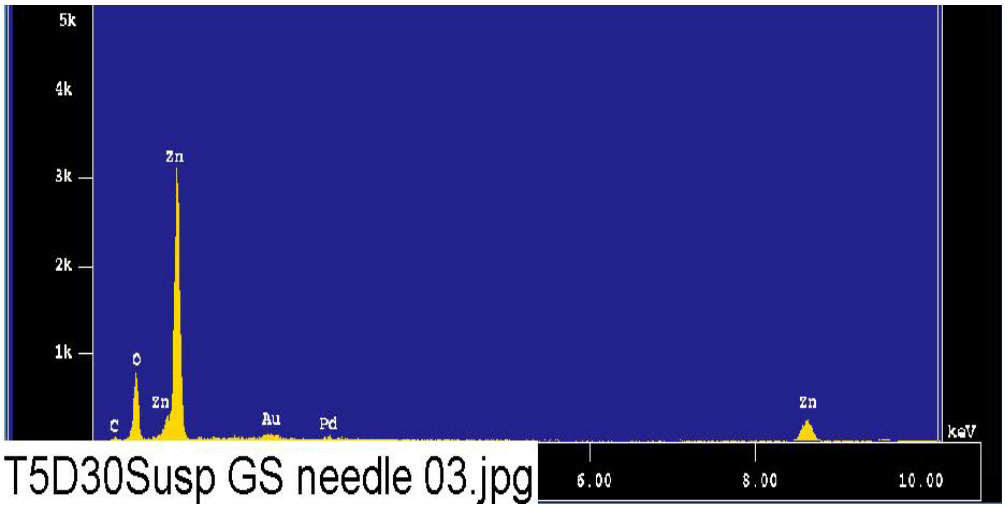


Figure E3-3: EDS counting spectrum for the needle-like crystal (EDS1) on the unsubmerged galvanized steel coupon surface shown in Figure E3-2. (T5D30Susp GS needle 03.jpg)

The results from the chemical composition analysis for T5D30Susp GS needle 03.jpg are given in Table E3-1

Table E3-1. Chemical Compositions for T5D30Susp GS needle 03.jpg, Figure E3-3

```
Group     : NRC
Sample    : T5D30  ID# : 9
Comment   : Needle crystal on suspended Gal-steel
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  60.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.005E-09 A
            Stage Point : X=44.601 Y=63.875 Z=11.848
            Acq. Date   : Tue Aug 30 14:27:56 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Zn K      Normal   8.19-10.06   30.9955   0.0087     3521 /       5
  O K      Normal   0.31- 0.74   24.2024   0.0125    22808 /      40
  C K      Normal   0.11- 0.47   29.4584   0.0106      297 /     123
-------------------------------------------------------------------
                              Chi_square =  7.1611

Element Mass%    Atomic%   ZAF      Z      A      F
    Zn   48.925  17.6401 1.1674 1.1710 0.9969 1.0000
     O   36.525  53.8072 1.1161 0.9160 1.2184 1.0000
     C   14.550  28.5527 0.3653 0.9195 0.3973 1.0000
-------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.3522
```

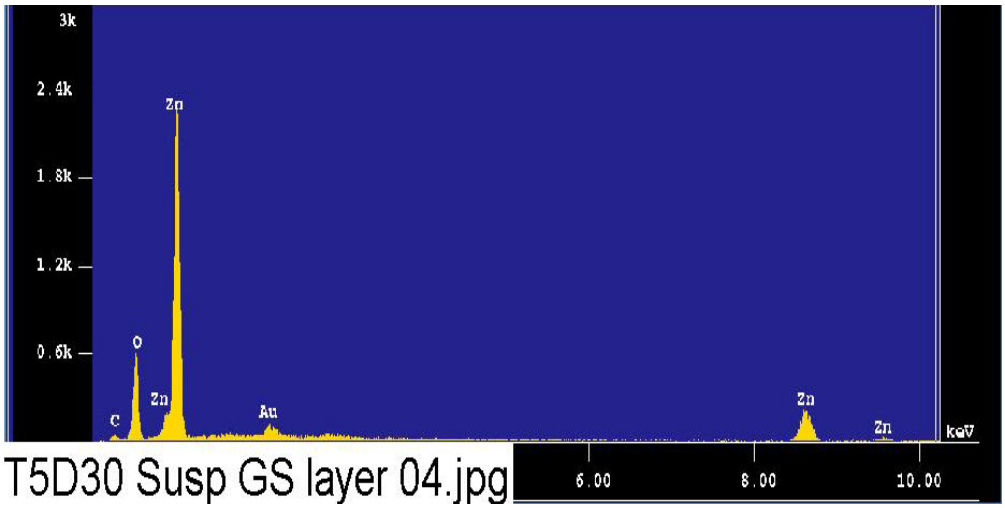


Figure E3-4: EDS counting spectrum for the cracked layer (EDS2) on the unsubmerged galvanized steel coupon surface shown in Figure E3-2. (T5D30 Susp GS layer 04.jpg)

The results from the chemical composition analysis for T5D30 Susp GS layer 04.jpg are given in Table E3-2

Table E3-2. Chemical Compositions for T5D30 Susp GS layer 04.jpg, Figure E3-4

```
Group     : NRC
Sample    : T5D30  ID# : 10
Comment   : Cracked layer on suspended Gal-steel
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  60.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.006E-09 A
            Stage Point : X=44.601 Y=63.875 Z=11.848
            Acq. Date   : Tue Aug 30 14:32:43 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Zn K      Normal   8.19-10.06   27.9430   0.0081     3177 /       6
  C K      Normal   0.11- 0.47   29.1554   0.0102      294 /     100
  O K      Normal   0.31- 0.74   19.0550   0.0110    17975 /      35
-------------------------------------------------------------------
                              Chi_square =  4.1819

Element Mass%    Atomic%   ZAF      Z      A      F
    Zn   49.823  17.9813 1.1635 1.1671 0.9970 1.0000
     C   16.397  32.2068 0.3670 0.9167 0.4003 1.0000
     O   33.780  49.8119 1.1568 0.9133 1.2667 1.0000
-------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.5324
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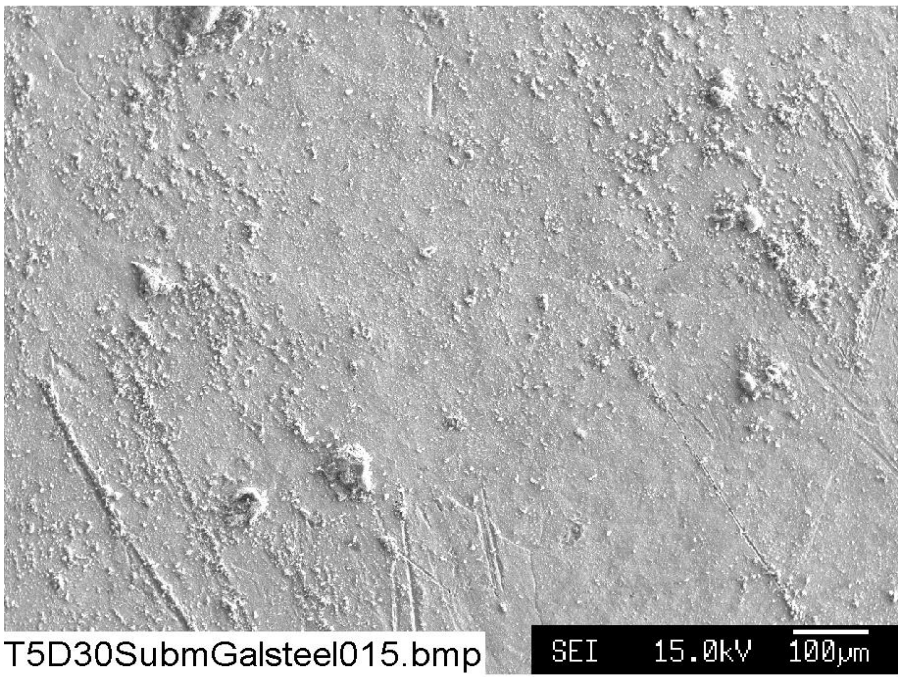

Figure E3-5: SEM image magnified 100 times for a Test #5 Day-30 submerged galvanized steel coupon sample. (T5D30SubmGalsteel015.bmp)

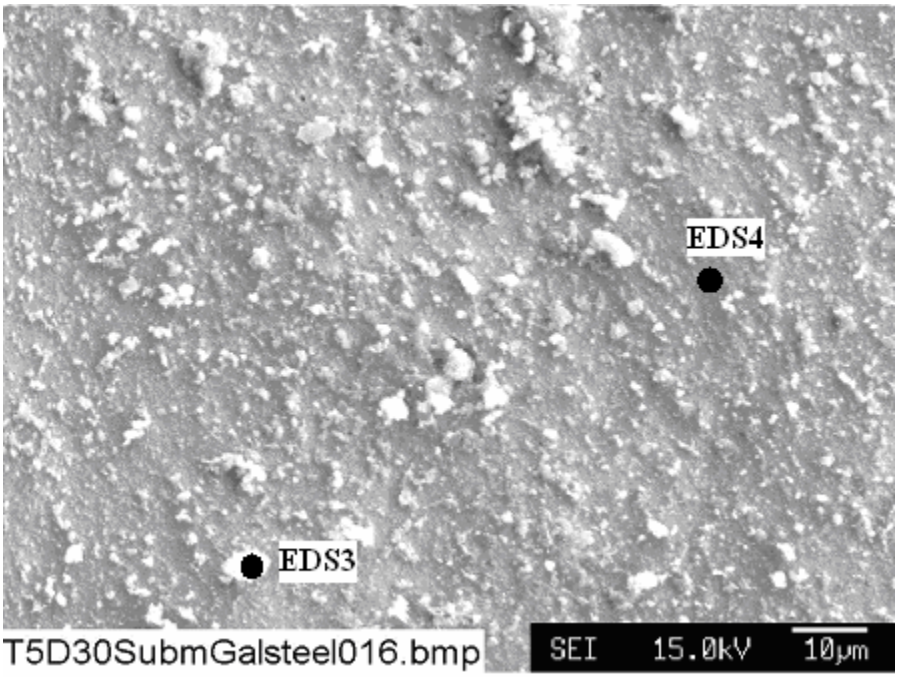

Figure E3-6: Annotated SEM image magnified 1000 times for a Test #5 Day-30 submerged galvanized steel coupon sample. (T5D30SubmGalsteel016.bmp)

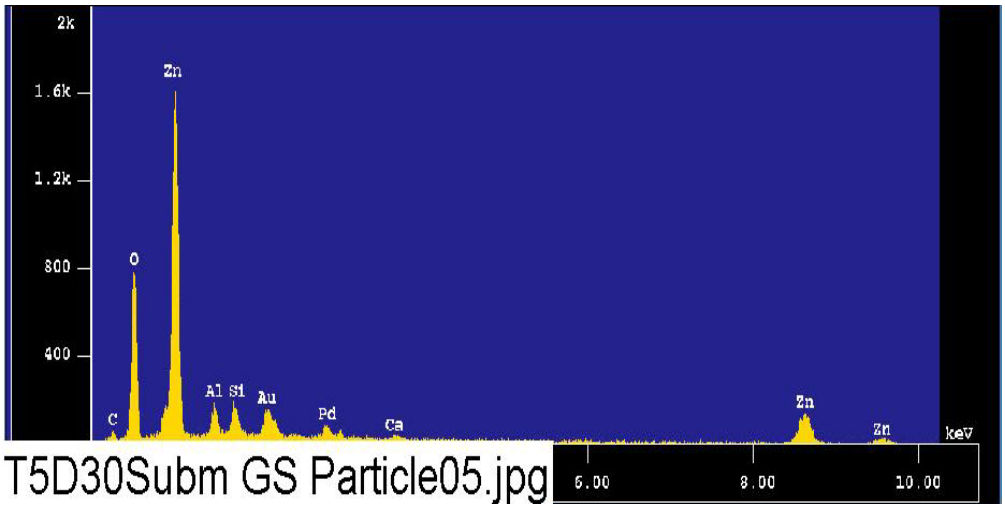

Figure E3-7: EDS counting spectrum for the light deposits (EDS3) on the submerged galvanized steel coupon surface shown in Figure E3-6. (T5D30Subm GS Particle05.jpg)

The results from the chemical composition analysis for T5D30Subm GS Particle05.jpg are given in Table E3-2

Table E3-3. Chemical Compositions for T5D30Subm GS Particle05.jpg, Figure E3-7

```
Group     : NRC
Sample    : T5D30  ID# : 11
Comment   : Particles on submerged Gal_steel surface
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  60.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.005E-09 A
            Stage Point : X=56.678 Y=62.490 Z=11.848
            Acq. Date   : Tue Aug 30 14:47:04 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Al K      Normal   1.26- 1.78    0.9951   0.0008     1109 /      70
 Si K      Normal   1.50- 2.07    0.9148   0.0005     1119 /     125
 Ca K      Normal   3.40- 4.30    0.4234   0.0049      250 /      25
 Zn K      Normal   8.19-10.06   17.3542   0.0070     1971 /       7
  C K      Normal   0.11- 0.47   19.5680   0.0121      197 /     121
  O K      Normal   0.31- 0.74   25.0700   0.0122    23626 /      28
-----------------------------------------------------------------
                              Chi_square =  5.5084

Element Mass%    Atomic%   ZAF      Z      A      F
    Al    2.628   2.0964 1.5366 0.9406 1.6342 0.9996
    Si    2.296   1.7594 1.4602 0.9328 1.5654 1.0000
    Ca    0.685   0.3677 0.9409 0.9394 1.0025 0.9991
    Zn   36.016  11.8565 1.2072 1.2114 0.9965 1.0000
     C   12.092  21.6651 0.3595 0.9447 0.3805 1.0000
     O   46.283  62.2547 1.0739 0.9412 1.1410 1.0000
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.7191
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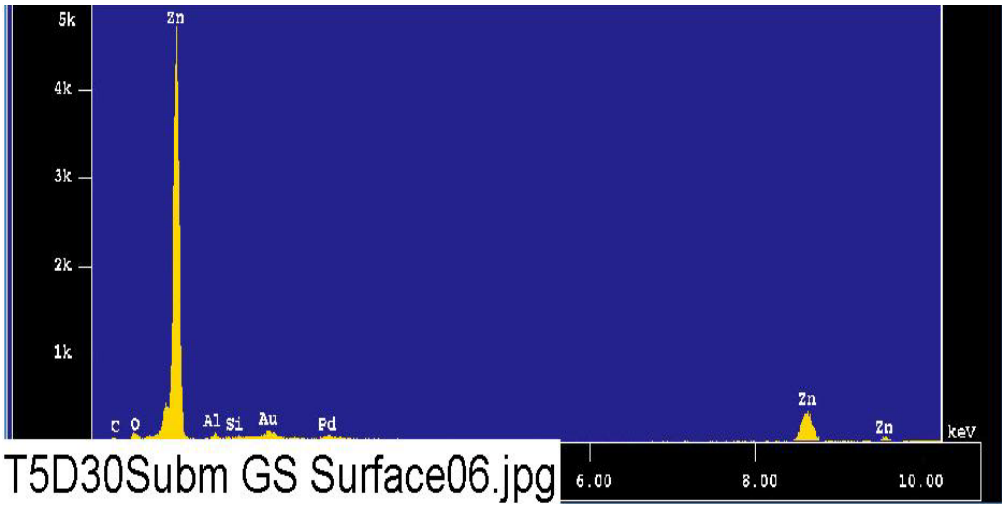

Figure E3-8: EDS counting spectrum for the grey coupon surface (EDS4) of the submerged galvanized steel shown in Figure E3-6. (T5D30Subm GS Surface06.jpg)

The results from the chemical composition analysis for T5D30Subm GS Surface06.jpg are given in Table E3-4

Table E3-4. Chemical Compositions for T5D30Subm GS Surface06.jpg, Figure E3-8

```
Group     : NRC
Sample    : T5D30  ID# : 12
Comment   : Surface of submerged Gal_steel
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  60.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.006E-09 A
            Stage Point : X=56.678 Y=62.490 Z=11.848
            Acq. Date   : Tue Aug 30 14:52:18 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Al K      Normal   1.26- 1.78    0.4278   0.0007      477 /      58
 Si K      Normal   1.50- 2.07    0.1268   0.0004      155 /      91
 Zn K      Normal   8.19-10.06   45.4077   0.0105     5163 /      10
  O K      Normal   0.31- 0.74    2.7433   0.0065     2588 /      58
  C K      Normal   0.11- 0.47   21.6706   0.0104      219 /      36
-----------------------------------------------------------------
                              Chi_square =  1.5563

Element Mass%    Atomic%   ZAF      Z      A      F
    Al    1.263   1.6261 1.8551 0.8522 2.1770 1.0000
    Si    0.331   0.4087 1.6372 0.8446 1.9384 1.0000
    Zn   77.389  41.1116 1.0705 1.0718 0.9988 1.0000
     O    5.427  11.7800 1.2426 0.8547 1.4540 0.9999
     C   15.590  45.0735 0.4519 0.8583 0.5265 1.0000
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.5921
```

Appendix E4

SEM/EDS Data for Test #5 Day-30 Steel Coupons

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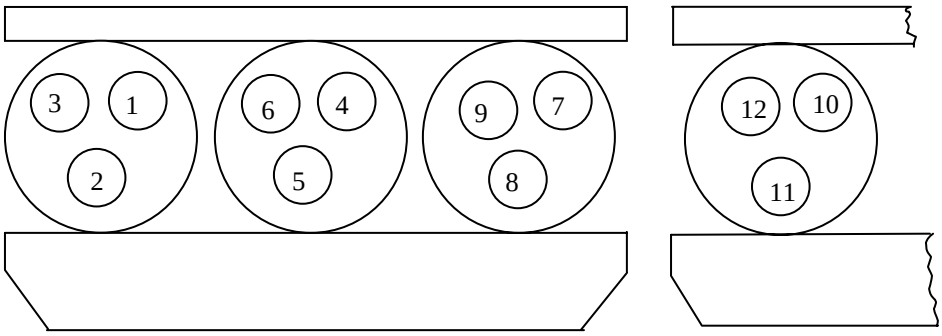
This appendix shows the SEM/EDS results for the metal steel coupons under two categories: (1) unsubmerged; (2) submerged. Unsubmerged coupons were only contacted with the solution during the initial 4-hour spray phase. In addition, the surface of the unsubmerged coupons may be affected during the test by the moist air in the tank gas space. Submerged refers to the coupons submerged in the solution during the test.

The coupon samples were collected on August 25, 2005 (the date Test #5 was shut down) and examined by SEM/EDS on August 30 and September 6, 2005. The steel coupon samples were dried in air before coating with Au/Pd for SEM examination. SEM results present the surface condition of the steel coupons. In addition, EDS results provide a semi-quantitative elemental analysis of the coupon surface and the corrosion products.

Transcribed Laboratory Log

Laboratory session from September 6, 2005.
SEM Test #5 Day-30 Steel Coupons.

Conditions: e=15.0kV, WD=11mm



1--Yellow Deposits on Submerged Rack	2--Sediment (T5D30)	3--Al-Unsubmerged
4--Al-Submerged	5--Gal-Steel Unsubmerged	6--Gal-Steel Submerged
7--Cu Unsubmerged	8--Cu-Submerged	9--Steel-Unsubmerged
10--Steel-Submerged	11--Drain Collar Interior	12--Drain Collar Outside Ext.

Unsubmerged Steel Coupon.

Image:	T5D30SuspSteel021	100 ×	SEM image	Figure E4-1
	T5D30SuspSteel022	1000 ×	SEM image higher magnification	Figure E4-2
EDS:	T5D30SuspSteel023	1000 ×	SEM annotated image	Figure E4-3
	T5D30Susp Steel Surface11		On smooth surface shown in image 023	Figure E4-4
	T5D30Susp Steel Particle12		On white particles shown in image 023	Figure E4-5

Submerged Steel Coupon.

Image:	T5D30SubmSteel024	100 ×	SEM image of fiberglass	Figure E4-6
	T5D30SubmSteel025	1000 ×	SEM Annotated image	Figure E4-7
EDS:	T5D30Subm Steel light particle13		EDS of light particles in 025	Figure E4-8
	T5D30Subm Steel dark particle14		EDS of dark particle in 025	Figure E4-9
	T5D30Subm Steel Surface15		EDS of surface in 025	Figure E4-10

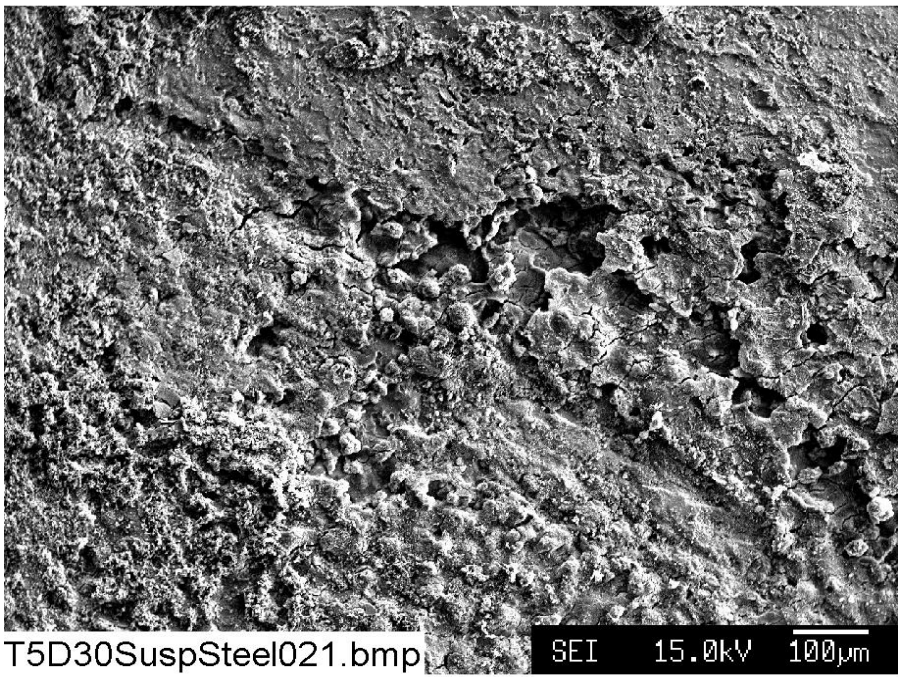


Figure E4-1: SEM image magnified 100 times for a Test #5 Day-30 unsubmerged steel coupon sample. (T5D30SuspSteel021.bmp)

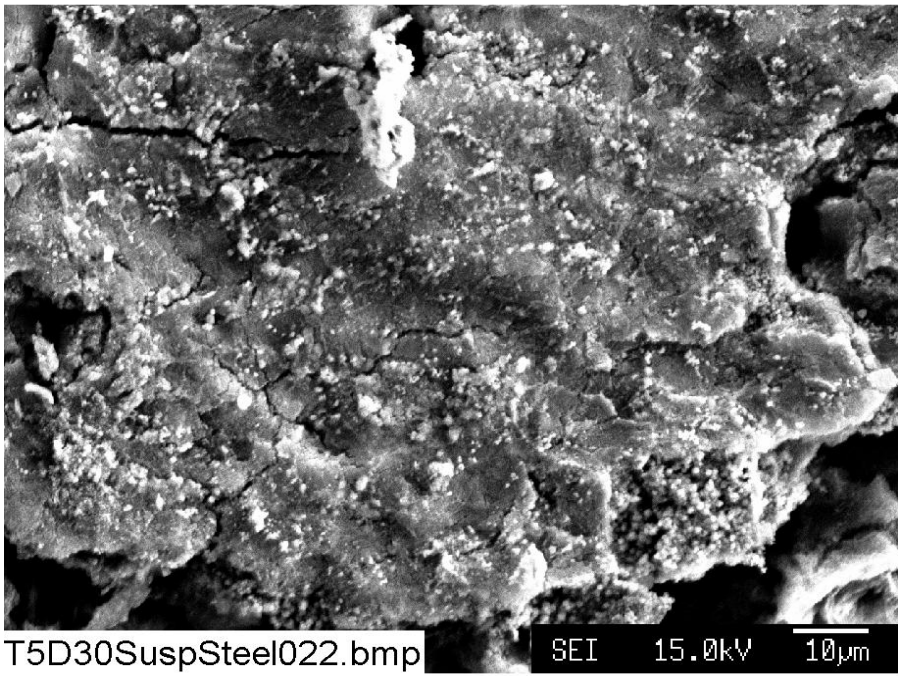


Figure E4-2: SEM image magnified 1000 times for a Test #5 Day-30 unsubmerged steel coupon sample. (T5D30SuspSteel022.bmp)

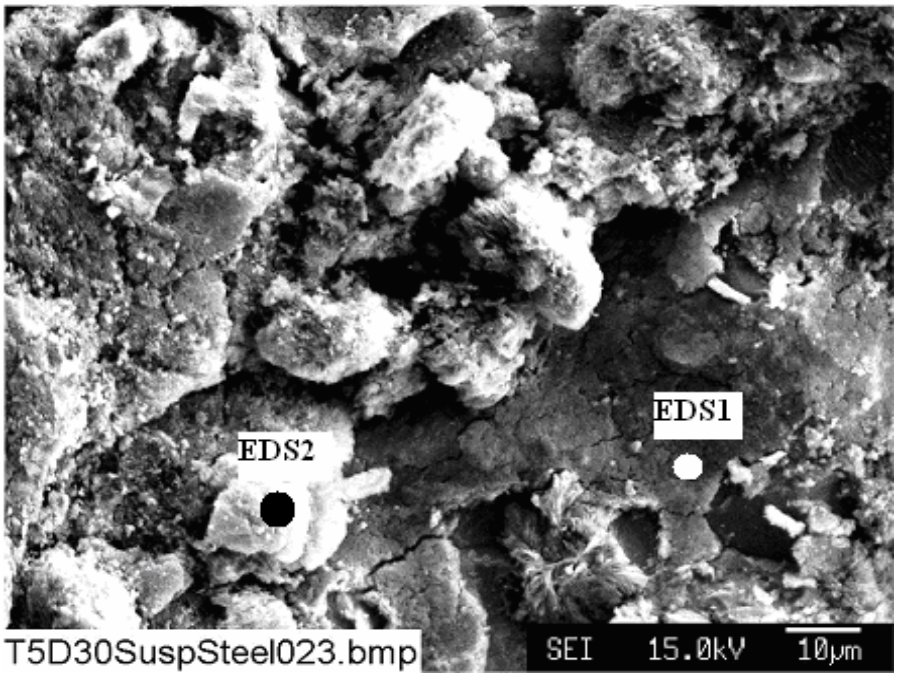


Figure E4-3: Annotated SEM image magnified 1000 times for a Test #5 Day-30 unsubmerged steel coupon sample. (T5D30SuspSteel023.bmp)

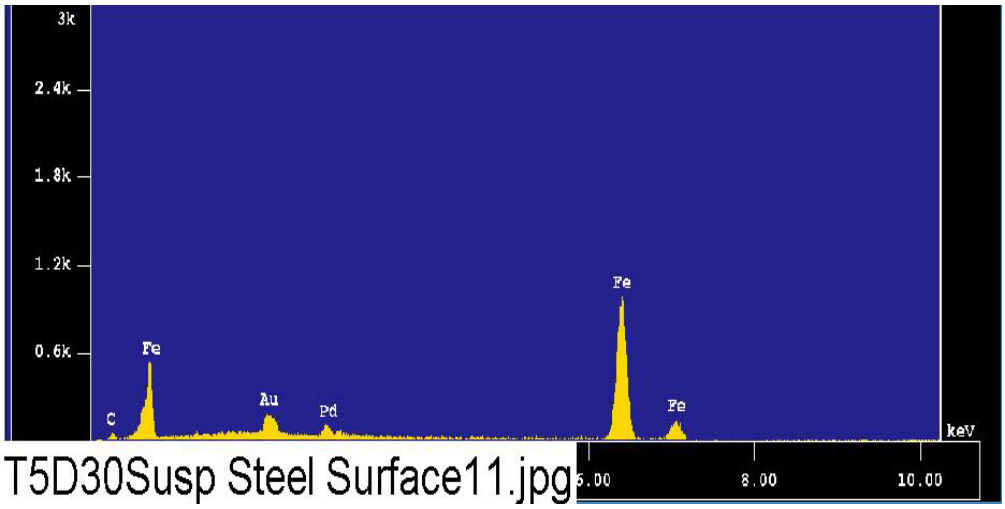


Figure E4-4: EDS counting spectrum for the dark coupon surface (EDS1) of the unsubmerged steel shown in Figure E4-3. (T5D30Susp Steel Surface11.jpg)

The results from the chemical composition analysis for T5D30Susp Steel Surface11.jpg are given in Table E4-1

Table E4-1. Chemical Compositions for T5D30Susp Steel Surface11.jpg, Figure E4-4

```
Group     : NRC
Sample    : T5D30  ID# : 17
Comment   : Surface of Suspended Steel
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  60.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.004E-09 A
            Stage Point : X=21.743 Y=66.867 Z=11.848
            Acq. Date   : Tue Aug 30 15:38:16 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Fe K      Normal   6.04- 7.40   57.1720   0.0094    15164 /      18
  C K      Normal   0.11- 0.47   25.4488   0.0098      256 /      25
-------------------------------------------------------------------
                             Chi_square =  3.9338

Element Mass%    Atomic%   ZAF      Z      A      F
    Fe   89.763  65.3492 1.0215 1.0227 0.9989 1.0000
     C   10.237  34.6509 0.2617 0.8592 0.3046 1.0000
-------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.5370
```

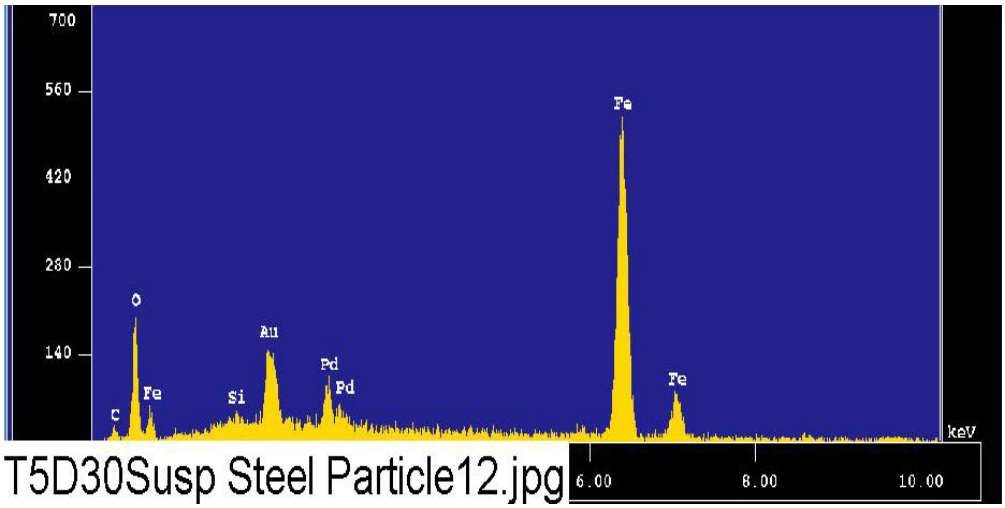


Figure E4-5: EDS counting spectrum for the light particle (EDS2) on the unsubmerged steel coupon surface shown in Figure E4-3. (T5D30Susp Steel Particle12.jpg)

The results from the chemical composition analysis for T5D30Susp Steel Particle12.jpg are given in Table E4-2

Table E4-2. Chemical Compositions for T5D30Susp Steel Particle12.jpg, Figure E4-5

```
Group     : NRC
Sample    : T5D30  ID# : 18
Comment   : Particles on Suspended Steel
Condition : Full Scale   : 20KeV(10eV/ch,2Kch)
            Live Time    :  60.000 sec     Aperture #     : 1
            Acc. Volt    : 15.0 KV         Probe Current : 1.005E-09 A
            Stage Point  : X=21.743 Y=66.867 Z=11.848
            Acq. Date    : Tue Aug 30 15:43:38 2005


Element      Mode      ROI(KeV)    K-ratio(%)  +/-    Net/Background
 Si K       Normal    1.50- 2.07     0.1045   0.0003      128 /      47
 Fe K       Normal    6.04- 7.40    29.4350   0.0070     7815 /      13
  C K       Normal    0.11- 0.47    15.9559   0.0066      161 /      35
  O K       Normal    0.31- 0.74     6.1537   0.0065     5799 /      21
-------------------------------------------------------------------
                              Chi_square =  2.8510

Element Mass%     Atomic%    ZAF       Z       A       F
    Si    0.348    0.4060 1.3604 0.8714 1.5612 0.9999
    Fe   76.176   44.6485 1.0562 1.0587 0.9976 1.0000
     C   10.179   27.7411 0.2604 0.8849 0.2942 1.0000
     O   13.297   27.2045 0.8818 0.8813 1.0011 0.9995
-------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  2.4503
```

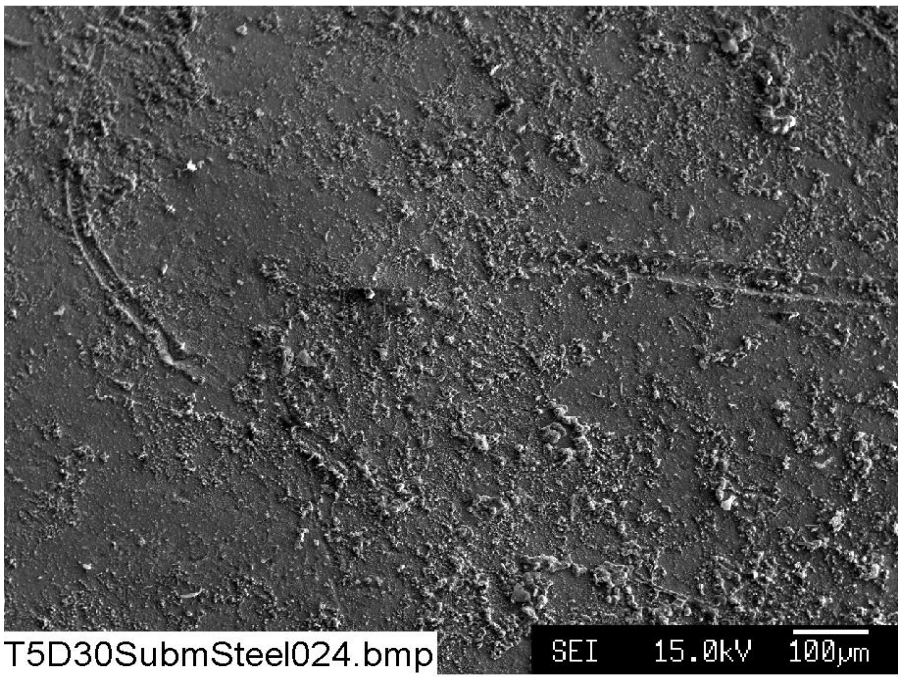


Figure E4-6: SEM image magnified 100 times for a Test #5 Day-30 submerged steel coupon sample. (T5D30SubmSteel024.bmp)

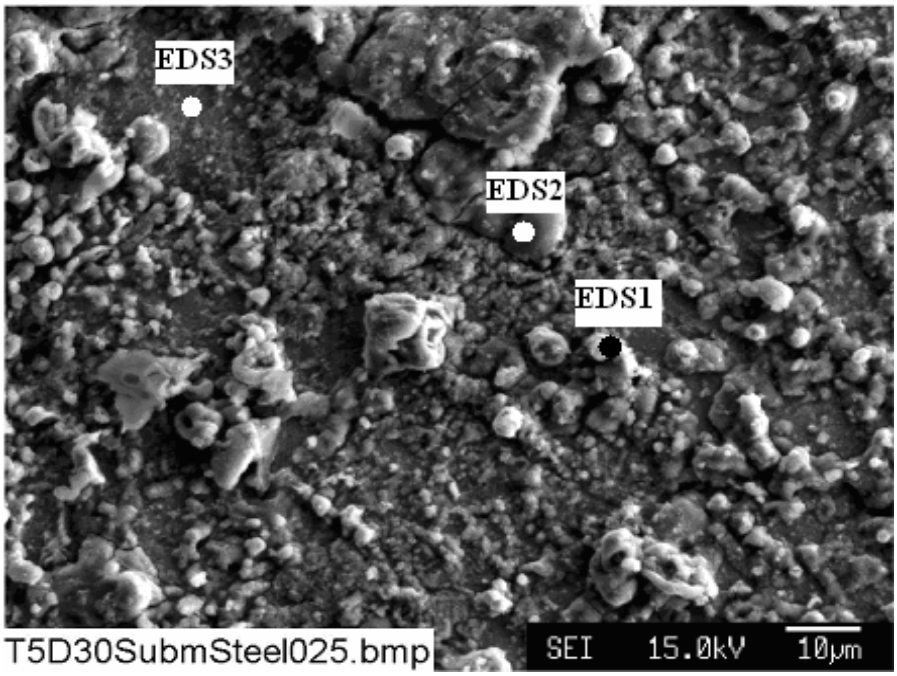


Figure E4-7: Annotated SEM image magnified 1000 times for a Test #5 Day-30 submerged steel coupon sample. (T5D30SubmSteel025.bmp)

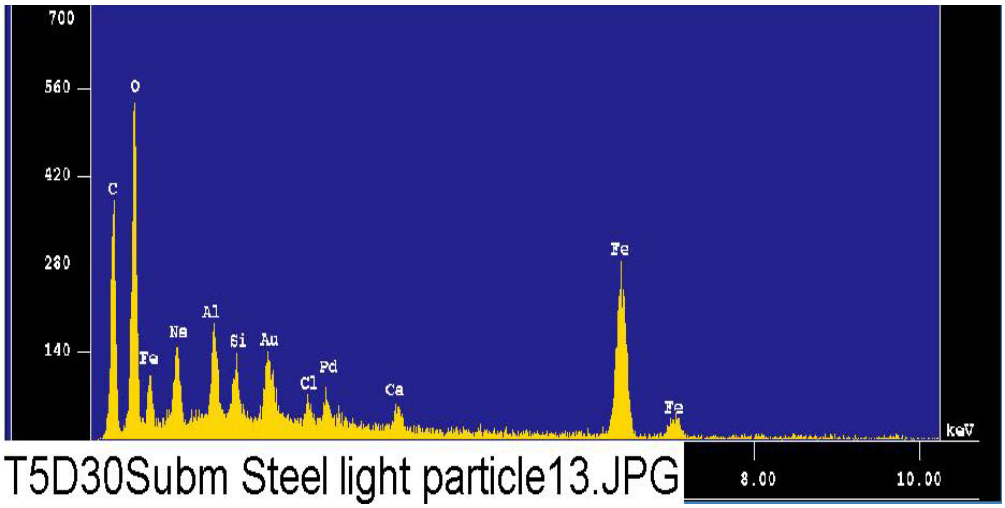


Figure E4-8: EDS counting spectrum for the light particle (EDS1) on the submerged steel coupon surface shown in Figure E4-7. (T5D30Subm Steel light particle13.jpg)

The results from the chemical composition analysis for T5D30Subm Steel light particle13.jpg are given in Table E4-3

Table E4-3. Chemical Compositions for T5D30Subm Steel light particle13.jpg, Figure E4-8

```
Group     : NRC
Sample    : T5D30  ID# : 19
Comment   : Light particle on submerged Steel
Condition : Full Scale   : 20KeV(10eV/ch,2Kch)
            Live Time    :  60.000 sec    Aperture #    : 1
            Acc. Volt    : 15.0 KV        Probe Current : 1.003E-09 A
            Stage Point  : X=76.455 Y=54.980 Z=11.000
            Acq. Date    : Tue Aug 30 16:26:04 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
  C K      Normal   0.11- 0.47  255.6311   0.0237     2571 /      99
  O K      Normal   0.35- 0.72   14.1923   0.0039     3803 /      62
 Na K      Normal   0.83- 1.28    1.2501   0.0063      923 /      34
 Al K      Normal   1.26- 1.78    1.1529   0.0008     1283 /      66
 Si K      Normal   1.50- 2.07    0.6334   0.0009      704 /     119
 Cl K      Normal   2.32- 3.09    0.5137   0.0007      485 /      36
 Ca K      Normal   3.40- 4.30    0.8103   0.0053      477 /      27
 Fe K      Normal   6.04- 7.40   15.7888   0.0051     4184 /       6
-------------------------------------------------------------------
                              Chi_square =  3.2813

Element Mass%    Atomic%   ZAF      Z      A      F
     C   49.124  65.0147 0.1870 0.9620 0.1944 1.0000
     O   26.855  26.6826 1.8416 0.9574 1.9235 0.9999
    Na    1.678   1.1603 1.3065 0.9520 1.3704 1.0015
    Al    1.365   0.8042 1.1523 0.9602 1.2004 0.9998
    Si    0.695   0.3932 1.0673 0.9487 1.1251 0.9999
    Cl    0.522   0.2339 0.9882 1.0049 0.9842 0.9991
    Ca    0.770   0.3053 0.9247 0.9632 0.9661 0.9937
    Fe   18.991   5.4057 1.1706 1.1807 0.9915 1.0000
-------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.0275
```

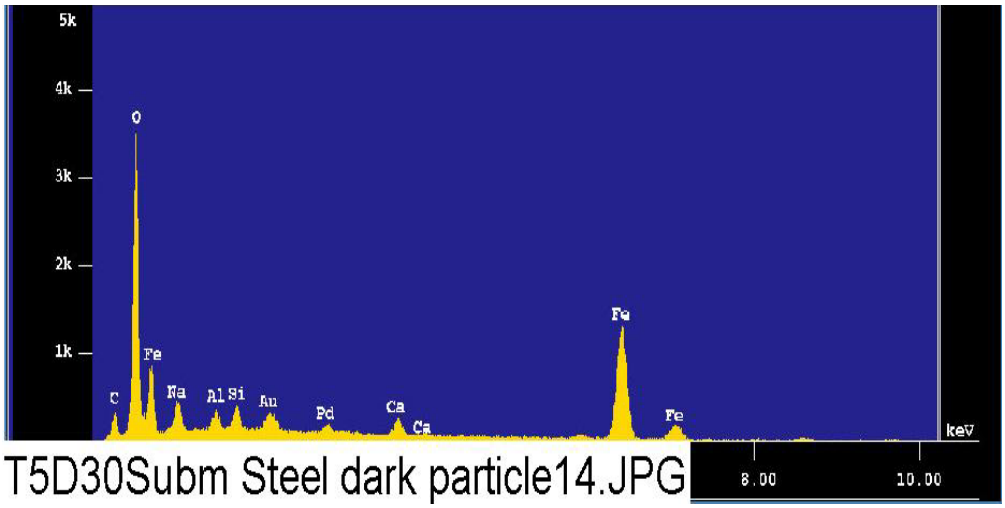


Figure E4-9: EDS counting spectrum for the dark particle (EDS2) on the submerged steel coupon surface shown in Figure E4-7. (T5D30Subm Steel dark particle14.jpg)

The results from the chemical composition analysis for T5D30Subm Steel dark particle14.jpg are given in Table E4-4

Table E4-4. Chemical Compositions for T5D30Subm Steel dark particle14.jpg, Figure E4-9

```
Group     : NRC
Sample    : T5D30  ID# : 20
Comment   : Dark particle on submerged Steel
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 191.130 sec    Aperture #     : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.003E-09 A
            Stage Point : X=76.455 Y=54.980 Z=11.000
            Acq. Date   : Tue Aug 30 17:08:10 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Na K      Normal   0.83- 1.28    0.9443   0.0114     2221 /     116
 Al K      Normal   1.26- 1.78    0.5316   0.0013     1884 /     212
 Si K      Normal   1.50- 2.07    0.5549   0.0008     2159 /     284
 Ca K      Normal   3.40- 4.30    1.2563   0.0109     2354 /      73
 Fe K      Normal   6.04- 7.40   24.3440   0.0113    20548 /      36
  O K      Normal   0.31- 0.74   33.9978   0.0267   101858 /     292
  C K      Normal   0.11- 0.47   58.5353   0.0260     1876 /     551
-------------------------------------------------------------------
                            Chi_square = 20.3524

Element Mass%    Atomic%   ZAF      Z      A      F
    Na    1.969   1.7446 1.6775 0.9412 1.7796 1.0015
    Al    0.872   0.6582 1.3195 0.9486 1.3913 0.9998
    Si    0.890   0.6453 1.2897 0.9408 1.3709 0.9999
    Ca    1.439   0.7313 0.9214 0.9484 0.9798 0.9916
    Fe   34.836  12.7034 1.1509 1.1573 0.9945 1.0000
     O   43.074  54.8293 1.0190 0.9488 1.0741 0.9999
     C   16.919  28.6879 0.2325 0.9522 0.2442 0.9999
-------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.2433
```

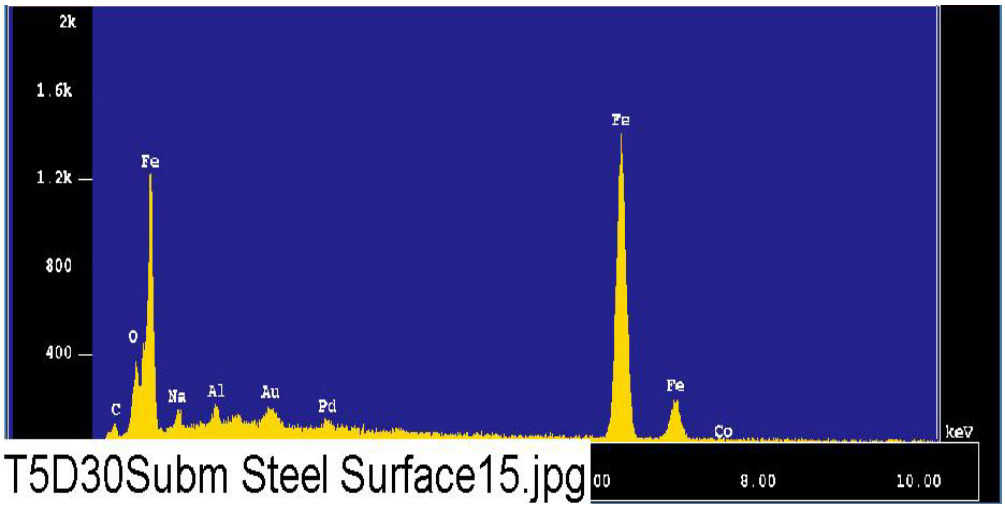


Figure E4-10: EDS counting spectrum for the submerged steel coupon surface (EDS3) shown in Figure E4-7. (T5D30Subm Steel Surface15.jpg)

The results from the chemical composition analysis for T5D30Subm Steel Surface15.jpg are given in Table E4-5

Table E4-5. Chemical Compositions for T5D30Subm Steel Surface15.jpg, Figure E4-10

```
Group     : NRC
Sample    : T5D30  ID# : 21
Comment   : Surface of submerged Steel
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  83.100 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.003E-09 A
            Stage Point : X=76.455 Y=54.980 Z=11.000
            Acq. Date   : Tue Aug 30 17:15:50 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Na K      Normal   0.83- 1.28    0.6403   0.0069      655 /      72
 Fe K      Normal   6.04- 7.40   58.7678   0.0113    21567 /      30
 Co K      Normal   6.53- 8.02    0.1879   0.0017       64 /      66
  O K      Normal   0.31- 0.74    5.4760   0.0120     7133 /     270
  C K      Normal   0.11- 0.47   28.0267   0.0128      390 /      86
 Al K      Normal   1.26- 1.78    0.5087   0.0009      784 /     108
-----------------------------------------------------------------
                            Chi_square =  8.3983

Element Mass%    Atomic%   ZAF      Z      A      F
    Na    1.768   2.7579 2.0987 0.8635 2.4268 1.0016
    Fe   80.660  51.7984 1.0432 1.0450 0.9983 1.0000
    Co    0.264   0.1607 1.0680 1.0710 0.9972 1.0000
     O    6.395  14.3354 0.8877 0.8717 1.0189 0.9994
     C    9.925  29.6345 0.2692 0.8754 0.3075 1.0000
    Al    0.988   1.3132 1.4761 0.8693 1.6981 1.0000
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.3156
```

Appendix F

SEM/EDS Data for Test #5 Day-30 Sediment

List of Figures

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Particulate sediments at the bottom of the tank directly relate to the corrosion products and debris generated during ICET tests. This appendix presents the probe SEM/EDS and XRD/XRF results for the sediment samples collected from the bottom of the tank on the date Test #5 was shut down (August 25, 2005). The purpose of these analyses is to provide the information of the morphology and the composition of the sediments.

The sediment samples were dried in air before coating with Au/Pd for probe SEM examination. EDS results provide a semi-quantitative elemental composition of the sediment. The SEM/EDS results of the Test #5 Day-30 sediment samples were obtained on September 6, 2005. XRD and XRF analysis was performed on October 3 and August 30, 2005, respectively. Based on XRD results, the sediment sample contained crystalline substance of quartz, which is likely from fiberglass debris. XRF results show the chemical composition of the sediment.

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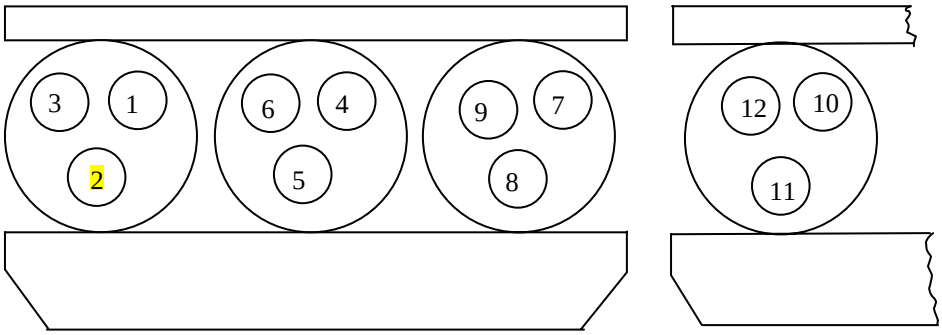
Transcribed Laboratory Log

Laboratory session from September 6, 2005.

SEM Test #5 Day-30 Sediment

Conditions: e=15.0kV, WD=11mm



1--Yellow Deposits on Submerged Rack	2--Sediment (T5D30)	3--Al-Suspended
4--Al-Submerged	5--Gal-Steel Suspended	6--Gal-Steel Submerged
7--Cu Suspended	8--Cu-Submerged	9--Steel-Suspended
10--Steel-Submerged	11--Drain Collar Interior	12--Drain Collar Outside Ext.

Sediment Sample

Image:	T5D30Sediment004	70 ×	Annotated SEM image	Figure F-1
EDS:	T5Sedmt03		EDS on big particle shown in 004	Figure F-2
Image:	T5D30Sediment005	300 ×	Annotated SEM image	Figure F-3
EDS:	T5Sedmt04		EDS on particles shown in 005	Figure F-4
Image:	T5D30Sediment006	600 ×	SEM at higher magnification	Figure F-5

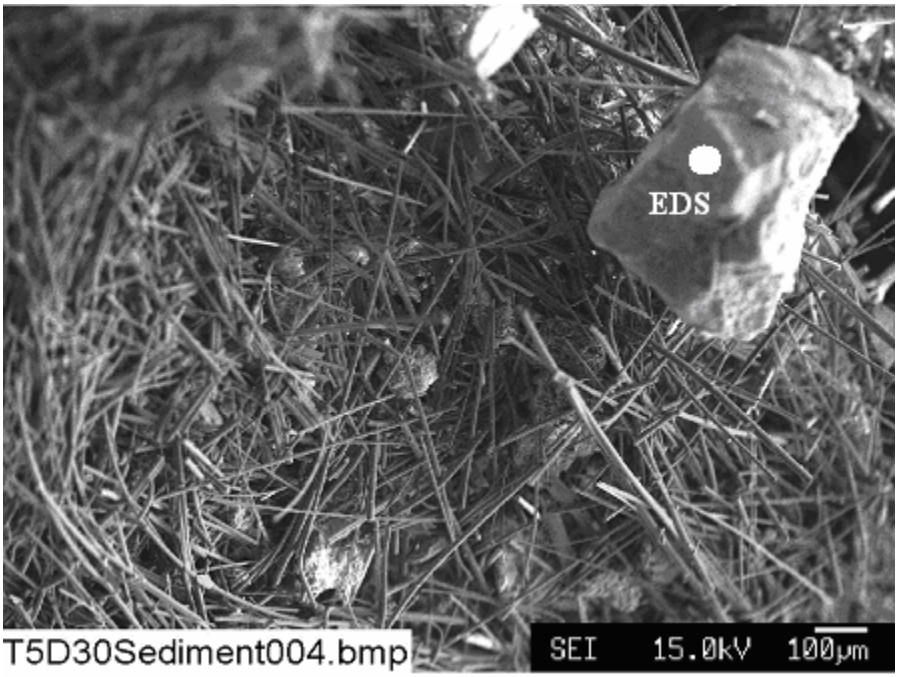


Figure F-1: Annotated SEM image magnified 70 times for the Test #5 Day-30 sediment at the bottom of the tank. (T5D30Sediment004.bmp)

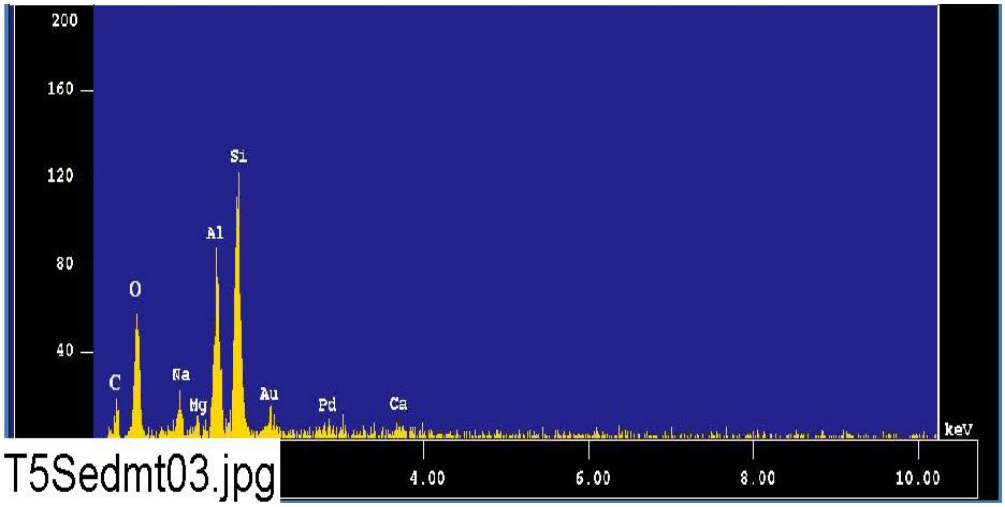


Figure F-2: EDS counting spectrum for the big particulate deposit shown in Figure F-1. (T5Sedmt03.jpg)

The results from the chemical composition analysis for T5Sedmt03.jpg are given in Table F-1

Table F-1. Chemical Compositions for T5Sedmt03.jpg, Figure F-2

```
Group     : NRC
Sample    : T5D30  ID# : 31
Comment   : Big particle in sediment
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 174.760 sec    Aperture #    : 5
            Acc. Volt   : 15.0 KV        Probe Current : 1.996E-08 A
            Stage Point : X=45.414 Y=58.152 Z=11.027
            Acq. Date   : Tue Sep  6 11:10:45 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Na K      Normal   0.83- 1.28    3.5839   0.0020      121 /       4
 Mg K      Normal   1.00- 1.53    1.2764   0.0001       37 /      21
 Al K      Normal   1.26- 1.78   12.2138   0.0005      622 /      33
 Si K      Normal   1.50- 2.07   18.2484   0.0003     1021 /      39
 Ca K      Normal   3.40- 4.30    2.8124   0.0018       76 /       2
  O K      Normal   0.31- 0.74   38.3211   0.0034     1650 /       2
  C K      Normal   0.11- 0.47  242.5716   0.0044      112 /      10
-----------------------------------------------------------------
                            Chi_square =  1.9507

Element Mass%    Atomic%   ZAF      Z      A      F
    Na    1.925   1.2891 0.9857 0.9838 1.0014 1.0006
    Mg    0.918   0.5814 1.3200 0.9736 1.3595 0.9973
    Al    6.842   3.9044 1.0282 0.9924 1.0399 0.9963
    Si   11.585   6.3513 1.1653 0.9848 1.1833 1.0000
    Ca    1.488   0.5718 0.9714 0.9964 0.9748 1.0001
     O   36.681  35.3029 1.7570 0.9908 1.7731 1.0000
     C   40.561  51.9992 0.3069 0.9939 0.3088 1.0000
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  0.5448
```

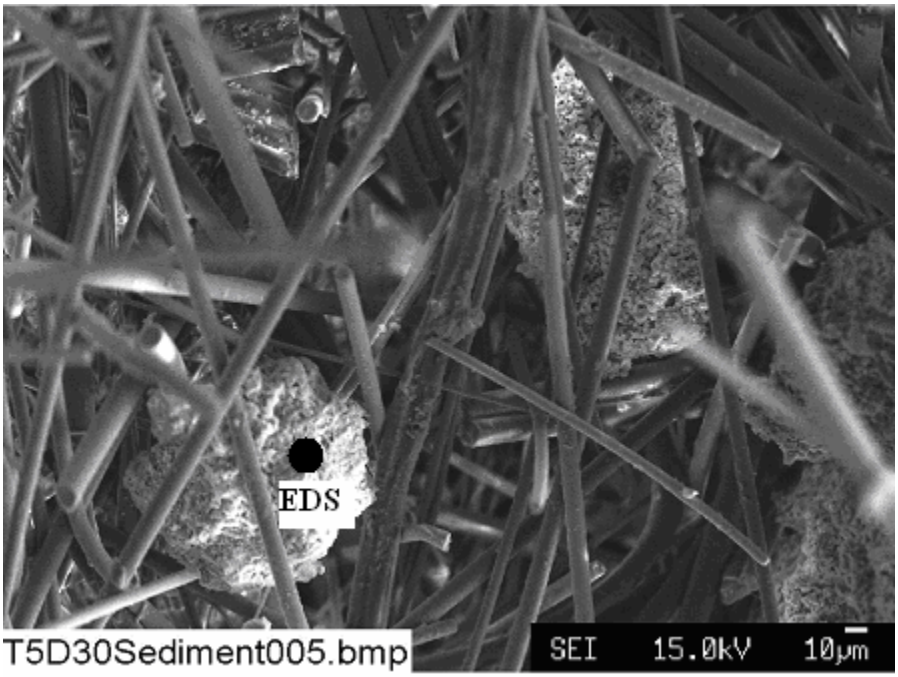


Figure F-3: Annotated SEM image magnified 300 times for the Test #5 Day-30 sediment at the bottom of the tank. (T5D30Sediment005.bmp)

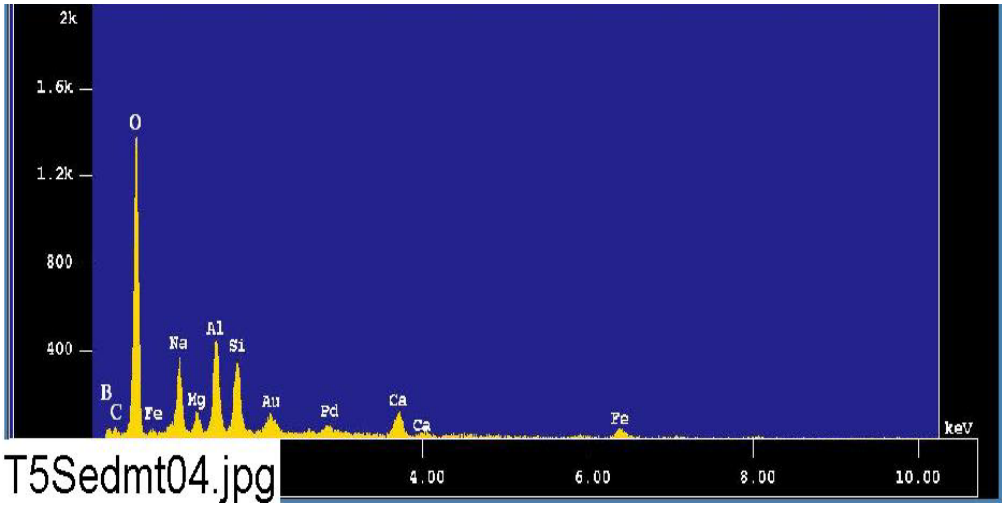


Figure F-4: EDS counting spectrum for the particulate deposit shown in Figure F-3. (T5Sedmt04.jpg)

The results from the chemical composition analysis for T5Sedmt04.jpg are given in Table F-2

Table F-2 Chemical Compositions for T5Sedmt04.jpg, Figure F-4

```
Group     : NRC
Sample    : T5D30  ID# : 32
Comment   : Small particle in sediment
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  84.130 sec    Aperture #    : 5
            Acc. Volt   : 15.0 KV        Probe Current : 1.996E-08 A
            Stage Point : X=45.414 Y=58.152 Z=11.027
            Acq. Date   : Tue Sep  6 11:20:28 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
 Na K      Normal   0.83- 1.28  153.4448   0.0087     2497 /      60
 Mg K      Normal   1.00- 1.53   61.4743   0.0005      850 /     209
 Al K      Normal   1.26- 1.78  151.9680   0.0012     3728 /     172
 Si K      Normal   1.50- 2.07  114.4332   0.0006     3081 /     250
 Ca K      Normal   3.40- 4.30  110.9579   0.0070     1439 /      18
 Fe K      Normal   6.04- 7.40   94.8041   0.0025      554 /       8
  O K      Normal   0.31- 0.74  1963.2505   0.0155    40703 /      31
  C K      Normal   0.11- 0.47  742.3510   0.0103      165 /     210
  B K      Normal   0.00- 0.40  8313.1895   0.2895       28 /      22
-----------------------------------------------------------------
                              Chi_square = 10.8445

Element Mass%    Atomic%   ZAF      Z      A      F
    Na    0.536   0.2721 0.7359 0.8962 0.8200 1.0014
    Mg    0.302   0.1447 1.0326 0.8873 1.1642 0.9997
    Al    0.607   0.2624 0.8408 0.9049 0.9295 0.9997
    Si    0.503   0.2090 0.9259 0.8984 1.0307 1.0000
    Ca    0.453   0.1317 0.8587 0.9126 0.9413 0.9997
    Fe    0.500   0.1045 1.1112 1.1262 0.9867 1.0000
     O   15.144  11.0404 1.6238 0.9016 1.8010 1.0001
     C    5.435   5.2784 1.5413 0.9038 1.7053 1.0000
     B   76.520  82.5568 1.9377 1.0331 1.8757 1.0000
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  0.0047
```

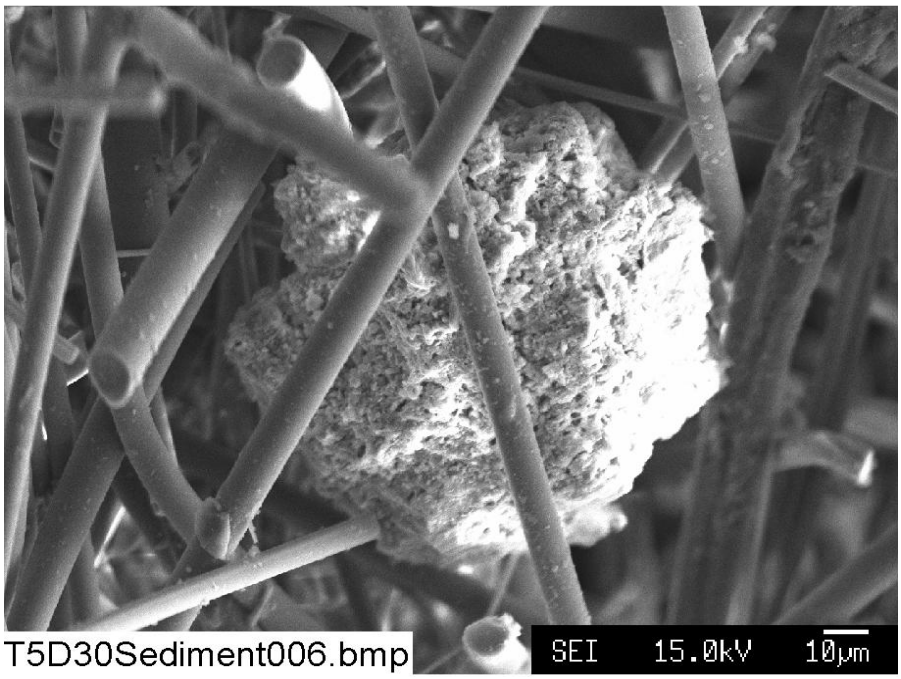


Figure F-5: SEM image magnified 600 times for the Test #5 Day-30 sediment at the bottom of the tank. (T5D30Sediment006.bmp)

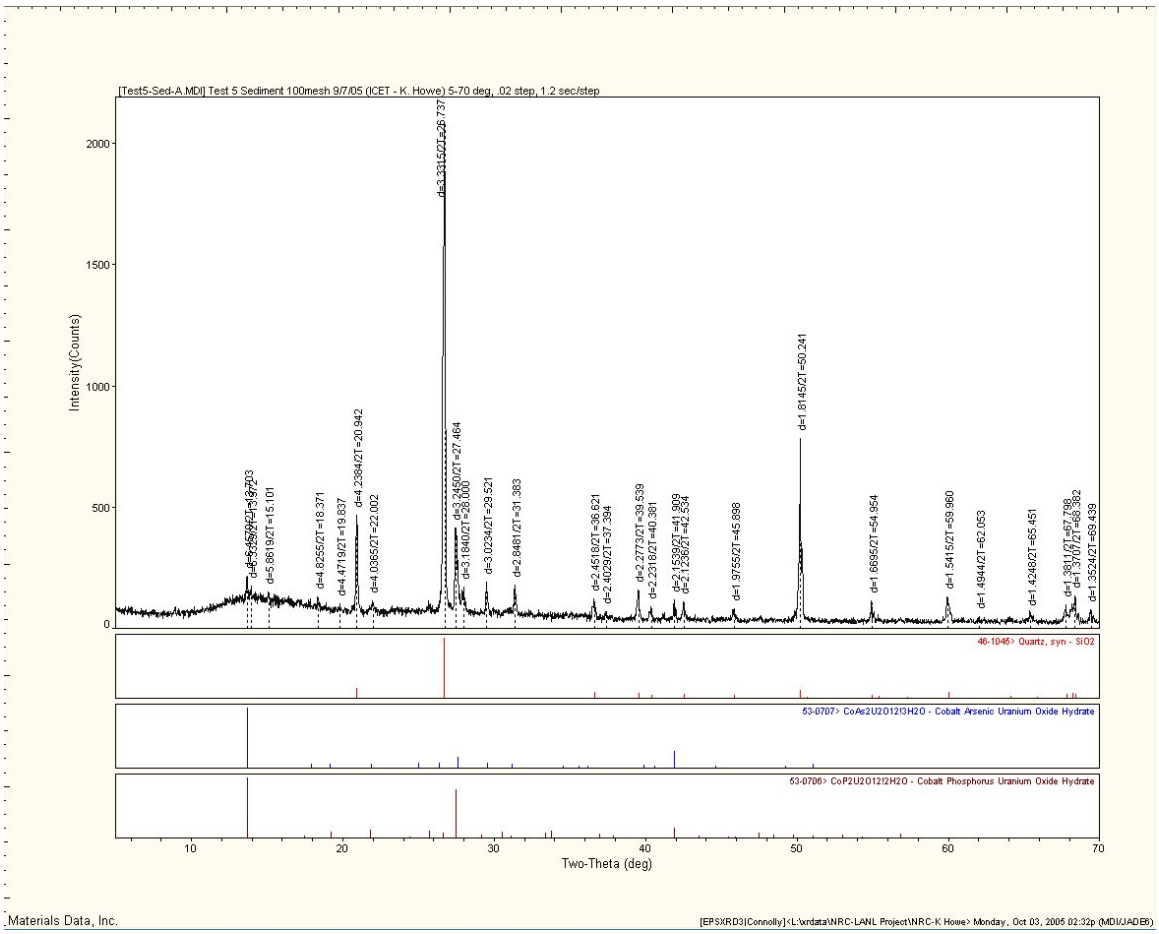


Figure F-6: The XRD result of Test #5 Day-30 sediment.

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Table F-3. Dry Mass Composition of the Test #5 Day-30 Sediment Sample by XRF Analysis.

Sample ID	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	$H_20(-)$	$H_2O(+)CO_2$	P_2O_5	Total	H2O(+)CO2 /DF (10) & Cover. To %
ICET Test #5	63.76	0.18	6.00	1.34	0.00	0.07	1.78	5.49	9.28	1.09	0.69	3.27	0.07	93.02	1.0033

Appendix G

TEM Data for Test #5 Solution Samples

List of Figures

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Figure G-3: TEM energy-dispersive x-ray spectrum for a Test #5 Day-4 unfiltered sample. The copper peak is likely from the copper sample holder for TEM analysis. (T5D4_UF_EDS.jpg) G-4

Figure G-4: TEM magnified 2,000 times for one Test #5 Day-15 unfiltered sample location. (T5D15~UF~2k.jpg) G-4

Figure G-5: Annotated TEM magnified 4,000 times for one Test #5 Day-15 unfiltered sample location. (T5D15~UF~4kEDS.jpg) G-5

Figure G-6: TEM energy-dispersive x-ray spectrum for a Test #5 Day-15 unfiltered sample. The copper peak is likely from the copper sample holder for TEM analysis. (T5D15_UF_EDS.jpg) G-5

Figure G-7: TEM magnified 2,000 times for one Test #5 Day-30 unfiltered sample location. (T5D30~UF~2k.jpg) G-6

Figure G-8: TEM magnified 4,000 times for one Test #5 Day-30 unfiltered sample location. (T5D30~UF~4k~2.jpg) G-6

Figure G-9: Annotated TEM magnified 8,000 times for one Test #5 Day-30 unfiltered sample location. (T5D30~UF~8k~EDS.jpg) G-7

Figure G-10: TEM energy-dispersive x-ray spectrum for a Test #5 Day-30 unfiltered sample. The copper peak is likely from the copper sample holder for TEM analysis. (T5D30_UF_EDS.jpg) G-7

Figure G-11: TEM magnified 10,000 times for one Test #5 Day-30 unfiltered sample location. (T5D30~UF~10k.jpg) G-8

This appendix presents TEM images and EDS results for Test #5 Day-4, Day-15, and Day-30 unfiltered solution samples. The unfiltered solution samples were directly extracted from the tank. A tiny drop of the testing solutions was transferred to a copper mesh followed by drying in air for TEM analysis. The TEM and EDS results were obtained on September 1, 2005.

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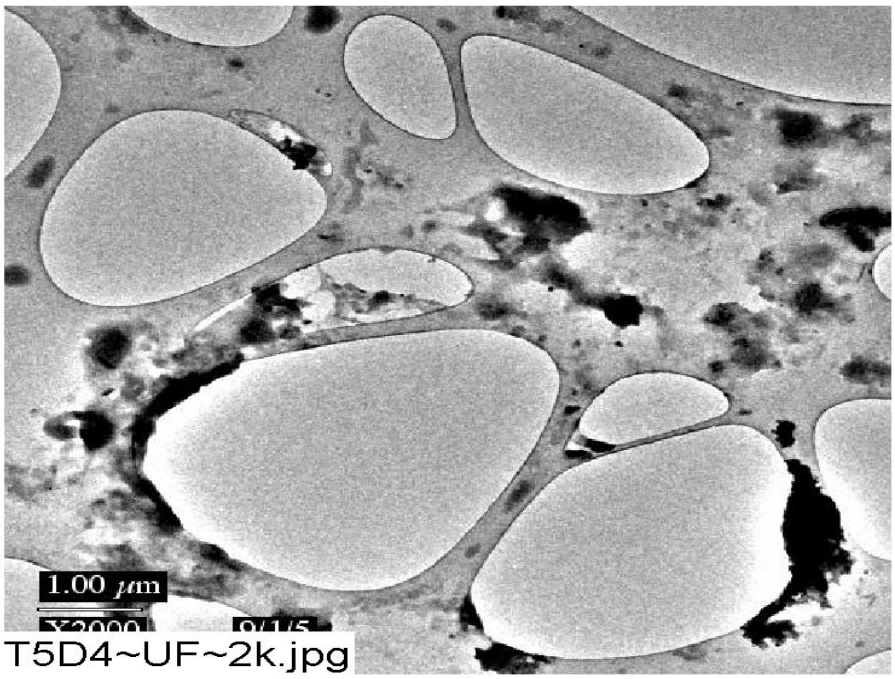

Figure G-1: TEM magnified 2,000 times for one Test #5 Day-4 unfiltered sample location. (T5D4~UF~2k.jpg)

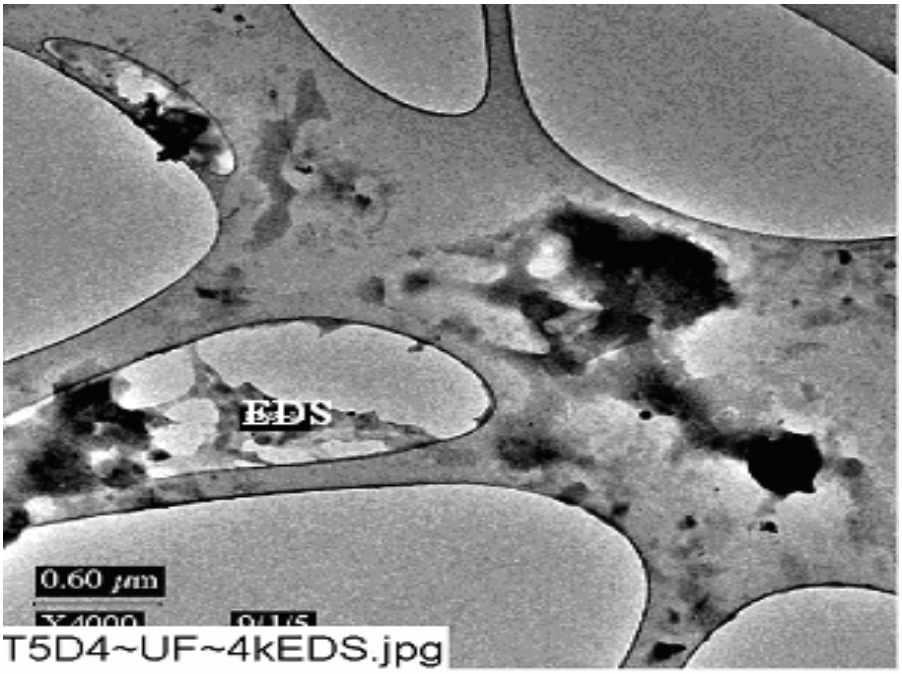

Figure G-2: Annotated TEM magnified 4,000 times for one Test #5 Day-4 unfiltered sample location. (T5D4~UF~4kEDS.jpg)

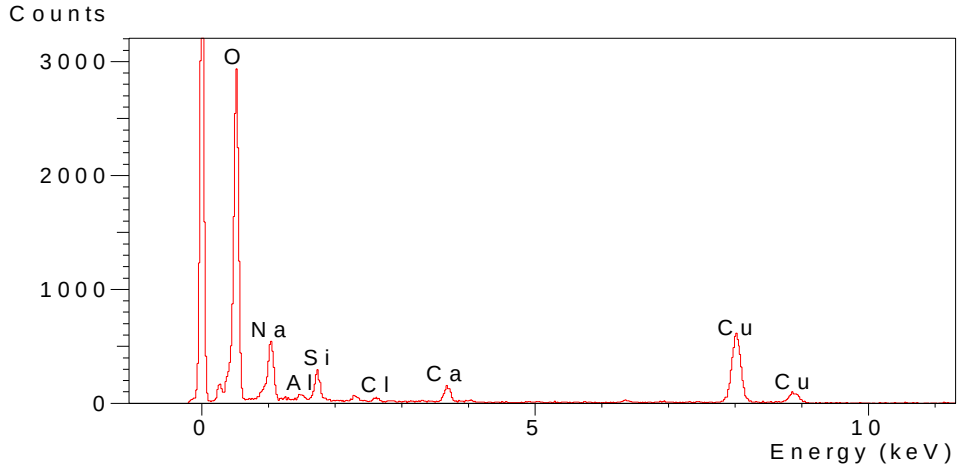

Figure G-3: TEM energy-dispersive x-ray spectrum for a Test #5 Day-4 unfiltered sample. The copper peak is likely from the copper sample holder for TEM analysis. (T5D4_UF_EDS.jpg)

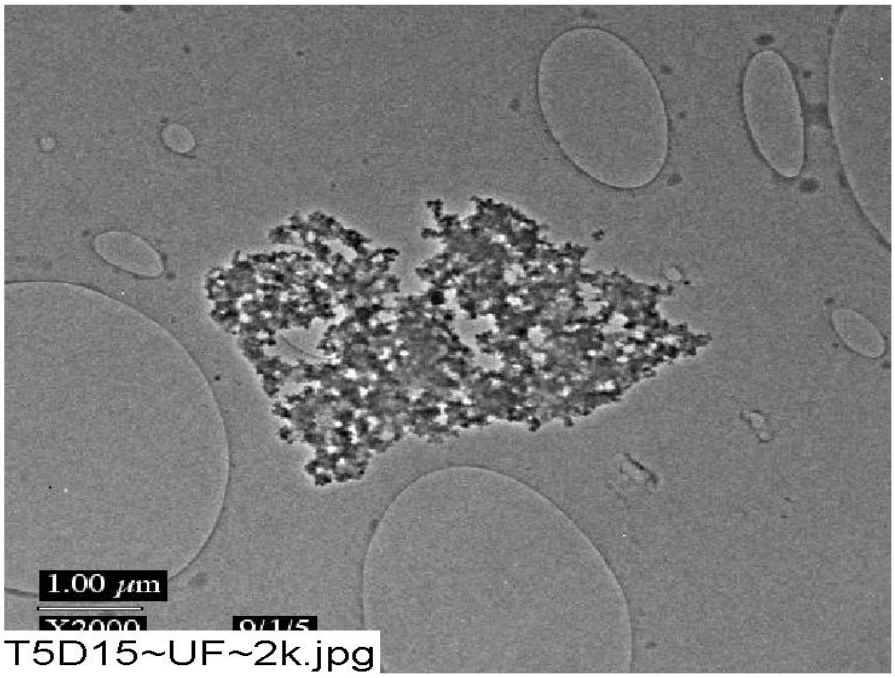

Figure G-4: TEM magnified 2,000 times for one Test #5 Day-15 unfiltered sample location. (T5D15~UF~2k.jpg)

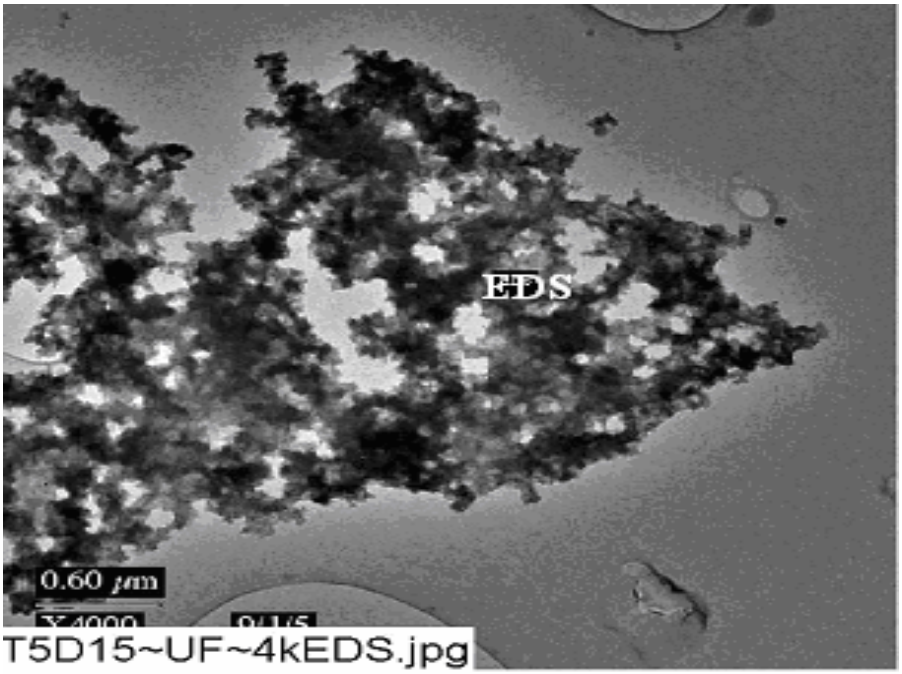

Figure G-5: Annotated TEM magnified 4,000 times for one Test #5 Day-15 unfiltered sample location. (T5D15~UF~4kEDS.jpg)

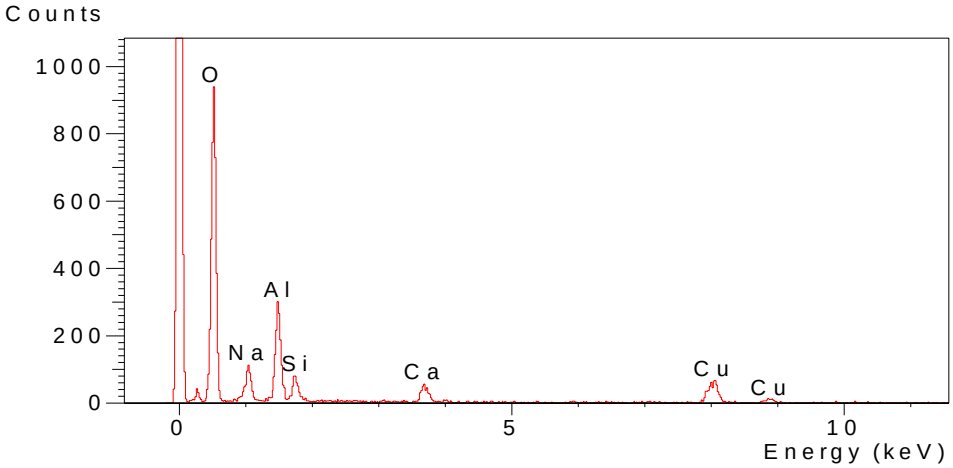

Figure G-6: TEM energy-dispersive x-ray spectrum for a Test #5 Day-15 unfiltered sample. The copper peak is likely from the copper sample holder for TEM analysis. (T5D15_UF_EDS.jpg)

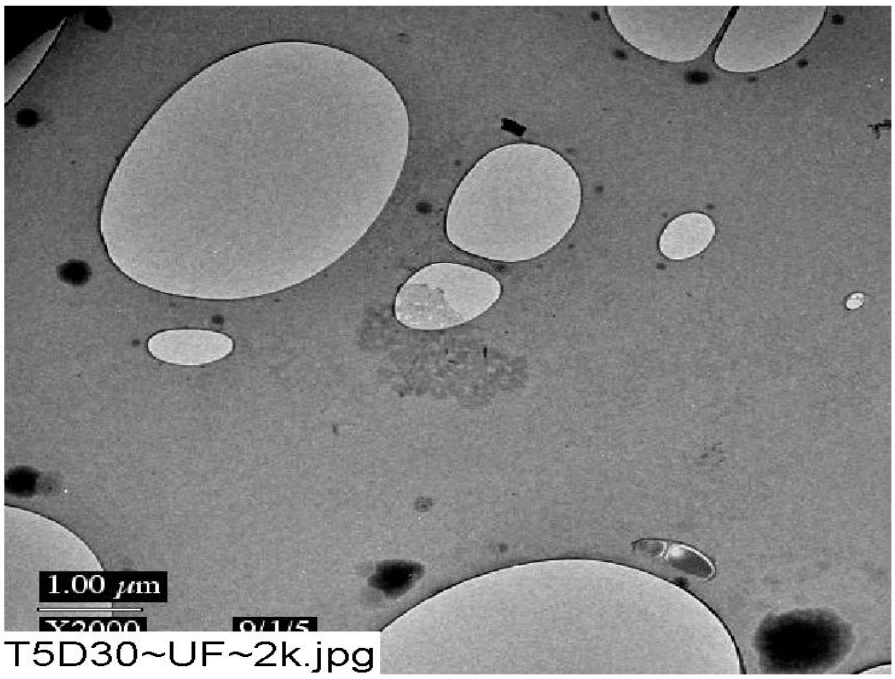

Figure G-7: TEM magnified 2,000 times for one Test #5 Day-30 unfiltered sample location. (T5D30~UF~2k.jpg)

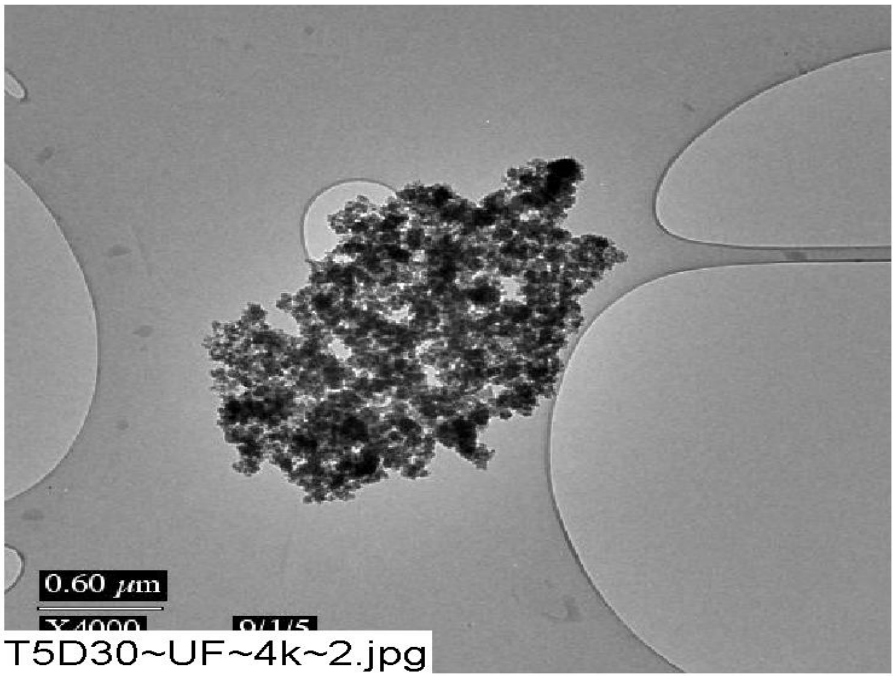

Figure G-8: TEM magnified 4,000 times for one Test #5 Day-30 unfiltered sample location. (T5D30~UF~4k~2.jpg)

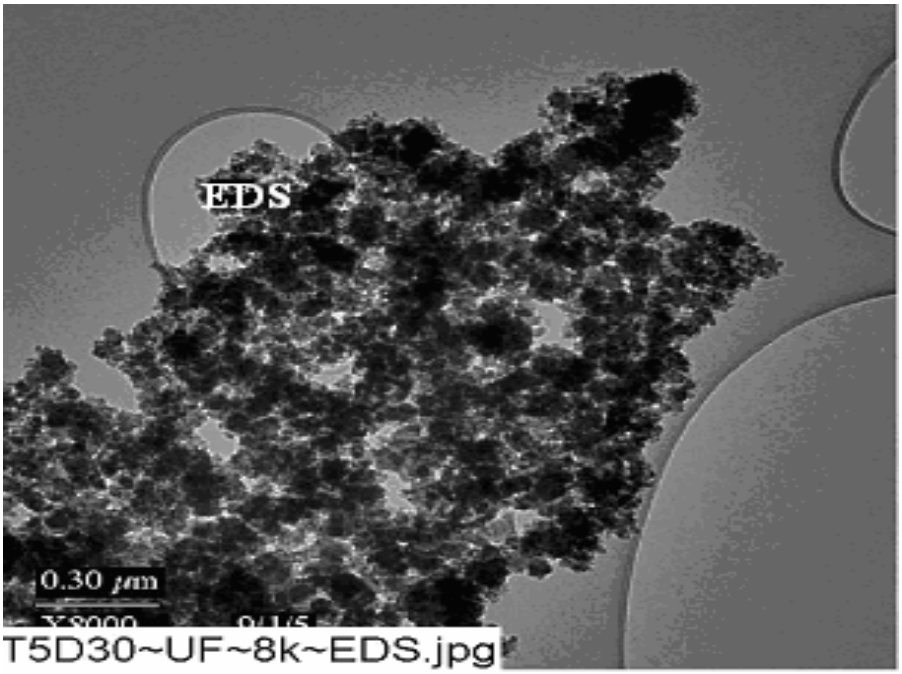

Figure G-9: Annotated TEM magnified 8,000 times for one Test #5 Day-30 unfiltered sample location. (T5D30~UF~8k~EDS.jpg)

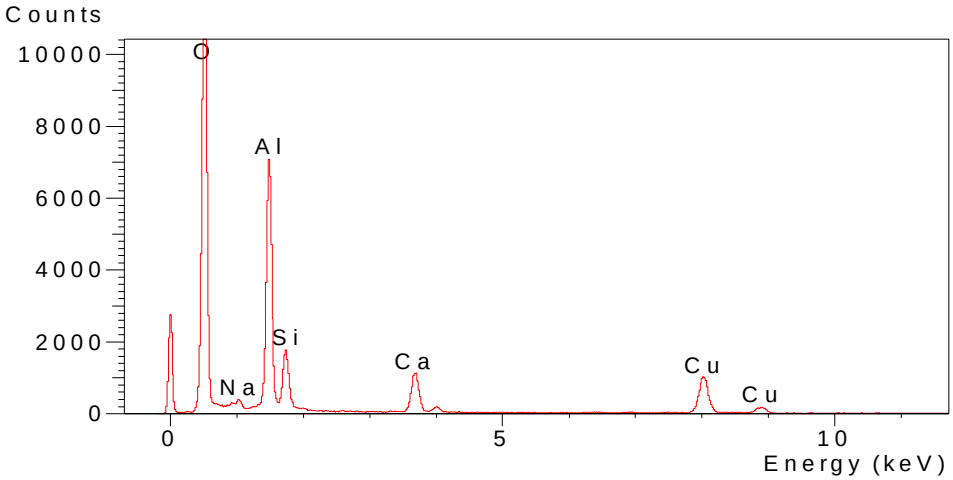

Figure G-10: TEM energy-dispersive x-ray spectrum for a Test #5 Day-30 unfiltered sample. The copper peak is likely from the copper sample holder for TEM analysis. (T5D30_UF_EDS.jpg)

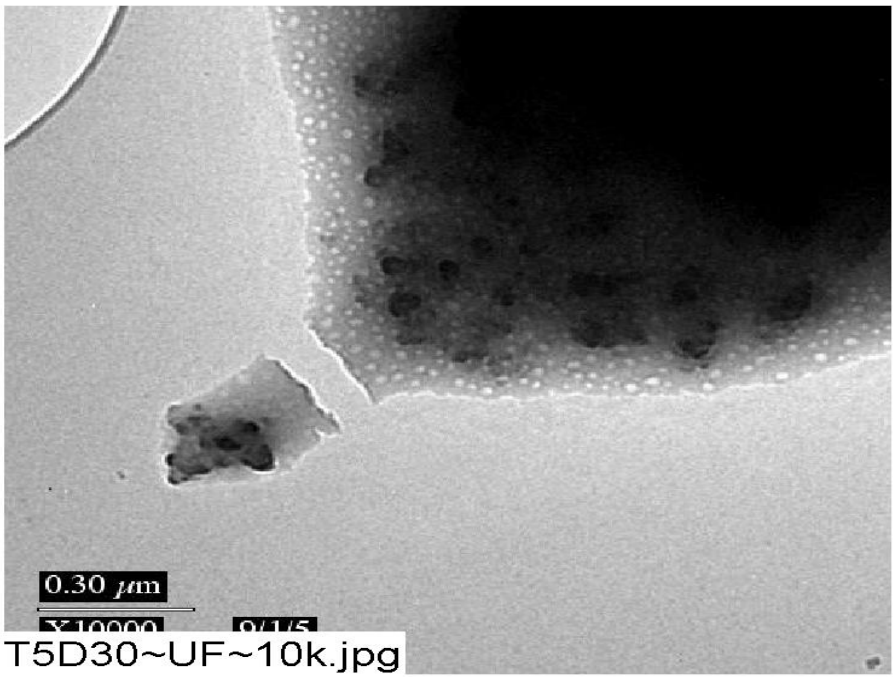

Figure G-11: TEM magnified 10,000 times for one Test #5 Day-30 unfiltered sample location. (T5D30~UF~10k.jpg)

Appendix H

UV Absorbance Spectrum – Day-30 Solution Samples

List of Figures

Figure H-1 UV Absorbance Spectrum for Test #5 Day-30 Solution Samples..... H-4

List of Tables

Table H-1: Test #5 Day-30 Solution Sample Laboratory Settings..... H-5

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This appendix presents the UV absorbance results of the Test #5 Day-30 solution sample. The purpose of this analysis was to find any distinguished absorbance peaks to identify the organics present in the solution. The solution sample was collected through a 0.7 μm fiberglass filter at $60^{o}C$ to remove particulate impurities, followed by being scanned over the wavelength ranging from 200 to 800 nm by a UV-visible spectrophotometer at room temperature. The spectrum of RO water was used as background subtraction. From the result, no distinguished absorbance peaks were found. The results were obtained on August 28, 2005.

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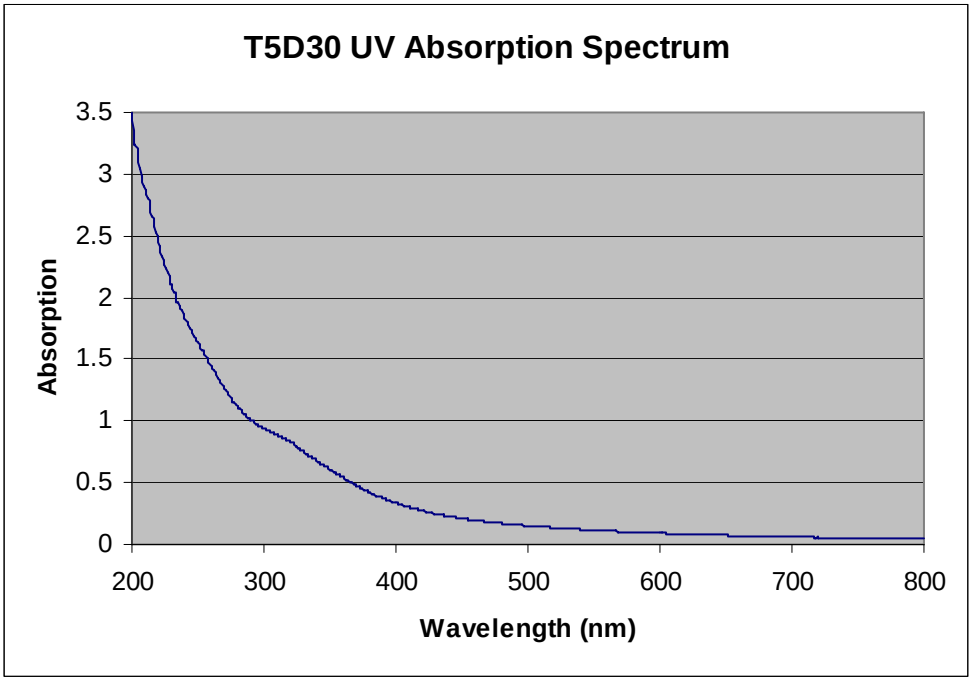

Figure H-1 UV Absorbance Spectrum for Test #5 Day-30 Solution Samples.

Table H-1: Test #5 Day-30 Solution Sample Laboratory Settings.

Test #5 Day-30	
Collection Time:	8/25/2005 5:26:10 PM
Operator Name:	
Scan Software Version:	3.00(182)
Parameter List :	
Instrument:	Cary 50
Instrument Version:	3.00
Start (nm):	800.0
Stop (nm):	200.0
X Mode:	Nanometers
Y Mode:	Abs
UV-Vis Scan Rate (nm/min):	600.00
UV-Vis Data Interval (nm):	1.00
UV-Vis Ave. Time (sec):	0.1000
Beam Mode:	Dual Beam
Baseline Correction:	On
Baseline Type:	Baseline correction
Baseline File Name:	
Baseline Std Ref File Name:	
Cycle Mode:	Off
Comments:	
Method Log:	
Method Name:	Default
Date/Time stamp:	8/25/2005 5:13:49 PM
Method Modifications:	
Cell Changer 6x6 Changed:	8/25/2005 5:13:53 PM / Old:1 / New:0
UVVIS SAT Changed:	8/25/2005 5:14:14 PM / Old:0.0125 / New:0.1000
NIR SAT Changed:	8/25/2005 5:14:14 PM / Old:0.0125 / New:0.1000
Common SAT Changed:	8/25/2005 5:14:14 PM / Old:0.0125 / New:0.1000
Baseline Correction Changed:	8/25/2005 5:14:14 PM / Old:0 / New:1
Temp Controller Changed:	8/25/2005 5:14:14 PM / Old:0 / New:2
Sipper Type Changed:	8/25/2005 5:14:14 PM / Old:Internal RSA / New:External sipper
End Method Modifications	
<Current Wavelength>	200

Appendix I

ICET Test #5: Pre-Test, Test, and Post-Test Project Instructions

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The ICET test series is conducted under the guidance of project instructions (PIs), which identify the steps to follow for certain activities. These PIs are revised or re-written as needed for each test. For Test #5, new PIs were written to address pre-test and test operations. The PI that addresses post-test operations was unchanged from Tests #3 and #4. These three PIs are included in this appendix to more completely describe the test apparatus and chemical solution preparations, the test startup and daily sampling, and the steps followed after test shutdown.

.

1.0 INTRODUCTION

1.1 PURPOSE

The purpose of this instruction is to ensure that all data acquisition, test samples, testing supplies, chemicals, and related materials are ready and accounted for prior to testing. In addition, this instruction provides instructions on preparing the chemical test apparatus for testing.

1.2 SCOPE

The pre-test operations preparation will ensure that successful initiation of the testing activity is achieved.

1.3 REFERENCES

- Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 13, July 20, 2005
- Chemical Additive Analysis Revisions – ICET-CALC-007, November 11, 2004; Test #5 Addendum, July 12, 2005
- Laboratory Safety Guidelines
- ASTM A 380 – 99, Standard Practice for Cleaning, Descaling, and Passivation of Stainless Steel Parts, Equipment, and Systems
- Material Safety Data Sheets (MSDS) for all chemicals involved

2.0 PREREQUISITES

The data acquisition setup and inspection; instrument calibration; and the coupon receipt, preparation, inspection, and storage tasks must be completed in full prior to the completion of this activity. Fiberglass and calcium silicate (cal-sil) samples must be weighed and their planned locations in the tank identified. That data must be recorded.

2.1 Training Requirements

The following personnel training is required for this task:

1) LabVIEW and computer data acquisition training
2) Chemical handling training, specifically for ethyl alcohol, ammonium hydroxide, and lithium hydroxide.
3) Safe lift execution training

2.2 Equipment Requirements

The following equipment is required to perform this activity: computer with installed LabVIEW software, data acquisition system, and fully assembled and calibrated ICET test apparatus.

Safety equipment must be available: goggles, gloves, lab coats, eye wash station.

3.0 DOCUMENTATION REQUIRED

MSDSs must be available for all chemicals used.

A lab notebook must be maintained throughout the pre-test operations instruction. Contained within the lab notebook will be the date, times, description of activities, and quantities of chemicals added, number of cleanings, and physical observations of the tank cleaning and preparation procedures.

4.0 HAZARDS

The hazards associated with this activity include potential injuries associated with chemical handling.

5.0 INSTRUCTIONS

1. Ensure that all testing materials and supplies are ready and on-site. See checklist at the end of this document. Verify that eye wash station is operational. Note: The following solutions are not used in this instruction, but are to be prepared in advance of entering ICET-PI-018, "Test Operations, Test #5 (fiberglass and sodium tetraborate at pH 8)." After preparation, clearly label the containers with the solutions and place in an area restricted for ICET Project test use.
2. Prepare 21. 2 g of concrete dust and 63.7 g of latent debris.
3. Prepare LiOH solution: dissolve 0.284 g of lithium hydroxide (LiOH) in about 100 mL water in a 250-mL sample container.
4. Prepare HCl solution for spray nozzle feed.
   a. Add 90.8 mL of 12.24 N HCl to 2 gallons of RO water in a 10-L container.
   b. Properly label and store the container.
5. Prepare laboratory control sample (LCS). See ICET-PI-005, "Chemical Sampling and Analysis," for details on the laboratory control sample.
6. Start the data acquisition system. Verify that the data acquisition system is monitoring flow rate, pump speed, temperature, and pH.
7. Clean the tank and piping.

a. Cleaning should commence as soon after a test is completed as possible, to prevent material from hardening in the tank or piping and to maximize the time available for cleaning.
b. Cleaning chemicals may consist of weak acids (e.g., acetic acid, citric acid, or dilute mineral acids), weak bases (e.g., ammonium hydroxide), week organic solvents (e.g., ethanol), or detergents/surfactants (e.g., trisodium phosphate, sodium dodecyl sulfate), as necessary. Cleaning solutions can be heated if necessary. Note that the discharge limit to the sanitary sewer is a maximum temperature of 140 °F and pH between 5.0 and 11.5. Cleaning solutions that are not within this range should be neutralized before discharge.
c. During cleaning, the pump should be run and water directed through both recirculation lines (through the spray nozzles and lower headers)
d. The sample line should be removed from the piping, physically cleaned, and carefully inspected. If the sample line cannot be adequately cleaned, it should be replaced.
e. After each cleaning step, the tank and piping should be thoroughly rinsed with tap water or demineralized water.
f. After each cleaning step, a segment of pipe should be removed, and the interior of the pipe visually inspected.
g. Cleanliness criteria: When the tank visually appears to be satisfactorily cleaned, the tank and piping should be thoroughly rinsed with demineralized water. The interior surfaces of the tank and piping shall be free of any deposits that can be removed by vigorous scrubbing. Demineralized water drained from the tank should have turbidity less than 0.3 NTU and conductivity less than 50 uS/cm.

8. Tank is now ready for testing. Proceed immediately to Instruction No. ICET-PI-018, "Test Operations, Test #5 (fiberglass and sodium tetraborate at pH 8).

6.0 ATTACHMENTS

No forms are attached to this document.

7.0 Materials Checklist

_____ lithium hydroxide, 0.284 g

_____ 90.8 mL of 12.24 N HCl

_____ tap water supply

_____ demineralized water production system

_____ chemical handling safety equipment (lab coat, goggles, rubber gloves)

_____ analytical balance

_____ top loading balance

_____ chemical spatula

_____ chemical scoop

_____ weigh boats

_____ 10-L plastic container

_____ 250 mL graduated cylinder

_____ 250-mL HDPE or PP bottle

_____ 2.5 gallons ethanol

_____ 2.5 gallons ammonium hydroxide

_____ turbidimeter and associated equipment

_____ conductivity meter and associated equipment

1.0 INTRODUCTION

1.1 PURPOSE

The intent of the instruction is to outline the steps that are to be followed during testing.

1.2 SCOPE

This activity forms the core of the entire Chemical Effects Testing project. All activities involved in this project affect and are affected by this activity.

1.3 REFERENCES

- Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 13, July 20, 2005
- ASTM Standard G 4-01
- ASTM Standard D 3370-95a
- ASTM Standard G 31-72
- Material Safety Data Sheets (MSDS) for all chemicals involved
- LabVIEW operation manual
- Laboratory Safety Guidelines
- Chemical Additive Analysis Revisions – ICET-CALC-007, November 11, 2004; Test #5 Addendum, July 12, 2005

2.0 PREREQUISITES

All sample coupons must be placed in their corresponding racks. Also, the pre-operation test preparation activity must be completed in full.

2.1 Training Requirements

The following personnel training is required for this task:
1) LabVIEW and computer data acquisition training.
2) Chemical handling training for all chemicals involved.

2.2 Equipment Requirements

The following equipment is required to perform this activity: computer with installed LabVIEW software, data acquisition system, and fully assembled and calibrated ICET test apparatus.

Safety equipment must be available: goggles, gloves, lab coats, hard hats, steel-toed shoes, eye wash station, hydrogen detector and hydrogen removal system.

3.0 DOCUMENTATION REQUIRED

A lab notebook must be maintained throughout the testing procedure. In addition, a binder will be maintained that includes pertinent test instructions and the completed daily log sheets (see Attachment A). The daily log sheet contains the date, times, physical description, and quantity of fiberglass and water samples obtained each day. In addition, the daily log sheet contains information from the data acquisition system (DAS), the water samples taken, and other test information.

The electronic data that are acquired are backed up daily and stored in a separate location each testing day. Refer to ICET-PI-001, Data Acquisition Setup and Inspection.

4.0 HAZARDS

The hazards associated with this activity include tipping of the chemical tank assembly, ingestion and/or respiration of any chemicals involved, and scalding and/or burning hazards involved in daily tank venting, and possible hydrogen gas generation from corrosion reactions. Appropriate measures to control hydrogen gas must be in place before operations commence.

Lifting hazards associated with the tank lid and coupon racks are also associated with this activity.

5.0 INSTRUCTIONS

1. Because of the time required for heating the tank contents and dissolving chemicals, this sequence should be started at least 48 hours before the scheduled time t = 0. Pre-test operations preparation should be complete before proceeding with this sequence.
2. Ensure that all testing materials and supplies are ready and on-site (see checklist at end of this instruction).
3. Add 248 gallons of RO water to the tank by pumping water from the RO skid through the totalizing flow meter. Record flow to the nearest 0.5 gallon.
4. Verify valves are positioned as follows:

Valve	Description	Position
V-1	tank drain	closed
V-2	pump isolation	open
V-3	instrument loop supply	open
V-4	instrument loop discharge	open
V-5	instrument loop bypass	closed

V-6	in-line filter isolation	open
V-7	recirculation line supply	open
V-8	tank spray nozzle supply	closed
V-9	sample line	closed
V-10	recirculation line injection	closed

5. Start pump and adjust to flow rate of approximately 25 gpm.
6. Start computer, start LabVIEW, verify that flow rate, pump speed, temperature, and pH are being recorded properly.
7. Turn on heater and allow water in tank to heat to 60 °C ± 2 °C. (This may take up to 20 hours.)
8. Add the pre-mixed LiOH solution.
9. Add 6.48 kg of boric acid (H_3BO_3), weighing in approximately 2 kg increments, recording the weight of each increment to the nearest 10 g.
10. Add 10.0 kg of Borax (sodium tetraborate) weighing in approximately 2 kg increments, recording the weight of each increment to the nearest 10 g.
11. Allow the water to circulate until the solution is visibly clear, indicating that the boric acid and Borax is completely dissolved. If it is observed that the chemicals are not remaining in solution, consider running the pump at a higher speed during pre-test operations.
12. Allow water in tank to heat to 65 °C ± 2 °C.
13. Take grab water sample for analysis for the parameters identified in steps a – h below. Also record physical appearance of the sample (clarity, presence of gelatinous material, etc). All Day 1 and subsequent samples will be analyzed by Assaigai Analytical Laboratory. In addition, periodic test samples and laboratory control samples (LCSs) will also be analyzed by the UNM laboratory.
    a. pH
    b. temperature
    c. turbidity
    d. viscosity
    e. total suspended solids (TSS)
    f. dissolved oxygen (DO)
    g. chloride
    h. metals (Al, B, Ca, Cu, Fe, Pb, Li, Mg, Ni, K, Si, Na, and Zn), total and dissolved
14. Add 21.2 g of concrete dust and 63.7 g of latent debris samples (prepared earlier), wait 10 minutes, take 100 mL water sample for particulate size distribution, density, and TSS.
15. Stop pump.
16. Place coupon racks and fiberglass holders into tank.
17. Verify locations of coupon racks and fiberglass holders.
18. Verify the tank temperature is 60-62 °C.
19. Start pump and adjust pump speed to 25 gpm.
20. Open valve V-8 (tank spray nozzle supply) to direct water to nozzles and adjust valves V-7 (recirculation line supply) and V-8 (tank spray nozzle supply) until nozzle

flowmeter is reading 3.5 gpm. Verify total flow is still 25 gpm and adjust variable frequency drive (VFD) if necessary.

21. Record date and time at which nozzle flow started. This is time t = 0 for the test.
22. During the first few hours of the test, if the test solution is cloudy, consider frequent measurements of turbidity, pH, and TSS.
23. At t = 2 hours, start chemical metering pump and inject pre-measured HCl solution into the tank. The objective here is to add the 2-gallon HCL solution in 2 hours.
24. At t = 4 hours, shut off chemical metering pump and isolate this line.
25. Take a measurement of hydrogen concentration. At 2-hour increments, repeat the hydrogen concentration measurement. If the concentration reaches 10% of the flammability limit, purge the tank atmosphere. This needs to be repeated until the hydrogen concentration has been determined to be below 10% of the flammability limit, and then the frequency of hydrogen concentration measurements is to be re-evaluated.
26. At t = 4 hours, stop the spray flow by closing valve V-8.
27. After closing valve V-8 (at t = 4 hours), take water grab sample for analysis for the parameters listed below. Record the time of sample collection.
    a. pH
    b. temperature
    c. turbidity
    d. viscosity
    e. chloride
    f. total suspended solids (TSS)
    g. dissolved oxygen (DO)
    h. metals (Al, B, Ca, Cu, Fe, Pb, Li, Mg, Ni, K, Si, Na, and Zn), total and dissolved.
28. At t = 24 hours, and daily thereafter, take water grab sample for analysis for the parameters listed below. (The LANL PI will propose a different sampling frequency to the project sponsors if test data support it.) Record the time of sample collection.
    a. pH
    b. turbidity
    c. viscosity
    d. temperature
    e. total suspended solids (TSS)
    f. metals (Al, B, Ca, Cu, Fe, Pb, Li, Mg, Ni, K, Si, Na, and Zn), total and dissolved. An exception is that B, Li, K, Pb, and chloride analyses will be performed only at t = days 15 and 30. Also, dissolved oxygen will be measured at day 30.
29. During each daily water sample collection, look inside tank (through windows) and record observations. If the tank water level indicates that the water volume is 245 gallons or less, add RO water to bring the volume up to 250 gallons and record the amount added.
30. At t = 24 hours, weekly thereafter, and at the end of the test, collect 100 mL water sample for particulate size distribution and density analysis, to be performed at AALI. The particulate size ranges to be used will be as close as possible to those called out in the test plan: (in microns), 1-10, 11-25, 26-50, 51-75, 76-100, and > 100 microns.

31. At t = 24 hours, weekly thereafter, and at the end of the test, collect water samples for strain rate viscosity measurements (see PI-010 for sample details.)
32. After 2 to 5 days of testing, it is anticipated that the solution will be stable and no suspended particles will be visible. If that is the case, insert the three types of fiberglass samples described in Attachment B. Note that one of the samples (long, narrow stainless steel holder) is to be placed in front of the water distribution headers, and the others are to be placed behind the headers. (The date and time of the addition of these samples will be recorded in the lab notebook.)
33. At 3 days $\leq t \leq$ 5 days, 14 days $\leq t \leq$ 16 days and at the end of the test, collect a sacrificial fiberglass sample to be inspected and examined with SEM.
34. At 24 hours, at 14 days $\leq t \leq$ 16 days and at the end of the test, run 1L of water through a nucleopore filter. The filter will be taken for SEM analysis as specified in ICET-PI-007. (Note that depending on the solution, some filter material will not work well for this operation. If possible, use a nucleopore filter for SEM analysis, and then collect a second sample on nitrocellulose filter for later digestion and ICP analysis.)
35. Shut down pump
36. Indicate end of test on the data acquisition system and shut down the data acquisition software.
37. Proceed directly to PI-008 Post-Test Operations.

6.0 ATTACHMENTS

Attachment A. Daily Log Sheet

Attachment B. Test #5 Fiberglass Sample Addition after Test Start

7.0 MATERIAL CHECKLIST

_____ boric acid, 6.48 kg

_____ Borax, 10.0 kg

_____ pre-mixed LiOH solution

_____ concrete dust, 21.2 g

_____ latent debris, 63.7 g

_____ Nucleopore filter

_____ chemical handling safety equipment (lab coat, goggles, rubber gloves)

_____ top-loading balance

_____ weigh pan for 2 kg aliquots of boric acid

_____ stainless steel filter paper holder

_____ 500 mL graduated cylinder (for TSS)

_____ totalizing flow meter

_____ sample containers (see Chemical Sampling Instruction)

_____ analytical equipment (see Chemical Sampling Instruction)

_____ pre-assembled coupon racks

_____ pre-assembled fiberglass baskets, total of 11 lb of fiberglass

_____ coupon handling safety equipment (hard hat, leather gloves, boots)

_____ computer disks for backup of Labview data

_____ Masterflex peristaltic pump and tubing

_____ demineralized water production system

Attachment A. Daily Log Sheet

Daily Log Sheet
Integrated Chemical Effects Test (Test # 5)

Date: _____ Time of sample collection: _____
Sample taking and data reduction by _____ and_____

Sample bottle identification:
Assaigai (total): _____
Assaigai (filtered): _____
UNM (total): _____
UNM (filtered): _____

Control system readings:
Temperature: _____ Flow: _____ pH: _____

Analyses:
Volume filtered for TSS: _____ pH: _____
Temperature: _____ Dissolved oxygen: _____
Turbidity (at 60 °C): _____ (at 23 °C; and 10 min.)_____
Viscosity, unfiltered (60 °C): _____ (at 23 °C) _____
Viscosity, filtered (60 °C): _____ (at 23 °C) _____
Water Level: _____ Water Added: _____
Hydrogen: _____ Other: _____
Fiberglass or other samples taken: _____
TSS filter #: _____ TSS (mg/L): _____

Comments:

Observations written in lab notebook by _____
☐ Continued on back

Attachment B. Test #5 Fiberglass Sample Addition after Test Start

Recent experience gained in ICET Test #3 with adding large quantities of Calcium Silicate (Cal-Sil) debris to the tank suggests that contamination of fiber samples with suspended particulate may complicate the post-test identification and analysis of chemical products that may be contained within the samples. Similar behavior without Cal-Sil in Tests #1 and #2 was also observed. Past experience also suggests that the circulating tank solution will clarify after 1 to 2 days, providing an opportunity to introduce fiber samples for immersion in the chemical environment without substantially shortening the exposure time and while avoiding the complications of particulate contamination. (Several other fiber samples are immersed at the initiation of the test so that they are directly exposed to large quantities of the particulates).

This attachment to ICET-PI-018 Rev 0 addresses the addition of three types of containers for fiberglass samples that are to be inserted in the test solution between Days 2 and 5 after substantial water clarity has been achieved and as needed to match operations schedules. These containers include: (A) a long (5-6 in.), thin (1/4 to ½ in.), narrow (1-2 in.) stainless steel mesh envelope that will be placed in front of a discharge hole on one of the water distribution headers (a "high-flow" area of the tank); (B) a nylon mesh envelope of approximately 4 to 6 inches square containing 5 to 10 g of fiberglass, and (C) 3 to 5, two-inch diameter pucks of fiberglass that are prepared in rings of CPVC and encased in typical envelopes of stainless steel mesh. Each container will hold less than 10 g of fiberglass and their fiberglass contents are considered negligible with respect to the total test amount of fiberglass. The nylon mesh envelope and pucks will be placed in "low-flow" areas of the tank behind the water distribution headers. Technical descriptions and justifications for each item follow.

Test Item (A): thin sample in high flow

This sample may provide evidence of whether chemical deposits are enhanced or inhibited by the direct impingement of water flow. The large aspect ratio of this envelope (length/width) is designed to avoid large perturbations in the inlet flow patterns that would occur by placing a large flat object near the distribution headers. Post-test examination of this sample will be made using typical ESEM and SEM/EDS survey techniques.

Test Item (B): nylon mesh envelope

ICET Test #1 results indicated a possible preference for chemical deposits to form near the interface between fiberglass and the stainless steel mesh that was used to form the sample envelopes. The introduction of a nonmetallic casing material may permit a comparison of this effect under exposure to a similar chemical environment. Nylon mesh was selected as a suitably inert material for constructing an envelope for this fiber. Based on qualitative assessments and recommendations of material performance made by

chemical supply vendors (see for instance, chemical resistance information given at www.eldonjames,com/html), nylon is expected to exhibit "Excellent" resistance to NaOH solutions of up to 50% concentration and "Good" resistance to industrial concentrations of boric acid. This container material has not been submitted for independent bench-scale leaching tests in the ICET solution. The small quantity of foreign material is not expected to perturb interpretation of results from ICET Test #5 regardless of its performance characteristics. Post test examination of this sample will be made using typical ESEM and SEM/EDS survey techniques.

Test Item (C): Fiber pucks

The MOU established between NRC and EPRI for conduct of the ICET series specifically excludes modification of the apparatus for the purpose of obtaining in-line flow head loss data. However, the presence of chemical deposits observed on and within fiberglass samples obtained from ICET Test #1 to Test #2 raises questions regarding the potential of these products to impede water flow and about their behavior under flowing conditions, for example, whether they will be adherent or wash out of the fiber matrix. Unanswered questions also remain regarding the possible formation of deposits in the presence of flow, but without a direct mechanism of studying formation under flow, it may be useful to examine whether the deposits can form in fiberglass that represents a prototypical debris bed. Two of the attributes that may distinguish fiberglass on a debris bed from fiberglass in a debris flock are (a) degree of mechanical separation between fibers, and (b) degree of compaction in the bed.

12 fiberglass pucks will be prepared as shown in Figure 1 for jacketing in a stainless steel envelope. Four of the pucks will be immersed in the Test #5 test solution at the start of the test. The remaining eight pucks will be immersed in the test solution after the water clarity has improved. Approximately 5-10 grams of dry fiberglass are required to fill the ½-inch thick, 2-in. diameter sample ring. The sample ring is cut from a 2-inch diameter CPVC pipe to provide a standard dimension for any flow testing that may be desired after the conclusion of Test #5 and to avoid the introduction of unapproved foreign materials in the test tank. Before introducing the fiberglass to the mold, it will first be agitated in a kitchen blender for at least 2 minutes in two batches, each batch containing approximately half the debris and approximately 2 quarts of water. The purpose of agitation is to separate fibers from the raw flocks of manufactured insulation. The batches will be sequential poured into the mold placed across a mesh screen. Gentle manual tamping may be required to ensure uniformity of the bed.

Figure 1. Example fiber pucks prepared for immersion in ICET test solution.

The introduction of fiberglass pucks will provide the following technical opportunities for post-test examination:

1) Any observed chemical deposits will be relatively free from contamination of suspended particulates.
2) ESEM and SEM/EDS examines can be made for the presence of deposits inside of a relatively compact debris bed.
3) If exams 1 and 2 are positive, the pucks will provide a concentrated quantity of the deposit for possible extraction, isolation and identification.
4) The pre-measured dry mass of the fiber may permit a determination of dry mass for any deposited chemical products. This may provide a first step towards quantifying rates and quantities of formation.
5) The convenient form of the debris in the mold will facilitate any head loss testing that is deemed interesting or necessary as a post-test analysis activity. Samples of this type could be placed either within a continuously circulating closed loop or within a static head drain column for measurement of flow loss. The primary objective of any such examinations would be the direct comparison of fresh, identically prepared samples with cultured samples that have been exposed to the test environment. Expected variability between the samples suggests that several replicates should be prepared for comparison. Any work of this type will be conducted under a separate approved procedure.

<u>Note:</u> The purpose of this attachment is to describe the samples to be added to Test #5 after test initiation. Any post-test evaluations of these samples other than ESEM and SEM/EDS will be done under separate procedure/project instructions. In

addition, any post-test head loss testing will require appropriate documentation and sponsor approvals.

1.0 INTRODUCTION

1.1 PURPOSE

The intent of this instruction is to ensure that the experimental samples are removed from the test apparatus, the test apparatus is rinsed and inspected, and the test apparatus is made ready for subsequent pre-test operations.

1.2 SCOPE

This activity marks the end of one chemical effects test run. Experimental sample removals and inspections, test apparatus rinsing, and preparations for cleaning and subsequent tests are addressed here.

1.3 REFERENCES

- Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 12.c, March 30, 2005
- ASTM Standard G 4-01
- ASTM Standard G 31-72
- ICET-PI-002, Coupon Receipt, Preparation, Inspection, and Storage, November 19, 2004
- ICET-PI-014, Rev. 0, Test Operations, Test #3 (cal-sil and fiberglass, with TSP, April 5, 2005
- ICET-PI-005, Rev. 1, Chemical Sampling and Analysis, February 3, 2005
- Laboratory safety guidelines
- ICET Project Safety Plan

2.0 PREREQUISITES

All test operation PI criteria must be completed prior to conducting this task.

2.1 Training Requirements

- Laboratory Safety Guidelines
- ICET Project Safety Plan

2.2 Equipment Requirements

A city tap water supply outlet is required for this activity and chemical handling and lifting safety equipment. A reverse osmosis unit is required for the final flush.

3.0 DOCUMENTATION REQUIRED

Documentation related to test parameters, chemical water analyses, coupon and fiberglass examinations, and daily test operations are outlined elsewhere. In this instruction, the steps required to remove samples from the test apparatus and to make it ready for the next test are outlined. In addition, observations as to the test apparatus' condition are obtained and recorded here.

4.0 HAZARDS

The hazards associated with this activity include ingestion/respiration and/or dermal and eye contact with residual chemicals. Lifting hazards associated with the tank lid and coupon racks are also associated with this activity.

5.0 INSTRUCTIONS

1) On the last day of testing, collect water samples and perform analyses as outlined in ICET-PI-014 and ICET-PI-005.
2) Remove 10L of water from the test apparatus and store at test temperature, for future analyses
3) Shut off the recirculation pump.
4) Remove the small fiberglass samples for SEM examination.
5) Leave one heater on and continue to monitor tank water temperature.
6) Isolate and drain the test apparatus piping.
7) Remove the tank lid.
8) Before removing coupon racks or insulation samples, examine and take photographs and notes of the inside of the tank, the coupons and racks, and the insulation samples.
9) Remove the six non-submerged coupon racks to a staging area for drying and post-test examinations (refer to ICET-PI-002).
10) Take additional photographs of the inside of the tank.
11) Drain the tank slowly, down to the level that uncovers the submerged rack, but keeping the water level above the heater.
12) Remove the submerged coupon rack to the staging area.
13) Repeat step # 10.
14) Turn off the heater.
15) Completely drain the tank, taking precautions so that the sediment on the bottom of the tank is not disturbed any more than necessary.
16) Store water that was drained from the test apparatus until it is cleared for disposal or shipment. (This step was just moved from later in the PI – the old step #26.)
17) When the tank is drained, repeat step # 10. Note especially the locations and orientations of the remaining samples.
18) Remove the remaining insulation samples to the staging area to dry.

19) Ensure that all samples removed from the tank are clearly marked as to their location and orientation within the tank.
20) After all samples have been removed, repeat step # 10.
21) Inspect the interior of the tank, noting any observations.
22) Note the presence of any sediment. Carefully remove as much sediment as possible, noting any unique aspects of it, such as location. Place the sediment in plastic containers with lids, marking the location of the sediment in the tank.
23) Remove the tank drain screen and remove the insulation sample for future analysis.
24) Remove the flow meter from the loop and take pictures of the flow meter interior.
25) Remove any deposits within the flow meter and place the deposits in plastic containers with lids. This is to keep the samples hydrated.
26) Remove a section of pipe, take pictures of the pipe interior, and remove and store any deposits there.
27) Replace the flow meter and piping section.
28) Rinse the tank with tap water and drain the water.
29) Fill the system with 250 gallons of tap water and circulate water through the spray nozzles and recirculation headers for at least 60 minutes. Repeat with de-mineralized water.
30) If any signs of deterioration are observed on the inside of the test apparatus tank, remove selected insulation on the tank. Inspect the stainless steel tank for any abnormalities.

6.0 ATTACHMENTS

No forms are attached to this document.