

Figure 3-16. Phosphate concentration.

3.2.3. Optical/TEM Images from Filtered and Unfiltered Samples

TEM/EDS and diffraction patterns analysis were performed for Test #3, Day-4, Day-15, and Day-30 filtered and unfiltered solution samples. The filtered solution samples were passed through a 0.7- μm fiberglass filter at 60°C. The unfiltered solution samples were extracted from the tank directly. The results showed no significant diffraction pattern, due to the amorphous nature of the samples. In addition, no significant presence of colloidal particles was observed. Appendix J contains the TEM data.

3.3. Insulation

Test #3 was the first ICET test that included cal-sil insulation in addition to NUKON™ fiberglass samples. The fiberglass samples received more thorough investigations, with samples removed from the tank on Day 4, Day 15, and Day 30. The cal-sil was analyzed based on its Day-30 character. In addition, analyses were performed on the raw cal-sil, both baked and not baked.

3.3.1. Deposits in Fiberglass Samples

The fiberglass debris was 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 experience direct water flow through the fiber. All were thoroughly immersed in the test solution until they were recovered from the tank.

After the fiberglass had been exposed in the test solution for some time, deposits have formed throughout the fiber matrix and appear to be chemically originated and/or physically retained or attached. Because there was no significant water flow directly through the fiber, the migration of particles into the fiberglass interior is likely insignificant. Therefore, the deposits found in the interior of the fiberglass samples were likely chemically originated, i.e., formed through precipitation. However, particulate deposits may have been physically retained or attached on the fiberglass exterior.

There were four fiberglass locations in the tank that were examined in this test, including the low-flow area, the high-flow area, the birdcage, and the drain collar. (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 3.3.1.7 give the ESEM/SEM/EDS results according to the location of the fiberglass samples in the tank and the sampling date. The different samples include Day-4 low flow, Day-15 low flow, Day-15 high flow, Day-30 low flow, Day-30 high flow, Day-30 drain collar, and Day-30 birdcage. The corresponding figures are Figures 3-17 through 3-77. Additional micrographs of fiberglass samples are presented in Appendices A, B, and C.

In general, the deposits appear to be more prevalent and/or to develop as the test proceeds. The particulate/flocculent deposits on Day-4 and Day-15 high- and low-flow fiberglass samples were likely originated through chemical precipitation during the drying process of the samples. The figures show that the deposits are pervasive throughout the fiber. Comparing probe SEM results with ESEM results reveals that much more significant flocculence was found with probe SEM analysis, possibly because ESEM samples were much moister than were probe SEM samples during the examination process. The drying process caused the formation of the flocculence through chemical precipitation.

Far more particulate deposits were found on Day-30 exterior samples, especially on the drain collar and the birdcage fiberglass samples, which showed the development of a continuous coating on their exteriors. The deposits on these samples include particulate deposits that were likely physically captured or attached. "Physically captured" means the deposits existed/formed in bulk solution first followed by attachment on the fiberglass. "Chemically originated" means the deposits formed directly in the fiberglass. Based on EDS analysis, the particulate deposits on the fiberglass exterior may be classified into two categories according to P and Si content. Particulate deposits of lower P and higher Si content were likely cal-sil particles; particulate deposits of high P and lower Si were likely composed of calcium phosphate precipitates (although the specific compound was not determined). Both kinds of deposits may be physically transported and/or deposited onto the fiberglass sample exterior. However, different from the exterior, the interior fiberglass samples were relatively clean. This result suggests that almost all of the particulate deposits were physically retained at the fiberglass exterior. The deposits in the fiberglass interior were probably formed by chemical precipitation during the sample

drying process for ESEM/SEM analysis. EDS analysis indicates that the flocculent deposits contained insignificant amounts of P, meaning that the deposits did not have a direct relation to the white gel (cream) that was seen forming during the injection of TSP on the first day of the test.

3.3.1.1. Day-4 Low-Flow Fiberglass Samples

Since there was no significant water flow through the fiberglass samples during the test, particle migration from the solution into the fiberglass interior is insignificant. Based on the ESEM/SEM results, deposits were found on both the exterior and the interior of the low-flow fiberglass samples after test Day 4. Because these deposits formed continuously among glass fibers and even coated the fibers, it is likely that these deposits are of chemical origin instead of being physically attached/retained. Comparing ESEM and SEM results for the same fiberglass sample reveals that some dark deposits were found with ESEM results only. However, when the fiberglass samples were totally dried for SEM analysis, only white flocculence deposits were found, possibly because the fiberglass samples were semidried (partially dehydrated) with ESEM analysis. It is likely that the dark deposits began to precipitate out when the fiberglass samples were partially dehydrated during ESEM analysis. However, these dark deposits were totally precipitated out and dehydrated for SEM analysis. As a result, a significant amount of the white flocculence was found with SEM results. EDS results show that the deposits were commonly composed of O, Si (possible), Na, Ca, and small amounts of Mg, Al, B, and P, whether they were found on the exterior or interior of the fiberglass samples. The uncertainty of Si is due to the fact that x-ray may be scattered and/or penetrate the deposits. As a result, the signal may be reflected to the detector by the fiberglass in addition to the deposits. Therefore, when Si peaks show up, the existence of Si in the deposits cannot be confirmed or excluded. In addition, it should be noted that the deposits contained insignificant amounts of P, which means that the deposits did not have a direct relation to the white particles observed in the tank during TSP injection on the first day of the test.

Comparing interior and exterior fiberglass samples reveals no significant difference in the amount of deposits, again probably because of a chemical origin for the deposits. Chemical precipitation occurs to the same degree on both exterior and the interior fiberglass samples. Figures 3-17 through 3-27 show the Day-4, low-flow fiberglass results.

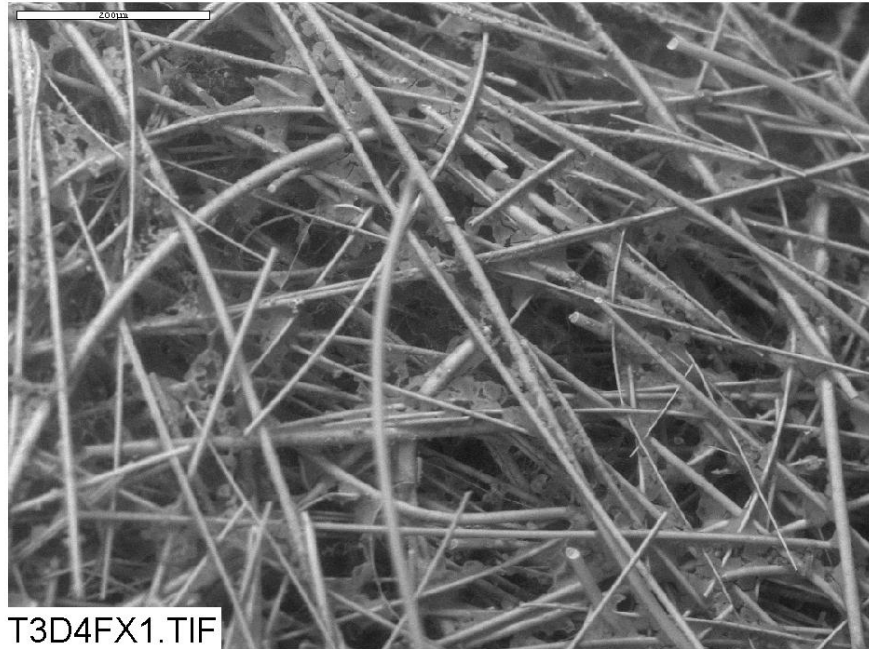


Figure 3-17. ESEM image of a Test #3, Day-4 low-flow exterior fiberglass sample, magnified 150 times. (T3D4FX1, 4/12/05)

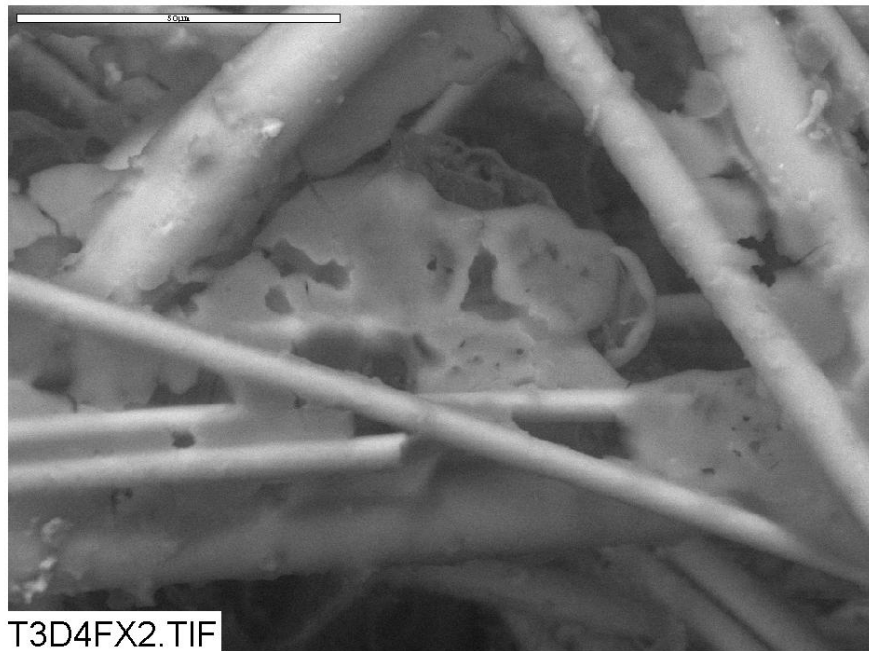


Figure 3-18. ESEM image for a Test #3, Day-4 low-flow exterior fiberglass sample, magnified 1000 times. (T3D4FX2, 4/12/05)

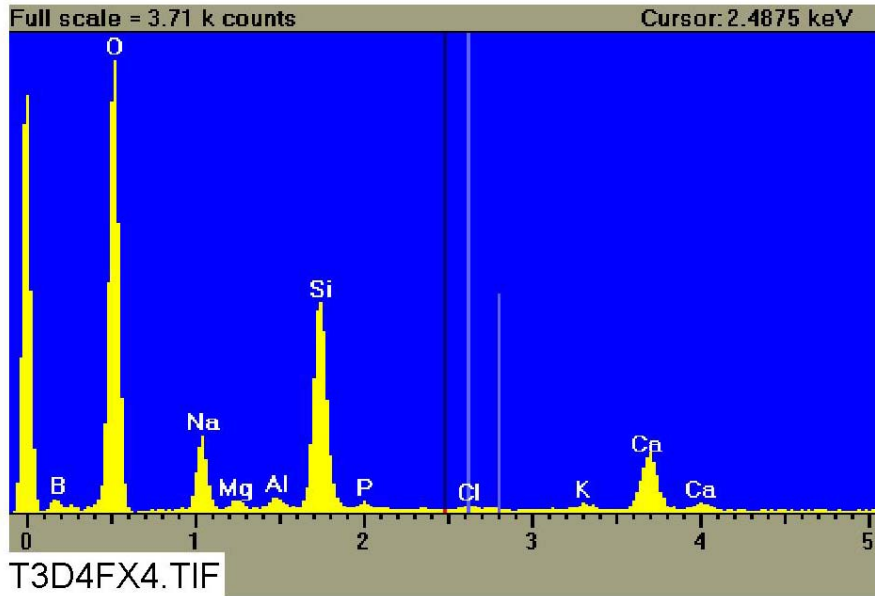


Figure 3-19. EDS counting spectrum (after calibration) for the deposits between the fibers on the ESEM image shown in Figure 3-18. (T3D4FX4, 4/12/05)

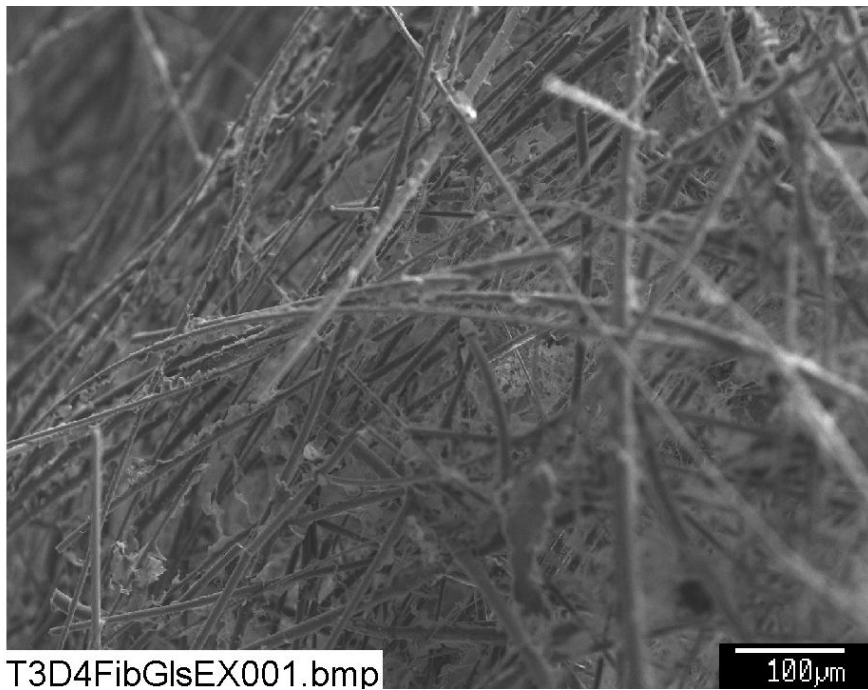


Figure 3-20. SEM image of a Test #3, Day-4 low-flow exterior fiberglass sample, magnified 150 times. (T3D4FibGlsEX001, 4/12/05)

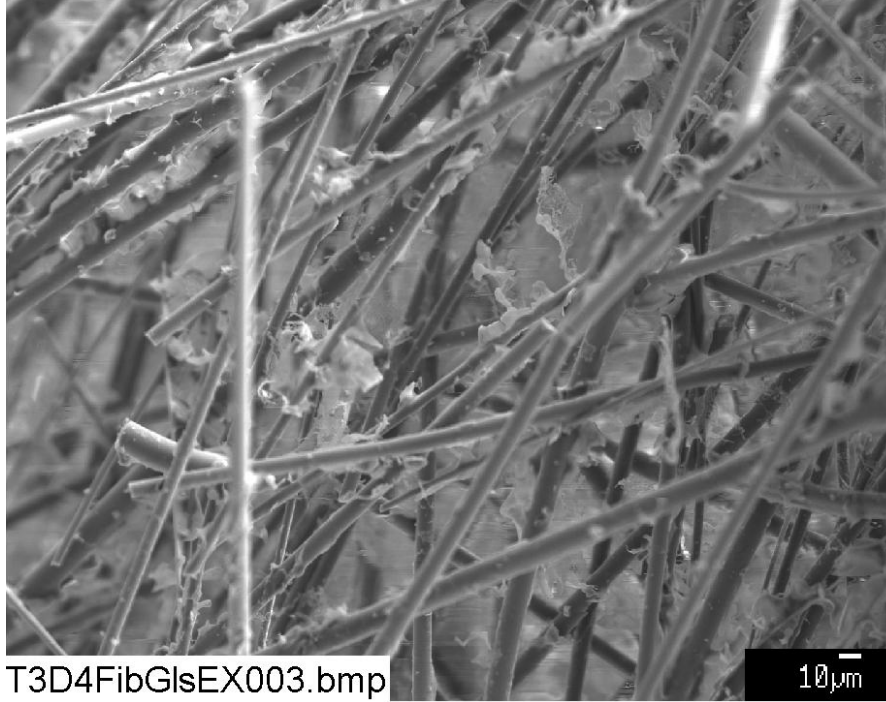


Figure 3-21. SEM image of a Test #3, Day-4 low-flow exterior fiberglass sample, magnified 300 times. (T3D4FibGlsEX003, 4/12/05)

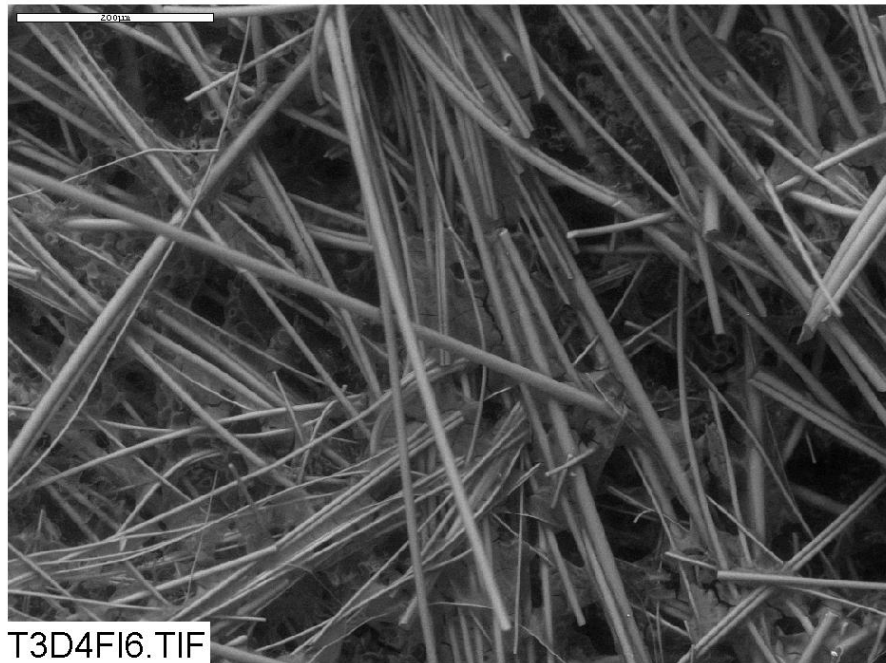


Figure 3-22. ESEM image of a Test #3, Day-4 low-flow interior fiberglass sample, magnified 150 times. (T3D4FI6, 4/12/05)

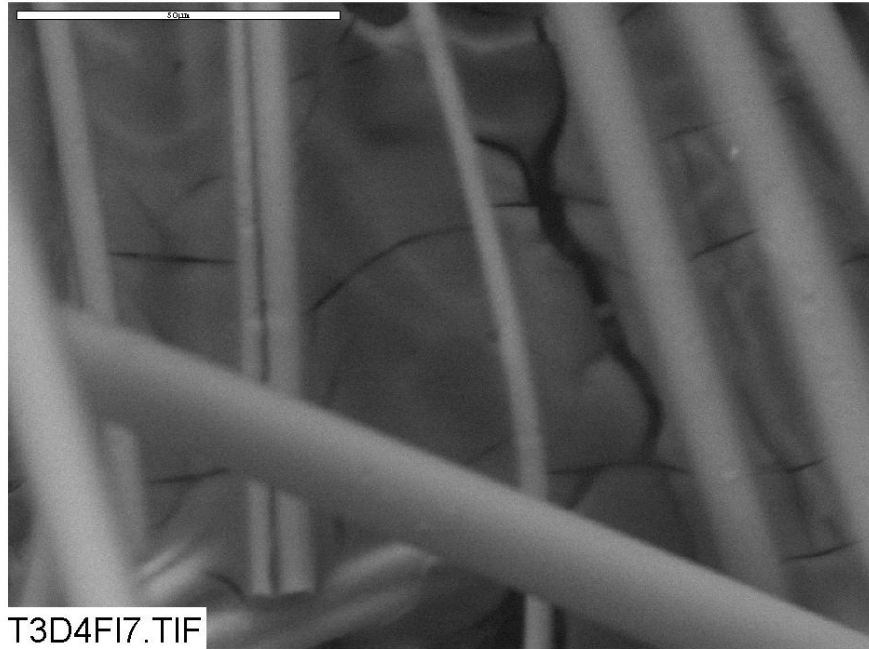


Figure 3-23. ESEM image of a Test #3, Day-4 low-flow interior fiberglass sample, magnified 1000 times. (T3D4FI7, 4/12/05)

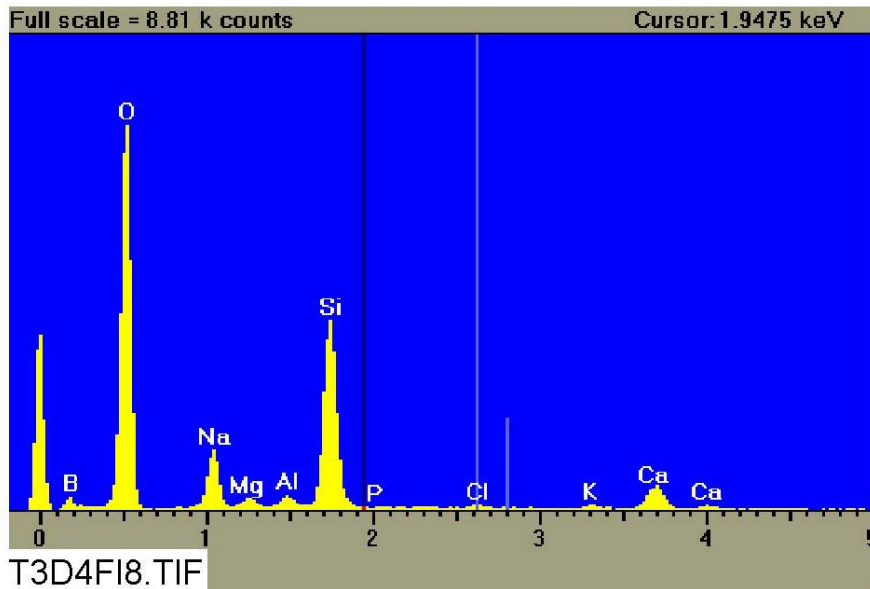


Figure 3-24. EDS counting spectrum (after calibration) for the deposits between the fibers on the ESEM image shown in Figure 3-23. (T3D4FI8, 4/12/05)

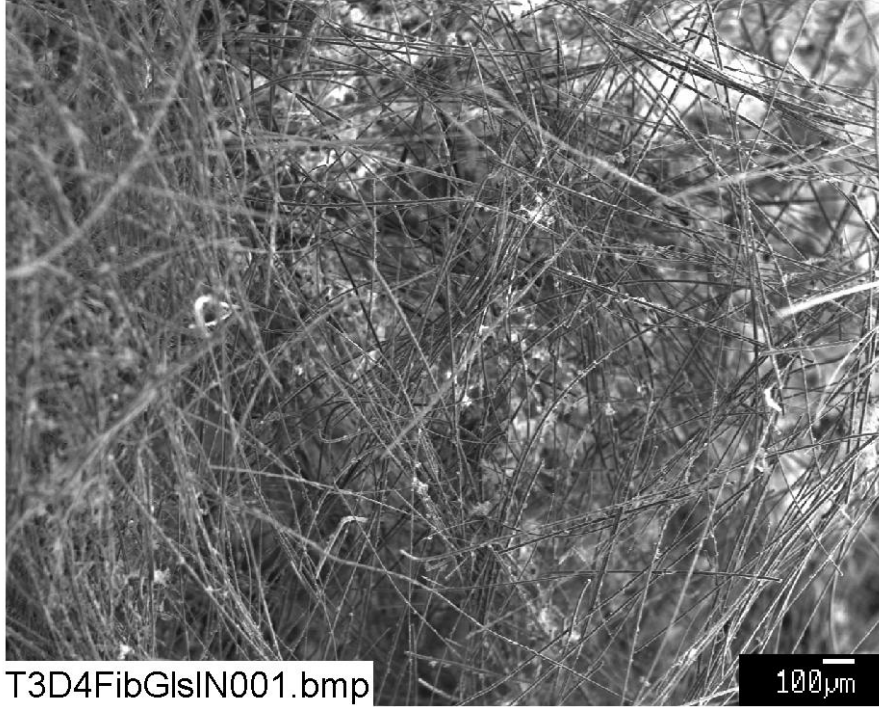


Figure 3-25. SEM image of a Test #3, Day-4 low-flow interior fiberglass sample, magnified 50 times. (T3D4FibGlsIN001, 4/12/05)

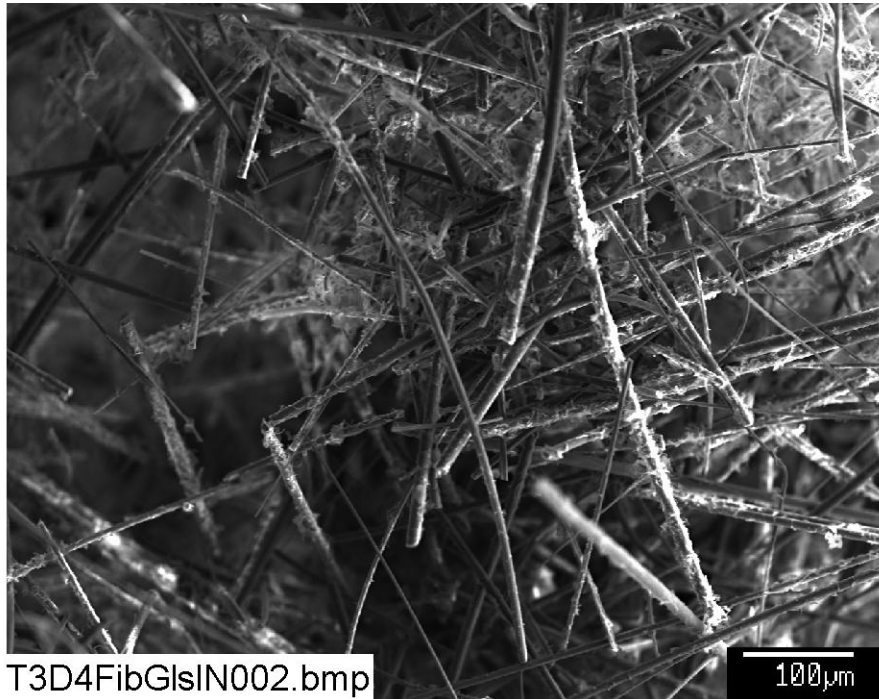


Figure 3-26. SEM image for a Test #3, Day-4 low-flow interior fiberglass sample, magnified 150 times. (T3D4FibGlsIN002, 4/12/05)

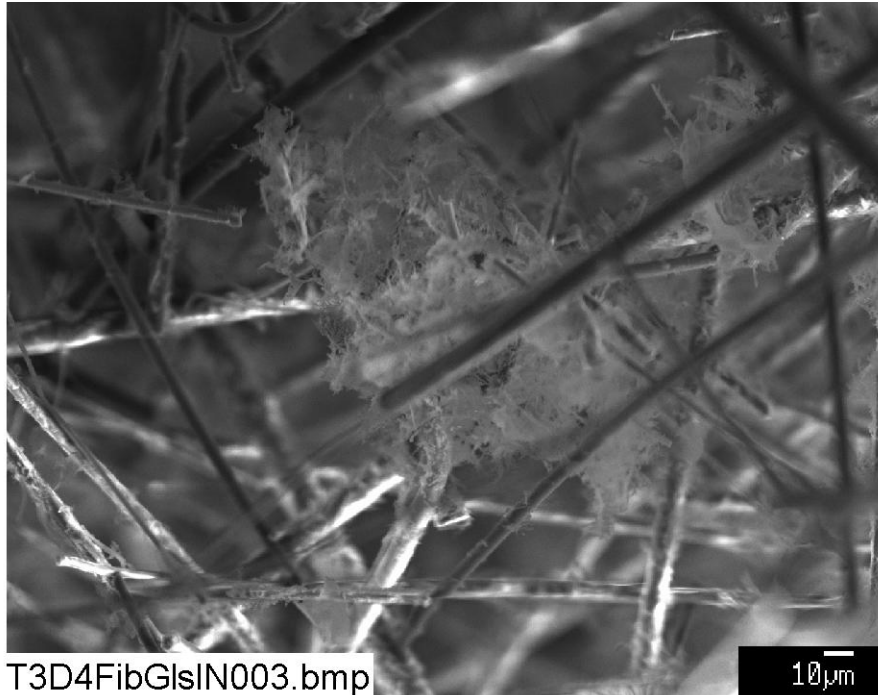


Figure 3-27. SEM image of a Test #3, Day-4 low-flow interior fiberglass sample, magnified 400 times. (T3D4FibGlsIN003, 4/12/05)

3.3.1.2. Day-15 Low-Flow Fiberglass Samples

As with Day-4 samples, dark deposits and white flocculence were found with ESEM and SEM results, respectively, on Day-15 low-flow fiberglass samples. There was no significant increase in the amount of deposits on Day-15 samples compared with Day-4 samples. Comparing the amount of deposits on the exterior and the interior Day-15 low-flow fiberglass samples revealed no significant difference. Again, EDS results show that the deposits on both of the exterior and the interior samples were commonly composed of O, Si (possible), Na, Ca, and small amounts of Mg, Al, B, and P, suggesting the deposits' likely chemical origin. Figures 3-28 through 3-38 show the Day-15 low-flow fiberglass results.

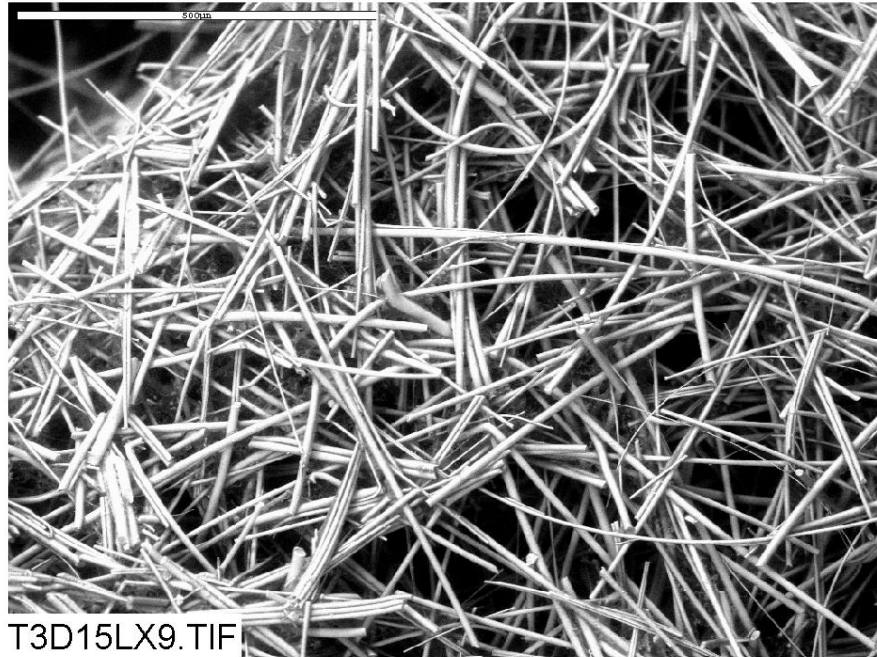


Figure 3-28. ESEM image of a Test #3, Day-15 low-flow exterior fiberglass sample, magnified 110 times. (T3D15LX9)

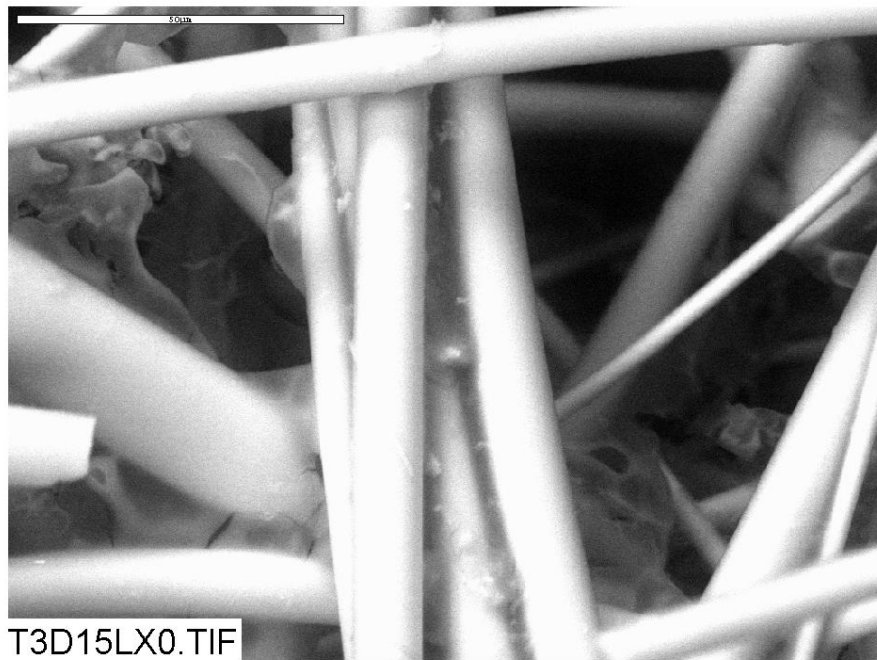


Figure 3-29. ESEM image of a Test #3, Day-15 low-flow exterior fiberglass sample, magnified 1000 times. (T3D15LX0)

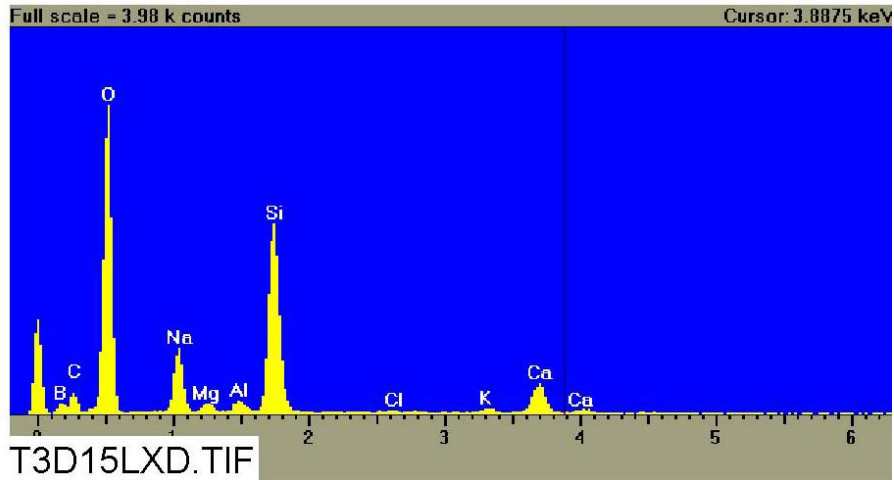


Figure 3-30. EDS counting spectrum for the deposits between the fibers on the ESEM image shown in Figure 3-29. (T3D15LXD)

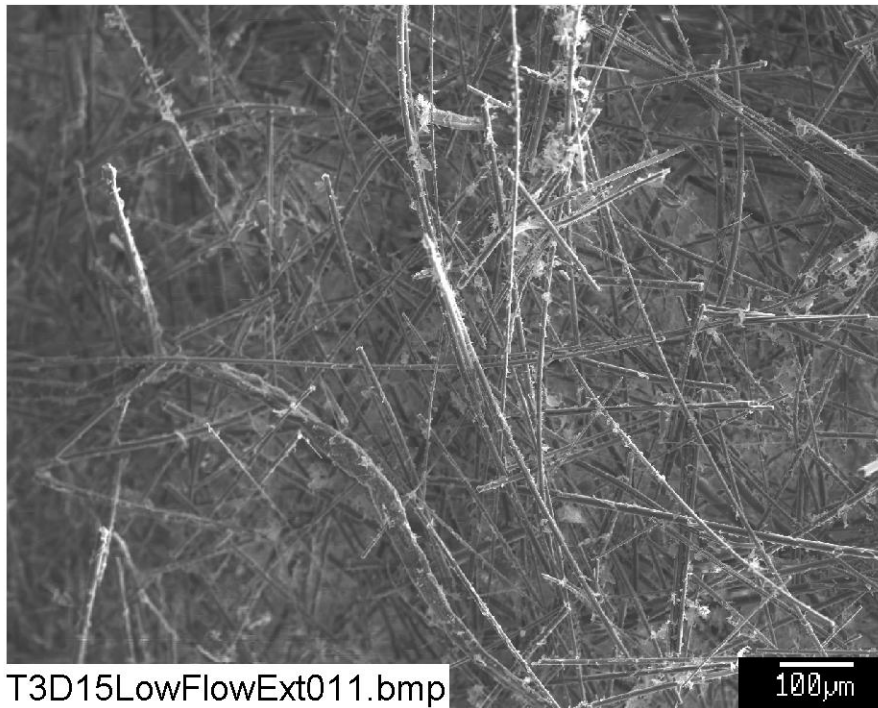


Figure 3-31. SEM image of a Test #3, Day-15 low-flow exterior fiberglass sample, magnified 100 times. (T3D15LowFlowExt011)



Figure 3-32. SEM image of a Test #3, Day-15 low-flow exterior fiberglass sample, magnified 1000 times. (T3D15LowFlowExt013)

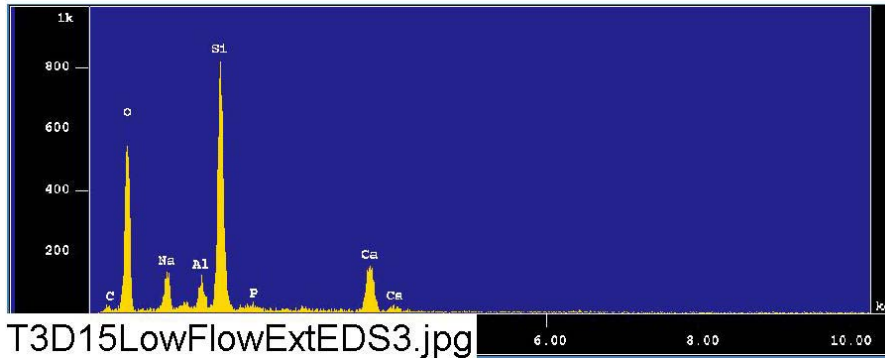


Figure 3-33. EDS counting spectrum for the flocculent deposits between the fibers on the SEM image shown in Figure 3-32. (T3D15LowFlowExtEDS3)

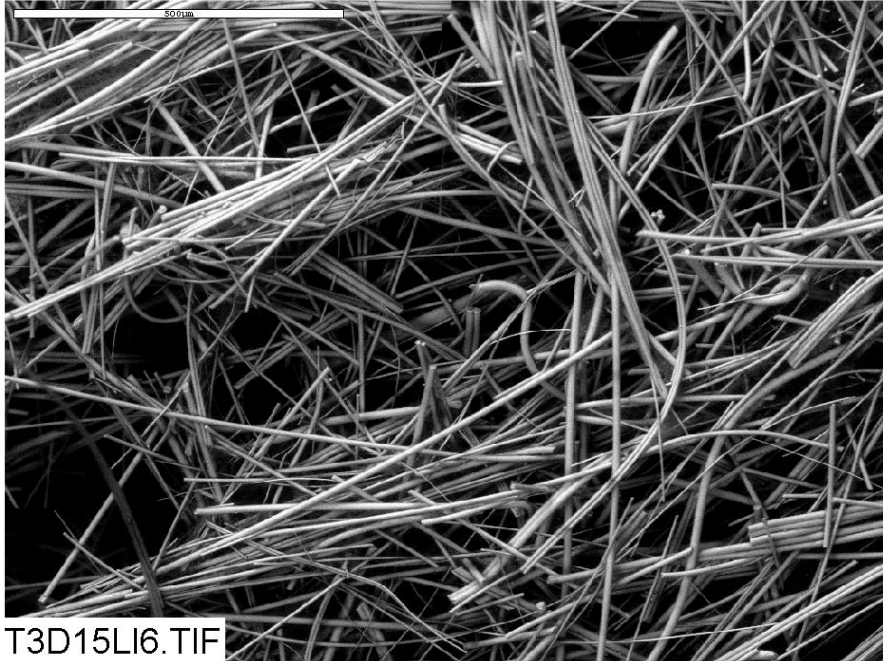


Figure 3-34. ESEM image of a Test #3, Day-15 low-flow interior fiberglass sample, magnified 100 times. (T3D15LI6)

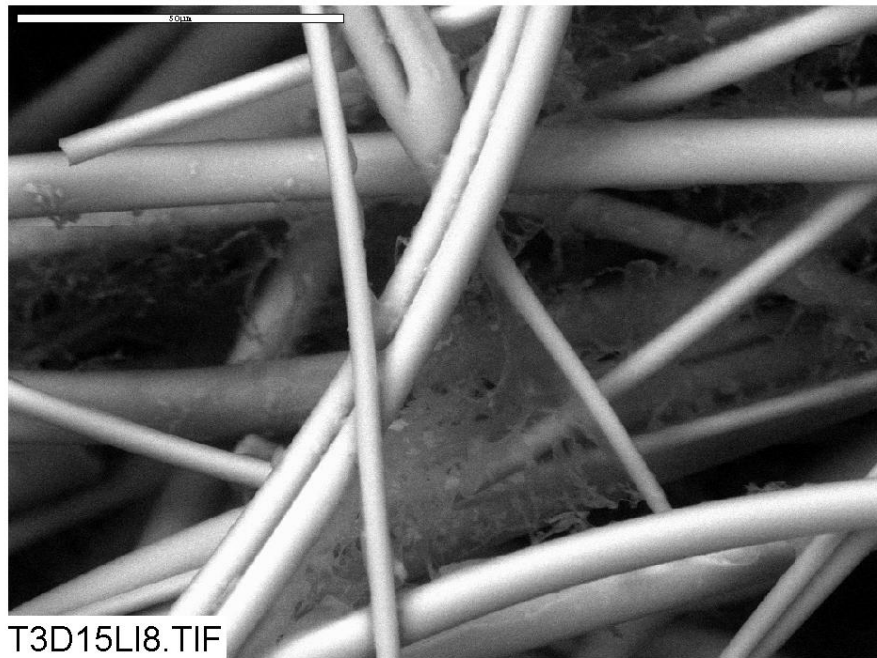


Figure 3-35. ESEM image of a Test# 3, Day-15 low-flow interior fiberglass sample, magnified 1000 times. (T3D15LI8)

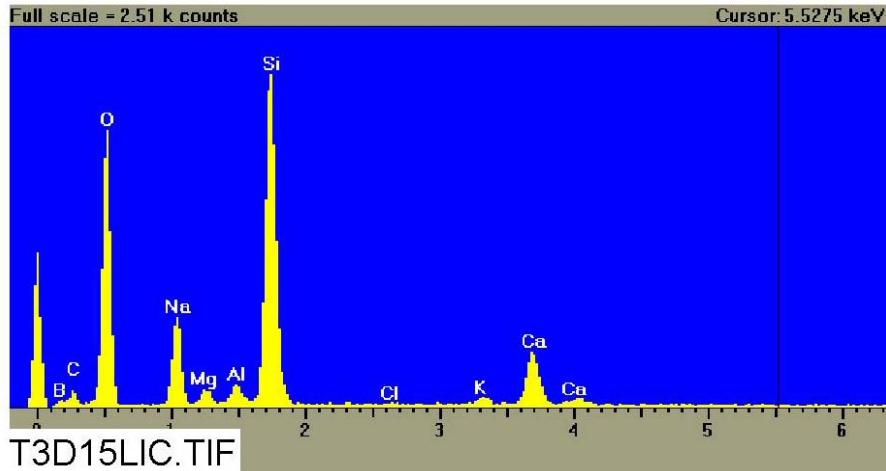


Figure 3-36. EDS counting spectrum for the flocculent deposits between the fibers on the ESEM image shown in Figure 3-35. (T3D15LIC)

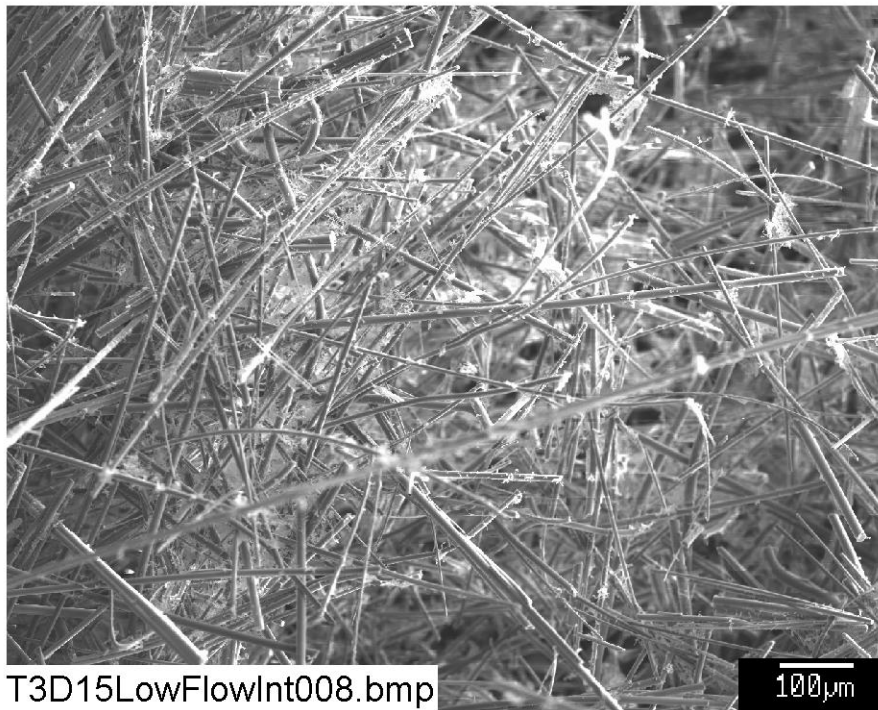


Figure 3-37. SEM image of a Test #3, Day-15 low-flow interior fiberglass sample, magnified 100 times. (T3D15LowFlowInt008)

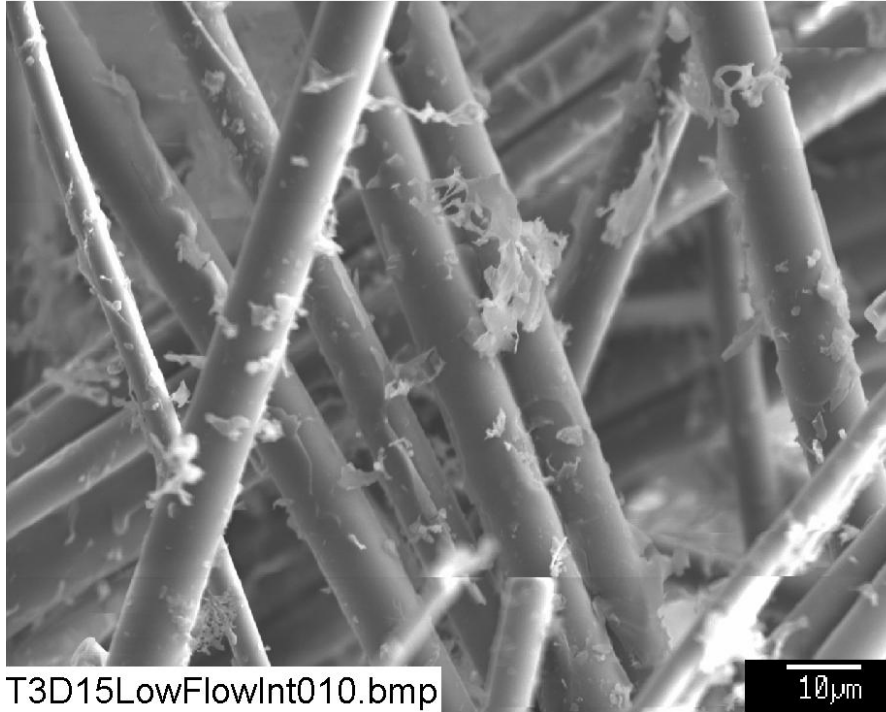


Figure 3-38. SEM image of a Test #3, Day-15 low-flow interior fiberglass sample, magnified 1000 times. (T3D15LowFlowInt010)

3.3.1.3. Day-15 High-Flow Fiberglass Samples

No significant difference was found between Day-15 high-flow and low-flow fiberglass samples, except for some large flat fibers found on the Day-15 high-flow exterior fiberglass samples (see lower left corner of Figure 3-39). These large flat fibers, which were likely from the submerged cal-sil samples (see Appendix H), were physically attached/retained on the exterior of the fiberglass samples. No large flat fibers were found in the interior of the fiberglass samples. Again, dark deposits and white flocculence were found with ESEM and SEM results, respectively, on Day-15 high-flow fiberglass samples. There was no significant difference in the amount of deposits on the exterior and interior fiberglass samples. Similarly, EDS results verified that the deposits on the exterior and interior samples were commonly composed of O, Si (possible), Na, Ca, and small amount of Mg, Al, B, and P, suggesting the deposits' likely chemical origin. Figures 3-39 through 3-50 show the Day-15 high-flow fiberglass results.

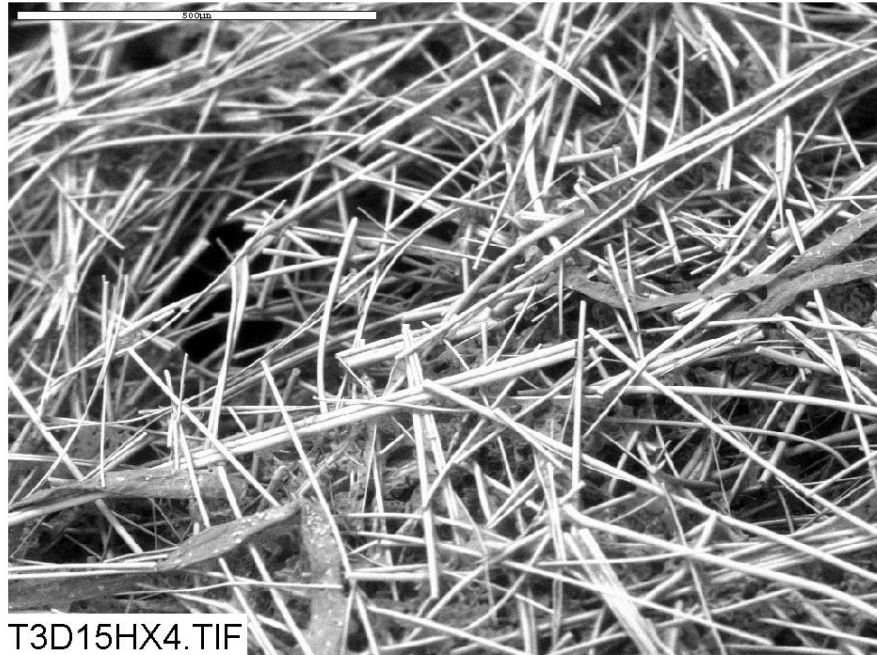


Figure 3-39. ESEM image of a Test #3, Day-15 high-flow exterior fiberglass sample, magnified 110 times. (T3D15HX4, 4/22/05)

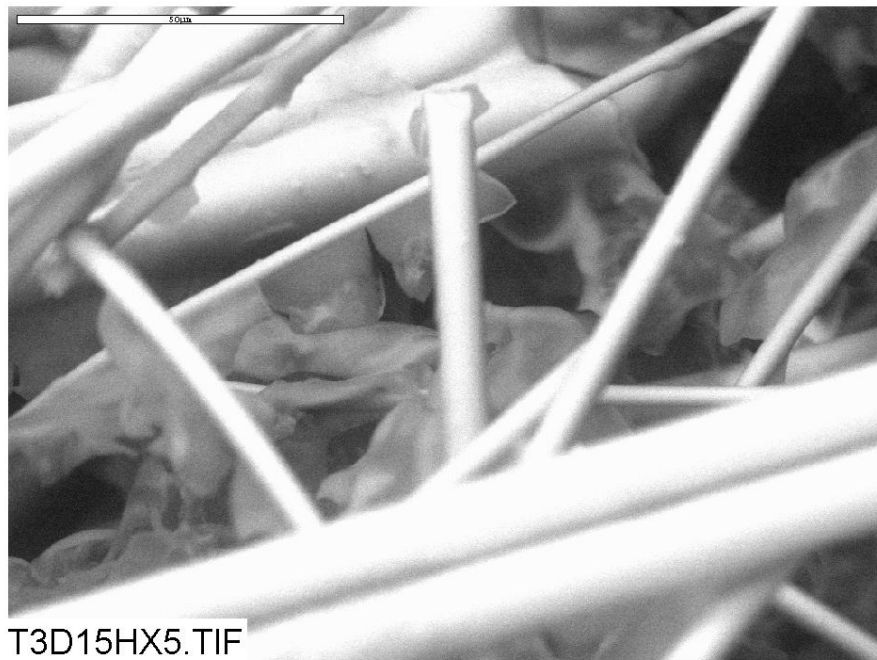


Figure 3-40. ESEM image of a Test #3, Day-15 high-flow exterior fiberglass sample, magnified 1000 times. (T3D15HX5, 4/22/05)

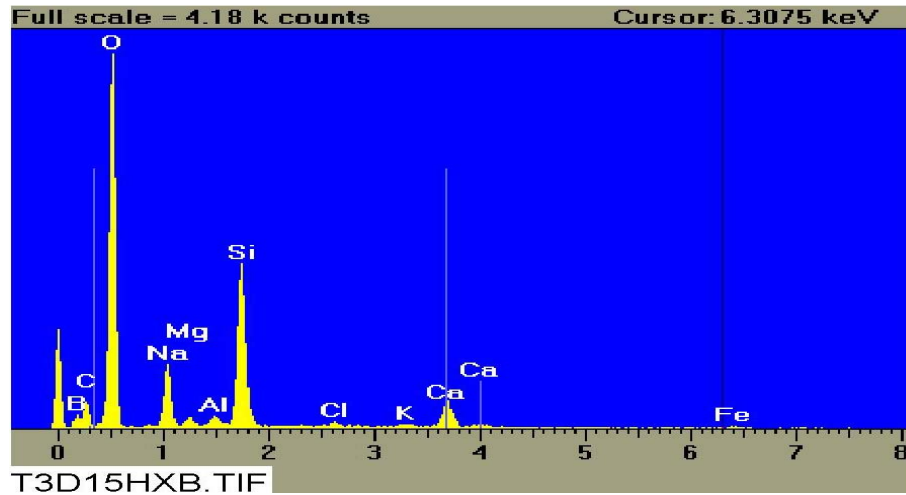


Figure 3-41. EDS counting spectrum for the deposits between the fibers on the ESEM image shown in Figure 3-40. (T3D15HIB, 4/22/05)

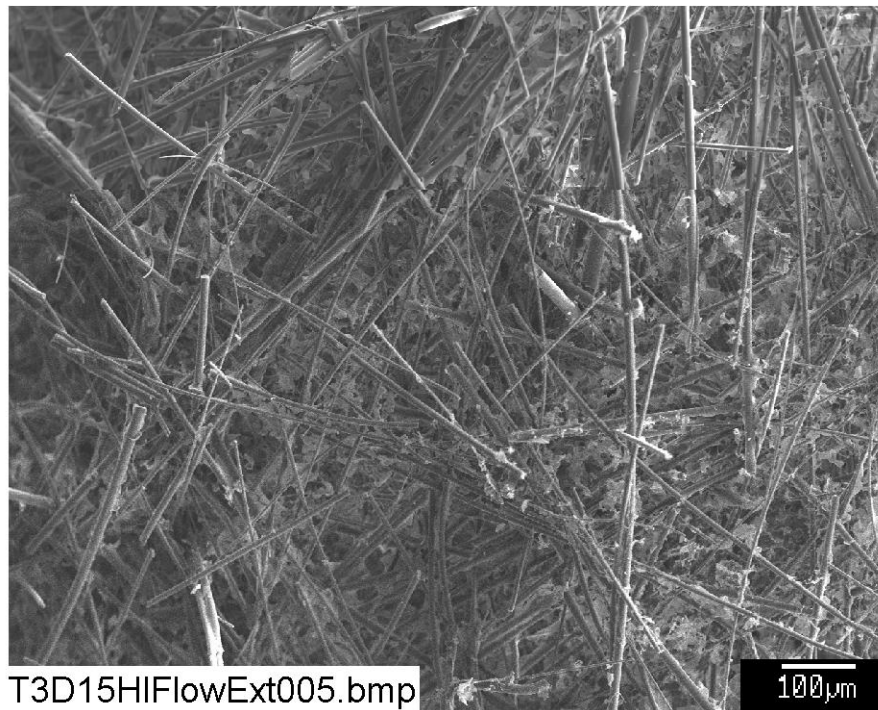


Figure 3-42. SEM image of a Test #3, Day-15 high-flow exterior fiberglass sample, magnified 100 times. (T3D15HIFlowExt005, 4/22/05)

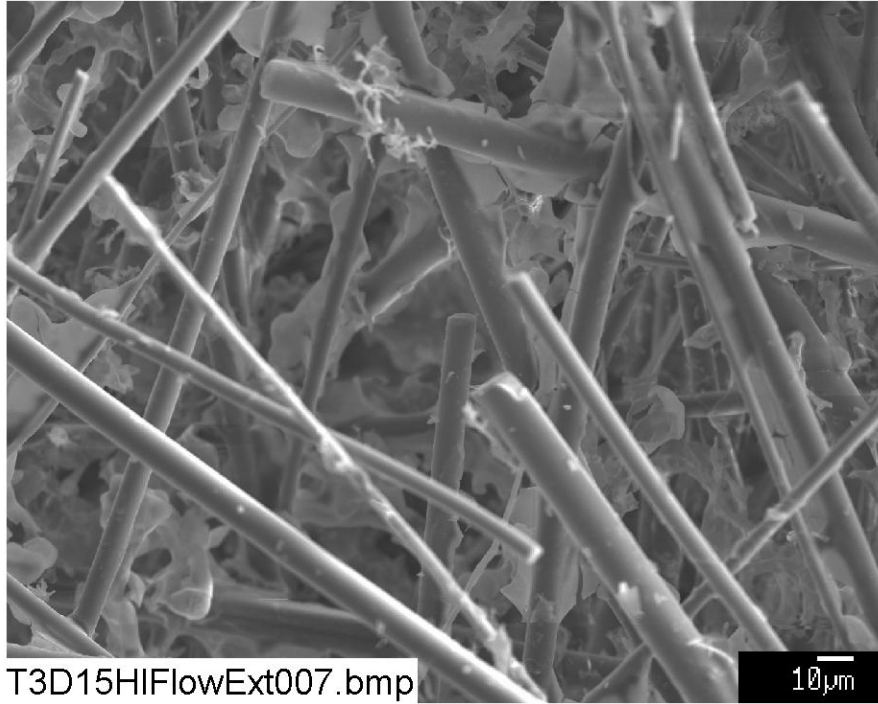


Figure 3-43. SEM image of a Test #3, Day-15 high-flow exterior fiberglass sample, magnified 500 times. (T3D15HiFlowExt007, 4/22/05)

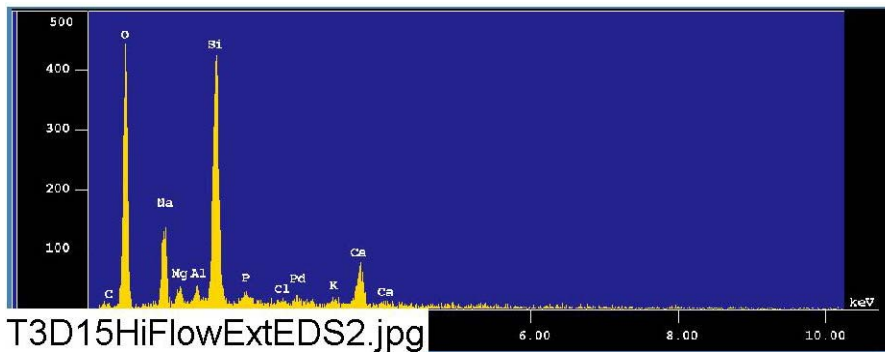


Figure 3-44. EDS counting spectrum for the flocculent deposits between the fibers on the SEM image shown in Figure 3-43. (T3D15HiFlowExtEDS2, 4/22/05)

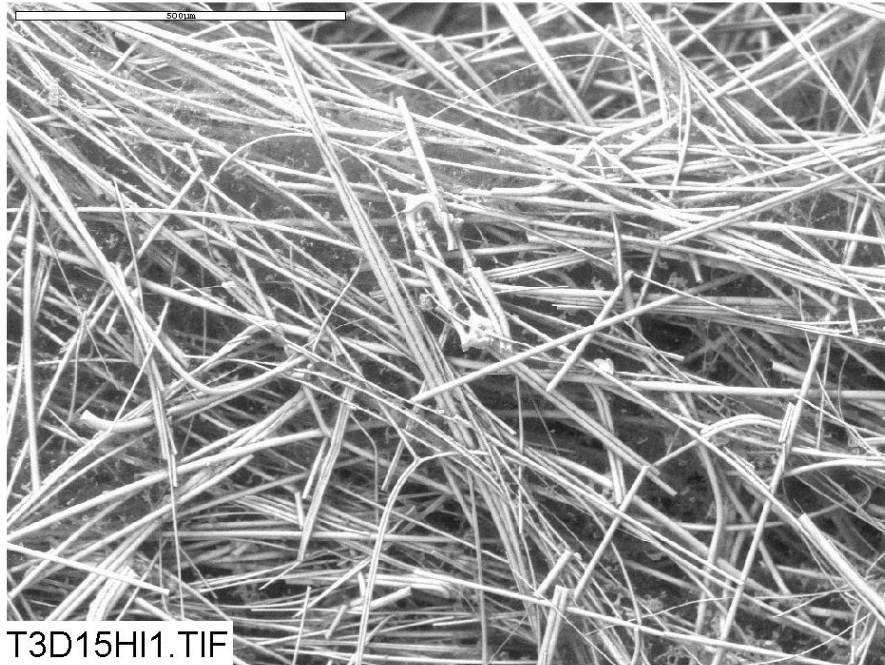


Figure 3-45. ESEM image of a Test #3, Day-15 high-flow interior fiberglass sample, magnified 100 times. (T3D15HI1, 4/22/05)

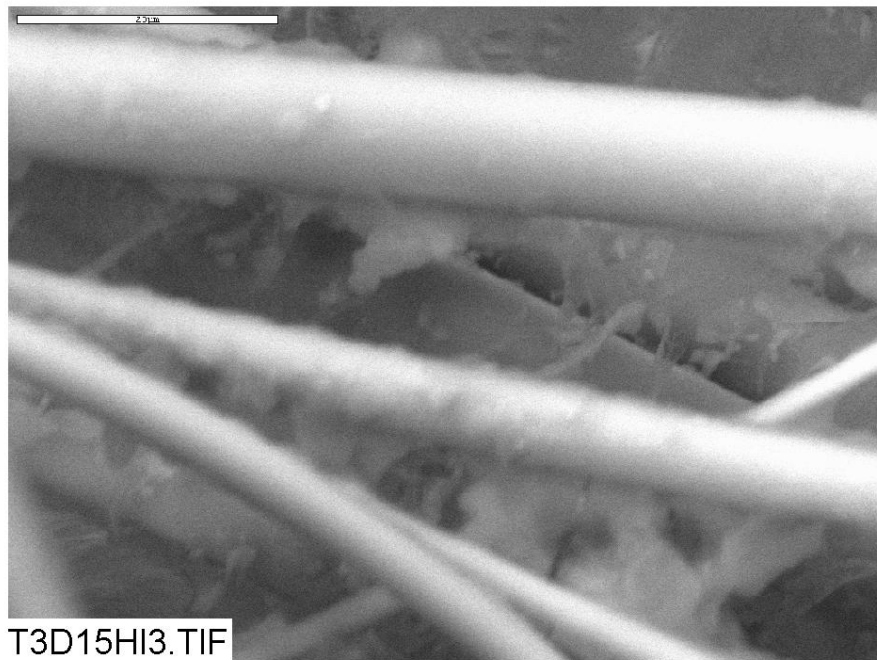


Figure 3-46. ESEM image of a Test #3, Day-15 high-flow interior fiberglass sample, magnified 2000 times. (T3D15HI3, 4/22/05)

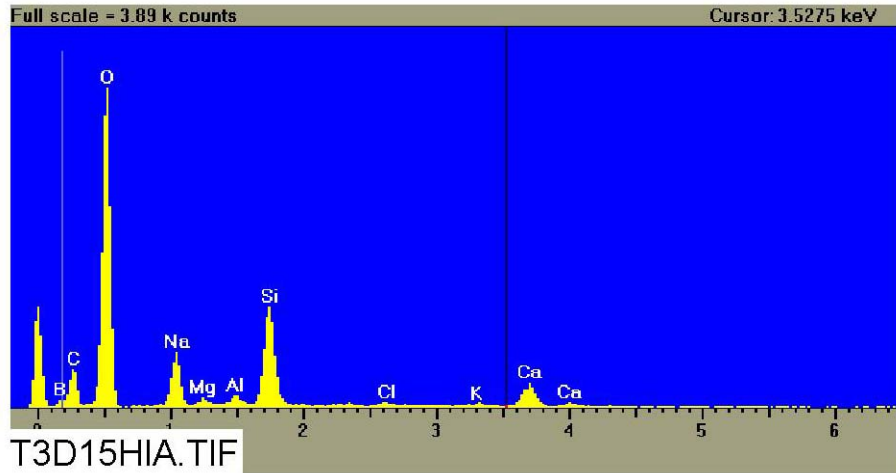


Figure 3-47. EDS counting spectrum for the flocculent deposits between the fibers on the ESEM image shown in Figure 3-46. (T3D15HIA, 4/22/05)

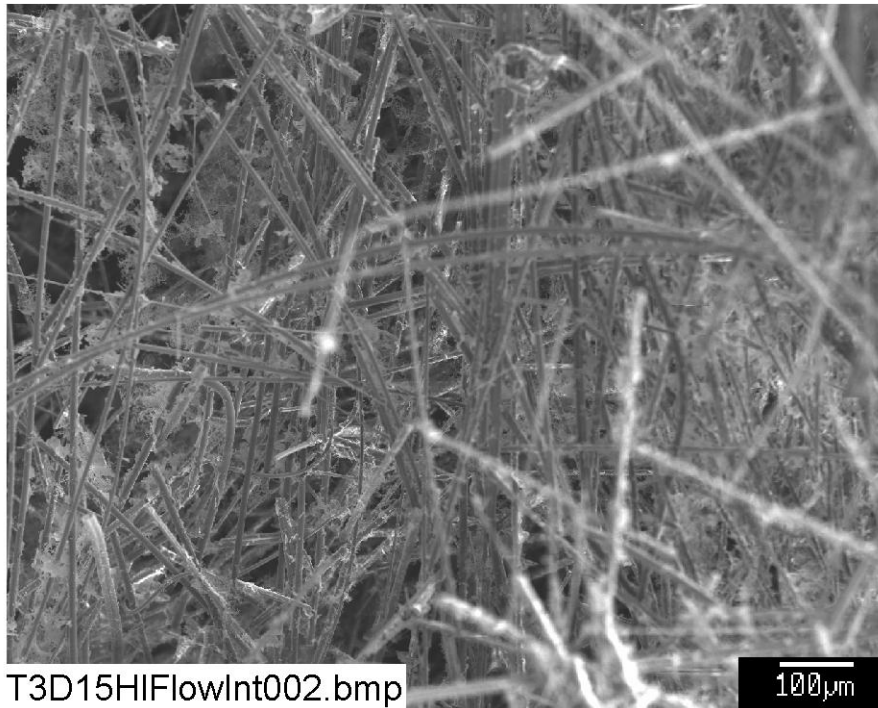


Figure 3-48. SEM image of a Test #3, Day-15 high-flow interior fiberglass sample, magnified 100 times. (T3D15HIFlowInt002, 4/22/05)

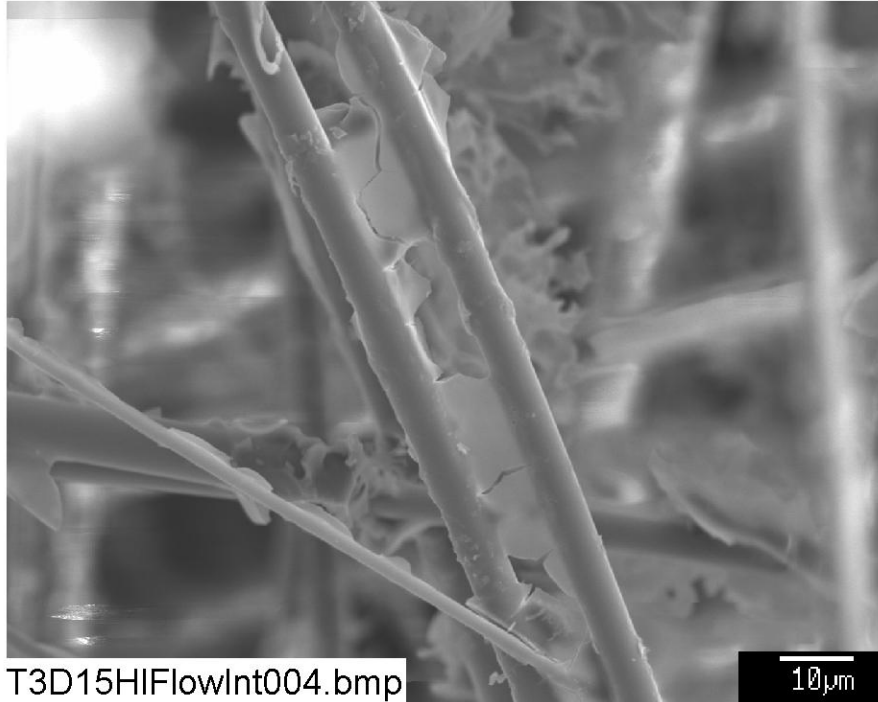


Figure 3-49. SEM image of a Test #3, Day-15 high-flow interior fiberglass sample, magnified 1000 times. (T3D15HiFlowInt004, 4/22/05)

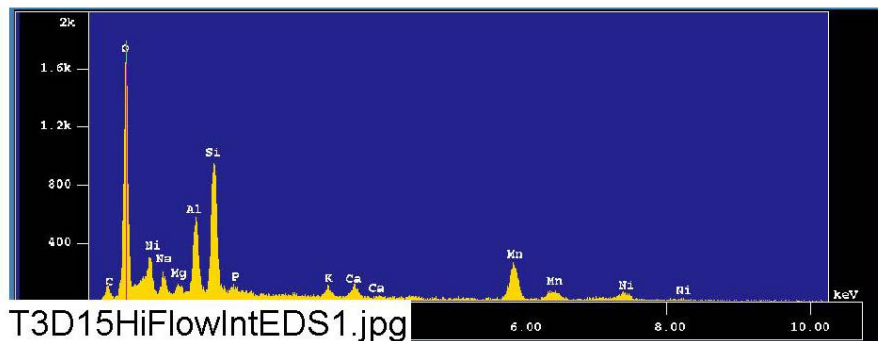


Figure 3-50. EDS counting spectrum for the flocculent deposits between the fibers on the SEM image shown in Figure 3-49. (T3D15HiFlowIntEDS1, 4/22/05)

3.3.1.4. Day-30 Low-Flow Fiberglass Samples

Comparing Day-30 low-flow fiberglass samples with Day-4 and Day-15 low-flow fiberglass samples revealed deposits that are similar in property and amount. In addition, there was no significant difference in the amount of deposits found in exterior and interior Day-30 low-flow fiberglass samples. Figures 3-51 through 3-54 show the Day-30 low-flow fiberglass results.

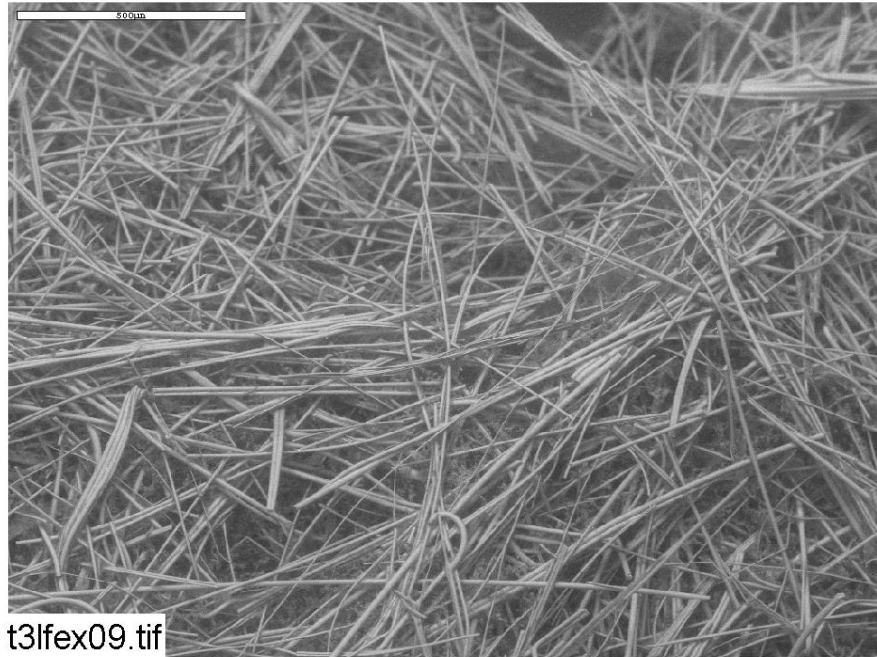


Figure 3-51. ESEM image of a Test #3, Day-30 exterior low-flow fiberglass sample, magnified 70 times. (t3lfex09, 5/6/05)

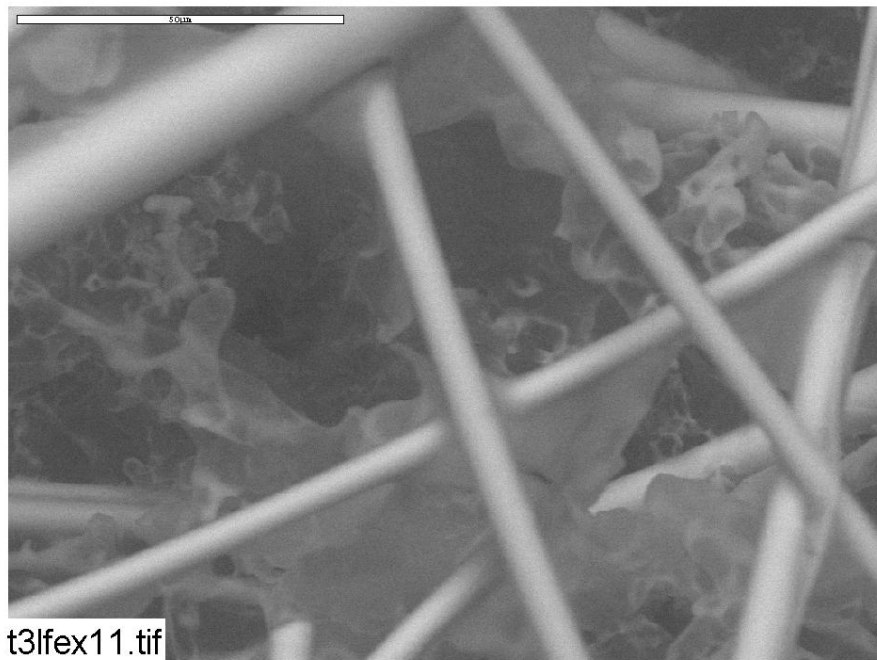


Figure 3-52. ESEM image of a Test #3, Day-30 exterior low-low fiberglass sample, magnified 1000 times. (t3lfex11, 5/6/05)

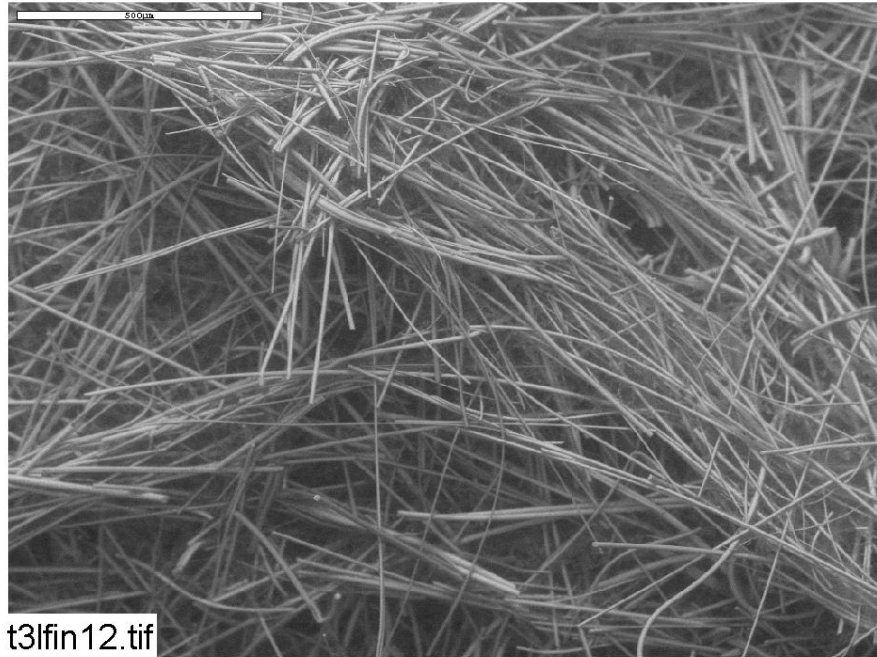


Figure 3-53. ESEM image of a Test #3, Day-30 interior low-flow fiberglass sample, magnified 70 times. (t3lfin12, 5/6/05)

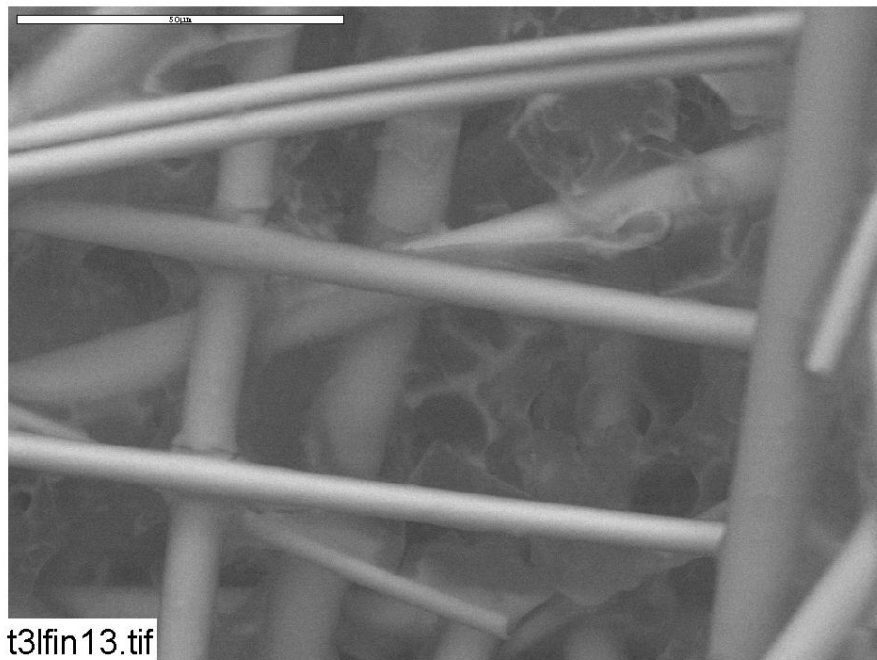


Figure 3-54. ESEM image of a Test #3, Day-30 interior low-flow fiberglass sample, magnified 1000 times. (t3lfin13, 5/6/05)

3.3.1.5. Day-30 High-Flow Fiberglass Samples

Compared with other high- and low-flow fiberglass samples, Day-30 high-flow exterior samples contained a significant amount of particulates. As opposed to the exterior samples, the interior samples were relatively clean, suggesting that the particulate deposits were physically attached/retained on the fiberglass exterior. EDS results show that the particulate deposits were composed of a significant amount of P, which is different from the previous fiberglass samples. The deposits' high P, Ca, and O content suggests that the deposits were $\text{Ca}_3(\text{PO}_4)_2$, which relates to the white gel (cream) formed during the injection of TSP. That $\text{Ca}_3(\text{PO}_4)_2$ was likely precipitated out from the testing solution, followed by sedimentation/transportation onto the Day-30 high-flow fiberglass exterior. Figures 3-55 through 3-59 show the Day-30 high-flow fiberglass results.

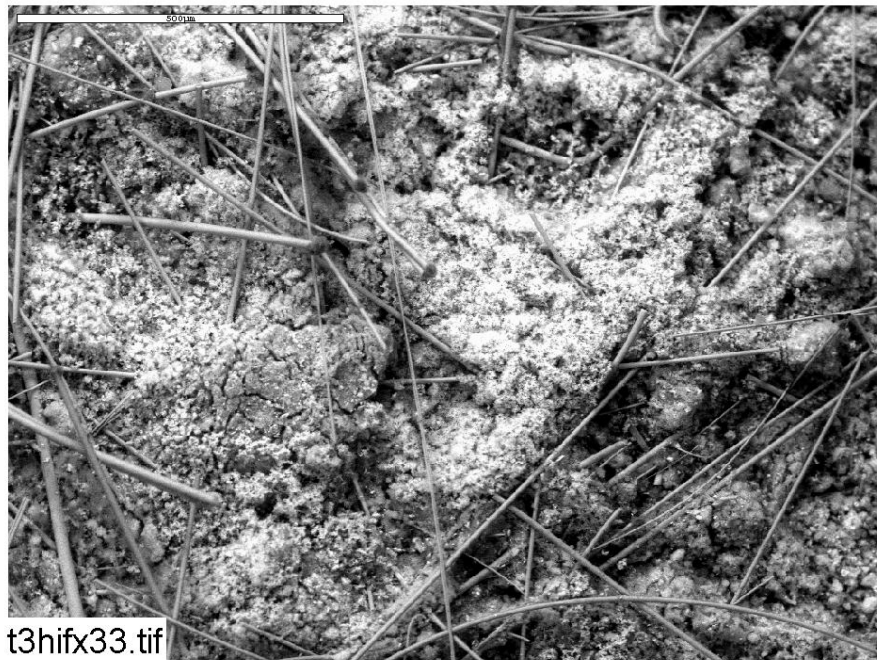


Figure 3-55. ESEM image of a Test #3, Day-30 exterior high-flow fiberglass sample, magnified 100 times. (t3hifx33, 5/11/05)

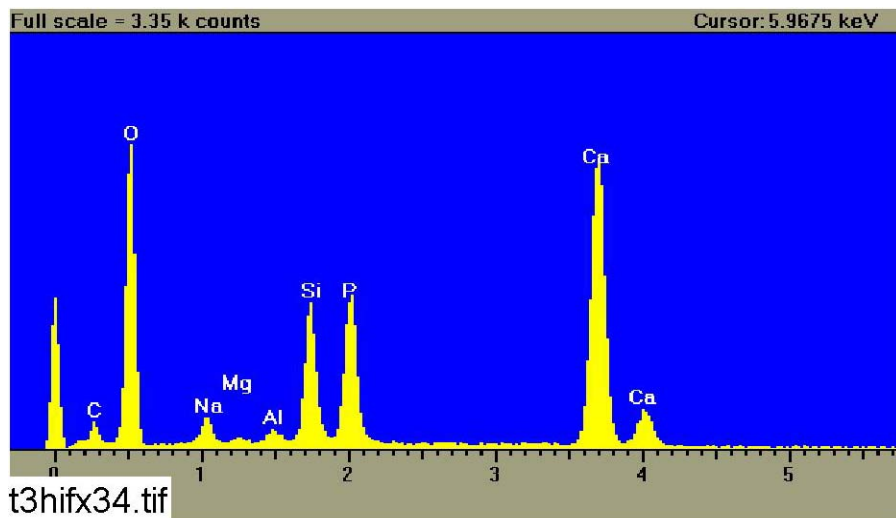


Figure 3-56. EDS counting spectrum for the large masses of particulate deposits shown in Figure 3-55. (t3hifx34, 5/11/05)



Figure 3-57. ESEM image of a Test #3, Day-30 exterior high-flow fiberglass sample, magnified 600 times. (t3hifx35, 5/11/05)

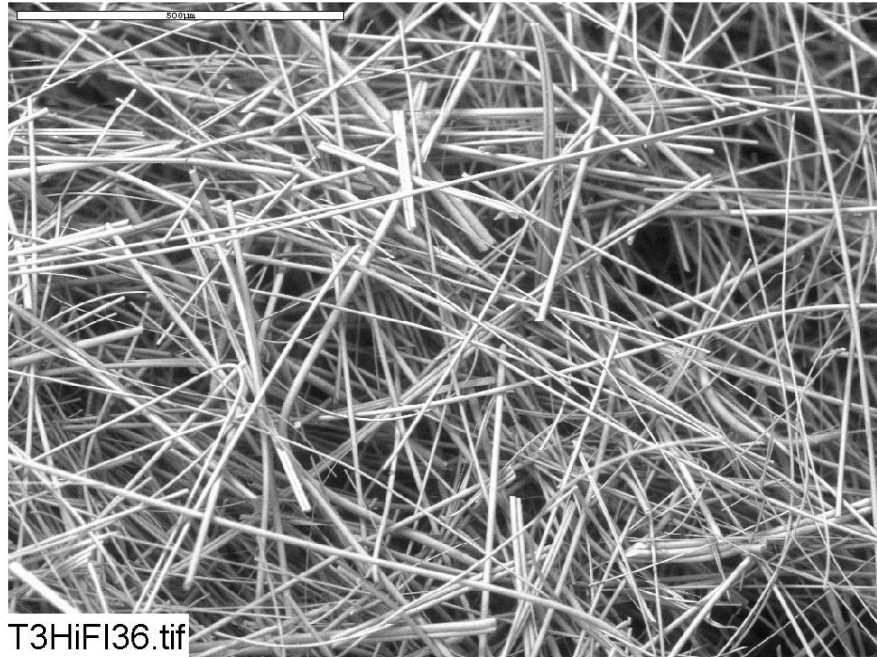


Figure 3-58. ESEM image of a Test #3, Day-30 interior high-flow fiberglass sample, magnified 100 times. (T3HiFI36, 5/11/05)

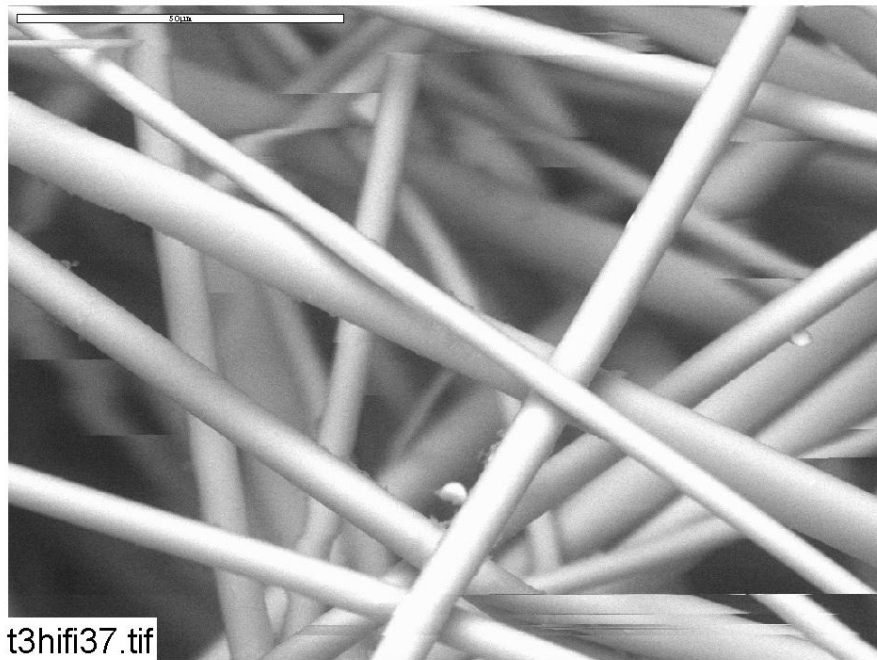


Figure 3-59. ESEM image of a Test #3, Day-30 interior high-flow fiberglass sample, magnified 1000 times. (t3hifi37, 5/11/05)

3.3.1.6. Day-30 Drain Collar Fiberglass Samples

Figure 3-60 shows the drain collar after it was removed from the tank. When the tank was drained, the drain collar was totally surrounded by sediment. Both the exterior fiberglass sample that was farthest from the drain screen and the exterior sample that was next to the drain screen have very significant amounts of particulate deposits. Inspection revealed the development of a continuous coating on the drain collar exterior, including particulate deposits that were likely physically retained or attached. The amount of deposits on the drain collar exterior was greater than on high- and low-flow fiberglass samples. The ESEM images showed that two types of material were retained; an amorphous material that appeared darker in the ESEM images and a lighter granular material. The EDS results indicate that these materials had different P and Si content. The lighter particulate deposits (see Figure 3-62) have a higher percentage of P and a lower percentage of Si than the dark deposits, suggesting that light particulate deposits are likely composed of $\text{Ca}_3(\text{PO}_4)_2$ precipitates and dark deposits of cal-sil particles. Both kinds of deposits could have been transported and/or deposited/retained on the drain collar fiberglass exterior. As opposed to what was found on the exterior sample, no significant deposits were found in the drain collar interior sample, suggesting that that almost all of the particulate deposits were physically retained at the fiberglass exterior. The result is consistent with findings for the Day-30 high-flow fiberglass samples. Figures 3-61 through 3-70 show the drain Day-30 drain collar fiberglass results.



Figure 3-60. Drain screen collar removed from the tank.

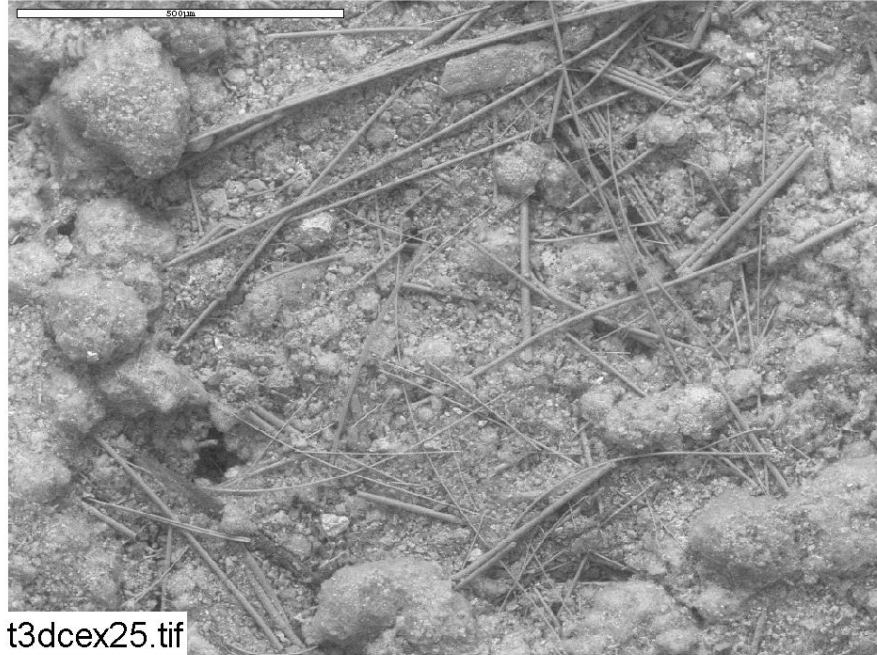


Figure 3-61. ESEM image of a Test #3, Day-30 exterior fiberglass sample on the drain collar (away from the drain screen), magnified 100 times. (t3dcex25, 5/6/05)

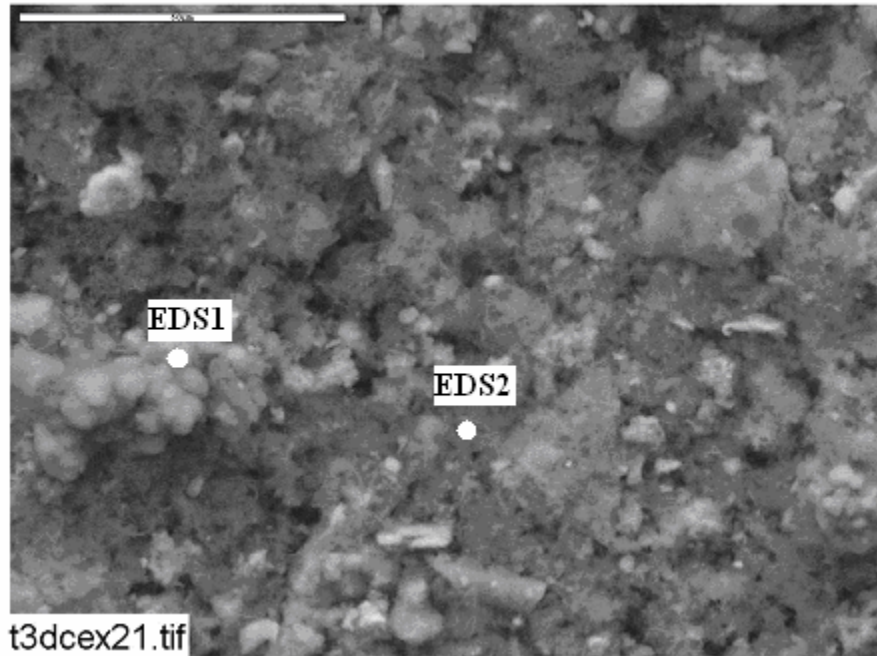


Figure 3-62. ESEM image of a Test #3, Day-30 exterior fiberglass sample on the drain collar (away from the drain screen), magnified 1000 times. (t3dcex21, 5/6/05)

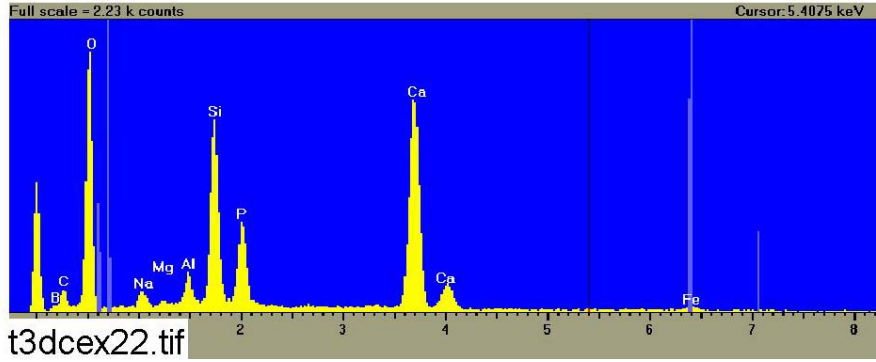


Figure 3-63. EDS counting spectrum for the light particulate deposits (EDS1) shown in Figure 3-62. (t3dcex22, 5/6/05)

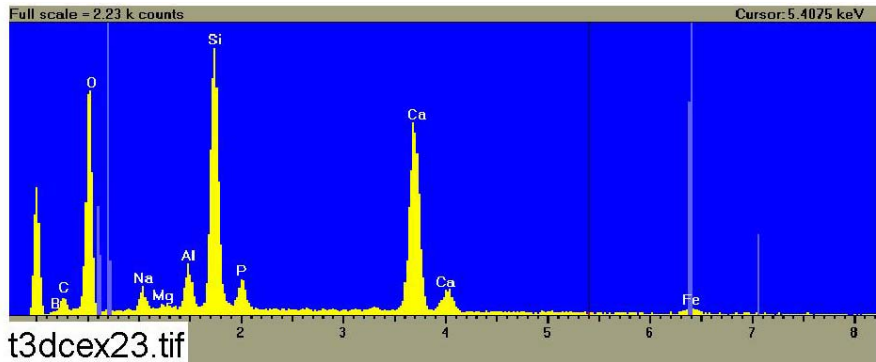


Figure 3-64. EDS counting spectrum for the dark deposits (EDS2) shown in Figure 3-62. (t3dcex23, 5/6/05)

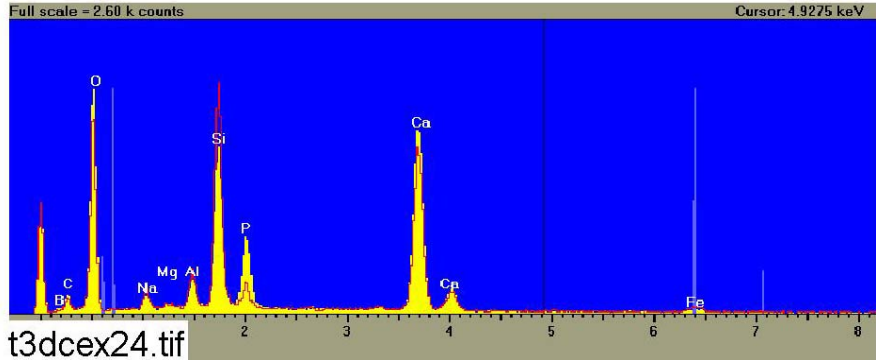


Figure 3-65. Comparison of EDS counting spectra between Figure 3-63 (yellow) and Figure 3-64 (red). (t3dcex24, 5/6/05)



Figure 3-66. ESEM image of a Test #3, Day-30 exterior fiberglass sample on the drain collar (adjacent to the drain screen), magnified 100 times. (t3DCSC16, 5/6/05)

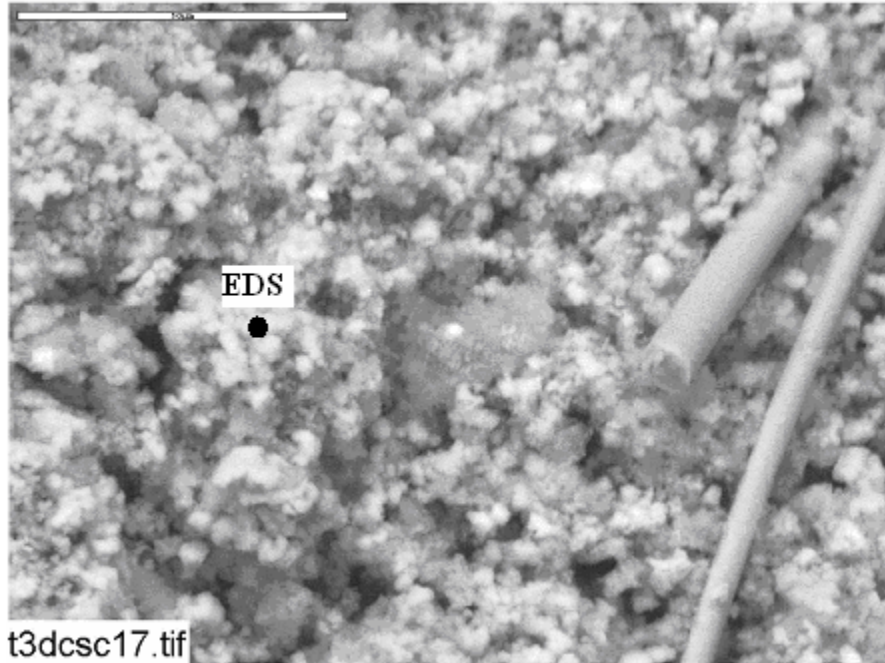


Figure 3-67. ESEM image of a Test #3, Day-30 exterior fiberglass sample on the drain collar (adjacent to the drain screen), magnified 1000 times. (t3dcsc17, 5/6/05)

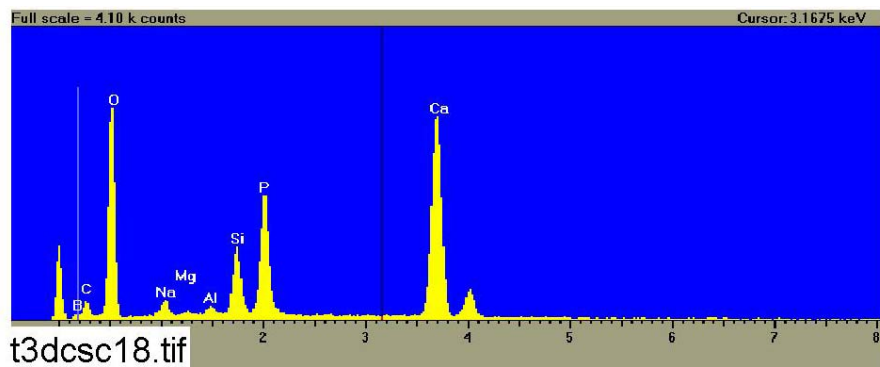


Figure 3-68. EDS counting spectrum for the particulate deposits shown in Figure 3-67. (t3dcsc18, 5/6/05)

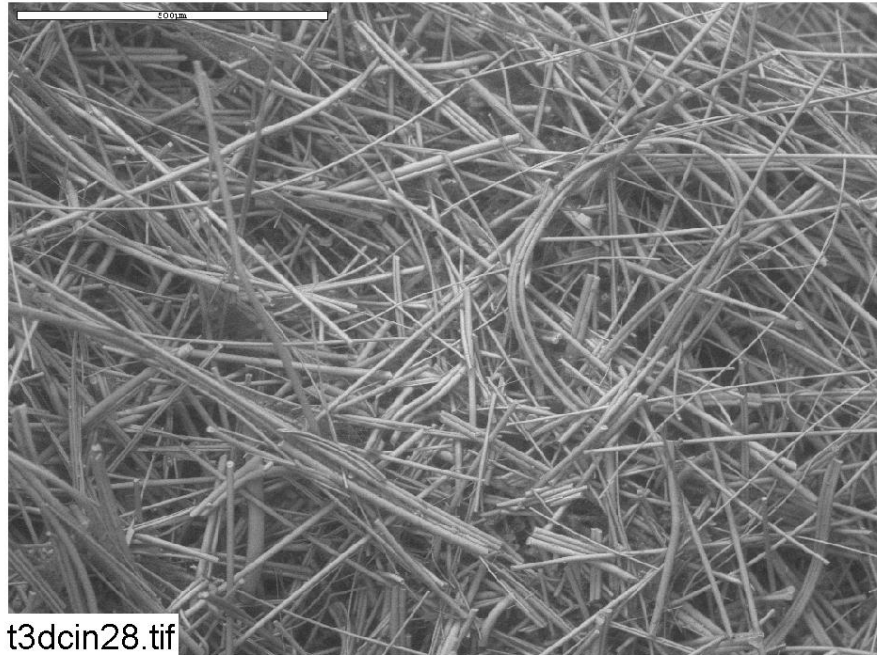


Figure 3-69. ESEM image of a Test #3, Day-30 interior fiberglass sample on the drain collar, magnified 100 times. (t3dcin28, 5/6/05)

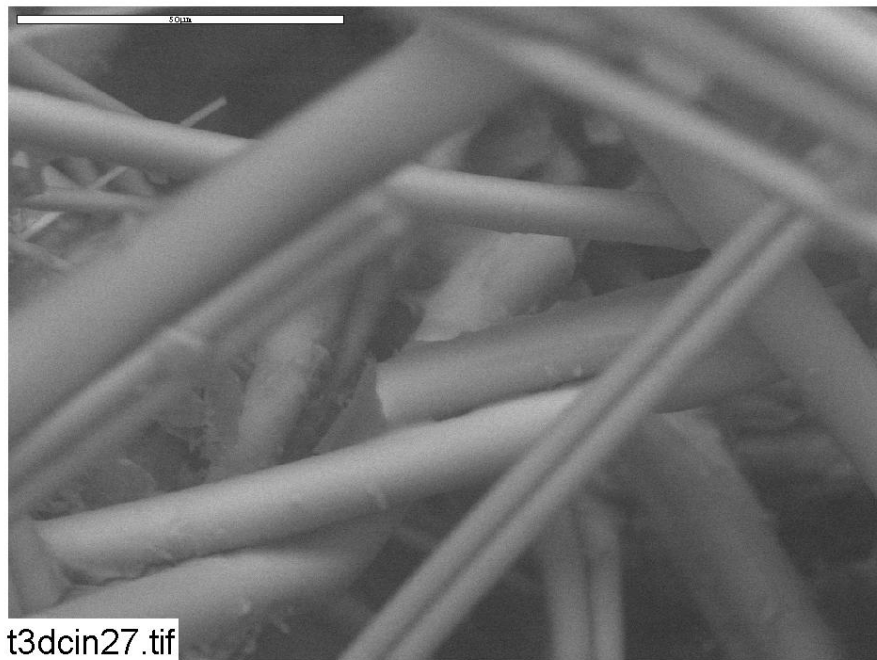


Figure 3-70. ESEM image of a Test #3, Day-30 interior fiberglass sample on the drain collar, magnified 1000 times. (t3dcin27, 5/6/05)

3.3.1.7. Day-30 Fiberglass Sample within the Birdcage

The test fluid was drained from the tank at the end of the 30-day test. Figure 3-71 shows the bottom of the tank after the tank was emptied. The birdcage is the cube in the center bottom of the figure, covered on its top with and sitting in light-colored gel-like material. For the Day-30 fiberglass sample within the birdcage, SEM images indicate large deposits (Figure 3-72) as well as a continuous coating (Figure 3-74) over the exterior of the fiberglass. The amount of particulate deposits within the birdcage was greater than on high- and low-flow fiberglass samples. The EDS result shows that the large particulate deposits had higher P and lower Si percentages than did the continuous coating shown in Figure 3-74. As with the particulate deposits on the drain collar, the large deposits are likely composed of $\text{Ca}_3(\text{PO}_4)_2$ precipitates, while the continuous coating was likely cal-sil particles. Both kinds of deposits were physically transported and/or deposited/retained on the birdcage fiberglass exterior. Compared with the exterior sample, the interior sample was relatively clean. Only small amounts of deposits were found. These deposits were similar to the deposits observed on high- and low-flow interior samples, which were 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 conditions on the Day-30 high-flow and drain-collar fiberglass samples. Figures 3-72 through 3-79 show the Day-30 birdcage fiberglass results.



Figure 3-71. Tank bottom after the test fluid was drained.

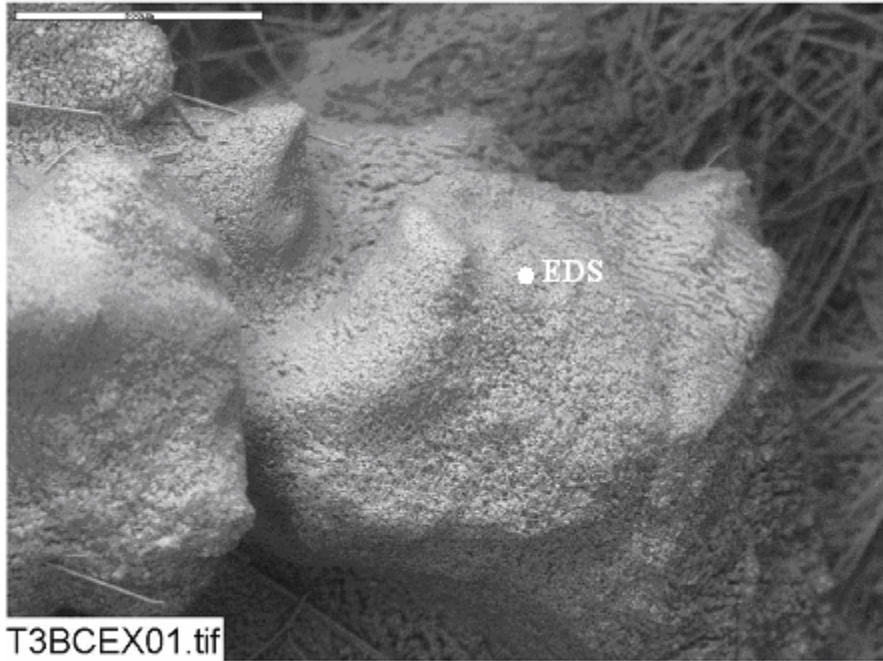


Figure 3-72. ESEM image of a Test #3, Day-30 exterior fiberglass sample within the birdcage, magnified 80 times. (T3BCEX01, 5/6/05)

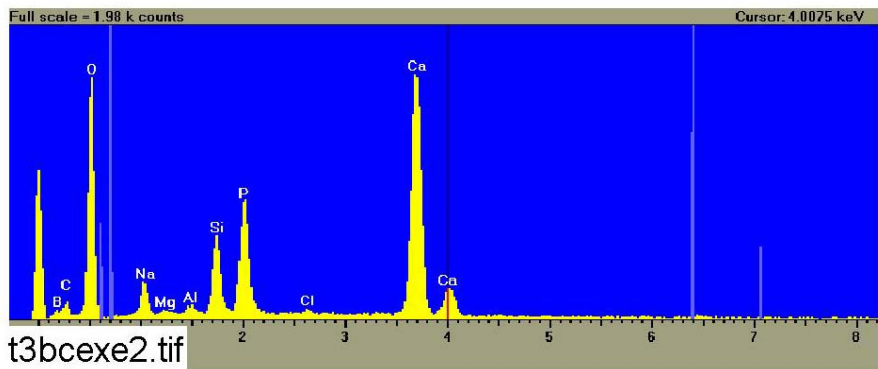


Figure 3-73. EDS counting spectrum for the large deposits shown in Figure 3-72. (t3bcexe2, 5/6/05)

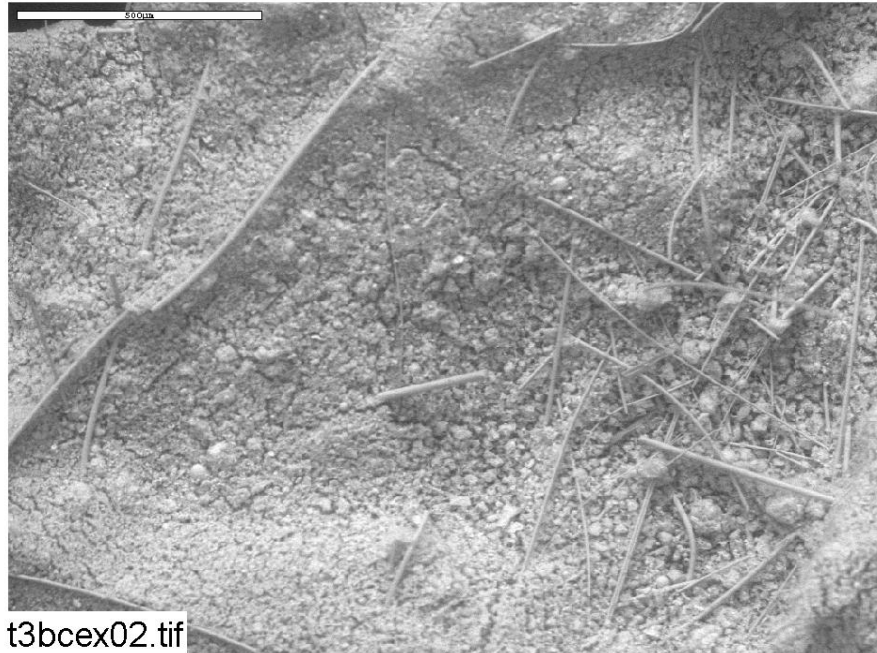


Figure 3-74. ESEM image of a Test #3, Day-30 exterior fiberglass sample within the birdcage, magnified 80 times. (t3bcex02, 5/6/05)

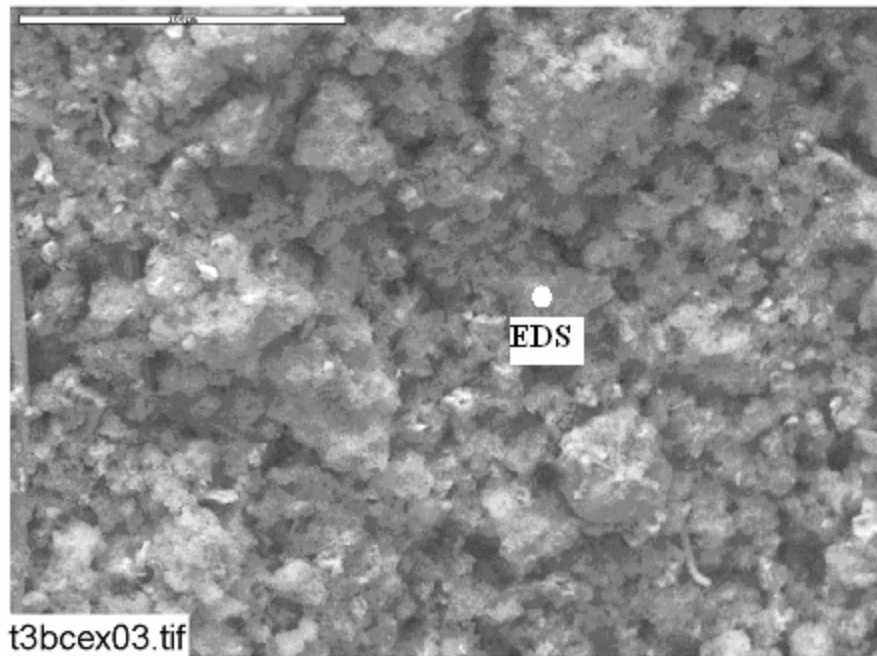


Figure 3-75. ESEM image of a Test #3, Day-30 exterior fiberglass sample within the birdcage, magnified 500 times. (t3bcex03, 5/6/05)

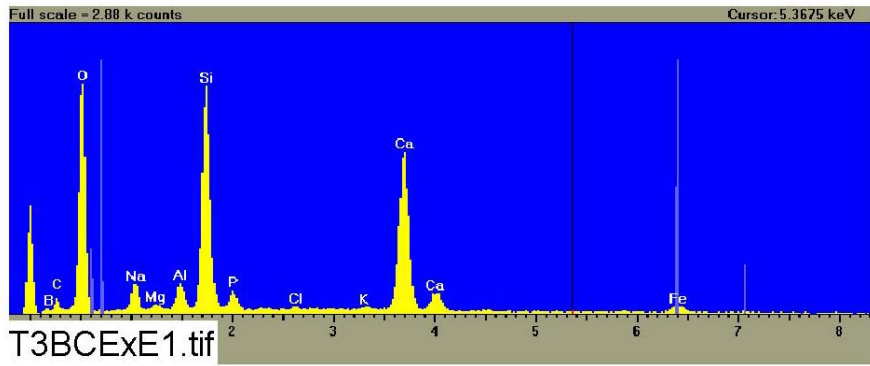


Figure 3-76. EDS counting spectrum for the deposits shown in Figure 3-75. (T3BCExE1, 5/6/05)

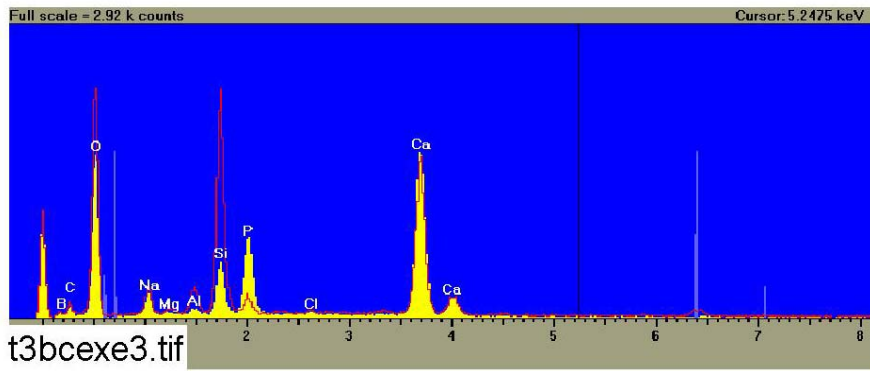


Figure 3-77. Comparison of EDS counting spectra of Figure 3-76 (red) and Figure 3-73 (yellow). (t3bcexe3, 5/6/05)

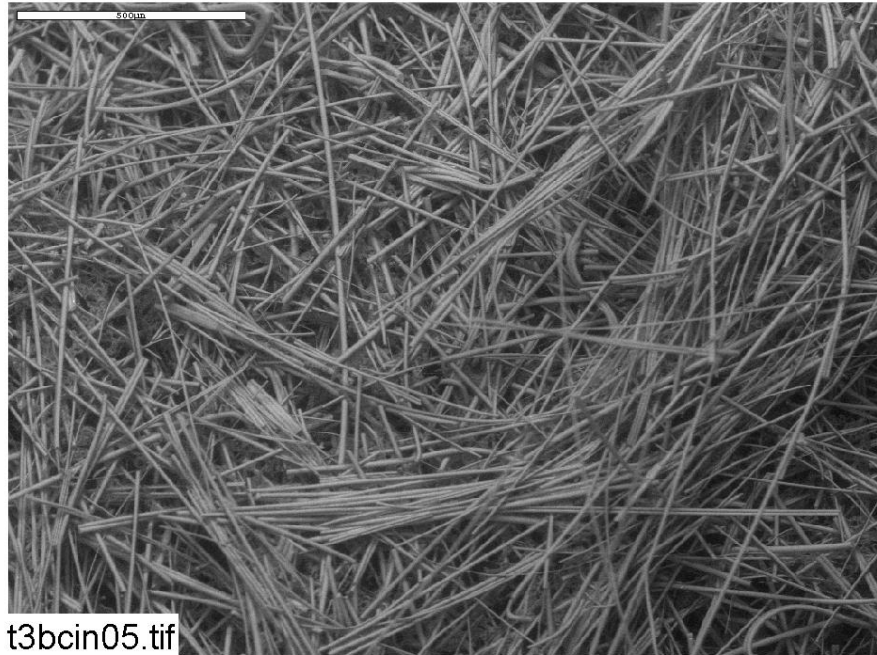


Figure 3-78. ESEM image of a Test #3, Day-30 interior fiberglass sample within the birdcage, magnified 80 times. (t3bcin05, 5/6/05)

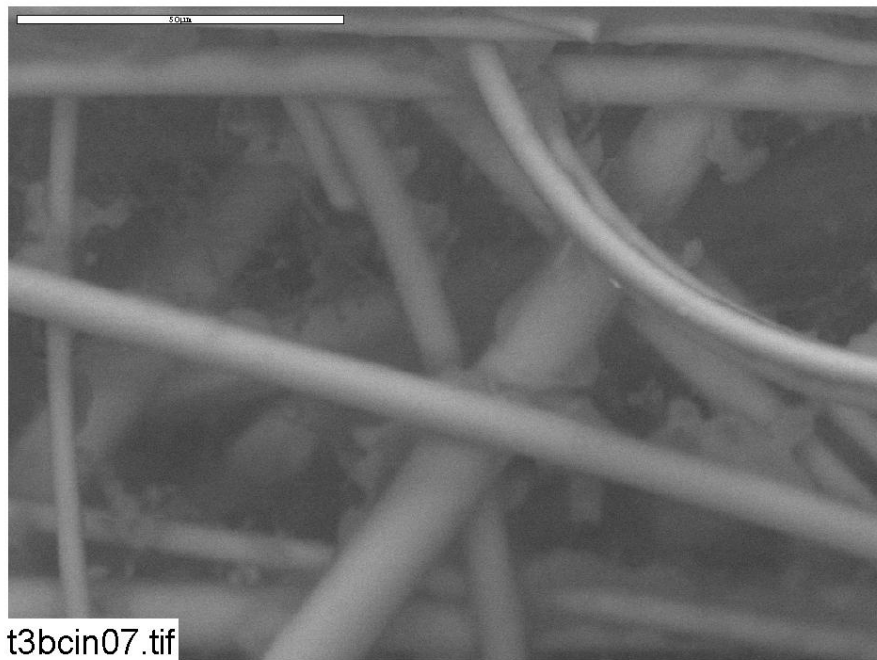


Figure 3-79. ESEM image of a Test #3, Day-30 interior fiberglass sample within the birdcage, magnified 1000 times. (t3bcin07, 5/6/05)

3.3.2. Cal-Sil Samples

Test #3 was the first ICET test that included cal-sil in addition to fiberglass samples. XRD/XRF results show the crystal structure and the chemical composition of the unused raw and unused baked cal-sil samples. Based on XRD results, both unused raw and unused baked cal-sil samples contained crystalline substances of tobermorite ($\text{Ca}_{2.25}(\text{Si}_3\text{O}_{7.5}(\text{OH})_{1.5})(\text{H}_2\text{O})$) and calcite (CaCO_3). XRF results indicated that the dominant elemental compositions of cal-sil include Si and Ca and small amount of Al, Fe, Na, and Mg. There was no significant difference in elemental composition between raw and baked unused cal-sil. After being baked in a laboratory oven at 260°C for 72 hours, the raw cal-sil color changed from yellow to pink. The possible property changes of cal-sil after being baked include loss of water and oxidation of reductive species such as organic carbon, Fe(0), and Fe(II), as well as possible mineral and crystal structural changes. Specifically, oxidation of Fe(0) and Fe(II) into Fe_2O_3 is likely responsible for the baked cal-sil's turning pink.

ESEM/SEM/EDS examined a Day-30 unbaked cal-sil sample that had been submerged in the birdcage and a Day-30 baked cal-sil sample that had been submerged in the high-flow zone. EDS results show a significant amount of P on the exterior of the submerged cal-sil samples, both baked and unbaked; almost no P was present in the interior of the submerged cal-sil. (The interior cal-sil sample was obtained by breaking a chunk of cal-sil in half, and the interior sample was examined with SEM.) This result may be explained by the cal-sil exterior surface's being exposed to the testing solution, likely causing phosphate to complex with Ca at the exterior surface. However, because of limited phosphate diffusion into the cal-sil interior, no P was found in the interior cal-sil samples. In addition, unlike fiberglass, cal-sil is granular, making it difficult to distinguish cal-sil particles from the foreign deposits/debris attached on the cal-sil samples. Appendix H includes ESEM and SEM/EDS data for the cal-sil.

3.4. Metallic and Concrete Samples

3.4.1. Weights and Visual Descriptions

3.4.1.1. Submerged Coupons

Examination of the 40 submerged coupons provides valuable insight 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 insertion in 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 complete drying of the coupons before the post-test pictures. 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. Generally, the submerged coupons experienced more dramatic changes in both appearance and weight.