3.2.3. Optical/TEM Images from Filtered and Unfiltered Samples

TEM/EDS and diffraction pattern analyses were performed for Test #4, Day-4, Day-15, and Day-30 unfiltered solution samples. 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 H contains the TEM data.

3.3. Insulation

Test #4 was the second ICET test that included cal-sil insulation in addition to NUKONTM fiberglass samples. The fiberglass samples received thorough investigations, with samples removed from the tank on Day 5, 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 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.

Fiberglass samples that were examined with SEM after they had been exposed in the test solution for several days exhibited deposits throughout the fiber matrix. Those would be either 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.

To understand the formation of the film deposits, control experiments were performed by gently rinsing the interior fiberglass samples with several drops of RO water before ESEM analysis. The results show that after being rinsed with RO water, the film deposits disappeared from the fiberglass samples. This fact suggests that the film is actually soluble, which is consistent with the explanation that the film was formed by chemical precipitation during the drying process of fiberglass. In other words, although the ESEM analysis maintains samples in a moister state than conventional SEM, the partial drying that took place during ESEM analysis was sufficient for some chemicals to precipitate and form the film deposits that were observed. Sections 3.3.1.6 and 3.3.1.7 contain results from rinsed fiberglass samples.

There were four fiberglass locations in the tank that were examined in this test, including in a low-flow area, a 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 3.3.1.10 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-5 low flow, Day-15 low flow, Day-15 high flow, Day-30 low flow, Day-30 low flow in high envelope, Day-30 high flow, Day-30 high flow, Day-30 high flow in front of header, Day-30 drain collar, and Day-30 birdcage. The corresponding figures are Figures 3-16 through 3-85. Additional micrographs of fiberglass samples are presented in Appendices A, B, and C.

In general, particulate deposits were found only on the exterior of the fiberglass. This result suggests that almost all of the particulate deposits were physically retained or attached on the fiberglass exterior. The amount of particulate deposits increases from Day-15 low- and high-flow to the Day-30 high-flow samples. Further increases in particulate deposits were observed in the Day-30 big envelope low-flow sample, with even more on the birdcage sample, and the most on the drain collar sample. EDS shows that these particulate deposits contain significant amount of Si and Ca, suggesting they are from cal-sil debris. In contrast, the interior of fiberglass samples at different locations was relatively clean. Only film deposits were observed. There was no significant trend with respect to the location and the time. The film deposits were primarily composed of O, Na, Ca, C, Mg, Al, and possibly Si. These deposits are likely formed by chemical precipitation during the dehydration process of the samples. Control experiments were performed by gently rinsing the fiberglass interior samples with RO water, followed by ESEM analysis. The film deposits disappeared after this preparation technique. These results suggest that the film is soluble, which is consistent with the explanation that the film was formed by chemical precipitation.

Results also show that the mesh material, i.e., stainless steel or nylon did not significantly affect the deposits on fiberglass. In addition, the Day-30 high-flow fiberglass (in front of the header and placed in the tank on Day 2) sample exterior had much less particulate deposits attached/retained than the other high-flow sample exterior that was placed in the tank on Day 0. That was due to the settling of suspended particles in the test solution during the first two days.

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 is insignificant. Based on the ESEM results, some deposits were found after 5 days of the test on both the exterior and the interior of the low-flow fiberglass samples. In these deposits, a few particulates were found on the fiberglass exterior only (see Figure 3-17). However, most of the deposits were formed continuously like a film among glass fibers and coatings on the fibers. Therefore, these deposits are likely of chemical origin instead of being physically attached/retained. EDS results indicated that the film was composed of C, O, Na, Ca, Mg, Al, and possibly Si. Comparing the amount of the film on the

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 3-24 show the Day-4 low-flow fiberglass results.



Figure 3-16. ESEM image magnified 80 times for a Test #4, Day-5 low-flow exterior fiberglass sample. (T4D5LFX1.jpg)



Figure 3-17. ESEM image magnified 1000 times for a Test #4, Day-5 low-flow exterior fiberglass sample. (t4d5lx11.jpg)



Figure 3-18. ESEM image magnified 500 times for a Test #4, Day-5 low-flow exterior fiberglass sample. (t4d5lx14.jpg)



Figure 3-19. ESEM image magnified 1000 times for a Test #4, Day-5 low-flow exterior fiberglass sample. (t4d5lx12.jpg)



Figure 3-20. EDS counting spectrum for the film between the fibers shown in Figure 3-19. (t4d5lx13.jpg)



Figure 3-21. ESEM image magnified 80 times for a Test #4, Day-5 low-flow interior fiberglass sample. (t4d5lfi5.jpg)



Figure 3-22. ESEM image magnified 500 times for a Test #4, Day-5 low-flow interior fiberglass sample. (t4d5lfi7.jpg)



Figure 3-23. ESEM image magnified 2000 times for a Test #4, Day-5 low-flow interior fiberglass sample. (t4d5lfi8.jpg)



Figure 3-24. ESEM image magnified 1000 times for a Test #4, Day-5 low-flow interior fiberglass sample. (t4d5lfi9.jpg)

3.3.1.2. Day-15 Low-Flow Fiberglass Samples

Similar to the Day-5 samples, a few particulate deposits were found on the fiberglass exterior. EDS analysis shows that the particulate deposits were mainly composed of O, Na, Ca, Al, Mg, B, C, K, and possibly Si. However, the film deposits were prevalent on both the exterior and the interior fiberglass samples. There was no significant increase in the amount of deposits on Day-15 samples compared to Day-5 samples. Comparing the amount of the film deposits on the exterior and the interior Day-15 low-flow fiberglass samples, the difference was insignificant. Figures 3-25 through 3-30 show the Day-15 low-flow fiberglass results.



Figure 3-25. ESEM image magnified 100 times for a Test #4, Day-15 exterior low-flow fiberglass sample. (t4d15lx1.jpg)



Figure 3-26. Annotated ESEM image magnified 100 times for a Test #4, Day-15 exterior low-flow fiberglass sample. (t4d15lx3.jpg)



Figure 3-27. EDS counting spectrum for the particulate deposit on fiberglass shown in Figure 3-26. (t4d15lx2.jpg)



Figure 3-28. ESEM image magnified 500 times for a Test #4, Day-15 exterior low-flow fiberglass sample. (t4d15lx4.jpg)



Figure 3-29. ESEM image magnified 100 times for a Test #4, Day-15 interior low-flow fiberglass sample. (t4d15li6.jpg)



Figure 3-30. ESEM image magnified 500 times for a Test #4, Day-15 interior low-flow fiberglass sample. (t4d15li7.jpg)

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. The film deposits were prevalent on both the exterior and interior fiberglass samples. Consistently, EDS analysis indicates that the film was composed of O, Na, Ca, C, Mg, Al, K, and possibly Si. There was no significant difference regarding the amount of the film deposits between the exterior and the interior fiberglass samples, suggesting the likely chemical origin of the film. In addition, no particulate deposits were observed on either the exterior or interior fiberglass samples. Figures 3-31 through 3-36 show the Day-15 high-flow fiberglass results.



Figure 3-31. ESEM image magnified 100 times for a Test #4, Day-15 exterior high-flow fiberglass sample. (t4d15hx8.jpg)



Figure 3-32. ESEM image magnified 400 times for a Test #4, Day-15 exterior high-flow fiberglass sample. (t4d15hx9.jpg)



Figure 3-33. ESEM image magnified 750 times for a Test #4, Day-15 exterior high-flow fiberglass sample. (t4d15hx7.jpg)



Figure 3-34. EDS counting spectrum for the spot on the film between the fibers shown in Figure 3-33. (t4d15hx6.jpg)



Figure 3-35. ESEM image magnified 100 times for a Test #4, Day-15 interior high-flow fiberglass sample. (t4d15hi3.jpg)



Figure 3-36. ESEM image magnified 800 times for a Test #4, Day-15 interior high-flow fiberglass sample. (t4d15hi2.jpg)

3.3.1.4. Day-30 Low-Flow Fiberglass Samples

A similar amount and composition of the film deposits were found with Day-30 low-flow fiberglass samples compared to Day-5 and Day-15 low-flow fiberglass samples. No significant difference was found regarding the amount of the film deposits between the exterior and the interior Day-30 low-flow fiberglass samples. As with the Day-5 and 15 samples, the film was composed of O, Na, Ca, Mg, Al, C, K, and possibly Si. Besides the film, a coating was found on the glass fibers (see Figure 3-39). The coating was likely formed by chemical precipitation. EDS analysis shows the coating had the same composition as the film, suggesting both are likely of chemical origin. Figures 3-37 through 3-43 show the Day-30 low-flow fiberglass results.



Figure 3-37. ESEM image magnified 100 times for a Test #4, Day-30 exterior low-flow fiberglass sample. (t4d30lx1.jpg)



Figure 3-38. ESEM image magnified 500 times for a Test #4, Day-30 exterior low-flow fiberglass sample. (t4d30lx2.jpg)



Figure 3-39. ESEM image magnified 1000 times for a Test #4, Day-30 low-flow fiberglass sample. (t4d30lx5.jpg)



Figure 3-40. EDS counting spectrum for the spot of coating substance on the fiberglass shown in Figure 3-39. (t4d30lx4.jpg)



Figure 3-41. ESEM image magnified 100 times for a Test #4, Day-30 interior low-flow fiberglass sample. (T4D30LI6.jpg)



Figure 3-42. ESEM image magnified 500 times for a Test #4, Day-30 interior low-flow fiberglass sample. (t4d30li9.jpg)



Figure 3-43. EDS counting spectrum for the film on the fiberglass shown in Figure 3-42. (t4d30li8.jpg)

3.3.1.5. Day-30 Fiberglass Inserted in Nylon Mesh in Low-Flow Zone

A 5 g fiberglass sample was enclosed in a nylon mesh and submerged in a low-flow zone of the tank on Day 3, to provide a comparison to all other fiberglass samples, which were enclosed in stainless steel mesh and placed in the tank on Day 0. 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 these results to Day-30 low-flow fiberglass samples, no significant difference was observed. The film was still the dominant deposit on both of the exterior and the interior samples. There were no particulate deposits found on the fiberglass. This result suggests that the mesh material did not significantly affect the deposits on fiberglass. In addition, ESEM images show that the nylon fiber was relatively clean and without significant difference was found compared to the low-flow fiberglass samples put in the tank at the start of the test. Figures 3-44 through 3-49 show the Day-30 nylon-enclosed fiberglass results.



Figure 3-44. ESEM image magnified 100 times for a Test #4, Day-30 nylon mesh submerged in lowflow area (inserted on Day 4). (t4d30nl1.jpg)



Figure 3-45. ESEM image magnified 500 times for a Test #4, Day-30 nylon mesh submerged in low-flow area (inserted on Day 4). (t4d30nl2.jpg)



Figure 3-46. ESEM image magnified 100 times for a Test #4, Day-30 exterior low-flow fiberglass sample contained in a nylon mesh (inserted on Day 4). (t4NLEx01.jpg)



Figure 3-47. ESEM image magnified 500 times for a Test #4, Day-30 exterior low-flow fiberglass sample contained in a nylon mesh (inserted on Day 4). (t4nlex02.jpg)



Figure 3-48. ESEM image magnified 100 times for a Test #4, Day-30 interior low-flow fiberglass sample contained in a nylon mesh (inserted on Day 4). (t4nlIn04.jpg)



Figure 3-49. ESEM image magnified 500 times for a Test #4, Day-30 interior low-flow fiberglass sample contained in a nylon mesh (inserted on Day 4). (t4nlin05.jpg)

3.3.1.6. Day-30 Low-Flow Fiberglass Samples in the Big Envelope

Compared to other Day-30 low-flow fiberglass samples, a significant amount of particulate deposits was observed on the exterior of the Day-30 low-flow samples in the big envelope. The big envelope sat on the tank bottom and was in contact with the test sediment on the bottom of the envelope. Figure 3-120 shows the sediment after the samples were lifted out of the tank. However, no particulate deposits were observed on the fiberglass interior. In addition, some large flat fibers were found on the exterior fiberglass samples (see Figure 3-51). These large flat fibers were likely from cal-sil (see Appendix D). It is possible that the particulate deposits and cal-sil fibers were physically attached/retained on the exterior of the fiberglass samples. Figures 3-50 through 3-53 show the results from the Day-30 low-flow fiberglass enclosed in the big envelope. In contrast to other samples, these samples were gently rinsed with RO water and no film was found on either the exterior or the interior of the fiberglass. The disappearance of the film is likely caused by the rinse of RO water to keep the sample moist before ESEM analysis. Based on the control experiment noted earlier (see Appendix C4), the film is soluble.



Figure 3-50. ESEM image magnified 100 times for a Test #4, Day-30 exterior low-flow fiberglass sample in a big envelope. (t4evlx3.jpg)



Figure 3-51. ESEM image magnified 500 times for a Test #4, Day-30 exterior low-flow fiberglass sample in a big envelope. (t4evlx2.jpg)



Figure 3-52. ESEM image magnified 100 times for a Test #4, Day-30 interior low-flow fiberglass sample in a big envelope. (t4evli4.jpg)



Figure 3-53. ESEM image magnified 500 times for a Test #4, Day-30 interior low-flow fiberglass sample in a big envelope. (t4evli7.jpg)

3.3.1.7. Day-30 High-Flow Fiberglass Samples

Compared to Day-30 low-flow fiberglass samples, a significant amount of particulate deposits and fiberglass debris were found on high-flow exterior samples. However, no particulate deposits were found on the interior. This result suggests that these particulate deposits were physically attached/retained on the fiberglass exterior. However, similar film deposits were found on both of the fiberglass exterior and the interior. The EDS result shows the film was composed of O, Na, Ca, C, Mg, Al, K, and possibly Si, which is similar to other EDS results. Figures 3-54 through 3-61 show the Day-30 high-flow fiberglass results. Figures 3-60 and 3-61 show fiberglass after it was rinsed with RO water.



Figure 3-54. ESEM image magnified 100 times for a Test #4, Day-30 exterior high-flow fiberglass sample. (T4HFEx01.jpg)



Figure 3-55. ESEM image magnified 500 times for a Test #4, Day-30 exterior high-flow fiberglass sample. (t4hfex07.jpg)



Figure 3-56. ESEM image magnified 800 times for a Test #4, Day-30 exterior high-flow fiberglass sample. (t4hfex06.jpg)



Figure 3-57. EDS counting spectrum for the spot of film on the fiberglass shown in Figure 3-56. (t4hfex05.jpg)



Figure 3-58. ESEM image magnified 100 times for a Test #4, Day-30 interior high-flow fiberglass sample. (t4hfin01.jpg)



Figure 3-59. ESEM image magnified 500 times for a Test #4, Day-30 interior high-flow fiberglass sample. (t4hfin04.jpg)



Figure 3-60. ESEM image magnified 100 times for a Test #4, Day-30 interior high-flow fiberglass sample. The sample was gently prerinsed with RO water. (T4Rnd01.jpg)



Figure 3-61. ESEM image magnified 500 times for a Test #4, Day-30 interior high-flow fiberglass sample. The sample was gently prerinsed with RO water. (t4rnd05.jpg)

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

The images of the fiberglass in front of a header are different from the conventional highflow fiberglass samples in Section 3.3.1.7. The header sample was put in the tank on Day 2 to compare with samples placed in the tank on Day 0. Due to settling of suspended particles and decrease in turbidity during the first two days of the test, no significant particulate deposits were found on the header fiberglass exterior, as shown by ESEM images. This suggests that the deposits observed on the exterior of the high-flow samples put in the tank on Day 0 are due to the higher suspended solids and turbidity present in the system during the first several days. However, the coating and film deposits were found on both the exterior and the interior of the header samples. Consistent with earlier observations, the coating and the film were composed of O, Na, Ca, Mg, Al, C, K, and possibly Si. As mentioned before, coating and film deposits are likely caused by chemical precipitation when the samples were partially dehydrated. Figures 3-62 through 3-68 show the results from the Day-30 high-flow fiberglass in front of a header.



Figure 3-62. ESEM image magnified 100 times for a Test #4, Day-30 exterior high-flow fiberglass sample in front of a header (inserted on Day 4). (t4d30hx1.jpg)



Figure 3-63. ESEM image magnified 1000 times for a Test #4, Day-30 exterior high-flow fiberglass sample in front of a header (inserted on Day 4). (t4d30hx5.jpg)



Figure 3-64. EDS counting spectrum for the spot of coating substance on fiberglass shown in Figure 3-63. (t4d30hx4.jpg)



Figure 3-65. ESEM image magnified 100 times for a Test #4, Day-30 interior high-flow fiberglass sample in front of a header (inserted on Day 4). (t4d30hI6.jpg)



Figure 3-66. ESEM image magnified 500 times for a Test #4, Day-30 interior high-flow fiberglass sample in front of a header (inserted on Day 4). (t4d30hI9.jpg)



Figure 3-67. EDS counting spectrum for the spot of substance attached on fiberglass shown in Figure 3-66. (t4d30hI8.jpg)



Figure 3-68. ESEM image magnified 1000 times for a Test #4, Day-30 interior high-flow fiberglass sample in front of a header (inserted on Day 4). (t4d30hi1.jpg)

3.3.1.9. Day-30 Drain Collar Fiberglass Samples

When the tank was drained, the drain collar was totally surrounded by sediment. Figure 3-121 shows the drain collar after the tank was drained. 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. 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. EDS results indicate that the particulate deposits were mainly composed of O, Si, Ca, Na, Al, and C, regardless of whether the drain collar exterior was farthest from the drain screen or next to the drain screen. The high content of Si and Ca in the particulate deposits suggests that they were probably from cal-sil debris. In contrast to the exterior, no significant particulate deposits were found in the drain collar interior sample, and only film-like deposits were found (see Figure 3-75). The drain collar interior appears similar to the other high- and low- flow fiberglass interior samples. This result suggests that almost all of the particulate deposits were physically retained at the fiberglass exterior, which is consistent with Day-30 high-flow fiberglass samples. Figures 3-69 through 3-79 show the drain collar fiberglass results.



Figure 3-69. ESEM image magnified 100 times for a Test #4, Day-30 exterior fiberglass sample on the drain collar (away from the drain screen). (t4dcox05.jpg)



Figure 3-70. ESEM image magnified 500 times for a Test #4, Day-30 exterior fiberglass sample on the drain collar (away from the drain screen). (t4dcox02.jpg)



Figure 3-71. ESEM image magnified 1000 times for a Test #4, Day-30 exterior fiberglass sample on the drain collar (away from the drain screen). (t4dcox03.jpg)



Figure 3-72. EDS counting spectrum for the large mass of particulate deposits on fiberglass shown in Figure 3-71. (t4dcox04.jpg)



Figure 3-73. ESEM image magnified 100 times for a Test #4, Day-30 interior fiberglass sample on the drain collar. (t4dcin11.jpg)



Figure 3-74. ESEM image magnified 500 times for a Test #4, Day-30 interior fiberglass sample on the drain collar. (t4dcin12.jpg)



Figure 3-75. ESEM image magnified 500 times for a Test #4, Day-30 interior fiberglass sample on the drain collar. (t4dcin15.jpg)



Figure 3-76. ESEM image magnified 100 times for a Test #4, Day-30 exterior fiberglass sample on the drain collar (next to the drain screen). (t4dcix06.jpg)



Figure 3-77. ESEM image magnified 500 times for a Test #4, Day-30 exterior fiberglass sample on the drain collar (next to the drain screen). (t4dcix07.jpg)



Figure 3-78. ESEM image magnified 1000 times for a Test #4, Day-30 exterior fiberglass sample on the drain collar (next to the drain screen). (t4dcix08.jpg)



Figure 3-79. EDS counting spectrum for the large mass of particulate deposits on fiberglass shown in Figure 3-78. (t4dcix09.jpg)

3.3.1.10. Day-30 Fiberglass Sample within the Birdcage

For the Day-30 fiberglass sample within the birdcage, the SEM images indicate a significant amount of particulate deposits (see Figure 3-80) on the exterior of the fiberglass. The location of the birdcage was on top of a large cal-sil sample that sat on the bottom of the tank and was in direct contact with the sediment. The birdcage, however, was not in direct contact with the sediment. The amount of particulate deposits was greater than the amounts on the high- and low-flow fiberglass samples, but less than the drain collar exterior. The EDS result shows that the particulate deposits were composed of O, Si, Ca, Na, Al, and C, which is consistent with the drain collar exterior. Again, the high content of Si and Ca suggests these particulate deposits were from cal-sil debris. Compared to the exterior, the interior sample was relatively clean. Only film deposits were found. These film deposits were similar to the observed high- and low-flow fiberglass 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 Day-30 high-flow and drain collar fiberglass samples. Figures 3-80 through 3-85 show the birdcage fiberglass results.



Figure 3-80. ESEM image magnified 100 times for a Test #4, Day-30 exterior fiberglass sample within the birdcage. (T4D30BX1.jpg)



Figure 3-81. ESEM image magnified 500 times for a Test #4, Day-30 exterior fiberglass sample within the birdcage. (T4D30bx2.jpg)



Figure 3-82. Annotated ESEM image magnified 500 times for a Test #4, Day-30 exterior fiberglass sample within the birdcage. (t4d30bx4.jpg)



Figure 3-83. EDS counting spectrum for the particulate deposits on fiberglass shown in Figure 3-82. (T4D30bx3.jpg)



Figure 3-84. ESEM image magnified 100 times for a Test #4, Day-30 interior fiberglass sample within the birdcage. (t4d30bi5.jpg)



Figure 3-85. ESEM image magnified 500 times for a Test #4, Day-30 interior fiberglass sample within the birdcage. (t4d30bi7.jpg)