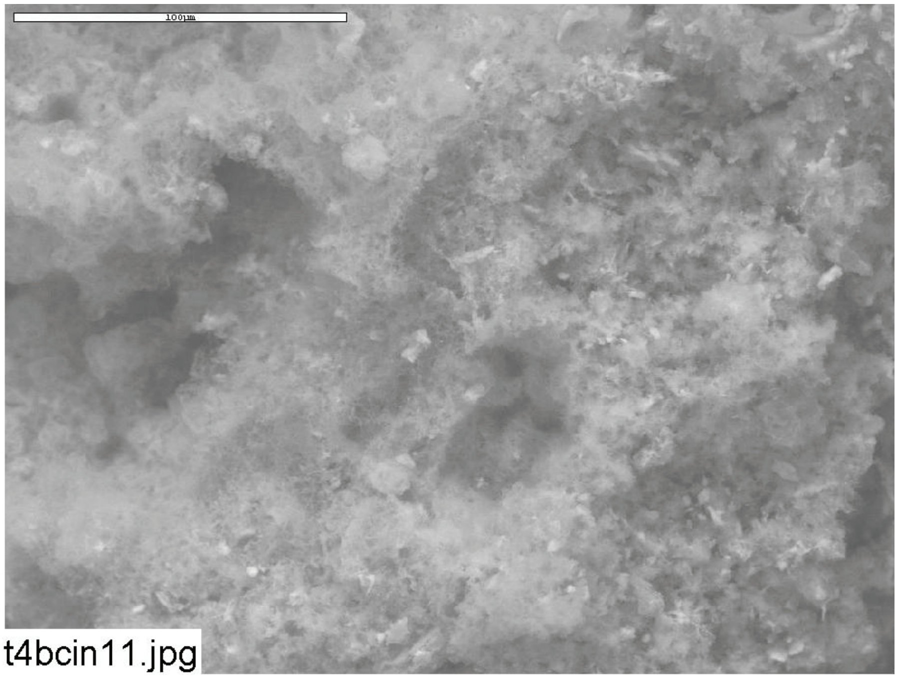

Figure 4-72. **ESEM image, magnified 500 times, of a Test #4, Day 30, low-flow interior baked cal-sil sample. (t4bcin11.jpg)**

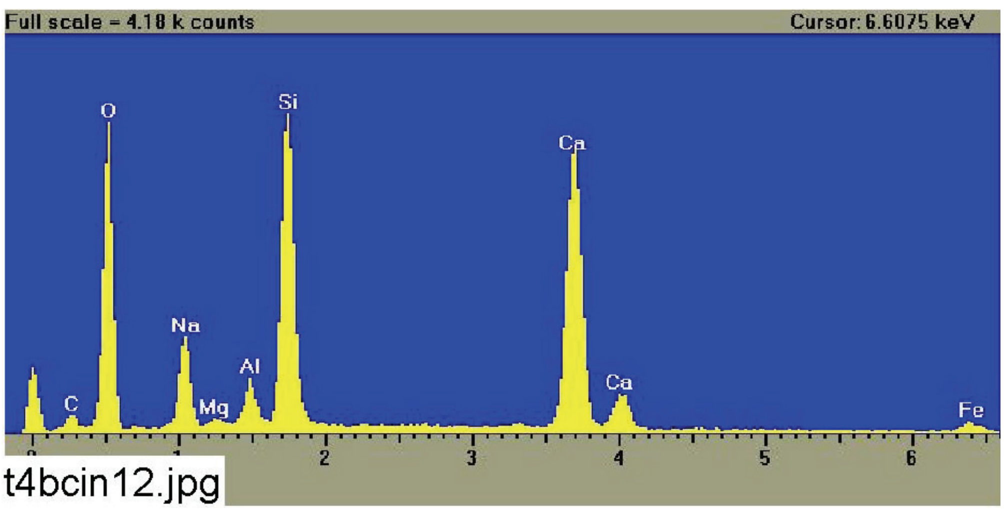

Figure 4-73. **EDS counting spectrum for the whole image shown in Figure 4-72. (t4bcin12.jpg)**

4.3. Sediment

According to photographic evidence and SEM/EDS analysis, the sediment samples from the five ICET tests were composed mainly of debris of insulation material (i.e., fiberglass and/or cal-sil), dirt, corrosion products, and chemical precipitates. However, because of the differences in solution conditions (i.e., pH and trisodium phosphate) and the insulation materials, the relative compositions and amounts of the sediments varied among the tests. As shown in Table 4-6, the largest amount of sediment was observed in Tests #3 and #4. Test #5 yielded the smallest amount of sediment.

As shown in Table 4-7, XRF analysis determined that silicon (Si) was the dominant element in the sediments of Tests #1, #2, and #5 because fiberglass was the sole insulation material used in these tests. Table 4-8 lists XRF results of unused insulation samples for reference. The XRF result for silicon (Si) is consistent with the SEM results showing large portions of fiberglass debris in the sediments. The fiber debris present at the bottom of the ICET tank represents fiberglass that escaped the stainless-steel mesh bags that were constructed to hold the primary volume of this debris type. The detection of quartz in XRD results of Tests #1, #2, and #5 sediment samples (Figures 4-78, 4-83, and 4-98) further verified the presence of some fiberglass debris in the sediments. However, in Tests #3 and #4, calcium content was higher than silicon in the sediment because 80% of the fiberglass was replaced by cal-sil as the insulation material in those two tests. As a result, cal-sil debris contributed largely to the sediments, as shown in the SEM/EDS results. The XRD results (Figures 4-89 and 4-94) indicate that Test #3 and Test #4 sediment contained crystalline of calcite ($CaCO_3$) and tobermorite in two forms, [$Ca_{2.25}(Si_3O_{7.5}(OH)_{1.5})(H_2O)$ and $Ca_4(Si_6O_{15}(OH)_2)(H_2O)_5$], which match the XRD results of unused, baked and unused, unbaked cal-sil samples. This result suggests that the sediment obtained from Tests #3 and #4 was mostly attributable to the addition of the cal-sil material.

In addition, because of the injection of trisodium phosphate in Tests #2 and #3, a significantly higher phosphorus content was found in the sediments of Tests #2 and #3 than in the other tests. The presence of aluminum, calcium, iron, magnesium, and manganese in the sediments are likely a result of corrosion products of metallic and concrete coupons and/or from the leaching of insulation materials.

Table 4-7. Elemental Composition of the Sediment Samples from Different Tests Based on XRF Analysis

Element	Test #1 (%)	Test #2 (%)	Test #3 (%)	Test #4 (%)	Test #5 (%)
Si	27.0	17.5	16.9	16.0	29.8
Ti	0.1	0.1	0.1	0.1	0.1
Al	3.3	8.5	2.6	2.5	3.2
Fe	1.4	0.5	1.6	1.5	0.9
Mn	0.2	0.1	0.0	0.0	0.1
Mg	1.3	0.9	0.4	0.4	1.1
Ca	3.9	3.6	19.4	20.4	3.9
Na	8.7	2.9	1.6	3.7	6.9
K	0.7	0.6	0.4	0.2	0.9
P	0.0	4.4	1.3	0.1	0.0
Wet Sediment Recovered (g)	292	256	78,000	86,000	89

Table 4-8. Elemental Composition of the Fiberglass Samples Based on XRF Analysis

Element	Clean Fiberglass	Unbaked Cal-sil	Baked Cal-Sil
Si	29.2	15.8	17.9
Ti	--	0.2	0.1
Al	1.9	2.3	2.7
Fe	--	1.5	1.8
Mn	--	0.1	0.1
Mg	2.1	0.8	0.5
Ca	5.9	24.8	24.8
Na	11.7	1.7	1.7
K	--	0.3	0.3
P	--	0.1	0.1

4.3.1. Test #1 Sediment

Figure 4-74 is a photograph of the sediment sample from the bottom of the tank after completion of Test #1. The photograph shows that a large portion of the sediment is fiberglass debris, which is consistent with SEM images in Figures 4-75, 4-76, and 4-77. Besides fiberglass debris, the particulate materials in the sediment mixture likely are from corrosion products, dirt, and chemical precipitates. A total of 292 g of wet sediment were recovered from the tank following the test.

In addition to the photograph and SEM images, XRD was used to determine the composition and the structure of some solid polycrystalline substances. As shown in Figure 4-78, the XRD spectrum indicates the presence of quartz in the sediment, which is consistent with the photograph and SEM images showing fiberglass debris in the sediment. Another possible crystal, sigloite, is not likely present in the sediment because the sigloite XRD peaks do not totally match the sample spectrum.

To further characterize other properties of the Test #1 sediment, the ratio between dry and wet sediment mass was determined and the qualitative resuspension and settling behavior of the sediment was performed. The ratio of dry to moist sediment mass was determined to be between 0.4 and 0.5. The sediment was shown to resuspend upon agitation and provide a turbidity measurement of 77 NTU after 40 minutes. Comparing this result to that of clean tap water (0.8 NTU), it can be concluded that a significant concentration of particles remained in solution after 40 minutes.

Figure 4-74. Sediment from south quadrant of tank after Test #1.

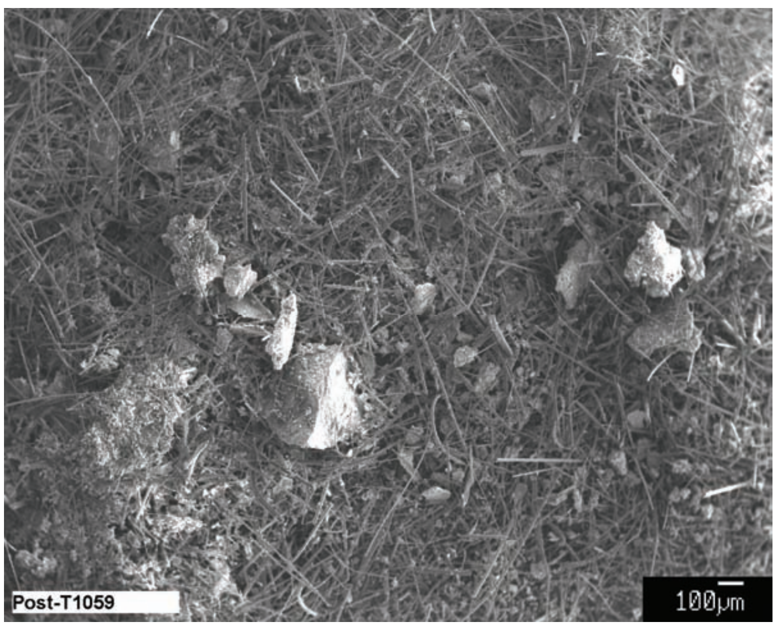

Figure 4-75. SEM overview image, magnified 40 times, of a Test #1, Day 30, sediment sample.

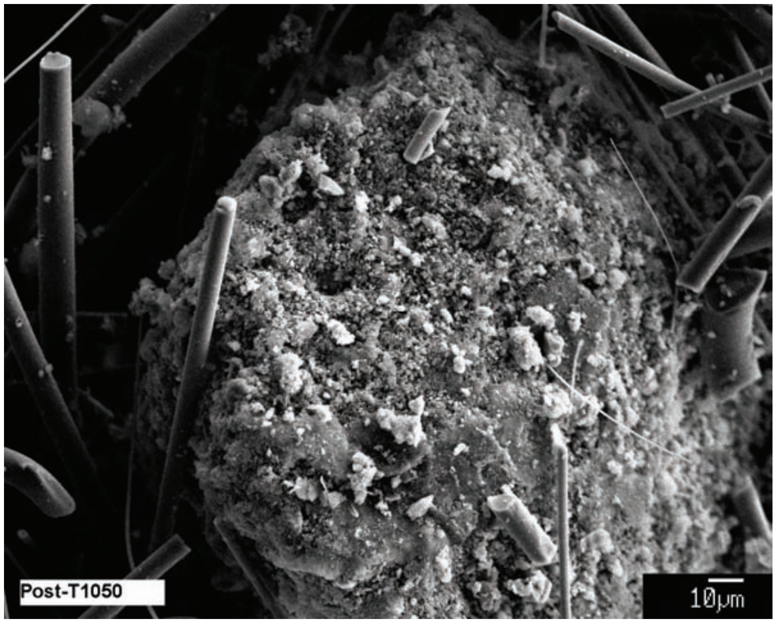

Figure 4-76. SEM image with a higher magnification on a Test #1, Day 30, sediment sample.

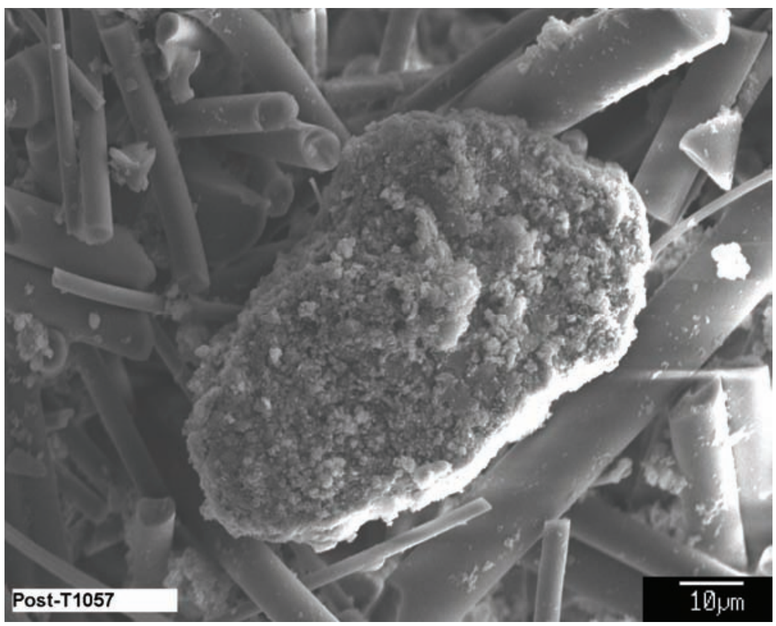

Figure 4-77. SEM image, magnified 1000 times, of a Test #1, Day 30, sediment sample.

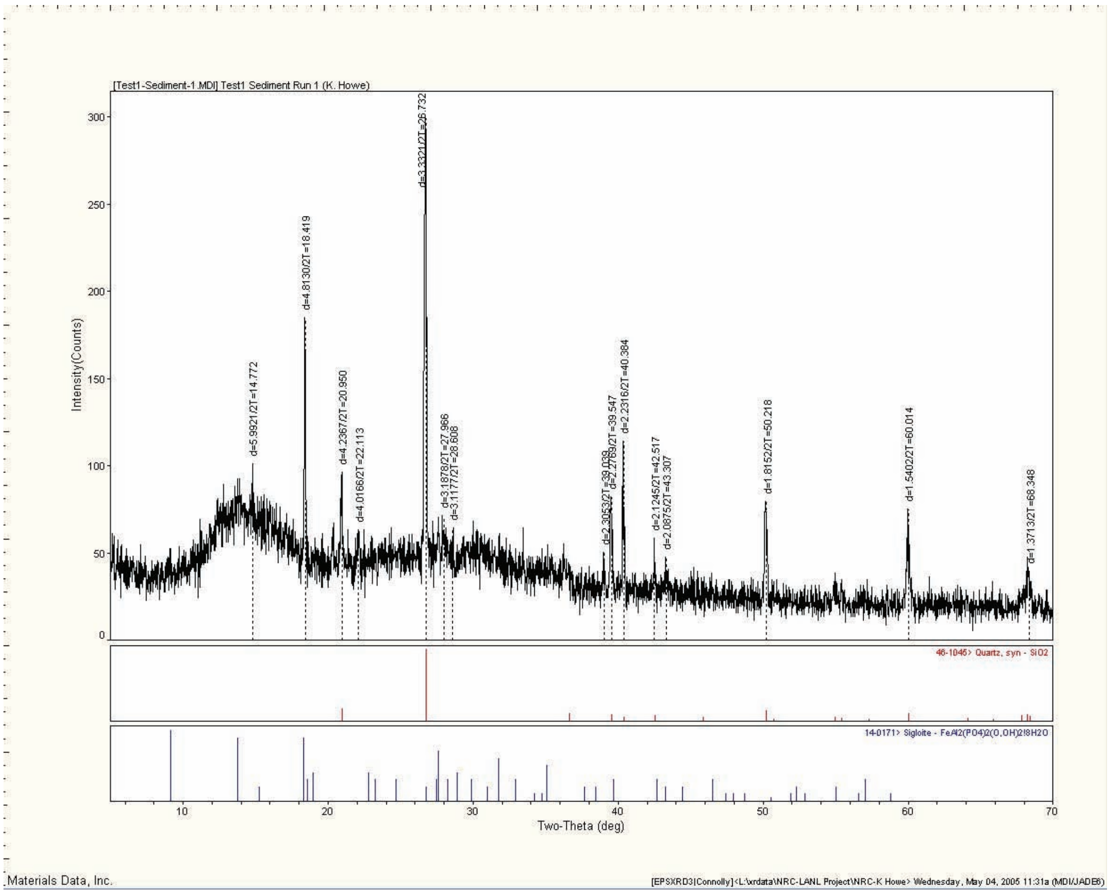

Figure 4-78. XRD result on a Test #1, Day 30, mixed-sediment sample.

4.3.2. Test #2 Sediment

In Test #2, the latent debris and crushed concrete material were observed to settle completely on the bottom of the tank over the course of several days. This particulate, in combination with fugitive fiberglass debris, forms the basic substrate of the sediment layer recovered from the tank at the end of Test #2. A total of 256 g of wet sediment was recovered from the tank following the test. Figure 4-79 shows a photo of a Test #2, Day 30, sediment sample, which is composed of particulate deposits and fiberglass debris. Figures 4-80, 4-81, and 4-82 show SEM images and EDS spectra of Test #2 sediment samples. Because of the heterogeneous nature of the sediment, this sediment is likely composed of corrosion products, concrete debris, dirt, and fiberglass. As in Test #1, the highest elemental content of the Test #2 sediment was silicon (Table 4-6), which likely came from the fiberglass debris. The ratio of dry to moist sediment mass was determined to be 0.5, which is consistent with the ratio found for the Test #1 sediment.

Figure 4-79. Picture of a Test #2, Day 30, sediment sample.

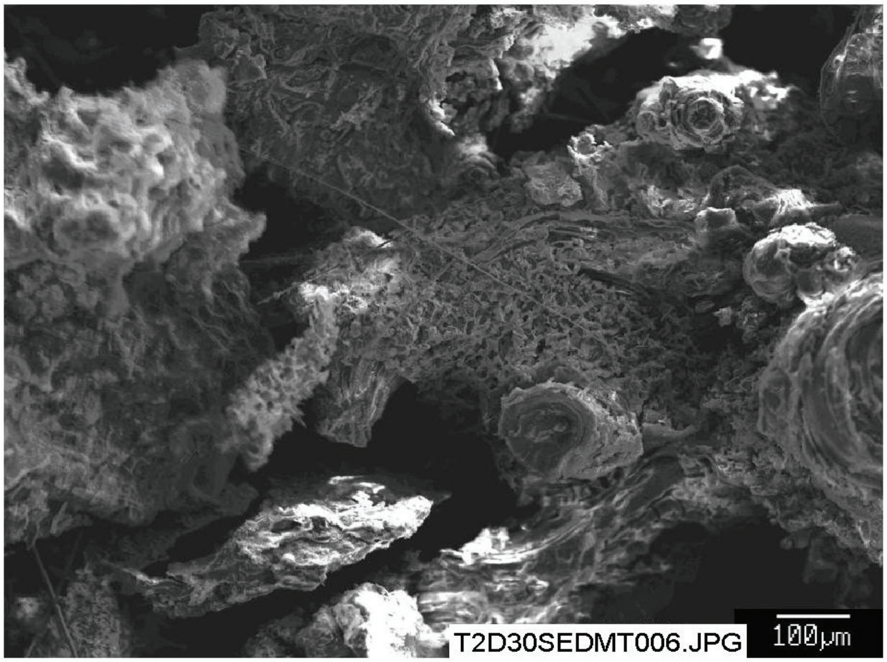

Figure 4-80. SEM image of a Test #2, Day 30, sediment sample.

Figures 4-81 and 4-82 provide a higher magnification and an EDS spectrum of the same Test #2 sediment shown in Figure 4-80. Underlying fibers are visible in this image, and the dominant elemental constituents of the particulate deposits are oxygen, magnesium, phosphorus, calcium, aluminum, silicon, carbon, and zinc. The XRD result of Test #2 sediment is shown in Figure 4-83. Again, the presence of quartz is consistent with the fiberglass debris in the sediment.

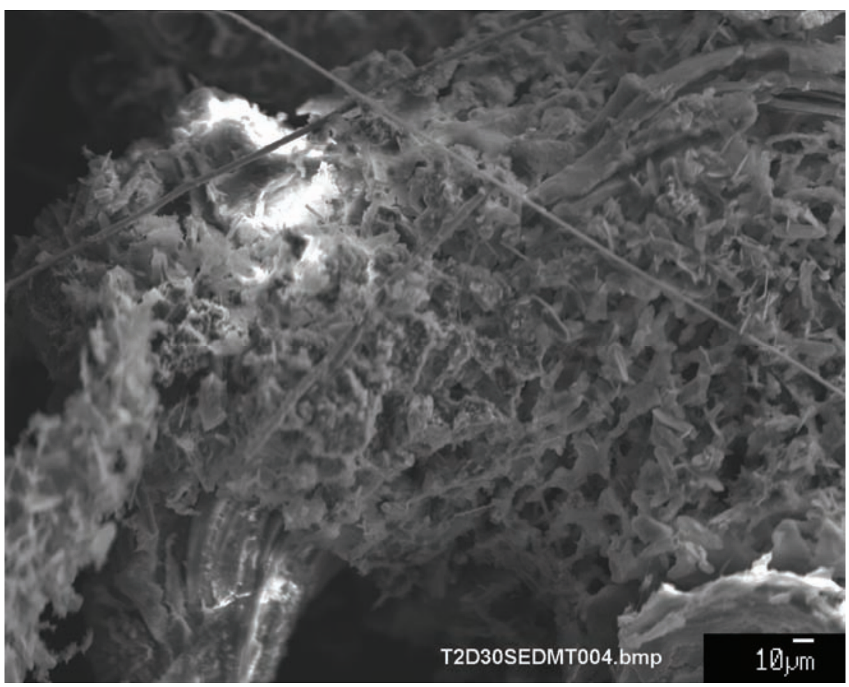

Figure 4-81. Higher-magnification SEM image of a Test #2, Day 30, sediment sample.

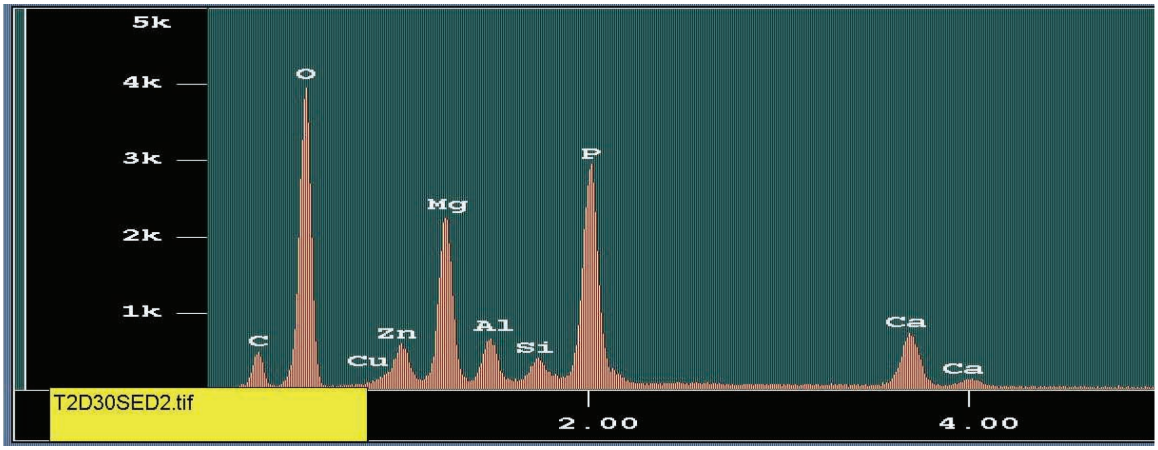

Figure 4-82. EDS counting spectrum for the porously structured material shown in Figure 4-81.

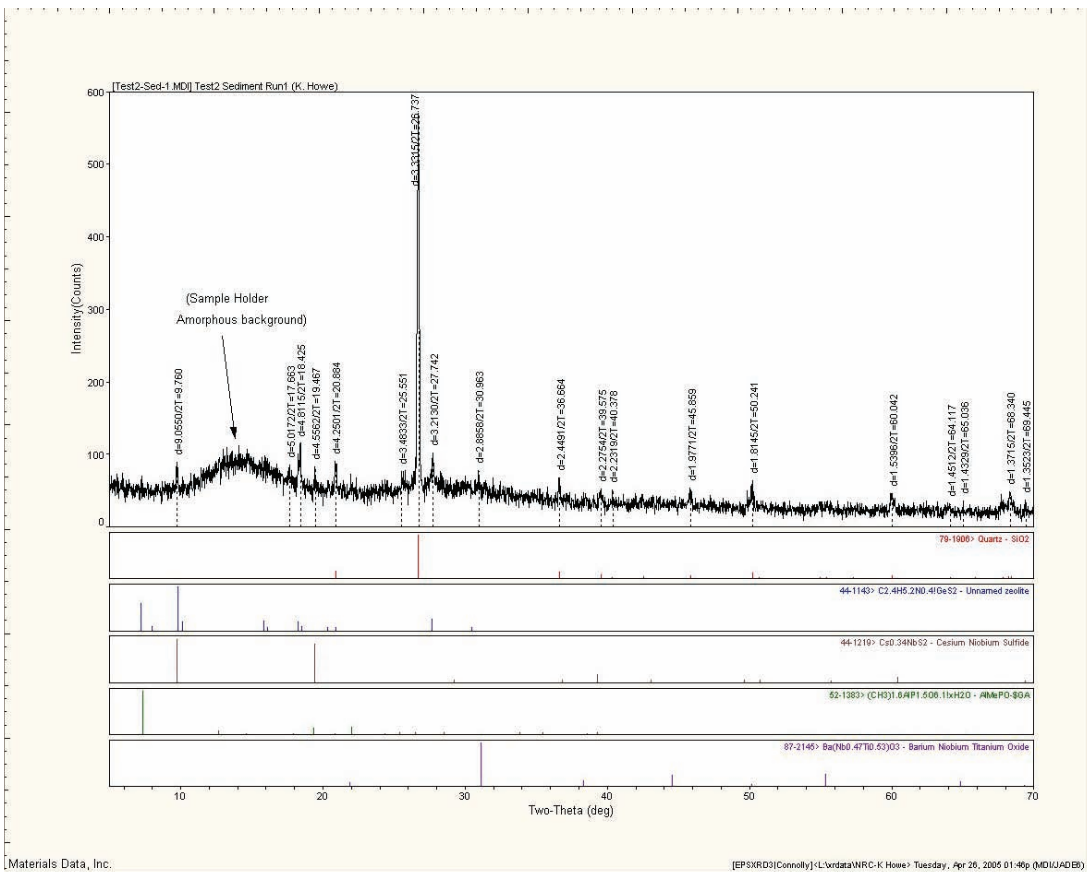

Figure 4-83. XRD results for Test #2, Day 30, mixed sediment.

Test #3 Sediment

Eighty percent of the fiberglass was replaced by cal-sil in Test #3. As a result, the cal-sil debris contributed significantly to the sediment, as shown in Figure 4-84, which is a photograph of the Test #3 sediment extracted from the bottom of the tank. The pink and yellow particles seen in the sediment correspond to baked and unbaked cal-sil. Over 78 kg of wet sediment were recovered from the tank following the test (approximately 20 kg of dry cal-sil dust was initially added to the tank).

ESEM/EDS results for pink and yellow sediment are shown in Figures 4-85, 4-86, 4-87, and 4-88. No significant difference was found regarding the composition of the pink and yellow sediments; both of them were composed mainly of oxygen, silicon, calcium, aluminum, sodium, magnesium, iron, boron, and carbon. Comparing the EDS results of the yellow and pink sediment with the unbaked and baked cal-sil samples, respectively, both yellow and pink sediments are of higher content of oxygen. Since the yellow and pink sediments from Test #3 were analyzed with ESEM (the sediment samples were wet), and the unbaked and baked cal-sil samples were dry and analyzed with the probe SEM, the higher oxygen content in the sediment samples was likely from water.

Figure 4-84. Sediment removed from the tank after Test #3.

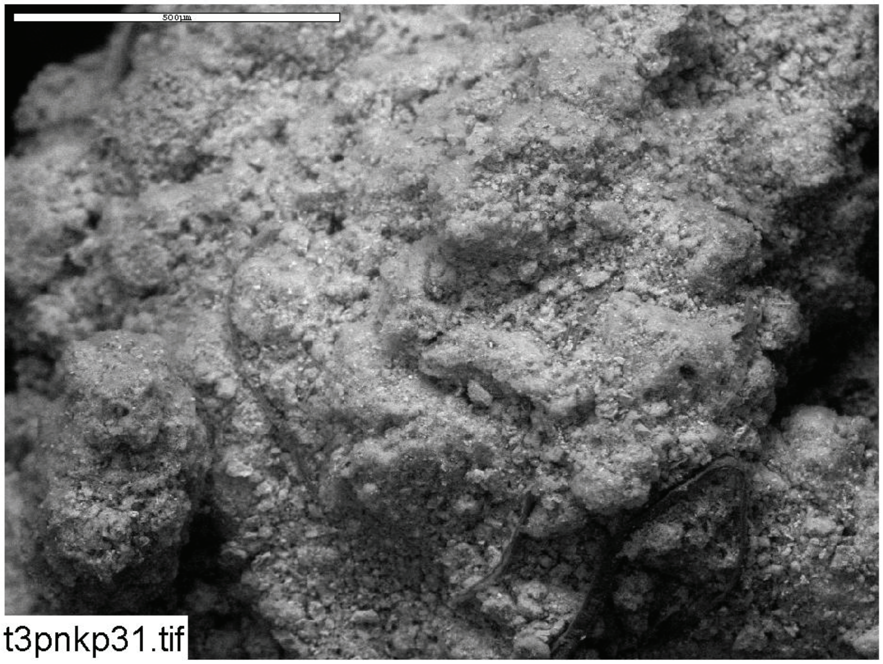

Figure 4-85. ESEM image of a Test #3, Day 30, pink sediment, magnified 100 times. (t3pnkp31, 5/6/05)

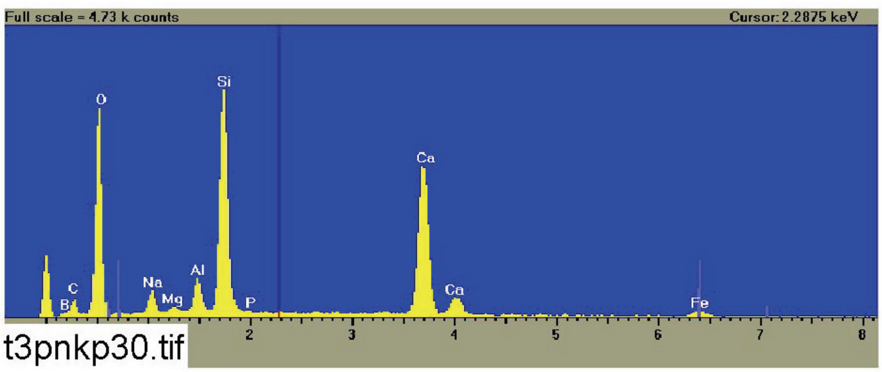

Figure 4-86. EDS counting spectrum for the pink sediment shown in Figure 4-85. (t3pnkp30, 5/6/05)

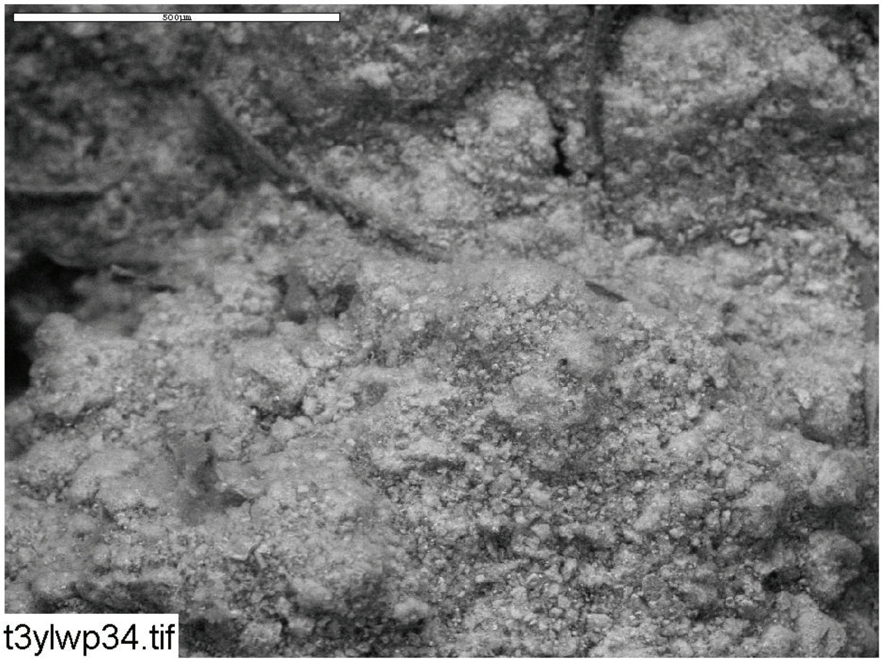

Figure 4-87. ESEM image of a Test #3, Day 30, yellow sediment, magnified 100 times. (t3ylwp34, 5/6/05)

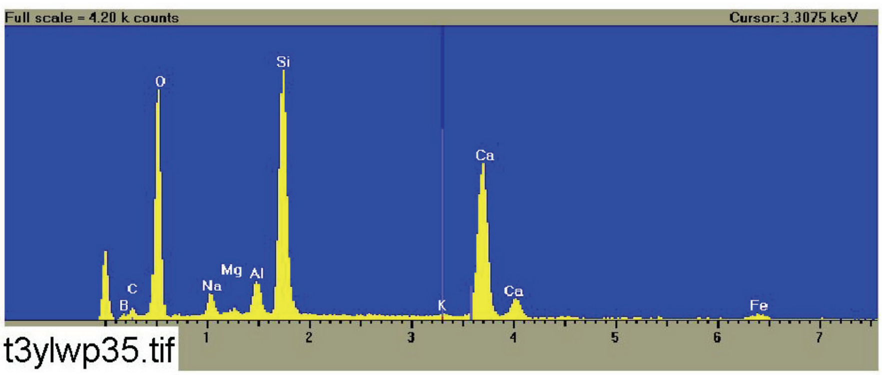

Figure 4-88. EDS counting spectrum for the particles shown in Figure 4-87. (t3ylwp35, 5/6/05)

The XRD results in Figure 4-89 indicate the sediment contained crystalline calcite ($CaCO_3$), tobermorite [$Ca_{2.25}(Si_3O_{7.5}(OH)_{1.5})(H_2O)$, and $Ca_4(Si_6O_{15}(OH)_2)(H_2O)_5$], which is consistent with the XRD results of unused baked and unused unbaked cal-sil samples (see Section 4.2.2). This result suggests that cal-sil debris contributed greatly to the sediment of Test #3, including both unbaked and baked cal-sil. It should be noted that other deposits, such as fiberglass debris and corrosion products, may also be present in the sediment.

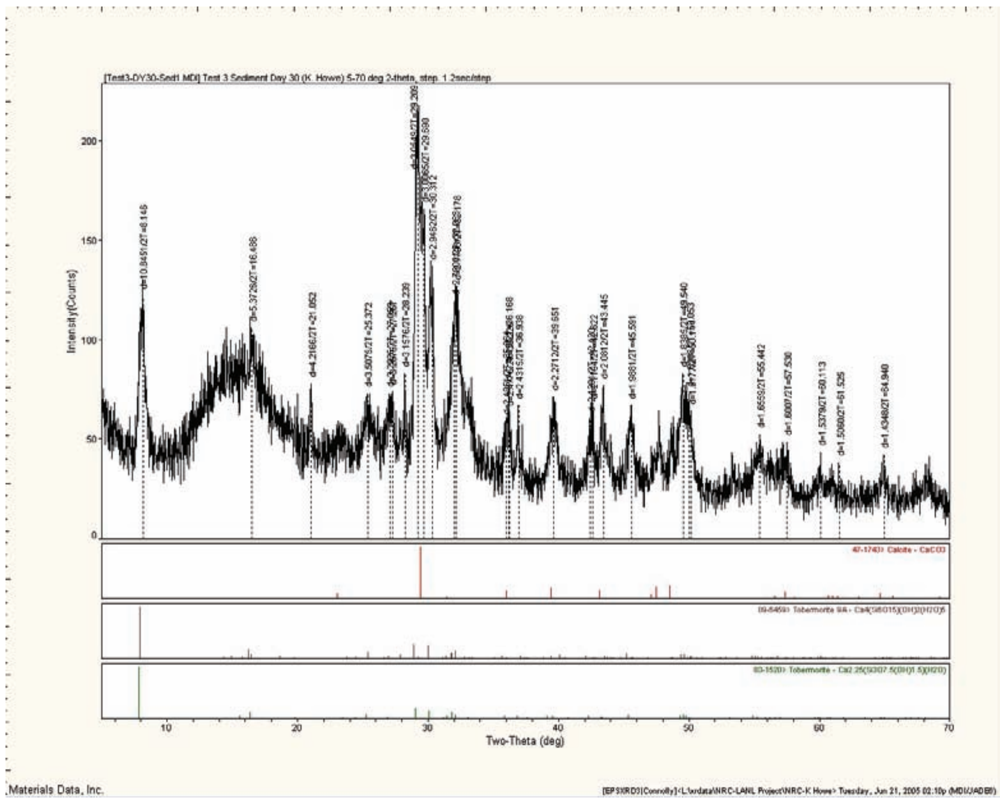

Figure 4-89. **XRD results for the Test #3, Day 30, mixed sediment.**

Test #4 Sediment

As in Test #3, 80% of the fiberglass was replaced by cal-sil in Test #4. Over 86 kg of wet sediment were recovered from the tank following the test (approximately 20 kg of dry cal-sil dust was initially added to the tank). Figure 4-90 shows a photograph of the sediment, which appears to be composed mainly of cal-sil debris. The SEM/EDS and XRD analysis provided information on the morphology and composition of Test #4 sediments, as shown in Figures 4-91, 4-92, 4-93, and 4-94. EDS results show that more than 84% of the sediment was composed of silicon, calcium, and oxygen. Similarly, the XRF results in Table 4-6 show that Si and Ca are the major elements in the composition of the sediment (oxygen is not detectable by XRF), plus small amounts of Na, Al, Fe, and Mg.

Based on the XRD results in Figure 4-94, the sediment sample contained crystalline substances of tobermorite [$Ca_{2.25}(Si_3O_{7.5}(OH)_{1.5})(H_2O)$ and $Ca_4(Si_6O_{15}(OH)_2)(H_2O)_5$], as well as calcite ($CaCO_3$), which were also in the unused unbaked or unused baked cal-sil samples. Considering the collective evidence from the EDS, XRF, and XRD analyses, it is likely that the sediment was composed of a significant amount of cal-sil debris, including both baked and unbaked cal-sil. It should be noted that other deposits, such as fiberglass debris and corrosion products, may also be present in the sediment.

Figure 4-90. Sediment removed from the tank after Test #4.

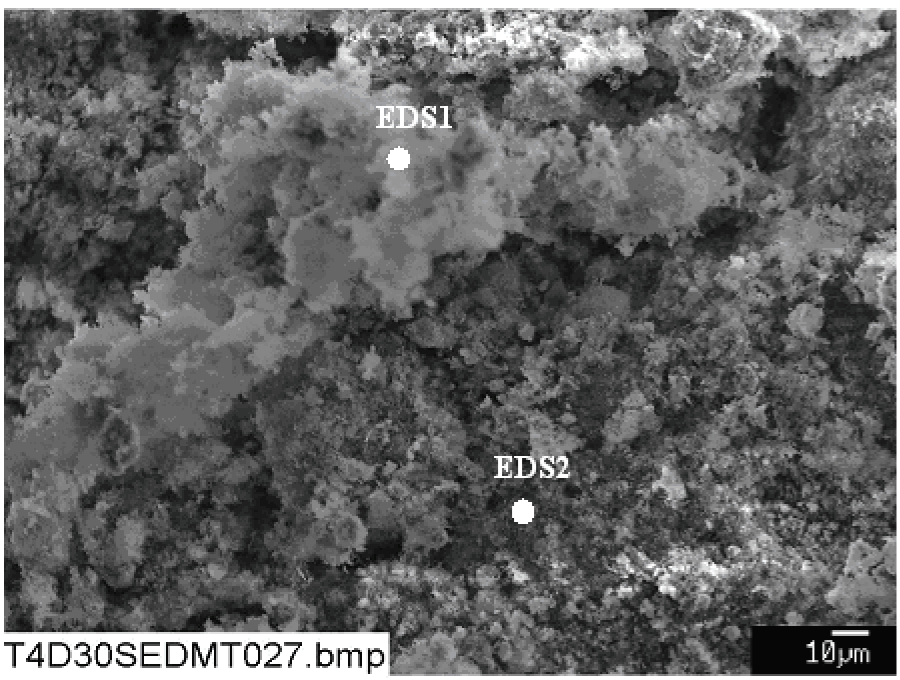

Figure 4-91. Annotated SEM image, magnified 500 times, for a Test #4, Day 30, sediment sample at the bottom of the tank. (T4D30SEDMT027.bmp)

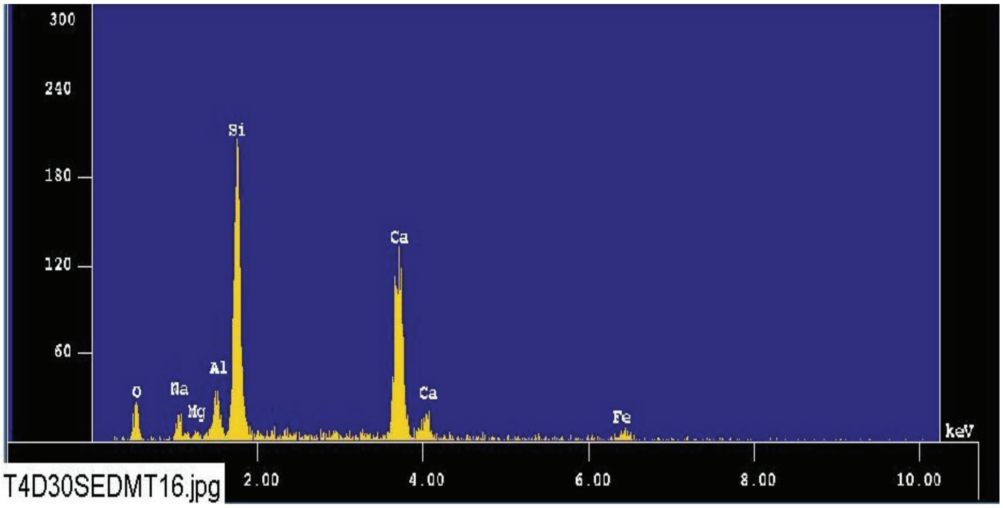

Figure 4-92. **EDS counting spectrum for the white snow-like deposits (EDS1) shown in Figure 4-91. (T4D30SEDMT16.jpg)**

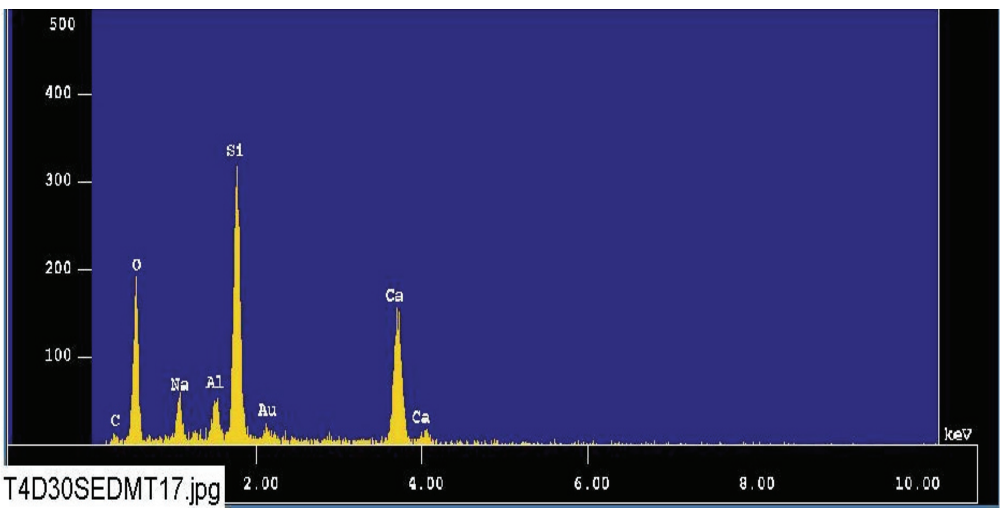

Figure 4-93. **EDS counting spectrum for the dark deposits (EDS2) shown in Figure 4-91. (T4D30SEDMT17.jpg)**

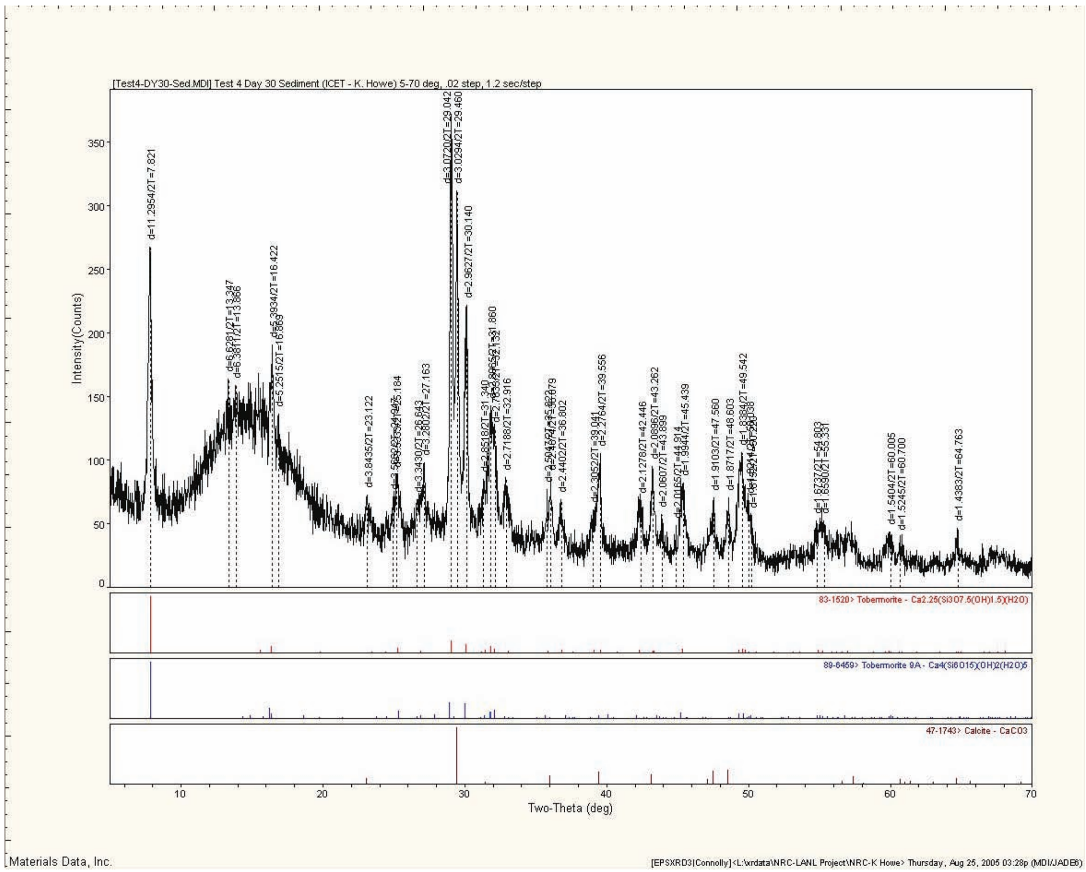

Figure 4-94. XRD results for the possible matching crystalline substances in the Test #4, Day 30, sediment.

Test #5 Sediment

Similar to Test #1 and Test #2, fiberglass was the only insulation material used in Test #5. The least amount (only 89 g) of wet sediment was recovered from the tank following the test. Figure 4-95 is a photograph of the sediment from Test #5, which shows a significant amount of fiberglass debris. The SEM image in Figure 4-96 also indicates a large amount of fiberglass debris, mixed with a few particulate deposits in the sediment. The particulate deposit is composed of silicon, aluminum, oxygen, carbon, sodium, and magnesium, as shown in Figure 4-97. The particulate deposits may originate from the corrosion products, dirt, and chemical precipitates. Consistent with the photograph and SEM images, the XRF results show that silicon was 29.8% of the total mass of the dried sediments, as shown in Table 4-6.

In addition, the XRD result in Figure 4-98 indicates the presence of quartz crystal in the sediment. The quartz likely derived from the fiberglass debris, which is consistent with the Tests #1 and #2 sediment. It should be noted that the XRD result also shows the possible crystalline match of cobalt and uranium compounds in the sediment. However, cobalt and uranium were not likely to be present. The XRD signature corresponding to these compounds likely results from the heterogeneous nature of the sediment and are not part of the sediment composition.

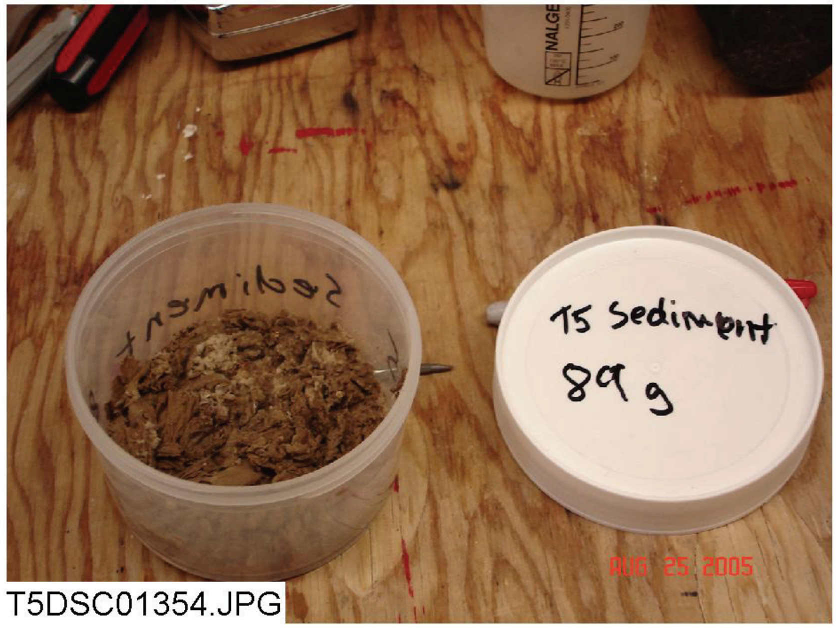

Figure 4-95. Sediment sample removed from the tank after Test #5.

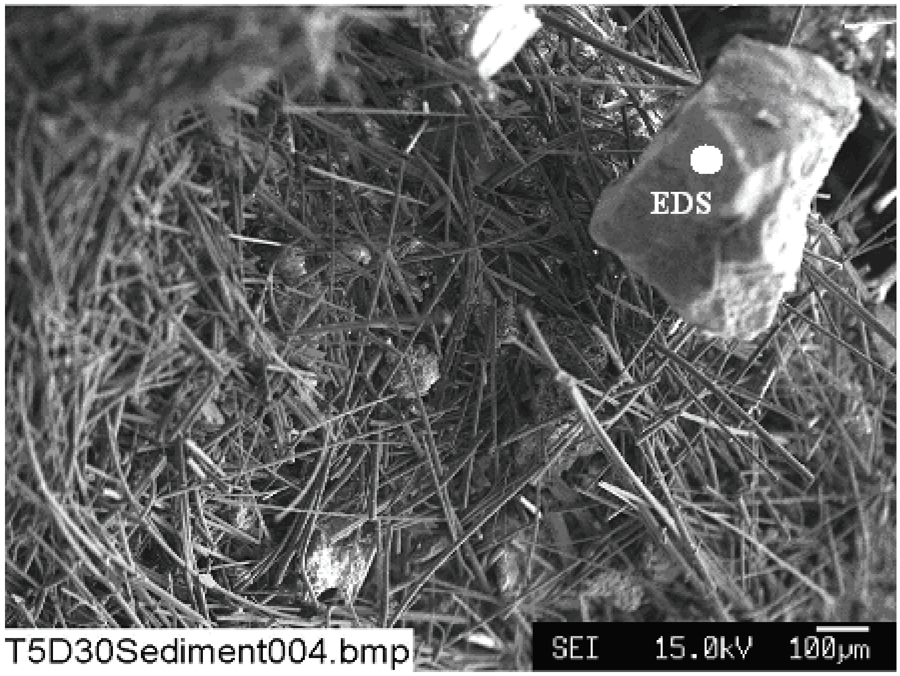

Figure 4-96. SEM image, magnified 70 times, of the Test #5, Day 30, sediment at the bottom of the tank.

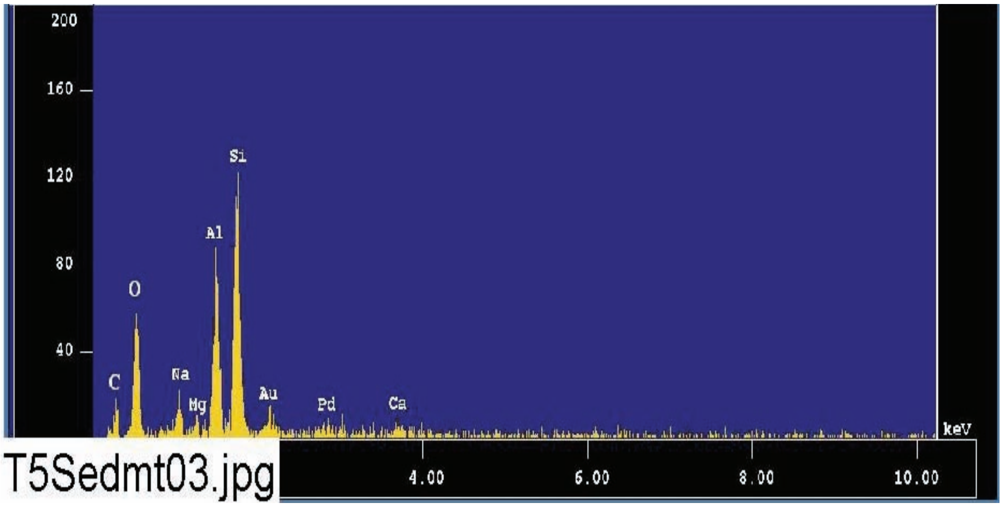

Figure 4-97. EDS counting spectrum for the big particulate deposit shown in Figure 4-96.

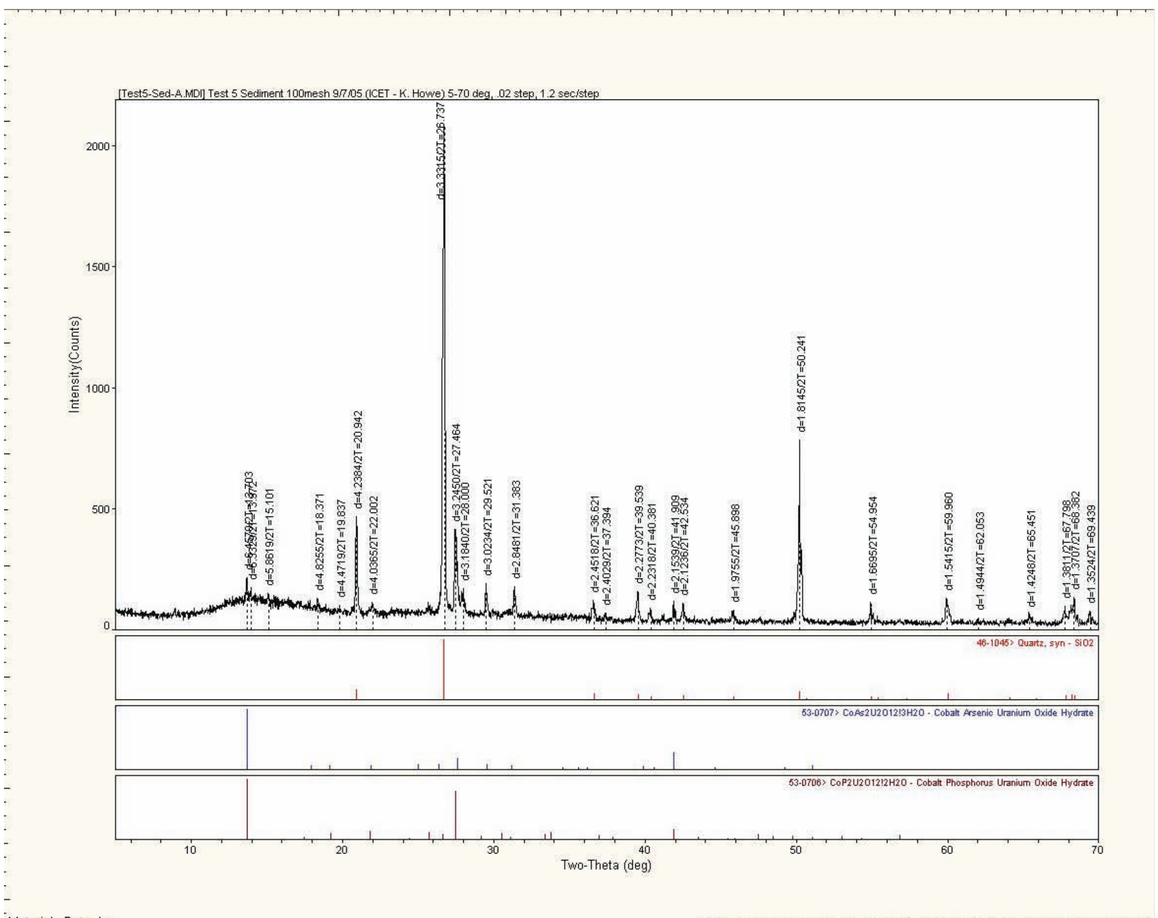

Figure 4-98. XRD results for the possible matching crystalline substances in Test #5, Day 30, sediment.