

### 3. TEST RESULTS

This section describes the results obtained from Test #4. An overview is first presented in the form of general observations. 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) insulation [NUKON™ fiberglass samples and cal-sil materials] (3) metallic and concrete samples, (4) sediment, and (5) deposition products.

#### 3.1. General Observations

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

Four hours after the test began, the solution was very murky and the cal-sil had not completely settled. The color of the tank solution was a yellowish-brown.

On Day 1, there was no observed sedimentation on the coupon racks or insulation holders. Also, the solution was clear with no observed presence of suspended particles. Most of the cal-sil had settled to the bottom of the tank.

On Day 3, a very thin round 2-in. diameter white deposit was observed on the submerged insulation holder on the north side of the tank. Five gallons of RO make-up water were added to the system into the tank outlet pipe, upstream of the recirculation pump. The turbidity before water addition was approximately 1.5 nephelometric turbidity units (NTU), and after the water addition, it was 9.8 NTU.

On Day 4, a collection of white particles was observed on the submerged insulation holder and the mesh birdcage on the north side of the tank.

On Day 8, 5 gallons of RO make-up water were added to the tank through the top using the recycle funnel. The flow remained steady at 25 gpm throughout this process. The water clarity declined slightly after RO water addition and turbidity was 3.0 NTU one hour after the water was added. This is greater than the 0.8 NTU value that was measured just before RO water addition.

On Day 11, a power outage took place. The recirculation pump was stopped for 2 hours and 15 minutes. Also, the maximum solution temperature rose to 62.4°C, which is 0.4°C above the target maximum. The maximum temperature was above 62.0°C for approximately 1 hour.

On Day 13, fiber-like material was observed to be deposited on some of the submerged coupons. Also on that day, 6 gallons of RO make-up water were added to the tank through the recycle funnel at the top of the tank. The flow remained steady throughout this process. The turbidity before water addition was 0.6 NTU; 1 hour after RO water addition, the turbidity was 1.6 NTU.

On Day 14, a high-flow sacrificial fiberglass sample was removed from the tank. This caused a disturbance in the tank and the solution became visibly more turbid. The fiberglass fibers that were first observed on Day 13 were no longer present.

On Day 16, small fibers similar to those observed previously were present on some submerged coupons.

On Day 17, 5 gal. of RO make-up water were added to the tank through the top using the recycle funnel. The flow remained steady at 25 gpm throughout this process. The water clarity declined slightly after RO water addition and turbidity was measured to be 0.9 NTU 30 minutes after the water was added.

On Day 22, 5 gal. of RO make-up water were added to the tank through the top of the recycle funnel. The flow remained unchanged during the process. The water clarity declined slightly after RO water addition and turbidity was measured to be 0.7 NTU thirty minutes after the water was added.

On Day 24, one of the three redundant thermocouples in the tank failed. The test solution temperature was subsequently recorded with the two remaining thermocouples.

On Day 27, 5 gal. of RO make-up water were added to the tank through the top using the recycle funnel. The flow remained steady at 25 gpm throughout this process. The water clarity declined slightly after RO water addition and turbidity was measured to be 0.6 NTU 30 minutes after the water was added.

Excluding Day 0, tank clarity and color remained constant throughout the test even with the addition of make-up RO water. The color was similar to weak, iced tea. No corrosion products were observed on the submerged coupons. A total of 31 gallons of make-up water were added to the system, and the system volume at the end of the test was 247 gal.

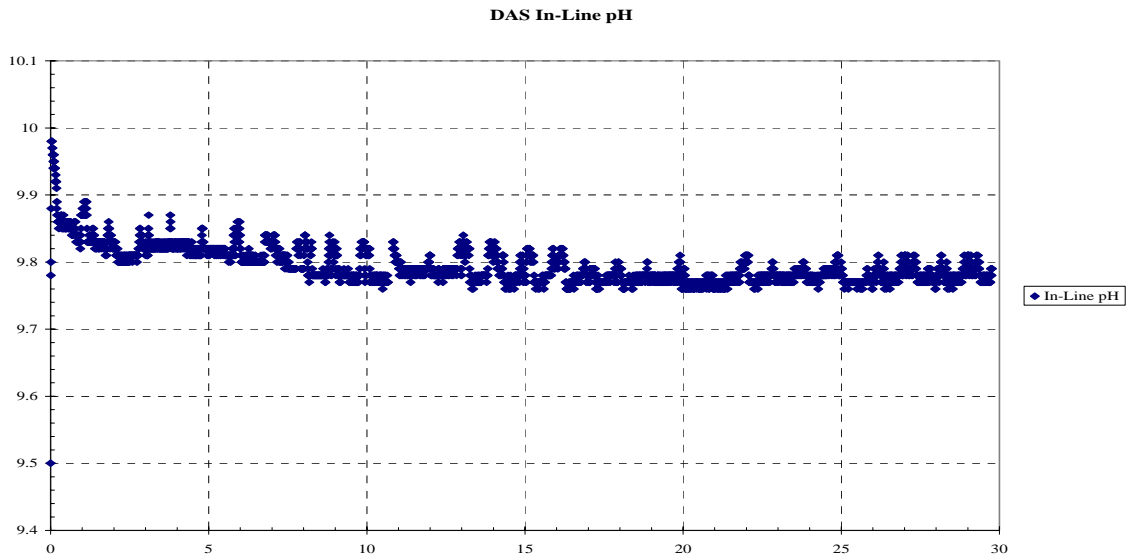
### **3.1.1. Control of Test Parameters**

**Recirculation Flow Rate:** Neglecting the power outage on Day 11, the average recirculation flow rate was 94.4 L/min (24.9 gpm). The recorded recirculation flow rate had a standard deviation of 0.04 L/min with a range of 93.9 to 95.8 L/min (24.8 to 25.3 gpm) excluding the spray cycle.

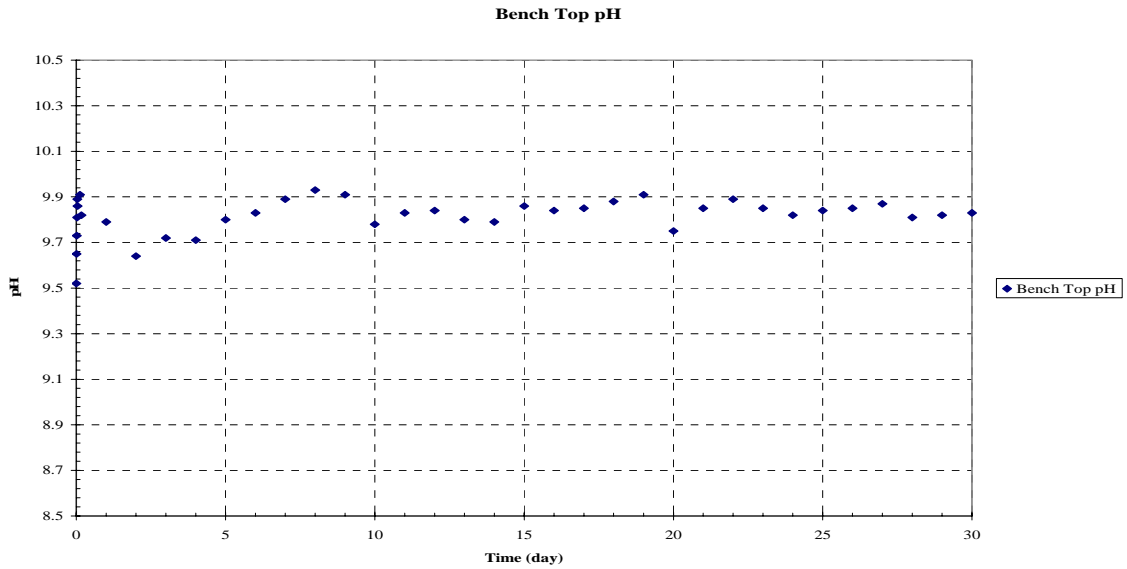
**Temperature:** Temperature is recorded at three submerged locations in the ICET tank. On Day 24, one of the three thermocouples failed. To reset the high temperature alarm, a thermocouple that measures room temperature replaced the bad thermocouple within the data acquisition system (DAS). Neglecting the deactivated thermocouple, the average recorded temperatures at the two locations were 60.8°C and 60.7°C (141.5°F and 141.2°F). The standard deviation in temperature recorded was within  $\pm 0.26^\circ\text{C}$  ( $\pm 0.46^\circ\text{F}$ ), with a maximum range of 58.1°C to 62.4°C (136.3 F to 144.4 F). The minimum temperature occurred during the addition of make-up water. The maximum temperature occurred during the power outage.

**pH:** Before time zero, 15.14 kg of boric acid, 8.47 kg of sodium hydroxide, 212 mL of 12.24 N hydrochloric acid solution, and 0.663 g of lithium hydroxide were dissolved into the ICET tank solution. The measured bench-top pH was 9.5. During the addition of the sodium hydroxide solution during the first 30 minutes, the pH rose to 9.8 and remained at that value through the spray cycle. The pH declined for the first 2 days to a value of 9.6, which was the predicted value for a solution in complete equilibrium with the atmosphere. After the second day the pH began to rise to a high value of 9.9 on Day 8. The pH varied between 9.7 and 9.9 from Day 9 to the end of the test. This can be seen in Figures 3-1 and 3-2.

The in-line pH probe produced the data in Figure 3-1. At four hours, the in-line pH measurement was 9.9 and at Day 30, it was 9.8. The bench-top pH measurements, which are calibrated daily, are presented in Figure 3-2. At 4 hours, the bench-top pH measurement was 9.8 and at Day 30, it was also 9.8.



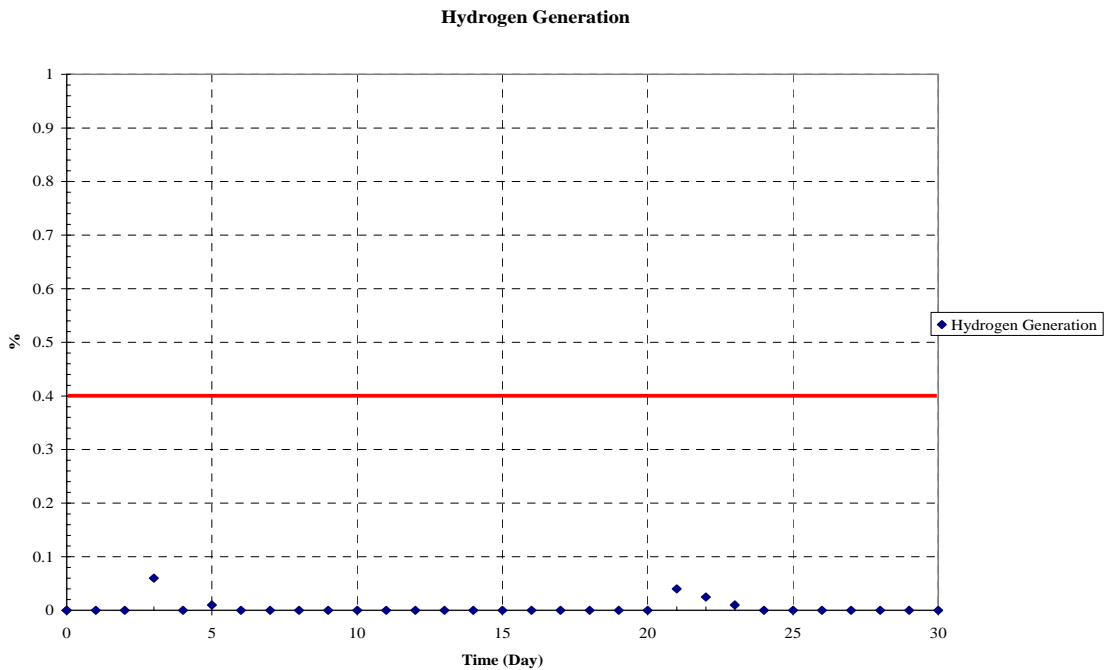
**Figure 3-1. In-line pH measurements.**



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

### 3.1.2. Hydrogen Generation

Hydrogen remained at or below 0.05% for the duration of the test as shown in Figure 3-3. All of the measured values are 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 after the latent debris and concrete dust were added, for the 23°C and 60°C water samples were 1.3 NTU and 0.9 NTU. After the addition of cal-sil, the tank solution became very cloudy. Upon the addition of the chemicals through the spray line, particulates were suspended in solution and the solution became even more cloudier.

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 which were 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, the turbidity gradually decreased from 129 NTU at the time-zero point to 36 NTU at the 4-hour point.

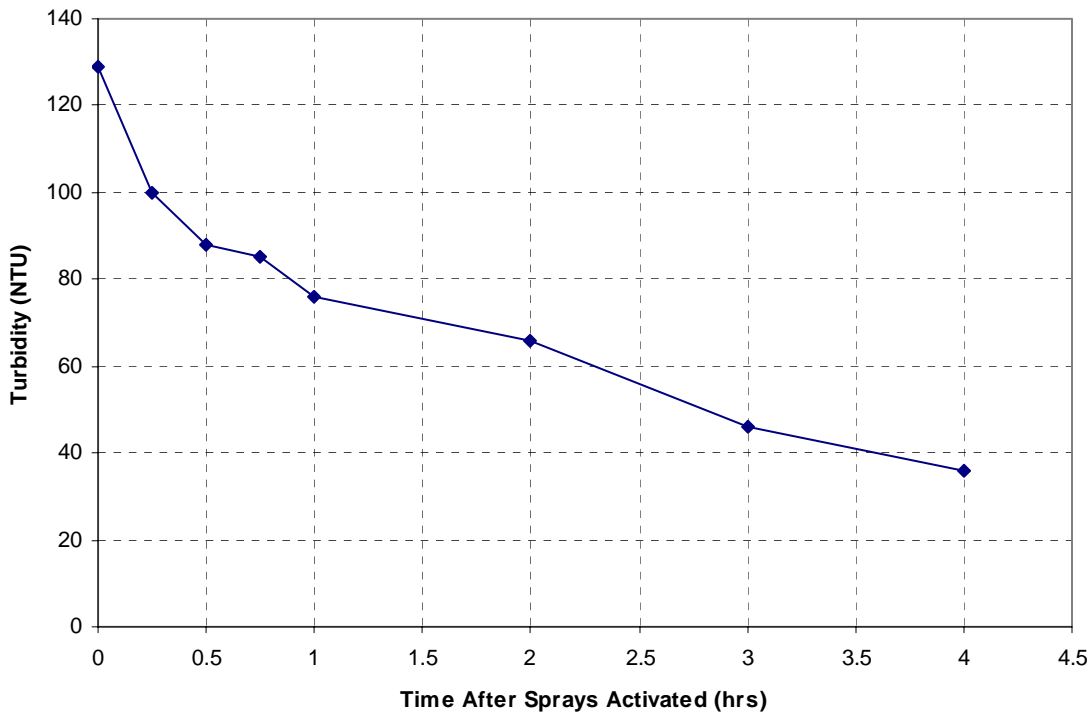


Figure 3-4. Day-1 turbidity results.

Figure 3-5 contains the daily turbidity values at 23°C and 60°C throughout the test. The trend of each curve exhibits an initial spike at the beginning of the spray cycle, followed by a sharp decline during the first four hours of the test. The turbidity results continued to decline and remained low from Day 1 until Day 30. During the last three weeks of

testing, the turbidity values for the 23°C and 60°C samples averaged 0.54 NTU and 0.56 NTU, respectively.

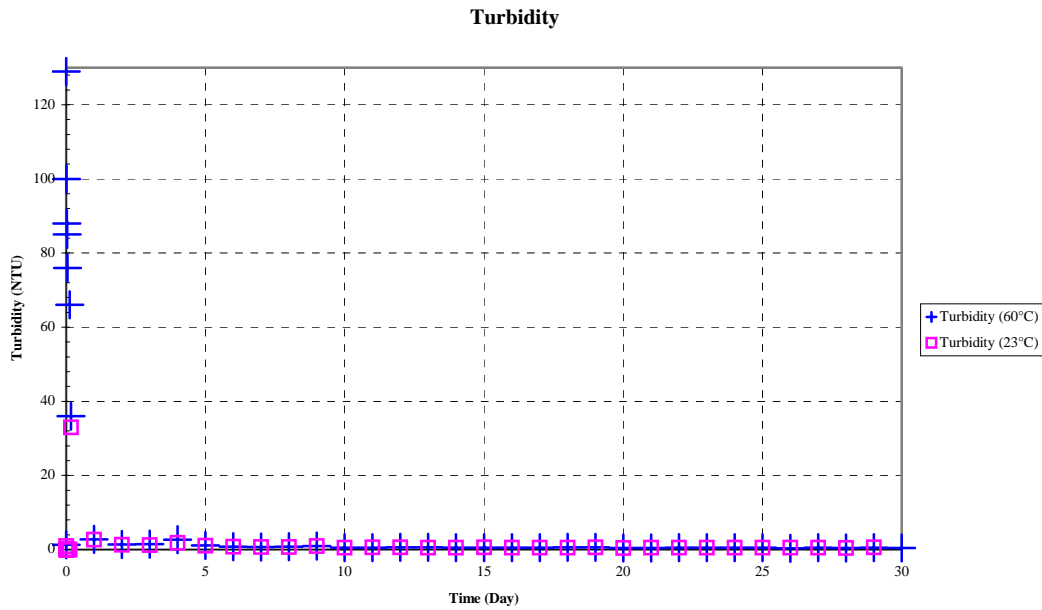
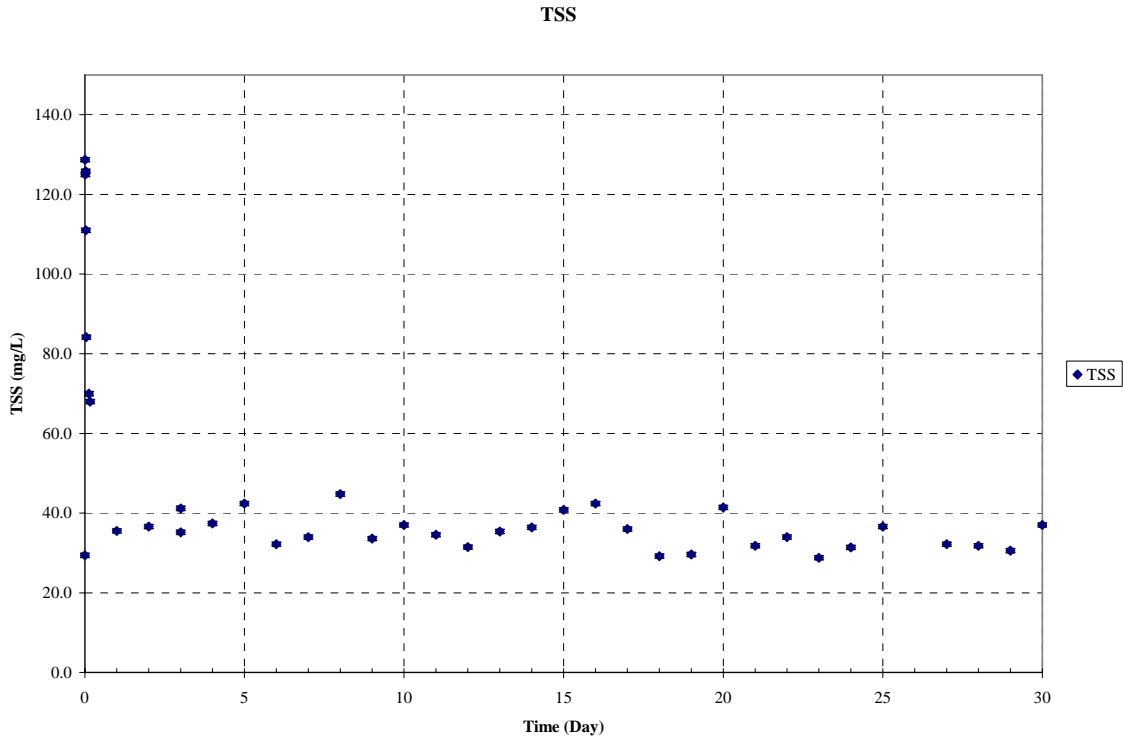


Figure 3-5. Daily turbidity results.

**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. 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 #4 TSS data as the experiment progressed. The baseline TSS measurement, taken before time zero, was 29.4 mg/L. Further TSS measurements were taken throughout the spray cycle. At the start of the spray cycle, the TSS measurement was at its highest value of 128.7 mg/L. At four hours, the TSS measurement had dropped to 68.0 mg/L. Following the spray cycle and beginning at 24 hours, the TSS measurements were performed daily. After 24 hours, TSS measurements varied approximately 16 mg/L over the 30-day test period, from a low of 28.8 mg/L to a high of 44.8 mg/L.



**Figure 3-6. Test #4 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 ( $\pm 1.0^\circ\text{C}$ ) [140°F ( $\pm 1.8^\circ\text{F}$ )] and 23°C ( $\pm 2.0^\circ\text{C}$ ) [73.4°F ( $\pm 3.6^\circ\text{F}$ )]. Viscosity of water 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). The measured viscosity is shown in Figure 3-7. The values were steady throughout the test.

Viscosity 60°C and 23°C

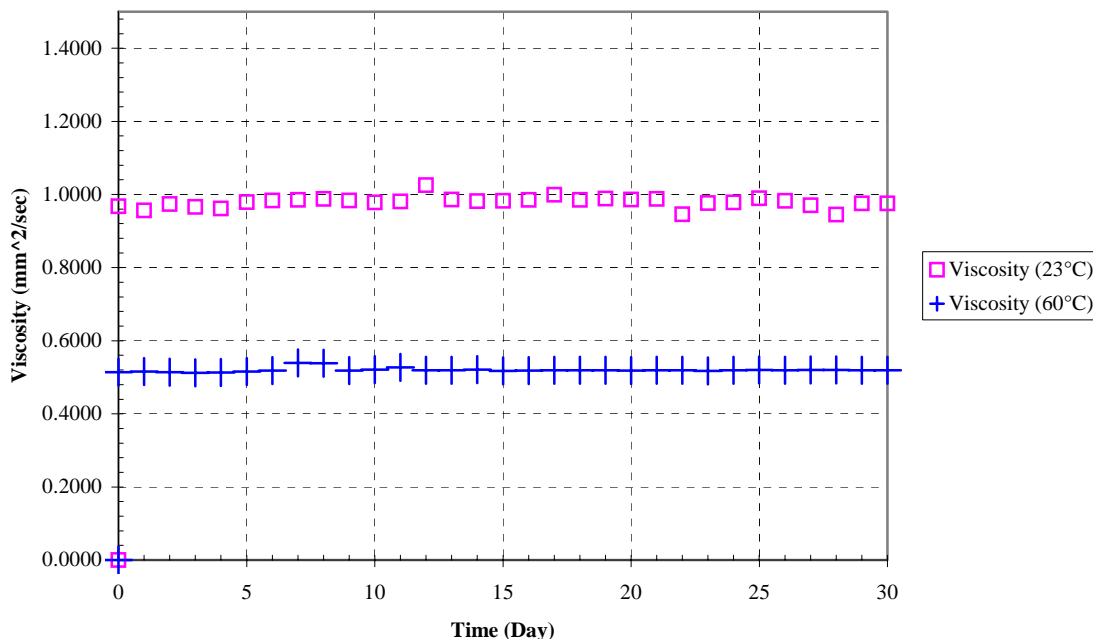


Figure 3-7. Viscosity at 60°C and 23°C.

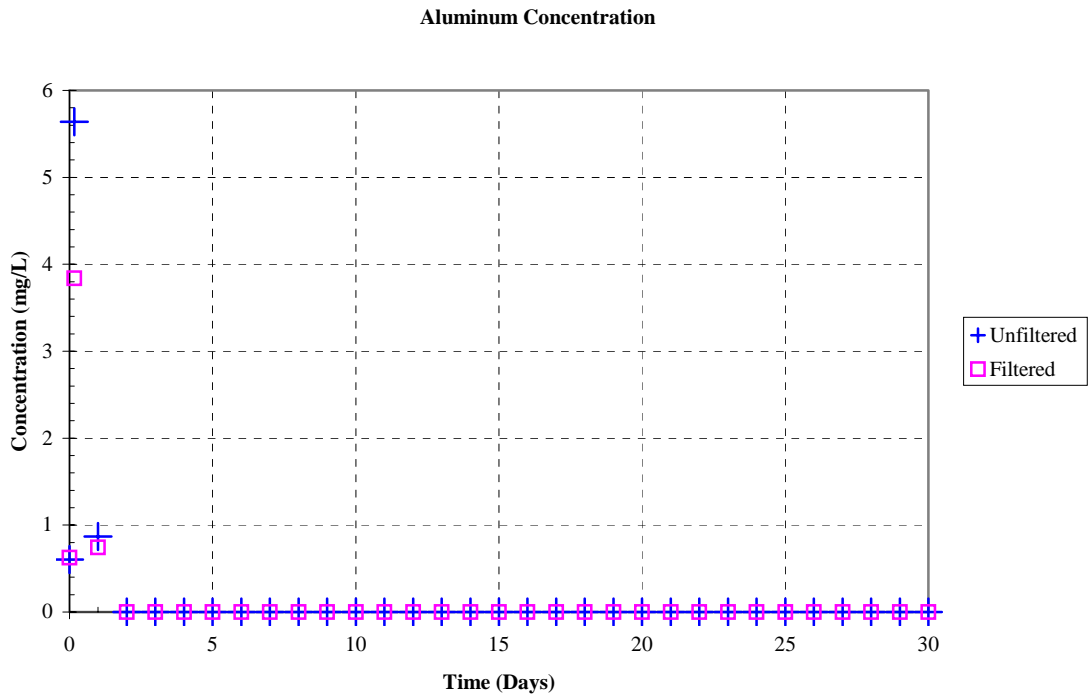
### 3.2.2. Metal Ion Concentration

ICP results for Test #4 are displayed in Figures 3-8 through 3-15, which are daily sample results. Table 3-1 contains ICP results for elements that were analyzed on Days 1, 17, and 30. Table 3-1 shows the chloride, boron, lead, lithium, and potassium concentrations. An examination of the figures reveals that aluminum, copper, iron, magnesium, and zinc were present in trace amounts, below 1 mg/L. It also can be seen that calcium, silica, and magnesium are present in higher concentrations.

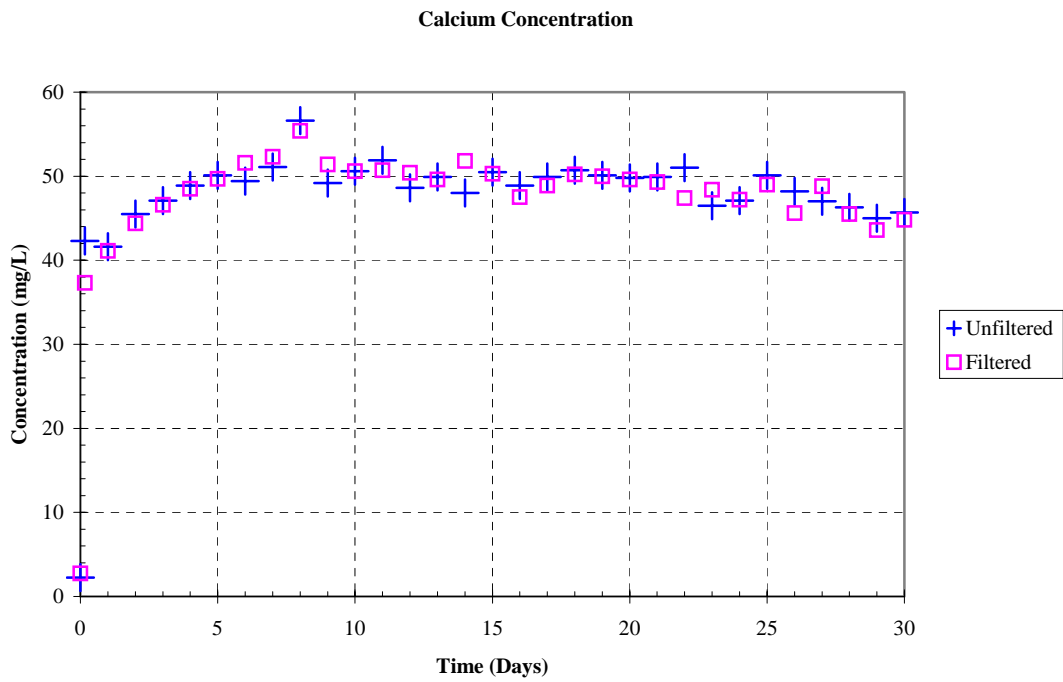
Table 3-1. ICP Results for Selected Elements

Sample Time	Unfiltered Samples				
	Chloride	Boron	Lead	Lithium	Potassium
	mg/L				
Baseline	83.5	2880	0.02	0.19	7.7
4 Hours	87.7	2830	0.02	0.23	46.9
Day 1	88.9	2880	0.02	0.22	52.0
Day 17	93.3	2860	0.02	0.29	67.1
Day 30	91.0	3390	0.02	0.26	39.9





**Figure 3-8. Aluminum concentration.**



**Figure 3-9. Calcium concentration.**

### Copper Concentration

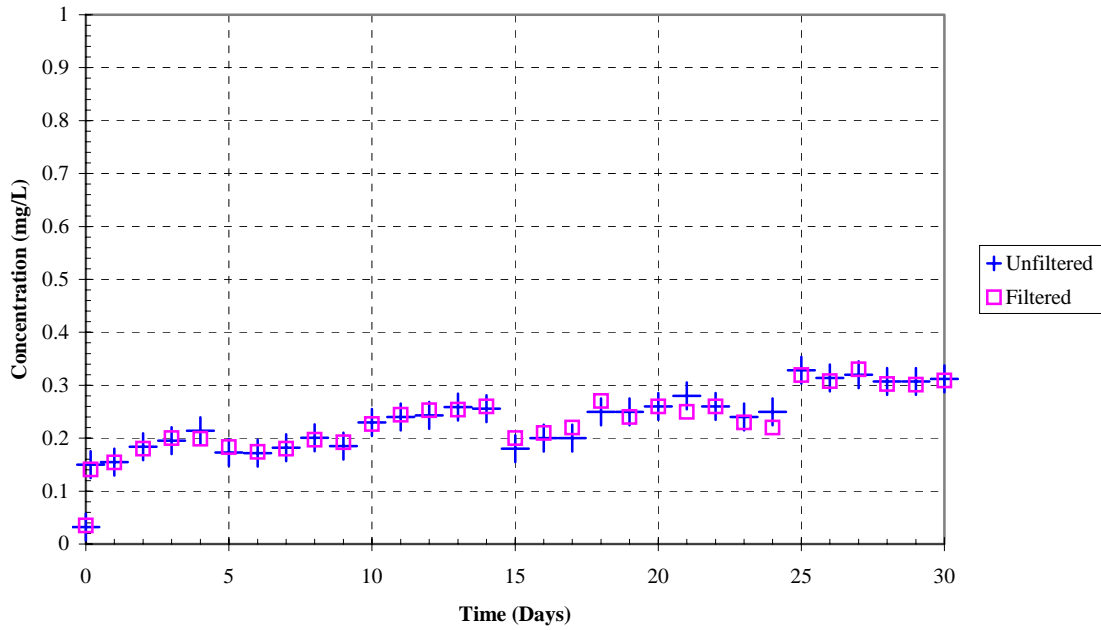


Figure 3-10. Copper concentration.

### Iron Concentration

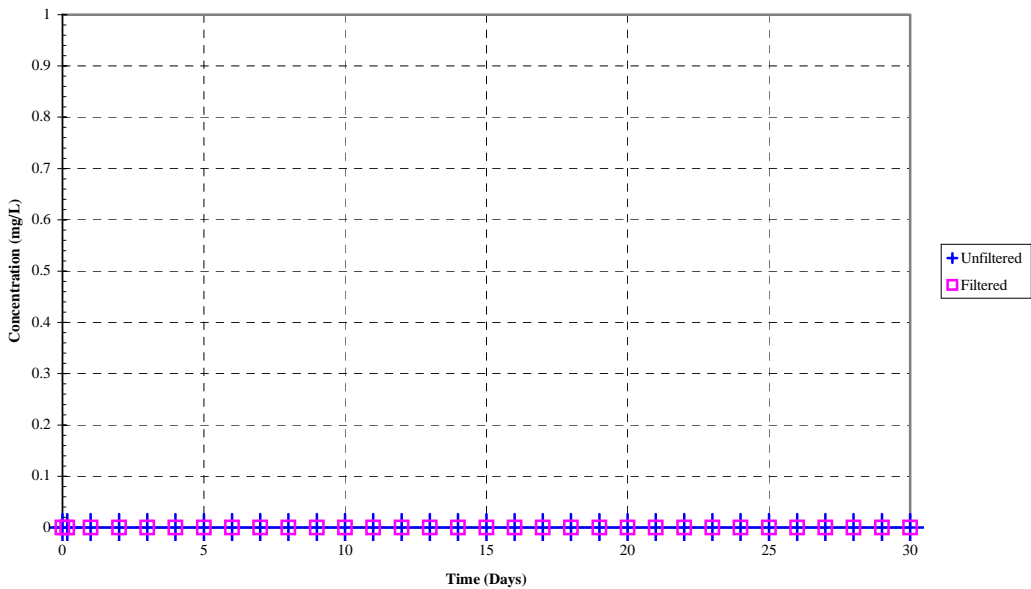
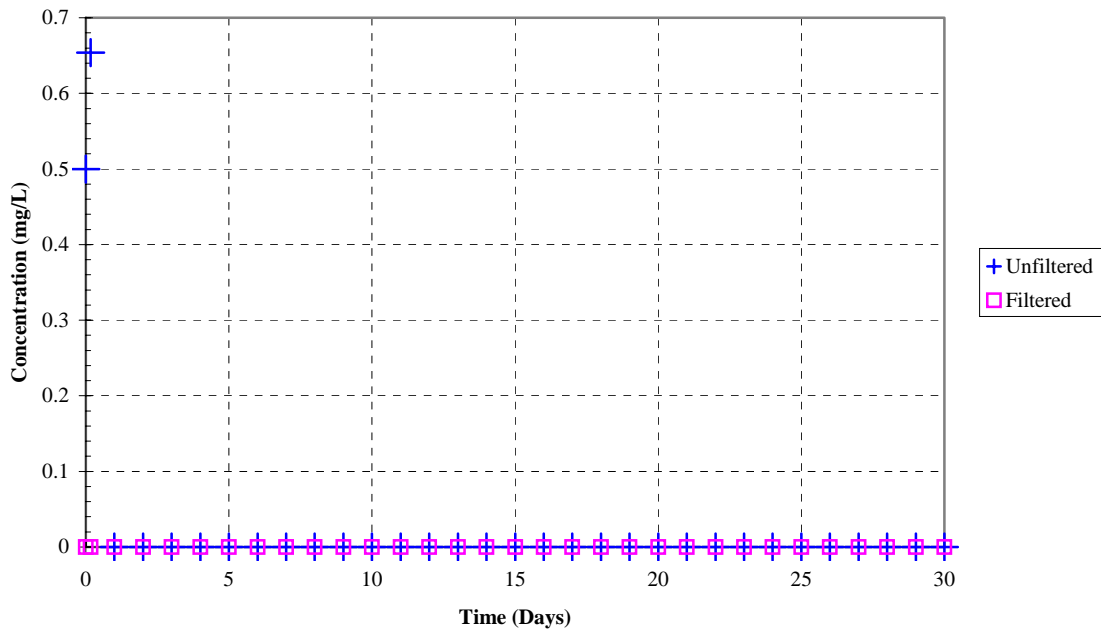


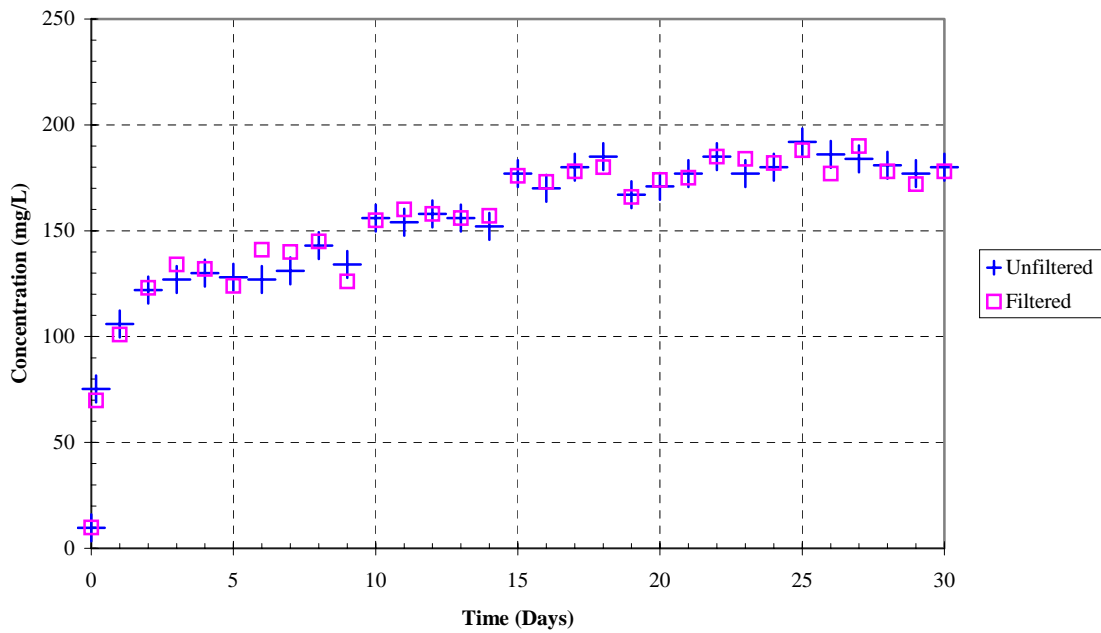
Figure 3-11. Iron concentration.

**Magnesium Concentration**

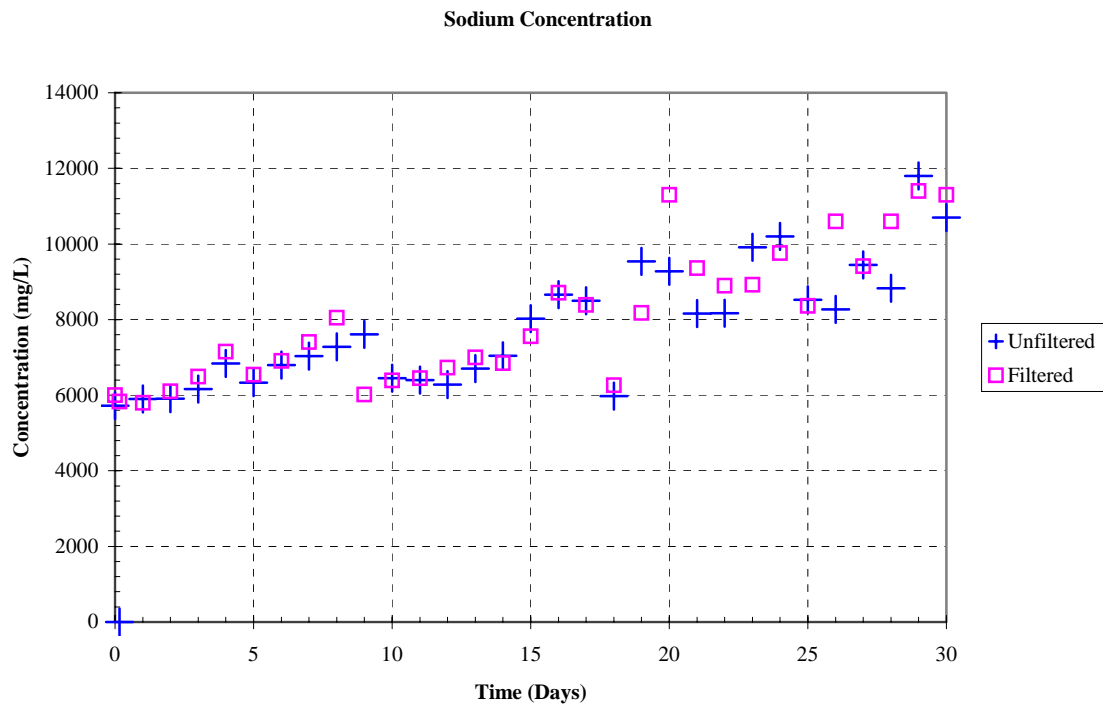


**Figure 3-12. Magnesium concentration.**

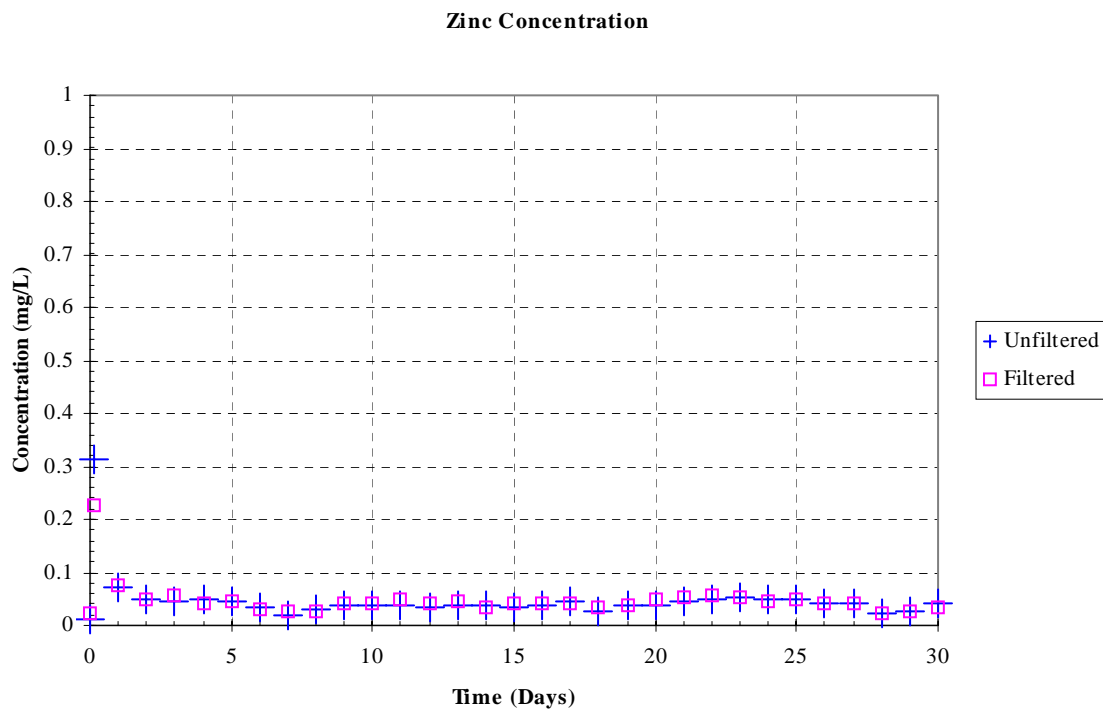
**Silica Concentration**



**Figure 3-13. Silica concentration.**



**Figure 3-14. Sodium concentration.**



**Figure 3-15. Zinc concentration.**

### **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 NUKON™ 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.