

Appendix D

ESEM/EDS Data for Test #4, Day-30 Low-Flow Cal-Sil Samples

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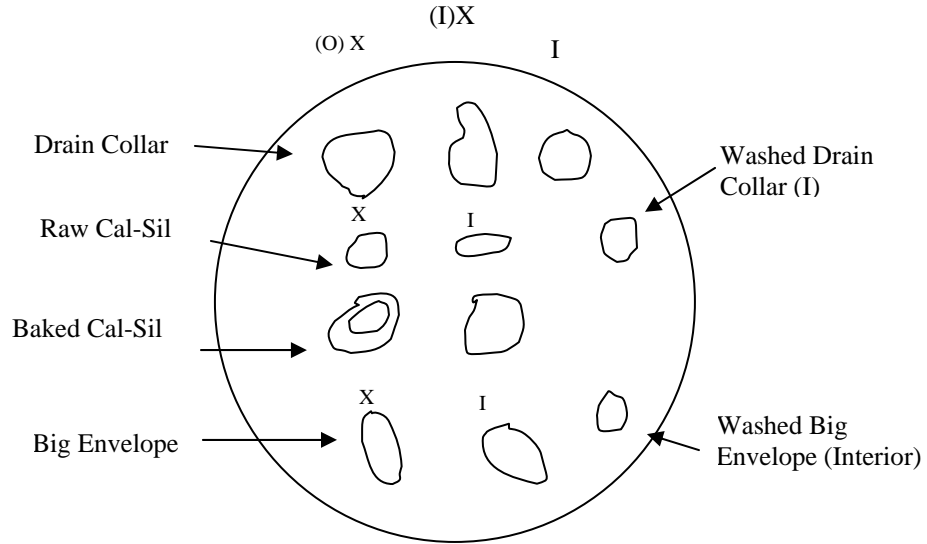
This appendix presents the ESEM/EDS results of Test #4, Day-30 raw and baked cal-sil samples submerged in a low-flow zone. The cal-sil samples were collected on the date Test #4 was shut down (June 23, 2005). ESEM was employed to analyze the hydrated cal-sil samples without any coating under a low-vacuum condition (i.e., 80 Pa) to minimize the modification of the cal-sil samples through a drying process. The ESEM/EDS analytical results of the cal-sil samples were obtained on June 30, 2005. EDS results provide a semi-quantitative elemental analysis of the sample compositions.

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Transcribed Laboratory Log

Laboratory session from June 30, 2005.

ESEM/EDS Test #4, Day-30 Low-Flow Cal-Sil



Submerged Raw Cal-Sil (Low-Flow) Exterior

Image:	T4RC EX01	100 ×	ESEM image	Figure D-1
	t4rcex02	500 ×	ESEM at higher magnification	Figure D-2
EDS:	t4rcex03		EDS on whole image t4rcex02	Figure D-3

Submerged Raw Cal-Sil (Low-Flow) Interior

Image:	t4rcin04	100 ×	ESEM image	Figure D-4
	t4rcin05	500 ×	ESEM at higher magnification	Figure D-5
EDS:	t4rcin06		EDS of whole image t4rcin05	Figure D-6

Low-Flow Exterior Submerged Baked Cal-Sil

Image:	T4BCEX07	100 ×	ESEM image	Figure D-7
	t4bcex08	500 ×	ESEM at higher magnification	Figure D-8
EDS:	t4bcex09		EDS of whole image of t4bcex08	Figure D-9

Low-Flow Interior Submerged Baked Cal-Sil

Image:	T4BCIN10	100 ×	ESEM image	Figure D-10
	t4bcin11	500 ×	ESEM at higher magnification	Figure D-11
EDS:	t4bcin12		EDS of whole image of t4bcin11	Figure D-12

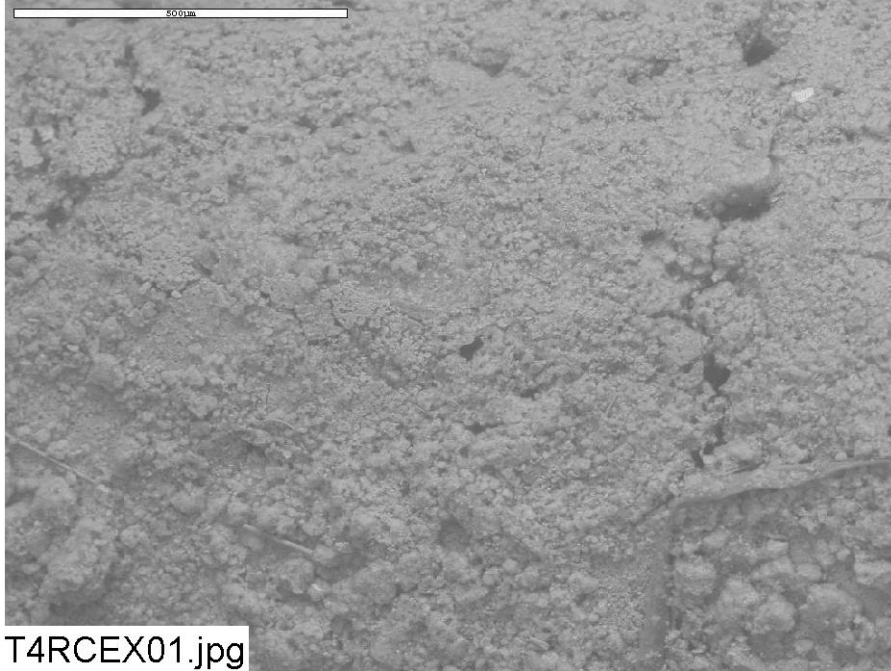


Figure D-1. ESEM image magnified 100 times for a Test #4, Day-30 low-flow exterior raw cal-sil sample. (T4RCEX01.jpg)

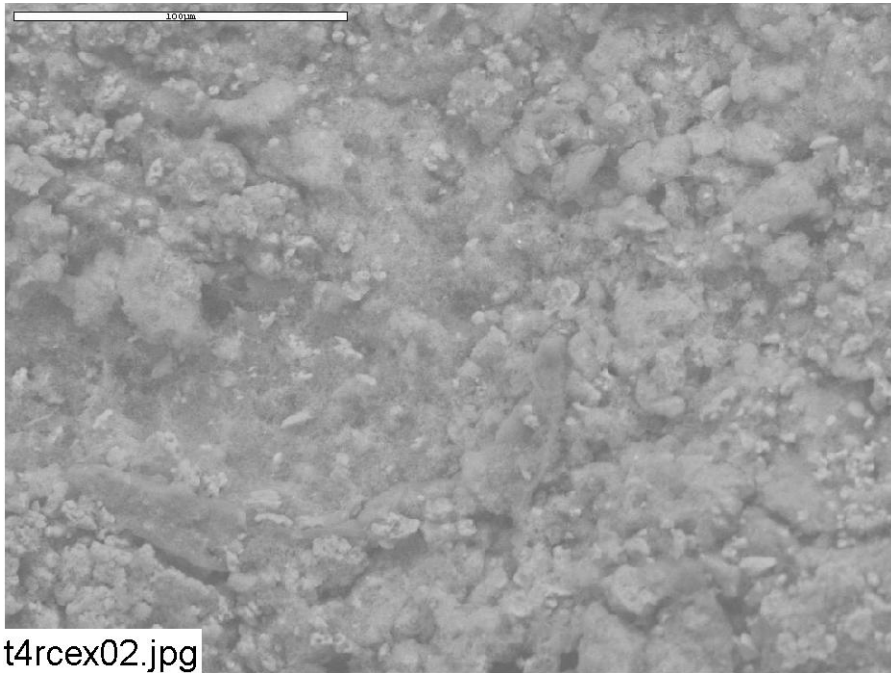


Figure D-2. ESEM image magnified 500 times for a Test #4, Day-30 low-flow exterior raw cal-sil sample. (t4rcex02.jpg)

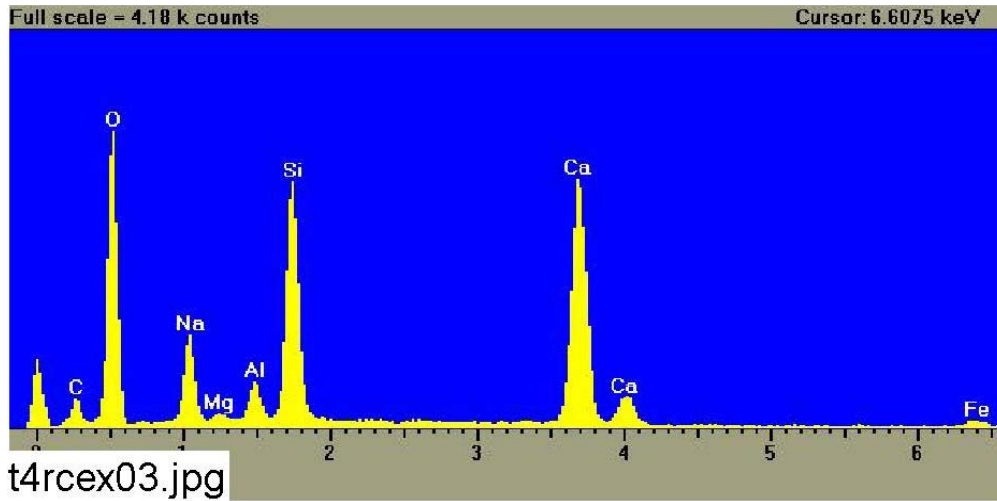


Figure D-3. EDS counting spectrum for the whole image shown in Figure D-2. (t4rcex03.jpg)

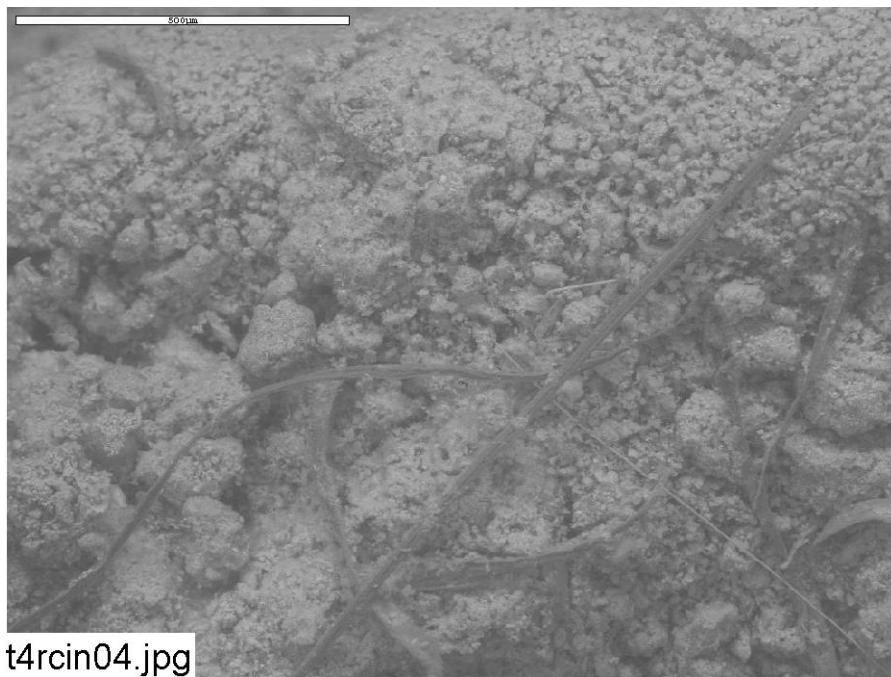


Figure D-4. ESEM image magnified 100 times for a Test #4, Day-30 low-flow interior raw cal-sil sample. (t4rcin04.jpg)

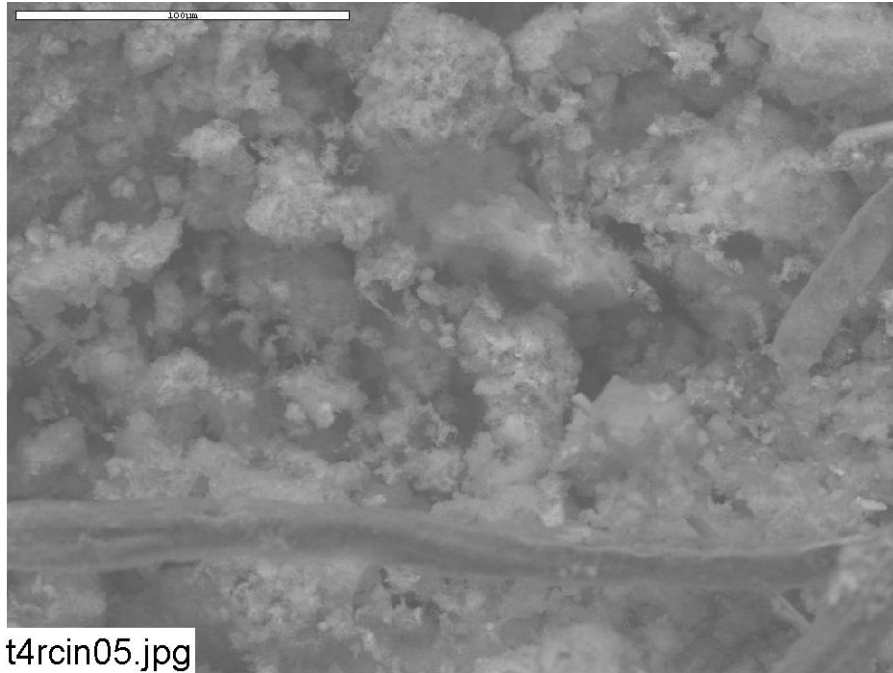


Figure D-5. ESEM image magnified 500 times for a Test #4, Day-30 low-flow interior raw cal-sil sample. (t4rcin05.jpg)

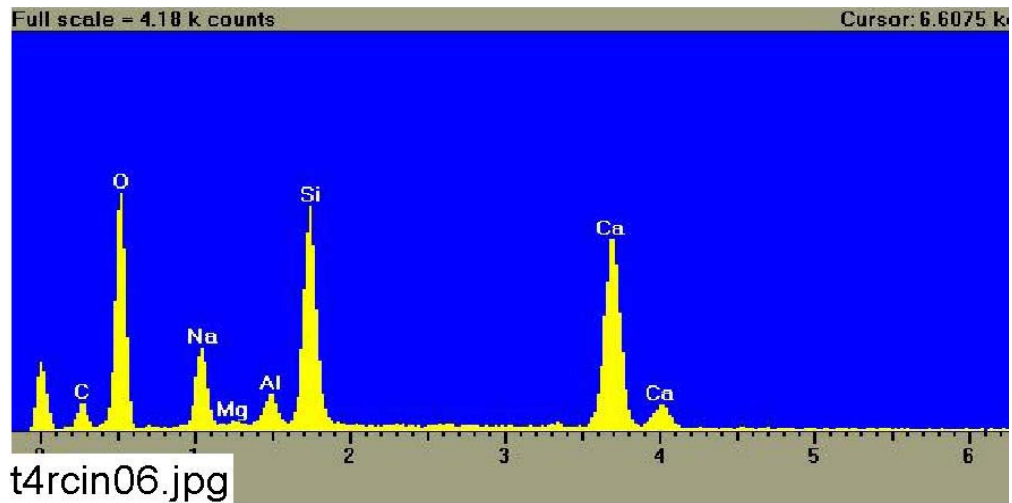


Figure D-6. EDS counting spectrum for the whole image shown in Figure D-5. (t4rcin06.jpg)

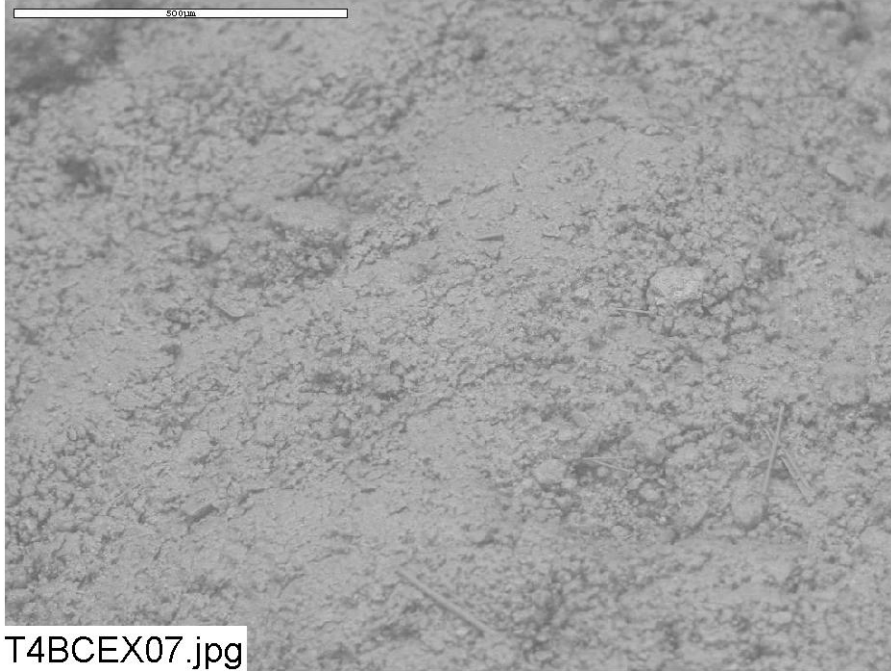


Figure D-7. ESEM image magnified 100 times for a Test #4, Day-30 low-flow exterior baked cal-sil sample. (T4BCEX07.jpg)

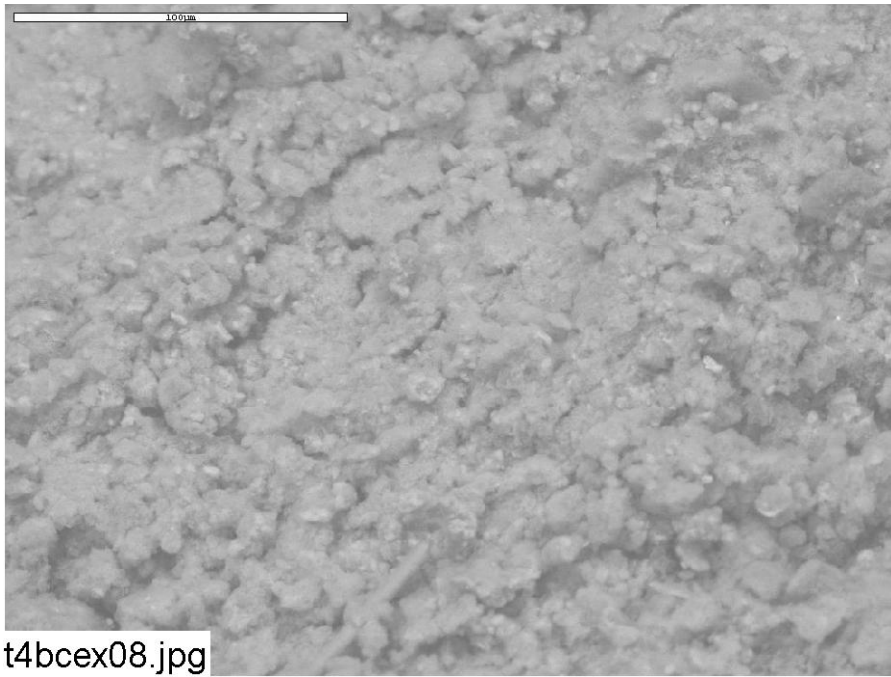


Figure D-8. ESEM image magnified 500 times for a Test #4, Day-30 low-flow exterior baked cal-sil sample. (t4bcex08.jpg)

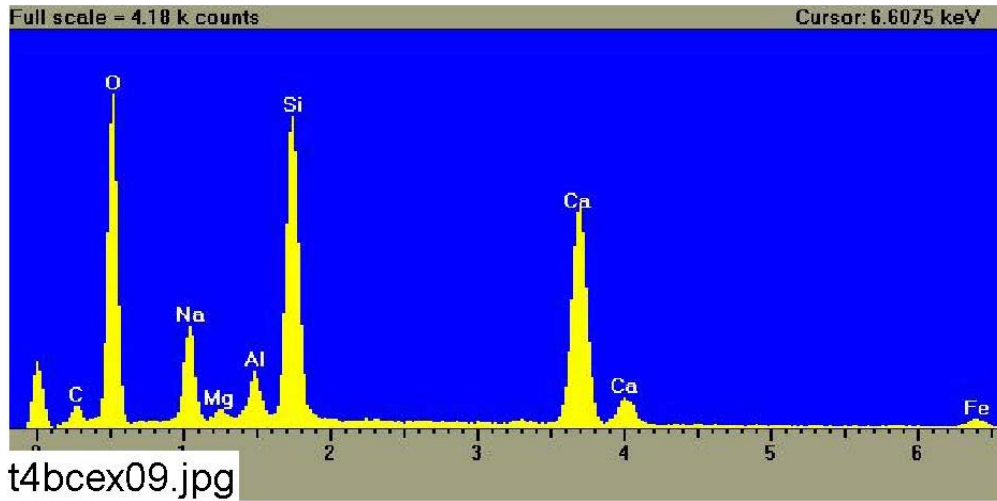


Figure D-9. EDS counting spectrum for the whole image shown in Figure D-7. (t4bcex09.jpg)

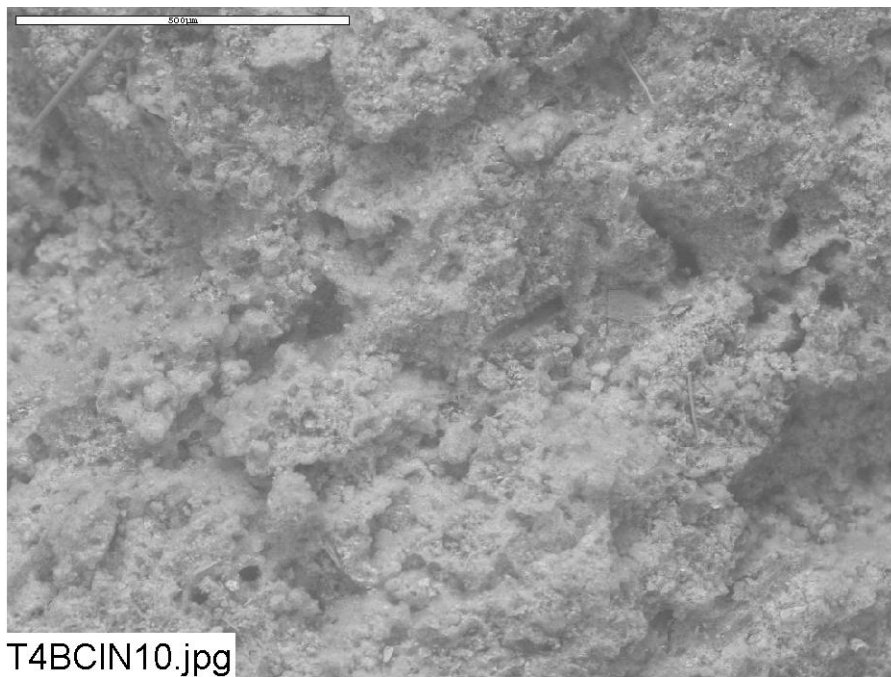


Figure D-10. ESEM image magnified 100 times for a Test #4, Day-30 low-flow interior baked cal-sil sample. (T4BCIN10.jpg)

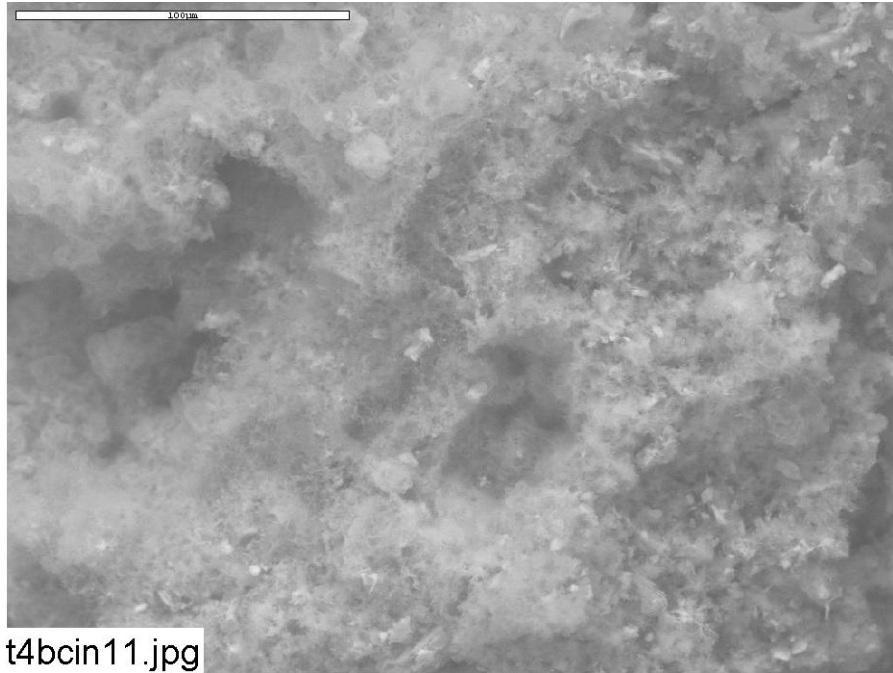


Figure D-11. ESEM image magnified 500 times for a Test #4, Day-30 low-flow interior baked cal-sil sample. (t4bcin11.jpg)

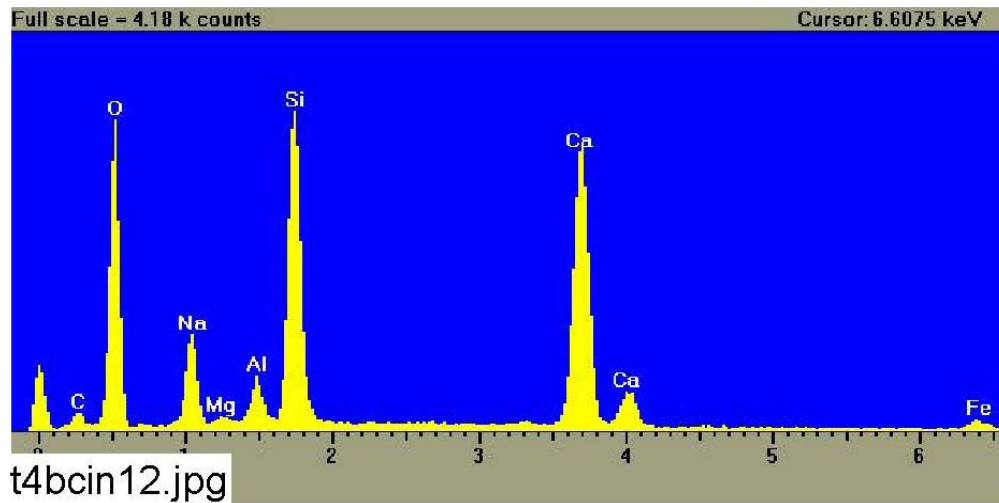


Figure D-12. EDS counting spectrum for the whole image shown in Figure D-11. (t4bcin12.jpg)

Appendix E

ESEM and SEM/EDS Data for Test #4, Day-30 Deposition Products

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For ICET, of interest is the corrosion/reaction effect of metal and concrete coupons, as well as the deposition of debris in the tank. To understand the corrosion processes that have occurred in the test, one direct way is the examination of the corrosion/deposition products after the test is completed. For this purpose, the corrosion/deposition products were collected on the date Test #4 was shut down (June 23, 2005). These products are fine powders on the submerged CPVC rack.

These products were collected by directly adhering onto double-sided carbon tapes for probe SEM/EDS examination. After the samples were dried in air, an Au/Pd coating was applied to enhance the surface conductivity of the samples and to prevent possible charging problems during SEM examination. Based on EDS results, a semi-quantitative elemental analysis was performed after calibration. This appendix presents the SEM/EDS data that were obtained on June 29, 2005.

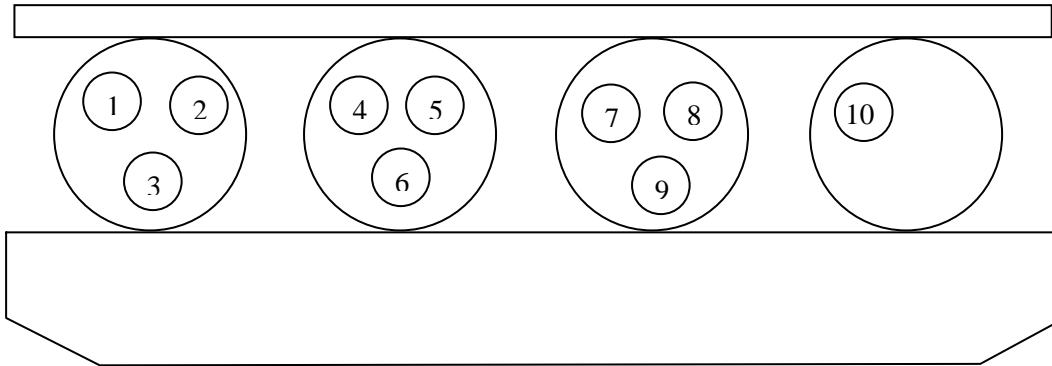
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Transcribed Laboratory Log

Laboratory session from June 29, 2005.

ESEM Test #4, Day-30 Deposition Products

- | | | | |
|-----------------|-------------------------|-------------------|---------------|
| 1. Suspended Al | 3. Sus. Cu | 5. Sus. Gal-Steel | 7. Sus. Steel |
| 2. Submerged Al | 4. Sub. Cu | 6. Sub. Gal Steel | 8. Sub. Steel |
| 9. Sediment | 10. Powder on Sub. Rack | | |



Powder on Submerged Rack

Image:	T4D30RackPowder029	500 ×	ESEM image	Figure E-1
	T4D30RackPowder030	1000 ×	ESEM at higher magnification	Figure E-2
EDS:	T4D30RackPowder018		EDS of whole of image 30	Figure E-3

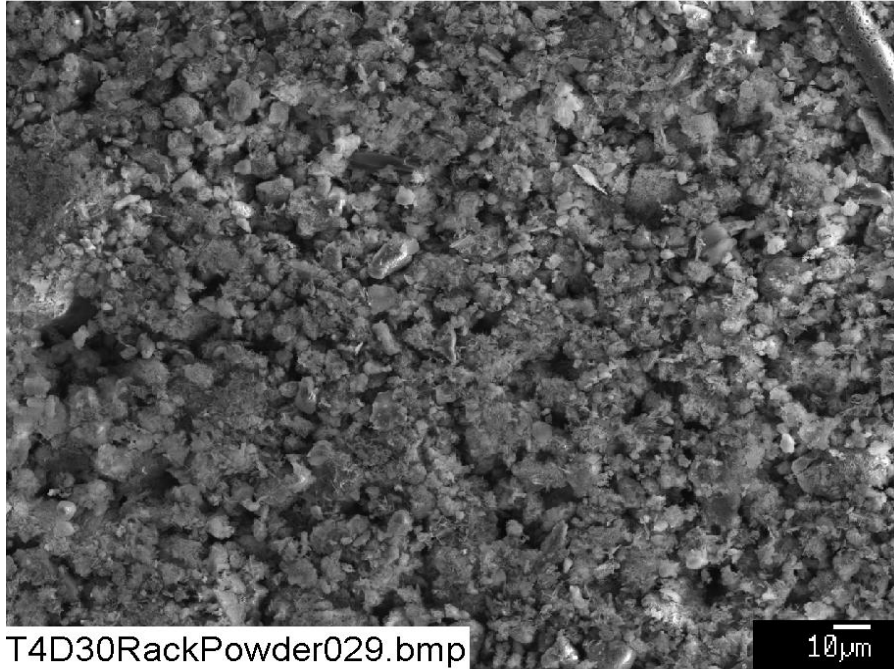


Figure E-1. SEM image magnified 500 times for a Test #4, Day-30 fine powder on the submerged rack. (T4D30RackPowder029.bmp)

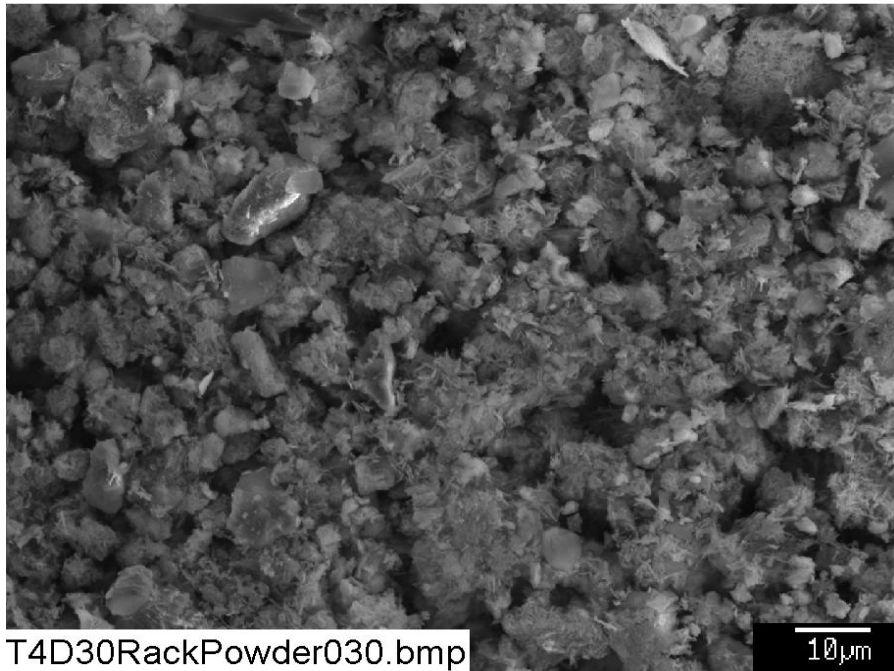


Figure E-2. SEM image magnified 1000 times for a Test #4, Day-30 fine powder on the submerged rack. (T4D30RackPowder030.bmp)

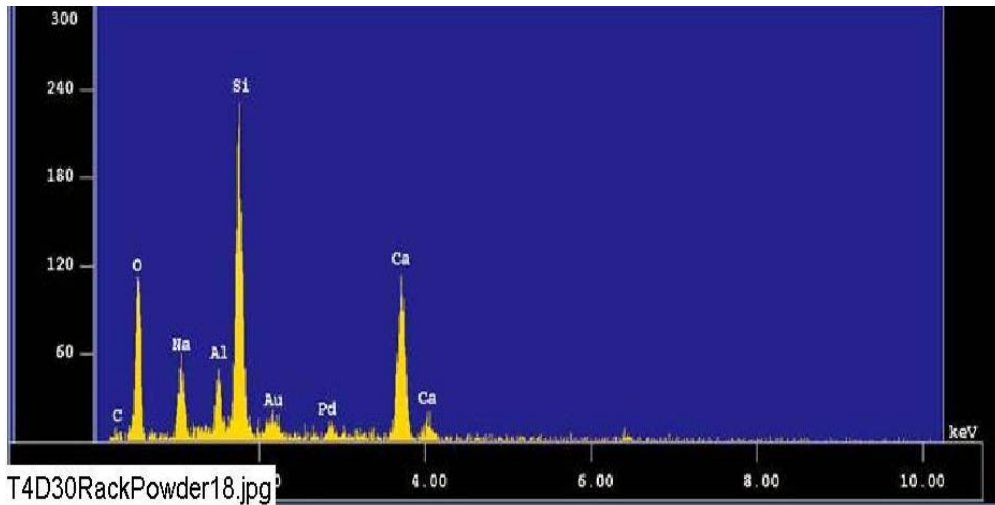


Figure E-3. EDS counting spectrum for the particles (whole image) shown in Figure E-2. (T4D30RackPowder18.jpg)

The results from the chemical composition analysis for T4D30RackPowder18.jpg are given in Table E-1.

Table E-1. Chemical Compositions for T4D30RackPowder18.jpg, Figure E-3

Jun 29 15:55 2005 /tmp/eds_pout.log Page 1

```

Group       : NRC
Sample      : T4D30 ID# : 18
Comment     : powder on submerged rack
Condition   : Full Scale : 20KeV(10eV/ch,2Kch)
              Live Time  : 60.000 sec   Aperture #   : 2
              Acc. Volt  : 15.0 KV      Probe Current : 1.068E-09 A
              Stage Point : X=86.234 Y=59.512 Z=11.000
              Acq. Date  : Wed Jun 29 15:53:49 2005
    
```

Element	Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Background	
O K	Normal	0.25- 0.77	30.9731	0.0014	1027 /	3
Na K	Normal	0.81- 1.27	4.6719	0.0006	445 /	8
Al K	Normal	1.26- 1.78	2.8315	0.0004	390 /	71
Si K	Normal	1.50- 2.07	10.6068	0.0007	1361 /	32
Ca K	Normal	3.40- 4.30	15.8585	0.0055	989 /	5
C K	Normal	0.09- 0.46	0.1513	0.0001	7 /	10

Chi_square = 1.6428

Element	Mass%	Atomic%	ZAF	Z	A	F
O	51.787	66.6327	1.3652	0.9856	1.3851	1.0000
Na	8.112	7.2632	1.4177	1.0401	1.3635	0.9996
Al	4.182	3.1907	1.2060	1.0015	1.2094	0.9957
Si	15.974	11.7078	1.2296	0.9896	1.2431	0.9995
Ca	19.144	9.8324	0.9857	0.9980	0.9875	1.0001
C	0.801	1.3733	4.3237	1.0335	4.1836	0.9999

Total 100.000 100.0000
Normalization factor = 1.2247

Appendix F1

SEM/EDS Data for Test #4, Day-30 Aluminum Coupons

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Table F1-4.	Chemical Compositions for T4D30Alsubm04.jpg, Figure F1-10	F1-13

This appendix shows the SEM/EDS results for the metal aluminum coupons under two categories: (1) unsubmerged and (2) submerged. Unsubmerged refers to coupons held in the test tank gas space above the water level of the solution during ICET. Unsubmerged coupons were contacted by the solution only during the 4-hour spraying period at the initial date of the test. In addition, the surface of the unsubmerged coupons may also have been affected by the moisture in the gas space during the test. Submerged refers to the coupons that were under the solution during the test.

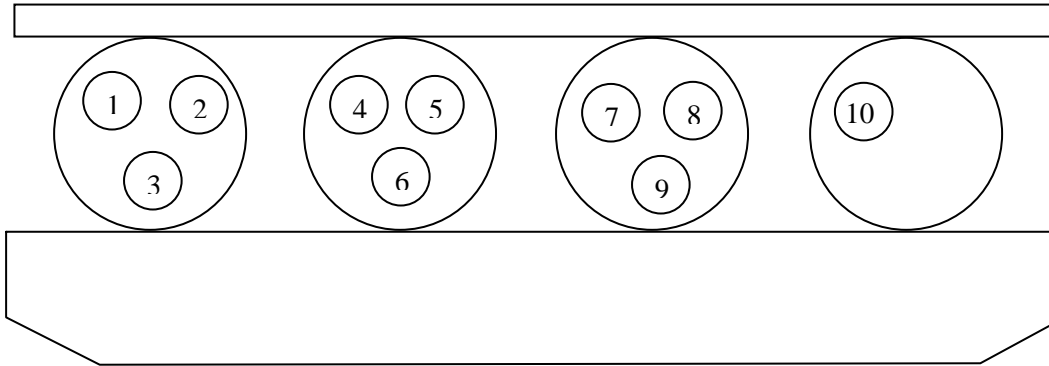
The coupon samples were collected on June 23, 2005 (the date Test #4 was shut down) and examined by SEM/EDS on June 29, 2005. The aluminum coupon samples were dried in air before being coated with Au/Pd for SEM examination. SEM results present the surface condition of the aluminum coupons. In addition, EDS results provide a semi-quantitative elemental analysis of the coupon surface and the corrosion products.

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Transcribed Laboratory Log

Laboratory session from June 29, 2005.
SEM Test #4, Day-30 Aluminum Coupons

- | | | | |
|-------------------|-------------------------|-------------------|---------------|
| 1. Unsubmerged Al | 3. Sus. Cu | 5. Sus. Gal-Steel | 7. Sus. Steel |
| 2. Submerged Al | 4. Sub. Cu | 6. Sub. Gal Steel | 8. Sub. Steel |
| 9. Sediment | 10. Powder on Sub. Rack | | |



Unsubmerged Aluminum Coupons

Image:	T4D30AlSusp001	100 ×	SEM image	Figure F1-1
	T4D30AlSusp002	500 ×	SEM image higher magnification	Figure F1-2
	T4D30AlSusp003	1000 ×	SEM annotated image	Figure F1-3
EDS:	T4D30AlSusp01		On particles at Al surface shown in image T4D30AlSusp003	Figure F1-4
	T4D30AlSusp02		On Al coupon surface shown in image T4D30AlSusp003	Figure F1-5

Submerged Al Coupon

Image:	T4D30AlSubm004	100 ×	SEM image of fiberglass	Figure F1-6
	T4D30AlSubm005	500 ×	SEM image higher magnification	Figure F1-7
	T4D30AlSubm006	1000 ×	SEM annotated image	Figure F1-8
EDS:	T4D30AlSubm03		EDS of particles shown in 006	Figure F1-9
	T4D30AlSubm04		EDS of Al surface in 006	Figure F1-10
Image:	T4D30AlSubm007	5000 ×	SEM image higher magnification	Figure F1-11

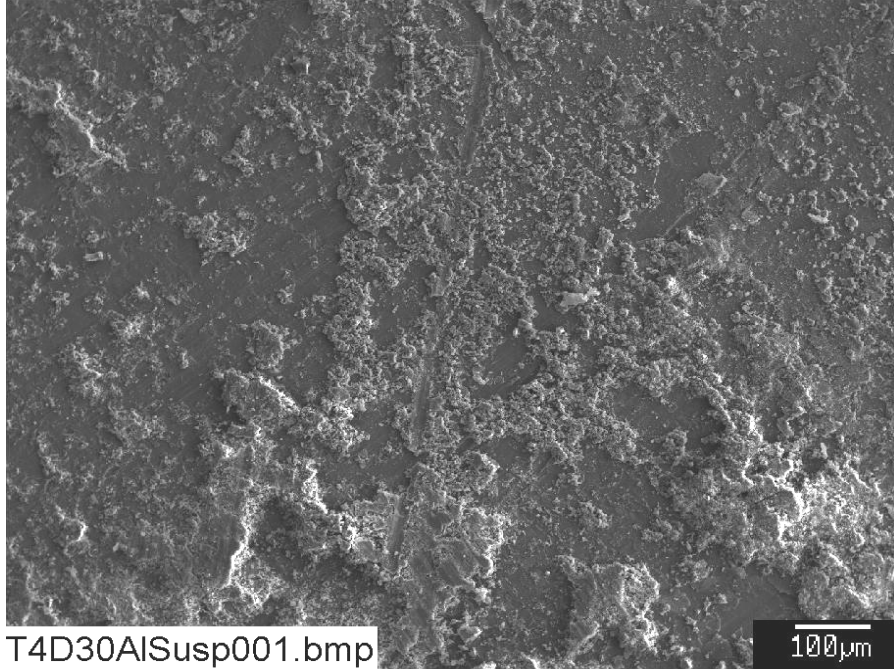


Figure F1-1. SEM image magnified 100 times for a Test #4, Day-30 unsubmerged aluminum coupon sample. (T4D30AlSusp001.bmp)

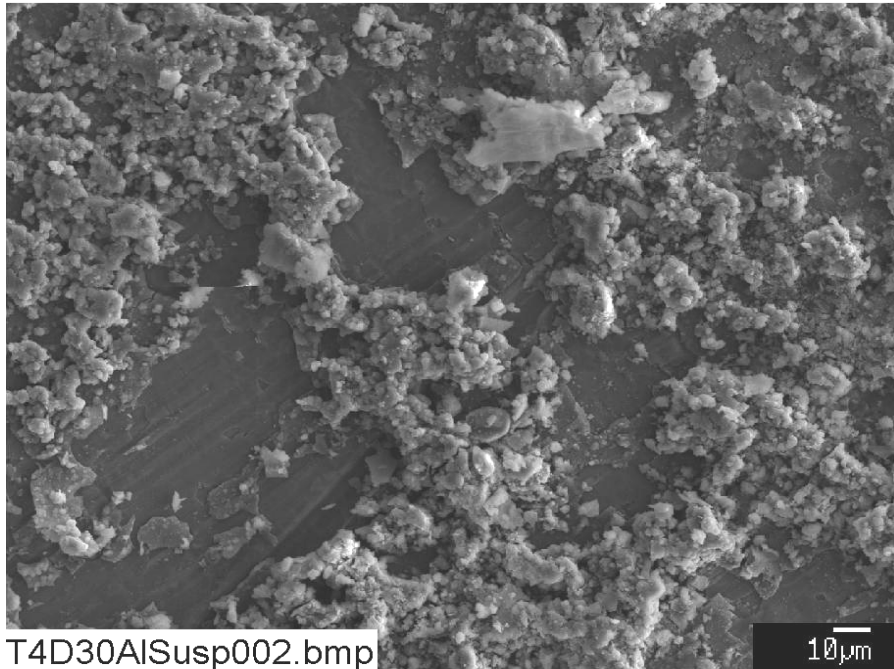


Figure F1-2. SEM image magnified 500 times for a Test #4, Day-30 unsubmerged aluminum coupon sample. (T4D30AlSusp002.bmp)

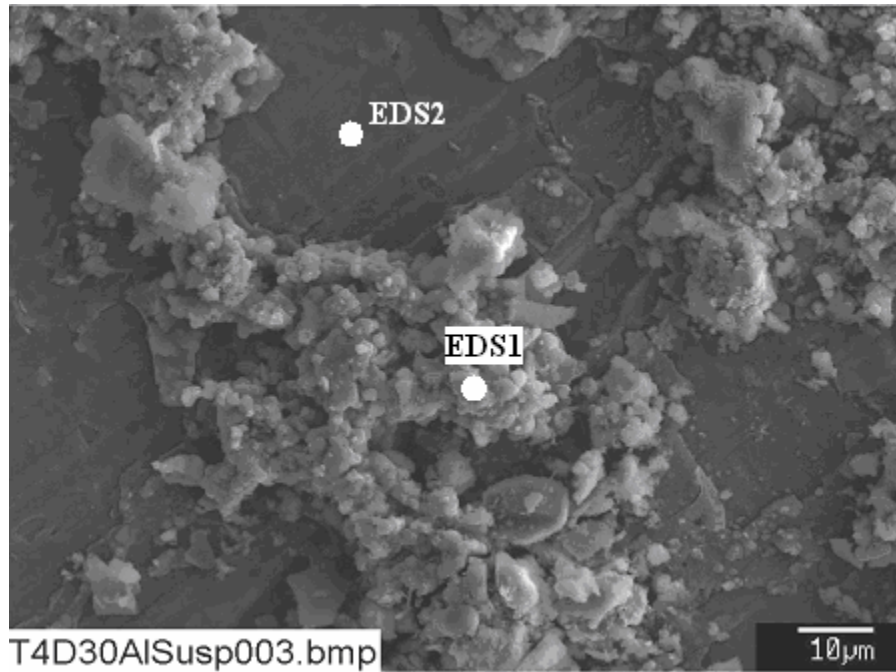


Figure F1-3. Annotated SEM image magnified 1000 times for a Test #4, Day-30 unsubmerged aluminum coupon sample. (T4D30AlSusp003.bmp)

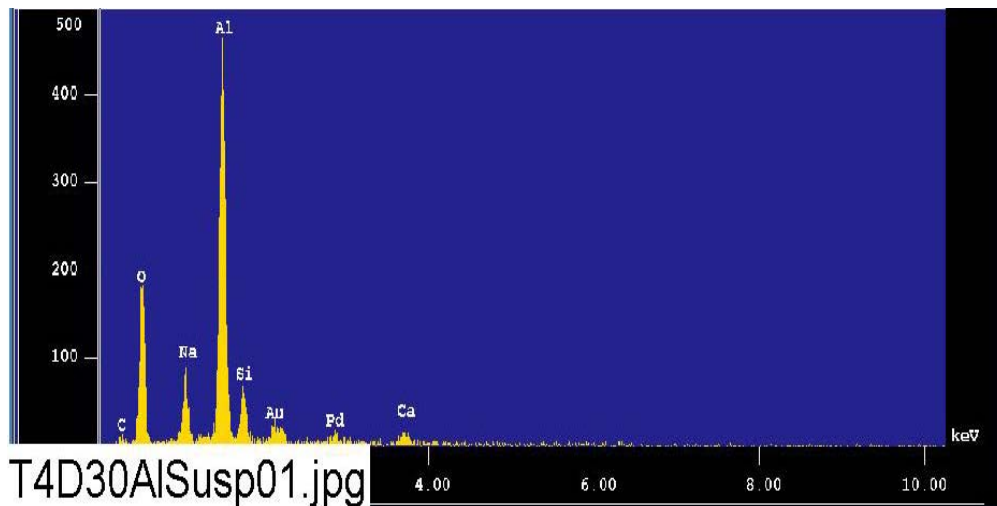


Figure F1-4. EDS counting spectrum for the deposits (EDS1) on the coupon surface shown in Figure F1-3. (T4D30AlSusp01.jpg)

The results from the chemical composition analysis for T4D30AlSusp01.jpg are given in Table F1-1.

Table F1-1. Chemical Compositions for T4D30AlSusp01.jpg, Figure F1-4

Jun 29 10:11 2005

```

Group       : NRC
Sample      : T4D30 ID# : 1
Comment     : Particle on suspended Al
Condition   : Full Scale : 20KeV(10eV/ch,2Kch)
              Live Time  : 60.000 sec   Aperture #   : 2
              Acc. Volt  : 15.0 KV      Probe Current : 1.069E-09 A
              Stage Point : X=86.836 Y=58.400 Z=10.786
              Acq. Date   : Wed Jun 29 10:06:34 2005
    
```

Element	Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	49.7713	0.0017	1653 / 8
Na K	Normal	0.81- 1.27	6.5744	0.0007	627 / 6
Al K	Normal	1.26- 1.78	29.7835	0.0010	4103 / 29
Si K	Normal	1.50- 2.07	3.1103	0.0004	400 / 238
Ca K	Normal	3.40- 4.30	1.8580	0.0027	116 / 2
C K	Normal	0.09- 0.46	0.1137	0.0001	6 / 16

 Chi_square = 3.1919

Element	Mass%	Atomic%	ZAF	Z	A	F
O	47.921	60.0922	0.9108	0.9888	0.9212	0.9999
Na	8.495	7.4130	1.2222	1.0435	1.1736	0.9980
Al	35.757	26.5870	1.1356	1.0048	1.1313	0.9990
Si	5.079	3.6282	1.5447	0.9930	1.5557	1.0000
Ca	1.975	0.9887	1.0056	1.0019	1.0036	1.0001
C	0.773	1.2909	6.4296	1.0368	6.2020	1.0000

 Total 100.000 100.0000
 Normalization factor = 1.0572

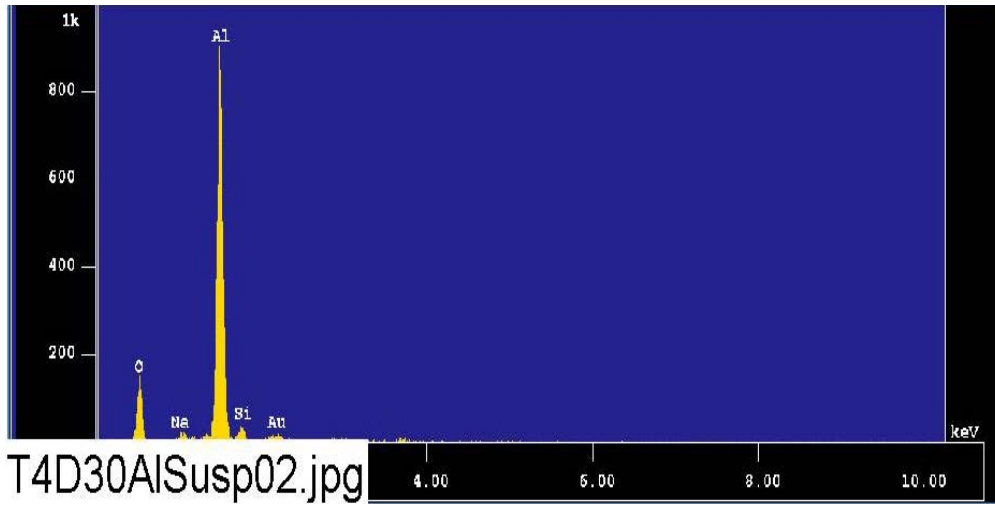


Figure F1-5. EDS counting spectrum for the flat coupon surface (EDS2) shown in Figure F1-3. (T4D30AlSusp02.jpg)

The results from the chemical composition analysis for T4D30AlSusp02.jpg are given in Table F1-2.

Table F1-2. Chemical Compositions for T4D30AlSusp02.jpg, Figure F1-5

Jun 29 10:15 2005

```

Group      : NRC
Sample     : T4D30 ID# : 2
Comment    : Surface of suspended Al
Condition  : Full Scale : 20KeV(10eV/ch,2Kch)
             Live Time  : 60.000 sec   Aperture #   : 2
             Acc. Volt   : 15.0 KV      Probe Current : 1.068E-09 A
             Stage Point : X=86.836 Y=58.400 Z=10.786
             Acq. Date   : Wed Jun 29 10:13:59 2005
    
```

Element	Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Background	
O K	Normal	0.25- 0.77	36.6938	0.0015	1217 /	4
Na K	Normal	0.81- 1.27	1.3775	0.0004	131 /	8
Al K	Normal	1.26- 1.78	57.7086	0.0014	7943 /	24
Si K	Normal	1.50- 2.07	1.7543	0.0003	225 /	430

 Chi_square = 4.6847

Element	Mass%	Atomic%	ZAF	Z	A	F
O	36.111	48.7449	0.9776	0.9856	0.9919	0.9999
Na	1.540	1.4468	1.1106	1.0400	1.0727	0.9956
Al	59.291	47.4571	1.0206	1.0013	1.0198	0.9995
Si	3.058	2.3512	1.7314	0.9893	1.7500	1.0000

 Total 100.000 100.0000
 Normalization factor = 1.0067

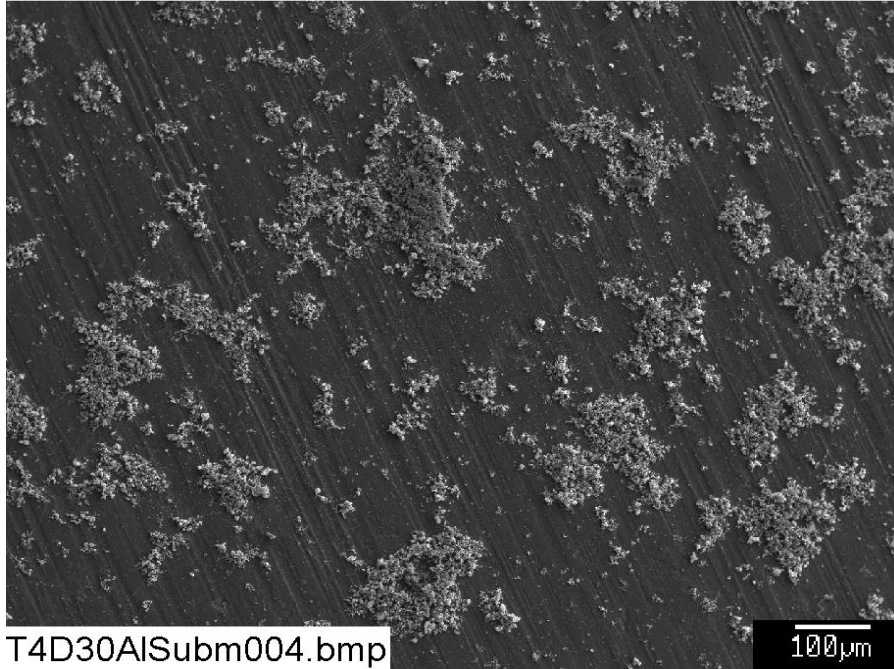


Figure F1-6. SEM image magnified 100 times for a Test #4, Day-30 submerged aluminum coupon sample. (T4D30AlSubm004.bmp)

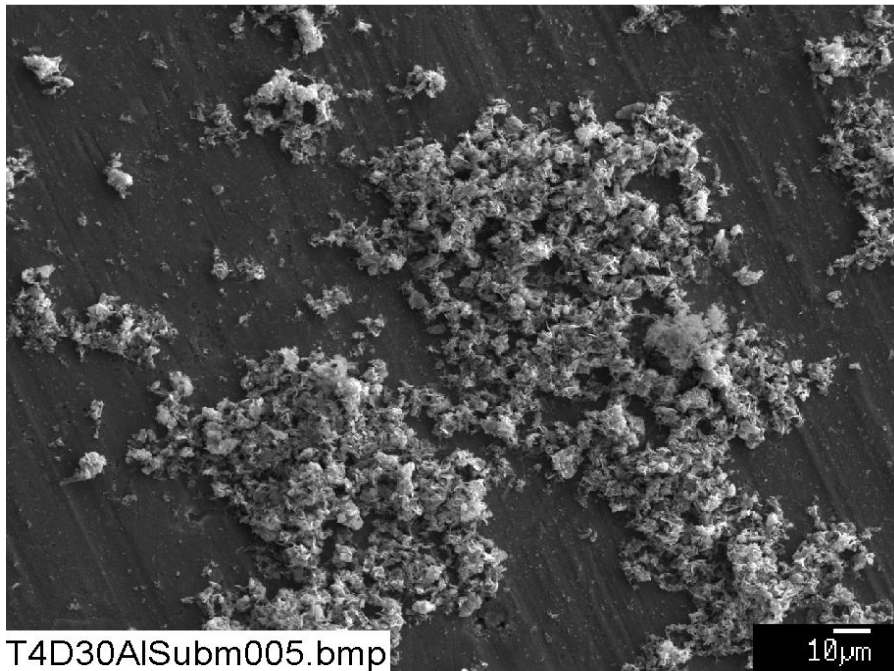


Figure F1-7. SEM image magnified 500 times for a Test #4, Day-30 submerged aluminum coupon sample. (T4D30AlSubm005.bmp)

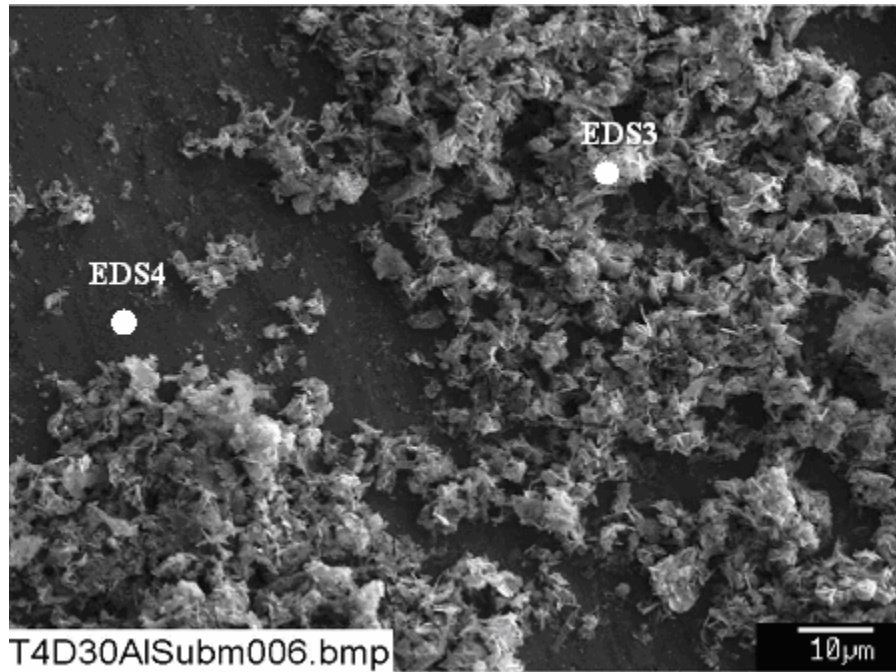


Figure F1-8. Annotated SEM image magnified 1000 times for a Test #4, Day-30 submerged aluminum coupon sample. (T4D30AlSubm006.bmp)

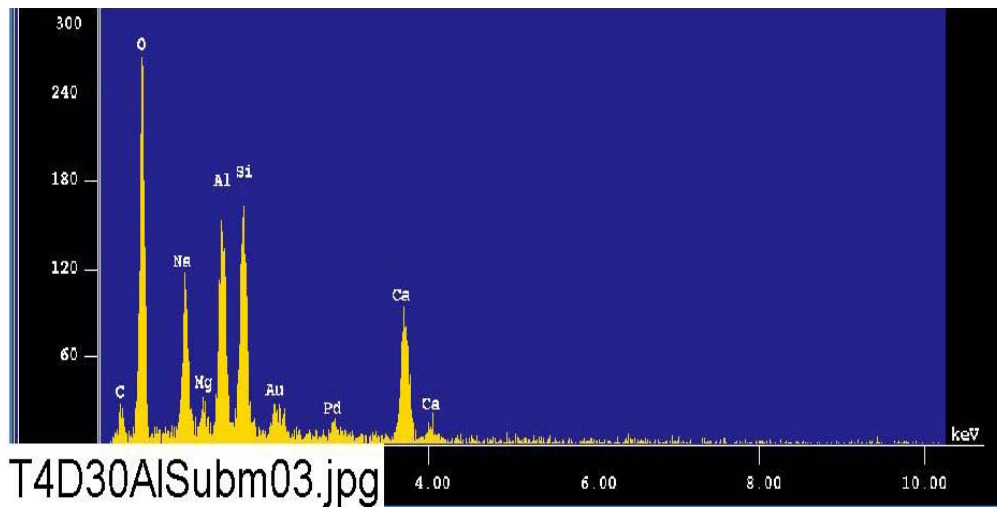


Figure F1-9. EDS counting spectrum for the deposits (EDS3) on the coupon surface shown in Figure F1-8. (T4D30AlSubm03.jpg)

The results from the chemical composition analysis for T4D30AlSubm03.jpg are given in Table F1-3.

Table F1-3. Chemical Compositions for T4D30AlSubm03.jpg, Figure F1-9

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```

Group       : NRC
Sample      : T4D30 ID# : 3
Comment     : Particles on submerged Al
Condition   : Full Scale : 20KeV(10eV/ch,2Kch)
              Live Time  : 60.000 sec   Aperture #   : 2
              Acc. Volt  : 15.0 KV      Probe Current : 1.067E-09 A
              Stage Point: X=74.706 Y=62.388 Z=10.786
              Acq. Date  : Wed Jun 29 10:30:29 2005
    
```

Element	Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Background	
C K	Normal	0.09- 0.46	0.5999	0.0002	29 /	15
O K	Normal	0.25- 0.77	68.4438	0.0020	2268 /	16
Na K	Normal	0.81- 1.27	9.1569	0.0008	872 /	18
Mg K	Normal	0.97- 1.57	1.2076	0.0002	167 /	24
Al K	Normal	1.26- 1.78	9.9577	0.0007	1369 /	79
Si K	Normal	1.50- 2.07	8.1480	0.0007	1045 /	80
Ca K	Normal	3.40- 4.30	13.3080	0.0052	829 /	4

Chi_square = 3.4940

Element	Mass%	Atomic%	ZAF	Z	A	F
C	2.021	3.2391	4.2355	1.0372	4.0840	0.9999
O	57.541	69.2225	1.0567	0.9892	1.0682	1.0000
Na	10.097	8.4534	1.3860	1.0441	1.3282	0.9994
Mg	1.535	1.2155	1.5981	0.9830	1.6294	0.9978
Al	9.810	6.9977	1.2382	1.0055	1.2340	0.9980
Si	8.534	5.8480	1.3164	0.9937	1.3251	0.9998
Ca	10.462	5.0238	0.9881	1.0029	0.9851	1.0001

Total 100.000 100.0000
Normalization factor = 0.7956

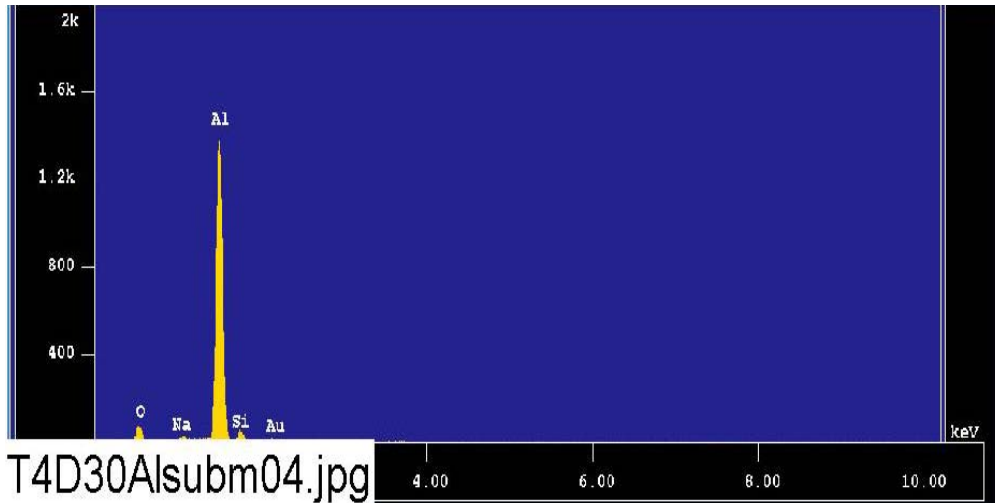


Figure F1-10. EDS counting spectrum for the flat coupon surface (EDS4) shown in Figure F1-8. (T4D30Alsubm04.jpg)

The results from the chemical composition analysis for T4D30A1subm04.jpg are given in Table F1-4.

Table F1-4. Chemical Compositions for T4D30A1subm04.jpg, Figure F1-10

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```

Group      : NRC
Sample     : T4D30 ID# : 4
Comment    : Surface of submerged Al
Condition  : Full Scale : 20KeV(10eV/ch,2Kch)
             Live Time  : 60.000 sec   Aperture #   : 2
             Acc. Volt  : 15.0 KV      Probe Current : 1.068E-09 A
             Stage Point : X=74.653 Y=62.211 Z=10.786
             Acq. Date   : Wed Jun 29 10:41:51 2005
    
```

Element	Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	23.8625	0.0013	792 / 7
Na K	Normal	0.81- 1.27	1.5073	0.0005	144 / 13
Al K	Normal	1.26- 1.78	98.1067	0.0019	13504 / 30
Si K	Normal	1.50- 2.07	2.4247	0.0004	311 / 724

 Chi_square = 2.7984

Element	Mass%	Atomic%	ZAF	Z	A	F
O	20.718	30.5721	1.1041	0.9815	1.1250	0.9999
Na	1.176	1.2077	0.9922	1.0354	0.9648	0.9933
Al	74.561	65.2404	0.9665	0.9967	0.9702	0.9995
Si	3.545	2.9798	1.8592	0.9847	1.8881	1.0000

 Total 100.000 100.0000
 Normalization factor = 0.7863

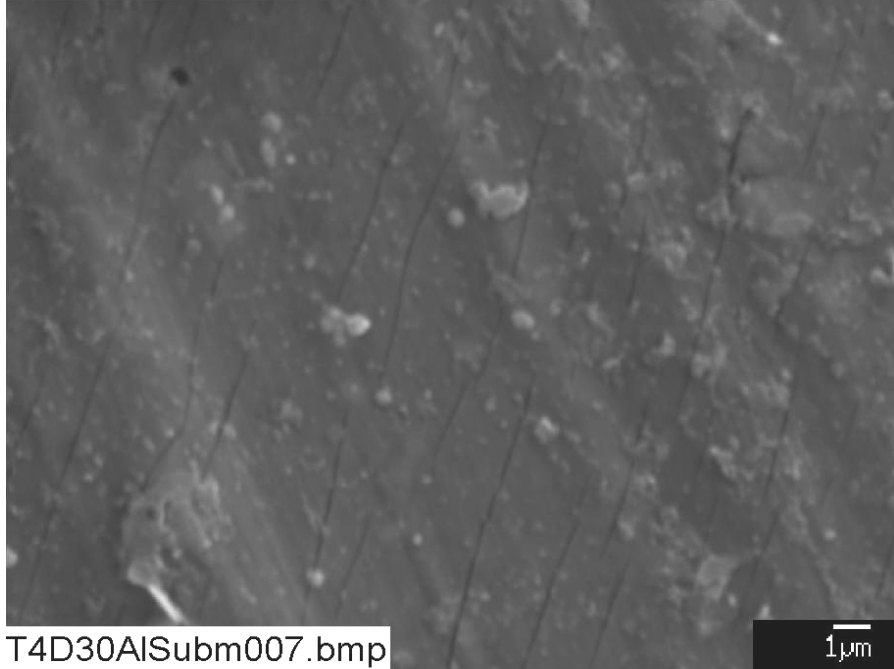


Figure F1-11. SEM image magnified 5000 times for a Test #4, Day-30 submerged aluminum coupon sample. (T4D30AlSubm007.bmp)