

Appendix H

SEM/EDS and ESEM/EDS Data for Test #3 Day-30 Cal-sil

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ICET Test #3 is the first ICET Test that incorporates calcium silicate (cal-sil) to represent insulation materials in a power plant along with fiberglass. This appendix presents the ESEM/SEM/EDS and XRD/XRF results of different cal-sil samples, including unused raw and unused baked cal-sil samples. The Test #3 Day-30 raw cal-sil samples were submerged in the birdcage, while the Test #3 Day-30 baked cal-sil sample were submerged in the high-flow zone.

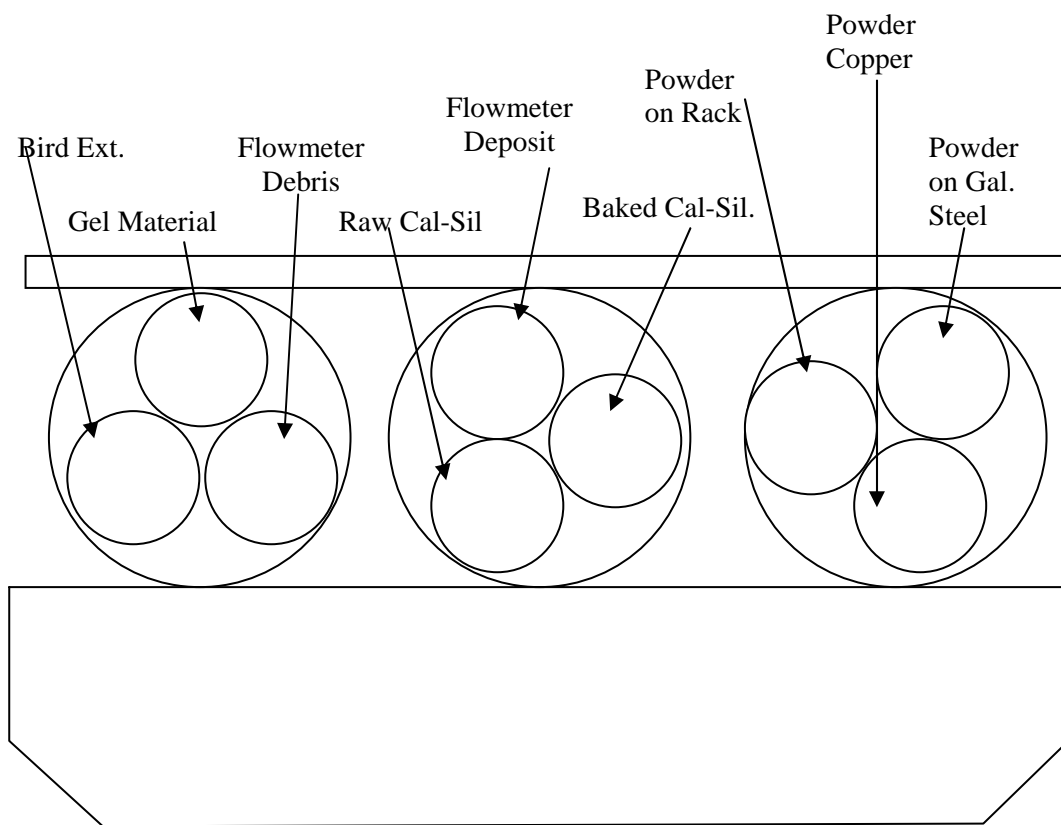
The submerged cal-sil samples were collected on the date Test #3 was shut down (May 5, 2005). For the SEM examination, the samples were dried in air before being coated with Au/Pd. EDS results provide a semi-quantitative elemental analysis of the sample compositions.

In addition, 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). Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

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Transcribed Laboratory Log

Laboratory session from May 9, 2005.
 Test #3 Day-30 Cal-sil



Raw Cal-Sil

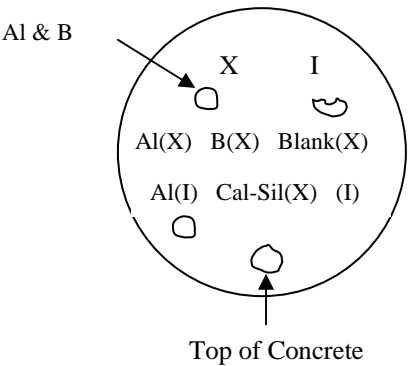
Image:	T3~RawCal-Sil007	100 ×	Figure H-1
	T3~RawCal-Sil008	1000 ×	Figure H-2
EDS:	T3Prtcle04	On particles of image 008	Figure H-
	T3fiber05	On fiber of image 008	Figure H-
	EDS06	Whole image of image 008	Figure H-
Image:	T3~RawCal-Sil009	100 ×	Figure H-

Baked Cal-Sil

Image:	T3~BakedCal-Sil010	200 ×	Figure H-
EDS:	EDS07	EDS of image 010	Figure H-
Image:	T3~BakedCal-Sil011	1000 ×	Figure H-

Transcribed Laboratory Log

Laboratory session from May 10, 2005.
Test #3 Day-30 ESEM Birdcage.



Cal-Sil Exterior in Birdcage

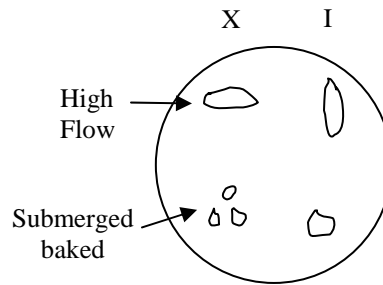
Image:	t3calx21	500 ×	Figure H-
	T3Calx22	500 ×	Figure H-
EDS:	t3calx23	EDS on T3Calx22	Figure H-

Cal-Sil Interior in Birdcage

Image:	T3CalI24	100 ×	Figure H-
	T3CalI25	500 ×	Figure H-
EDS:	T3cali26	EDS on T3CalI25	Figure H-
Image:	t3cali27	500 ×	Figure H-

Transcribed Laboratory Log

Laboratory session from May 11, 2005.
Test #3 Day-30 Baked Cal-sil



Submerged Baked Cal-Sil Exterior

Image:	T3bcal39	100 ×		Figure H-
	t3bcal40	1000 ×	Particles	Figure H-
EDS	t3bcal41		EDS on white particles shown in t3bcal40	Figure H-
	t3bcal42		EDS on dark particles shown in t3bcal40	Figure H-
Image:	T3Bcal43	100 ×		Figure H-
	t3bcal44	1000 ×		Figure H-3
	t3bcal45	1000 ×		Figure H-
EDS:	t3bcal46		EDS on whole screen of image 45	Figure H-

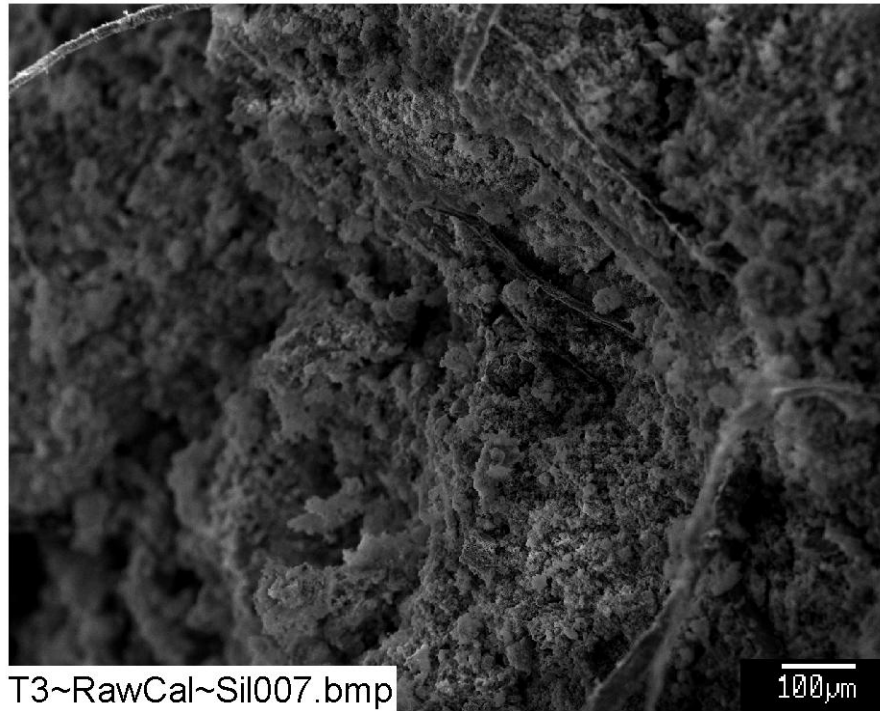


Figure H-1: SEM image magnified 100 times for a raw cal-sil sample. (T3~RawCal_Sil007)

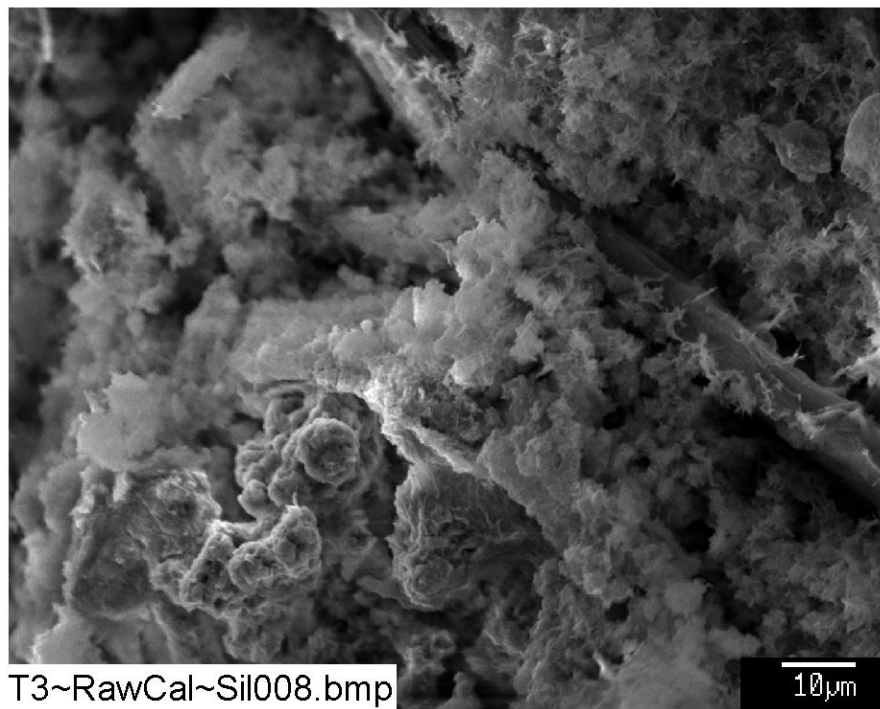
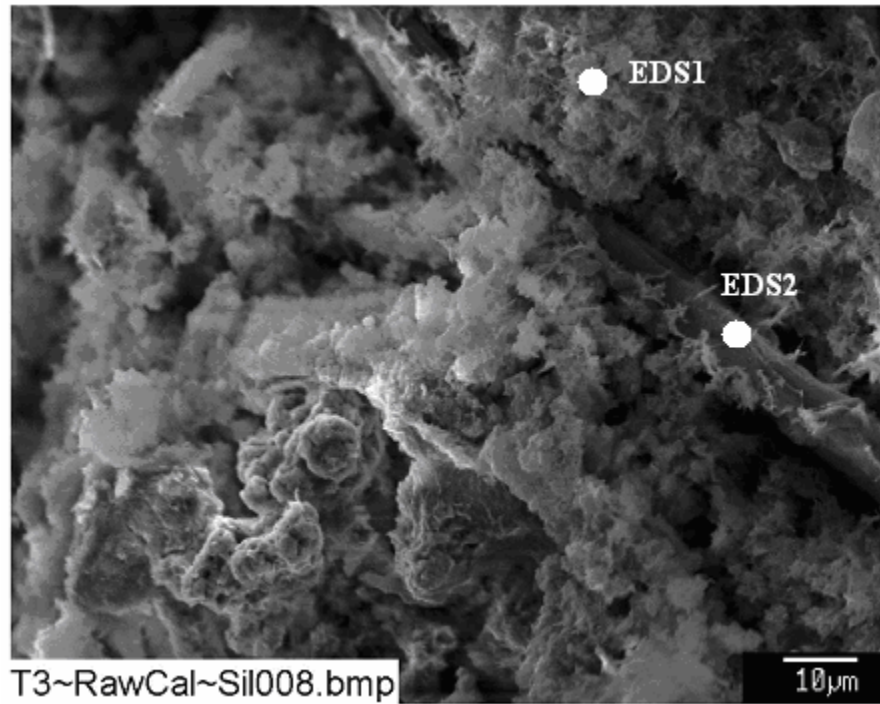
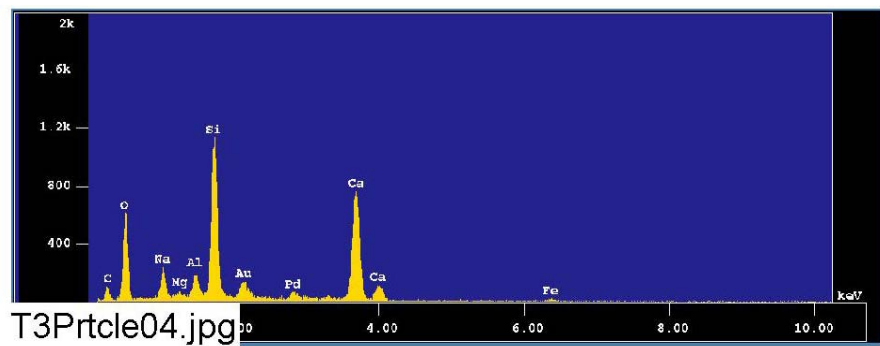


Figure H-2: SEM image magnified 1000 times for a raw cal-sil sample. (T3~RawCal_Sil008)



**Figure H-3: Annotated SEM image magnified 1000 times for a raw cal-sil sample.
(T3~RawCal_Sil008)**



**Figure H-4: EDS counting spectrum for the particles (EDS1) shown in Figure H-23.
(T3Prtcle04)**

The results from the chemical composition analysis for T3Prtcle04 are given in Table H-1.

Table H-1. Chemical Compositions for T3Prtcle04, Figure H-4.

May 9 2005

Group : NRC
Sample : T3D30 ID# : 4
Comment : Raw Cal-Sil Particle
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.604E-09 A
Stage Point : X=58.349 Y=61.668 Z=12.516
Acq. Date : Mon May 9 14:05:35 2005

Element	Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	0.9637	0.0005	537 / 119
O K	Normal	0.25- 0.77	10.7009	0.0031	4017 / 65
Na K	Normal	0.81- 1.27	1.1322	0.0012	1221 / 43
Mg K	Normal	0.97- 1.57	0.1062	0.0003	166 / 80
Al K	Normal	1.19- 1.83	0.9046	0.0004	1409 / 80
Si K	Normal	1.50- 2.05	6.6718	0.0010	9692 / 107
Ca K	Normal	3.39- 4.30	13.6411	0.0036	9631 / 25
Pd L	Normal	2.22- 3.81	1.1981	0.0013	878 / 68
Fe K	Normal	6.00- 7.44	0.8951	0.0012	257 / 18

Chi_square = 21.7880

Element	Mass%	Atomic%	ZAF	Z	A	F
C	7.204	13.1026	3.7872	1.0155	3.7297	0.9999
O	38.993	53.2408	1.8460	0.9683	1.9064	1.0000
Na	3.324	3.1581	1.4872	1.0216	1.4562	0.9997
Mg	0.323	0.2898	1.5385	0.9616	1.6029	0.9982
Al	2.373	1.9210	1.3288	0.9940	1.3428	0.9956
Si	15.630	12.1570	1.1868	0.9716	1.2226	0.9991
Ca	26.877	14.6492	0.9982	0.9904	1.0081	0.9997
Pd	3.135	0.6437	1.3257	1.3709	0.9776	0.9891
Fe	2.142	0.8379	1.2123	1.1945	1.0148	1.0000

Total 100.000 100.0000
Normalization factor = 1.9740

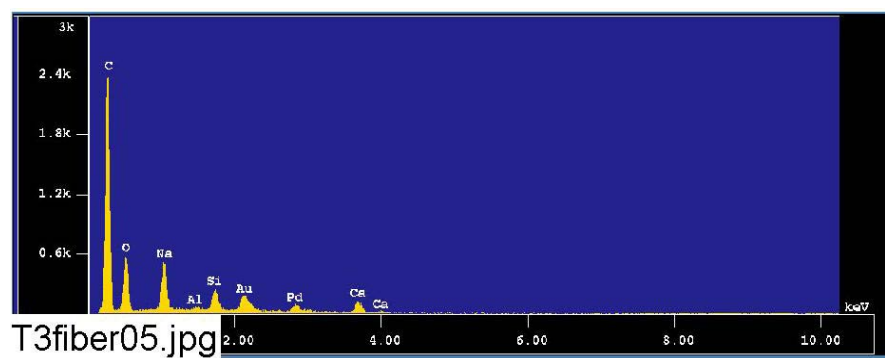


Figure H-5: EDS counting spectrum for the fiber (EDS2) shown in Figure H-23. (T3fiber05)

The results from the chemical composition analysis for T3fiber05 are given in Table H-2.

Table H-2. Chemical Compositions for T3fiber05, Figure H-5.

May 9 2005

Group : NRC
Sample : T3D30 ID# : 5
Comment : Raw Cal-Sil Fiber
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.603E-09 A
Stage Point : X=58.273 Y=61.617 Z=12.516
Acq. Date : Mon May 9 13:56:08 2005

Element	Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	33.8965	0.0016	18866 / 99
O K	Normal	0.25- 0.77	10.3921	0.0032	3899 / 1192
Na K	Normal	0.81- 1.27	3.1096	0.0017	3352 / 52
Al K	Normal	1.19- 1.83	0.1520	0.0003	237 / 64
Si K	Normal	1.50- 2.05	1.1022	0.0005	1600 / 74
Ca K	Normal	3.39- 4.30	2.0189	0.0016	1425 / 17

Chi_square = 50.8203

Element	Mass%	Atomic%	ZAF	Z	A	F
C	65.848	74.0755	1.6099	1.0143	1.5873	1.0000
O	25.742	21.7397	2.0528	0.9679	2.1208	1.0000
Na	4.419	2.5969	1.1775	1.0228	1.1510	1.0002
Al	0.219	0.1098	1.1954	0.9966	1.2002	0.9994
Si	1.434	0.6899	1.0783	0.9750	1.1061	0.9999
Ca	2.338	0.7882	0.9598	0.9997	0.9601	1.0000

Total 100.000 100.0000
Normalization factor = 1.2067

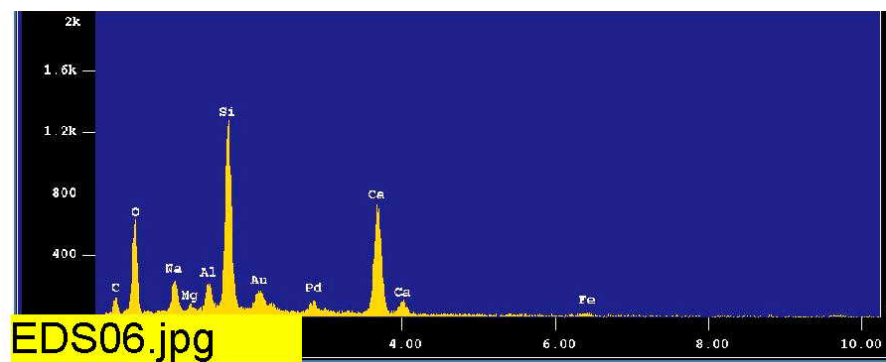


Figure H-6: EDS counting spectrum for the whole image shown in Figure H-23. (EDS06)

The results from the chemical composition analysis for EDS06 are given in Table H-3.

Table H-3. Chemical Compositions for EDS06, Figure H-6.

May 9 2005

Group : NRC
Sample : T3D30 ID# : 6
Comment : Raw Cal-Sil
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.606E-09 A
Stage Point : X=57.876 Y=61.138 Z=12.516
Acq. Date : Mon May 9 14:28:12 2005

Element	Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	1.1389	0.0005	635 / 112
O K	Normal	0.25- 0.77	11.5668	0.0033	4348 / 73
Na K	Normal	0.81- 1.27	1.5352	0.0013	1658 / 56
Mg K	Normal	0.97- 1.57	0.1356	0.0003	213 / 79
Al K	Normal	1.19- 1.83	1.1066	0.0005	1726 / 98
Si K	Normal	1.50- 2.05	7.1883	0.0010	10455 / 146
Ca K	Normal	3.39- 4.30	12.0212	0.0034	8498 / 24
Fe K	Normal	6.00- 7.44	0.9544	0.0012	274 / 11

Chi_square = 26.8019

Element	Mass%	Atomic%	ZAF	Z	A	F
C	9.300	16.0597	4.1007	1.0233	4.0075	0.9999
O	39.825	51.6305	1.7291	0.9759	1.7719	1.0000
Na	4.382	3.9538	1.4336	1.0297	1.3927	0.9996
Mg	0.411	0.3503	1.5204	0.9694	1.5717	0.9979
Al	2.909	2.2365	1.3203	1.0021	1.3240	0.9952
Si	17.041	12.5848	1.1905	0.9797	1.2160	0.9994
Ca	23.814	12.3235	0.9948	0.9993	0.9959	0.9996
Fe	2.318	0.8608	1.2196	1.2068	1.0106	1.0000

Total 100.000 100.0000

Normalization factor = 1.9913



Figure H-7: SEM image magnified 100 times for a raw cal-sil sample. (T3~RawCal_Sil009)

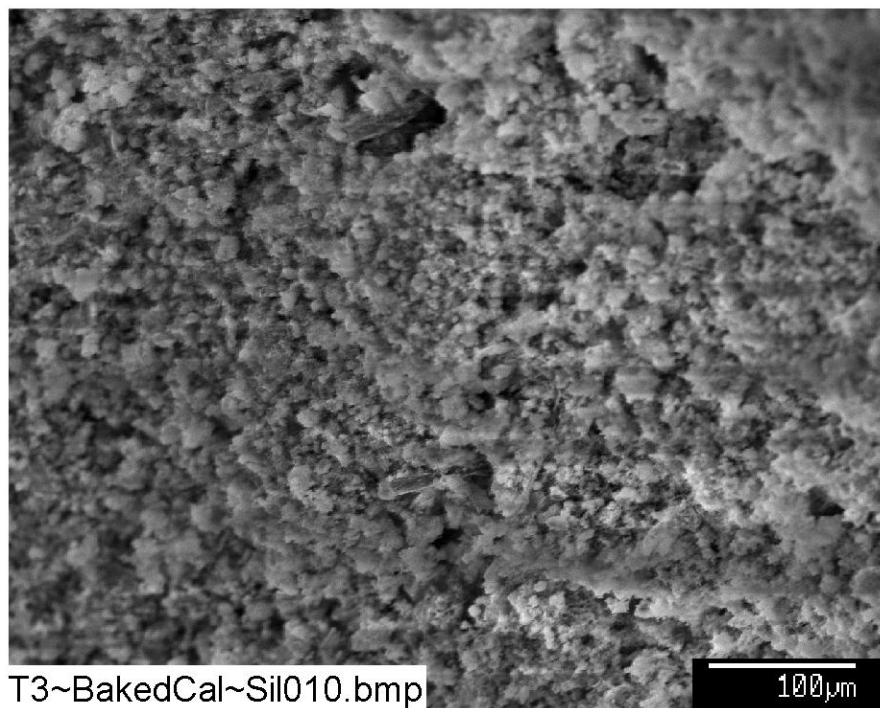


Figure H-8: SEM image magnified 200 times for a baked cal-sil sample (T3~BakedCal~Sil010)

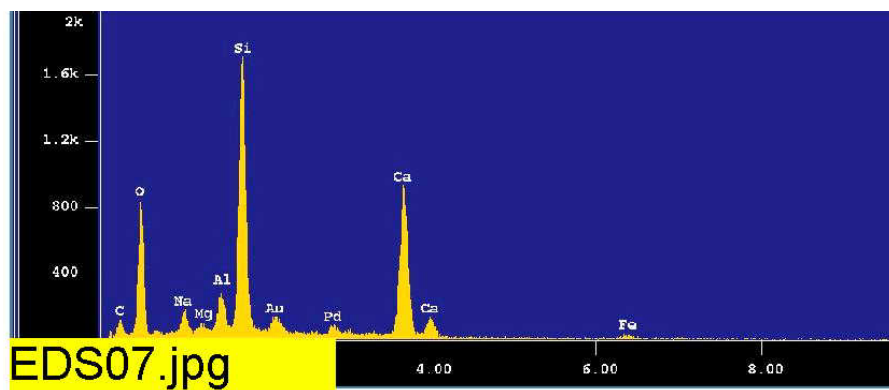


Figure H-9: EDS counting spectrum for the baked cal-sil sample (whole image) shown in Figure H-. (EDS07)

The results from the chemical composition analysis for EDS07 are given in Table H-4.

Table H-4. Chemical Compositions for EDS07, Figure H-9.

May 9 2005

Group : NRC
Sample : T3D30 ID# : 7
Comment : Baked Cal-Sil
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.604E-09 A
Stage Point : X=49.364 Y=57.751 Z=12.516
Acq. Date : Mon May 9 14:20:15 2005

Element	Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	0.9259	0.0006	516 / 161
O K	Normal	0.25- 0.77	14.9326	0.0037	5606 / 77
Na K	Normal	0.81- 1.27	1.0025	0.0012	1081 / 65
Mg K	Normal	0.97- 1.57	0.1609	0.0003	252 / 79
Al K	Normal	1.19- 1.83	1.4816	0.0005	2308 / 124
Si K	Normal	1.50- 2.05	9.5017	0.0012	13802 / 172
Ca K	Normal	3.39- 4.30	15.8398	0.0038	11184 / 30
Fe K	Normal	6.00- 7.44	0.7928	0.0013	227 / 10

Chi_square = 33.5587

Element	Mass%	Atomic%	ZAF	Z	A	F
C	6.304	11.1172	4.1881	1.0251	4.0858	0.9999
O	42.250	55.9339	1.7404	0.9776	1.7803	1.0000
Na	2.361	2.1752	1.4487	1.0315	1.4051	0.9996
Mg	0.391	0.3407	1.4950	0.9710	1.5432	0.9977
Al	3.143	2.4673	1.3049	1.0037	1.3069	0.9947
Si	18.294	13.7966	1.1843	0.9812	1.2077	0.9994
Ca	25.681	13.5714	0.9973	1.0007	0.9968	0.9998
Fe	1.576	0.5977	1.2226	1.2082	1.0119	1.0000

Total 100.000 100.0000

Normalization factor = 1.6257

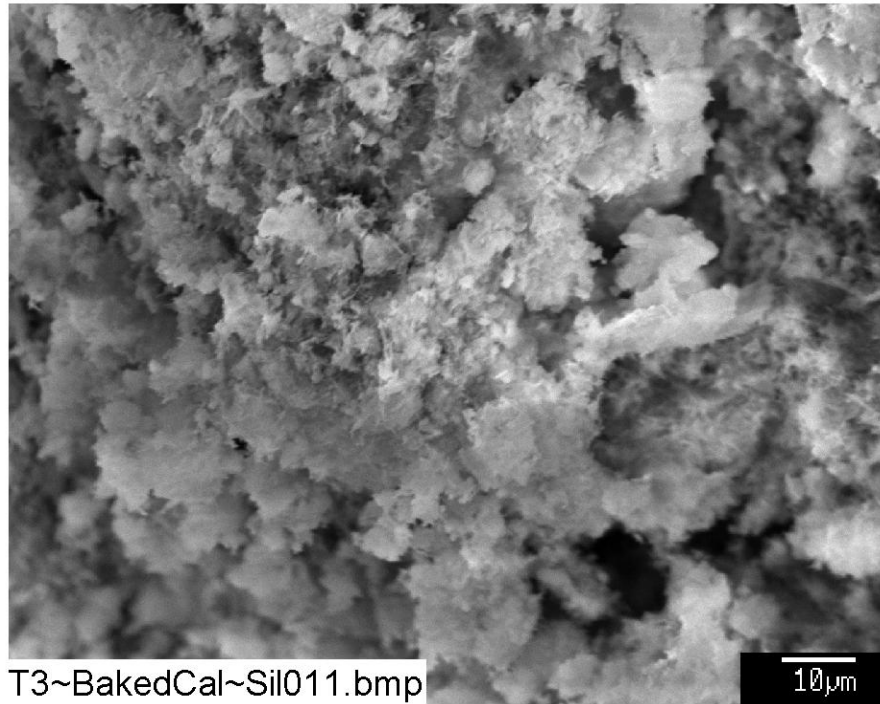


Figure H-10: SEM image magnified 1000 times for a baked cal-sil sample. (T3~BakedCal~Sil011)

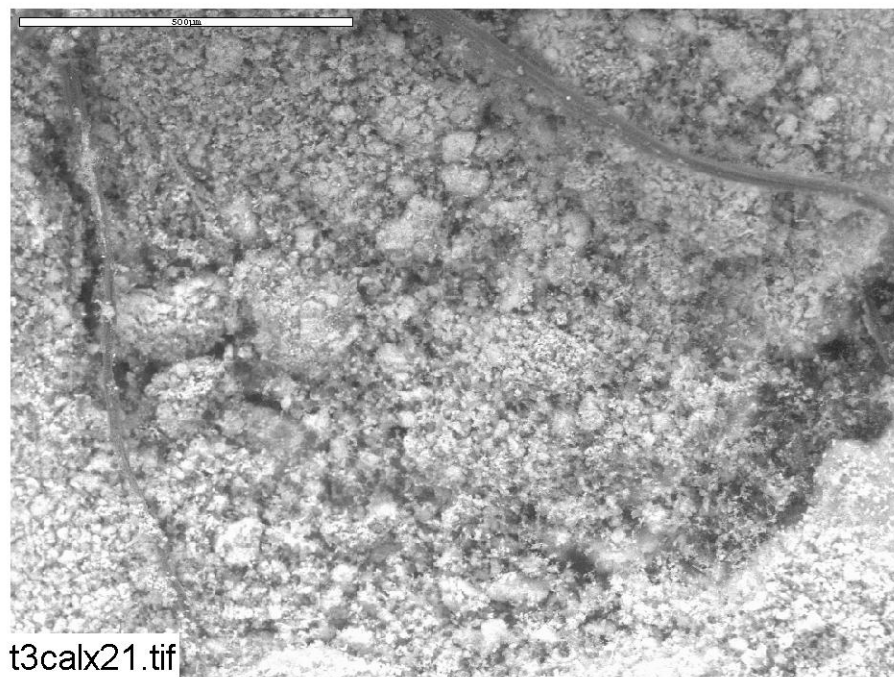


Figure H-11: ESEM image magnified 100 times for the exterior of a Test#3 Day-30 raw cal-sil sample submerged in the birdcage. (t3calx21)

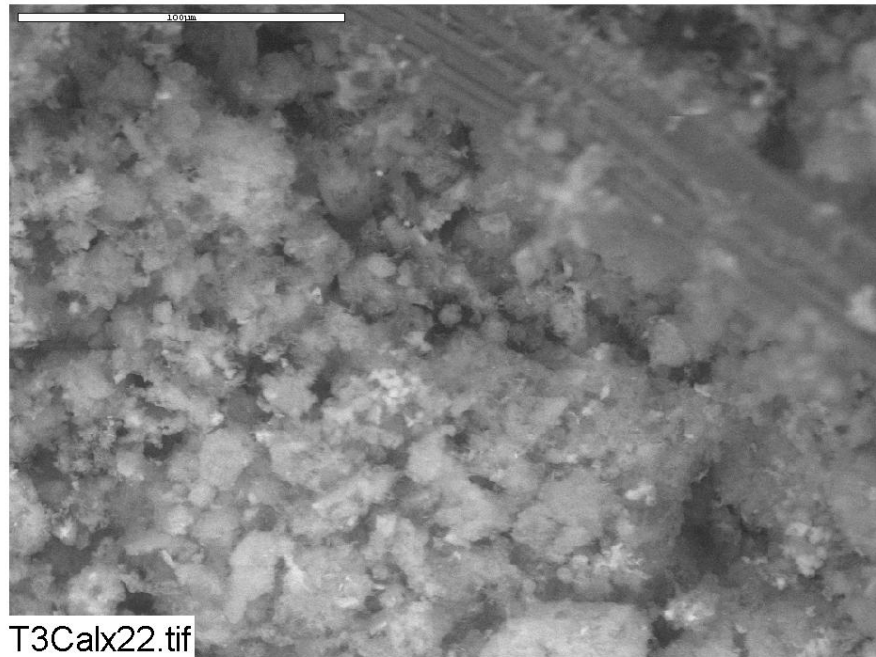


Figure H-12: ESEM image magnified 500 times for the exterior of a Test#3 Day-30 raw cal-sil sample submerged in the birdcage. (T3Calx22)

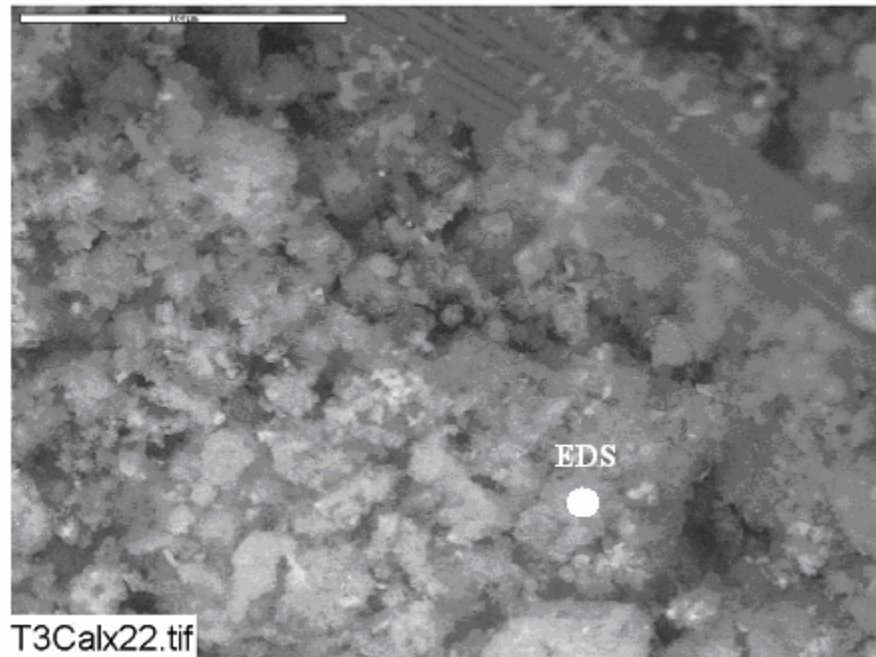


Figure H-13: Annotated ESEM image magnified 500 times for the exterior of a Test#3 Day-30 raw cal-sil sample submerged in the birdcage. (T3Calx22)

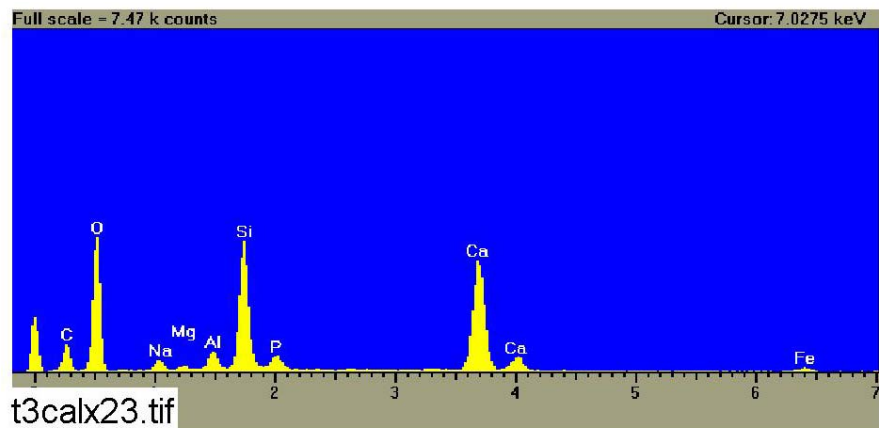


Figure H-14: EDS counting spectrum for the particles shown in Figure H-3. (t3calx23)

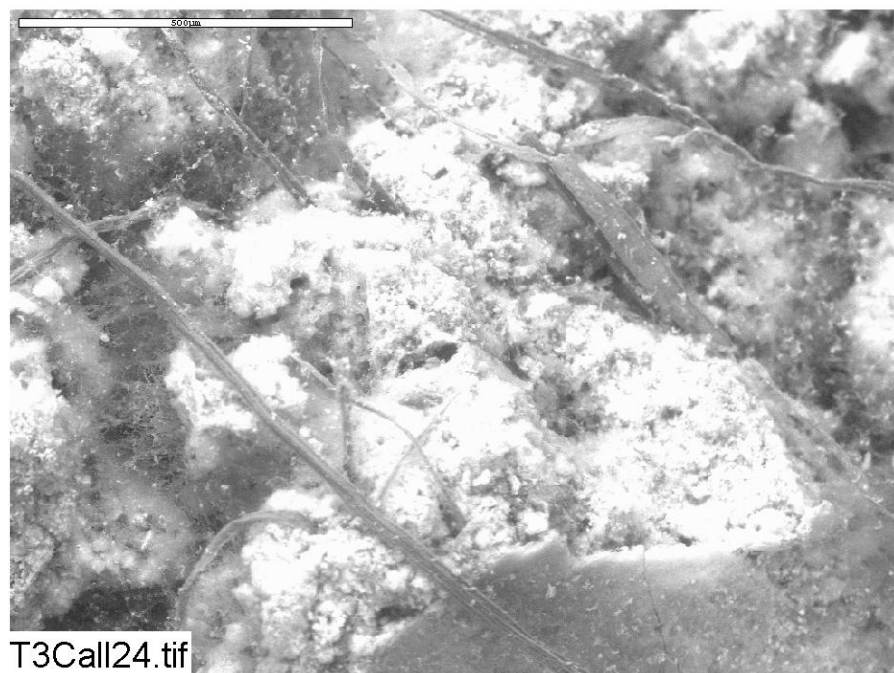


Figure H-15: ESEM image magnified 100 times for the interior of a Test#3 Day-30 raw cal-sil sample submerged in the birdcage. (T3CalI24)

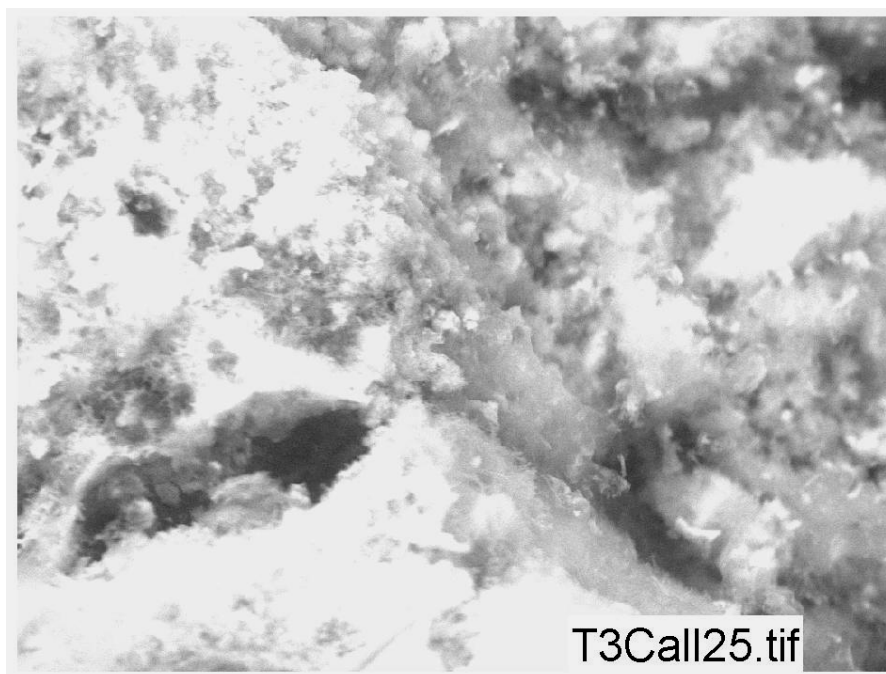


Figure H-16: ESEM image magnified 500 times for the interior of a Test#3 Day-30 raw cal-sil sample submerged in the birdcage. (T3Call25)

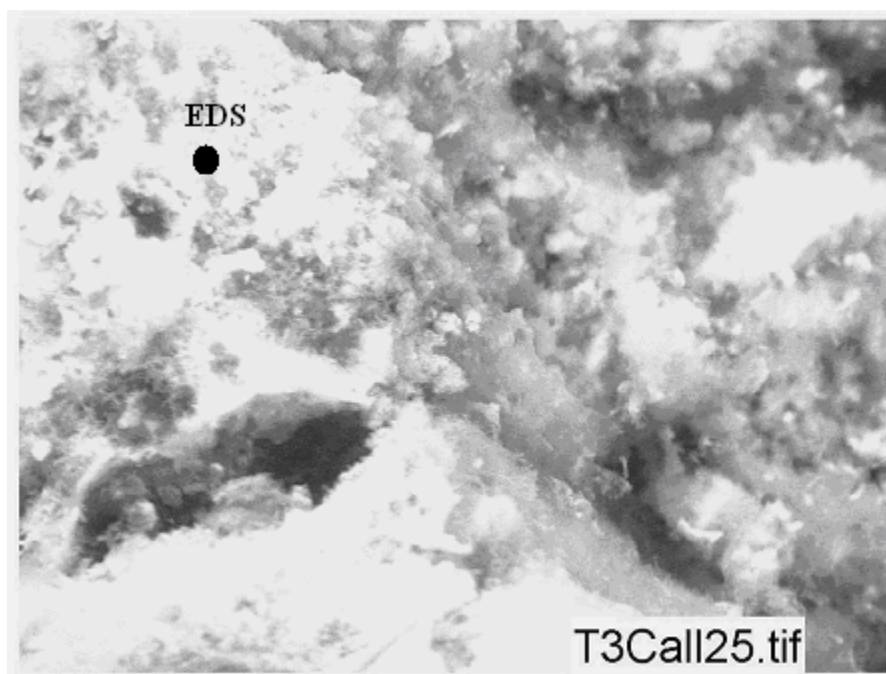


Figure H-17: Annotated ESEM image magnified 500 times for the interior of a Test#3 Day-30 raw cal-sil sample submerged in the birdcage. (T3Call25)

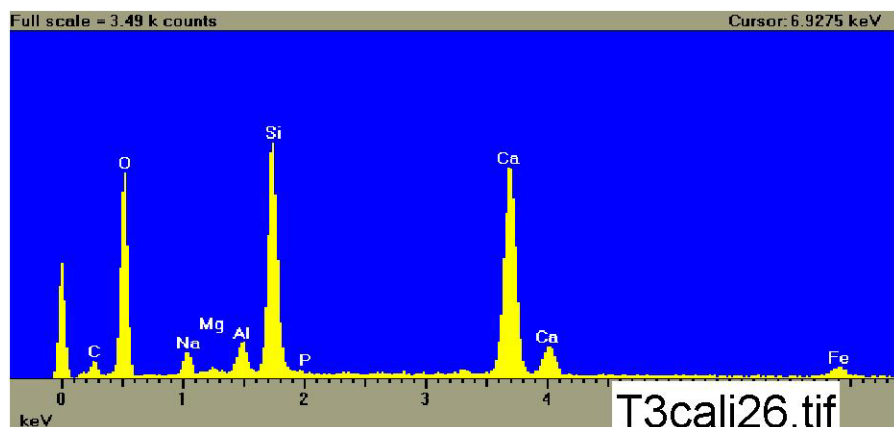


Figure H-18: EDS counting spectrum for the particles shown in Figure H-7. (T3cali26)



Figure H-19: ESEM image magnified 500 times for the interior of a Test#3 Day-30 raw cal-sil sample submerged in the birdcage. (T3CalI27)

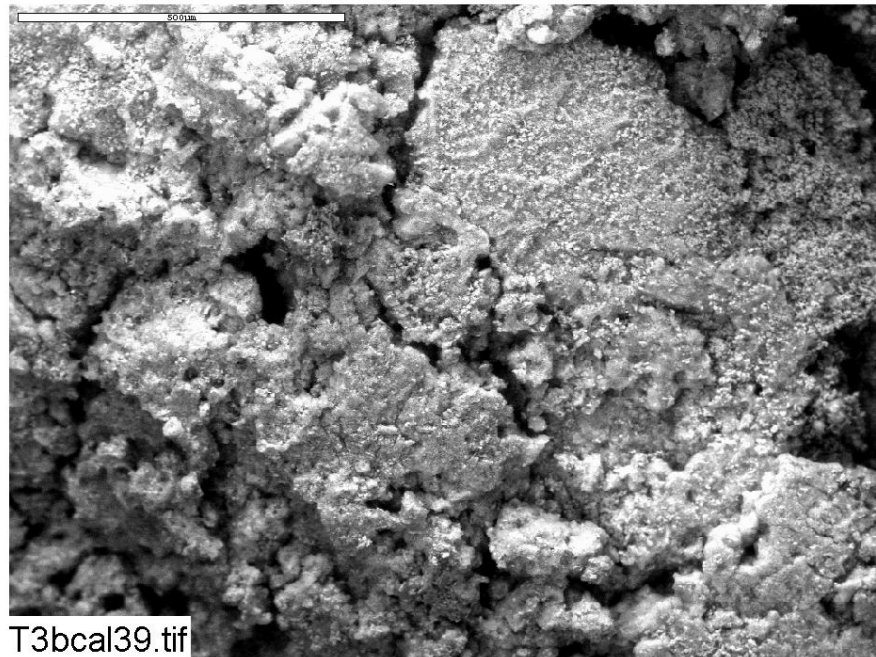


Figure H-20: ESEM image magnified 100 times for the exterior of a Test#3 Day-30 submerged high-flow baked cal-sil sample. (T3bcal39)

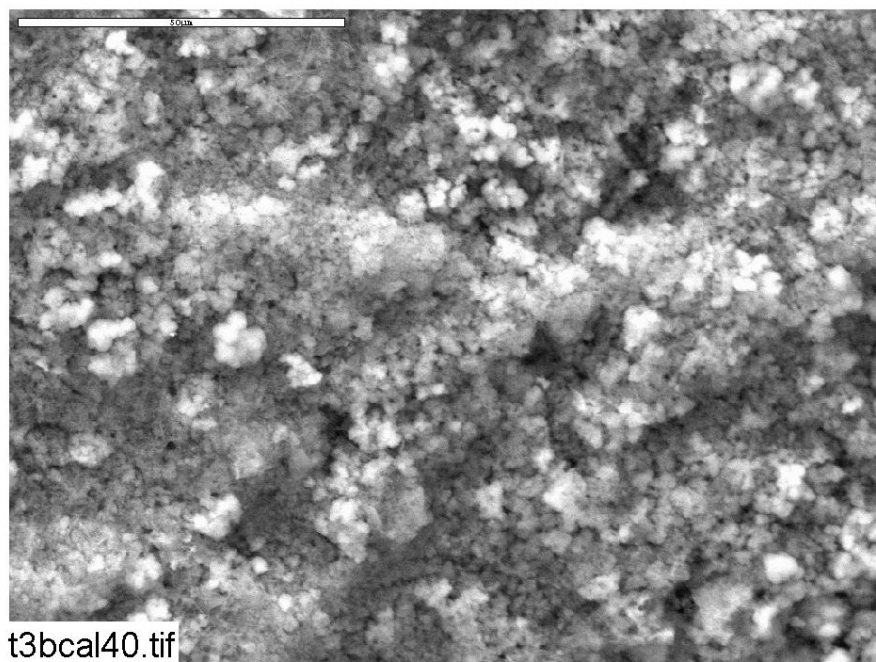


Figure H-21: ESEM image magnified 1000 times for the exterior of a Test#3 Day-30 submerged high-flow baked cal-sil sample. (t3bcal40)

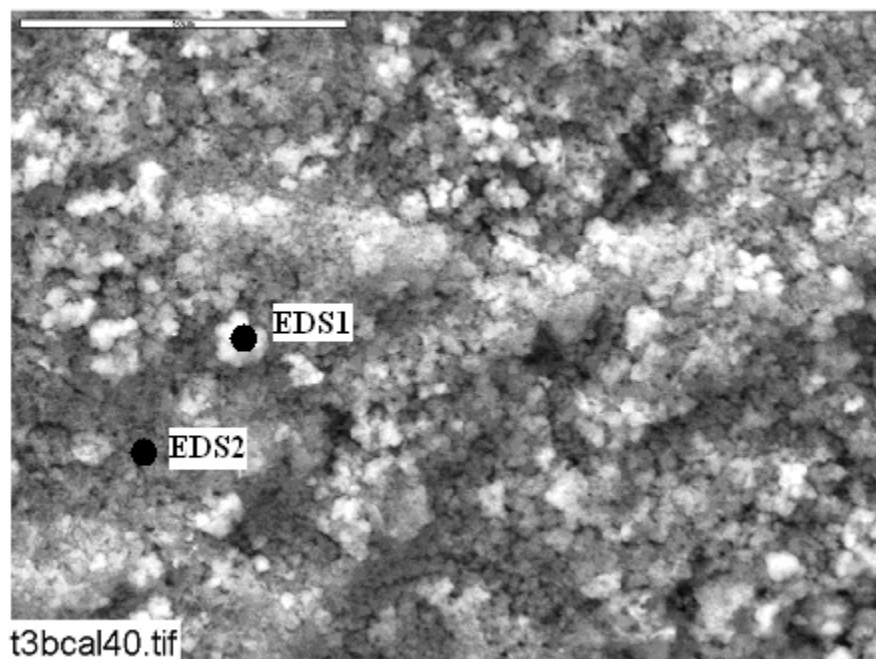


Figure H-22: Annotated ESEM image magnified 1000 times for the exterior of a Test#3 Day-30 submerged high-flow baked cal-sil sample. (t3bcal40)

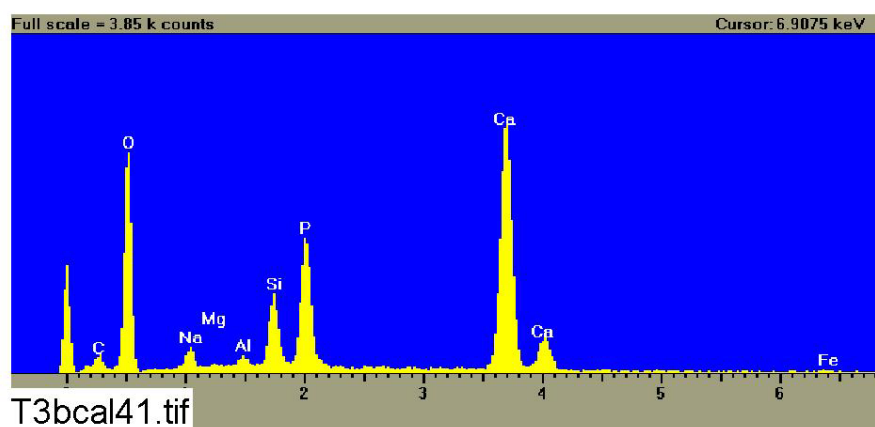


Figure H-23: EDS counting spectrum for the light particles (EDS1) shown in Figure H-22. (T3bcal41)

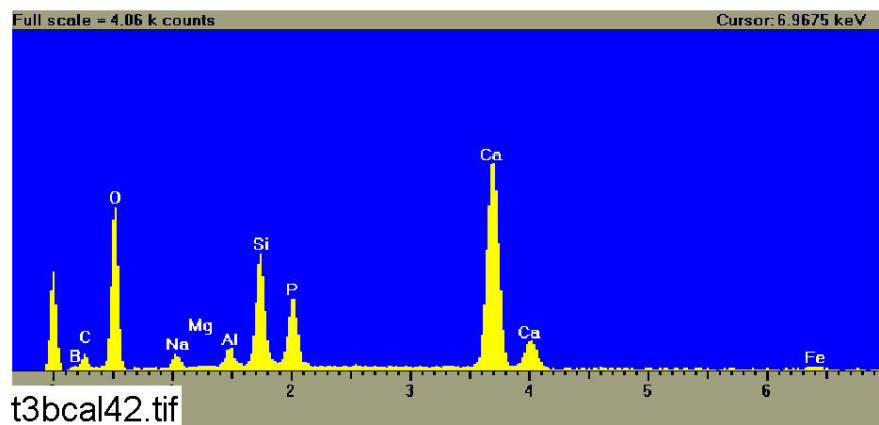


Figure H-24: EDS counting spectrum for the dark particles (EDS2) shown in Figure H-22. (t3bcal42)

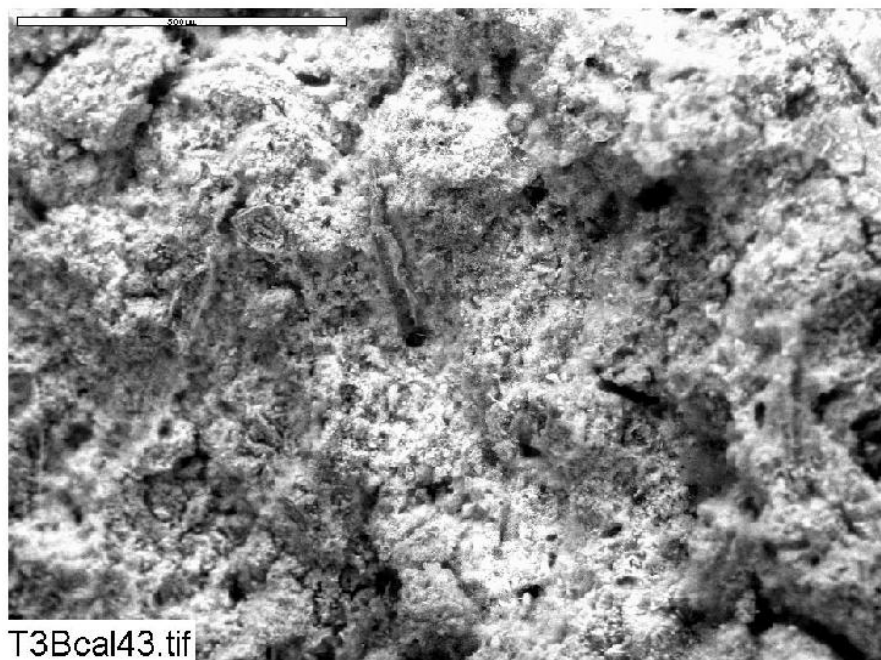


Figure H-25: ESEM image magnified 100 times for the interior of a Test#3 Day-30 submerged high-flow baked cal-sil sample. (T3Bcal43)

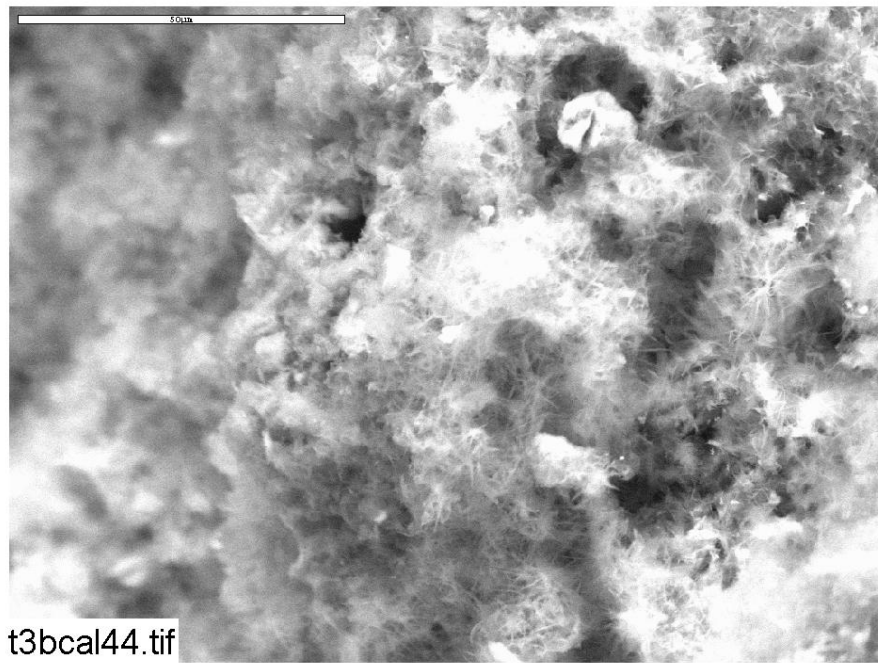


Figure H-36: ESEM image magnified 1000 times for the interior of a Test#3 Day-30 submerged high-flow baked cal-sil sample. (t3bcal44)

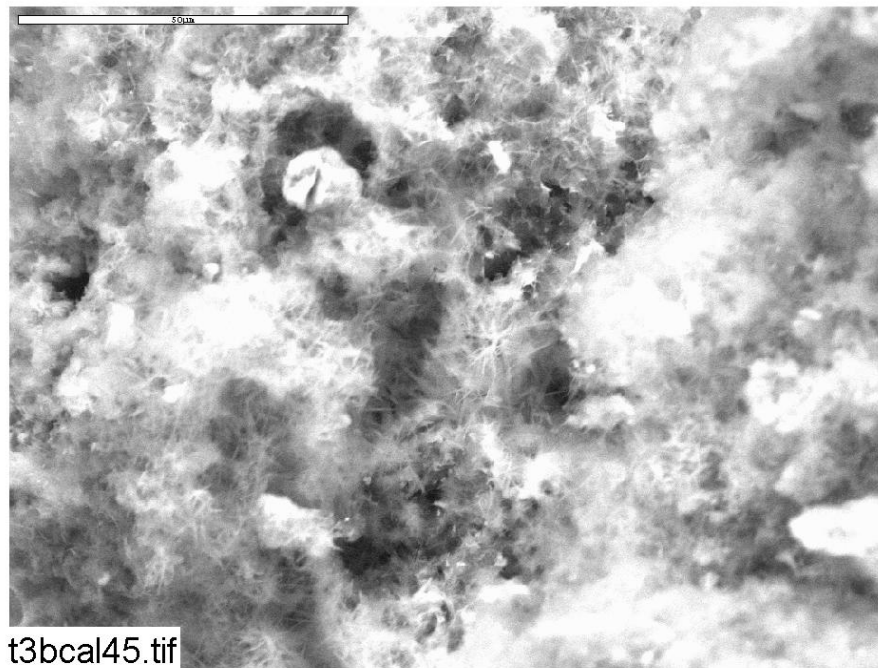


Figure H-27: ESEM image magnified 1000 times for the interior of a Test#3 Day-30 submerged high-flow baked cal-sil sample. (t3bcal45)

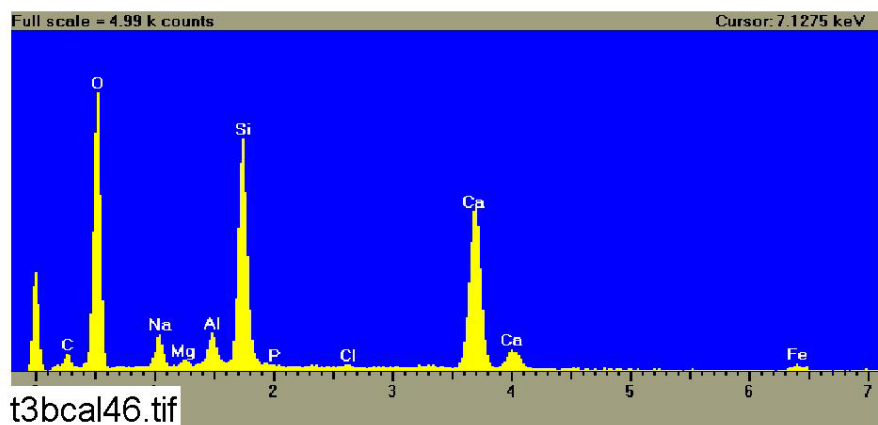


Figure H-28: EDS counting spectrum for the whole image shown in Figure H-7. (t3bcal46)

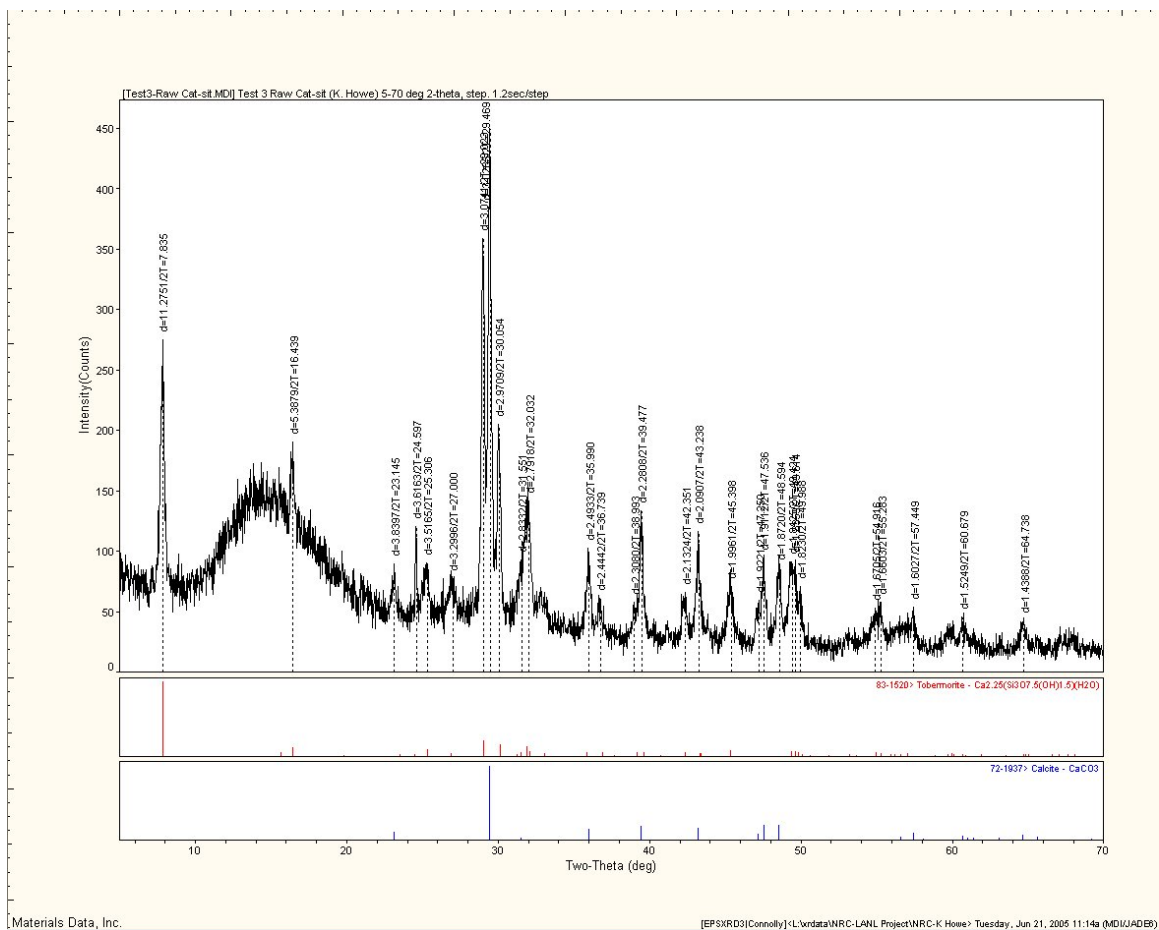


Figure H-29. XRD Results for the Unused Raw Cal-sil Sample

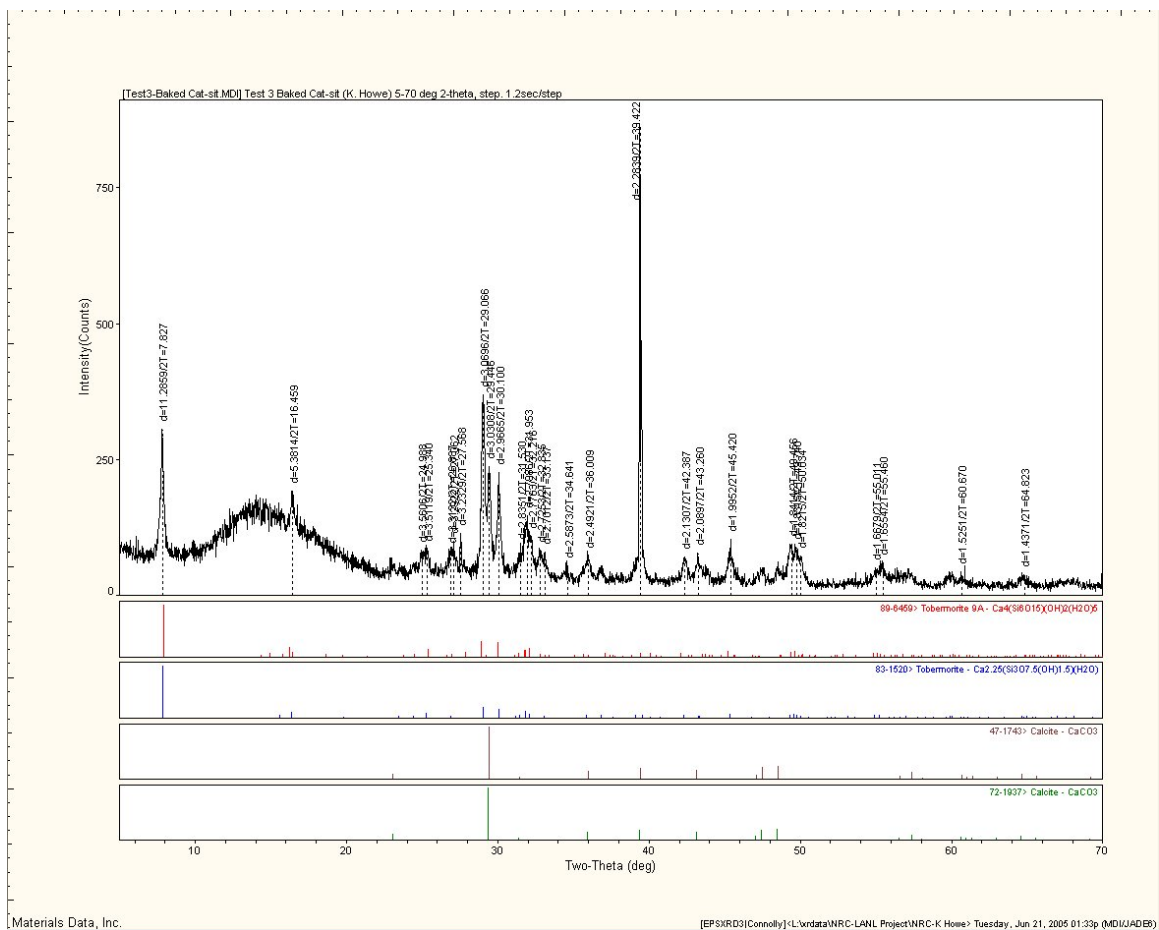


Figure H-30. XRD Results for the Unused Baked Cal-sil Sample

Table H-5. Dry Mass Composition of Unused Cal Sil Samples by XRF Analysis

Sample ID	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	FeO	MnO	MgO	CaO	Na ₂ O	K ₂ O	H ₂ O(-CO ₂)	H ₂ O(+CO ₂)	P ₂ O ₅	Total	H ₂ O(+CO ₂)/DF (10) & Cover. To %
Baked Cal Sil	38.34	0.18	5.02	2.54	0.00	0.06	0.79	34.76	2.32	0.42	0.35	18.75	0.15	103.67	1.0191
Raw Cal Sil	33.87	0.36	4.27	2.07	0.00	0.05	1.35	34.66	2.27	0.35	0.56	1.59	0.12	81.50	1.0016