Results of Sorption/Desorption Experiments with IONSIV IE-911 Crystalline Silicotianate

by

W. R. Wilmarth

Westinghouse Savannah River Company Savannah River Site Aiken, South Carolina 29808

J. T. Mills

V. B. Dukes

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Results of Sorption/Desorption Experiments with IONSIV⁰IE-911 Crystalline Silicotitanate

W. R. Wilmarth, J. T. Mills, and V. H. Dukes Waste Processing Technology Section Savannah River Technology Center

Summary

This report describes the results of cesium loading studies with IONSIV[®] IE-911, crystalline silicotitanate sorbent (CST). The experiments investigated the effect of time at elevated temperature. The results from tests conducted at temperatures of 35 and 55 °C indicate the following.

- Pretreating the IONSIV[®] IE-911 with sodium hydroxide lowers the equilibrium distribution coefficient, K_d , from 2323 ± 72 mL/g to 2117 ± 77 mL/g for average Savannah River Site waste simulant starting with a cesium concentration of 18 mg/L. This difference is accounted by the difference in formula weight between the hydrogen and sodium forms of the CST.
- Elevating the temperature to 55 °C for a short duration (1 day) lowered the K_d value measured at 25 °C by 7%. The 7% reduction is larger than the 3.4% variance observed in replicate K_d testing at 25 °C. Raising the temperature to 35 °C showed no effect on the K_d .
- The loss in cesium capacity is magnified when represented as K_d . After a 55 °C temperature excursion, only 0.4% of the initially sorbed cesium did not resorb.
- Data from tests conducted with a temperature excursion (55 °C) of 14 days indicate a detrimental effect (20% reduction) on K_d or 1 2% change in the percentage of cesium initially removed. Again, raising the temperature to 35 °C showed no effect.
- Loss of K_d occurred in waste simulants devoid of added aluminum and silicon.
- Solid state characterization of the crystalline silicotitanate surfaces shows formation of sodium aluminosilicate, or sodalite, and formation correlates with time at elevated temperature. The sodalite deposition, however, does not correlate to loss of cesium K_d.

Introduction

The Salt Disposition Systems Engineering Team identified non-elutable ion exchange as one of three preferred alternatives to replace the In-Tank Precipitation process used to decontaminate soluble radioactive waste at the Savannah River Site (SRS). Crystalline silicotitanate (CST) in the engineered form (UOP, LLC. IONSIV[®] IE-911) serves as the ion exchange medium in this process option. Previous studies examined the performance of the IONSIV[®] IE-911 crystalline silicotitanate for removing cesium with simulated and

actual SRS tank wastes.^{1,2} The results of these tests confirmed critical aspects of the proposed facility design including column sizing.

Moreover, the stability of the IONSIV[®] IE-911 material in SRS simulated wastes has undergone extensive study. Excess materials added during the synthesis of the IONSIV[®] IE-910 precursor leach into the highly caustic salt solutions.³ These materials, silicon and niobium, subsequently precipitate as aluminosilicate and oxides, respectively. Work by Taylor⁴ at Oak Ridge showed an irreversibility of cesium sorption behavior onto CST after exposure at elevated temperature. In these tests, CST that was loaded with cesium, was heated for extended periods of time (60 days). Measurements taken after the material was brought to ambient temperature (~25 °C) showed the cesium did not resorb. Therefore, High Level Waste Engineering requested^{5,6} the Savannah River Technology Center (SRTC) to examine this irreversibility in greater detail.

Experimental

Personnel loaded IONSIV[®] IE-911 with cesium at room temperature in batch tests using simulated waste solutions. Table 1 contains the composition of these waste solutions. They then exposed the loaded sorbent and solution to higher temperatures for a period of time. Finally, researchers cooled the slurry and held at a lower temperature for a period of time. Personnel measured the extent of cesium sorption (at low temperature), desorption (at higher temperature), and re-sorption (at low temperature). Variables in these tests included time (0 to 4 weeks), temperature (25 to 55 °C), and waste composition (average, high hydroxide, and high nitrate at 2 sodium ion concentrations). Tests compared as-received and pretreated IONSIV[®] IE-911. Researchers analyzed selected samples of treated IONSIV[®] IE-911 for changes in composition and structure. Researchers shook all samples in a New Brunswick Scientific Classic Series C-24 Classic Benchtop Incubator Shaker. The experiments used a liquid-to-solid ratio of 0.2 g of CST to 20 mL of solution.

Personnel recorded data gathered during these experiments in accordance with Procedure 4.10⁷ of the L1 Manual, SRTC Procedures Manual. The laboratory notebooks, WSRC-NB-99-00254 and WSRC-NB-2000-00089 provide lifetime storage as a record. Gamma spectra came from an in-house instrument using an approved procedure.⁸

The authors made water weight percent measurements and used the results to correct the weights of the crystalline silicotitanate resin for calculation of equilibrium distribution coefficients, K_d . The drying occurred at 400 °C and Table 2 provides the data. The weight percent water in the as-received IONSIV[®] IE-911 measured 14.25% and 16.22% for the pretreated material.

Component	Average	High OH ⁻	High NO ₃
Na ⁺	5.6	5.6	5.6
K^+	0.015	0.030	0.0041
Cs^+	0.00014	0.00037	0.00014
OH	1.91	3.05	1.17
NO ₃ ⁻	2.14	1.10	2.84
NO_2^-	0.52	0.74	0.37
AlO_2^-	0.31	0.27	0.32
CO_{3}^{2}	0.16	0.17	0.16
SO_4^{2-}	0.15	0.030	0.040
Cľ	0.025	0.010	0.040
F⁻	0.032	0.010	0.050
PO_4^{3-}	0.010	0.008	0.010
$C_2 O_4^{2-}$	0.008	0.008	0.008
$\mathrm{SiO_3}^{2-}$	0.004	0.004	0.004
MoO_4^{2-}	0.0002	0.0002	0.0002

Table 1.	Composition	(M) of SRS	Waste Simulants	
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	As-Rec.	Pretreated
Wt crucible empty Weight crucible W/ CST	13.8928 g 14.8968 g	14.7472 g 15.7647 g
Wt dried crucible % water	14.7546 g 14.22%	15.6015 g 16.32%
wt crucible empty Weight crucible w/ CST	14.7174 g 15.7401 g	14.9694 g 15.9782 g
wt dried crucible % water	15.5973 g 14.28%	15.8171 g 16.11%

Results and Discussion

Table 3 contains a compilation of the initial equilibrium distribution coefficients, K_d , obtained from the various salt solutions. For the average salt solution, the K_d value for the as-received IONSIV[®] IE-911averaged 2323 ± 72 (3.1 %) mL/g. The results from the sodium hydroxide pre-treated samples exhibited a slightly lower average K_d at 2117 ± 77 (3.6 %) mL/g. This lower K_d may originate from the difference in density of the sodium-form of the crystalline silicotitanate. Both of these values compare well with that calculated from the ZAM computer model⁹ for cesium removal in the average salt matrix. Good agreement also results between experiment and the ZAM model for the other two solution matrices. For the high hydroxide and high nitrate solutions the K_d values averaged 2551 ± 136 (5.3%) mL/g and 1800 ± 60 (3.3%) mL/g, respectively. These agree with the 2500 mL/g and 1850 mL/g values of the ZAM predictions for the high hydroxide and high nitrate matrices, respectively.

Table 3.	Initial	Equilibrium	Distribution	Coefficients
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-Received	Pre-Treated
2323	2117
n 72	77
2160	
2551	
n 136	
2500	
1800	
n 60	
1850	
	s-Received 2323 m 72 2160 2551 m 136 2500 m 1800 m 60 1850

One Day High Temperature Treatment

Two experiments examined the influence of a short term (1 day) exposure at two different temperatures (35 and 55 °C). Figure 1 shows the results of the loss of K_d after a 1-day temperature excursion after allowing the cooled system (25 °C) to return to equilibrium (1 week). At 35 °C, the measured K_d with pre-treated CST in average salt

solution after the 1-day temperature excursion measured 2106 mL/g compared to 2090 mL/g as initially measured. This indicates no effect on the cesium sorption capability. Likewise, the change in the percentage of cesium removed by the CST after the high temperature treatment showed similar behavior. The percentage change in the amount of cesium removed from the salt solution after 1 day at elevated temperature was -0.2%.

However, the K_d measured after a 1-day excursion to 55 °C measured 1885 mL/g compared to 2035 mL/g initially. This loss in Cs K_d at 55 °C equals 7.4%. This variance (7.4%) is about twice the variance (3.6%) observed at 25 °C for nine replicate tests with no temperature excursion. Also shown in Figure 1 are the results displayed for the 55 °C excursion in terms of percent cesium removal. After a day at 55 °C, the IONSIV[®] IE-911 shows a 0.4% loss in the percentage of initial cesium removed from solution. In other words, initially 94.6% of the cesium was removed by the CST in this test. After the temperature excursion, 94.2% of the cesium was removed leaving a 0.4% difference. A data compilation for the K_d and cesium removal percentage is given in Appendix 1 and 2, respectively.





Figure 1. Results from 1 Day Temperature Excursion

Fourteen Day High Temperature Treatment

We obtained a significantly larger data set for the 14-day excursion to elevated temperature. Figures 2a and 2b show the loss of K_d and difference in the percentage of cesium removed for the 14-day tests, respectively. The authors conducted tests in five salt solutions including average, average solution diluted to 2.8 M sodium ion concentration, high hydroxide, high nitrate and an average solution that did not contain added aluminum or silicon compounds. In preparing these solutions, personnel added sodium nitrate as needed to maintain the sodium ion concentration at 5.6 M. Moreover, tests occurred at two temperatures (35 and 55 °C) with replicate tests conducted at 35 and 55 °C for the average salt solution and pretreated IONSIV[®] IE-911.

The data for cesium K_d in average waste solution after 14 days at elevated temperature (35 °C) followed by a 1-week low temperature equilibration showed a slight loss on the order of 6 to 8%. Two tests used pretreated IONSIV[®] IE-911 and one used as-received material. The test with the as-received material yielded the smallest loss in K_d (6.2%). The replicate tests with the pretreated CST provide an estimate of the error in this type of test. The loss of K_d in these tests measured 7.4 and 8.7%. Therefore, the data suggests an experimental error in replicate tests as low as 1%.



Figure 2a. 14-Day Loss of Kd after Elevated Temperature Treatment

No loss in K_d occurred at 35 °C for the test conducted in average salt solution without added silicon or aluminum. These data support the theory that the loss in K_d relates to aluminosilicate formation. Wilmarth et al.¹⁰ previously showed that simulants containing typical amounts of silicon and aluminum would produce aluminosilicate even at 35 °C. Loss of K_d proved larger in the 50% diluted average waste and in the high nitrate

simulant. In these tests, the loss of cesium K_d measured 12.8 and 12.6%, respectively. Lastly, in the case of the high hydroxide solution the K_d actually improved with the high



14 Day Elevated Temperature Tests followed by 1 Week at 25 oC

Figure 2b. 14-Day Change in Cs removal after Elevated Temperature Treatment

temperature treatment at 35 °C. If aluminosilicate formation provides the mechanism by which loss of K_d occurs, then the increased solubility of the aluminosilicate with increased hydroxide concentration¹¹ may explain the lack of a temperature effect. Similarly, the low rate of formation of the aluminosilicate¹⁰ may contribute to the lack of temperature effects in this high hydroxide test.

A very similar plot is shown in Figure 2b that shows the change in the percentage of cesium removed from the solution. In every case at 35 °C, the change was very small and was less than 0.5 %. This basically indicates that at 35 °C the cesium that desorbed at the higher temperature re-sorbed at 25 °C. In one test (high hydroxide), a small additional amount of cesium was sorbed by the IONSIV[®] IE-911.

The data from the tests conducted with a 14-day temperature excursion to 55 $^{\circ}$ C (see Figure 2a and 2b) showed a detrimental effect on the cesium K_d after high temperature

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treatment. Likewise, there is a change in the percentage of cesium removed from solution. The loss in K_d ranged from 17.5% in average salt solution with pretreated IONSIV[®] IE-911 to 30.3% with as-received material; whereas, the change in cesium removal is between 1.5 and 2 %. The estimate of error in these K_d tests proves slightly larger than the lower temperature tests and measured 6% based on the replicates of pretreated CST. The deviation proved slightly lower (4.6%) if one expands the basis set to include all of the tests. The improved performance of the IONSIV[®] IE-911 observed in the heat treatments at 35 °C in the high hydroxide and average solutions without silicon and aluminum did not occur at 55 °C.

The loss of K_d (21.2%) and the change in percent cesium removed (1 - 2%) in the tests conducted in the average salt solution without Si and Al indicates that if aluminosilicate precipitates on the surface of the CST the precipitate does not significantly alter the performance at these conditions. Therefore, the authors conducted a second set of tests using a simulant devoid of silicon and aluminum. The initial set of experiments used a simulant prepared by replacing the equivalent sodium concentration derived from the Si and Al components with sodium nitrate. Personnel made no adjustment to the total amount of hydroxide. This protocol increased the free hydroxide level substantially. In the second set of experiments we maintained the sodium level at 5.6 M and the free hydroxide at 1.9 M. The K_d for this simulated waste equaled 1670 mL/g at 25 °C. The results after a three-week excursion to 35 °C showed some resorption of cesium with a measured 1435 mL/g, a 14.3% reduction. The results at 55 °C showed a 19.4% reduction in K_d. Therefore, the effect also occurs in the absence of aluminum and silicon.

Twenty-eight Day High Temperature Treatment

Four tests occurred with a 28-day temperature excursion at two temperatures (35 and 55 °C). Two tests used as-received IONSIV[®] IE-911 and two tests used pretreated IONSIV[®] IE-911. All tests used average waste simulant. Figure 3 contains a bar graph depicting the effect of time at temperature on the loss of K_d for pretreated IONSIV[®] IE-911. At 35 °C, a large variance occurred in the final measurement of K_d after resorption. The K_d measured with pretreated IONSIV[®] IE-911 after a 14-day excursion to 35 °C showed a loss of K_d of about 8%. However, the K_d with the same material measured no loss after a 28-day excursion to 35 °C. Almost identical data resulted from the tests with as-received IONSIV[®] IE-911. In those tests at 35 °C, the loss of K_d after 14 days measured 6.2%; but the loss after 28 days measured only 2.9%. Therefore, the data suggests little influence on the ambient temperature absorption of cesium after raising the temperature to 35 °C.

The results from the 55 °C tests do, however, show a significant loss of K_d as observed in Figure 3. As previously mentioned in this report, an ~ 20% loss of cesium K_d occurred for pretreated IONSIV[®] IE-911 in average waste simulant after a 14-day temperature excursion to 55 °C. The results from the 28-day excursion validate the loss in K_d . The loss equaled 21.7% nearly matching the results from the 14-day test. The detrimental

effect did not worsen at 28 days which disagrees agree with previous results of Taylor, et al. 12

Table 4 contains a comparison of the K_d data to that of the cesium removal data. As with the other data sets, the two response variables (K_d and % Cs removed from solution) agree. The amount of cesium removed by the CST resin in each of these tests is about 95%. The table depicts the change in the percentage of cesium removed after the elevated temperature excursion. At a maximum, there is less than a 2% change in the percentage of cesium removed by the CST. This maximum occurs after 14 days at 55 °C.



Figure 3. Effect of Time and Temperature on K_d for Pretreated IONSIV[®] IE-911 in Average Waste

Solid State Characterization

Personnel used X-ray diffraction and energy dispersive X-ray spectroscopy to characterize the IONSIV[®] IE-911 materials after the tests. The characterization attempted to determine if chemical deposition caused the loss of K_d after exposure to elevated temperature.

	Kd Data		Delta in % Cs Removed			
	Temp (°C)	Temp (°C)	Temp (°C)	Temp (°C)		
Time (d)	35	55	35	55		
1	-0.8	7.4	-0.2	0.4		
14	8.1	21.9	0.4	1.3		
28	-0.6	21.7	0	1.2		

 Table 4. Comparison of Time-Temperature Effects



Figure 4. SEM and EDX Images of CST

Figure 4 shows the energy dispersive X-ray (EDX) spectra and a scanning electron microscopy (SEM) micrograph of the surface of IONSIV[®] IE-911 particle after exposure to elevated temperature. Small (10 – 15 micron) spherical beads appear visible adhering to the surface of the IONSIV[®] IE-911 particle. The EDX spectrum from spot A shows the characteristic spectrum of the CST material. The spectrum obtained from spot B shows peaks from sodium, aluminum and silicon. The spectrum agrees with spectra

taken from the nitrate-salt of sodalite.¹³ This aluminosilicate has a molecular formula of $Na_8Al_6Si_6O_{24}(NO_3)_2-4H_2O$.



Figure 5. X-ray Powder Patterns from CST Samples

Figure 5 shows the X-ray diffraction powder patterns in the angular region of 20 to 30° 20. The first pattern comes from as-received IONSIV® IE-911. The blue vertical lines represent the pattern from reference materials. The second pattern comes from CST material after 1 day at 55 °C and shows two differences from the as-received material. First, the low intensity peak 22° 20 shifts to 22.7° 20 and corresponds with changes from hydrogen form of the material to the sodium form of CST. The second change involves the appearance of a very small reflection 24.2° 20 overlapping with a red vertical line corresponding to a reflection from the nitrate-salt of sodalite. The next two patterns come from similar materials exposed for increasing duration at elevated temperature. This peak from the aluminosilicate increases with longer duration at elevated temperature and appears consistent with known formation kinetics.¹⁰ The last pattern comes from CST exposed to elevated temperature in a simulant devoid of aluminum and silicon. In this case, the CST does not show evidence for aluminosilicate formation as expected. However, this material did show a loss in cesium-loading performance.

Conclusions

The thermal and chemical stability of IONSIV[®] IE-911 proves fundamental to the crystalline silicotitanate flowsheet proposed for treatment of Savannah River Site waste. Adverse effect of chemical leaching or thermal degradation from an operational viewpoint can pose significant risk. Data obtained in initial testing of the thermal stability of CST indicated an irreversible cesium absorption phenomenon. Therefore, this series of tests examined the influence of exposure as a function of time at temperature.

The results of these tests confirmed a high temperature "effect". Sorbent exposed for extended periods (i.e., greater than 14 days) at high temperature (55 °C) did show some loss of cesium removal efficiency (~ 1 - 2%). The tests did not show any effect at lower temperature (35 °C) even following exposures for 28 days. Moreover, the tests conducted with short duration at elevated temperature show only minor effects (i.e., <10% loss in K_d).

Solid state characterization of the CST surfaces exhibited evidence for aluminosilicate formation following exposure to wastes containing both silicon and aluminum. Previously personnel speculated that formation of the aluminosilicate might explain the irreversibility of the cesium desorption. However, tests conducted in waste simulants devoid of aluminum and silicon showed the same loss of cesium K_d after high temperature excursions. Additional work needs to examine the mechanism for this loss of K_d .

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Approvals Authors W. R. Wilmarth, Waste Processing Technology UH Dukes fu

V. H. Dukes, Waste Processing Technology

J. T. Mills, Waste Processing Technology

Reviewer Mauer F. F. Fondeur, Waste Processing Technology

Management

S. D. Fink, Level 4 Manager, Waste Processing Technology

J. T. Carter, Director of Engineering Salt Waste Disposition Facility

MOPHIC

W. L. Tamosaitis, Manager Waste Processing Technology

D. W. Wester, TFA System Lead Pacific Northwest National Laboratory

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Appendix K_d Experimental Data

Test No.	Pretreatment	Salt Sol,n	Desorption Time	Temperature	Initial K_d	High Temp K_d	Final K_d	% Loss Initial	% Loss Avg
1	As-Received	Avg	0	25	2367	***	2278	3.8	2.0
2	As-Received	Avg	14	35	2321	1480	2178	6.2	6.3
3	As-Received	Avg	28	35	2201	1307	2136	2.9	8.1
4	As-Received	Avg	14	55	2380	508	1658	30.3	28.6
5	As-Received	Avg	28	55	2348		1683	28.3	27.6
6	Pretreated	Avg	0	25	2074	***	2251	-8.5	-6.4
7	Pretreated	Avg	1	35	2090	1560	2106	-0.8	0.5
8	Pretreated	Avg	14	35	2105	1184	1949	7.4	7.9
9	Pretreated	Avg	28	35	2140	1240	2153	-0.6	-1.7
10	Pretreated	Avg	14	35	2108	1243	1925	8.7	9.0
11	Pretreated	Avg	1	55	2035	860	1885	7.4	10.9
12	Pretreated	Avg	14	55	2231	576	1647	26.2	22.2
13	Pretreated	Avg	28	55	2241		1754	21.7	17.1
14	Pretreated	Avg	14	55	2024	514	1670	17.5	21.1
15	Pretreated	Avg-Si and Al	0	25	2051	***	***	***	#VALUE!
16	Pretreated	Avg-Si and Al	14	35	2790	1698	2835	-1.6	-22.0
17	Pretreated	Avg-Si and Al	14	55	2235	591	1761	21.2	24.2
18	Pretreated	1/2 Avg	0	25	5075	***	5068	0.1	0.1
19	Pretreated	1/2 Avg	14	35	4882	2902	4255	12.8	16.1
20	Pretreated	1/2 Avg	14	55	5099	1304	3777	25.9	25.6
21	Pretreated	High OH	0	25	2458	***	2910	-18.4	-14.1
22	Pretreated	High OH	14	35	2487	1652	2771	-11.4	-8.6
23	Pretreated	High OH	14	55	2708	840	2206	18.5	13.5
24	Pretreated	High NO3	0	25	1733	***	2037	-17.5	-13.2
25	Pretreated	High NO3	14	35	1818	1017	1590	12.6	11.7
26	Pretreated	High NO3	14	55	1848	416	1449	21.6	19.5

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Percentage Changes in Cesium Removal

					Starting Count	25 °C	% ()s	High Temp	% Cs	Final Count	% ()	Delta % Cs
Test No.	Pretreatment	Salt Sol,n	Desorption Time	Temperature	Rate	Rate	Removed	Count Rate	Removed	Rate	Removed	Removed
1	As-Received	Avg	0	25	3716	160.7	95.7	***	***	166.7	95.2	0.2
2	As-Received	Avg	14	35	3716	165.9	95.5	254.2	93.2	176.3	95.3	0.2
3	As-Received	Avg	28	35	3716	182.5	95.1	297.2	92	187.7	95.0	0.1
4	As-Received	Avg	14	55	3716	160.7	95.7	648.7	82.5	226.4	93.9	1.8
5	As-Received	Avg	28	55	3716	168.6	95.5	590.8	84.1	231.1	93.8	1.7
6	Pretreated	Avg	0	25	3716	196.8	94.7	***	***	182.1	95.1	-0.4
7	Pretreated	Avg	1	35	3716	195.3	94.7	193.9	94.8	188.7	94.9	-0.2
8	Pretreated	Avg	14	35	3716	188.4	94.9	322.3	91.3	202.7	94.5	0.4
9	Pretreated	Avg	28	35	3716	185.6	95.0	309	91.6	184.5	95.0	0.0
10	Pretreated	Avg	14	35	3716	185.2	95.0	303.6	91.8	201.8	94.6	0.4
11	Pretreated	Avg	1	55	3716	200.1	94.6	500.1	86.5	215.1	94.2	0.4
12	Pretreated	Avg	14	55	3716	181.2	95.1	615.9	83.4	241.3	93.5	1.6
13	Pretreated	Avg	28	55	3716	172.9	95.3	589.4	84.1	218.1	94.1	1.2
14	Pretreated	Avg	14	55	3716	198.9	94.6	677.2	81.8	238.5	93.6	1.0
15	Pretreated	Avg-Si and Al	0	25	4138.9	215.7	94.8					
16	Pretreated	Avg-Si and Al	14	35	4138.9	166.2	96.0	266.3	93.6	163.7	96.0	0.0
17	Pretreated	Avg-Si and Al	14	55	4138.9	195.6	95.3	591.3	95.7	245.2	94.1	1.2
18	Pretreated	1/2 Avg	0	25	3659.6	80.9	97.8					
19	Pretreated	1/2 Avg	14	35	3659.6	87.3	97.6	144.5	96.1	99.8	97.3	0.3
20	Pretreated	1/2 Avg	14	55	3659.6	81.9	97.8	300.6	91.8	109.7	97.0	0.8
21	Pretreated	High OH	0	25	3834.3	175.7	95.4					
22	Pretreated	High OH	14	35	3834.3	168.5	95.6	239	93.8	151.9	96.0	-0.4
23	Pretreated	High OH	14	55	3834.3	155.5	95.9	443.1	88.4	181.8	95.3	0.6
24	Pretreated	High NO3	0	25	3854.8	261.7	93.2			222.6	94.2	-1.0
25	Pretreated	High NO3	14	35	3854.8	245.2	93.6	393.8	89.8	261.4	93.2	0.4
26	Pretreated	High NO3	14	55	3854.8	219.4	94.3	814.8	78.9	275.6	92.9	1.4

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