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ALKALI METAL VAPOR REMOVAL FROM PRESSURIZED FLUIDIZED-BED COMBUSTOR FLUE GAS

Quarterly Report
April—June 1980

by

Irving Johnson, W. M. Swift,
S. H. D. Lee, and W. A. Boyd

MASTER

ARGONNE NATIONAL LABORATORY, ARGONNE, ILLINOIS

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Chemical Engineering Division

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ABSTRACT

In the application of pressurized fluidized-bed combustors (PFBC) to the generation of electricity, hot corrosion of gas turbine components by alkali metal compounds is a potential problem. The objective of this investigation is to develop a method for removing these gaseous alkali metal compounds from the high-pressure high-temperature gas from a PFBC before the gas enters the gas turbine. A granular-bed filter, using either diatomaceous earth or activated bauxite as the bed material, is the concept currently being studied.

Results are presented for the testing of diatomaceous earth for alkali vapor sorption at 800°C and 9-atm pressure, using a simulated flue gas.

Activated bauxite sorbent can be regenerated by leaching with water, and the kinetics of the leaching is under study.

SUMMARY

In the proposed application of pressurized fluidized-bed combustion of coal to power generation, corrosion of the turbine blades in a downstream gas turbine due to the presence of alkali metal compounds in the hot flue gas may be a problem. The objective of this task is to develop a hot, granular fixed-bed filter to sorb alkali metal compounds from the hot flue gas before the gas is expanded through a gas turbine.

Previous studies have shown that both diatomaceous earth and activated bauxite are very effective sorbents for removing NaCl, KCl, and K_2SO_4 vapors from hot simulated flue gas of PFBC. Activated bauxite has also been demonstrated to be easily and effectively regenerated by use of a simple water-leaching process.

Experimental studies to test these two sorbents for alkali vapor sorption under pressurized conditions have been continued. Four experiments were completed at 800°C and 9-atm pressure to test the sorption of NaCl vapor from simulated PFBC flue gas by diatomaceous earth. In three of the experiments, excursions from experimental conditions were observed due to unexpected decreases in gas flow to the sorption test unit. Results obtained from the successfully completed experiment with a 5-in. thick bed operated 25 h onstream time at 800°C and 9 atm indicate that 90% of the NaCl vapor was removed from a simulated flue gas containing $\sim$3 ppmV NaCl vapor.

Studies have also been initiated to obtain a better understanding of the kinetics of the water-leaching regeneration of NaCl-adsorbed activated bauxite. The regenerated activated bauxite samples from the kinetic studies will also be used in tests to characterize its alkali sorption behavior under pressurized conditions. Two seven-cycle sets of experiments of NaCl-vapor sorption and water-leaching regeneration have been completed. Preliminary results from these leaching studies show that the maximum leaching rate of the adsorbed NaCl from the activated bauxite occurs during the first five minutes of leaching. After the first five minutes of leaching, the rate rapidly decreases and approaches a constant value. The leaching rate also appears to increase with an increasing number of water-leaching regeneration cycles.

Minor modifications made to the pressurized sorption test unit are also discussed in this report.

I. REMOVAL OF ALKALI COMPOUNDS FROM HOT FLUE GAS FROM COAL COMBUSTION

In the application of pressurized fluidized-bed combustion of coal to power generation, corrosion of the turbine blades of the gas turbine due to attack by alkali metal compounds in the hot flue gas is a possible problem. This problem can be solved by reducing the concentration of alkali metal compounds in the hot flue gas to a level which can be tolerated by a gas turbine. A way to accomplish this would be with a filter containing a hot fixed bed of sorbent to remove the alkali metal compounds from the hot flue gas before the gas is expanded into the turbine. The objectives of this task are to develop an effective sorbent and to determine the necessary characteristic sorption parameters for the design of a hot fixed-bed filter.

Diatomaceous earth and activated bauxite have been identified to be very effective in removing NaCl, KCl, and K_2SO_4 vapors from hot simulated flue gas of PFBC.[1] Also, activated bauxite has been demonstrated to be easily and effectively regenerated using a simple water-leaching process.[2] Regeneration of the alkali sorbent not only provides a possible economic benefit to the alkali-removal process, but also alleviates the spent sorbent disposal problem.

During this report period, a series of experiments has been initiated (1) to test the alkali-sorption performance of diatomaceous earth under pressurized conditions using the pressurized sorption test unit and (2) to obtain kinetic data on the water-leaching regeneration of activated bauxite. Experimental results from these studies are presented. Also described are minor modifications of the pressurized sorption test unit (ANL/CEN/FE-80-4).

A. Sorption Tests

In four experiments (PHGC-D-1 to -4) completed using the pressurized sorption test unit, the sorption of NaCl vapor from a simulated PFBC flue gas by diatomaceous earth was measured as a function of experiment duration (7, 16, 20, and 25 h). The details of the pressurized sorption test unit were reported previously (ANL/CEN/FE-80-4). In each experiment, a 5-in.-thick bed of sorbent was divided into five equal sections with platinum gauze dividers as described in the preceding report in this series (ANL/CEN/FE-80-5). The tests were performed at a bed temperature of 800°C, a system pressure of 9 atm absolute, and a superficial gas velocity of 30 cm/s.

The volumetric composition of the simulated flue gas was 5.1% O_2, 18.2% CO_2, 3.3% H_2O, and the balance N_2. To prevent corrosion of the test system, no SO_2 was contained in the simulated PFBC flue gas.

The particle size of the diatomaceous earth used was -8 +10 mesh, which is equivalent to 2-mm-diameter particles. The diatomaceous earth particles had been heat-treated at 900°C in an air stream for 15 h prior to testing in order to remove any volatile alkali metal compounds initially present.

At the end of each sorption test, the amount of NaCl vaporized and transported in the simulated PFBC flue gas to the sorbent bed was determined; each section of a sorbent bed was separately collected and analyzed for sodium content; condensates on the cold trap, the glass-wool filter (a back-up filter), and Al_2O_3 sorption tube were collected by washing the parts with distilled water and analyzing for sodium concentration. From the above determinations, a NaCl material balance was made around the sorbent bed.

Close control of the bed temperature along the length of the sorbent bed is essential. In order to verify close control, the temperature profile along the entire 5-in. bed of diatomaceous earth is measured with five thermocouples and the measured temperature profile along the bed as a function of experiment duration in a blank run are shown in Fig. 1.

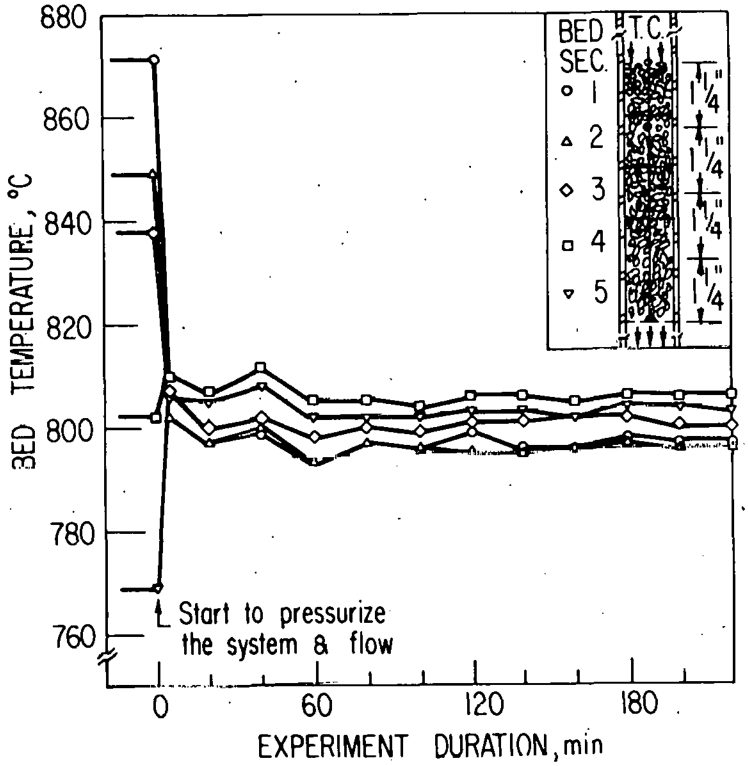

Fig. 1. Temperature Profile Measurement along 5-in. Bed of Diatomaceous Earth (8-10 mesh)

The temperature profile measurements were done under the same experimental conditions as those used in sorption tests--namely, a bed temperature of 800°C, a system pressure of 9 atm, and a superficial gas velocity of 30 cm/s. As shown in Fig. 1, the temperature of the entire 5-in. bed can be controlled within 12°C, with the center of the bed at about 800°C, the upstream bed on the low side of the selected temperature, and the downstream bed on the high side of the selected temperature.

Table 1 presents the material balances for the four tests completed. Except in experiment PHGC-D-1 (7-h run), there were periods of unattended overnight operation. To provide uninterrupted gas flow throughout each run, two or more cylinders of each flue gas component were connected in series, with each cylinder controlled by its own flow regulator. When this gas flow arrangement was used, excursions occurred during portions of the first three experiments (PHGC-D-1, -2, and -3) due to decreases in nitrogen or nitrogen and CO_2 gas flows to the system, a result of improper regulation of the delivery pressure at each cylinder that caused gas flow to decrease during

Table 1. Material Balances for Experiments Testing Sectioned Beds of Diatomaceous Earth for NaCl Vapor Capture[a]

Experiment PHCG-D-	1	2	3	4
Experiment Duration, h	7	16	20	25
INPUT (I)				
(1) NaCl Vaporized, mg	70	267	146	228
OUTPUT (O)				
(2) NaCl Collected by				
(a) Cold Trap, mg	9	1	1	9
(b) Glass-Wool Filter, mg	1	14	8	12
(c) Al_2C_3 Sorption Tube, mg	2	2	2	2
(3) NaCl Captured by Sorbent,[b] mg	28	160	75	204
(4) Sum, mg	40	177	86	228
Loss (I - O), mg (%)	30 (42.9%)	90 (33.7%)	60 (41.1%)	0 (0%)

[a] Tested at 800°C and 9 atm. Superficial gas velocity was 30 cm/s.

[b] Sodium content of sorbent was obtained by dissolving two representative samples of ground sorbent (200 mesh) in a mixture of H_2SO_4, HF, and HNO_3 and then analyzing the solution by flame emission spectrometry (FE). FE was done by R. Bane.

switching from one cylinder to another in periods of unattended operation. Excursions in gas flow rates could cause excursions in sorbent bed temperature and NaCl sample temperature so that the rate of NaCl vaporization would vary during an experiment. A proper and careful adjustment of each cylinder's delivery pressure was made and experiment PHGC-D-4 (the 25-h run) was completed smoothly without any problems.

As can be seen in Table 1, material balances indicated that 34-43% of the total NaCl vaporized cannot be accounted for in experiments PHGC-1, 2, and 3, in which excursions were observed. Whether the observed losses of NaCl are related to the excursions or to other factors is not known at this time. Further investigation is needed to clarify this discrepancy. The material balance for experiment PHGC-D-4 (the smooth, successful run) shows excellent agreement of the NaCl "Input" and "Output".

The weight of sorbent and the amount of NaCl sorbed in each section of the bed in each of the completed four experiments are given in Table 2. It is apparent from this table that NaCl vapor in the simulated flue gas was essentially captured by the first section of the bed. Based on the results of experiment PHGC-D-4, the 5-in. bed of diatomaceous earth removed (on the average) 90% of the NaCl vapor present in a flue gas containing ∿3 ppmV NaCl vapor for 25 h onstream time at 800°C bed temperature and 9-atm system pressure.

During this series of tests, agglomeration of sorbent particles was observed in the first section of the bed. Some particles were found to adhere to the wall of the alumina sorption tube. It is believed that diatomaceous earth reacts with sorbed NaCl vapor, forming sodium silicates that melt at the bed temperature (800°C). From a practical viewpoint, formation of "sticky" sodium silicate melt on the surface of sorbent particles could cause operational difficulties in a large granular-bed filter.

B. Modification of the Pressurized Sorption Test Unit

A pressurized alkali-vapor sorption test unit has been assembled (ANL/CEN/FE-80-4). Because of the high rate of nitrogen gas consumption during an experiment, a liquid nitrogen cylinder equipped with a built-in vaporizer (Model ARLG-45, Airco Welding Supply) has been installed to replace the pressurized nitrogen cylinders (220 cu ft) used previously. The new cylinder has a liquid nitrogen capacity of 3500 cu ft and will supply nitrogen gas at a rate of 325 cu ft/h at 250 psig during continuous operation. The normal vaporization rate (leak rate) is 2.4%/day (or 87 cu ft/day). Based on the nitrogen consumption rate in typical experiments and the leak rate, replacement of an ARLG-45 liquid nitrogen cylinder is expected to be required only once every 1.5 to 2 months. This modification will save time and labor required to replace cylinders, will conserve laboratory space for the storage of cylinders, and most importantly, will prevent flow excursions during experiments.

A pressure switch has been installed to control the lower limit of the operating pressure in the system (a pressure switch to control overpressure of the system was previously installed). In case the Al_2O_3 sorption tube fails or a gas leak occurs during an experiment, the newly installed pressure switch will be activated to shut off all furnaces and gas supplies and to vent the systems.

Table 2. Weight and Sodium Analyses of each Section of the Diatomaceous Earth Bed

Exp. PHGC-D-	Section No.	Wt of Sorbent, g	NaCl Capture, mg NaCl/g sorbent	Total NaCl Captured, mg	Avg % NaCl Vapor Captured[a]
1	1	4.7671	5.5	26.3	
	2	4.8451	0.3	1.5	
	3	4.7117	0	0	
	4	4.8416	0	0	
	5	4.7793	0	0	
	total	23.9448		27.8	
2	1	4.9200	31.9	157.0	
	2	4.7703	0.5	2.5	
	3	4.8457	0	0	
	4	4.8140	0	0	
	5	4.8289	0	0	
	total	24.1789		159.5	
3	1	4.6560	15.3	71.2	
	2	4.9889	0.8	4.0	
	3	4.7405	0	0	
	4	4.7049	0	0	
	5	4.8576	0	0	
	total	23.9479		75.2	
4	1	4.9835	37.7	187.7	82.3
	2	4.7089	2.4	11.4	5.0
	3	4.7804	0.5	2.2	1.0
	4	4.7712	0.3	1.2	0.5
	5	4.7864	0.4	1.8	0.8
	total	24.0304		204.3	89.6

[a] Obtained by dividing the total NaCl captured (Column 5) in each section by the total amount of NaCl vapor transported to the bed, which is given in row (1) of Table 1.

C. NaCl Vapor Sorption and Water-Leaching Regeneration of Activated Bauxite

This study has two objectives. The first objective is to obtain a better understanding of the water-leaching regeneration kinetics of NaCl-adsorbed activated bauxite. The second objective is to produce regenerated activated bauxite samples for subsequent characterization of their alkali sorption behavior in the pressurized sorption test unit. In the water-leaching kinetics study, activated bauxite is cyclically tested for NaCl vapor sorption and water-leaching regeneraion. The procedures for the cyclic experiment are shown and explained in Fig. 2. The NaCl vapor sorption experiments were carried out using the laboratory-scale, horizontal batch fixed-bed combustor.[3]

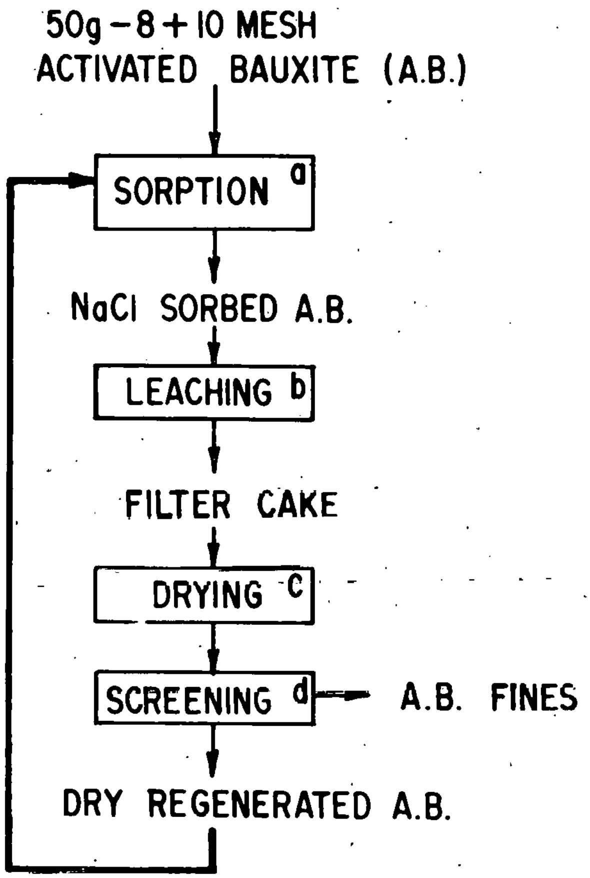

Fig. 2.

Flow Sheet Showing NaCl Vapor Sorption Step and Water-Leaching Regeneration Step for Activated Bauxite

a. Sorption Experiment Conditions: 800°C at atmospheric pressure in a simulated dry flue gas of PFBC containing 3% O_2, 16% CO_2, 300 ppm SO_2, and the balance N_2; superficial gas velocity = 66 cm/s; GHSV $\cong$ 18,600 h^{-1} (or contact time of 0.2 s); experiment duration = 6 h.

b. Leaching Conditions: leached with distilled water in a beaker at gently boiling temperature (∿95°C) for a specific duration.

c. Drying Conditions: 400°C for 1 h in an air flow.

d. Sieved with 10-mesh screen to discard fines.

To determine the leaching rate of adsorbed NaCl from activated bauxite, the following experimental procedures were used: (1) the NaCl-adsorbed activated bauxite obtained from each sorption cycle was divided into four portions by weighing. Each portion of the sample was then leached with 100 mL of distilled water at 95°C for a specific period of time--5, 10, 30, or 60 min.

Distilled water was intermitently added to each leaching solution to compensate for vaporization losses and thereby maintain a constant amount of leaching medium. At the end of each leaching period, the leachate was collected and analyzed for its sodium content. The leached activated bauxite samples (from all four leachings) were then recombined and subjected to Soxhlet extraction for ∿15 h. Again, distilled water was used as the extraction medium. By repeated extractions for a long duration with pure water, Soxhlet extraction can achieve practically complete extraction of the adsorbed NaCl from the sorbent. At the end of extraction, the extracting solution was also analyzed for sodium content. Subsequent treatment of the extracted activated bauxite sample then proceeded by the procedures shown in Fig. 2.

In this NaCl vapor sorption and water-leaching regeneration work, two sets of cyclic experiments (HGC-88 series and HGC-89 series) were conducted.

Tables 3 and 4 show the amounts of sorbent being leached and the amounts of NaCl leached out as a function of leaching duration for each cycle of experiments HGC-88 and HGC-89, respectively. During the water-leaching stage, activated bauxite fines were produced due to attrition of the sorbent particles. As shown in Fig. 2, activated bauxite fines (<10 mesh particles) were separated by screening and discarded before the regenerated activated bauxite was further tested for NaCl vapor sorption in the next cycle; therefore, the total amount of regenerated sorbent decreased continuously in later regeneration cycles, as shown in Tables 3 and 4.

Table 3. Summary of Water-Leaching Regeneration of NaCl-Adsorbed Activated Bauxite for Experiment HGC-88

Leaching-Regeneration Cycle No.	Leaching Time, min	Weight of Sorbent Leached, g	Total NaCl Leached, mg	NaCl Leached per gram Sorbent,[a] mg	% NaCl Leached per gram Sorbent[b]
1	5	12.50	51.2	4.10	67.3
	10	12.50	55.3	4.42	72.6
	30	12.50	58.1	4.65	76.4
	60	12.50	65.8	5.26	86.4
	15 h[c]	50.00	74.3		
	Sum	50.00	304.7	6.09	
2	5	12.51	48.6	3.89	72.2
	10	12.50	49.6	3.97	73.7
	30	12.50	54.8	4.38	81.3
	60	11.65	47.9	4.11	76.3
	15 h[c]	49.16	63.9		
	Sum	49.16	264.8	5.39	
3	5	12.49	31.1	2.49	74.1
	10	12.39	29.6	2.39	71.1
	30	12.36	32.7	2.65	78.9
	60	10.21	30.9	3.03	90.2
	15 h[c]	47.45	35.1		
	Sum	47.45	159.4	3.36	

(Contd)

Table 3. (Contd)

Leaching-Regeneration Cycle No.	Leaching Time, min	Weight of Sorbent Leached, g	Total NaCl Leached, mg	NaCl Leached per gram Sorbent,[a] mg	% NaCl Leached per gram Sorbent[b]
4	5	12.49	27.4	2.19	79.1
	10	12.30	28.0	2.28	82.3
	30	12.36	30.0	2.43	87.7
	60	9.20	22.3	2.42	87.4
	15 h[c]	46.35	20.5		
	Sum	46.35	128.2	2.77	
5	5	12.50	51.2	4.10	88.4
	10	12.49	47.6	3.81	82.1
	30	12.49	47.0	3.76	81.0
	60	8.26	35.6	4.31	92.9
	15 h[c]	45.74	30.7		
	Sum	45.74	212.2	4.64	
6	5	12.50	54.8	4.38	84.7
	10	12.49	54.9	4.40	85.1
	30	12.49	53.7	4.30	83.2
	60	7.24	35.3	4.88	94.4
	15 h[c]	44.72	32.4		
	Sum	44.72	231.0	5.17	
7	5	11.00	52.1	4.74	86.5
	10	11.00	53.3	4.85	88.5
	30	11.00	53.7	4.88	89.1
	60	10.59	50.0	4.72	86.1
	15 h[c]	43.59	29.8		
	Sum	43.59	238.9	5.48	

[a] Obtained by dividing values in column 4 by values in column 3.

[b] Obtained by dividing values in column 5 by the average amount of NaCl adsorbed on one gram of sorbent. The latter is obtained by dividing the sum of the total NaCl leached in each cycle by the total weight of sorbent leached in the cycle.

[c] Duration of Soxhlet extraction in hours.

It should be pointed out that in these series of experiments, the amount of NaCl vapor transported to the sorbent bed varied in each cycle. This is probably due to loose control of the NaCl sample temperature. As a result, the amount of NaCl captured by the sorbent bed varied in each cycle, as reflected in the different amounts of NaCl leached out in the different cycles (column 4 in both Tables 3 and 4). It was also noticed that the sorbent bed became more loosely packed as the amount of regenerated sorbent decreased, resulting in (1) a decrease in the NaCl vapor capture efficiency of the bed and (2)--usually--a decrease in the total amount of NaCl leached (Tables 3 and 4). The decrease in NaCl vapor capture efficiency by the sorbent bed was reflected in more NaCl being captured by cold traps downstream from the

Table 4. Summary of Water-Leaching Regeneration of NaCl-Adsorbed Activated Bauxite for Experiment HGC-89

Leaching-Regeneration Cycle No.	Leaching Time, min	Weight of Sorbent Leached, g	Total NaCl Leached, mg	NaCl Leached per gram Sorbent,[a] mg	% NaCl Leached per gram Sorbent[b]
1	5	12.50	65.6	5.25	78.1
	10	12.50	62.3	4.98	74.1
	30	12.50	60.8	4.86	72.3
	60	13.26	68.3	5.15	76.6
	15 h[c]	50.76	84.2		
	Sum	50.76	341.2	6.72	
2	5	12.50	27.4	2.19	50.3
	10	12.50	42.8	3.43	78.9
	30	12.46	47.7	3.83	88.0
	60	11.18	46.6	4.17	95.9
	15 h[c]	48.64	47.3		
	Sum	48.64	211.8	4.35	
3	N.A.[d]				
4	5	12.50	43.3	3.46	69.8
	10	12.49	43.5	3.48	70.2
	30	12.48	49.4	3.96	79.8
	60	8.70	37.7	4.33	87.3
	15 h[c]	46.17	54.9		
	Sum	46.17	228.8	4.96	
5	5	12.50	45.1	3.61	77.1
	10	12.50	42.7	3.42	73.1
	30	12.50	50.2	4.02	85.9
	60	7.33	31.6	4.31	92.1
	15 h[c]	44.83	40.1		
	Sum	44.83	209.7	4.68	
6	5	11.00	60.3	5.48	82.8
	10	11.09	62.1	5.65	85.3
	30	11.10	61.8	5.61	84.7
	60	10.93	59.4	5.43	82.0
	15 h[c]	43.94	47.1		
	Sum	43.94	290.7	6.62	

(Contd)

Table 4. (Contd)

Leaching-Regeneration Cycle No.	Leaching Time, min	Weight of Sorbent Leached, g	Total NaCl Leached, mg	NaCl Leached per gram Sorbent,[a] mg	% NaCl Leached per gram Sorbent[b]
7	5	11.00	52.7	4.79	72.8
	10	11.00	60.8	5.53	84.0
	30	11.00	65.4	5.95	90.4
	60	9.08	54.9	6.05	92.0
	15 h[c]	42.08	43.1		
	Sum	42.08	276.9	6.58	

[a] Obtained by dividing values in column 4 by values in column 3.

[b] Obtained by dividing values in column 5 by the average amount of NaCl adsorbed on one gram of sorbent. The latter is obtained by dividing the sum of the total NaCl leached in each cycle by the total weight of sorbent leached in the cycle.

[c] Duration of Soxhlet extraction in hours.

[d] N.A. - Not analyzed due to incomplete sorption test caused by power failure. Adsorbed NaCl was completely leached from sorbent before it was tested in the next cycle.

sorbent bed. Beginning with cycle 4 of experiment HGC-89, a step was taken to correct the variation in bed packing by introducing an extra platinum gauze to support the bed. This corrective measure effectively increased the capture efficiency of the bed and thus increased the amount of NaCl leached, as shown in Tables 3 and 4.

Figure 3 is a plot of the percent of the NaCl leached as a function of leaching duration. The values plotted in this figure each represent the average for the percentages of NaCl leached in corresponding cycles of experiments HGC-88 and HGC-89 (last columns of Tables 3 and 4). As shown in this figure, the data points are somewhat scattered. This is believed due to the difficulty of dividing the NaCl-adsorbed activated bauxite sample into equally homogeneous portions for leaching.

Despite the scatter in the data, Fig. 3 shows that adsorbed NaCl is leached from the activated bauxite at the maximum rate during the first five minutes of leaching. After that, the rate quickly decreases, approaching a constant value.

Figure 3 also shows that the rate of leaching increases with leaching cycle. This is further illustrated in Fig. 4, which is a plot of the average percentages of NaCl leached in experiments HGC-88 and HGC-89 (last columns of Table 3 and 4) as a function of leaching cycle. It is evident from Fig. 4 that for each leaching duration, the percent NaCl leached increased with each subsequent leaching regeneration cycle. On the average, 75% of the sorbed NaCl was leached in the first five minutes of leaching and this amount increased to ∿85% in one hour of leaching.

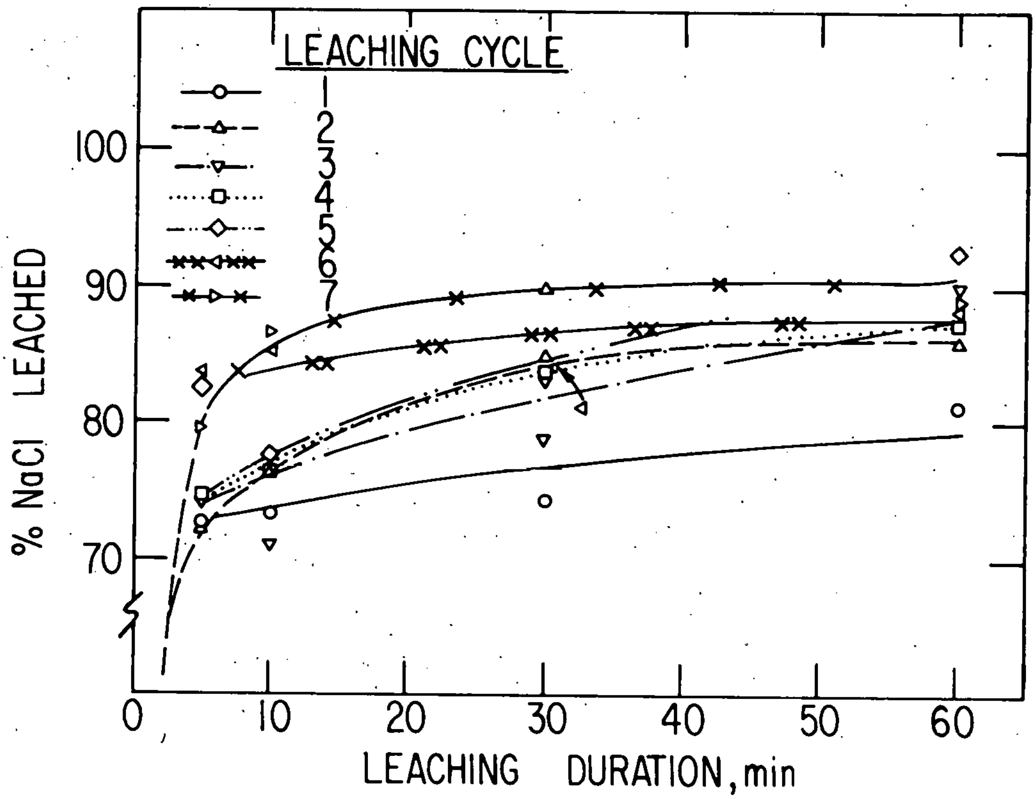

Fig. 3. Amount of Leachable NaCl Leached as a Function of Leaching Duration. Each point represents the average of an Exp. HGC-88 value and an Exp. HGC-89 value.

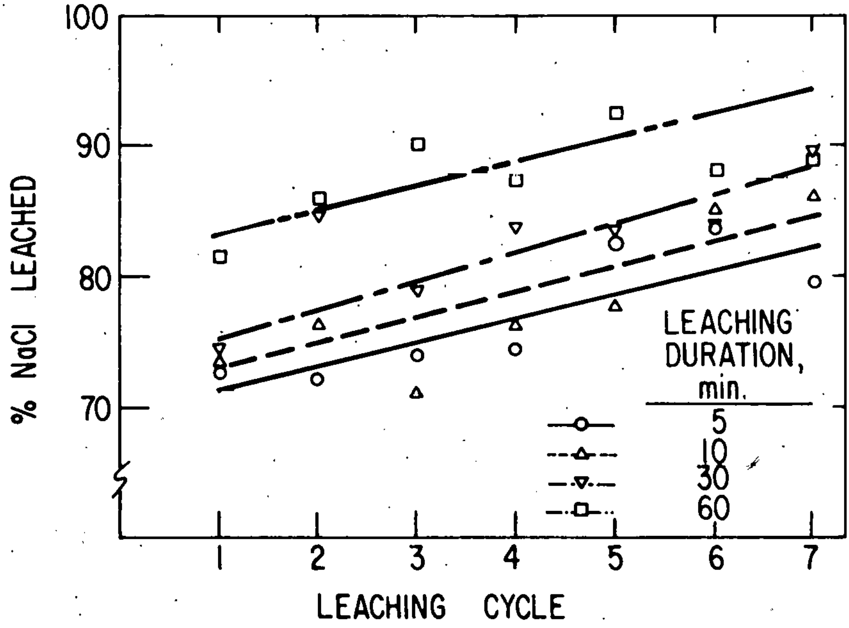

Fig. 4. Amount of Leachable NaCl Leached from NaCl-Adsorbed Activated Bauxite as a Function of Leaching Cycle

In previous studies,[2] it was found that activated bauxite that has been regenerated by water leaching continuously gains in pore volume as the number of water-leaching regeneration cycles increases. Also, a fraction of the NaCl vapor captured by activated bauxite was found to react with SiO_2 and clay minerals present as impurities to form water-insoluble compounds (such as sodium aluminum silicates). Apparently, these reactions open up the structure of activated bauxite and therefore open up the pores of activated bauxite and create more fine pores. The increase in pore size reduces the resistance to diffusion of water into the pores for extracting the adsorbed NaCl molecules. Consequently, the rate of leaching increases with increasing leaching regeneration cycle, as is observed in Fig. 4.

D. Future Work

Experimental studies will be continued to test activated bauxite under pressurized conditions and to obtain the technical data base required for the design of large-scale granular-bed filters. Alkali sorption isotherm and alkali breakthrough data will be obtained in the first phase of this work. Studies on the water-leaching of activated bauxite will also be continued in order to investigate the effects on leaching rate of (1) adsorbed alkali loading in activated bauxite and (2) leaching temperature.

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