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DIATOMACEOUS EARTH AND ACTIVATED BAUXITE USED AS GRANULAR SORBENTS FOR THE REMOVAL OF SODIUM CHLORIDE VAPOR FROM HOT FLUE GAS

by

Sheldon H. D. Lee, William M. Swift,
and Irving Johnson

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ABSTRACT

Diatomaceous earth and activated bauxite were tested as granular sorbents for use as filter media in granular-bed filters for the removal of gaseous alkali metal compounds from the hot (800°C) flue gas of PFBC. Tests were performed at atmospheric pressure, using NaCl vapor transported in relatively dry simulated flue gas of PFBC. Either a fixed-bed combustor or a high-temperature sorption test rig was used. The effects of sorbent bed temperature, superficial gas velocity, gas hourly space velocity, and NaCl-vapor concentration in flue gas on the sorption behavior of these two sorbents and their ultimate sorption capacities were determined. Both diatomaceous earth and activated bauxite were found to be very effective in removing NaCl vapor from flue gas. Preliminary cost evaluations showed that they are economically attractive as granular sorbents for cleaning alkali vapor from simulated flue gas.

I. INTRODUCTION

Pressurized fluidized-bed combustion (PFBC) offers a coal combustion technique having the potential for improved thermal efficiency, reduced costs, and acceptable environmental impact.[1] This technique involves the combustion of coal in a fluidized bed of chemically active sorbent that is capable of capturing sulfur dioxide released during coal combustion. Limestone and dolomite are widely used as sorbents.

A potential application of PFBC is in combined-cycle power generation systems.[2] In such systems, the high-pressure hot flue gas will be expanded through a gas turbine to recover energy.

Past experience obtained when operating gas turbines with hot flue gas from (1) the combustion of liquid fuels[3] and (2) the direct combustion of pulverized coal[4] has indicated that particulates of unburned coal and ash and alkali metal compounds (such as chlorides and sulfates of sodium and potassium) in the flue gas cause catastrophic erosion, corrosion, and fouling of gas turbine hardware. The turbine erosion and corrosion problems in PFBC combined-cycle systems have recently been reviewed by Stringer and his associates.[5] For the successful operation of combined-cycle power plants, cleanup of the hot flue gas to remove particulates and alkali metal compounds upstream from the gas turbine is imperative.

Of the corrosive agents present in the flue gas of coal combustion, alkali metal compounds are thought to cause hot corrosion of turbine hardware.[6,7] Sodium and potassium compounds exist in coal in similar chemical forms. Sodium chloride and potassium chloride are present in saline groundwater that permeates the rock and fills the pores and cracks in the coal bed. Both sodium and potassium are also constituents of clay minerals in coal, such as illite and montmorillonite. Sodium chloride is a principal form of sodium in coal; in contrast, potassium in coal occurs largely as nonvolatile alumino-silicates. However, potassium can be released from silicates as potassium chloride by exchange reactions with sodium chloride vapor.[8-10] Since both sodium chloride and potassium chloride have appreciable vapor pressures (0.4 and 0.7 kPa, respectively, at 900°C) at the fluidized-bed combustion temperature range, their vapors are expected to be in the flue gas of PFBC. Thermodynamic calculations indicate that gaseous sodium chloride and potassium chloride are the major sodium and potassium carriers, respectively, in the flue gas from the fluidized-bed combustion of coal.[11] Alkali metal sulfates can be formed by the reactions of alkali chlorides with gaseous sulfur compounds.

A way to eliminate the hot corrosion of gas turbine hardware is to reduce the concentration of alkali metal compounds in hot flue gas to a level tolerable for turbine blades. The current industrial gas turbine specification limit placed upon alkali metals is equivalent to 0.024 ppm in the combustion gases entering the turbine.[2] A direct method for accomplishing the alkali vapor reduction in flue gas is by controlling combustion to minimize the evolution of alkali metal compounds. Combustion Power Company has demonstrated that the addition of an aluminum silicate clay to the combustion zone of PFBCs effectively reduces the emission of alkali vapors.[12] However, this measure has not shown the capability of reducing alkali concentration in the flue gas to a tolerable level for gas turbines.

An alternative method for reducing alkali concentration in the flue gas is the removal of alkali metal compounds from flue gas after combustion but before it is expanded into a turbine. However, no commercially reliable and economically feasible high-temperature and high-pressure (HTHP) gas purification equipment for HTHP cleanup of flue gas is yet available.[13] The hot gas cleanup systems for PFBC combined-cycle systems have been reviewed and assessed by Stringer and his associates.[5] Granular-bed filters are among many devices being actively developed for high-temperature, high-pressure particulate cleanup of flue gas.[5,13-15] They hold great promise based on their potential advantages over other devices for particulate removal.[16] Above all, a granular-bed filter with a suitable bed material will simultaneously remove particulates and alkali metal compounds. Therefore, the cleanup of alkali corrodents from flue gas can be achieved if an effective granular sorbent that is capable of capturing alkali metal compounds from flue gas can be found. The objective of this study is to develop such a granular sorbent.

In the screening tests of six commercially available products, the authors found activated bauxite and diatomaceous earth to be very effective in removing NaCl, KCl, and K_2SO_4 vapors from simulated hot flue gas of PFBC.[17] To pursue these findings and obtain the technical data base needed for the design of a large-scale granular filter, studies of these two promising sorbents were continued. In these studies, sodium chloride vapor transported in hot, relatively dry simulated flue gas of PFBC was used to test NaCl-vapor sorption capabilities of the sorbents at atmospheric pressure.

The results obtained from these atmospheric studies provide an understanding of the fundamentals of the sorption processes and the guideline for further studies at elevated pressures to simulate PFBC conditions. NaCl and KCl have similar chemical properties. The sorption behaviors of diatomaceous earth and activated bauxite toward both NaCl and KCl vapors are also similar;[17] therefore, it is also reasonable to assume the applicability of the results obtained from these NaCl-vapor studies to the sorption phenomenon of the sorbents toward KCl vapor, which is believed to be the major potassium compound present in the flue gas of fluidized-bed combustion of coal.[11]

Presented in this report are experimental results obtained on the effects of operating variables on the NaCl-vapor sorption behavior of diatomaceous earth and activated bauxite. Preliminary cost evaluations based on the experimental results are also discussed.

II. EXPERIMENTAL

A. Material

Diatomaceous earth used in this study is "Celatom MP-91" diatomaceous silica, a product of Eagle-Picher Industries, Inc., Ohio. Activated bauxite is a high-alumina "Porocel" bauxite product obtained from Engelhard Minerals and Chemicals Corporation, New Jersey. Table 1 shows their typical chemical compositions as supplied by the manufacturer. Both diatomaceous earth and

Table 1. Typical Chemical Compositions of Diatomaceous Earth and Activated Bauxite

Compound	Diatomaceous Earth, wt %	Activated Bauxite, wt %
SiO_2	92.0	10.0
Al_2O_3	5.0	81.5
Fe_2O_3	1.8	3.0
CaO	0.2	N.A.[a]
MgO	0.3	N.A.
TiO_2	N.A.	3.5
Others	0.3	2.0
	99.6	100.0

[a] N.A. - not analyzed.

activated bauxite tested in this study were −8 +10 mesh particles obtained by sieving. Before each test, each sorbent was preheated in a flowing air stream at 900°C for ∼15 h to remove volatile alkali metal compounds.

B. Equipment

A laboratory-scale, batch-type, fixed-bed combustor system was used to test sorbents for the removal of gaseous sodium chloride. Figure 1 is a schematic diagram of the combustor system. A detailed description of the system is presented elsewhere.[18]

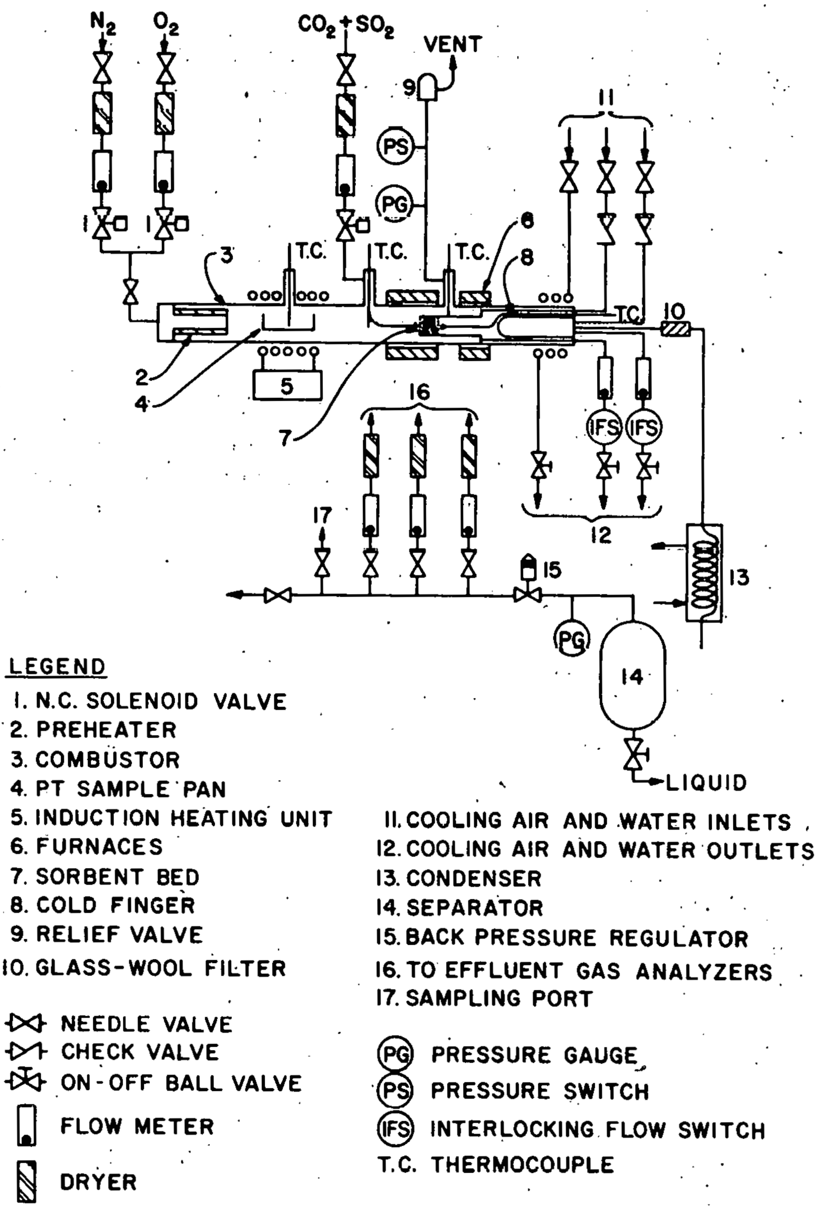

Fig. 1. Batch-Type Fixed-Bed Combustor System

The combustor is an 8.9-cm-ID 1.8-m-long horizontal stainless steel pipe and consists of four sections: preheating, combustion, filtration, and cold-trap sections. The sorbent is placed between two perforated steel plates in a stainless steel tube, which is fastened upstream from a cold trap tube into which a cold finger is inserted; thus, the sorbent is in the filtration section of the combustor. A weighed quantity of pure sodium chloride in a platinum sample pan is heated (by induction heating) in the combustion section of the combustor and is vaporized. The vapor is carried by preheated flowing gas downstream through the sorbent bed, and any remaining vapor is condensed on the cold trap tube, the cold finger, and the backup filter (<u>i.e.</u>, the glass-wool filter).

A small-scale sorption test rig (Fig. 2) was used to determine the ultimate sorption capacity of the sorbent. In this test rig, a known amount of sodium chloride contained in a platinum sample pan is vaporized inside a pure Al_2O_3 tube that is heated by a tubular furnace. The preheated simulated PFBC flue gas then carries the sodium chloride vapor downstream through the bed of sorbent to be tested and through a condenser, and finally the gas is vented to an exhaust. The sorbent bed is supported by a platinum gauze and quartz tubing inserted concentrically inside the Al_2O_3 tube. The Al_2O_3 tube is capped on both ends with lava end caps. A ceramic fiber gasket is used as the seal between the Al_2O_3 tube and the end cap.

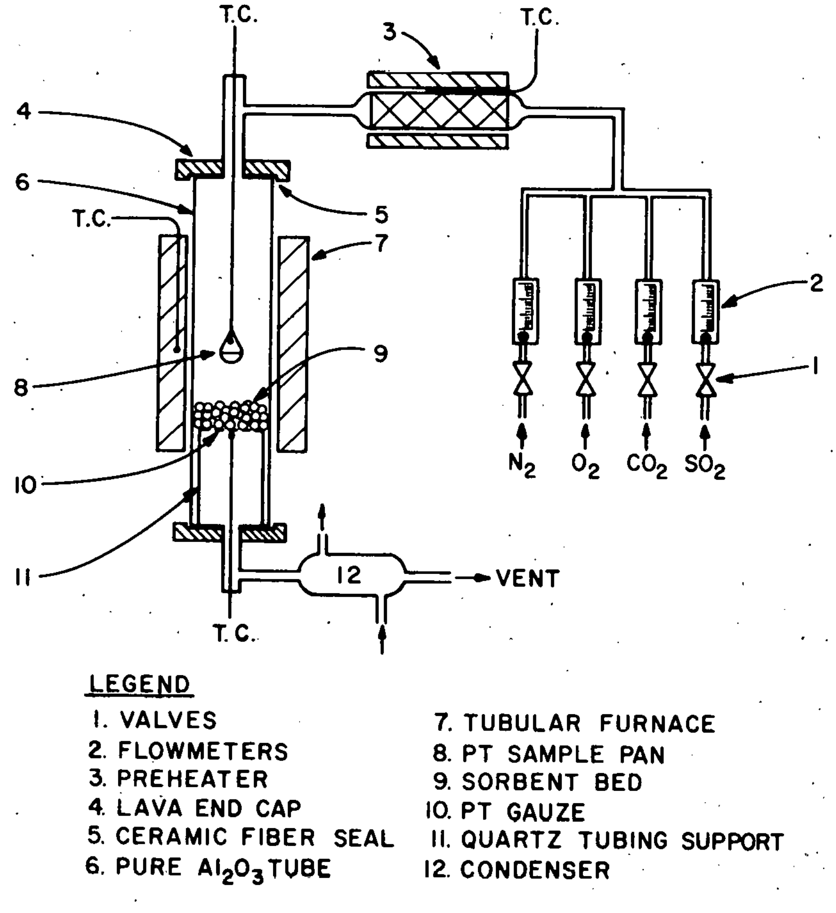

Fig. 2. NaCl-Vapor Sorption Test Apparatus

C. Procedures

1. Sorption Tests

All tests conducted in this study were performed at atmospheric pressure. Sorbents were tested using simulated relatively dry flue gas of PFBC. The volumetric composition of the simulated flue gas was 3% O_2, 16% CO_2, ∿180 ppm H_2O, ∿300 ppm SO_2, sodium chloride vapor ranging from 24 to 150 ppm, and the balance N_2. The trace amount of moisture was the moisture present in O_2, CO_2, and N_2 cylinders. Although the estimated alkali concentration in the flue gas of PFBC is on the order of 5 ppm, a fairly high alkali concentration in the flue gas was used in this work so that a detectable amount of alkali vapor could be obtained within a reasonable experimental period. The NaCl sample temperature was controlled at 900°C, which was greater than the sorbent bed temperature of about 800°C. Because of the substantial dilution of the sodium chloride vapor by the hot carrying gas, the sodium chloride in the carrying gas should be in the gas phase. This was supported by vapor pressure calculations showing that the sodium chloride vapor pressure in the carrying gas was significantly lower than its saturation vapor pressure at the sorbent bed temperature. The amount of sodium chloride vaporized for a test was controlled by varying either the size of the sample pan or the sodium chloride sample temperature.

At the end of each experiment, the weight loss of sodium chloride in the sample pan was measured; the condensates were collected by washing with distilled water and were analyzed for sodium. The amount of sodium chloride vapor captured by the sorbent was obtained by the direct analysis of the sodium content of the sorbent. The sorption capability of a sorbent was evaluated by its effectiveness in removing sodium chloride vapor from flue gas.

2. Sorbent Preparation for Sodium Content Analysis

To obtain a representative sample of the sorbent for analysis, the entire amount of the sorbent tested was first ground to about -200 mesh powder. After thorough mixing of the powder, two random samples (one gram for each diatomaceous earth sample and one-half gram for each activated bauxite sample) were taken for the preparation of a solution for analysis.

For diatomaceous earth, the sorbent sample was dissolved in a mixture of H_2SO_4 and HF (10 mL 1:1 H_2SO_4 and 25 mL 49% HF) in a plastic container. The solution was evaporated on a water bath until most of the HF was removed, then 3 mL HNO_3 was added, and the heating continued until strong fumes of SO_3 evolved. Distilled water was added and then the solution was heated on a water bath for one-half hour. The solution was cooled and analyzed for sodium concentration.

For activated bauxite, the sample was fused with 15 grams of high-purity (98%) NH_4HF_2 in a platinum crucible in an oil batch (at ∿170°C). After fusion of the sample was complete (∿2 h), the fused product was dissolved in 150 mL distilled water, and then 25 mL HCl was added to dissolve the precipitate. The solution was then analyzed for sodium concentration.

D. Analytical Methods

Sodium concentrations in either water or acid solutions were determined by flame emission spectrometry. An Instrumental Laboratory Model 153 spectrometer was used for the analyses.

III. SUMMARY OF EARLIER SCREENING TESTS

Six commercial products--alundum (pure Al_2O_3), Celatom MP-91 diatomaceous earth, silica gel, Burgess No. 10 pigment, Attapulgus clay, and activated bauxite--were screened as filter media in granular-bed filters for removal of gaseous alkali metal compounds from the hot flue gas of PFBCs.[17] Tests were performed using a simulated relatively dry flue gas of PFBC in the laboratory-scale, fixed-bed combustor (Fig. 1) and the high-temperature sorption test rig (Fig. 2). Tests were conducted at atmospheric pressure.

Experimental results show that both diatomaceous earth and activated bauxite are very effective granular sorbents for the removal of gaseous NaCl, KCl, and K_2SO_4 from hot (800-880°C) simulated flue gas of PFBC. Alkali chloride vapor removal efficiencies ranging from 95 to 98% for both sorbents were achieved at the experimental conditions of superficial gas velocity of 25 cm/s, a gas hourly space velocity (GHSV) of 12,000 h^{-1} (or a contact time of 0.3 s between the flue gas and the sorbent bed), and $\sim$83 ppm alkali chloride vapor in the flue gas. The K_2SO_4-vapor-removal efficiencies were 72 and 93% for diatomaceous earth and activated bauxite, respectively, at a fairly high gas hourly space velocity of 62,000 h^{-1} or a contact time of only 0.05 s with $\sim$3 ppm K_2SO_4 vapor in the flue gas. Improved capabilities of these sorbents for retaining K_2SO_4 vapor can be expected if the contact time of the flue gas with the sorbent bed is increased.

The retention of gaseous alkali metal compounds by diatomaceous earth is attributed to chemical reactions of diatomaceous earth with alkali metal compounds to form water-insoluble or difficultly soluble alkali metal silicates.

$$2\ NaCl(g) + H_2O(g) + n\ SiO_2(s) = Na_2O \cdot n\ SiO_2(s) + 2\ HCl(g)$$

$$\Delta H_{800°C} = -5.71 \text{ kcal mol}^{-1} \text{ for } n = 1$$

K_2SO_4 vapor reacts with activated bauxite to form water-insoluble compounds; however, capture of gaseous alkali metal chlorides by activated bauxite is primarily by an adsorption mechanism.

IV. RESULTS AND DISCUSSION

A. Effect of Sorbent Bed Temperature

Table 2 shows experimental conditions for testing both diatomaceous earth and activated bauxite for NaCl-vapor capture as a function of sorbent bed temperature and the duration of the experiment. Results of these tests are tabulated in Tables 3 and 4, which show the distributions of NaCl when NaCl vapor is captured by diatomaceous earth and activated bauxite, respectively.

Table 2. Experimental Conditions for Testing Diatomaceous Earth and Activated Bauxite for NaCl-Vapor Capture

Avg. Sorbent-Bed Temperature	800 and 880°C
System Pressure	100 kPa
Particle Size of Sorbent	-8 +10 mesh
Thickness of Sorbent Bed	1.3 cm
Flowing Gas Composition	3% O_2 16% CO_2 $\sim$300 ppm SO_2 $\sim$180 ppm H_2O 69-98 ppm NaCl vapor Balance N_2
Gas Flow Rate	11.3 L/min
Superficial Gas Velocity	25 cm/s at 800°C
GHSV (gas hourly space velocity)	67,000 h^{-1} at 800°C

Since total NaCl vapor transported in the 880°C series of experiments was substantially higher than that in the 800°C series, the quantity of NaCl sorbed from the 880°C series were recalculated to an 800°C basis and are given in parentheses in row 4 of both tables. These quantities sorbed were used in plots (Fig. 3), allowing sorption performance to be compared on the same basis.

As can be seen in Fig. 3, the amount of NaCl captured by diatomaceous earth is substantially greater than that captured by activated bauxite on a weight basis (i.e., milligrams of NaCl per gram of sorbent). This is due to the activated bauxite being more dense than diatomaceous earth. For all of these experiments, a 1.3-cm-thick sorbent bed was used; therefore, on a volume basis (i.e., mg NaCl/mL sorbent) the quantity of NaCl sorbed by activated bauxite is greater than that of diatomaceous earth.

Figure 3 also shows that the amount of NaCl captured by diatomaceous earth increases with sorbent-bed temperature. This indicates that the reaction between NaCl vapor and diatomaceous earth are endothermic. In contrast to diatomaceous earth, the amount of NaCl sorbed by activated bauxite decreases with increasing sorbent-bed temperature. Since a physical adsorption process is always exothermic, the amount of adsorbate sorbed on an adsorbent must always decrease with increasing temperature according to the principle of Le Chatelier; therefore, the present observation substantiates the conclusion made earlier[17] that NaCl vapor is captured by activated bauxite through an adsorption process. Figure 3 also shows that the amount of sorption of both diatomaceous earth and activated bauxite increases nonlinearly with time. This suggests that under the experimental conditions, neither the reaction rate between NaCl and diatomaceous earth nor the rate of adsorption of NaCl on activated bauxite is controlled by the rate of mass transfer of NaCl vapor from the bulk flue gas to the external surface of the sorbent.[19]

Table 3. Capture of Gaseous NaCl by Diatomaceous Earth as a Function of Temperature and Duration of Exposure

	Experiment[a]							
	HGC-19	HGC-21	HGC-20	HGC-34	HGC-22	HGC-23	HGC-24	HGC-29[b]
Sorbent Bed Temperature, °C	880	880	880	880	800	800	800	800
Exposure Time, h	1	2	3	4.5	1	2	3	6
	Total NaCl, mg							
(1) NaCl Collected by								
(a) Cold Traps	7	27	65	95	5	42	56	182
(b) Glass-Wool Filter	3	15	37	47	2	9	31	107
(2) NaCl Captured by Sorbent[c]	129	296	388	516	124	251	343	544
(3) Total	139	338	490	658	131	302	430	833
(4) Quantity Sorbed, mg NaCl/g sorbent [(2)/15]	8.6 (8.1)[d]	19.7 (17.6)[d]	25.9 (22.7)[d]	34.4 (32.9)[d]	8.3	16.7	22.9	36.3

---

[a] 15.0 g sorbent was tested at atmospheric pressure in a simulated relatively dry PFBC flue gas at a superficial gas velocity of 25 cm/s and GHSV = 67,000 h^{-1}.

[b] The system pressure increased to 146 kPa at the end of the experiment. This was because the glass-wool filter gradually became compacted owing to the high gas flow.

[c] Sodium concentrations of the sorbent were obtained by dissolving representative samples of sorbent and then analyzing the solutions.

[d] Values that would be obtained if the corresponding amounts of NaCl vapor were transported as in the 800°C series of experiments. For example, 8.1 was obtained from $\frac{129}{139} \times \frac{131}{15}$.

Table 4. Capture of Gaseous NaCl by Activated Bauxite as a Function of Temperature and Duration of Exposure

	Experiment[a]							
	HGC-25	HGC-26	HGC-27	HGC-28[b]	HGC-30	HGC-32	HGC-31	HGC-33[b]
Sorbent Bed Temperature, °C	800	800	800	800	880	880	880	880
Exposure Time, h	1	2	3	6	1	2	3	4.5
	Total NaCl, mg							
(1) NaCl Collected by								
(a) Cold Traps	6	22	40	160	16	38	106	180
(b) Glass-Wool Filter	4	10	22	100	6	19	58	103
(2) NaCl Captured by Sorbent[c]	170	343	444	734	203	271	394	489
(3) Total	180	375	506	994	225	328	558	772
(4) Quantity Sorbed, mg NaCl/g sorbent [(2)/15]	5.7	11.4	14.8	24.5	6.8 (5.4)[d]	9.0 (10.3)[d]	13.1 (11.9)[d]	16.3 (15.6)[d]

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[a] 30.0 g sorbent was tested at atmospheric pressure in a simulated relatively dry PFBC flue gas at a superficial gas velocity of 25 cm/s and GHSV = 67,000 h^{-1}.

[b] System pressure increased to 169 kPa at the end of the experiments. This was because the glass-wool filter gradually became compacted owing to the high gas flow.

[c] Sodium concentration of the sorbent was obtained by fusing representative samples of sorbent, and then analyzing the solutions.

[d] Values that would be obtained if the corresponding amounts of NaCl vapor were transported as in the 800°C series of experiments. For example, 5.4 was obtained from $\frac{203}{225} \times \frac{180}{30}$.

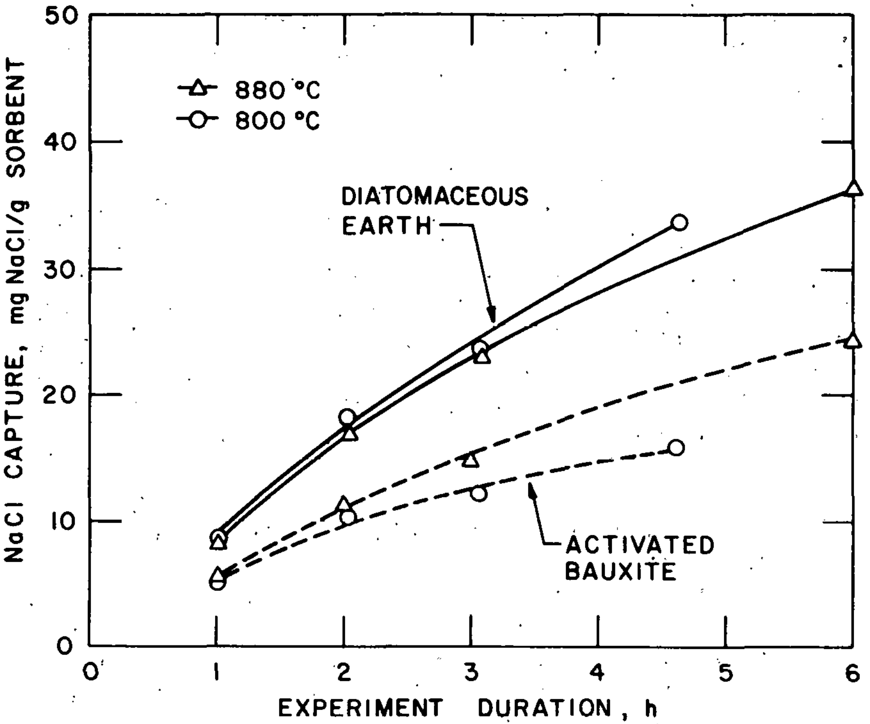

Fig. 3. Effect of Sorbent Bed Temperature on NaCl-Vapor Capture as a Function of Exposure Time

B. Effect of Superficial Gas Velocity at Constant Gas Hourly Space Velocity (GHSV)

In gas-solid reactions such as the system involved in the present study, the effect of the superficial gas velocity of flue gas on the rate of NaCl capture provides another means of measuring the role played by the mass transfer of NaCl vapor from the bulk of the flue gas to the external surface of the sorbent. For mass-transfer-controlled reactions, an increase in superficial gas velocity increases the mass transfer coefficient, thereby increasing the extent of reaction. The superficial gas velocity is also an important parameter when designing a sorber because it determines the cross-sectional area of the bed for a given volumetric flow rate of flue gas.

For these reasons, a series of tests has been conducted to study the effect of superficial gas velocity on the sorption capacity of both diatomaceous earth and activated bauxite. In this series of experiments, sorbent was tested at 800°C and atmospheric pressure in a simulated PFBC flue gas (3% O_2, 16% CO_2, 180 ppm H_2O, 300 ppm SO_2, and the balance N_2) at superficial gas velocities of 25, 66, and 155 cm/s, and at a constant GHSV of 67,000 h^{-1}. Increases in the superficial gas velocity of the flue gas at the sorbent bed were accomplished by reducing the cross section of the sorbent bed, but the amount of sorbent packed was kept constant to maintain a constant GHSV. For each superficial gas velocity, the sorbent was tested at durations of both 2 and 3 h. The NaCl-vapor concentration in the simulated flue gas ranged from 69 to 98 ppm.

Tables 5 and 6 show NaCl-vapor capture by diatomaceous earth and activated bauxite, respectively, as a function of superficial gas velocity. Calculated average rates of NaCl capture (in mg NaCl/g sorbent/h) [row (4) in both Tables 5 and 6, divided by experiment duration] are plotted as a function of superficial gas velocity in Fig. 4.

Within the limits of experimental and analytical errors, the rates of NaCl capture for both diatomaceous earth and activated bauxite remain constant irrespective of the superficial gas velocity, as shown in Fig. 4. These results verify the conclusion (drawn previously) that under the experimental conditions, the rates of NaCl capture by both diatomaceous earth and activated bauxite are not controlled by the mass transfer of NaCl vapor from the bulk of flue gas to the external surfaces of the sorbent. Rather, the rate of NaCl capture is controlled by either diffusion of NaCl vapor through internal pores, adsorption of NaCl vapor on the active sites of the sorbent, or chemical kinetics.

Also shown in Fig. 4 are the rates of NaCl capture for both sorbents calculated from results of experiments HGC-4 and HGC-8 in Table 2 of Ref. 17 with the amounts of NaCl vapor transported revised to conform to the amounts transported in the 25-cm/s series of experiments. For those two experiments, sorbent was tested at 870°C in an air flow at a superficial gas velocity of 8 cm/s and with a GHSV of 3500 h^{-1}. Because the sorbents in those two experiments were not tested under experimental conditions quantitatively comparable to those at high superficial gas velocities, the results are included in the figure for qualitative comparison. It is seen that the rate of NaCl capture is substantially lower at a superficial gas velocity of 8 cm/s than at higher superficial gas velocities. This indicates that at such a low superficial gas velocity, the rate of NaCl capture by these sorbents was probably mass transfer controlled.

C. Effect of GHSV at Constant Superficial Gas Velocity

The gas hourly space velocity (GHSV) is defined as the volumetric flow rate of flue gas in units of sorbent volume per hour. The reciprocal of the GHSV, which is space time, is related to the contact time of the flue gas with the sorbent bed. The GHSV determines the sorbent volume required for a given volumetric flow rate of flue gas to be treated. Both diatomaceous earth and activated bauxite were tested as a function of GHSV at 800°C and a constant superficial gas velocity of 66 cm/s for 2 h. All experiments were carried out at a constant flow rate of flue gas; therefore, GHSV was varied by varying the volume of the sorbent bed. Table 7 shows NaCl-vapor capture by diatomaceous earth and activated bauxite as a function of GHSV. Figure 5 is a plot of the results given in Table 7.

Figure 5 indicates, as expected, that for both sorbents, increased contact time of flue gas with the sorbent bed increases the NaCl-vapor capture. At the fairly short contact time of about 0.2 s in an experiment of 2-h duration, 98.5 and 97% removal of NaCl vapor from hot flue gas by activated bauxite and diatomaceous earth, respectively, can be achieved.

Table 5. Capture of Gaseous NaCl by Diatomaceous Earth as a Function of Superficial Gas Velocity

	Experiment[a]					
	HGC-23	HGC-24	HGC-37	HGC-38	HGC-42	HGC-41R
Exposure Time, h	2	3	2	3	2	3
Superficial Gas Velocity, cm/s	25	25	66	66	155	155
	Total NaCl, mg					
(1) NaCl Collected by						
(a) Cold Traps	42	56	29	52	29	82
(b) Glass-Wool Filter	9	31	14	28	18	52
(2) NaCl Captured by Sorbent[b]	251	343	328	422	332	397
(3) Total	302	430	371	502	379	531
(4) Quantity Sorbed, mg NaCl/g sorbent [(2)/15]	16.7	22.9	21.9 (17.8)[c]	28.1 (24.1)[c]	22.1 (17.6)[c]	26.5 (21.4)[c]

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[a] Sorbent (15.0 g) was tested at 800°C and atmospheric pressure in a simulated PFBC dry flue gas at GHSV = 67,000 h^{-1}.

[b] Sodium concentrations in the sorbent were obtained by dissolving representative samples of the sorbent and then analyzing the solutions.

[c] To allow comparison, these are the values which would be obtained if the NaCl vapor were transported as in the 25-cm/s series of experiments. For example, 17.8 was obtained from $\frac{328}{371} \times \frac{302}{15}$.

Table 6. Capture of Gaseous NaCl by Activated Bauxite as a Function of Superficial Gas Velocity

	Experiment[a]					
	HGC-26	HGC-27	HGC-40	HGC-39	HGC-43R	HGC-44
Exposure Time, h	2	3	2	3	2	3
Superficial Gas Velocity, cm/s	25	25	66	66	155	155
	Total NaCl, mg					
(1) NaCl Collected by						
(a) Cold Traps	22	40	42	62	33	47
(b) Glass-Wool Filter	10	22	25	33	20	27
(2) NaCl Captured by Sorbent[b]	343	444	281	430	351	498
(3) Total	375	506	348	525	404	582
(4) Quantity Sorbed, mg NaCl/g sorbent [(2)/15]	11.4	14.8	9.4 (10.1)[c]	14.3 (13.8)[c]	11.7 (10.9)[c]	16.6 (14.4)[c]

---

[a] Sorbent (30.0 g) was tested at 800°C and atmospheric pressure in a simulated PFBC dry flue gas at GHSV = 67,000 h^{-1}.

[b] Sodium concentrations in the sorbent were obtained by fusing representative samples of the sorbent and then analyzing the solutions.

[c] To allow comparison, these are the values which would be obtained if the NaCl vapor were transported as in the 25-cm/s series of experiments. For example, 10.1 was obtained from $\frac{281}{348} \times \frac{375}{30}$.

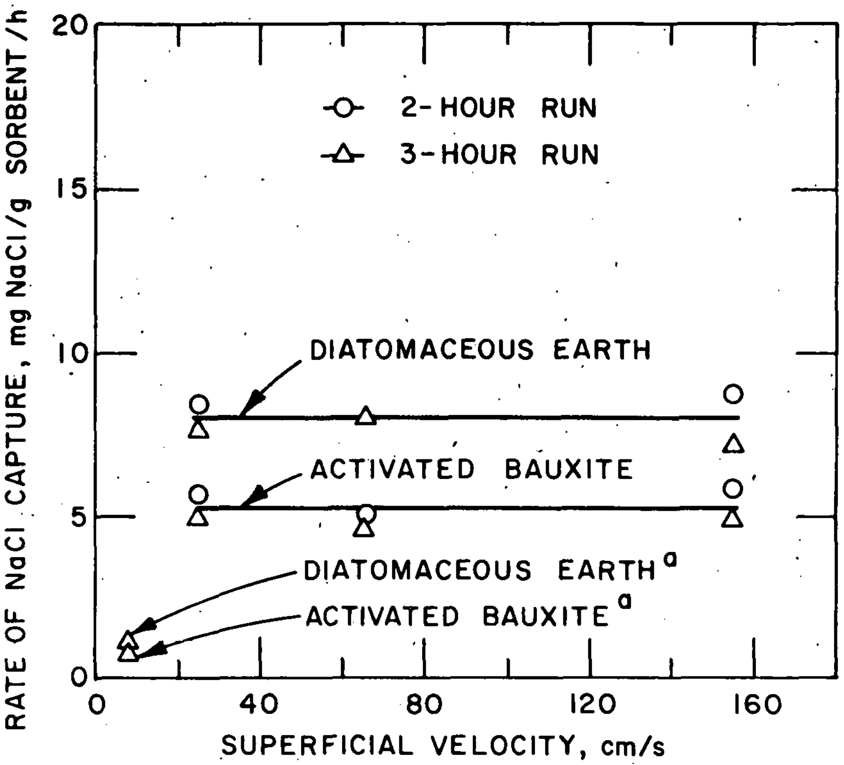

Fig. 4. Effect of Superficial Gas Velocity on Rate of NaCl-Vapor Capture

[a] Data calculated from results of experiments HGC-4 and HGC-8 in Table 2 of Reference 17. Two values were revised to conform to the amounts of NaCl that would be transported in the 25-cm/s series of experiments. In these two experiments, sorbent was tested at 870°C and atmospheric pressure and in an air flow at GHSV = 3500 h^{-1}.

D. Effect of NaCl-Vapor Concentration in Flue Gas

Tests that have been completed were generally conducted using a simulated flue gas of PFBC containing ∿80 ppmV of alkali metal compound vapor. On the basis of experimental measurements[12,20,21] and thermodynamic calculations,[11,22] the alkali vapor concentration in the flue gas in fluidized-bed combustion of coal is expected to be of the order of 5 ppmV, as shown in Fig. 6.

For the purpose of studying the effect of NaCl-vapor concentration in flue gas on sorption performance, a series of experiments were carried out to test these two sorbents in flue gas containing from 24 to 107 ppmV NaCl vapor. Alkali concentrations greater than 5 ppmV were used so that a detectable amount of alkali vapor could be obtained within a reasonable experimental period. The effect was investigated at two gas hourly space velocities (GHSV): 33,500 h^{-1} (or a contact time of 0.11 s) and 18,600 h^{-1} (or a contact time of 0.2 s). The superficial gas velocity of flue gas passing through the sorbent bed was 66 cm/s.

Table 7. Capture of Gaseous NaCl by Diatomaceous Earth and Activated Bauxite as a Function of Gas Hourly Space Velocity (GHSV)

	Experiment[a]				
	HGC-47	HGC-52R	HGC-40	HGC-50	HGC-51
Sorbent	← Diatomaceous Earth →		← Activated Bauxite →		
GHSV, h^{-1} (Contact Time, s)	33,500 (0.11)	18,600 (0.19)	67,000 (0.05)	33,500 (0.11)	18,600 (0.19)
	Total NaCl, mg				
(1) NaCl Collected by					
(a) Cold Traps	29	6	42	19	3
(b) Glass-Wool Filter	18	4	25	11	2
(2) NaCl Captured by Sorbent[b]	292	318	281	348	330
(3) Total	339	328	348	378	335
(4) NaCl Captured by Sorbent, [(2)/(3)] x 100	86%	97%	81%	92%	99%

[a] Sorbent was tested at 800°C, atmospheric pressure, and a superficial gas velocity of 66 cm/s in a simulated PFBC dry flue gas. Duration of each experiment was 2 h.

[b] Sodium content of the sorbent was obtained by dissolving two representative samples and then analyzing the solutions.

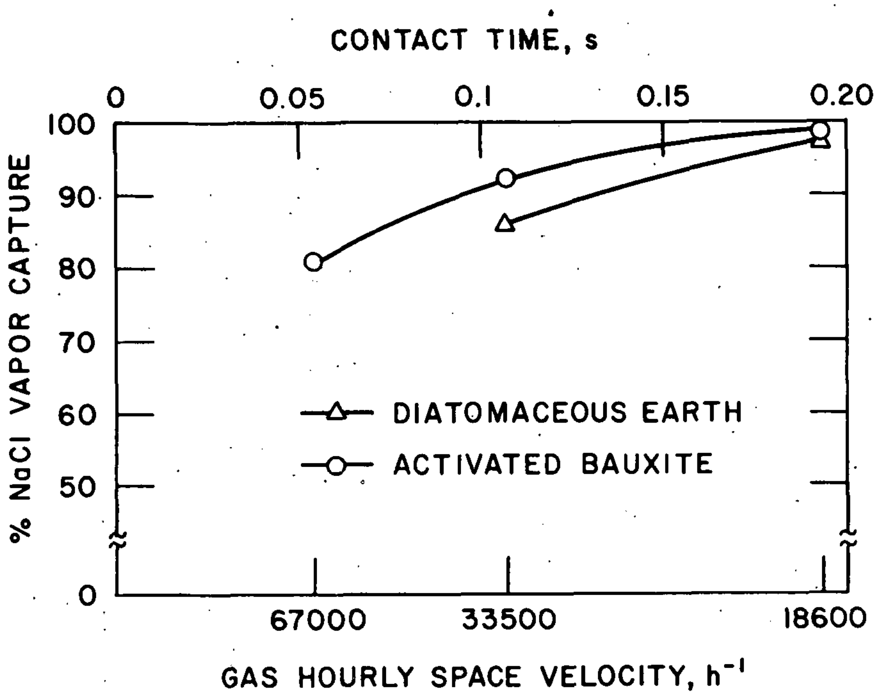

Fig. 5. Effect of GHSV of Flue Gas on NaCl-Vapor Capture

The experimentally measured distribution of NaCl obtained from testing both diatomaceous earth and activated bauxite as functions of NaCl-vapor concentration in flue gas, exposure time, and gas hourly space velocity are shown in Tables 8 and 9, respectively. The results are also plotted in Figs. 7 to 10. Figures 7 and 8 are plots of the average rate of capture of gaseous NaCl in milligrams of NaCl per hour per gram of sorbent, as a function of NaCl concentration in parts per million. In Figs. 9 and 10, the percent NaCl vapor captured is plotted as a function of exposure time (in hours).

As shown in Figs. 7 and 8, the average rates of gaseous NaCl capture by both diatomaceous earth and activated bauxite are linear functions of the NaCl-vapor concentration in the flue gas. This linear relationship was observed for both gas hourly space velocity conditions tested (GHSVs of 18,600 and 33,500 h^{-1}). However, depending upon the NaCl concentration, this linear relationship was observed to hold only for a limited period of exposure time--longer for lower NaCl-vapor concentration.

It is seen in Figs. 9 and 10 that at the NaCl levels used in the flue gas, the efficiency in terms of percent NaCl vapor captured by the sorbent decreases with exposure time. When the NaCl concentration in the flue gas is high, the NaCl capture efficiency is noted to drop rapidly with exposure time; however, when the NaCl concentration is lower, the efficiencies remain at significantly higher levels for longer periods of time than at higher NaCl concentrations. Figures 9 and 10 show that the same levels of efficiency obtained at high NaCl-vapor concentration (∿100 ppm) can also be obtained when the NaCl-vapor concentration in the flue gas is reduced about one-third of the high value, indicating that the sorption efficiencies of the sorbents are

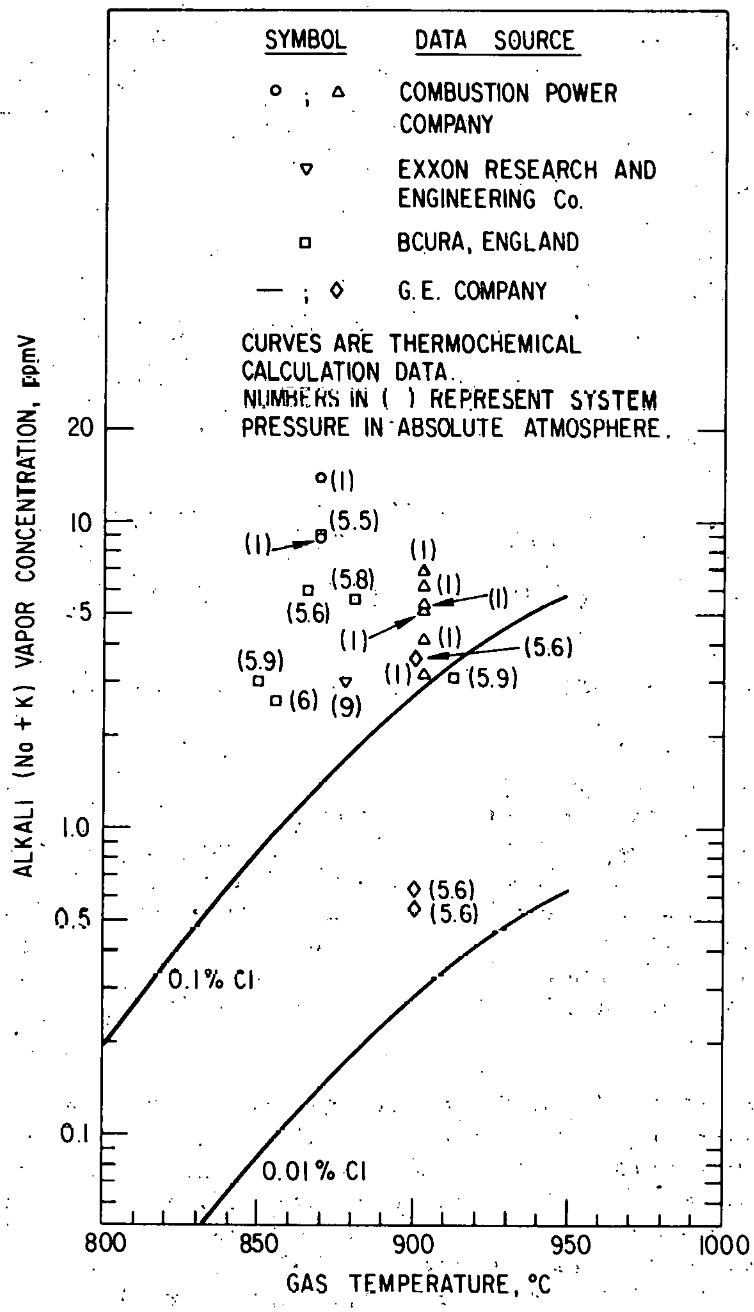

Fig. 6. Alkali-Vapor Concentration in Flue Gas from Fluidized-Bed Combustion of Coal (Ref. 12, 20-23)

Table 8. Capture of Gaseous NaCl by Diatomaceous Earth as a Function of Exposure Time, NaCl Concentration, and Gas Hourly Space Velocity

	Experiment HGC-[a]														
	47	66	65	57	57R	58	58R	56	62	55	52R	68	67	75	74
Exposure Time, h	2	2.5	3.5	2	2	3	3	5	5	8	2	3	5	3	5
Avg. NaCl Conc. in Flue Gas, ppmV	96	97	105	37	24	33	28	27	27	29	93	43	59	34	38
Quantity of Sorbent, g	30	30	30	13	13	13	13	13	13	13	50	22	22	22	22
GHSV, h^{-1}	← 33,500 →										← 18,600 →				
	NaCl, mg														
(1) NaCl Collected by															
(a) Cold Trap	29	58	100	5	3	4	7	7	11	51	6	3	11	3	7
(b) Glass-Wool Filter	18	37	65	2	2	1	3	3	5	22	4	1	4	1	2
(2) NaCl Captured by Sorbent[b]	292	334	483	49	31	66	57	90	89	104	318	93	206	73	132
(3) Total	339	429	648	56	36	71	67	100	105	177	328	97	221	77	141
(4) Rate of Sorption mg NaCl/h-g sorbent	4.9	4.4	4.6	1.9	1.2	1.7	1.5	1.4	1.4	1.0	3.2	1.4	1.9	1.1	1.2
(5) % of NaCl Vapor Captured, [(2)/(3)] x 100	86.1	77.9	74.5	87.5	86.1	93.0	85.1	90	84.8	58.8	97.0	95.9	93.2	94.8	93.6

[a] The sorbent was tested at 800°C and atmospheric pressure in a simulated flue gas of PFBC at a superficial gas velocity of 66 cm/s.

[b] NaCl concentration of the sorbent was measured by dissolving representative samples of the sorbent and then analyzing the solutions.

Table 9. Capture of Gaseous NaCl by Activated Bauxite as a Function of Exposure Time, NaCl Concentration, and Gas Hourly Space Velocity

	Experiment HGC-[a]									
	50	60	59	54	53	51	71	77	78	79
Exposure Time, h^{-1}	2	2	3	5	10	2	5	7	10	15
Avg. NaCl Vapor Conc. in Flue Gas, ppmV	107	33	35	53	43	107	37	31	32	36
Amount of Sorbent, g	60	27	27	27	27	115	50	50	50	50
GHSV, h^{-1}	← 33,500 →					← 18,600 →				
	NaCl, mg									
(1) NaCl Collected by Cold Trap and Glass-Wool Filter	30	6	6	23	123	5	3	7	6	49
(2) NaCl Collected by Sorbent[b]	348	44	72	176	201	372	136	157	236	353
(3) Sum [(1) + (2)]	378	50	78	199	324	377	139	164	242	402
(4) Avg. Rate of Sorption, mg NaCl/h-g sorbent	2.90	0.82	0.89	1.30	0.74	1.62	0.54	0.45	0.47	0.47
(5) % of NaCl Captured	92.1	88.0	92.3	88.4	62.0	98.7	97.8	95.7	97.5	87.8

[a] Sorbent was tested at 800°C and atmospheric pressure in a simulated dry flue gas of PFBC at a superficial gas velocity of 66 cm/s.

[b] Na concentration of the sorbent was obtained by fusing representative samples of the sorbent and then analyzing the solutions.

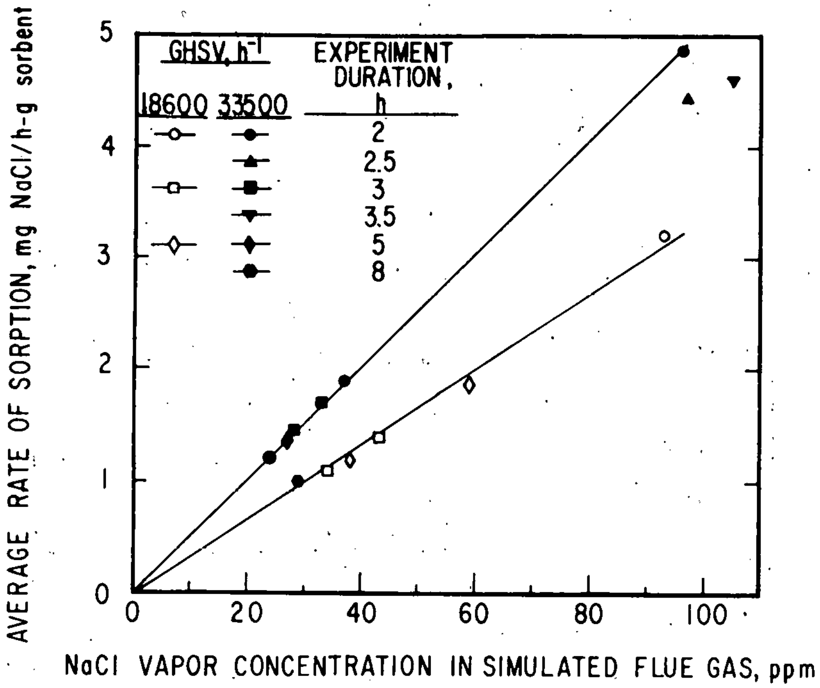

Fig. 7. Average Rate of Capture of Gaseous NaCl by Diatomaceous Earth as a Function of NaCl Concentration

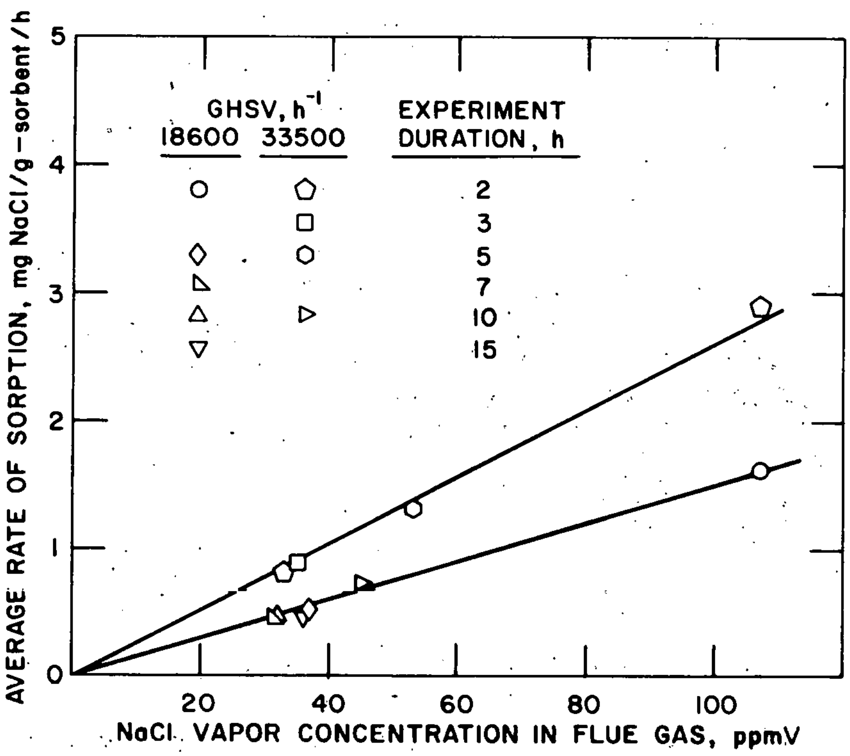

Fig. 8. Average Rate of Capture of Gaseous NaCl by Activated Bauxite as a Function of NaCl Concentration

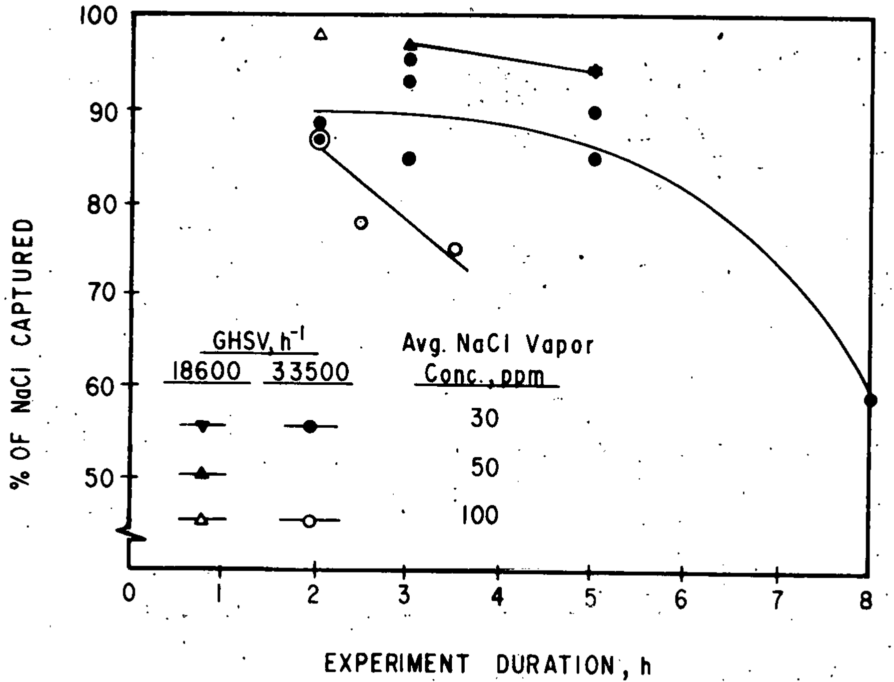

Fig. 9. Effect of NaCl Concentration on the Capture of Gaseous NaCl by Diatomaceous Earth as a Function of Exposure Time

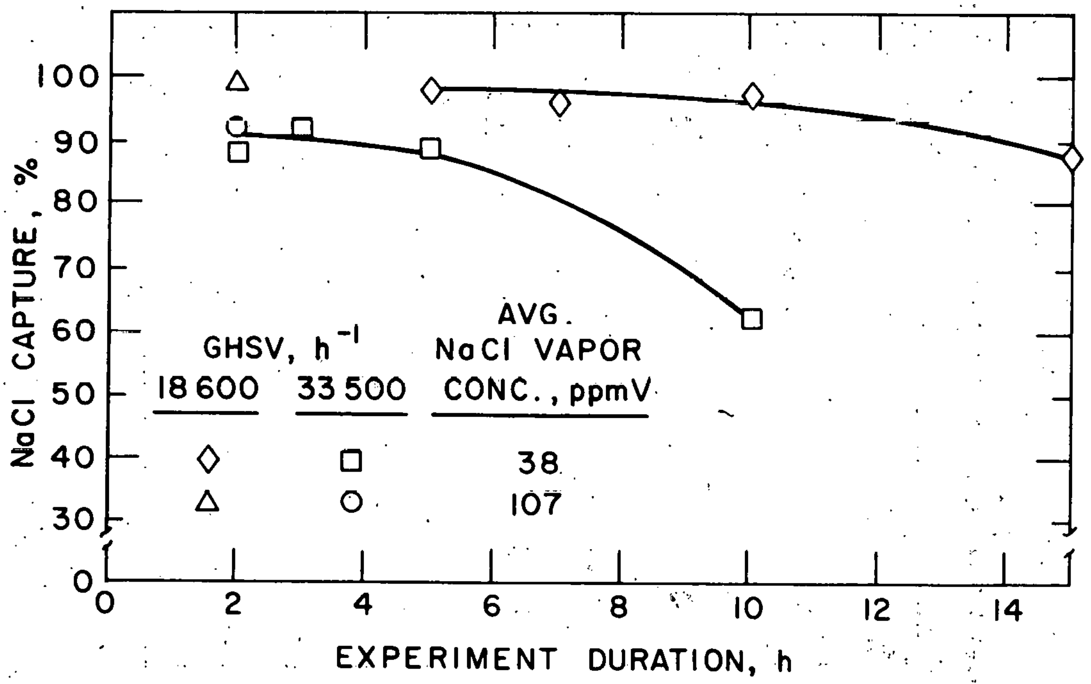

Fig. 10. Effect of NaCl Concentration on the Capture of Gaseous NaCl by Activated Bauxite as a Function of Exposure Time

not reduced by reducing the NaCl-vapor concentration in the flue gas. These data also show that for a given exposure time, when gas hourly space velocity is reduced (i.e., when the contact time of the flue gas and the sorbent bed is increased), the efficiency is substantially increased. This is in agreement with the previous observations (Section IV. C.).

It can be concluded from this series of experiments that the average rates of gaseous NaCl capture by diatomaceous earth and activated bauxite are a function of NaCl-vapor concentration in the flue gas. Within the NaCl concentration range of 30 to ~100 ppmV, the reduction of NaCl concentration in flue gas does not reduce the sorption efficiency of the sorbent.

E. Ultimate NaCl-Vapor Sorption Capacity

Ultimate sorption capacity is defined here as the maximum amount of alkali vapor the sorbent can hold at a given temperature and pressure. A series of tests have been carried out to obtain the ultimate NaCl-vapor sorption capacities of diatomaceous earth and activated bauxite. Tests were conducted using the small-scale sorption test rig (Fig. 2). In this series of tests, a layer of one-sorbent-particulate thick was evenly spread out on the platinum gauze so that each sorbent particle had a maximum and equal chance of being exposed to NaCl-bearing simulated dry flue gas.

Figure 11 shows the plots of sorption capacities (in mg NaCl per gram sorbent) as a function of exposure time (in hours) when -8 +10 mesh sorbent was tested at 800°C and atmospheric pressure for two different average NaCl-vapor concentrations in the flue gas. The superficial gas velocity of flue gas passing through the sorbent layer was 27 cm/s. Under this experimental condition, as has been shown previously (Section IV. B.), the sorption of NaCl vapor by both diatomaceous earth and activated bauxite is not mass-transfer controlled.

As shown in Fig. 11, the NaCl-vapor sorption capacity of activated bauxite increases with exposure time; however, it levels off and approaches an ultimate sorption capacity of about 25 mg/g sorbent. In contrast, the sorption capacity of diatomaceous earth shows a continuously linear increase to 150 mg NaCl/g sorbent and does not show a tendency to level off; therefore, the ultimate NaCl-vapor sorption capacity of diatomaceous earth is greater than 150 mg NaCl/g sorbent.

F. Preliminary Cost Evaluations

The effectiveness of both diatomaceous earth and activated bauxite (alkali sorbents) for removal of alkali metal compounds from hot simulated flue gas of PFBC has been experimentally demonstrated. These two sorbents were evaluated from a practical economic point of view. To allow comparison, the cost of an SO_2-sorbent (Tymochtee dolomite) was also included in this evaluation. Table 10 shows the assumptions and computation base for this preliminary cost evaluation for a 200-MWe PFBC demonstration plant.

On the basis of the assumptions in Table 10, a material balance around a 200-MWe PFBC demonstration plant combustor was made and is shown in Fig. 12. The material balance shows that 206.9 ML/h flue gas at 800°C and 1 MPa

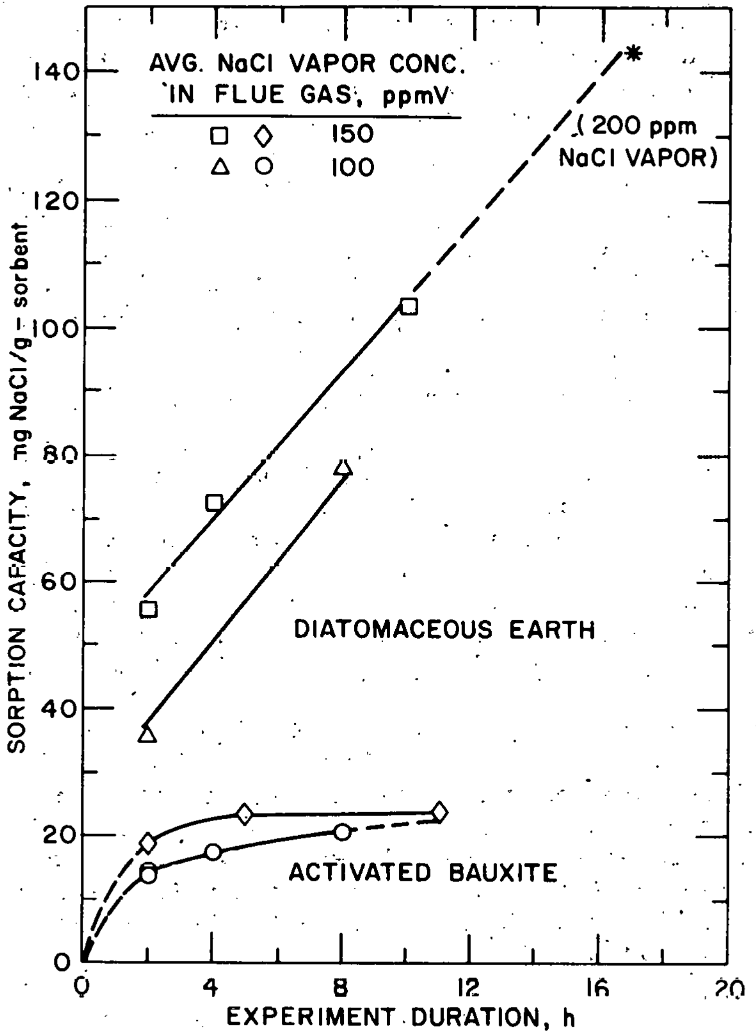

Fig. 11. NaCl-Vapor Sorption Capacities of Diatomaceous Earth and Activated Bauxite as a Function of Exposure Time

(10 atm) will be produced which must be treated for removal of alkali metals. Experimental results showed that both diatomaceous earth and activated bauxite can achieve 97% removal of alkali metals from the flue gas at a gas hourly space velocity (GHSV) of 33,500 h^{-1}. The GHSV is defined as the volumetric flow rate of flue gas per units of sorbent volume per hour. Therefore, the volumetric flow rates of alkali sorbents required to treat 206.9 ML/h flue gas can be calculated. With known bulk densities and current prices of alkali sorbents, as shown in Table 10, the mass flow rate of alkali sorbent needed and the cost of the sorbent can be computed. Table 11 shows the computed results.

As compared with the 0.82-mill cost for SO_2-sorbent per kWh of electricity produced, 0.96 and 1.85 mill would be the costs of using diatomaceous earth and activated bauxite, respectively, to achieve removal of 97% of alkalis from the hot flue gas from a 200-MWe PFBC demonstration plant. Although

Table 10. Assumptions and Computation Base for Preliminary Cost Evaluations of Granular Sorbents for the Removal of Alkali Metal Compounds from Hot Flue Gas of a 200-MWe PFBC Demonstration Plant

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Assumptions for 200-MWe PFBC Demonstration Plant

(1) Coal: Typical Illinois coal with a heating value of 29 MJ/kg of coal (12,500 Btu/lb and an ultimate analysis of 70.3% C, 5.0% H, 1.3% N, 3.3% S, 11.4% ash, and 8.7% O.

(2) SO_2 Sorbent: Tymocthee dolomite containing 51.8% $CaCO_3$ (20.7% Ca) and 43.3% $MgCO_3$; once-through operation.

(3) Excess Air: 20%

(4) Ca/S Mole Ratio for 80% sulfur removal: 1.5

(5) Over-all Cycle Efficiency: 40%

Computation Base for Alkali-Sorbent Evaluation

(1) 97% removal of alkali metal compounds from hot flue gas (800°C). This basis is achievable, based on the experimental results obtained with both diatomaceous earth and activated bauxite at 800°C and atmospheric-pressure operation.

(2) Bulk Densities of Granular Sorbents (ρ given by manufacturer)

ρ_{DE} = 24 lb/ft^3 (385 g/L)
ρ_{AB} = 55 lb/ft^3 (882 g/L)

(3) Current Prices of Granular Sorbents (August 1978)

$80/ton diatomaceous earth
$275/ton activated bauxite
$8/ton SO_2-sorbent (assumed)

(4) Once-through operation for alkali sorbents

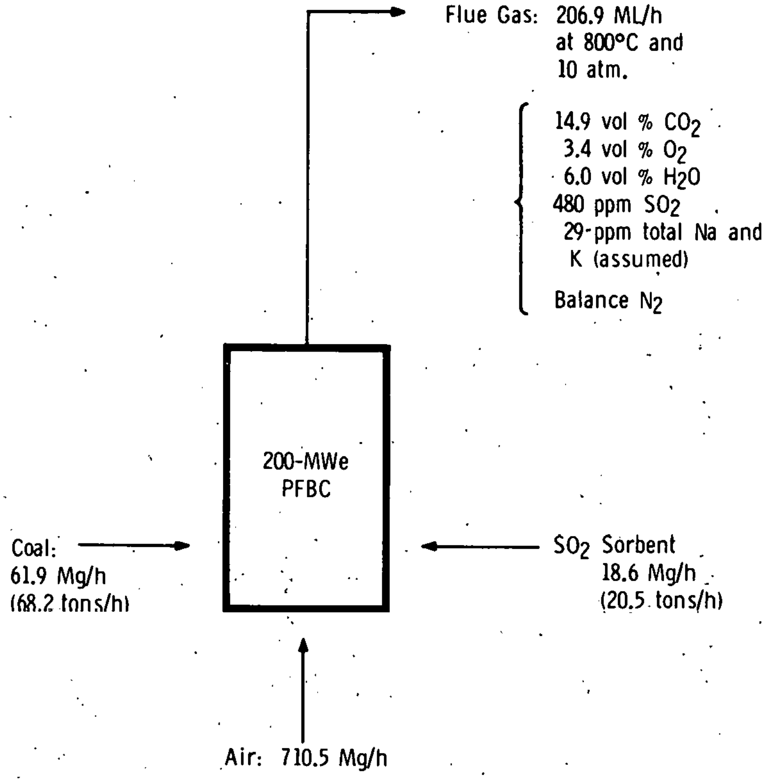

Fig. 12. Material Balance for Conceptual 200-MWe PFBC Demonstration Plant, Based on Assumptions in Table 10.

Table 11. Cost Evaluations of Granular Sorbents for 97%-Alkali and 80%-Sulfur Removals for a 200-MWe Demonstration Plant

Sorbent	Sorbent Required per Ton of Coal Burned, kg	Cost per kg Coal Burned, ¢	Cost per kWh Electricity Produced, mill
Diatomaceous Earth	32	0.31	0.96
Activated Bauxite	40	0.61	1.85
Tymochtee Dolomite	273	0.26	0.82

the cost of using activated bauxite is about twice the cost of diatomaceous earth on a once-through basis, activated bauxite has been demonstrated to be easily and effectively regenerated by a simple water-leaching process;[24] therefore, the cost of using activated bauxite could be significantly reduced. This calculation will be made when more information is obtained about the water-leaching process.

V. SUMMARY

In the prospective application of pressurized fluidized-bed combustion (PFBC) of coal for power generation, corrosion of gas turbine blades due to attack by alkali metal compounds in the hot flue gas is a potential problem. Earlier studies showed that both granular diatomaceous earth and granular activated bauxite, used as filter media in granular-bed filters, effectively remove gaseous alkali metal compounds from the hot (800-880°C) simulated flue gas of PFBCs. To continue this work, studies were performed to obtain the effects of operating variables on the sorption behavior of these two sorbents. In tests, NaCl vapor was transported in a relatively dry simulated PFBC flue gas, using a laboratory-scale, fixed-bed combustor and a high-temperature sorption test rig.

Results of these studies show that the amounts of NaCl-vapor sorbed by diatomaceous earth and activated bauxite (1) increase with the bed temperature for diatomaceous earth, indicating endothermic reactions between the NaCl vapor and diatomaceous earth, but decrease with the bed temperature for activated bauxite, indicating an adsorption phenomenon for NaCl-vapor sorption by the sorbent, and (2) increase with a decrease of the gas hourly space velocity of flue gas passing through the sorbent bed. Greater than 97% removal of NaCl vapor from the flue gas by both sorbents can be achieved at the fairly short contact time of 0.2 s. The rates of NaCl-vapor capture by both diatomaceous earth and activated bauxite are independent of the superficial velocity of flue gas in the range of 25 to 155 cm/s, indicating that the capture rate is not mass-transfer controlled. However, the rates of NaCl-vapor capture increase with increasing NaCl-vapor concentration in flue gas. Within the NaCl-vapor concentration of 30 to 100 ppmV, the reduction in NaCl-vapor concentration in flue gas does not reduce the sorption efficiency for both diatomaceous earth and activated bauxite.

At 800°C and atmospheric pressure, the ultimate NaCl-vapor sorption capacity for activated bauxite is found to be 25 mg NaCl/g sorbent and that for diatomaceous earth to be greater than 150 mg NaCl/g sorbent. Preliminary cost evaluations for using diatomaceous earth and activated bauxite to achieve 97% removal of alkalis from hot flue gas (generated from a 200-MWe demonstration plant with 40% over-all cycle efficiency) gave estimates of 0.31¢ and 0.61¢ per kilogram of coal burned, respectively, on a once-through basis. These costs may be compared with 0.26¢ per kilogram of coal burned for the cost of the SO_2 sorbent. Activated bauxite has been demonstrated to be easily and effectively regenerated by a simple water-leaching process;[24] therefore, the cost of using activated bauxite could be reduced by recycling this sorbent.

VI. CONCLUSIONS AND RECOMMENDATIONS

Tests and cost evaluations of diatomaceous earth and activated bauxite indicate their effectiveness and economic attractiveness as filter media in granular-bed filters, for cleaning up alkali corrodents from the hot flue gas of PFBCs. Data obtained on the effects of several operating parameters on the sorption performance of these two sorbents constitute a significant technical data base for the design of a large-scale granular-bed filter.

It is recommended that parametric studies be continued to complete the technical data base needed for design. It is desired to learn (1) the effects of operating parameters on breakthrough data of alkali vapors through the sorbent bed at elevated pressures, and (2) the pressure drop across the sorbent bed as functions of operating parameters.

ACKNOWLEDGMENTS

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