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MEASUREMENT OF ALKALI-VAPOR EMISSION FROM PRESSURIZED FLUIDIZED-BED COMBUSTION OF ILLINOIS COALS

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ABSTRACT

Two Illinois Herrin No. 6 coals and one Illinois Springfield No. 5 coal were separately combusted in a laboratory-scale (15-cm dia) pressurized fluidized-bed combustor (PFBC) combined with an alkali sorber. These coals were combusted in a fluidized bed of Tymochtee dolomite at temperatures ranging from 910 to 950°C and a system pressure of 9.2 atm absolute. Alkali-vapor emission (Na and K) in the PFBC flue gas was determined by the analytical activated-bauxite sorber bed technique developed at Argonne National Laboratory. The test results showed that sodium is the major alkali-vapor species present in the PFBC flue gas, and that the level of sodium-vapor emission increases linearly with both Na and Cl contents in the coals. This suggests that the sodium-vapor emission results from direct vaporization of NaCl present in the coals. The measured alkali-vapor concentration (Na + K), 67 to 90 ppbW, is more than 2.5 times greater than the allowable alkali limit of 24 ppb for an industrial gas turbine. Combusting these coals in a PFBC for power generation may require developing a method to control alkali vapors.

INTRODUCTION

The pressurized fluidized-bed combustion/gas-turbine combined-cycle system (PFBC/GTCCS)

shows promise for the direct combustion of coal for power generation in a cost-effective, reliable, and environmentally acceptable manner. Several demonstration and commercial plants are already being operated^[1-3]. In PFBC/GTCCS, the high-temperature, high-pressure (HTHP) flue gas exiting from the PFBC combustor is expanded through a gas turbine to recover thermal energy for improved overall cycle efficiency. To protect the gas turbine from erosion and hot corrosion, the entrained ash particulates and alkalis (such as chlorides and sulfates of sodium and potassium) in the PFBC flue gas must be reduced to acceptable levels.

Alkali exists in both coal and the sorbent. In PFBC, alkali can be transported to the gas turbine as particulates and vapors. Several HTHP particulate cleanup devices (such as SiC candle, ceramic membrane cross-flow, and granular-bed filters) have been demonstrated to achieve acceptable particulate reduction for gas turbine systems. However, alkali vapor is still a concern because of its potential penetrability through the particulate cleanup devices. Alkali-vapor emission from coal can be functions of coal and sorbent mineralogy and PFBC operating conditions. The current industrial specification limit for total alkalis in the combustion gas entering a gas turbine is equivalent to 24 ppb at an air/fuel (petroleum distillate) ratio of 50^[4].

Table 1. Chemical Analyses of Coals

	Coal BS		Coal WA		Coal OB	
	As Received	Dry	As Received	Dry	As Received	Dry
Proximate Analysis, %						
Moisture	9.4	--	10.1	--	6.7	--
Fixed Carbon	46.2	51.0	46.1	51.3	46.5	49.8
Volatile Matter	33.7	37.2	30.9	34.3	37.7	40.4
Ash	10.7	11.8	12.9	14.4	9.1	9.8
	100.00	100.00	100.00	100.00	100.00	100.00
Ultimate Analysis, %						
Moisture	9.44	--	10.08	--	6.68	--
C	62.14	68.62	61.98	68.93	69.10	74.05
H	4.18	4.62	3.93	4.37	4.81	5.15
N	1.27	1.40	1.54	1.71	1.55	1.66
Cl	0.09	0.10	0.20	0.22	0.32	0.34
S	3.70	4.09	1.38	1.54	2.50	2.68
Ash	10.69	11.80	12.90	14.35	9.13	9.78
O (Diff.)	8.49	9.37	7.99	8.88	5.91	6.34
	100.00	100.00	100.00	100.00	100.00	100.00
Alkali Analysis, %						
Na		0.07		0.09		0.13
K		0.18		0.39		0.18

The alkali vapor in PFBC flue gas has been experimentally quantified by several institutions over a wide range of PFBC operating parameters. Using a conventional batch-type grab sampling of the flue gas followed by the analysis of the condensate, the alkali vapor concentration was measured as 0.1-10 ppm^[5-7]. The chemical form(s) of these alkali species was not identified. Under the support of the U.S. Department of Energy, an on-line, real-time alkali analyzer has been developed and widely used in the field in recent years^[8,9]. Lee and Carls^[10] recently developed an analytical

activated-bauxite sorber bed technique and measured 1.3-1.5 ppmW sodium and 0.1 ppmW potassium in the flue gas from PFBC of lignite. The sodium material balance showed that 0.24% of the total sodium in lignite was released as vapor species. Greene^[11] suggested that sodium in lignite evolves in a form of fume.

Illinois has the largest bituminous coal reserves in the U.S. These coals generally have a high chlorine content. Chlorine in coal tends to enhance alkali emission during coal combustion^[12]. Fouling and

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In addition to the AASB, an Ames on-line, real-time alkali analyzer and a batch-type alkali and particulate sampling train (APST) were installed to measure the alkali concentration in the sorber inlet gas stream. Unlike the AABS, both these devices employ a heated stainless steel sampling line, despite the unknown extent of the alkali capture by the line^[13].

Analytical Activated-Bauxite Sorber Bed (AASB)

Each AASB (separated into 15 sections by Pt gauze dividers), which is 2.54-cm dia, 38.1-cm long (3-in. dia, 15-in. long), is separately connected to a downstream gas conditioning and control train that contains a quartz-wool filter. The flow rate of the exit gas passing through each AASB is separately regulated and measured by a flowmeter and a wet-test meter. At the end of the test, the activated-bauxite sorbent is collected from each section of the AASB. The downstream line and the filter are rinsed with deionized water to collect any condensed alkalis on the line and the filter. Both the sorbent and the rinse are analyzed for alkalis (Na and K) by atomic absorption. With a known amount of PFBC flue gas flowing through the AASB (as measured by the wet-test meter) and a known amount of total alkali captured, a time-averaged concentration for the vapor-phase alkali in the PFBC flue gas can be determined.

Alkali and Particulate Sampling Train (APST)

A batch-type APST, based on the conventional condensation grab-sampling method, was also installed for measuring alkali vapors. The APST consists of (1) a cold trap, (2) three bubblers, and (3) a pressure gauge and flow control and measurement devices. The alkali vapor is cooled and condensed on the cold trap. At the end of each measuring period (normally 1 h), the cold trap is rinsed with deionized water. The rinse and bubbler solutions are analyzed for alkalis by atomic absorption.

Ames Alkali Analyzer

Under the support of the Department of Energy through Morgantown Energy Technology Center, the Ames alkali analyzer was installed to obtain real-time measurements of alkalis in the PFBC flue

gas. The analyzer is a flame atomic emission spectrometer that has a dual channel, and is designed to determine parts per billion (ppb) concentrations of sodium and potassium in a gas stream. This analyzer has been used in field tests for measuring alkali content in the gas streams of both coal gasification and combustion systems. Details of the analyzer and the field test results are reported elsewhere^[9].

ALKALI MEASUREMENTS

All three tests were conducted under similar operating conditions, as shown in Table 2. A total of 46.5, 35, and 49.5 h of steady-state combustion was accumulated for the tests of Coal BS, Coal WA, and Coal OB, respectively.

Ames Alkali Analyzer

The PFBC flue gas was continuously monitored for its alkali concentration by the Ames alkali analyzer through a Type 304 stainless steel sampling line that is 1-m long, 0.635-cm OD (3.28-ft long, 1/4-in. OD). The line was maintained at 800-900°C by using electrical heating rods to prevent the possible condensation of alkali vapors in the line. Figure 3 shows typical alkali results measured by the Ames alkali analyzer for tests of Coal BS and Coal WA. The quick drops in alkali concentrations shown in the figure were in response to the sharp decrease in the sampling-line temperature caused by temporary power termination to the sampling-line heaters. This effect suggests that the measured alkalis are either present as (1) vapors that condensed out on the sampling line as the line cooled or (2) sub-micron particulates that penetrated through the SiC candle filters and deposited on the cooled sampling line due to a thermophoresis effect (small particles in a temperature gradient are driven from the high- to low-temperature regions). The sharp peaks shown in Fig. 3 are attributed to the fly ash dusts generated by the dumps of the bed overflow and the cyclone ash collection hoppers. The alkali concentrations measured by the Ames alkali analyzer were in the range of 30-50 ppbW sodium and 10-30 ppbW potassium for Coal BS, 50-80 ppbW sodium and 20-80 ppbW potassium for the Coal WA, and 30-50 ppbW sodium and 10-30 ppbW potassium for Coal OB.

Table 2. Nominal Operating Conditions of PFBC/Alkali Sorber Facility and AASB

<u>Combuster</u>			
Sorbent	Tymochtee Dolomite		
Avg. bed temperature, °C	910-950		
System pressure, atm	9.2		
Fluidizing velocity, m/s	0.91		
Expanded bed height, cm	81.3		
<u>Alkali Sorber System</u>			
Sorber bed material	Activated Bauxite		
Sorber vessel interior temperature, °C	850		
<u>AASB</u>	<u>Coal BS</u>	<u>Coal WA</u>	<u>Coal OB</u>
Steady-state on-stream duration, h	46.5	35	49.5
Superficial gas velocity ^a , cm/s	48.0	52.7	35.5
Gas hourly space velocity ^b , h ⁻¹ (space time, s)	4540 (0.79)	4980 (0.72)	3360 (1.07)
Total amount of flow passing through the bed, g	122,000	100,640	106,290

^aRepresents the velocity of PFBC flue gas passing through the empty bed at operating conditions.

^bRepresents the volumetric flow rate of PFBC flue gas in units of bed volume per hour at operating conditions.

Alkali and Particulate Sampling Train

The batch-type APST was also installed through a type 304 stainless steel sampling line that is ~ 1-m long, 0.635-cm OD (3.28-ft long, 1/4-in. OD). The line was also maintained at 800-900°C by electrical heating rods. Table 3 summarizes the results of the APST measurements for tests of Coal BS and Coal WA. The measured alkali concentrations for the test of Coal WA were consistently higher than those of Coal BS. This trend agrees with the results obtained by the Ames alkali analyzer. The APST measurement for the test with Coal OB was not successful, probably due to contamination of the cold trap.

Analytical Activated-Bauxite Sorber Bed

Only one AASB was analyzed for its alkali (Na and K) capture for each coal test. The results (Table 4) include only the water-soluble alkalis (from water leaching) because the amount of acid-soluble alkalis in water-leached sorbent is negligible.

After each test, a thin layer of brownish ash particles had accumulated on the first upstream section of AASB. This is probably the result of a minor leak in the seal between the activated-bauxite sorber tube and the tube sheet. Because of this ash contamination, the sodium and potassium contents of the first section are exceptionally high compared to the rest of the bed sections, as shown in Table 4. Thus the amount of alkali-vapor capture by this

Fig. 3. Sodium and Potassium Concentrations In PFBC Flue Gas Measured by Ames Alkali Analyzer

Table 3. Alkali Measurement by APST

Measure- ment No.	Alkali Concentration, ppbW			
	Coal BS		Coal WA	
	Na	K	Na	K
1	13 ± 5	8 ± 5	90 ± 10	54 ± 10
2	16 ± 6	10 ± 6	58 ± 9	67 ± 9
3	30 ± 9	12 ± 9	44 ± 8	44 ± 8
4	18 ± 7	<5 ± 5	94 ± 9	37 ± 9
5	14 ± 10	<5 ± 5	N.A.	N.A

section was corrected for the alkali contribution from the ash contamination. As illustrated in Fig. 4, this was done by (1) plotting the amount of alkali-vapor capture in each section as a function of the bed length and (2) extrapolating the least-square-fit

curve to intercept the y-axis. The intercept value is considered to be a reasonable amount of alkali vapor being captured by the first section of AASB.

Fig. 4. Plots of Sectional Alkali Content in AASB for Test of Coal WA

Table 4 shows that the last section of the AASB captured an appreciable amount of sodium, suggesting the breakthrough of the vapor-phase sodium from the AASB. Any alkali vapor that breaks through the AASB is cooled through the gas transport line, condensed to form aerosols, and captured by the quartz-wool filter. The amount of sodium captured by the gas-transport line and the quartz-wool filter is also given in Table 4. From the known amount of total flow passed through the AASB (shown in Table 2) and the known amount of total vapor-phase alkalis captured by both the AASB and the downstream gas conditioning and control train, we calculated the time-averaged alkali-vapor concentration in the PFBC flue gas (after being cleaned of ash particulates by the hot cyclone and SiC candle filters with >99.9% particulate removal

TABLE 4. Alkali Analysis for Each Section of AASB

Section No.	Amount of Alkali Capture per Section ^a , μg					
	Coal BS		Coal WA		Coal OB	
	Na	K	Na	K	Na	K
1	1920 ± 50 (760 ± 5) ^b	2740 ± 80 (280 ± 5) ^b	1860 ± 60 (660 ± 5) ^b	2180 ± 60 (300 ± 5) ^b	1570 ± 10 (840 ± 10) ^b	1090 ± 10 (240 ± 10) ^b
2	655 ± 4	188 ± 4	573 ± 4	221 ± 4	780 ± 10	140 ± 10
3	484 ± 5	100 ± 4	625 ± 4	161 ± 4	720 ± 10	130 ± 10
4	667 ± 4	71 ± 4	530 ± 4	119 ± 4	710 ± 10	90 ± 10
5	524 ± 4	55 ± 4	471 ± 4	102 ± 4	670 ± 10	50 ± 10
6	517 ± 4	63 ± 4	450 ± 4	122 ± 4	640 ± 10	60 ± 10
7	537 ± 4	61 ± 4	415 ± 4	23 ± 4	570 ± 10	50 ± 10
8	360 ± 4	71 ± 4	401 ± 5	33 ± 4	590 ± 10	0
9	529 ± 5	3 ± 4	405 ± 5	34 ± 4	570 ± 10	0
10	417 ± 4	21 ± 4	451 ± 5	0	490 ± 10	0
11	278 ± 5	17 ± 4	331 ± 5	0	450 ± 10	0
12	320 ± 5	0	249 ± 4	0	420 ± 10	0
13	306 ± 5	0	300 ± 4	0	360 ± 10	0
14	236 ± 4	0	269 ± 4	0	350 ± 10	0
15	283 ± 5	0	163 ± 4	0	310 ± 10	0
Sum (vapor only)	6870 ± 70	930 ± 50	6290 ± 70	1120 ± 40	8470 ± 150	760 ± 70
Alkali vapor captured by gas-transport line and quartz-wool filter, μg	300 ± 10	0	360 ± 10	0	330 ± 20	0
Total alkali vapor captured, μg	7170 ± 80	930 ± 50	6650 ± 80	1120 ± 40	8800 ± 170	760 ± 70
Time averaged alkali-vapor concentration in PFBC flue gas ^c , ppbW	59 ± 1	8 ± 1	66 ± 1	11 ± 1	83 ± 2	7 ± 1

^aResults have been corrected for background alkalis in the solvent.
^bEstimated amount captured from vapor-phase alkalis after corrected for (1) the alkali contribution from fly ash captured by the section, as shown in Fig. 4, and (2) the background alkalis.
^cObtained by dividing the total alkali-vapor captured by the total amount of flow through the bed, which is given in Table 2.

efficiency). As shown in the last row of Table 4, this concentration was determined to be 59 ± 1 ppbW sodium and 8 ± 1 ppbW potassium for Coal BS, 66 ± 1 ppbW sodium and 11 ± 1 ppbW potassium for Coal WA, and 83 ± 2 ppbW sodium and 7 ± 1 ppbW potassium for Coal OB. These alkali-vapor concentrations are more than 2.5 times greater than the currently suggested alkali specification limit of 24 ppb for an industrial gas turbine. The data suggest that the alkali vapors may need to be controlled if these Illinois coals are to be used in a PFBC/GTCCS for power generation.

The sodium-vapor concentrations measured by the AASB for the coals BS and OB are significantly higher than those measured by the Ames alkali analyzer or APST. This discrepancy can be attributed, at least in part, to the capture of sodium vapor by the stainless steel sampling line used in the Ames alkali analyzer and the APST.^[13] The AASB used no sampling line. Somewhat unexpectedly, the sodium-vapor concentrations measured by the AASB for coal WA were similar to those measured with the other two techniques. This finding can be explained by the presence of ash dust in the sorber inlet gas stream for the measurements with the Ames analyzer and APST (for the AASB, the ash dust was captured in the first bed section). The dust presence in the gas stream is indicated by the significantly higher potassium levels measured by the Ames analyzer and the APST for the test of Coal WA, which has more than twice the potassium content compared with Coal BS or OB (see Table 1). The source of the dust was probably a leak in the seal between the activated-bauxite sorber tube and the tube sheet. The high dust content contributed to the higher sodium and potassium concentrations measured by the Ames analyzer and APST for the test of coal WA.

RELATIONSHIP OF SODIUM-VAPOR EMISSION WITH SODIUM AND CHLORINE CONTENTS

Sodium is the major vapor-phase alkali (Na and K) species emitted during the PFBC of these three Illinois coals. This is not surprising because potassium in coal occurs largely as non-volatile aluminosilicates. The distribution and mode of occurrence of sodium and chlorine in Illinois Basin coals (including Herrin No. 6 and Springfield No. 5

seams) have been well studied and documented by researchers at the Illinois State Geological Survey^[14,15]. In Illinois coals, portions of Na and Cl occur as NaCl dissolved in pore water contained in micro- and meso-pores. The concentration of this salt may be controlled by a common geological factor, such as salinity of groundwater. The remaining sodium in coal is present as non-volatile clay minerals, while the remaining chlorine occurs as chloride ions (Cl⁻) adsorbed on the inner surfaces of the micropores in coal macerals^[15]. The three coals studied in this work show a linear relationship between Na and Cl contents (Fig. 5.), suggesting that portions of the total Na and Cl are associated as NaCl. Sodium chloride is volatile and is known to have an appreciable vapor pressure (0.4 kPa or 3.0 mm Hg at 900°C) in the PFBC temperature range.

Fig. 5. Relationship of Sodium and Chlorine Contents in Coal

In Fig. 6, the sodium-vapor emission data in Table 4 correlated with Na and Cl contents in the three coals. Sodium-vapor emission from the PFBC shows a linear relationship with the Na and Cl contents. This relationship suggests that the sodium-vapor emission results from vaporization of the NaCl in these coals.

Sodium chloride vapor can react with SO_2 in the gas phase to form condensed Na_2SO_4 nuclei:

$2 \text{NaCl} + \text{SO}_2 + 1/2 \text{O}_2 + \text{H}_2\text{O} = \text{Na}_2\text{SO}_4 + 2 \text{HCl}$
The sulfate nuclei can be readily adsorbed on ash particles. The measured sodium vapor could represent the thermo-dynamically equilibrated or kinetically limited amount of the reaction. More study is needed to verify this.

Fig. 6. Sodium-Vapor Concentration as Function of Sodium and Chlorine Contents in Coal

CONCLUSION

Conclusions from this work are as follows:

- (1) Sodium is the major alkali-vapor species (Na and K) present in the PFBC flue gas of these three Illinois coals.
- (2) Sodium-vapor emissions from these tests showed linear relationship with both Na and Cl contents in the coals. These coals also showed a linear relationship between their Na and Cl contents, which suggests that portions of the Na and Cl are associated (e.g., as NaCl). The sodium-vapor emission could have resulted from direct vaporization of NaCl.

- (3) The time-averaged alkali-vapor concentration (Na + K) ranges from 67 ± 2 to 90 ± 3 ppbW, which is more than 2.5 times greater than the allowable alkali limit of 24 ppb for an industrial gas turbine. A method to reduce these alkali vapors may be needed if these coals are to be used in PFBC/GTCC for power generation.

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