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FUNDAMENTAL MECHANISMS IN FLUE GAS CONDITIONING

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INTRODUCTION

This project is divided into four tasks. We developed our Management Plan in Task 1. Task 2, Evaluation of Mechanisms in FGD Sorbent and Ash Interactions, focuses on characteristics of binary mixtures of these distinct powders. Task 3, Evaluation of Mechanisms in Conditioning Agents and Ash, is designed to examine effects of various conditioning agents on fine ash particles to determine mechanisms by which these agents alter physical properties of ash. We began Tasks 2 and 3 with an extensive literature search and assembly of existing theories. We completed this phase of the project with publication of two special Topical Reports. During the past quarter we continued our analyses of the samples in our database. We will be continuing these laboratory analyses through the next two quarters. Pilot-scale evaluations will follow these laboratory investigations. We will include the results of laboratory and pilot-scale work performed under Tasks 2 and 3 in a Flue Gas Conditioning Model that we will issue under Task 4. The Final Report for the project will also be prepared under Task 4.

Task 2 and Task 3 each include several groups of experiments. Each experiment has a title and a corresponding number. We described all of these experiments and tests that are included under each experiment (except Experiment 3.5, which is discussed in this report) in our fourth Quarterly Report (April - June 1992). In our literature reviews reported in Topical Reports 1 and 2, we emphasized the roles adsorbed water can have in controlling bulk properties of powders. The experiments we are performing are designed to define the extent to which water affects key properties of ashes, powders, and mixtures of sorbents and ashes. We have included selected data from prior quarterly reports in this report for the sake of completeness, and for comparisons with newly-acquired data. In several places in this report samples are referred to by their identification (SID) numbers. Table 1 provides a brief description of all the samples we have used in this project along with their SID numbers.

Table 1
Identification of Samples

	SID	Description of Sample
SORBENTS	2870	Muskingum base line ESP hopper ash
	2871	DITF dry $\text{Ca}(\text{OH})_2$ injection
AND	2872	DITF $\text{Ca}(\text{OH})_2$ slurry injection
	2873	DITF $\text{Ca}(\text{OH})_2$ slurry injection with CaCl_2 addition
BASE LINE	2884	Kintigh base line ESP hopper ash
	2885	spray dryer ash (High Sulfur Test Center)
ASHES	2883	Edgewater furnace $\text{Ca}(\text{OH})_2$ injection
	2890	ADVACATE injection
DUST CAKE	2101	Scholz dust cake ash
	2029	Monticello dust cake ash
ASHES	2515	Harrington dust cake ash
	1994	AFBC dust cake ash
LEACHED	2101-W	leached Scholz dust cake ash (first set)
	2029-W	leached Monticello dust cake ash (first set)
DUST CAKE	2515-W	leached Harrington dust cake ash (first set)
	1994-W	leached AFBC dust cake ash (first set)
ASHES	2101-L	leached Scholz dust cake ash (second set)
	2029-L	leached Monticello dust cake ash (second set)
	2515-L	leached Harrington dust cake ash (second set)
	1994-L	leached AFBC dust cake ash (second set)
POWDERS	1856	glass beads
	3063	silicon dioxide (Cabosil L-90)
	3064	silicon dioxide (Cabosil M-5)
	3065	silicon dioxide (Cabosil EH-5)
	3238	aluminosilicate (Neutralite)
	2769	silicon dioxide (Kenite 900)
	2874	alumina powder

SUMMARY

Our recent laboratory efforts were primarily directed toward the determination of the effects of adsorbed water on the tensile strength of powders and the development of apparatus for Experiment 3.5. As suggested by our literature review, our data indicate that water adsorption depends on particle morphology and on surface chemistry. Our measurements of tensile strength show that, for many of the samples we have analyzed, a relative minimum in tensile strength exists for samples conditioned and tested at about 30% relative humidity. Under Experiment 3.5, which began during this last quarter, we have been developing a system capable of conditioning selected samples with $(\text{NH}_4)_2\text{SO}_4$, NH_4HSO_4 , CaCl_2 , organosiloxane, and SO_3 . Details of this experiment are discussed under section 3.5.

Outside of the laboratory, we also prepared two papers for presentation at symposia in June and July of this year. We have prepared and submitted draft and final manuscripts entitled "How Particulate Properties Affect the Collection of Fine Particles" to the Air and Waste Management Association for review. We will make a presentation of project results at their 86th Annual Meeting and Exhibition which will be held in Denver on June 13-18, 1993. We have also prepared a paper entitled "Continuing Work on the Fundamental Mechanisms in Flue Gas Conditioning" that will be presented at the Ninth Annual Coal Preparation, Utilization and Environmental Control Contractor's Conference sponsored by the Department of Energy's Pittsburgh Energy Technology Center in Pittsburgh on July 19-22, 1993.

TASK 2: ANALYSES OF MIXTURES OF SORBENTS AND ASHES

E 2.1 Standard Characterization of Mixtures of Sorbents and Ashes

During the past quarter, we began analyses on a sample of ash from the injection of recycled ADVACATE material from tests conducted at SRI's one million-Btu/hr pilot-scale coal combustor (SID 2890). The sample was collected from the hopper of the pulse-jet baghouse serving the combustor. SEM pictures of the ADVACATE injection ash are shown in Figure 1. As with the other mixtures of sorbents and ashes, this sample apparently contains a mixture of relatively smooth, spherical ash particles and irregularly-shaped sorbent particles. Cumulative and differential size distributions of this sample are presented in Figure 2. The specific surface area of this sample is $15.1 \text{ m}^2/\text{g}$, and its true density is $2.45 \text{ g}/\text{cm}^3$. The size distribution of this ash is shown in Figure 2. Its Stokes' MMD was determined to be $9.4 \text{ }\mu\text{m}$. The chemical characteristics of this sample are included in Table 2.

a

b

c

d

Figure 1. SEM photographs of the mixture of ADVACATE sorbent and ash (SID 2890). These photographs were taken at magnifications of a) 100X, b) 500X, c) 1000X, and d) 5000X.

Figure 2. Cumulative and differential size distribution data measured with the Shimadzu Centrifugal Particle Size Analyzer for the mixture of ADVACATE sorbent and ash (SID 2890). The MMD of this ash was determined to be 9.4 μm .

Table 2
Chemical Characteristics of Ashes and Sorbent and Ash Mixtures, % wt.*

SID	2870	2871	2872	2873	2884	2885	2883	2890
LiO ₂	0.05	0.02	0.02	0.03	0.03	0.01	0.01	0.02
Na ₂ O	0.57	0.27	0.46	0.59	0.62	0.67	0.17	0.62
K ₂ O	1.9	0.83	0.87	0.97	1.6	0.48	0.63	1.6
MgO	1.3	0.9	1.1	1.0	0.8	0.6	1.0	0.88
CaO	2.1	48.6	38.5	31.5	3.0	36.5	46.9	17.3
Fe ₂ O ₃	20.7	8.2	8.0	9.1	20.9	4.1	10.5	12.1
Al ₂ O ₃	22.9	9.9	9.7	11.1	24.1	7.0	7.9	17.8
SiO ₂	48.7	20.9	20.0	23.1	46.9	12.4	16.2	37.4
TiO ₂	1.0	0.6	0.5	0.5	0.9	0.3	0.40	0.67
P ₂ O ₅	0.20	0.09	0.11	0.10	0.46	0.19	0.08	0.12
SO ₃	0.33	9.1	20.3	21.1	0.23	36.0	13.9	12.1
LOI	1.1	12.5	12.3	6.1	4.9	6.2	13.6	15.2
soluble SO ₄ ⁼	0.78	10.4	24.2	25.3	0.76	43.8	17.4	14.2
Equilibrium pH	5.8	11.3	11.7	11.0	9.1	10.5	11.3	9.5

* Equilibrium pH is a dimensionless quantity.

The ADVACATE ash has a general chemical makeup like those of the other mixtures of ashes and sorbents. However, it has a higher specific surface area, has higher value for loss-on-ignition (LOI), and is less alkaline, than the other mixtures of sorbent and ash. Ongoing measurements under Experiments 2.2 and 2.3 will help determine the effects these differences may have on the behavior of this sample.

E 2.3 Characterization of Water-Conditioned Sorbent and Ash Mixtures

Tensile strength determinations were performed on samples that had been conditioned at a variety of relative humidities. Results of these measurements are included in Table 3.

Table 3
Tensile Strengths of Ashes and Mixtures of Sorbents and Ashes
from Task 2 Studies as a Function of Relative Humidity, N/m²

SID	-Relative Humidity used to Condition and Test Samples, %							
	1	11	22	34	50	75	83	93
2870				1.1	1.6		> 30	
2871	> 32	> 32	24	3.1	3.7		3.4	8.0
2872				> 28	> 9.5		15	> 24
2873				> 18	> 6.8		> 14	
2884				0.6	0.6		0.50	
2885	> 30	18		2.8	4.2		6.8	12
2883				31			> 23	
2890	> 32		13	11				> 32

Like many of the other ashes and powders we have tested, the ADVACATE ash has higher tensile strength in very dry or very moist environments than at moderate relative humidities. Based on the measurements we have made, its minimum tensile strength is the greatest of all of the mixtures of sorbents and ashes from low-temperature applications that we have tested. Additional tensile strength measurements that we have planned may elucidate this behavior.

TASK 3: ANALYSES OF ASHES AND POWDERS

Measurements made under Task 3 during the past quarter were limited to selected tensile strength determinations. Experiment 3.5 consumed the majority of effort under Task 3 during the past quarter.

E 3.3 Characterization of Water-Conditioned Powders and Fly Ashes

Table 4 presents a summary of all tensile strength measurements performed under Experiments 3.3 and 3.4. Except for the diatomaceous earth (SID 2769), and the leached and unleached AFBC dust cake ashes (SID's 1994-W and 1994), the tensile strength of each sample is at a minimum at a moderate value of relative humidity. Tensile strength increases as humidity is either increased or decreased from this condition. (The three samples listed above may also

have this same behavior, but our measurements have not been fully successful on these samples.)

Table 4
Tensile Strengths of Ashes and Mixtures of Sorbents and Ashes
from Task 3 Studies as a Function of Relative Humidity, N/m²

	-Relative Humidity used to Condition and Test Samples, %							
SID	1	11	22	34	50	75	83	93
2101	11	4.7		1.5	5.1	5.4	28	
2029	> 32	19		4.1	8.2	8.5	8.5	12
2515		> 32	> 32	19	15	> 26	30	
1994				> 13	> 7.7			
2101-W	15	6.3		1.8	2.8		3.9	3.1
2029-W	> 30	15		3.1	3.2	3.6	3.8	6.1
2515-W	> 32	8.8		2.0	1.6	2.5	3.4	3.8
1994-W				> 14	> 9.5			
1856	7.9	3.6		0.6	0.8	> 27	32	
3238	> 30	4.9		2.0	1.4		1.9	3.4
2769				> 6.6	> 4.4			
2874	3.3	1.5		0.8	0.8		1.2	

E 3.5 Determination of the Effects of the Deposition of Chemical Compounds on Particle Surfaces

Experiment 3.5 was designed to determine if the characteristics of ashes or powders can be significantly altered by the surface deposition of soluble salts, silanes, or SO₃. These compounds will be deposited on the surfaces of the ash or powder particles, and key characteristics of these conditioned materials will be measured. Potential effects of these compounds include changes in appearance, surface area, affinity for adsorption of water, and tensile strength. The results of these analyses will be compared with analyses performed on unconditioned samples under Experiments 3.1, 3.2, 3.3, and 3.4.

Table 5 details the division of Experiment 3.5 into three parts based on the type of compound to be deposited on the particles. Table 6 identifies which samples will be tested under Experiment 3.5.

Table 5
Areas of Investigation under Experiment 3.5

Experiment/Area	compound(s) to be deposited
3.5.1	soluble salts: $(\text{NH}_4)_2\text{SO}_4$, NH_4HSO_4 , and CaCl_2
3.5.2	organosiloxane (Trade name Prosil 28; proprietary formula)
3.5.3	SO_3

Table 6
Samples to be Tested under Experiment 3.5

DETERMINATION OF THE EFFECTS OF THE DEPOSITION OF CHEMICAL COMPOUNDS ON PARTICLE SURFACES				
		sample to be tested under		
SID	Description of sample	E 3.5.1	E 3.5.2	E 3.5.3
2101	Scholz dust cake ash			X
2029	Monticello dust cake ash			X
2515	Harrington dust cake ash			X
1994	TVA 20 MW AFBC dust cake ash			X
2101-L	leached Scholz dust cake ash	X	X	X
2029-L	leached Monticello dust cake ash	X	X	X
2515-L	leached Harrington dust cake ash	X	X	X
1994-L	leached TVA 20 MW AFBC dust cake ash	X	X	X
1856	glass beads	X	X	X
3238	aluminosilicate (Neutralite)	X	X	X
2769	silicon dioxide (Kenite 900)	X	X	X
2874	alumina powder	X	X	X

Work began with the assembly of a system designed to deposit the compounds on particle surfaces. Our plan was to deposit the soluble salts and organosiloxane with a spray drying

system. Our efforts were concentrated on establishing a constant, reliable feed of the slurry of ash particles suspended in water. We have tried three different devices for spraying the slurry. Although each of the sprayers (a Paasche air brush, a Binks air brush, and a Colison nebulizer) did create a suitably fine spray, only the Colison nebulizer was able to run unattended for a significant period of time. The air brushes, which produced finer sprays than the nebulizer, would clog up after 10 to 15 minutes. The nebulizer sprayed slurry continuously for up to six hours. We placed the nebulizer in a spraying chamber and passed the fine droplet particles through a diffusional dryer for final collection in a ceramic thimble filter. We operated the nebulizer with the ash slurry for about 12 to 14 hours. Although the fine droplet particles produced with this technique entered the diffusional dryer, we saw no evidence that ash particles were carried into the ceramic thimble filter.

The system for deposition of SO_3 on particle surfaces will probably consist of a flowing stream of SO_3 vapor passing through a porous bed of the powder or ash and then on through a NaOH solution to neutralize excess SO_3 . We may cool the bed of powder or ash to condense sulfuric acid on the particles.

Following the successful deposition of the various compounds on the particle surfaces, we plan to perform the following analyses:

Measure the water content of samples (with the Karl Fischer Device) after they have been conditioned in atmospheres having specific relative humidities. (These analyses will be performed in the same way as analyses performed under E 3.2.)

Measure tensile strengths of samples (with the Electrostatic Tensiometer) after they have been conditioned in atmospheres having specific relative humidities. (These analyses will be performed in the same way as analyses performed under E 3.3.)

Measure specific surface area of the samples exposed to the various chemical compounds.

Perform chemical analyses to determine the concentration of compound that was successfully deposited on the particles.

Take SEM photographs of the samples prepared under E 3.5 if other data warrants visual observation of the particle surface.

PLANS FOR THE UPCOMING QUARTER

Under Task 2, we will be performing a variety of analyses on ashes and mixtures of ashes and sorbents with emphasis on Karl Fischer titration, tensile strength measurements, resistivity measurements, and further uncompacted bulk porosity determinations. Measurements of the water content of conditioned samples will resume under E 3.2. Additional measurements of tensile strength are planned under Experiment 3.3. Alternate means of depositing soluble compounds on the ashes and powders under E 3.5 will be investigated.

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