MASTER

DEVELOPMENT OF
REGENERABLE SORBENTS FOR FLUIDIZED BED COMBUSTION

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SUMMARY

In the initialization of the current program, equipment employed in the original pioneering study, including a thermogravimetric analyzer, an associated gas-mixing panel, and sorbent pellet preparation and conditioning hardware, was accumulated, tested for operability, refurbished, reinspected and recalibrated for future use.

Specifications were prepared for the fabrication of a laboratory-scale hot fluidized bed test unit (LSHFB), and a design for this system was completed by our Mechanical Division.

As a means to reinitiate the laboratory program and to test our equipment, new formulations incorporating the calcium aluminate cements (CAC) and barium titanate (BaTiO_3) materials, which were found to be the most promising sorbent candidates in the original study, were prepared and tested. Titanium dioxide, when incorporated into calcium aluminate cement formulations, produced an improvement in SO_2 absorption performance and, up to a level of about 5 weight percent, did not degrade crush strengths appreciably. Incorporation of carbon black and/or flyash into TiO_2 -containing CAC formulations produced additional absorption improvement, but significantly reduced sorbent crush strengths.

Several promising leads were pursued in the reformulation of barium titanate compositions. But it is apparent that this line of research must be considerably extended to achieve improvements of the type desired.

1. INTRODUCTION

Fluidized bed combustion (FBC) may be the way to burn high-sulfur coals in the steam boilers of power plants to achieve environmentally acceptable flue gas emissions at lowest costs-of-electricity. A practical FBC system will require a low-cost sorbent (bed material), resistant to attrition at the conditions of the combustion, which may be repeatedly regenerated to minimize both the rates at which new sorbent must be added, and waste sorbent be removed, from the system. Limestone and dolomite have been the principal sorbent materials used to date, but definite limitations in attrition-resistance and regenerability have been encountered with these substances.

This project is concerned with the continuing development of superior sorbents whose use as sulfur-capture agents in coal-fired FBC boilers will prove cost-effective. Candidate sorbents include calcium aluminate cements (CAC), and formulations of barium and calcium titanates with bentonite clays. The research will consist of the following principal tasks:

1. Design and fabrication of a laboratory-scale, fluidized-bed system which may be used to simulate large-scale sorption and desorption performance.
2. Experimental preparation of pellets of the candidate sorbents directed toward optimization of the attrition-resistance and sorption properties determined in the laboratory-scale system.
3. Preparation of optimized sorbent pellet formulations and large-scale testing in an existing pilot-scale, coal-fired fluidized-bed combustor.
4. Economic assessment projected for full-scale systems based on pilot-scale results.

2. WORK PROGRESS

During the September-December quarter, our efforts were directed to the reconversion of analytical and processing equipment used in the previous, pioneering study, including the DuPont Thermogravimetric Analyzer (TGA), associated gas-blending facilities, and pellet-forming and treating apparatus, and to the design of the laboratory-scale fluidized-bed system, which is to be used to extend our sorbent testing capability.

We began also our investigation of methods to improve the utilization activity, and the attrition-resistance of candidate sorbent formulations. Modifications in the basic formulations, as well as in the techniques for pellet preparation, were introduced for the calcium aluminate cements and for the barium titanate formulas in particular.

2.1. Experimental Equipment

1. The DuPont Model 951 Thermogravimetric Analyzer (TGA)

The thermogravimetric instrument, as previously modified to permit operation with reactive gases, was refurbished and brought on-stream. For our purposes, this device is operated as a differential reactor to measure the weight changes accompanying the sulfation and regeneration of a sorbent sample held in a stream of flowing gases. Sulfation, or the absorption of SO_2 , is studied by flowing a simulated flue gas mixture (0.25%, or 2500 PPM, SO_2 plus 5% O_2 in N_2) over the sorbent at 870-900°C. Regeneration is accomplished by flowing a reducing gas mixture (5% CO in N_2) over the sulfated sorbent at 1100°C.

Shortly after we began to test the TGA for our program, the instrument's furnace failed. We were able to install a spare furnace from the store of spare parts on hand from a previous program, while the failed component was being returned to DuPont for repair. Within a week, however, the spare furnace which we had installed also failed. After consultation with DuPont, it was decided to order a new furnace to expedite the instrument's utilization.

The new furnace was received about the first of November and, after some minor initial complications, has functioned normally.

2. TGA Gas-Mixing Panel

The sulfation and regeneration gas mixtures which we employ with the TGA are prepared from standard gases by continuous mixing through a metering-mixing panel. All of the inspections on the gas regulators and safety relief valves in the system were found to be outdated, requiring routine maintenance and reinspection.

3. Materials

Initial quantities of consumable materials, including cements, chemicals, chart papers, recorder pens, standard gases, and quartz furnace tubes were ordered.

4. Pellet Preparation Facilities

Substantial effort was directed to consideration of possible methods by which sorbent pellet production might be expanded. Heretofore, very small quantities of pellets have sufficed for screening in the TGA, and for the small-scale attrition-resistance testing we have conducted. In the case of the previous larger-scale fluidized-bed tests, which required a significant quantity of sorbent pellets, pellets were prepared by outside agencies (catalyst manufacturers). In the present program, it is estimated that five to ten pounds of each formulation may be required in connection with the laboratory-scale testing sequences, and that, probably, several hundred pounds of sorbent pellets will be required to perform projected testing in the bench-scale, coal-fired, fluidized-bed system.

A canvass of relevant preparation facilities existing in other laboratories at this site, which might become available to our project, proved unrewarding. Similarly, initial telephone contacts to prospective suppliers of custom sorbent pellet formulations, in the quantities foreseen, have so far not been encouraging.

We have consequently turned our attention to assessing low-budget modification of our pellet preparation techniques and equipment with view toward producing sufficient quantities of pellets to meet our initial needs in the laboratory-scale testing program. Devices intended to enhance our capabilities to extrude, chop and to condition and humidify pellets in particular have been designed, and are in process of being fabricated.

We intend, at the same time, to persist in our search for outside custom vendors who may agree to undertake to provide our larger sorbent requirements.

5. Laboratory-scale Hot Fluidized Bed Test System

A design for the laboratory-scale hot fluidized bed system (LSHFB) has been prepared, and preliminary cost estimates are being made. This system (see Figure 1 in the Appendix) comprises a quartz reactor held in an electric furnace. Provisions for admitting and metering sulfation and regeneration gases at required rates, and for the analytical determination of SO_2 concentration in the reactor effluent gas stream, have been included. Similarly, the control of the system temperature and pressure, as well as suitable pressure-relief arrangements, have been considered.

Determination of the SO₂ concentration in the reactor effluent gas during a sulfation cycle, at which time the concentration will be in the very low PPM range, will require a different instrument than that which may be used to analyze the effluent gas during regeneration, when the SO₂ concentration may be expected to be in the percent region. A Model 400 DuPont SO₂ Analyzer may become available for use by the project as the PPM analyzer, and either, or both, and Infra-Red Industries instrument, or a Model 865 Beckman SO₂ analyzer, may become available to handle the regeneration gas stream analysis.

The LSHFB system will also be equipped with suitable reactor effluent gas filtration equipment to permit quantitative assessment of the attrition-resistance of candidate sorbents.

The quartz reactor tubes have been ordered, and necessary glass-blowing modifications have been scheduled. Fabrication and installation of the rest of the system is dependent on receipt of the electric tube furnace, and is scheduled for 1st quarter, 1980.

2.2 Calcium Aluminate Cement (CAC) Sorbent Formulations

During this quarter, we reviewed the findings of the initial study in this area (FINAL REPORT: "Regenerable Sorbents for Fluidized Bed Combustion", June, 1978, L. A. Ruth and G. M. Varga, Jr.). We considered that effort should be concentrated to improve the performance of the calcium aluminate cement formulations with respect especially to utilization (fraction converted to sulfate) and activity, while preserving, or enhancing, demonstrated resistance to attrition.

1. Effect of Addition of TiO₂ to CAC Formulations

Titanium dioxide (Reagent Grade TiO₂), when tested in the sulfation mode in our TGA apparatus, was found to be inert, i.e., no weight gain, or sulfur absorption, was observed. TiO₂ was incorporated into a number of CAC formulations at varying concentrations to determine whether the utilization activity of CAC would be influenced thereby. In addition, some of the TiO₂-containing formulations also incorporated flyash or carbon black as additional additives.

2. Techniques for CAC Pellet Preparation

The techniques used to form, cure, and calcine CAC sorbent pellets, and the formulation compositions tested in this case, are detailed in Table 1.

3. TGA Testing of CAC Sorbent Pellets

The CAC pellet formulations prepared as outlined in Table 1 were tested in the DuPont Thermogravimetric Analyzer (TGA). Figures 2 through 5 in the Appendix are representative of the form of the raw data observed for the CAC formulation containing five weight percent TiO₂ additive. In all cases, the sample weight is seen to increase during the sulfation portion of a cycle, and to decrease during the regeneration part of the cycle. In this instance, the particular sample underwent four successive complete sulfation-regeneration cycles.

Table 1

Techniques for Preparing CAC Pellets

I. Pellet Compositions

A. Calcium Aluminate Cement (CAC) + TiO₂

1. CAC powder with 4.76 weight percent TiO₂, extruded.
2. CAC powder with 2.00 weight percent TiO₂, granulated.

B. CAC + TiO₂ + Fly Ash

1. CAC powder with 4.5 weight percent TiO₂ and 4.5 weight percent fly ash, granulated

C. CAC + TiO₂ + Carbon Black

1. CAC powder with 12.5 weight percent TiO₂ and 4 weight percent carbon black (Vulcan X6-72)
2. CAC powder with 4.5 weight percent TiO₂ and 4.5 weight percent carbon black (Vulcan XC-72)

II. Pellet Forming and Curing

A. Extruded Pellets

1. Cement powder mixed with water (usually 22% water by weight).
2. Extruded through 1/8" I.D. glass tube.
3. Cut continuous extrudate to form 1/8"x1/8" cylinders.
4. Place in 100% humidity atmosphere at room temperature for three days.
5. Heat in 110°C oven overnight (at least 12 hours).
6. Heat treat (see III).

B. Spherical Pellet

1. A thin (1/16") layer of CAC on bottom of flat container is atomized with water.
2. Container isolated with circular motion as if sifting.
3. Add water to obtain larger spheres.
4. Spheres are screened at this point to uniform size
5. Spheres are misted, and placed in 100% humidity atmosphere for three days.
6. Heat in 110°C oven overnight (at least 12 hours).
7. Heat treat (see III)
8. Screen again if necessary to achieve size uniformity.

Table 1 (cont.)

III. Heat Treatment (calcination)

A. Standard Heat treatment

1. Heat in 200°C furnace for 12 hours.
2. Increase temperature to 300°C, hold for two hours. Repeat and hold at 400, 500, 600, 700, 800, 900, 1000°C.
3. 1100°C for overnight (at least 12 hours).

B. Fast Heat Treatment

1. Raise temperature according to schedule.

<u>Temp.</u>	<u>Hours from start</u>
200°C	0
400	2
700	3 to 3-1/2
900	4-1/2 to 5
1100	6

2. Hold at 1100°C for two hours.

In Figure 6 of the Appendix are compared this sequence of TGA cycling data with those previously obtained under essentially identical test conditions for a CAC sorbent pellet which did not contain the TiO_2 additive. It is clear that the TiO_2 addition has increased the apparent performance of the CAC sorbent, though at this point, the precise cause of the effect is not known.

4. Effect of Additional Additives with TiO_2 in CAC Sorbent Formulations

Both the carbon black and flyash additives incorporated, along with TiO_2 , into CAC formulations are primarily intended to increase the active surface area of the final prepared pellets. In both cases, carbon is burned out in the process of calcination, and serves to increase final porosity. Minor impurities present in the carbon black, or the refractory (major) portion of the flyash, would be expected to have very little, if any, chemical effect on sorbent performance; but the flyash, in particular, might influence the nature of the surface of the final prepared pellets. And both materials, by virtue of the induced porosity effect, could be expected to decrease the strength of the final pellets.

In all of the present cases, TGA testing of CAC formulations containing either 5% flyash or 5% carbon black, in addition to 5% TiO_2 , showed improved absorption activity. Figure 7 of the Appendix compares the cycling performance of the three formulations. The effect of the carbon black is greater, in this respect, than that of the flyash, as might be expected on the basis of a higher burnable fraction on calcination.

5. Attrition of CAC Formulations

Crush testing of finished sorbent pellets is performed with a Chittillon Spring Tester. The crush strengths of the CAC pellet formulations discussed above are given in Table 2. Note the relatively high crush strength for the formulation containing only TiO_2 additive, and the large, order-of-magnitude decrease in crush strength resulting from the incorporation either of flyash or carbon black.

Attrition-testing is performed in a small, fluidized bed agitation device (see final report, op. cit.). Because of the very poor crush strengths of the flyash or carbon black formulations, attrition-testing of these materials was not performed. The testing of the CAC - TiO_2 formulation, however, confirmed that TiO_2 addition did not materially degrade the high attrition-resistance observed for CAC alone. A measurement in our testing device showed an attrition of only 0.79% for four hours at an average velocity of 8.05 ft/sec. for the CAC formulation containing 5% TiO_2 .

2.3 Barium Titanate (BaTiO_3) Sorbent Formulations

In contrast with the CAC formulations discussed above, which in, and of themselves, demonstrate high attrition resistance, the highly active barium and calcium titanate formulations so far developed are considered deficient in this respect. Consequently, our efforts are being directed toward improvement of the physical properties of these sorbent materials, while preserving their demonstrated high utilization activities.

Table 2

Crush Strengths of CAC Sorbent Formulations

<u>Sample</u>	<u>Crush Strength Before Cycling, (lbs.)</u>
CAC + 5% TiO ₂	38.75
CAC + 5% TiO ₂ + 5% Carbon Black	3.50
CAC + 5% TiO ₂ + 5% Flyash	1.25

1. BaTiO₃ Formulations Incorporating Ceramacast 505

The aluminas, as a class, are among the strongest of the substrates employed in the preparation of high-temperature catalysts. In general, however, it is difficult to incorporate substantial fractions of additive materials into these substances, which have rather low active surface areas relative to other substrates employed in catalysis.

Ceramacast 505 is a mixture of alumina (Al₂O₃) and magnesia (MgO), along with a silicate binder, which may be slurried with water, cast, and fired at high temperature to produce a high-strength alumina cement. We prepared mixtures of Ceramacast 505 with BaTiO₃ at 10 and 25 weight percent levels. These mixtures formed into water pastes, as indicated in Table 3, were easily extrudable through a 1/8-inch glass tube. The finished pellets were heat-treated at only 200°C.

2. TGA Testing of Ceramacast 505 Formulations

The Ceramacast 505/BaTiO₃ formulations showed high sulfation activity in the initial cycle on the TGA, and good regeneration performance also on the initial cycle. Sulfation activity had dropped to a third of the initial activity on the second cycle, however, and to near-zero on a third cycle. It appeared that the sorbents were undergoing surface sintering in the TGA apparatus.

Consequently, other sorbent pellets, prepared as outlined in Table 3, were additionally calcined in an electric tube furnace at 1100°C, the normal regeneration temperature employed in the TGA tests, prior to testing in the TGA. Such pre-calcined pellets showed zero sulfation activity, reinforcing the notion that excessive sintering had occurred, probably due to formation of low-melting eutectic combinations among the magnesia, alumina, silica, and barium oxide components.

In a separate sequence of experiments, mixtures of MgO and TiO₂; Al₂O₃, BaO, and TiO₂; and BaO, TiO₂, Al₂O₃, and MgO were prepared and calcined in the same tube furnace at 1100°C. No evidence of melting or sintering was observed in any of these tests; however. The manufacturer of Ceramacast 505 would not disclose the nature of the silicate binder used in his formulation. Our tentative conclusion, therefore, was that the fusion we observed in our sorbent pellet formulations was due to this silicate component.

3. Silbond H-6 Formulations

Attempts to prepare BaTiO₃ sorbent pellet formulations on silica substrates, or in admixture with silicas, was pursued separately as a means to improve the strength of the barium titanate formulations.

The use of Silbond H-6, a silicate binder, in the preparation of silica-based catalysts is described in U.S. Patent No. 3, 645, 914. The composition and preparation procedures we adopted for our materials are shown in Table 4.

Table 3

Compositions of Ceramacast 505/BaTiO₃
Extruded Pellets

<u>Sample</u>	<u>Composition</u>	<u>wt%</u>
1	Ceramacast 505	100
2	BaTiO ₃	25
	Ceramacast 505	75
3	BaTiO ₃	10
	Ceramacast 505	90

Preparation

1. Mixed by hand for 15 minutes
2. Wet with distilled water
3. Extruded through a 1/8" I.D. glass tube
4. Let air-cure overnight (at least 12 hours)
5. Heated 2 hours at 200°C

Table 4

Composition of BaTiO₃/Silbond H-6
Pellet Preparations

1. 48.2 grams _____ Al₂O₃
2. 3.0 grams _____ SiO₂
3. 14.5 grams _____ BaTiO₃
4. 75.0 grams _____ distilled H₂O
5. 2.7 grams _____ Silbond H-6
6. 1.8 ml _____ 190% EtOH
7. 0.2 ml _____ MeOH
8. 0.2 ml _____ 4 N HNO₃

Procedure for Preparation of
BaTiO₃/Silbond H-6 Pellets

1. Weigh out Al₂O₃ + SiO₂ (200 micron); mix 30 minutes
2. Add distilled H₂O; soak overnight
3. Filter
4. Then add, in order, BaTiO₃, H₂O, Silbond H-6, 190% EtOH, MeOH, and 4N HNO₃
5. Mix for 30 minutes
6. Heat treat @ 200°F for 21 hours in sealed container
7. Add 6.5 ml distilled H₂O
8. Mix for 15 minutes
9. Extrude to 1/8" x 1/8" pellets with 1/8" I.D. glass tube
10. Dry at 230°F 21 hours
11. Calcine at 1000°C for 2 hours

Pellets prepared according to the method outlined in Table 4 showed high sulfation utilization. However, the crush strength of such pellets was very low.

Following a lead mentioned in a Russian literature reference, we subjected low-strength pellets to phosphoric acid treatment, followed by heat treatment at 150°C, and to additional calcination at 1200°C. The treatment effected an enormous increase in crush strength, but testing in the TGA showed that sulfation activity had been decreased just as dramatically. Figure 8 in the Appendix demonstrates the comparative performance of these Silbond H-6 pellet formulations before and after the acid treatment.

3. WORK PLANNED FOR FIRST QUARTER, 1980

Technical effort will be primarily directed toward fabrication, installation, and startup of the laboratory scale hot fluidized bed test unit.

As a necessary adjunct, the preparation of candidate sorbent pellet formulations will be pursued, both within and without our laboratory. We shall attempt to engage the services of an outside vendor to prepare the significant quantities of pellets we deem to be required, especially for the bench-scale test program.

We shall attempt, also, to expand on our efforts to improve the characteristics of the most promising candidate sorbents.

Figure 1 Laboratory-Scale Hot Fluidized Bed Sorbent Test Unit

DATE	REVISION	
LSHFB UNIT		
FLOW DIAGRAM		
O'DONN RESEARCH AND ENGINEERING COMPANY		
RESEARCH TECHNOLOGY SERVICES DIVISION		
LINDEN, N. J.		
NAME	DATE	PROJECT NO.
C. MORANDI	1/1/74	71480
DESIGNED BY	DATE	100%
C.M.	1/1/74	
APPROVED BY	DATE	

Figure 2 Thermogravimetric Analysis
5% TiO₂ In CAC

Cycle 1

PART NO. 990089

RUN NO. 1 DATE 11-21-79	T-AXIS	DTA-DSC	TGA	TMA	Legend TGA B. & N.
OPERATOR	SCALE, °C/in 10	SCALE, °C/in	SCALE, mg/in 25	SCALE, mils/in	
SAMPLE: 5.02% TiO ₂ In CAC	PROG. RATE, °C/min 10	(mcal/sec)/in	SUPPRESSION, mg 31.5	MODE	
ATM. 100% O ₂ B. & N.	HEAT COOL ISO X	WEIGHT, mg	WEIGHT, mg 31.5	SAMPLE SIZE	
FLOW RATE 100 ml/min	SHIFT, in	REFERENCE	TIME CONST., sec 1	LOAD, g	
	TIME BASE, min/in 5		dY, (mg/min)/in	dY, (10X), (mils/min)/in	

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Figure 3 Thermogravimetric Analysis
5% TiO₂ In CAC

Cycle 2

PART NO. 990089

RUN NO. 11 DATE: 11-11	T-AXIS	DTA-DSC	TGA	TMA	Reg. 5% CO Bal N ₂
OPERATOR	SCALE, °C/in. 20	SCALE, °C/in.	SCALE, mg/in. 1	SCALE, mils/in.	
SAMPLE: 7427-17 TiO ₂ 5% CAC	PROG. RATE, °C/min. X	(mcal/sec)/in.	SUPPRESSION, mg. 36	MODE	
ATM. 152.50 (101.30) 8.00	HEAT COOL ISO 0	WEIGHT, mg.	WEIGHT, mg.	SAMPLE SIZE	
FLOW RATE 1000/min	SHIFT, in. 5	REFERENCE	TIME CONST., sec. 1	LOAD, g.	
	TIME BASE, min/in. 5		dY, (mg/min)/in.	dY, (10X), (mils/min)/in.	

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MEASURED VARIABLE

Figure 4 Thermogravimetric Analysis
5% TiO₂ In CAC

Cycle 3

PART NO. 990089

RUN NO. 12 ² DATE 11-21-74	T-AXIS	DTA-DSC	TGA	TMA	Regan C&C 5% CAC
OPERATOR	SCALE, °C/in. 1000	SCALE, °C/in. (mcal/sec)/in.	SCALE, mg/in. 1	SCALE, mils/in.	
SAMPLE: 7429-13 TiO ₂ 5% in CAC	PROG. RATE, °C/min 100	WEIGHT, mg	SUPPRESSION, mg 36.2	MODE	
ATM. 25.2% O ₂ 75.0% N ₂	HEAT COOL ISO	REFERENCE	WEIGHT, mg	SAMPLE SIZE	
FLOW RATE 100 cc/min	SHIFT, in. 0		TIME CONST., sec 1	LOAD, g	
	TIME BASE, min/in. 5		dY, (mg/min)/in.	dY, (10X), (mils/min)/in.	

DUPONT Instruments

MEASURED VARIABLE

Figure 5 Thermogravimetric Analysis
5% TiO₂ In CAC
Cycle 4

PART NO. 990089

RUN NO. <u>1</u> / DATE <u>11/24/77</u>	T-AXIS	DTA-DSC	TGA	TMA
OPERATOR <u>J. J. J.</u>	SCALE, °C/in <u>100</u>	SCALE, °C/in <u>1</u>	SCALE, mg/in <u>1</u>	SCALE, mils/in <u>1</u>
SAMPLE <u>7-127-1</u>	PROG. RATE, °C/min <u>10</u>	(mcal/sec)/in <u>1</u>	SUPPRESSION, mg <u>5.6</u>	MODE <u>1</u>
ATM. <u>25% O₂, 5% CO₂, 70% N₂</u>	HEAT <u>COOL</u> <u>ISO</u>	WEIGHT, mg <u>1</u>	WEIGHT, mg <u>1</u>	SAMPLE SIZE <u>1</u>
FLOW RATE <u>100 mL/min</u>	SHIFT, in <u>0</u>	REFERENCE <u>1</u>	TIME CONST., sec <u>1</u>	LOAD, g <u>1</u>
	TIME BASE, min/in <u>5</u>		dY, (mg/min)/in <u>1</u>	dY, (10X), (mils/min)/in <u>1</u>

DUPONT Instruments

MEASURED VARIABLE

Figure 6

Comparison of Cycling Performance of
CAC Pellets Incorporating TiO_2

Figure 7

Comparison of Cycling Performance of CAC
+ TiO_2 With Carbon Black Or Flyash

Figure 8

Comparison Data For Silbond H-6/BaTiO₃
Pellets Before and After H₃PO₄ Treatment