MASTER

**REGENERATIVE PROCESS FOR DESULFURIZATION OF
HIGH TEMPERATURE COMBUSTION AND FUEL GASES**

PROGRESS REPORT NO. 14

OCTOBER 1 - DECEMBER 31, 1979

A.S. ALBANESE AND D.S. SETHI

March 1980

DEPARTMENT OF ENERGY AND ENVIRONMENT

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Abstract

The characteristics of Portland Type III cement (PC III) as a potential sorbent for use in a regenerative process for desulfurization of coal in fluidized-bed combustion were examined. The sulfation rates for PC III + 5% amorphous SiO_2 at temperatures between 860°C and 1100°C and at 1 atm were studied thermogravimetrically, using a simulated combustion gas mixture containing 0.25% SO_2 . The sulfation rates were found to be independent of temperature between 950°C and 1050°C . The isothermal regeneration with a mixture consisting of 5% CO , 15% CO_2 and the balance N_2 , in the above temperature interval of 100°C approached an equilibrium value of over 90% within thirty minutes. Sulfation/regeneration efficiency did not decrease with cycling and was unaffected by additives introduced to increase the porosity and/or conversion of free CaO in PC III to silicates which are known to be more regenerative than CaSO_4 . Pressurized TGA experiments within the optimum sulfation temperature range were conducted at 5 and 10 atm to establish the viability of this sorbent for pressurized fluidized-bed combustion use. Within the experimental accuracy, no pressure dependence of sulfation rate was observed. Our experiments indicate that the attrition resistance of PC III is significantly better than natural limestones.

I. Introduction

The results of our experiments reported previously,¹ suggest that Portland Type PC III cement (PC III) is a very reactive sorbent for SO₂ absorption with excellent isothermal regeneration efficiency. In this communication, we present the results of our experiments conducted to characterize PC III with regard to:

- 1) the effect of temperature on sulfation rates and capacity,
- 2) efficiency of sulfation/regeneration cycling,
- 3) the effect of additives on sulfation yield,
- 4) the pressure dependence of sulfation conversion,
- 5) the temperature dependence of regeneration, and
- 6) attrition resistance.

The objective of these experiments and the preliminary data on attrition tests on PC III reported here, is of course, to evaluate this material for fluidized-bed combustion application. Comparison of PC III with other sorbents such as Greer limestone and Tymochtee dolomite under their optimal sulfation capacity is also presented.

Sulfation and regeneration experiments were performed in a DuPont TGA and a custom designed vertical TGA set up² for pressurized runs. Sulfation was conducted at the desired temperature and pressure by using a simulated combustion gas of 0.25% SO₂, 5% O₂, 15% CO₂ and the balance N₂. The regeneration mixture consisted of 5% CO, 15% CO₂ and the balance N₂.

The flow rates for the sulfation and regeneration in the DuPont TGA and the pressurized unit were 500 mL/min and 1 L/min, respectively. Sulfation rates were monitored for between two to over six hours.

Regeneration approached equilibrium value after about twenty to thirty minutes at the above mentioned flow rates. After this time interval, the sample was treated with 5% O₂, 15% CO₂ and the balance N₂ until no weight increase was observed. This, to account for the weight correction for reducibles such as metal oxides in the PC III. The integrated optimal regeneration efficiency values obtained by this procedure are given in Table III.

II. Effect of Temperature on Sulfation Rate

The effect of temperature on the sulfation rate for PC III with 5% amorphous SiO₂ at 1 atm. is shown in Figure 1. The optimum temperature for sulfation is somewhere between 950°C and 1050°C. For a temperature lowering of about 65°C from the optimal temperature interval, the sulfation rate is decreased by about 15%, whereas a temperature increase of 50° results in a sulfation rate lowering of around 35%. The optimum temperature range³ in fluidized-bed combustion of coal for SO₂ absorption using natural limestone is between 830°C and 900°C. The higher observed optimum temperature for sulfation in the case of PC III is an advantage over the limestone, because combustion and power cycle efficiencies can be increased if the bed material can be maintained at a high temperature. The upper limit on the equilibrium partial pressure of SO₂ in this optimum temperature range⁴ is somewhere between 10⁻⁵ and 10⁻⁴ atm. for a 1 atm. total pressure, which is below the current EPA effluent limit. In pressurized fluidized bed systems, the situation will be even more favorable.

III. Sulfation/Regeneration Cycling Efficiency

The results of a typical isothermal sulfation/regeneration experiment conducted at a temperature within the optimal range (982°C) are presented in Table I. Ignoring the caveat of a much higher sulfation capacity for cycle number 1 for the moment, it is reasonable to conclude that the two hour sulfation capacity of about 45 percent or better and the equilibrium approaching regeneration value of about 95 percent do not deteriorate with cycling. No change in reactivity after repeated cycling of the same material indicates no sorbent sintering activity. Experiments wherein the regeneration was carried out at a temperature 50°C lower than the sulfation within the 950 to 1050°C optimal temperature range showed no significant difference from the data reported in Table I. This implies that any minor energy loss in the desired isothermal transfer of the hot sulfated pellets for the regeneration will not necessitate additional energy expenditure in this step. The higher initial sulfation capacity cannot be accounted for as due to the formation of CaSO_4 by sulfation of free lime (2%) in PC III. It most likely results from the sulfation of metal oxides such as Al_2O_3 , which may be incapable of regeneration at the temperatures used in this study. Such an explanation could account for the observation of a limiting fraction of about 0.95 in column 3 of Table I for the regeneration efficiency. Experiments are currently underway to elucidate the mechanism of the regeneration. These results will be presented at a later date.

IV. Effect of Additives on the Sulfation Yield

In a previous report¹, preliminary data on the effect of additives was presented. It was suggested that additives such as SiO_2 may improve the regeneration capacity by forming calcium silicates by reaction with the free lime in PC III through cycling. These silicates were observed to be more regenerative than CaSO_4 . Thus, the higher sulfation/regeneration capacity in recycling. Therefore, all the experimental results presented in Sections II, III and V in this work were conducted with 5% amorphous silica as an additive. We previously noted² that the sulfation yield was affected by the combined porosity and water content of the pellets. The decoupling of these two variables by careful control of the amount of water used in the pellet preparation shows (Table II) that as long as the water content in the initial preparation is below about 8% the sulfation yield is not affected. This introduces a reasonable degree of flexibility in pellet preparation operations. Wherever, the results in sections II, III and V of this report can be compared with similar experiments wherein porosity was introduced by the incorporation of burnable carbon,⁴ it was found that the sulfation capacity and sulfation/regeneration cycling efficiency were identical within the experimental error. Thus, it appears that at least for PC III and PC III +5% amorphous SiO_2 as sorbents, the porosity of the silicate structure is not changed to any appreciable degree by the addition of burnable carbon. In a recent paper⁵, it was suggested that the Ca(OH)_2 generated by the reaction of water with the free lime in cement results in the breakdown of cement due to alkali-silica reaction. This may conceivably lead to an increase in the porosity due to the opening of the silicate

structure and hence better sorbent characteristics. We tested this hypothesis by adding 2% lime to PC III +5% amorphous SiO_2 . As can be seen from the last entry in Table II, no increase in the sulfation capacity was observed.

We have initiated work with natural clays as additives. Improvement in sulfation capacity is only one aspect of this work. Another objective of this phase of work is to conduct attrition tests as to the viability of the various mixtures. Attrition losses are important not only from the point of view of total material attrition, but also in establishing the extent of submicron fluid bed material ejection into the atmosphere. Polycyclic aromatic hydrocarbons, analogous heterocyclic compounds and/or adducts of sulfur oxides⁷ have been reported to be present in the airborne particulate matter from coal combustion. Most of this particulate matter which is not trapped by the dust collectors is probably fly ash from the coal. Nevertheless, it can be expected to contain some sorbent material also. The organic compounds are suspected mutagens and carcinogens⁸. The flyash with adsorbed SO_2 through heterogeneous photo-oxidation can give rise to SO_3 with nontrivial quantum yields followed by H_2SO_4 aerosol formation. Other sulfur containing compounds such as dimethyl sulfate have been observed in flyash and in other particulate matter from coal combustion⁷. Thus, it appears to us that attrition tests as to total material loss as well as airborne loss are relevant in establishing the identity of a suitable sorbent. Experiments along these lines will be initiated soon.

V. Pressure Dependence of Sulfation Conversion

Pressurized TGA experiments were undertaken to determine the effect of high CO_2 partial pressure (P_{CO_2}) on the sulfation capacity of PC III. This, to establish the viability of this sorbent for pressurized fluidized-bed combustion. The experimental data on PC III + 5% SiO_2 given in Figure 2 for all three pressures of 1, 5 and 10 atm represent the mean values of seven experiments in each case. The three curves are within one standard deviation of each other. Thus, the sulfation rate of PC III + 5% amorphous SiO_2 is independent of pressure up to at least 10 atm. For comparison we have also plotted, on this figure, previously obtained data on sulfation rates at 1 and 10 atm for Greer limestone and Tymochtee dolomite under their optimal sorbent activity. The decrease in the sulfation capacity at the higher pressure for both of these is due to the fact that calcination of CaCO_3 at a P_{CO_2} of 1.5 atm. is noticeably less effective⁹ than at P_{CO_2} of 0.15 atm. Therefore, PC III in comparison to limestones appears to be a suitable sorbent for not only atmospheric fluid-bed application, but in pressurized combustion also, where higher combustion, power cycle and sulfur retention efficiencies, reduced reaction vessel size, and lower NO_x emissions represent some important advantages.

VI. Regeneration of Sulfated Portland III Pellets and Greer Limestone

TGA regeneration experiments were run on sulfated Portland III pellets (16/20 mesh, 5% SiO_2 , 6.1% H_2O) and sulfated Greer limestone to determine the extent of regeneration under several sets of operating conditions. The Portland III pellets were sulfated and regenerated isothermally and isobarically. The Greer was precalcined at 900°C and 1 atm. using 5% O_2 , 15% CO_2 and 80% N_2 . Sulfation and regeneration were carried out at 1 atm. using the same gas compositions (see p.1) as for the Portland III pellets. The results of our experiments are summarized in Table III. The rates of regeneration, which are approximate, are given in the last column of the table as SO_2 rate in mg/min. They were estimated from the entire linear portion of the TGA regeneration curves.

At a system pressure of 1 atm., the regeneration rates and efficiencies of both the Portland III pellets and Greer increase with increasing temperature, over the temperature ranges considered, which is consistent with the endothermic nature of the regeneration reactions. However, Portland III pellets regenerate much more efficiently than Greer.

The rate and extent of regeneration of sulfated Portland III at 10 atm. are comparable to those at 1 atm., for a regeneration temperature of about 957°C . Therefore, considering its sulfation capability, its high SO_2 equilibrium partial pressure in reductive decomposition, and its regeneration efficiency at 10 atm. pressure, it appears that Portland III pellets would be suitable not only for

pressurized-fluidized bed (PFB) service but also for isobaric sulfation/regeneration operations. Operating a commercial regenerative PFBC system isobarically could facilitate the transfer of solids between the fluidized-bed combustor and sorbent regenerator.

VII. Attrition of Portland III and Greer

A small scale fluidized-bed operated at 1000°C was used to determine the rates of attrition of 16/20 mesh Portland III pellets and +40 mesh Greer limestone. The pellets were made by the procedure described in section IX of this report. They were cured for 28 days at room temperature in an atmosphere of 100% humidity and had an initial water content of 7.6%. The Greer limestone was obtained from Argonne National Laboratory. The partially sulfated Greer was collected by the cyclone above Argonne's fluidized-bed pilot unit and has a wide particle size distribution. A grab sample of the Argonne material was screened and the fraction less than 40 mesh discarded; the +40 mesh fraction was used in our attrition tests.

The experiments were run at 1000°C in an electrically heated, 30 mm. I.D. by 30 cm. long quartz tube. Fluidization gases were preheated and then passed through a coarse frit which served as bed support and gas distributor. A total gas flow of 7500 SCCM was used in all experiments, which corresponds to a superficial bed velocity of 75.5 cm/sec (2.5 ft/sec) at operating conditions. A 20 g. sample (after pre conditioning) was used in all cases. This amount of material filled the quartz tube to a height of about 20 mm. under static bed conditions and to between 25 and 30 mm. when the bed was fluidized.

Before starting each run, a sample of the material to be tested was heated to 1000°C for 10 minutes in the reactor tube. A small amount of air was then pulsed through the bed every minute to mix the bed material (Portland or Greer) and drive off any moisture and volatiles. The material was allowed to cool, and subsequently removed with a vacuum trap. A 20 g. batch of this pre-conditioned material was loaded into the quartz tube, and brought to 1000°C within 5 to 8 minutes. Following this, the preheated fluidization gas mixture flow was initiated. At the end of 1 hour, the gas flow was stopped and the electric heater turned off. The sample was allowed to cool for about 5 minutes, then removed from the quartz tube, weighed and put back. The difference between the initial and final weights of the sample was taken as the amount of material elutriated from the bed due to attrition. The procedure was repeated for 8 cycles (8 hours).

For a superficial velocity of 2.5 ft/sec, it is roughly estimated (by terminal velocity calculations) that only particles less than 0.16 mm diameter will be elutriated from the fluidized bed when using air at 1000°C as fluidizing medium. The particle size in our experiments was between 0.85 and 1.00 mm for PC III and greater than 0.400 mm for Greer. Thus, it is appropriate to attribute the weight loss to material attrition.

The results of our experiments are presented in Figure 3 for the 16/20 mesh Portland III pellets and in Figure 4 for the Argonne +40 mesh Greer. In unsulfated Portland III, the lowest attrition losses occurred when air was used as fluidizing medium, which amounted to 4% of the original weight of sample after 8 hours of operation. The corresponding loss in oxygen was 5%, whereas, about 6% losses occurred when either

nitrogen or a mixture of 95% air and 5% CO₂ was used. Fully sulfated Portland III showed about a 6% loss after 8 hours in air, which is 1.5 times higher than for the unsulfated material.

Our data on +40 mesh, partially sulfated Greer indicate an attrition loss of about 14% after 8 hours of fluidization by air. This is 3.5 times greater than that obtained on unsulfated Portland III and 2.3 times greater than for sulfated Portland III. Thus, although these losses are not necessarily indicative, in an absolute sense, of what will occur in commercial size equipment, they do give a relative indication of the superior attrition resistance of agglomerated calcium silicates over natural Greer limestone.

In similar attrition experiments run at Argonne¹⁰, in a 5 cm. I.D. tube at 870°C, the losses for 14/30 mesh sulfated Greer were 1.8 times greater than for unsulfated material after an 8 hour fluidization period. These results are thus in agreement with our Portland III results in regards to the relative attrition resistance of sulfated and unsulfated material. However, sulfated Tymochtee dolomite and limestone 2203 were considerably more attrition resistant than the corresponding unsulfated materials. The reductions in attrition due to sulfation for Tymochtee and 2203 were 70% and 60%, respectively. Although no firm conclusion can be drawn at this time, it appears that sulfation tends to strengthen the relatively soft materials whereas it weakens the harder ones.

VIII. Kiln Regeneration Process

In order to get an idea of the possible SO₂ concentrations attainable by reductive decomposition of Portland III pellets in a kiln apparatus, we ran tests in our 1" batch kiln, described in BNL 50809, using CO as reducing medium.

The reaction

is assumed to occur according to the following two step sequence:

For Portland III, x takes on values of 2 and 3 which correspond to dicalcium silicate and tricalcium silicate, respectively.

Experiments were conducted on 10 g. samples of fully sulfated, 16/20 mesh PC III pellets, at temperatures ranging from 925 to 1000°C, using CO and N₂. At 925°C and 25 SCCM CO/25 SCCM N₂, the SO₂ concentration of the kiln off-gas was measured at 13.5% SO₂. When the kiln temperature was increased to 950°C, at the same flow conditions, the SO₂ concentration increased to 16.8%. For 1000°C operation and a feed gas of 40 SCCM CO and 25 SCCM of N₂, the concentration of SO₂ was 20.1%.

With coal as reductant in the 1" batch kiln, segregation of coal particles and sulfated PC III particles occurred resulting in low SO₂ off-gas concentrations.

IX. Preparation of Portland Cement Pellets

The PC III pellets used in our experiments are currently prepared at Brookhaven National Laboratory, on a small batch basis, using a vertically mounted drum (pelletizer) 5 in. deep by 18 in. in diameter. The drum is direct-driven by a small electric motor which operates at a speed of about 50 rpm.

The cement powder is first mixed with a fine spray of water to bring its moisture content to a maximum of 10% by weight. The powder must be pre-moistened before it is placed into the pelletizer drum or it will

slide and not tumble when the drum is rotated. The conditioned powder is uniformly fed into the pelletizer pan and intermittently sprayed with a light mist of water. During operation, the cement particles remain at the bottom of the pan as the drum surface rotates around them. The continuous rolling and tumbling of powder results in the formation of nuclei. Seed formation and growth is accomplished by the continuous layering of fines and liquid on the rolling bed of cement particles. If pellets of a wide size distribution are desired, the operation is continued until the desired size distribution is achieved (within the constraints of the equipment). In our experiments, we used pellets of a narrow size distribution (16/20 U.S. mesh). Therefore, once the particle nuclei were formed, the machine was turned off and emptied and the seed pellets were screened to a uniform size, smaller than the desired diameter of the finished pellets. The seeds were then re-fed into the pelletizer and the agglomeration procedure continued until the 16/20 mesh size was attained.

The water content of each batch of pellets (which is kept separate from others) is determined by weighing a representative sample of the batch and then re-weighing after drying in a vacuum oven at 100°C for one hour. Water content between a range of 2 to 8%, does not appear to affect the sorption capacity of Portland III pellets (see Table II, p16); however, it may affect other properties of the agglomerates (e.g., attrition resistance). The finished pellets are kept at 100% humidity for 28 days (curing period). After curing, the pellets are removed from the curing apparatus and lightly hammered to separate particles that have stuck together.

We have recently ordered new equipment for making agglomerates which will allow us to prepare pellets of desired diameter and water content, automatically, up to 100 lbs/day.

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Table I.

Isothermal Sulfation/Regeneration Cycling of PC III +5%
Amorphous SiO₂ + 6.1% H₂O^a at 982°C and 1 atm.

Cycle #	Sulfation, ^b %	Regeneration ^c %
1	55.3	93.9
2	47.6	93.8
3	46.1	94.7
4	46.8	94.6
5	46.4	94.9
6	46.6	94.6
7	45.9	94.2
8	45.2	91.0
9	39.9	94.8
10	45.6	95.1

^a See Text p. 11 for pellet preparation details.

^b Integrated sulfation over a 2 hour period.

^c Procedure for this evaluation described on p 2.

Table II.

Effect of Additives on the 2 Hour Sulfation Capacity of PC III +5%
Amorphous SiO₂ + 6.1% H₂O,^a at 982°C and 1 atm.

Additive	Additive wt%	Sulfation, ^b %
Water	2.1	51.0
Water	7.4	52.3
Water	7.1	50.1
Water	6.3	46.0 ^c
Water	12.3	37.5 ^c
Water	22.9	28.5 ^c
Lime: 1st cycle	2.0	51.0 ^d
2nd cycle		44.0 ^d
3rd cycle		45.1 ^d

^a See Text p. 11 for pellet preparation details.

^b Integrated sulfation capacity

^c Taken from Ref. 1

^d Regeneration efficiencies for cycles 1 & 2: 91.0 & 94.0%, respectively.

Table III.
Regeneration of Sulfated Portland III and Greer Limestone.

Material	Sulfation, %	Regeneration Temp., °C	Regeneration Pres., atm.	Weight Loss, mg.	Time, ^a min.	Regeneration, %	SO ₂ Rate mg/min. ^a
Portland III ^b	49.5	957	1	18.1	22	84.3	0.65
	59.0	1050	1	22.7	22	86.6	0.83
	38.8	1100	1	16.0	14	94.6	0.91
	58.5 ^c	958	10	22.9 ^c	26 ^c	91.0 ^c	0.70 ^c
Greer ^d	21.0 ^e	950	1	3.9	22	37.6	0.41
	18.1 ^e	1000	1	5.1	13	55.3	0.31
	18.0 ^e	1050	1	6.6	10	73.7	0.53

^a Over linear portion of regeneration curve.

^b 16/20 mesh pellets, 5% SiO₂ and 6.1% H₂O.

^c Average value for 12 sulfation/regeneration runs.

^d 16/20 mesh particles.

^e Calcined at 900°C using 30% N₂, 15% CO₂ and 5% O₂.

Figure 1. The Effect of Temperature on Sulfation Rate at 1 Atm.,
For Portland Type III Pellets With 5% Amorphous SiO_2 Added

Figure 2. Sulfation Rates of 16/20 Mesh Portland Type III Pellets,
Greer Limestone and Tymochtee Dolomite at Various Pressures and
Temperatures

Figure 3. Attrition Rates of 16/20 Mesh Portland III Cement (7.6% H₂O) in a 30mm Diameter Fluidized-Bed Operated at 1000°C, 1 Atm. and a Flow Rate of 7500 SCCM (Superficial Velocity = 2.5 Ft./Sec.)

Figure 4. Attrition Rate of +40 Mesh Greer Limestone, Which Was Collected in the Cyclone Above Argonne's FBC Unit. Tests Were Run in a 30mm Diameter Fluidized Bed at 1000°C, 1 Atm. and at a Flow Rate of 7500 SCCM

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