REGENERATIVE PROCESS FOR DESULFURIZATION OF HIGH TEMPERATURE COMBUSTION AND FUEL GASES

PROGRESS REPORT NO. 13
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SUMMARY

Various types of portland cement were subjected to sulfation/regeneration cycling for application in fluidized bed combustion (FBC) systems. Among them, Type III was the most reactive sorbent for SO_2 absorption. Sulfated portland cement is highly superior in its resistance to attrition than sulfated limestone which has serious attrition loss problems in fluidized-bed combustors. An isothermal sulfation/regeneration cycle for portland cement Type III pellets has been evaluated. Sulfation reactivity was optimized as a function of the quantity of carbon and water used in preparing the pellets. The sulfation rate was independent of the type of carbon used. Cement kiln dust, a waste product, was also tested as an absorbent for SO_2 . TGA experiments on the extent of regeneration and SO_2 concentration in the off-gas under various pressures and temperatures are being conducted. This information is of importance to pressurized fluidized bed (PFC) operations. Cycling of Type III portland cement pellets between sulfation and regeneration conditions indicate good maintenance of capacity with little deterioration after 20 cycles. These cycling experiments are continuing.

Experiments are being made with a 3" rotary kiln to provide information on kiln performance under various operating conditions. Among the variables considered are temperature, residence time, extent of regeneration and SO_2 concentration in the off gas. The feasibility of an alternative regeneration scheme has been studied. The first step is the reduction of CaSO_4 to CaS , and the second step the conversion of CaS to CaO with CO_2 . However, the regenerability of CaS deteriorated with each sulfation/regeneration cycle.

1. Alternative Sorbent Systems

1.1 Sorption and Regeneration

Portland cements have very favorable properties for sulfur absorption and regeneration for fluidized bed combustion applications.^{1,2} Various types of portland cement were subjected to sulfation/regeneration cycling as fine particulates (powders) in a TGA. Sulfation was performed at 900°C using a simulated combustion gas, 0.25% SO₂, 5% O₂, 15% CO₂, and the balance N₂, at a total pressure of one atmosphere. The sulfation period utilized was 2 hours. Regeneration of the sulfated product was accomplished by flowing 5% CO, 20% CO₂, and balance nitrogen at 1000°C over the sample. When regeneration was completed, before sulfation was begun, about 5% O₂, 15% CO₂, and balance N₂ was added until no weight gain was observed (the residual CaS and reduced metals were oxidized). The cycling results obtained are presented in Table 1. The sulfation capacity of the second or third cycle decreased due to incomplete regeneration of the previous cycle. Portland cement Type III was found to be the most reactive, while portland cement Type V retains its sulfation reactivity longest (after eight cycles). Portland Type I and Type III cements show moderate losses in sequential cycles, while Type II appears to exhibit the largest cycling loss.

The experimental results of sulfation/regeneration cycling of portland cements indicates the extent of sulfation decreases due to incomplete regeneration of previous cycles. Portland cement contains about 2% free or uncombined lime,³ and it is thought, because of CaSO₄ formation, it is more difficult to regenerate than sulfated silicates. A possible explanation is that SO₂ has to diffuse through the unregenerated

CaSO_4 product layer, thereby decreasing the sulfation rate. An experiment was performed to try to understand the deterioration in sulfation/regeneration cycling of portland cement. A mixture of 20% reagent grade CaO powder with 80% β -dicalcium silicate powder (EDS), was subjected to sulfation/regeneration cycling in a TGA. The results are listed in Table 2 and confirm our explanation of this decrease. The addition of a few percent SiO_2 (clay, etc.) to portland cement would help by reacting with the free lime, after several cycles, to form calcium silicates. The calcium silicates being more regenerable than CaSO_4 would thereby sulfate to higher levels.

The initial investigation into the performance of portland cement pellets was designed to help us understand, and eventually optimize the reactivity/attrition characteristics of portland cement as a function of burnables or water used to prepare them. This would vary the porosity and surface area. Portland cement was mixed with varying percentages of burnables (carbon) or water to form pellets. It was found that the greater the quantity of burnables used, the higher the sulfation capacity but the lower the strength of the pellet formed.²

Granulated cement pellets were prepared using varying percentages of water and were cured for 30 days. They were then sulfated. Figure 1 shows the results. The overall rate increases with decreasing percentages of water. The sulfation rates of the same materials after having been soaked in water for 24 hours and then vacuum dried are also shown in Figure 2. A similar trend was observed. Table 3 shows the surface areas of these materials. Before measuring the surface area, the materials were heated to 900°C for four hours in a N_2 flow. The results

indicate that the surface area increases with increasing percentages of water. The cause of the increased sulfation rate due to a decrease in water content (decreased surface area) of portland cement pellets is still unknown. It is suspected that the structure of these portland cement pellets has undergone a change. Pore size distribution of these materials will be measured to provide a better understanding of the mechanism involved.

Portland cement is composed of a number of compounds the reaction of which with water leads to the setting and hardening of the cement. All the compounds present in portland cement clinker are anhydrous, but when brought into contact with water they are all decomposed, forming hydrated compounds. Since the original anhydrous compounds cannot exist in equilibrium with aqueous solutions, the ultimate result of the action of water must be complete hydration.³

The sulfation rates of portland cement Type III pellets, which were prepared using a range of carbon to cement ratios, were tested. Previously, it was shown², the greater the percent carbon addition, the higher the sulfation capacity. However, it does not increase drastically above 1% carbon addition. This trend is consistent with the surface areas of these materials (as shown in Table 3). The greater the percent carbon addition, the greater the surface area.

In a series of experiments the sulfation rate of portland cement Type III was tested by adding different types of carbonaceous materials. All the coals or carbonaceous materials used were 170/230 U. S. mesh in size (plant grind size), and were burned off before the first sulfation cycle. As shown in Figure 3, with 5% addition of different

types of coals in portland cement, the sulfation rates are approximately the same order of magnitude, about twice that of portland cement without carbon addition (including coconut charcoal² reported previously). The sulfation rate was essentially independent of the type of carbon used. However, silica fume additive had the highest sulfation reactivity among these. Silica fume is a waste material from Ferro-silicon electric furnace smelting plants, and was kindly supplied by the Hanna Mining Company. Analysis of the material showed⁴ it contained ~8% carbon and > 90% Si along with trace amounts of SiC, FeO, Al₂O₃, MgO, CaO, Na₂O, K₂O. The addition of a few percent silica fume to portland cement would help in the formation of calcium silicates. At steady state, in a continuous cyclic sulfation/regeneration, calcium silicate would form and its content would increase with the number of cycles, because it would stay in the structure once it has formed.

Because the regeneration rate of portland cement is substantially higher than that of lime, portland cement can be regenerated at lower temperatures. We attempted to run cyclic isothermal sulfation and regeneration at 900°C, but because of the low regenerability at 900°C, we then went to 950°C. The optimal temperature range in fluidized-bed combustion of coal using natural limestones is between 830°C and 900°C because of sintering and pore size development.⁵ Combustion and power cycle efficiency will be increased substantially if the temperature is raised to 950°C using portland cement as bed material. The silicates remain in calcined form and do not sinter at this higher temperature. In fact, an optimal temperature (~ 950°C) of maximum sulfur retention is observed for portland cement Type III pellet (16/20 U. S. mesh, 6.3% H₂O) as shown in Table 4.

Cyclic isothermal sulfation/regeneration reactions were run at 950°C using portland cement Type III granulated spherical pellets. The spherical pellets were 16/20 U. S. mesh in size and contained 5% Illinois No. 6 coal powder before the first sulfation cycle. Figure 4 shows the typical sulfation/regeneration cycling in a TGA. Sulfation was undertaken at one atmosphere pressure and a gas composition of 0.25% SO₂, 5% O₂ with the balance being N₂. The sulfation period utilized was 2 hours. Regeneration was performed at one atmosphere with a gas composition of 5% CO and 20% CO₂ in nitrogen. The results indicate that there is only slight sorbent reactivity decay after twenty cycles, even though some free lime is present to form CaSO₄ as previously suggested. The reason for the reactivity of the regenerated sorbent remaining high is that the lower regeneration temperature (950°C) seemed to prevent the sorbent from sintering. The combined effect of CaSO₄ formation and sintering probably lowered the cycling capacity. At lower regeneration temperatures (950°C), sintering was negligible, and cycling sulfation capacity decreased slightly. Higher regenerated SO₂ off gas concentration for a sulfur recovery process is also benefited by isothermal operations because additional internal heat (burning fuel to produce heat) is not needed to raise the regeneration temperature, hence no dilution of SO₂ off gas would take place.

1.2 Attrition Rates

The attrition rate of sulfated portland cement Type III was compared with that of sulfated Greer limestone. Attrition tests were performed at ambient temperature in a 2.2 cm diameter fluidized bed, having an air velocity of 5 ft/sec. Starting materials for attrition tests

were all in the size range of 16/20 mesh, weighing approximately 10 grams. The materials were screened after each test. The samples with sizes smaller than 20 mesh ($850\text{ }\mu\text{m}$), including material lost from the bed, were measured as a function of time. The results are shown in Figure 5 and are rather striking. Sulfated portland cement Type III pellets after being tested for 40 hours had little attrition losses ($\sim 7\%$). Sulfated Greer limestone was soft and consequently had a high attrition rate ($\sim 45\%$ attrition losses).

Figure 6 shows the particle size distributions for both materials after 40-hr attrition tests. The sulfated portland cement Type III pellets had a much smaller particle diameter distribution change than sulfated Greer limestone. These results clearly indicate that portland cement is highly superior in its resistance to attrition than natural limestone. Natural limestone has serious attrition loss problems when used in a fluidized-bed combustor.

1.3 High Pressure TGA

Portland cement pellets were tested in a CO_2 environment similar to that in pressurized fluidized-bed combustion of coal. For example, sulfation was undertaken using 90% CO_2 , 0.25% SO_2 , 5% O_2 with the balance being N_2 at a total pressure of one atmosphere. Since portland cement is not carbonated in this high CO_2 atmosphere, it can be readily sulfated. The sulfation capacity of portland cement Type III pellets after 2 hours at 800°C was about 20%. At 800°C , the equilibrium partial pressure of CO_2 above limestone is about 0.21 atm. Thus, portland cement when exposed to a higher partial pressure of 0.9 atm CO_2 , which is the equivalent gas composition in a pressurized fluidized-bed combustion of coal, still indicates a significant capacity for sulfation.

Since it is possible to regenerate calcium silicates at lower temperatures as well as in pressurized systems⁶, TGA experiments on the extent of regeneration at pressures above atmospheric are being planned.

1.4 Cement Kiln Dust

Cement kiln dust is a waste product that piles up nation-wide at the rate of 10 million tons per year. It is an inexpensive abundant material to utilize as an absorbent for SO_2 . Therefore we obtained some cement kiln dust from Lone Star Industries, Inc. Typical analysis of this cement precipitator dust is listed in Table 5. The composition is similar to natural limestone, CaO being the predominant constituent.

Sulfation capacity of the cement kiln dust pellets was about 20%. This is about the same order of magnitude as limestone. Silica can be added in a stoichiometric ratio to CaO in cement kiln dust, and in a continuous cyclic process, would react to form calcium silicates. Burnable carbon can be added during the pelletizing of the cement kiln dust to increase porosity, similar to portland cement pellets, and thereby increase the sulfation capacity.

2. Catalytic Sulfation

Most of the testing of sorbents in the TGA was conducted without the presence of water vapor in the gas atmosphere. Since in the combustion of coal, water vapor is always present, we therefore performed TGA runs to determine its effect on the sorption of portland cement samples. A water vapor concentration of 5% in the gas atmosphere was maintained. Figure 7 shows the sulfation rate of portland cement Type III powder with and without 5% steam. Steam catalyzes the sulfation reaction for lime, calcium silicates⁷, and portland cement.

Morgantown Energy Technology Center asked whether Al_2O_3 in calcium silicates or portland cement lowers the sulfation reactivity. The effect of addition of Al_2O_3 on the sulfation of CaSiO_3 (ROC/RIC) and portland cement Type III is shown in Table 6. No effect on the sulfation rates of these materials was shown.

The question was also asked whether some mineral oxides (such as iron oxide) in fly ash catalyze the sulfation reactivity of sorbent pellets and fly ash mixtures. Analysis of secondary cyclone fly ash LST-7B supplied by Argonne National Laboratory is as follows: Fe_2O_3 , 12.4%; SiO_2 , 30.6%; Al_2O_3 , 20.2%.

Addition of iron oxide has been shown to be catalytic to the sulfation of CaO ⁸ and silicates⁶. For example a Greer limestone blank was made by soaking it for 30 min in distilled water at 70°C and air dried. A similar method was used except instead of distilled water we used a saturated solution of $\text{Fe}_2(\text{SO}_4)_3$. Before sulfation began, the $\text{Fe}_2(\text{SO}_4)_3$ was decomposed to Fe_2O_3 . The results obtained are shown in Figure 8. The sulfation rate of Greer lime was approximately 25% higher with impregnated 0.5% Fe_2O_3 than the blank.

3. Alternative Regeneration Scheme

We studied the feasibility of the following regeneration reactions:

In fluidized-bed coal combustion, a two step regeneration process as above, is possible. The first step is the reduction of CaSO_4 to CaS . In the second step the CaS is converted to CaO with CO_2 . The results of

our experiments indicate that the regenerability of CaS deteriorates with each sulfation/regeneration cycle. The poor regenerability of CaS is also a concern for dolomite regeneration in fuel gas desulfurization⁹ where CaS is converted to CaCO_3 with CO_2 and steam.

4. The Kiln Regeneration Process

Although the 3" kiln has been operated at various angles and rotational speeds, in practice a 2° angle at 4.5 rpm gives us the 30 minute residence time needed for regeneration. 2200 grams of sorbent fills the entire length of the kiln, and a feed rate of 60 to 70 grams/min maintains this loading for continuous operation.

Listed below are residence times for portland cement pellets and limestone for two angles and fixed rotational speed.

<u>Angle</u>	<u>Kiln rpm</u>	<u>Residence time</u>	<u>Sorbent</u>	<u>Comment</u>
2°	4	60 min	Limestone	As is from Argonne FBC
2°	4	40 min	Cement pellets	16/20 US mesh spherical
2.5°	4	30 min	Limestone	As is from Argonne FBC
2.5°	4	20 min	Cement pellets	16/20 US mesh spherical

Kiln Feed Rate

<u>Screw feed rpm</u>	<u>Wt. of sorbent grams/min</u>
1	4.2
2	10.4
4	24.0
6	36.0
10	60.1

Studies are being conducted with the 3" rotary kiln to provide information on kiln performance under various operating conditions. Among the variables being considered are temperature, residence time, extent of regeneration and SO₂ concentration in the off gas. The effect of CO₂ concentration on the reductive decomposition of the sulfated sorbent will also continue.

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Table 1. Cycling of various types of portland cement powder between sulfation and regeneration conditions.

	Type I		Type II		Type III		Type V	
	Sulfation	Regeneration	Sulfation	Regeneration	Sulfation	Regeneration	Sulfation	Regeneration
1	51.1	90.4	45.8	90.7	55.5	93.4	41.9	88.6
2	43.8	84.9	35.8	100.0	48.4	100.0	37.2	97.3
3	36.6	100.0	33.1	100.0	46.2	92.1	33.9	100.0
4	34.0	100.0	31.8	82.6	43.4	98.1	35.7	100.0
5	34.5	91.7	27.6	100.0	42.0	100.0	36.5	94.6
6	33.9	100.0	32.2	98.7	41.1	99.7	34.8	100.0
7	37.1	97.8	30.0	100.0	41.1	100.0	36.3	100.0
8	36.3	—	29.4	—	40.4	—	36.0	—

Table 2. Effect of free lime on sulfation/regeneration of dicalcium silicate. 80% C₂S + 20% CaO (powder).

Cycle	Sulfation ^a	Regeneration ^b
1	37.95%	76.23%
2	35.70%	65.29%
3	31.81%	65.29%
4	24.40%	

^a 0.25% SO₂, 5% O₂, and balance N₂ at 900°C in 2 hrs.

^b 5% CO and 95% N₂ at 1000°C.

Table 3. Surface area measurements of portland cement pellet mixed with varying percentages of carbon and water.

% H ₂ O	Surface area (m ² /g)
6.3	1.61
12.3	1.95
22.9	2.05
<hr/>	
% C	
0	2.48
1	2.56
5	2.57

Table 4. The effect of temperature on the sulfation of portland cement Type III granulated pellets (16/20 mesh, 6.3% H₂O) in TGA.

<u>Sulfation temperature (°C)</u>	<u>% Sulfation after 2 hours</u>
800	32.8
850	36.9
900	39.7
950	43.0
1000	41.8

Sulfation condition: 0.25% SO₂, 5% O₂, 15% CO₂, and the balance N₂.

Table 5. Chemical analysis of Lone Star cement kiln dust.

SiO ₂	9.10
Al ₂ O ₃	2.33
Fe ₂ O ₃	2.30
CaO	44.51
MgO	1.49
SO ₃	0.54
Loss of ignition	37.64
K ₂ O	1.51
Na ₂ O	<u>0.16</u>
Total	99.58

Table 6. The effect of Al_2O_3 addition on the sulfation of CaSiO_3 and portland cement Type III.

Material	Additive	% sulfation in 2 hours
CaSiO_3	0	27.4
Port. III	0	51.9
CaSiO_3	5% Al_2O_3	29.8
CaSiO_3	10% Al_2O_3	25.0
Port. III		53.1
CaSiO_3	50% Al_2O_3	22.9
Port. III		47.4

Figure 1. Effect of % water on the sulfation of portland cement Type III granulated pellets (30 day cure).

Figure 2. Effect of % water on the sulfation of portland cement Type III granulated pellets which have been soaked (after 30 day cure) for 24 hrs and then vacuum dried.

Figure 3. Sulfation rates of portland cement Type III granulated pellet mixed with 5% different types of carbonaceous materials.

Figure 4. Cycling of portland cement Type III granulated spherical pellets (16/20 mesh, 5% Illinois No. 6 coal powder) between sulfation and regeneration conditions.

Figure 5. Attrition rates of sulfated sorbents for sample weight loss > 20 mesh with initial particle size 16/20 mesh.

Figure 6. Particle size distribution of sulfated sorbents after 40 hour FB test at room temperature.

Figure 7. Sulfation rate of portland cement Type III with 5% steam and without steam at 900°C in 0.25% SO₂, 5% O₂, 15% CO₂, and a balance of N₂.

Figure 8. Calcination and subsequent sulfation of 16/20 mesh Greer limestone with 0.25% SO₂, 5% O₂, and balance N₂ at 900°C.
 O Greer limestone soaked in 70°C distilled water for 30 min and then dried in air.
 Δ Greer limestone soaked in 70°C Fe₂(SO₄)₃ aqueous solution (distilled water) for 30 min and then dried in air (0.5% Fe₂O₃).

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