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REGENERATIVE PROCESS FOR DESULFURIZATION OF HIGH TEMPERATURE COMBUSTION AND FUEL GASES

PROGRESS REPORT NO. 15
JANUARY 1 - MARCH 31, 1980

April 1980

DEPARTMENT OF ENERGY AND ENVIRONMENT

BROOKHAVEN NATIONAL LABORATORY
UPTON, NEW YORK 11973

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(Coal Conversion and Utilization-
Direct Combustion of Coal - TID-4500)

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April 1980

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TABLE OF CONTENTS

	PAGE
ABSTRACT	iv
I. INTRODUCTION	1
II. BASIC CHEMISTRY	2
Desulfurization	2
Regeneration.	4
III. PROCESS DESCRIPTION	5
Sorbent Preparation	6
Desulfurization	6
Sorbent Regeneration.	9
IV. PROCESS APPLICATION	9
Power Generation/Desulfurization.	11
Sorbent Regeneration.	12
V. COMPARISON OF PLANTS AND COSTS.	14

AN ENGINEERING STUDY
ON THE USE OF
REGENERATIVE CALCIUM SILICATE SORBENTS
FOR
AFB POWER GENERATION
FROM
HIGH SULFUR COAL

P.J. McGauley and A.S. Albanese
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ABSTRACT

A new regenerative sorbent process for desulfurizing fluidized-bed combustion gases is presented. The sorbent was developed by Brookhaven National Laboratory and is prepared from commercial silicate bearing portland cement. The chemical reactions and mechanical procedures involved in preparing the sorbent, in desulfurizing the flue gas, in regenerating the spent sorbent, and in producing elemental sulfur from the regenerator product gas are discussed. The performance of natural limestone sorbents is compared with that of portland cement pellets in each of the above operations. Conceptual engineering designs and cost data for atmospheric fluidized-bed (AFB) power generation plants using limestone and portland cement sorbents are also presented. Cost and limestone process data were obtained from U.S. Government publication PRS-23 EPA-600/7-77-126 which was prepared by General Electric, Bechtel and Foster Wheeler Energy Corporations and the Tennessee Valley Authority. The results of our analyses indicate that the cost of generating electricity by a regenerative portland cement sorbent plant would be about 12% lower than for "once-through" limestone AFB plants, about 17% lower than conventional power plants using wet limestone scrubbers and about 12% lower than regenerative magnesium oxide (MgO) scrubber plants.

I. INTRODUCTION

For the last several years, the Process Sciences Division of the Department of Energy and Environment at Brookhaven National Laboratory has been investigating calcium silicates for use as regenerative sorbents in the desulfurization of flue gases. The results of these studies are reported in BNL Progress Reports, Nos: BNL 50582, 50620, 50669, 50706, 50750, 50809, 50866, 50891, 50944, 50992, 51040 and 51106.

During the past year, we have identified portland cement as a low cost source of calcium silicates for potential use as regenerative sorbents in the removal of sulfur from hot flue gases. Cyclic sulfation/regeneration thermogravimetric (TGA) experiments have been run using simulated fluidized-bed combustion (FBC) and regeneration gases, as well as, hot (1000°C) attrition loss experiments in small scale fluidized-bed equipment. We have found that the sulfur reactivity of 16/20 mesh Portland III pellets is not appreciably diminished after twenty sulfation/regeneration cycles. Our results also indicate that these pellets are superior to Greer limestone in both sulfur dioxide (SO₂) capacity and attrition resistance. Furthermore, calcium silicates are suitable for use as sorbents in pressurized systems. The SO₂ equilibrium partial pressures for reductive decomposition of these sorbents are so high that, unlike limestone, thermodynamic equilibrium will not limit the concentration of SO₂ achievable in the regenerator off-gas. Moreover, regeneration rates of sulfated Portland III pellets are appreciably higher than for sulfated lime (Greer).

The optimum temperature range for sulfur absorption by natural limestones in coal-fired fluidized-bed combustors has been found to lie

between 830° and 900°C; above 900°C the limestone sorbents become less effective and may become deactivated. From a reactivity standpoint, combustion temperatures can be increased to between 975 and 1025°F when using portland cement as bed material. Calcium silicates do not sinter at these higher temperatures and maintain their reactivity. In fact, we have found (by TGA experiments) that the optimum temperature for sulfur retention by Type III Portland Cement pellets (16/20 U.S. mesh, 6.3% H₂O) is about 1000°C. Operating at these higher temperatures could result in combustion and power cycle efficiency increases and in a reduction in boiler cost.

II. BASIC CHEMISTRY

Both the BNL sorbent process and limestone and dolomite sorbent processes involve heterogeneous reactions between hot gaseous sulfur dioxide and dry solid calcium compounds. The chemical reactions of both processes are similar and are discussed and compared below.

Desulfurization

The principal reactions occurring in a fluidized-bed combustor when limestone sorbents are used may be summarized as follows:

Reactions (1) through (3) are the oxidation reactions of the carbon, sulfur and hydrogen contained in the coal feedstock and are obviously common to all coal combustion systems. Reaction (4) is the decomposition, or calcination, of limestone and Reaction (5) the

sulfation reaction of calcined limestone. Reaction (4) is highly endothermic, requiring about 42.6 kcal/mole of limestone, while Reaction (5) is exothermic. The direct reaction between limestone and sulfur dioxide,

may also occur; however, the porosity of raw limestone is low so that sulfation of calcium carbonate occurs at a slow rate. Therefore, in order to achieve high rates of SO₂ removal, Reaction (4) must readily occur under the prevailing operating conditions of the fluidized-bed combustor.

Calcination of limestone occurs only when the decomposition equilibrium partial pressure of CO₂ is greater than the prevailing partial pressure of CO₂ in the fluidized-bed combustor. Dolomite behaves similar to limestone except that decomposition of the magnesium carbonate component of the dolomite occurs at lower temperatures than calcium carbonate.

In the case of calcium silicates, no calcination is required and all major reactions occurring in the fluidized-bed are exothermic. For silicates, the SO₂ sorption reactions may be summarized as follows:

where x is 1 for mono-, 2 for di- and 3 for tricalcium silicate. At 880°C and atmospheric pressure, all of these silicates are thermodynamically capable of reducing the concentration of SO₂ in fluidized-bed combustor flue gas to the recently promulgated EPA limit (90% SO₂ removal for high sulfur coals). At 1000°C tricalcium silicate can still meet EPA requirements, whereas, the SO₂ equilibrium partial pressures for mono- and dicalcium silicates are too high. However, most commercial grades of

portland cements contain a large fraction of tricalcium silicate (e.g. 73 to 86% of the silicates present in Portland Type III is tricalcium silicate), therefore, they should be suitable (thermodynamically) for service at this higher temperature, if desired.

Regeneration

Regeneration of the CaSO_4 produced in the fluidized-bed by the desulfurization reaction can be accomplished by reacting the sulfate with an appropriate reducing medium. With a CO reducing gas, the overall reaction is:

which proceeds according to the following two sequential steps:

Experimental evidence indicates that the second step, Reaction (10), is rate controlling and determines the maximum concentration of SO_2 that can be produced during regeneration.

The main objectives of a regeneration process are: (1) to obtain a high degree of regeneration in a short period of time, (2) to obtain a high SO_2 partial pressure in the regenerator off-gas and (3) to effect the above without reducing the sulfation reactivity of the sorbent. The rate of regeneration and SO_2 partial pressure increase with increasing temperature but sorbent reactivity tends to decrease. With limestones sorbents the deactivation becomes prominent at regeneration temperatures above 1050°C . Thermal decomposition of CaSO_4 in the absence of a reducing medium is theoretically possible but is not feasible because temperature requirements are prohibitively high, i.e., the sorbent becomes deactivated during regeneration due to sintering.

The reactions for regenerating sulfated calcium silicates are similar to those for regenerating calcium sulfate and may be indicated as follows:

where x takes on integer values of from 1 to 3. As in the case of lime, Reaction (13) controls the rate of regeneration and the maximum concentration of SO_2 achievable in the regenerator off-gas. However, the SO_2 equilibrium partial pressures for these sorbents are appreciably higher than for limestone (e.g. 4 atm. at 1000°C for sulfated tricalcium silicate versus 0.1 atm for sulfated lime); therefore, higher SO_2 concentrations should be attainable during regeneration and thus more economical conversion of SO_2 to sulfur or sulfuric acid. Furthermore, sulfated silicates can be regenerated under pressure. Thus, for pressurized fluidized-bed combustion systems, the regenerator and combustor can be operated isobarically, facilitating the transfer of solids between combustor and regenerator.

III. PROCESS DESCRIPTION

The basic unit operations/processes for desulfurizing fuels by the silicate sorbent process are indicated in the block flow diagram of Figure I and are described below. A comparison with once-through limestone system is also presented.

Sorbent Preparation

The sorbent preparation procedures for the limestone process include the selection of suitable deposits of natural limestone, limestone mining, crushing and sizing, and the transport of large quantities of prepared limestone to the site of the power generating plant. During preparation all fractions of the mined limestone that are unsuitable in particle size for use as sorbent are rejected and sent to disposal.

In the silicates process, cement powder is first mixed with a fine spray of water to bring its moisture content up to a maximum of 10% by weight. Minor quantities of finely divided conditioning agents may be premixed with the cement prior to moistening. The conditioned mix is then blended in the presence of moisture. Seed formation and growth is accomplished by the continuous layering of fines and water on the moving bed of cement particles. These blending and agglomerating procedures are not energy intensive and are used in commercial plants that produce low cost iron ore pellets for use as blast furnace feed. Curing, sizing, and drying of the moist agglomerated sorbent are also required. The procedures and equipment employed in these operations are commonly used in the high volume, low cost processing of minerals in the metallurgical industry.

Desulfurization

Desulfurization in both the limestone and silicate sorbent processes occurs simultaneous with the combustion of coal in the fluidized-beds of AFB steam generators. That is, the sulfur dioxide generated by the combustion of coal is removed insitu by reaction with these sorbents. In most AFB steam generators of current design, the beds of fluidized sorbent are enclosed by

steam generating water walls. Additional heat transfer tubing for steam generation and superheating is located within the beds. The beds are maintained at a fixed temperature, below the fusion point of the coal ash. Gas flow is usually maintained at a rate that elutriates essentially all of the coal ash from the sorbent beds.

Both fuel and sorbent are fed to the fluidized-beds continuously. The feed rate of fuel is regulated to produce steam at the desired rate; the feed rate of sorbent is adjusted to control the concentration of sulfur dioxide in the combustor flue gas at or below a required or desired level. Bed depth is maintained at an approximately constant level by withdrawing spent sorbent from the bed at rates proportional to the fresh sorbent feed.

In the limestone process, crushed and sized sorbent is fed to the hot beds of the combustor without preheating. Upon entering the bed, the fresh limestone is dried, heated to the operating temperature of the bed and calcined. The calcination process generates carbon dioxide by the reaction: $\text{CaCO}_3 = \text{CaO} + \text{CO}_2$. The energy for these endothermic operations is supplied by the combustion of additional quantities of coal (above that required for the desired rate of steam production) in the combustor. The energy consumption for calcining limestone in commercial kilns normally ranges from 2.5 to 3.0 million BTU per ton.

As discussed above, the calcination of limestone generates calcium oxide (CaO) for use in desulfurization. For good SO_2 sorption, calcination must precede desulfurization. Therefore, the limestone sorbent process is normally operated at bed conditions that are favorable to calcination (about 900°C at an operating pressure of 1 atm.). Furthermore, maximum calcium utilization has been found to occur at temperatures between 830 and 900°C .

At higher operating temperatures the limestone sorbents become less effective and may become deactivated. Thus, fluidized-bed combustors utilizing limestone are usually designed for operation at temperatures between 830 and 900°C.

When limestone is calcined, carbon dioxide is expelled from the outer surfaces and from within the limestone particles. The expulsion of gas from the interior regions results in an increase in porosity and thus sorbent reactivity; however, this weakens the limestone particles and reduces their resistance to attrition. Thermal fracture also generates fines. The major fraction of these fines are elutriated from the fluidized-beds by the combustor flue gas and accumulate as an undesirable dust load in the downstream equipment. Furthermore, for a given dust collection system, the higher the concentration of particulates in the flue gas, the greater the fugitive emissions. Increase fugitive emissions increases potential health hazards.

In the regenerative silicates process, hot sorbent which is recycled from the regenerator is the major source of sorbent feed to the AFB combustor. Makeup sorbent is only a small fraction of the total sorbent feed stream to the combustor. All of the chemical reactions occurring in the fluidized-beds are exothermic and do not consume fuel values. These reactions do not include calcination and do not result in the expulsion of gases from the interior of the sorbent particles. Thus, sorbent losses are reduced to relatively minor quantities of finely divided material that separate from the parent particles due to particle-particle contact and particle-wall contact. Furthermore, with Portland cement as bed material, combustion temperatures can be increased to between 975 and 1025°C.

Operation at these higher temperatures could result in increased combustion and power cycle efficiencies, elimination of carbon burnup cells, and a decrease in boiler cost. The thermal efficiency of regeneration also improves with increased bed temperature.

Sorbent Regeneration

Most AFB steam generators of commercial design use limestone on a "once-through" basis because limestone is relatively cheap and because an economic process for regenerating limestone sorbents is not available. Moreover, attrition losses are high and reactivity decreases with cyclic usage of limestone.

Sulfated calcium silicate sorbents can be regenerated by contacting the hot solids with fuel and air in a fluidized-bed regenerator. The supplies of both fuel and air to the regenerator are regulated to provide both energy and reducing agents to satisfy the chemical reactions. The operating temperature of the regenerator is maintained between the same limits as those of the fluidized bed combustor. Isothermal operation of the combustor and regeneration eliminates preheating of the feed and reduces the heat duty of the regenerator. Moreover, the lower demand for fuel and air results in a higher concentration of SO_2 in the product gas from the regenerator.

IV. PROCESS APPLICATION

U.S. Government Report No. PRS-23 EPA-600/7-77-126 (November 1977) entitled "Utility Boiler Design/Cost Comparison: Fluidized-Bed Combustion Versus Flue Gas Desulfurization", provides process and cost comparisons between three types of utility power plants: (1) a conventional coal-fired

power plant with stack gas scrubber (CWS unit), (2) a "once-through" limestone sorbent atmospheric fluidized-bed power plant (AFB unit), and (3) a "once-through" dolomite sorbent pressurized (10 atm.) fluidized-bed plant (PFB unit). All plants were designed for about the same feed rate of coal of a specified type. The output of the CWS and AFB plants were estimated at about 800 MWe, while that of the PFB unit was about 900 MWe.

The above report was prepared under a prime contract between the Environmental Protection Agency (EPA) and the General Electric Corporation (GE), and subcontracts with Bechtel and Foster Wheeler Energy Corporations. It was reviewed for conformity with utility operating and financial practices, and revised, by the Tennessee Valley Authority (TVA). Capital and operating costs were evaluated on the basis of historical costs for mid-year 1975.

The GE AFB power plant uses limestone on a "once-through" basis and was designed to operate in compliance with former EPA regulations that required 83% sulfur capture and permitted 1.2 pounds of SO_2 in the effluent gas per million BTU of feed coal energy. A calcium to sulfur ratio of 2 to 1 was assumed by GE. In their review of the GE report, the EPA protested this ratio as being unrealistically low. Furthermore, on the basis of current EPA regulations that require 90% sulfur capture, we believe that a calcium to sulfur ratio of 4 to 1 is more reasonable. At this ratio, the feed rate for limestone sorbent would be double that assumed by GE.

The flowshceme for the AFB limestone plant (GE) is illustrated in Figure II. The material flows indicated are for 90% capture of sulfur, a calcium to sulfur ratio of 4 and a net electrical output of 814 MW.

The corresponding flowscheme for the regenerative silicates AFB plant at 90% sulfur capture and an output of 814 MWe is presented in Figure III. Hot regenerated sorbent (217,000 lb/hr) provides the major fraction of the sorbent feed, while fresh makeup sorbent (10,800 lb/hr) provides the minor fraction. Makeup sorbent, which is consumed at rates determined by the attrition rate of the portland pellets, is prepared from commercially available portland cement in off-site facilities by the agglomeration procedures described previously and developed at BNL. We have estimated these losses at about 5% of the circulating sorbent load. This estimate appears to be conservative based on hot (1000°C) attrition tests run at BNL on 16/20 U.S. mesh Portland III pellets in a small-scale fluidized-bed. Our results indicate that about 4% of the initial charge of material is lost by attrition after 8 hours of operation and that recycling tends to strengthen the particulates. In commercial size equipment attrition rates may be lower than in our laboratory equipment.

Power Generation/Desulfurization

In the AFB plant of GE design, sized and dried limestone prepared at the site is blended with coal and fed to the fluidized-beds in a fixed proportion corresponding to the required calcium to sulfur ratio. Spent sorbent is drained by gravity at rates that maintain the beds at constant level. The beds are designed to operate at a temperature of about 840°C, and a superficial velocity of up to 12 feet per second with 20% excess air. Coal fines amounting to a maximum of 15% of the heating value of coal feed are assumed to be elutriated from the beds. The fines are captured in dust collection equipment and fed to carbon burnup cells that operate in a non-desulfurizing mode at about 1100°C with 30% excess air. Waste solids are

transported to an on-site disposal area. Facilities for mining and transporting limestone to the power plant site are not included in the scope of the GE study.

In the regenerative AFB plant, the facilities for preparing limestone sorbent are replaced by facilities for transporting and handling makeup and hot regenerated sorbent. Fresh sorbent is prepared in off-site facilities. A system of 20 to 25 mobile, insulated cylindrical vessels equipped for transport by crane or rail and hoist are provided. These vessels receive hot sorbent from the regenerator by gravity flow and transport it to combustor feed bins where makeup sorbent is added. The feed bins are insulated and located above the combustor. The combined sorbent stream is fed by gravity to the fluidized-bed combustors in proportion to the coal feed. Hot spent sorbent from the beds is transported to the regenerator by a similar procedure. The design of the horizontal transport system would be similar to those used in the steelmaking industry for transporting liquid "hot metal" from blast furnaces to refining (steelmaking) furnaces. The vertical hoists would be similar in design to the hoists used in underground coal and metal mines for delivering product and waste to surface facilities. Each mobile vessel is sized to receive a two hour supply of sorbent from the steam generator when the plant is operating at design capacity.

Sorbent Regeneration

No regeneration process is involved in the power plant of the GE study. The silicates process uses a single insulated fluidized-bed regenerator and auxiliary equipment. The regenerator is approximately 25 feet internal diameter by 40 feet high (excluding heads) and is equipped with a plenum windbox and damper system that permits 70% turndown of fluidization gases.

The auxiliary equipment includes two insulated surge bins, one located above and the other below the regenerator. Each is sized to hold enough sorbent for five hours of operation at the design capacity of the power plant.

The regenerator is connected to the surge vessels by internally insulated vertical piping. The flow of sorbent between the surge bins and the regenerator is by gravity and is modulated by motor operated angle valves. Sorbent is transferred between combustor and regenerator by the transport system described above. The regenerator and its associated sulfur plant (described below) are located central to the steam generators. One regenerator services the entire power plant.

The air supply to the regenerator and sulfur furnace, which would normally be less than 4% of the flow of combustion air to the steam generator, is preheated by the steam generator (FBC's) preheater. Tailgas from the sulfur plant is recycled to the FBC's, where it is desulfurized, and exhausted with the combustion flue gas.

Wet coal, of the same type used for combustion, is fed to the regenerator and sulfur furnaces at a rate equal to about 5.5% of the feed to the steam generators. The coal is pulverized and dried in a hammer mill by contacting it with a slip-stream of tempered regenerator flue gas. A portion of the dried coal is fed to the regenerator to reduce the sulfated silicates; the remainder is fed to the sulfur plant.

Sulfur Production

The hot (955°C) off-gas from the regenerator, estimated at 14 to 18% SO₂, is reacted with dry pulverized coal in a sulfur reduction furnace. The coal feed rate to the furnace is regulated to produce a furnace product gas of about 1100°C containing hydrogen sulfide and sulfur dioxide in a molar

ratio of 2 to 1. This ratio is required by the downstream catalytic section of the Claus plant. The product gas from the furnace, which contains elemental sulfur, is cooled by heat exchange with water in a steam generating sulfur condenser. The resultant steam is available for export to the power generating plant. The cooled gas is reheated and passed through a series of catalysts beds, producing additional sulfur by reactions between H_2S and SO_2 . This sulfur is condensed by cooling the product gas in condensers that generate steam at about 20 psig.

The modified Claus process discussed above is similar to the sulfur recovery processes used in the "flash smelting" section of metallurgical plants of industrial design. These plants are commercially available and are operating at a number of copper and nickel smelters in the free world.

V. COMPARISON OF PLANTS AND COSTS

Material and energy balances and cost data for nine power plants of similar capacity are presented in Tables I and II, respectively. The data for three of the plants (Items 1, 6, and 8) were extracted directly from the GE report and correspond to 83% sulfur capture. The balances and cost data for the plants identified by Items 2, 3, 7 and 9 were modified to reflect the new EPA source standard of 90% sulfur removal. The data for the regenerative silicates process are presented in rows 4 and 5. The capital cost for the silicates process was estimated by comparing the costs of the limestone and regenerative magnesium oxide scrubber processes and by comparing the equipment items of the regenerative silicates system with the regenerative magnesium oxide scrubber process. In the silicates case, no credit was taken for increased combustion and power cycle efficiencies and byproduct sulfur was assumed to have zero market value.

The results of our analyses indicate that the cost of generating electricity by the AFB regenerative silicates sorbent plant is about 12% lower than "once-through" limestone AFB plants, about 17% lower than wet limestone scrubber plants and about 12% lower than regenerator MgO scrubber plants.

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TABLE I - COMPARISON OF PROCESSES

ATMOSPHERIC FLUIDIZED BED (AFB) PROCESSES							
TYPE OF SORBENT	SORBENT		COAL	PRODUCTS			
	POUNDS PER HOUR		Wet Basis	POWER MWe	POUNDS PER HOUR		
	New	Recycle			ASH	SPENT SORBENT	SULFUR
1. *Limestone 83% S capture	184,969	NONE	715,425	814.2	68,680	169,300	NONE
2. Limestone 90% S capture	359,000	NONE	715,425	790.5	68,680	272,800	NONE
3. Limestone 90% S capture	370,000	NONE	736,800	814.2	70,740	281,200	NONE
4. BNL Regenerative 90% S capture	10,625	213,500	715,425	801.0	68,680	10,625	24,700
5. BNL Regenerative 90% S capture	10,800	217,000	727,200	814.2	69,330	10,800	25,400
STACK GAS SCRUBBER PROCESSES							H ₂ SO ₄
6. *Limestone 83% S capture	125,000	NONE	730,600	795.5	70,140	109,200	NONE
7. Limestone 90% S capture	135,500	NONE	730,600	795.5	70,140	118,400	NONE
8. *Mgo. Regenerative 83% S capture	750	35,500	730,600	795.5	70,140	NONE	75,000
9. Mgo. Regenerative 90% S capture	820	38,500	730,600	795.0	70,140	NONE	78,500

*Data for these plants from report ~PRS-23 EPA-600/7-77-126.

TABLE II - COMPARISON OF COSTS

AFB PROCESSES		COST OF ELECTRICITY (Mills/KWH)			
Type of Sorbent	\$/KW	CAPITAL	O. & M.	FUEL	TOTAL
1. *Limestone 83% S capture	712.4	22.52	2.61	9.53	34.66
2. Limestone 90% S capture	756.0	24.61	3.55	9.82	37.98
3. Limestone 90% S capture	734.0	23.20	3.47	9.81	36.48
4. BNL Regenerative 90% S capture	653.8	21.01	1.85	9.69	32.55
5. BNL Regenerative 90% S capture	653.8	20.67	1.82	9.69	32.18
S T A C K G A S S C R U B B E R P R O C E S S E S					
6. *Limestone 83% S capture	782.3	24.73	3.74	10.10	38.57
7. Limestone 90% S capture	793.2	25.07	3.93	10.10	39.10
8. *MgO. Regenerative 83% S capture	728.0	23.01	3.55	10.10	36.66
9. MgO. Regenerative 90% S capture	728.0	23.01	3.57	10.10	36.68

*Data for these plants from report ~PRS-23 EPA-600/7-77-126.

Figure I. Basic Flow Diagram - BNL Process Regenerative Desulfurization of Coal in AFB Power Plants

Figure II. AFB Power Plant - Limestone Sorbent Flowscheme, Material & Energy Balance. Basis - Publication # PRS-23-EPA-600/7-77-126 Utility Boiler Design/Cost Comparison. November 1977.

Figure III. AFB Power Plant - Regenerative BNL Sorbent Flowscheme, Material & Energy Balance