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MULTI-CONTAMINANT CONTROL GRANULAR-BED FILTER

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5.6 Multi-Contaminant Control Granular Bed Filter

CONTRACT INFORMATION

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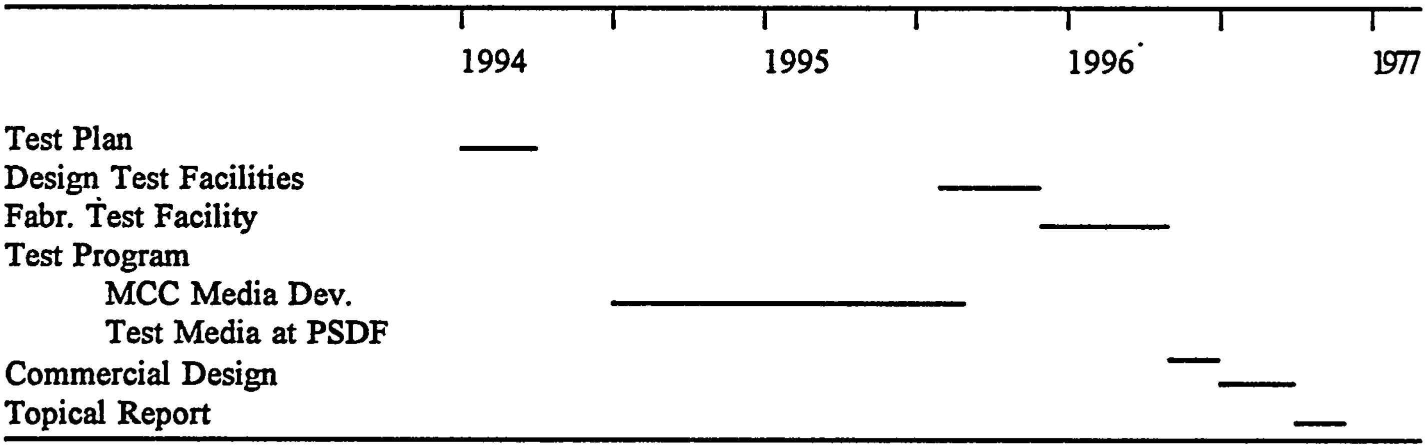
Period of Performance	October 1992 to April 1994

Schedule and Milestones

FY94-96 Program Schedule



OBJECTIVES

The objective of this phase of the Moving Granular Bed Filter (GBF) Development Program is to develop a GBF for the control of particulates and other contaminants found in high pressure and high temperature coal-derived gas streams. The filter should be able to remove particulates and one or more contaminants such as sulfur compounds, nitrogen compounds, alkali compounds, halogenated compounds, heavy metals and tars. The multi-contaminant control granular bed filter should be applicable to reducing and/or oxidizing conditions.

Specific objectives of the program are:

- To identify and define an approach to multi-contaminant control using a moving granular bed filter. The approach chosen for multi-contaminant control is justified with supporting data and information from the literature.

- To develop a test plan which includes the conceptual design of component test facilities, a description of tests which includes experimental procedures, operating conditions, duration of tests, number of tests and data to be collected.

- To design, procure and install experimental test facilities.

- To conduct tests to evaluate the moving granular bed filter for multi-contaminant control.

- To develop a commercial-scale design and economic analysis for a multi-contaminant control granular bed filter in PFBC or IGCC applications.

BACKGROUND INFORMATION

The granular bed filter was developed through low pressure, high temperature (1600°F) testing in the late 1970's and early 1980's (Guillory, 1980). Collection efficiencies over 99% were obtained. In 1988, high pressure, high temperature testing was completed at New York University, Westbury, N.Y., utilizing a coal-fired pressurized, fluidized bed combustor. High particulate removal efficiencies were confirmed as it was shown that both New Source Performance Standards and turbine tolerance limits could be met (Wilson, 1989).

The early scale-up work of the granular bed filter indicated potential limitations due to size, cost, and mechanical complexity. These limitations were addressed in the base contract of the present program (Wilson et al, 1992). It is currently proposed to use large diameter filters with 6 mm spherical filter medium to increase filtration capacity and reduce system complexity. Figure 1 shows a granular bed filter designed for a 100 MWe KRW (air) gasifier. The filter has an inside diameter of 14 ft with a nominal bed depth of 5 ft.

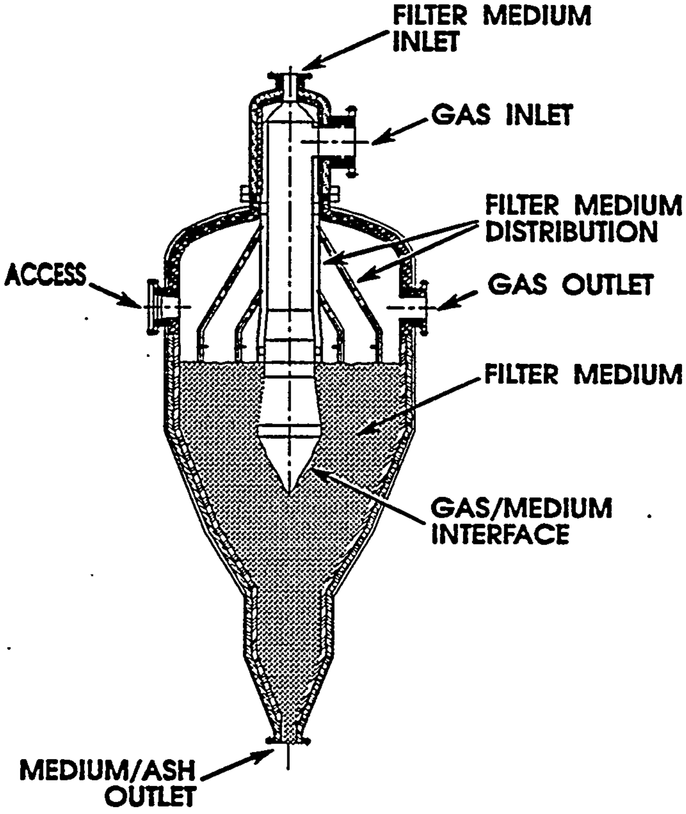


Figure 1. **Granular Bed Filter for 100 MWe KRW (Air) Gasifier**

PROJECT DESCRIPTION

A multi-contaminant control filter requires that either a chemically reactive sorbent be added to the gas stream and then removed in the filter or that the filter medium itself be chemically reactive. An application in which a fine grained sorbent is added to the gas stream and is then removed with the filter would be applicable to a granular bed filter as well as other types of filtration processes. We have chosen to use a chemically reactive filter medium to take advantage of the inherent characteristics of a granular bed filter which are long residence times for both the filter medium and the gas which is in contact with the filter medium.

The chemically reactive filter medium can be either a regenerable or non-regenerable sorbent. Others are currently working on the development of regenerable zinc titinate sorbents (Ayala et al, 1992). Such a sorbent would have potential use as a granular bed filter medium if it possesses sufficient strength and attrition resistance for many cycles through a granular bed filter and a sorbent regenerator. Another approach is to use a sorbent which has a finite life and is removed from the filter system after it is spent.

We propose to investigate a non-regenerable filter medium composed of a mixture of limestone and clay for the control of sulfur and alkali contaminants in coal-derived gas streams. Such a sorbent would also have the potential for the control of halogenated compounds, trace metals and tars.

In this concept, the filter medium would be composed of 6 mm chemically reactive spheres which are the same size as the filter medium used for particulate control. The rate of circulation of the filter medium in the GBF for particulate control is much greater than the rate at which reactive filter medium would need to be added to the filter for multi-contaminant control. Fresh reactive filter medium would be added to the circulating filter medium before it enters the filter. After the filter, spent filter medium would be removed from the circulation loop at the rate at which it is added.

Limestone for Sulfur Control

Limestone is widely used for the control of sulfur contaminants in both coal gasification and combustion processes because of its effectiveness, low cost and wide availability. Crushed limestone may not be an effective GBF filter medium because its irregular shape does not have the solids flow characteristics needed for a GBF and may have high attrition rates due to the loss of edges and corners formed in the crushing process.

Rather than using crushed limestone as the filter medium, it will first be pulverized, mixed with clay for alkali control, and then agglomerated into 6 mm spheres using suitable binders. The agglomerated limestone/clay spheres have the advantages of an improved shape for flow in a GBF, an increased micro-pore size and improved attrition properties.

Previous investigators have found that agglomerating finely powdered limestone improved its chemical reactivity and the temperature range of its reactivity, and that the addition of binders during agglomeration could improve mechanical strength. Shen and Albanese (1978), Voss (1983), and Spitsbergen et al. (1988) all demonstrated that calcined agglomerates of powdered limestone have considerably higher reactivity than calcined, similar-sized particles of the naturally occurring stone and may have increased mechanical strength. Zhang et al. (1989) demonstrated that not only is the chemical reactivity of calcined limestone improved by agglomeration, but also the temperature range for chemical reactivity.

Limestone can react directly with H_2S by the reaction route shown in equation 1. If the temperature is high enough, the limestone first calcines to calcium oxide and reacts by the route shown in equations 2 and 3.

$$CaCO_3 + H_2S = CaS + CO_2 + H_2O \qquad (1)$$

$$CaCO_3 = CaO + CO_2 \qquad (2)$$

$$CaO + H_2S = CaS + H_2O \qquad (3)$$

$$CaO + COS = CaS + CO_2 \qquad (4)$$

$$CO + H_2O = CO_2 + H_2 \qquad (5)$$

Carbonyl sulfide also reacts with calcium oxide as shown in equation 4. The extent of the above reactions are limited by the equilibrium concentrations.

Figure 2 shows the calculated equilibrium concentrations for H_2S and COS in the presence of limestone for fuel gas resulting from the KRW (air) gasifier. The equilibrium H_2S concentration decreases with temperature until a minimum value of 90 ppmv is reached at the calcination temperature of calcium carbonate, 1692°F. After the calcium carbonate calcines to calcium oxide, the equilibrium concentration of H_2S increases with temperature. The equilibrium concentration of COS depends on the concentration of H_2S. The minimum equilibrium COS concentration is 11 ppmv and it also occurs at 1692°F.

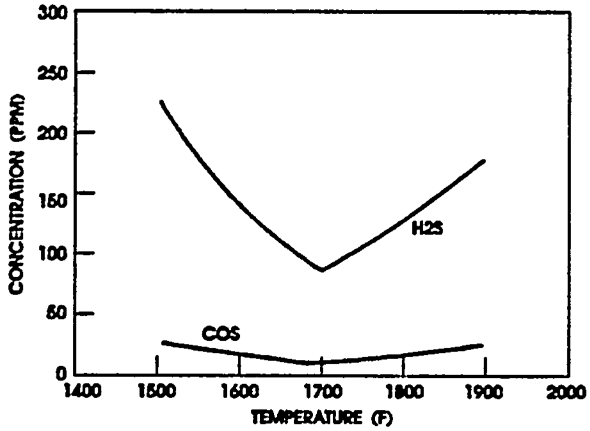


Figure 2 **Equilibrium Concentration for H_2S and COS for KRW (Air) Gas**

Pilot plant tests conducted at IGT (Goyal, 1988) showed that in their gasifier, it is possible to achieve 85% or more approach to the equilibrium concentration of H_2S. Assuming an 85% approach to the equilibrium concentration of H_2S, the H_2S concentration at the outlet of the GBF would be 106 ppmv. If the COS has the same approach to its equilibrium concentration, its concentration would be 13 ppmv. The combined concentration of H_2S and COS would be 119 ppmv which corresponds to 97.3% sulfur removal for the 2.68% sulfur coal used in the Wansley study (Southern Company Services, 1991).

Unlike the control of hydrogen sulfide with limestone, the control of sulfur dioxide is not thermodynamically limited in a pressurized fluidized-bed combustor operating at 10 atmosphere pressure at temperatures below 1050°C (Newby et al., 1989). Assuming that the PFBC has a limestone bed, a GBF with limestone medium would be used as a polishing sulfur dioxide absorber and as a particulate filter. In such an application, the limestone/clay medium would circulate through the GBF many times since the inlet concentration of sulfur dioxide would be low. The medium would have to have a high attrition resistance to be able to circulate many times through the filter.

Clay for Alkali Control

Several investigators have reported successful alkali removal from high temperature gas streams with sorbents of activated bauxite, attapulgus clay, calcium montmorillonite clay, diatomaceous earth, kaolin clay, and emathlite clay.

Emathlite, a type of fullers earth, was found to be a leading getter of alkali (Bachovchin et al., 1986). The clay had a high capacity for sodium and binds the sodium irreversibly. At extreme conversions, the clay was found to become sticky. This could be a problem but is unlikely to occur as these extreme conversions are not realistically obtained and the anticipated fraction of clay within the agglomerated pellet is small. Kaolin, bauxite, and emathlite were all found to be capable of removing alkali from coal conversion streams (Uberoi et al., 1990). Kaolin and emathlite sorption of alkali was an irreversible process. The maximum sorption capacity of the kaolin was about 25% while that of bauxite and emathlite was about 15%. During screening of alkali sorbents, calcium montmorillonite clay was found to be superior and was chosen for further investigation (McLaughlin, 1990).

All of these sorbents are capable of alkali removal but only a few can be considered within the scope of this program. Of these alkali sorbents tested, bauxite was reported to be fractionally irreversible with alkali removal being 10% chemical sorption and 90% physisorption (physically absorbed as water soluble alkali) (Lee and Johnson, 1980) while only kaolin and emathlite were reported to react irreversibly with alkali (Bachovchin et al., 1986; Uberoi et al., 1990). Because bauxite is also a relatively expensive sorbent which would not be suitable as a non-regenerable sorbent. Work on limestone agglomeration found attapulgus clay to be an effective binding agent (Voss, 1983). With these points in mind, an alkali sorbent of kaolin, attapulgus or emathlite clay in conjunction with limestone will be investigated.

Trace Metal Control

Work at the University of Arizona indicates the potential of porous solids such as bauxite, kaolin or activated alumina for the absorption of heavy metals such as lead or cadmium (Uberoi and Shadman, 1991a). Results have shown that bauxite was considerably more effective than kaolinite for the sorption of cadmium vapors (Uberoi and Shadman, 1991b). The lower effectiveness of kaolinite was explained during examination of the particle which showed an almost completely reacted surface with an unreacted interior. This surface reaction is also typical of dense, natural limestones reacting with sulfur. Therefore, the use of kaolinite in an agglomerated pellet with large micro-pores may allow higher utilization just as it does for the sorption of sulfur species by limestone agglomerates. The kaolinite had a lower water soluble fraction of sorbed cadmium than the bauxite which is desirable from the point of view of ultimate disposal. Also studied was the sorption of lead vapors by various sorbents and kaolinite was found to be the most effective (Uberoi, 1990).

Limestone and dolomite were found to be effective for the removal of zinc and lead vapors from simulated flue gases (Mojtahedi et al., 1989). Dolomite, with its more open pore structure, removed 82% of the lead vapors and 19% of the zinc vapors. Limestone removed 41% of the lead vapors and 81% of the zinc vapors. The sorption of lead vapors may possibly be improved by the agglomeration of limestone to give a more open pore structure similar to dolomite.

From the work of these investigators, a high temperature sorbent composed of kaolinite, bauxite, limestone or dolomite could be capable of heavy metals removal from coal process streams. These same materials have proven to be effective for the capture of alkalies and sulfur species.

Filter Media Preparation, Characterization and Evaluation

In order to evaluate the concept of a GBF multi-contaminant control filter medium, a bench scale laboratory program is planned to be followed by testing in a pilot scale GBF. The bench-scale laboratory program consists of filter media preparation, physical characterization and evaluation of chemical reactivity.

The filter media will be prepared from pulverized limestone, either 80% minus 150 microns or 80% minus 44 microns, an alkali absorbing clay and suitable binders. A disc pelletizer will be used to prepare 6 mm and 2 mm spherical pellets. Major emphasis will be on evaluation of 6 mm media. The 2 mm media will be evaluated only if the 6 mm media does not have suitable properties. Should disk pelletizing not produce a suitable filter medium, extruded pellets which are rounded into spherical shapes with a disc pelletizer would then be evaluated.

The clay to be used in the filter medium as an alkali sorbent will be determined on the basis of a screening test developed by McLaughlin (McLaughlin, 1990). The candidate clays to be evaluated are: kaolin clay, bauxitic kaolin clay, ematholite (calcium montmorillonite clay), attapulgite clay and low swelling bentonite clay. A mixture of clay and NaCl is heated in a thermogravimetric analyzer (TGA). The mixture with the lowest weight loss would be the best alkali sorbent. Mixtures of limestone, clay and NaCl will also be evaluated. The preferred clay will be mixed with limestone at 5% weight concentration to form the multi-contaminant control filter medium.

In order to make a strong, attrition resistant filter medium from the clay and limestone mixture, a binder may be needed. The following lists the binders which will be evaluated and their weight concentration.

- 15% attapulgus clay with 2% boric acid
- 2% and 4% sodium silicate
- 2% and 4% corn starch
- 2% sodium bentonite with 10% calcium sulfate
- 2% and 5% sodium bentonite
- 2% and 5% calcium montmorillonite
- 5% and 10% calcium sulfate hemihydrate
- 10% and 15% portland cement
- 10% and 15% FBC bed ash

The filter media prepared from the above combinations will be evaluated in terms of their physical properties. Pellet green strength is evaluated by a drop test which relates to the ability of the pellet to be handled before curing. After curing, calcined pellets are subjected to a crush test which relates to the ability of the pellets to maintain shape in the GBF. Pellet attrition resistance is measured using ASTM D4164-88 procedure. The attrition resistance of the most promising formulation will be evaluated in a special test apparatus which simulates the particle motion in a GBF. The apparatus consists of a lift pipe and return seal leg.

For the five most promising formulations more extensive physical characterizations will be performed. These include bulk density measurements, measurement of pore volume and pore diameter, determination of optimum pellet moisture for maximum strength and SEM microstructure of reacted pellets.

Media reactivity will be evaluated in sulfidation, sulfation and alkali absorption tests. Initial TGA sulfidation tests will be run on 15 formulations of 6 mm pellets and 3 sample of 2 mm pellets. The five best formulations will then be sulfided in a larger batch apparatus so that the reacted medium can be evaluated for crush strength and ASTM attrition resistance. For the most promising formulation, additional TGA will be run to obtain detailed kinetic data. In these tests, gas composition and temperature will be varied. A series of pressurized TGA tests will be run to access the effects of pressure on reaction kinetics. A larger scale fixed bed reactor will be used to sulfide filter medium for large scale attrition tests and to obtain kinetic data for comparison with the TGA data. This test series will evaluate the medium's reactivity with respect to the sorption of H_2S.

Prior to disposal, sulfided filter medium will have to be converted to the sulfate form. It is expected that the larger micro-pores of the pelletized limestone will facilitate the conversion of sulfide to sulfate as occurs with dolomite. Atmospheric TGA tests will be used to determine the kinetics of the oxidation of the sulfide filter medium.

The multi-contaminant control GBF could be used with combustion gases to remove particulate, sulfur dioxide, and alkali compounds. The five best formulations determined from the sulfidation tests will be evaluated for SO_2 removal. TGA tests will evaluate the kinetics of the sulfation reaction. Batch sulfation tests will generate sufficient quantities of reacted filter medium for crush strength tests and ASTM attrition tests.

Fixed-bed alkali absorption tests will be used to obtain kinetic data on the reactivity of the prepared sorbents with respect to the absorption of alkali and sulfur compounds. A heated sample holder whose change in weight is monitored with a micro balance heats salt crystals which vaporize into a carrier stream. The rate of evaporation is controlled by the temperature of the sample holder. The alkali vapors are mixed with either a gas stream containing H_2S or one containing SO_2 and are carried into the packed bed of sorbent. The packed bed section will be 3 inches in diameter by 12 inches long, in a tube of alumina which is inert with respect to the alkali vapors. Test durations are between 24 and 100 hours. The first tests use a carrier gas containing N_2, 5% H_2O, and additives of H_2S and HCl. In later tests, the carrier gas contains 95% CO_2 and 5% H_2O with additives of SO_2 and HCl. The first five tests use a shallow bed which will be analyzed for the average alkali and sulfur sorption. After the first five tests, the bed will be sectioned into ninths and analyzed for alkali and sulfur content as a function of the position in the bed. In some of the tests, the carrier gas will contain HCl which is know to inhibit the sorption of alkali.

The kinetic data collected on sorbent reactivity will be used to create a model of the sorbent's chemical reactivity in a GBF. The model will take into account the sorbent reactivity with respect to sulfur species and alkali. The model will provide information on the required bed depth of the GBF, the expected outlet concentration of sulfur and alkali species and the extent of reaction of the sorbent.

Pilot Plant Testing

Bench scale tests are used to develop and evaluate multi-contaminant control media. The developed media is evaluated in terms of its chemical and physical properties in a bench scale environment. The next phase of evaluation will be at the pilot plant scale. Combustion Power Co. is participating in the DOE sponsored tests at the Power Systems Development Facility (PSDF) to be installed in a Southern Company Services Facility in Wilsonville, AL. The first year of testing at the PSDF will be dedicated to the evaluation of the GBF for particulate control. For these tests, the GBF is connected to M.W. Kellogg's transport reactor which can be operated in either a gasification or combustion mode. After the evaluation of a GBF for particulate control, the opportunity exists to evaluate the GBF with a reactive medium for the control of sulfur and alkali compounds.

The modification necessary to allow testing at the PSDF GBF are relatively straight forward. Lock hoppers will be installed for the addition and removal of multi-contaminant control filter medium. The additional feed and removal systems can be accommodated within the existing structure. Nine tons of clay/limestone filter medium will be prepared for the test series which will provide for 80 hours of testing.

Cost Estimate for Production of GBF Multi-Contaminant Filter Medium

To evaluate sorbent feasibility, plant scale processes were developed to estimate the capital and operating costs to produce a GBF medium of limestone and clay at three different production rates. The production rates chosen were 5,000, 50,000 and 500,000 ton/yr.

To develop the plant processes and cost estimates for the three production rates, assumptions were made concerning the medium composition, i.e. the sorbent formulation. Of the 68 possible formulations to be evaluated, a single formulation was chosen as the base case. This formulation is 6 mm pellets of 80% less than 149 micron limestone with 5% montmorillonite clay and 10% dry binder addition of portland cement (PC III). The pellets would be formed by disc pelletization requiring 20% pellet moisture and moist environment curing for 72 hours. Cost estimates were also made for lower and upper limits based on different formulations and pelletizing procedures.

Table 1 shows the cost estimates; for the base case, the production cost for 50,000 tons/year would be $39.72 per ton.

FUTURE WORK

The first task in the GBF multi-contaminant development program of identifying a concept and a test plan is completed. The next phase will be the execution of bench scale tests to determine the feasibility of the proposed approach to multi-contaminant control using a GBF. Following bench scale testing, testing will be conducted on a pilot scale GBF at the PSDF. After completion of testing, the cost and design of a commercial scale GBF with multi-contaminant control will be determined.

Table 1 Cost Estimate for GBF Medium Production

Production Rate ton/yr	Capital Cost M$		Operating Cost $/ton	
	Base	Range	Base	Range
5,000	2.5	2.1 - 2.8	152.0	137.8 - 200.3
50,000	4.3	3.6 - 5.1	39.7	31.1 - 86.0
500,000	15.2	12.3 -17.0	26.9	190.2- 71.9

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