

# RESOURCE RECOVERY FROM WASTEWATER TREATMENT SLUDGE CONTAINING GYPSUM

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## INTRODUCTION

The disposal of wastewater treatment sludge generated at the Radford Army Ammunition Plant (RAAP) is a serious problem. The sludge is produced by neutralizing spent acid contained in the wastewater with lime, and consists principally of very finely divided wet gypsum (calcium sulfate dihydrate). Although the sludge is presently being disposed of in a landfill, the sludge is difficult to handle and convert into a load-bearing material. Therefore an alternative method of disposal is being developed and evaluated.

The alternative method involves drying and granulating the sludge, followed by high temperature calcination in a fluidized bed reactor to recover usable sulfur dioxide and lime. If the method is adopted, these products would be used within the plant with considerable cost savings. The sulfur dioxide would be added to the feed stream of an oleum manufacturing facility and the lime would be re-used in wastewater treatment.

The process under development is similar to one which was demonstrated recently for utilizing phosphogypsum, a waste product of the phosphoric acid manufacturing industry [1, 2]. Like the RAAP wastewater treatment sludge, phosphogypsum consists principally of wet, microscopic particles of gypsum. However, the two materials differ in impurity content with the phosphogypsum containing significant amounts of phosphate, fluoride, and silica, and the RAAP sludge containing small amounts of organic compounds. Since this difference in impurity content could affect subsequent processing, it was not known whether the same process conditions would apply to both

materials. Also, additional development and demonstration of the process was needed in any case for a more accurate evaluation of the process.

A preliminary investigation showed that the particles of dried wastewater treatment sludge were too small for economical processing in a conventional fluidized bed reactor because they would be blown out of the reactor at any reasonable gas velocity. Therefore the material would have to undergo a size enlargement step. Subsequent testing showed that the dried sludge could be briquetted without a binder by application of pressure alone. The briquetted material could then be crushed and the particles screened to provide a suitable feed for a fluidized bed reactor. Small samples of feed prepared in this way were subsequently calcined in a laboratory furnace under a controlled atmosphere. A series of experiments were conducted in which reaction time, temperature, gas composition, and particle size were varied. The results indicated that a high conversion of calcium sulfate to calcium oxide and sulfur dioxide could be achieved in a reasonable time by proper choice of conditions. Therefore a program was undertaken for demonstrating the process in bench-scale and larger equipment. The principal results of this demonstration are described below. This effort has been sponsored by the U.S. Army Research and Development center with funding provided by the U.S. Army Hazardous and Toxic Materials Agency.

## PROCESS DESCRIPTION

The proposed processing scheme for treating wastewater treatment sludge is illustrated by Fig. 1. The wet

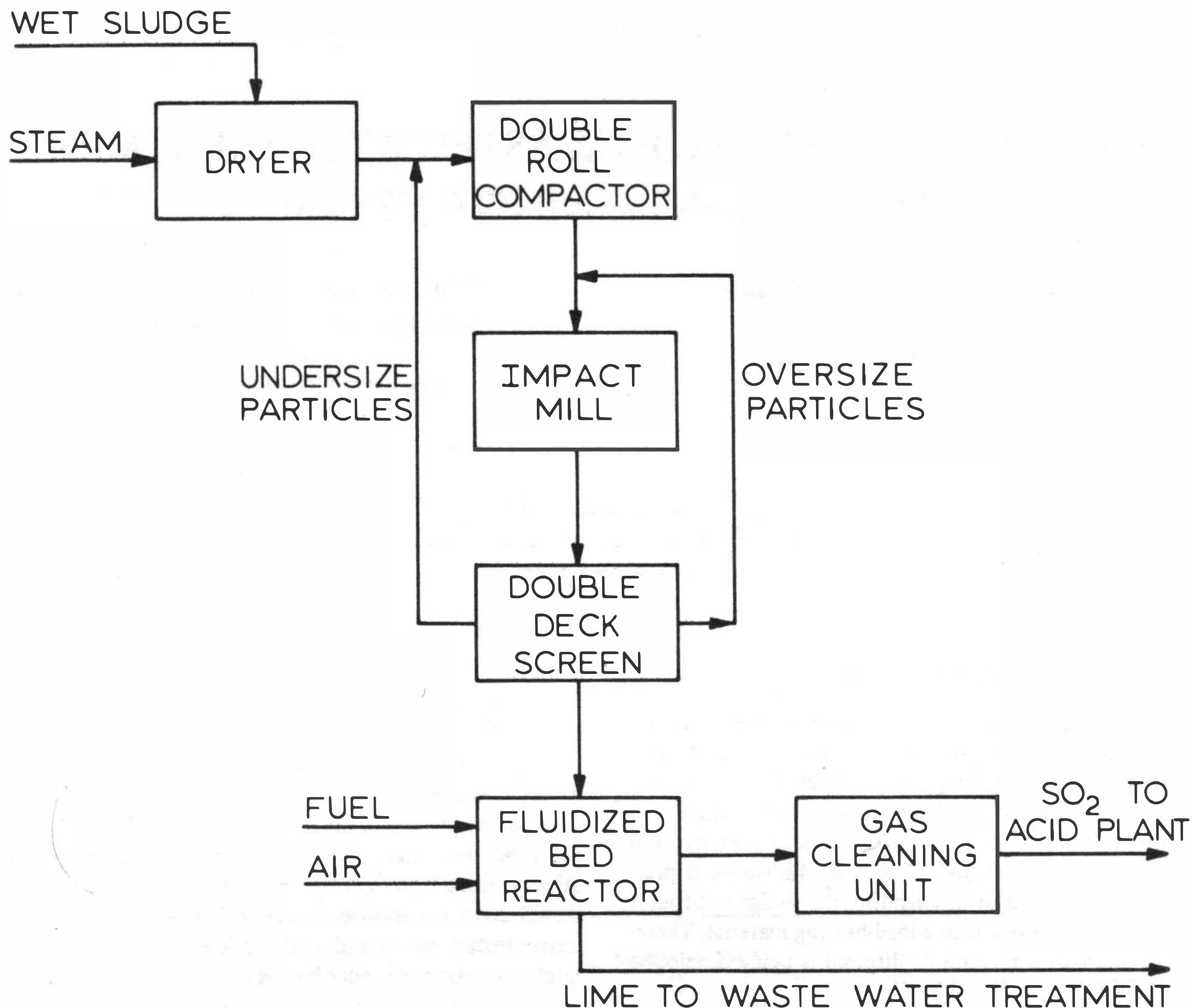
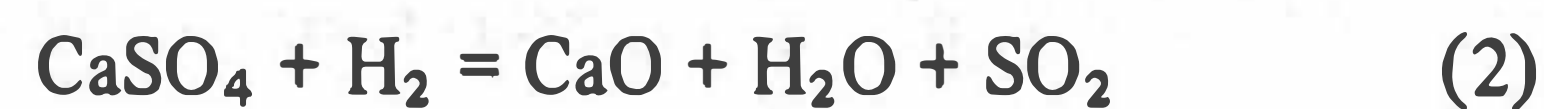


FIG. 1 PROCESS FOR UTILIZING WASTEWATER TREATMENT SLUDGE

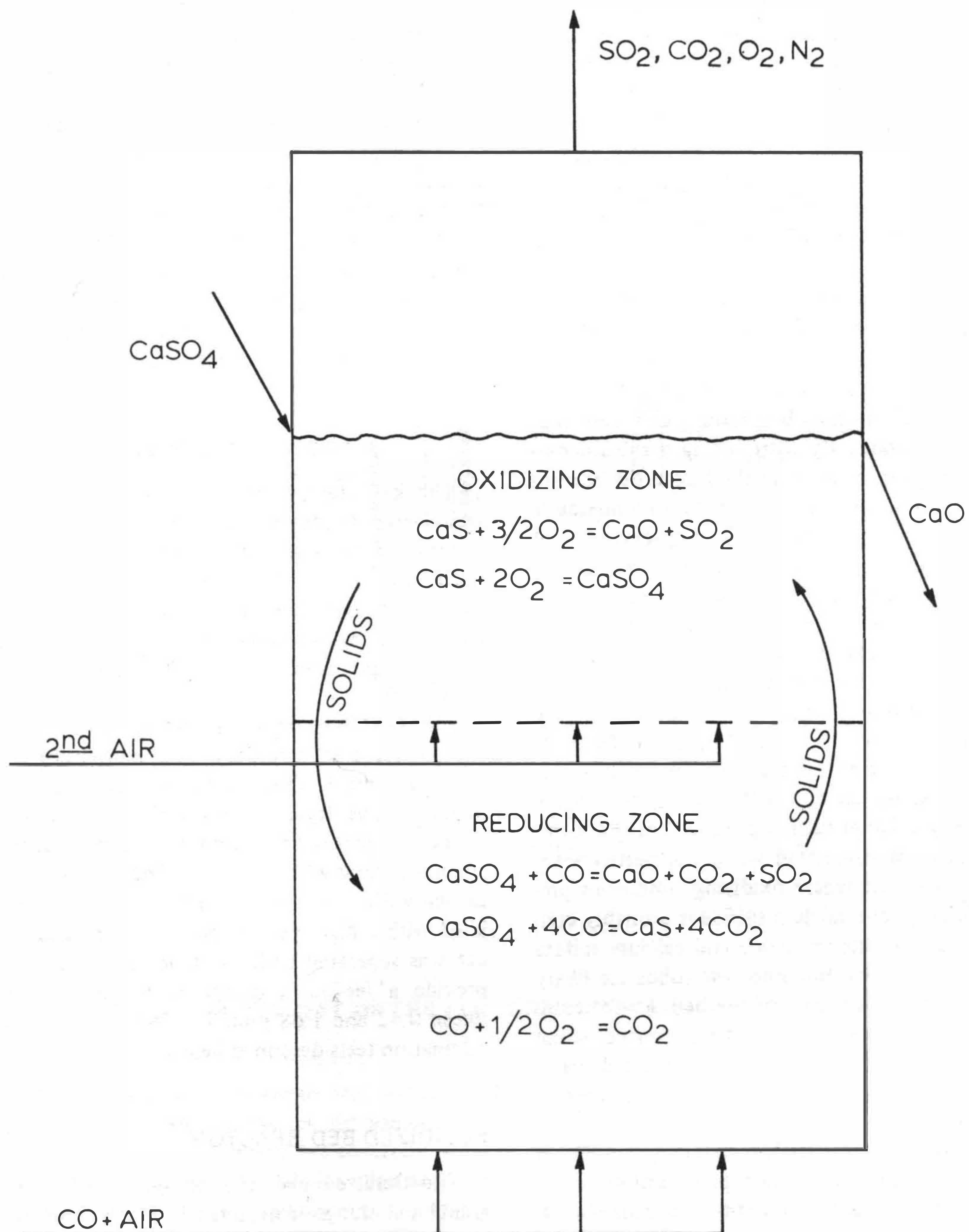
sludge would first be dried to remove surface moisture and a major portion of the water of crystallization. Dried sludge would be needed for compacting or briquetting the feed. Also, any moisture in the feed for the fluidized bed reactor would result in a greater heat load for the calcination step and a more dilute product gas. The dried powder would be briquetted with a double-roll compactor capable of applying high pressures and producing either briquettes or a solid ribbon of material. This material would be crushed with a hammer mill or impact mill and the resulting particles would be separated by a double-deck, vibrating screen to recover particles within the proper size range for the fluidized bed reactor. The oversize particles would be recycled through the impact

mill and the undersize particles would be returned to the compactor to be reprocessed.

The feed prepared in the above manner would be calcined continuously in a special two-zone fluidized bed reactor supplied with a hydrocarbon fuel and air. Although natural gas is presently the fuel of choice, other fuels such as powdered coal might be used. A substoichiometric amount of air would be supplied in order to generate carbon monoxide and hydrogen as well as heat through partial combustion of the fuel. These gases would react subsequently with the calcium sulfate as follows:







**FIG. 2 TWO-ZONE FLUIDIZED BED REACTOR CONCEPT**

Although these reactions are endothermic, sufficient heat would be released by partial combustion of fuel to supply the reaction needs. The sulfur dioxide produced by the preceding reactions as well as various combustion products would be carried away in the reactor off-gas. The off-gas would be cooled, cleaned, and dried before utilizing it in an oleum manufacturing facility. Calcined solids consisting chiefly of calcium oxide would be withdrawn from the reactor and cooled before being utilized for wastewater treatment.

Previous work at Iowa State University (ISU) has indicated that it would be advantageous to use a special two-zone fluidized bed reactor for the above process [3, 4]. The concept of a two-zone reactor is illustrated by Fig. 2. For purposes of illustration carbon monoxide is shown as both fuel and reductant. By introducing a substoichiometric amount of primary air with the fuel in the bottom of the reactor, only a portion of the carbon monoxide is burned to supply the necessary heat so the remainder of the carbon monoxide is available to react with calcium sulfate. Thus a reducing zone is created in the bottom of the reactor which causes the indicated reactions to take place. These reactions convert calcium sulfate to a mixture of calcium oxide and calcium sulfide. The latter is an undesirable by-product which can be eliminated by creating an oxidizing zone in the upper part of the fluidized bed by introducing excess secondary air in this part of the bed. Because the solids circulate freely within the fluidized bed, any calcium sulfide produced in the lower part of the reactor is eliminated when the particles are carried to the upper part where oxidizing conditions prevail. In the oxidizing zone calcium sulfide is probably converted to a mixture of calcium oxide and calcium sulfate by the indicated reactions. But since the solids are likely to be returned to the lower part of the bed, any calcium sulfate formed in the upper part will be converted in the lower part. Individual particles may be subjected many times to an oxidation-reduction cycle while passing through the reactor.

With a two-zone reactor, highly reducing conditions can be maintained which promote rapid reaction of the calcium sulfate. While such conditions also cause undesirable side reactions, the problem is overcome by providing a means for reoxidizing any calcium sulfide formed.

A detailed analysis of the preceding reaction scheme has shown the importance of efficient heat recovery [5]. The results of material and energy balance calculations are shown in Table 1 for two cases involving complete conversion of calcium sulfate to calcium oxide at 1175°C using methane as a fuel. For these calculations it was assumed that pure anhydrous calcium sulfate, fuel, and air would be fed continuously to a well-mixed, adiabatic reactor operating at steady-state and that all the prod-

ucts leaving the reactor would be in chemical equilibrium.

In the first case, none of the sensible heat of the products would be utilized for preheating the reactants and the reactants would be supplied at ambient temperature to the reactor. Consequently fuel and air requirements would be a maximum and sulfur dioxide concentration in the off-gas a minimum. In the second case, as much sensible heat as possible would be recovered from the reaction products and used to preheat the calcium sulfate and air supplied to the reactor in the most efficient way possible. For this case the fuel requirement would be only 43 percent of that for the first case while the sulfur dioxide concentration would be 2.3 times as great.

## PROCESS DEMONSTRATION

The key steps in the proposed process for utilizing wastewater treatment sludge were demonstrated with bench-scale or larger equipment. Wet sludge produced at the Radford Army Ammunition Plant was sent to various equipment manufacturers for drying, briquetting, crushing, and sizing. The prepared feed was then calcined in a two-zone fluidized bed reactor located at Iowa State University.

The feed preparation steps indicated in Fig. 1 were demonstrated satisfactorily using conventional industrial-type equipment. The wet sludge was dried with a screw-conveyer-type dryer which was fitted with a steam-heated cylindrical jacket and swept by a stream of warm air in cocurrent flow with the solids. The dried sludge was compacted with a double-roll press and then crushed or granulated with a high-speed impact mill. The granulated product was separated with a double-deck vibrating screen to provide a feedstock composed of particles ranging between 0.42 and 1.68 mm. The feedstock was used in the calcination tests described below.

## FLUIDIZED BED REACTOR

The fluidized bed reaction system used for calcining granulated sludge is depicted in Fig. 3. The reactor itself had an inside diameter of 15 cm and overall height of 2.1 m. The tubular body of the reactor was made of silicon carbide. The tube was fitted with a ceramic gas distributor and refractory-lined cap. A mixture of primary air and natural gas was admitted through four cone-shaped openings which were cast into the distributor. Feed particles were introduced with secondary air through an axial dip tube which was suspended from the top of the reactor. The reactor was also fitted with a protected thermocouple for measuring the temperature of the fluidized bed and with an overflow tube for removing reacted



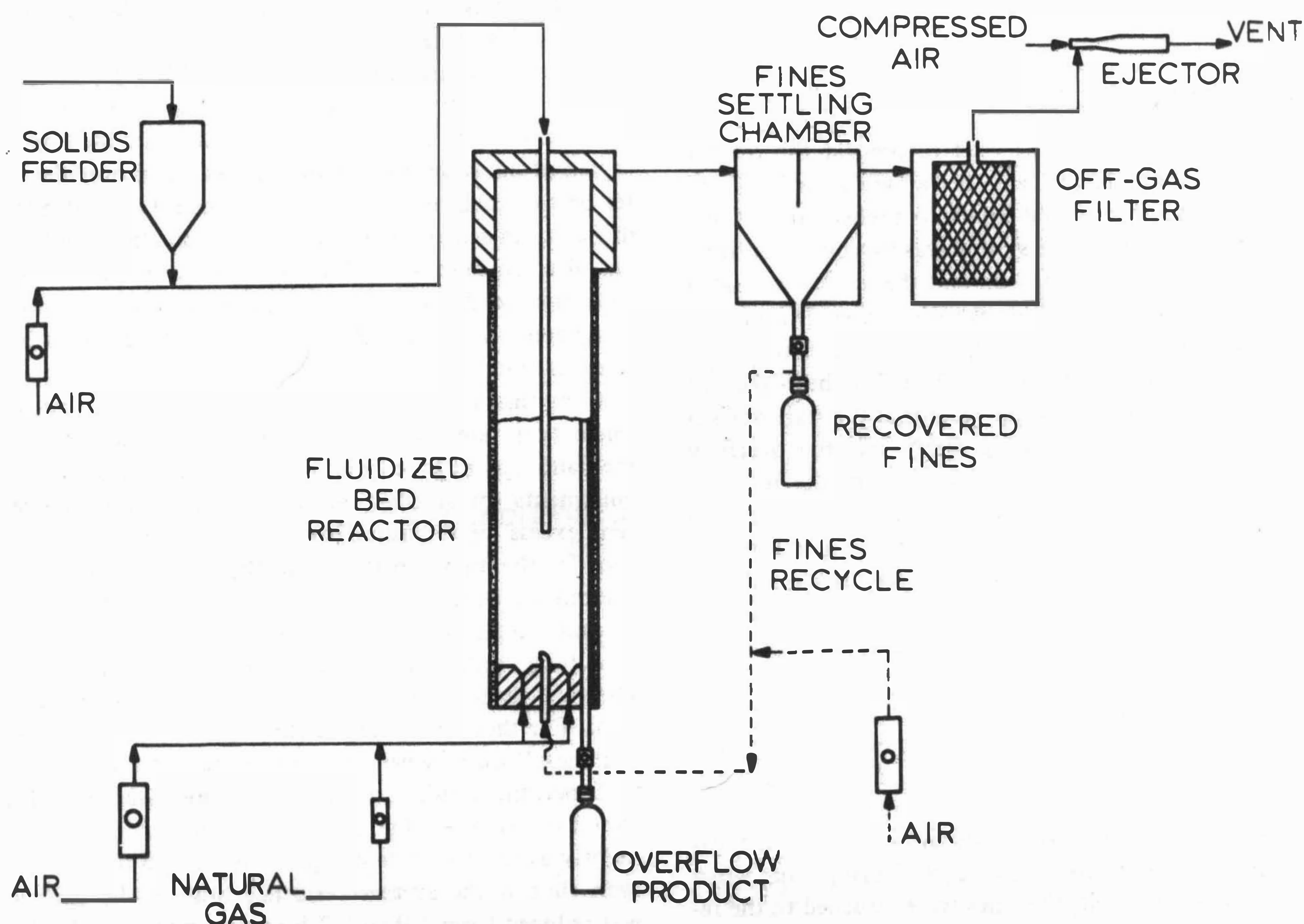


FIG. 3 FLOWSHEET OF THE FLUIDIZED BED REACTION SYSTEM

solids which were collected in a canister that was emptied periodically. The main body of the reactor was surrounded by an electric furnace for temperature control.

A solids feeder supplied granulated sludge at a controlled rate and the material was conveyed pneumatically into the reactor. Air and natural gas flows were indicated by calibrated rotameters and controlled manually. The reactor off-gas was conducted through a large settling chamber to remove the bulk of the fines entrained in the reactor. The gas then passed through a fiber glass filter to remove most of the remaining dust before venting to the atmosphere. The entire off-gas handling system was operated at slightly subatmospheric pressure to prevent escape of toxic gases into the laboratory where the reactor was located. The reduced pressure was created by an ejector mounted at the end of the vent pipe and ac-

tuated by compressed air.

During some runs the fines collected in the settling chamber were returned to the reactor, but during other runs the fines were merely recovered and analyzed. Whenever the fines were recycled, they were added to a portion of the primary air and introduced through an axial quartz tube in the bottom of the reactor. The upper end of this tube was bent to discharge the stream horizontally and prevent the development of a vertical spout in the bed.

Samples of the reactor off-gas were withdrawn from the top of the reactor, filtered, and analyzed with either a gas chromatograph or a standard Orsat apparatus. The chromatograph was capable of resolving the gas into four peaks: (1) nitrogen, (2) carbon dioxide, (3) water vapor, and (4) sulfur dioxide. However, the nitrogen peak also included argon and oxygen or carbon monoxide if pres-



ent. The Orsat apparatus provided an indication of the relative concentrations of acidic gases, oxygen, and carbon monoxide.

## CALCINATION PROCEDURE

The electric furnace was used to preheat the reactor overnight to about 1000°C and then air was passed through the hot reactor for several hours to preheat the off-gas handling system. When the system was thoroughly heated, the solids feeder was turned on and the reactor was filled gradually to the top of the overflow tube. During this operation, sufficient air was supplied to fluidize the solids in the reactor. Generally solids which had been reacted previously were used to fill the reactor at the start of each run. After solids were passing freely out the overflow tube, the flow of natural gas was started and the bed temperature rose rapidly. The feed was changed to fresh, unreacted material and the desired operating conditions were soon established. Once the desired flow rates and bed temperature were established, operating conditions were kept constant for several hours to allow the system to approach steady-state.

During the operation, the canisters which received solids issuing from the overflow tube and from the settling chamber were emptied periodically and the solids were weighed and sampled for analysis. The reactor off-gas was sampled intermittently and analyzed with both the gas chromatograph and Orsat apparatus. During runs when the fines were recycled, the fines were returned to the reactor at essentially the same rate as they were collected in the settling chamber.

After the operation was completed, the unit was shut down and allowed to cool to ambient temperature. The reactor was emptied and the system disassembled, cleaned, and prepared for the next run.

The percent desulfurization of the reacted particles was calculated by means of the following expression:

$$\text{Desulf.(\%)} = 100 - 100 \left[ \frac{\% \text{Ca}}{\% \text{SO}_3} \right]_{\text{Feed}} \left[ \frac{\% \text{SO}_3 + 2.5(\% \text{S}^-)}{\% \text{Ca}} \right]_{\text{Product}}$$

## EXPERIMENTAL RESULTS

A number of successful runs were made to demonstrate the calcination of granulated sludge in the two-zone fluidized bed reactor and to study the effects of different operating conditions on the results. Stable, continuous operation of the reactor was achieved in most cases and only a few operating problems were encountered. The ranges of operating conditions employed and results obtained are indicated in Table 2 for runs made both with and without

fines recycling. Also specific operating conditions and detailed results are presented in Table 3 for several typical runs. The runs are listed in order of increasing feed rate with the first three runs representing operation without fines recycling and the fourth run (H-17) representing operation with fines recycling. Most runs were continued for 6 h or more and the results listed in Tables 2 and 3 represent an average of measurements taken over the latter part of each run when conditions had stabilized. Some difficulty in achieving steady operating conditions was observed when fines were recycled because the rate of fines collection tended to vary. Conditions were not stabilized completely when run H-17 was discontinued after 5 h of operation.

Operating results were generally satisfactory when a calcination temperature of 1150°C was employed and fuel and air rates corresponded to the theoretical requirements for an ideal adiabatic reaction plus 5-10 percent excess air to insure having a pronounced oxidizing zone in the upper part of the fluidized bed. With this amount of excess air, the off-gas usually contained 1-2 percent oxygen and the solids which passed out the overflow tube contained a negligible amount of sulfide ( $\text{S}^-$ ). The runs listed in Table 3 were made under such conditions. For these runs the overflow product was 96-97 percent desulfurized when the fines were not recycled and 91.5 percent desulfurized when the fines were recycled. Desulfurization of the overflow product declined only slightly as the feed rate was raised from 5.58 to 10.4 kg/h even though the average residence time of the particles was reduced from 1.9 to 0.7 h as determined by dividing the total mass of calcium in the bed by the calcium feed rate. On the other hand, the rate of particle entrainment in the off-gas increased greatly as the operating rate increased and consequently a smaller percentage of the solids was recovered in the overflow product. A calcium balance showed that 91 percent of the calcium fed was recovered in the overflow product at the lowest feed rate but only 66 percent at the highest feed rate when none of the fines were recycled. Not all of the calcium was accounted for because some particles were too small to be captured by the settling chamber.

For the first three runs in Table 3, desulfurization of the fines recovered in the settling chamber was 88-89 percent which was significantly less than that of the overflow product. Because the entrained particles were not highly desulfurized, several runs were made in which the fines recovered in the settling chamber were recycled. The results of run H-17 were fairly typical. Recycling the fines increased the solids overflow rate from the reactor noticeably but somewhat reduced the desulfurization of the overflow product as can be seen by comparing the results of run H-17 with those of run H-2 made at similar

**TABLE 1 MATERIAL AND ENERGY BALANCE REQUIREMENTS FOR TWO IDEAL CASES INVOLVING COMPLETE CONVERSION OF CALCIUM SULFATE TO CALCIUM OXIDE AT 1175°C**

Case	$\frac{m \text{ CH}_4}{m \text{ CaSO}_4}$	$\frac{m \text{ Air}}{m \text{ CaSO}_4}$	SO <sub>2</sub> conc., %	
			wet	dry
No heat recovery	1.50	11.9	7.2	9.1
Max. heat recovery	0.65	3.8	16.6	21.0

**TABLE 2 RANGE OF OPERATING CONDITIONS AND RESULTS FOR THE RUNS MADE WITH FLUIDIZED BED REACTOR**

Parameter	Range	
	Without recycle	With recycle
Feed rate, kg/h	7.1 - 13.2	10.9 - 13.1
Feed moisture content, %	4.7 - 21.4	3.5 - 19.0
Nat. gas rate, m <sup>3</sup> /h	1.39 - 2.77	2.75 - 2.77
Total air rate, m <sup>3</sup> /h	11.6 - 23.1	13.5 - 13.6
Mole CH <sub>4</sub> /mole CaSO <sub>4</sub>	1.3 - 1.7	1.5 - 1.6
Mole air/mole CaSO <sub>4</sub>	10.8 - 14.3	12.2 - 12.9
Max. superficial velocity, m/s	0.95 - 1.90	1.82 - 1.89
Bed temperature, °C	1100 - 1150	1090 - 1150
Estimated SO <sub>2</sub> conc., %	4.2 - 6.2	3.9 - 6.0
Solids overflow rate, kg/h	2.28 - 3.77	4.01 - 5.21
Fines collection rate, kg/h	0.10 - 1.53	- - - - -
Fines recycle rate, kg/h	- - - - -	2.40 - 7.37
Particle residence time, h	0.68 - 1.89	0.74 - 0.91
Desulf. of overflow solids, %	73.5 - 98.6	74.0 - 95.0
Desulf. of collected fines, %	70.0 - 91.7	- - - - -



**TABLE 3 SPECIFIC OPERATING CONDITIONS AND RESULTS FOR TYPICAL RUNS WITH FLUIDIZED BED REACTOR**

Parameter	Run H-3	Run H-1	Run H-2	Run H-17 <sup>a</sup>
Feed rate (dry basis), kg/h	5.58	7.08	10.4	11.1
Feed composition				
Sulfate as SO <sub>3</sub> , %	44.9	44.9	44.9	52.8
Calcium as Ca, %	23.3	23.3	23.3	26.3
Moisture as H <sub>2</sub> O, %	21.4	21.4	21.4	3.5
Nat. gas rate, m <sup>3</sup> /h <sup>b</sup>	1.39	1.90	2.77	2.77
Total air rate, m <sup>3</sup> /h <sup>b</sup>	11.55	15.87	23.13	23.01
Mole CH <sub>4</sub> /mole CaSO <sub>4</sub>	1.44	1.55	1.55	1.52
Mole air/mole CaSO <sub>4</sub>	12.0	12.9	12.9	12.7
Max. superficial vel., m/s	0.95	1.30	1.90	1.89
Bed temperature, °C	1150	1150	1150	1150
Estimated SO <sub>2</sub> conc., %	5.6	5.0	5.2	6.0
Solids overflow rate, kg/h	2.28	2.77	3.16	4.48
Fines collection rate, kg/h	0.10	0.26	1.53	- - -
Fines recycle rate, kg/h	- - -	- - -	- - -	7.37
Particle residence time, h	1.9	1.3	0.7	0.9
Overflow solids composition:				
Sulfate as SO <sub>3</sub> , %	3.3	3.9	5.6	9.9
Sulfide as S, %	0	0	0	0.2
Calcium as Ca, %	66.0	62.3	64.4	61.5
Desulfurization, %	97.4	96.8	95.5	91.5
Fines composition:				
Sulfate as SO <sub>3</sub> , %	13.2	13.1	11.5	7.4
Sulfide as S, %	0.4	0.1	0.5	0
Calcium as Ca, %	59.0	58.7	60.9	62.0
Desulfurization, %	87.5	88.2	89.1	94.0
Ca recovered in overflow, %	91.1	82.1	65.9	92.2
Ca in collected fines, %	3.4	7.4	30.3	- - -
Total Ca accounted for, %	94.5	89.5	96.2	92.2

<sup>a</sup> Run made with fines recycle.

<sup>b</sup> Measured at 22°C and 1 atm.



TABLE 4 SIZE DISTRIBUTION AND DESULFURIZATION DATA FOR RUN H-1

Size, mm	Feed, wt %	Final Bed		Overflow Product		Entrained Fines	
		wt %	Des %	wt %	Des %	wt %	Des %
-1.68/+1.19	26	18	94.7	16	94.1		
-1.19/+0.84	40	44	97.5	44	97.1		
-0.84/+0.59	28	27	98.4	27	97.2	5	97.9
-0.59/+0.42	6	10	97.8	13	97.3	50	96.2
-0.42/+0.30		1	88.4			11	91.6
-0.30/+0.15						11	72.6
-0.15/+0.074						9	54.4
-0.074						14	40.4

feed rates. By recycling the fines, 92 percent of the calcium was recovered in the overflow product compared to 66 percent without recycling. Interestingly, a sample of the fines being recycled was collected at the end of run H-17 and it had the composition shown in Table 3 corresponding to 94 percent desulfurization. Therefore, recycling did improve the conversion of the fines.

Further insight was gained by separating samples of the final bed material, overflow product, and recovered fines into various size fractions by screening and then chemically analyzing these fractions. Results such as those for run H-1 listed in Table 4 showed that significant decrepitation of the particles occurred during calcination because the products were more heavily weighted with the finer sizes and less heavily weighted with the largest size than the feed. Also the data indicated that the fluidized bed was well mixed because the size distribution and chemical composition of the various sizes of the overflow product were similar to those of the bed. Furthermore it was apparent that desulfurization of the entrained particles decreased as particle size decreased which indicated that the finer particles were elutriated from the bed before being completely reacted.

For the runs discussed above, the sulfur dioxide content of the off-gas ranged between 5 and 6 percent. These values were estimated by material balance and verified by measurement with the gas chromatograph. The values

were not as high as the theoretical value of 7.2 percent for an ideal system because the feed was not completely dehydrated nor the calcium sulfate completely desulfurized.

When the granulated sludge was calcined at 1100°C using conditions which were otherwise similar to those listed in Table 3, the overflow product was not desulfurized as well as when it was calcined at 1150°C. At the higher temperature, increasing the fuel and air rates slightly for a given feed rate increased desulfurization of the solids to over 98 percent whereas decreasing the fuel and air rates reduced desulfurization.

Most of the problems encountered with the fluidized bed reactor were related to the relatively large rate of particle entrainment in the off-gas at high operating rates. The entrainment rate was large because of particle decrepitation, a low freeboard (90 cm) above the fluidized bed, and a high ratio of bed depth to diameter which was conducive to slugging and rough fluidization. Also the feed entered the reactor in a succession of small slugs, and since the feed contained water of crystallization, the sudden release of water vapor as each slug entered the hot bed produced a succession of pressure surges which also contributed to rough fluidization.

While much of the entrained material was recovered in the settling chamber, a significant amount escaped. Therefore only a portion of the material could be recycled. Some difficulty was experienced in controlling the recycle



rate because the equipment installed for this purpose was relatively crude.

Some of the entrained material accumulated inside the off-gas piping and formed scale which eventually would have plugged the piping. Two types of scale were observed: a hard scale which formed at high temperature in the piping between the reactor and settling chamber and a soft scale which formed at lower temperature between the settling chamber and gas filter.

## FUTURE DEMONSTRATION

Because of the encouraging results achieved with the bench-scale fluidized bed reactor, preparations have been made to demonstrate the calcination of granulated sludge in a larger reactor which will have an inside diameter of 25.4 cm and overall height of 3 m. This reactor will have about three times the capacity of the smaller reactor used to date and, therefore, will be capable of processing up to 1000 kg/day of sludge. Although the design of the larger reactor is similar to that of the smaller one, it will have a larger freeboard and will be operated with a lower bed height to diameter ratio so that a lower particle entrainment rate should be observed. Also the reactor will be equipped with more efficient dust collectors so that a larger percentage of the entrained material will be recovered and recycled. Construction of the larger reactor has been completed and it is undergoing shakedown tests.

## CONCLUSIONS

The technical feasibility of a process for drying, granulating, and calcining wastewater treatment sludge produced at the RAAP was demonstrated with bench-scale or larger equipment. It was shown that a satisfactory feedstock for a fluidized bed calciner can be prepared by dry-

ing the sludge followed by compacting or briquetting the material and then crushing and sizing the compacted solids. The prepared feed was calcined at rates up to 13 kg/h in an experimental two-zone fluidized bed reactor fired with natural gas. This reactor was operated with a lower reducing zone and an upper oxidizing zone so that the fluidized solids were alternately reduced and oxidized. As a consequence of this treatment, the calcium sulfate in the sludge was largely converted to calcium oxide and sulfur dioxide. When the treatment was conducted at 1150°C with the proper amounts of fuel and air, the bulk of the material was 96 to 97 percent desulfurized and a reactor off-gas containing 5 to 6 percent sulfur dioxide was produced. Calculations showed that much higher concentrations of sulfur dioxide could be produced by improving the thermal efficiency of the system. A larger fluidized bed reactor has been constructed and will be used for calcining granulated sludge at rates up to 1000 kg/day.

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