DEVELOPMENT OF A CALCIUM-BASED SORBENT FOR HOT GAS CLEANUP

Semi-Annual Technical Progress Report

April 1, 1999 - September 30, 1999

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ABSTRACT

The development and testing of potential calcium-based sorbents for hot gas cleanup continued. One of the most promising materials combines powdered limestone and a calcium aluminate cement by two step pelletization followed by steam curing. Reasonably strong pellets are produced with good adsorption characteristics by incorporating 20 wt.% cement in the core and 40 wt.% cement in the shell. The resulting 4.76 mm diameter pellets are capable of withstanding a crushing force approaching 11.5 N/mm before breaking and are also capable of removing H₂S from dilute, hot gas streams. The pellets are also regenerable and reusable.

Another promising material combines calcium carbonate powder and finely ground calcined alumina in tablet form. The small tablets are prepared by mixing the materials with water to form a thick paste which is then molded and dried. The tablets are hardened by calcining at either 1000 to 1100°C. The resulting tablets are strong and capable of removing H_2S from a dilute, hot gas stream.

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EXECUTIVE SUMMARY

The overall purpose of this project is the development of a superior calcium-based sorbent for hot gas cleanup in IGCC power generation systems. The sorbent should be capable of removing H_2S and COS from hot coal gas and should withstand repeated loading and regeneration.

A number of sorbent preparation methods have been investigated and tested. One method which stands out combines powdered limestone and a calcium aluminate cement into small spherical pellets made by a two step pelletization process which produces spherical pellets with a relatively weak core encased in a much stronger shell. The pellets are greatly strengthened by steam curing for up to three days, although most of the gain in strength is accomplished in the first 24 hr. The best overall results have been achieved by incorporating 20 wt.% calcium aluminate cement in the core and 40 wt.% in the shell. Cured pellets made with this composition and having an outside diameter of 4.76 mm were capable of withstanding a crushing force approaching 33.2 N/mm before breaking. After the pellets had been calcined at 1000°C, the force required to crush the pellets decreased to 11.5 N/mm which is considered acceptable. When the pellets were exposed to a gas stream containing 1.1% H_2S at 880°C for 60 min., they experienced a gain in weight of 7.8% which corresponded to 58% of the maximum possible gain in weight if all the calcium introduced in the form of limestone were utilized. These values increased to 11.1% and 82.5% when the temperature was raised to 920°C. The reacted pellets were also shown to be regenerable and their performance and compressive strength did not appear to suffer appreciably after 10 cycles of loading and regeneration.

Another promising sorbent was prepared by mixing calcium carbonate powder and finely ground calcined alumina with water to form a thick paste which was then molded into small tablets. After drying, the tablets were calcined at either 1000 or 1100° C to gain strength. Tablets having a diameter of 6.35 mm and thickness of 6.35 mm and made with 60 wt.% calcium carbonate were comparable in compressive strength to the 6.35 mm diameter spherical pellets described above. However, the gain in weight of the tablets was only 6.0% when they were treated with a gas stream containing 1.1% H₂S at 880°C for 60 min.

INTRODUCTION

To achieve the primary goal of this project, which is the development of a strong and regenerable, calcium-based sorbent for hot gas cleanup, various methods of combining lime or limestone with other materials have been investigated (1-5). While lime is an excellent sorbent for H_2S at high temperature, it lacks the necessary physical strength and stability for repeated regeneration and reuse. By combining lime with another material to impart the necessary physical properties, a better sorbent material can be achieved.

One of the most promising ways of combining lime with another material is through pelletization of the powdered materials in a revolving drum using a fine water spray. Although pellets with different structures and physical properties can be produced by this technique, it has proved advantageous to produce pellets with a relatively soft but reactive core made of limestone encased in a hard, porous shell containing both limestone and a hydraulic cement (5). A calcium aluminate cement designed for high temperature applications shows promise for this use, but it requires curing under moist conditions. It has

been found that the curing process can be accelerated by using steam. After curing, the pellets are calcined at high temperature.

Much of the work reported below was directed towards improving the pelletization process by investigating the effects of different variables on the ultimate strength and adsorption capacity of the core-in-shell pellets. The variables included curing time, the relative proportions of limestone and cement in the shell, and shell thickness. In addition, the adsorption and regeneration characteristics of the best material were evaluated.

Another method of producing a composite material for use as a sorbent was also evaluated. This method combines calcium carbonate or limestone with either the calcium aluminate cement or calcined alumina. The finely ground powders are mixed with water to form a thick paste which is then molded into small tablets that are subsequently dried and calcined. Tablets made with the calcium aluminate cement are also steam cured. The effects of tablet composition and size as well as calcination temperature on the properties of the material were investigated.

RESULTS AND DISCUSSION

Core in Shell Spherical Pellets

Although the limestone core in cement shell structure showed considerable promise for a high temperature sorbent, it was soon discovered that the composition of both the core and shell was critical. Initial experiments indicated that the shell tended to develop small cracks if the core was made entirely of limestone or the shell was made entirely of cement. The cracking tendency was overcome by adding some cement to the core and some limestone to the shell. Cracking was avoided when the core contained 20 wt.% calcium aluminate cement

and the shell contained 60 wt.% limestone. To investigate the importance of shell composition further, pellets were produced with 40, 60 and 80 wt.% limestone in the shell. Half the pellets were made with a shell thickness of 0.40 mm and half with a shell thickness of 0.83 mm. The limestone used for this purpose was obtained from the Three Rivers Quarry in Kentucky, and it contained 92.8 wt.% CaCO₃, 5.9% MgCO₃, and 1.1 wt.% SiO₂ according to the supplier. The material was ground and screened to provide $-297/+210 \,\mu\text{m}$ size particles for pelletization. The calcium aluminate cement was type CA-14 from Alcoa, and it was composed of $-44/+37 \,\mu\text{m}$ size particles.

To prepare the pellets cores with 80 wt.% limestone and 20 wt.% cement, the dry powders were premixed and placed in a pelletizing drum. As the drum revolved, a fine water spray was applied every 6-8 min. Although the material balled up rapidly, the sphericity and uniformity of the pellets improved by continuing to tumble the pellets for 1.0 hr. The pellets were subsequently screened carefully, and the -4.76/+3.96 mm size pellets were returned to the pelletizer drum for coating. The coating was applied by adding a mixture of limestone and cement particles to the tumbling pellets and by applying a fine water spray as needed. While the pellets were completely coated within a few minutes, they were kept in the revolving drum for 2.0 hr to improve their physical properties. During this time a fine water spray was applied at 6-8 min. intervals to keep the pellets moist. Afterwards the pellets were screened again, and the -5.83/+4.76 mm and -6.68/+5.63 mm sizes were selected for testing. These pellets were generally air-dried for one day and then steam cured at 100°C for three days. One portion of the cured pellets was set aside for various physical tests while another portion was calcined at 1000°C for 2.0 hr before testing.

,	Shell Thick.,	Limestone	Breaking for	Wt. gain, ^a	
	mm	conc., wt.%	Cured	Calcined	%
5.60	0.83	40	55.0 ± 6.7	20.1 ± 1.1	3.2 ± 0.2
5.60	0.83	60	33.4 ± 7.0	13.2 ± 6.1	5.6 ± 0.3
5.60	0.83	80	8.1 ± 1.8	2.5 ± 0.2	10.5 ± 1.8
4.76	0.40	40	47.5 ± 5.7	18.8 ± 3.5	4.8 ± 0.4
4.76	0.40	60	26.7 ± 2.6	12.9 ± 0.5	8.4 ± 0.1
4.76	0.40	80	7.5 ± 1.6	1.8 ± 0.1	11.0 ± 0.7

Table 1. Properties of core in shell pellets made with different concentrations of limestone in the shell.

^aTreated with 1.1% H_2S at 880°C for 1.0 hr.

The prepared pellets were subjected to various physical tests to determine their compressive strength, pore volume, surface area, and apparent porosity. The force required to break a pellet under compression was measured and divided either by the pellet diameter or by the pellet cross sectional area. This test was repeated five times for each batch of pellets. The apparent porosity of the pellets was measured by gas pycnometry. The surface area was determined by using the BET method and measuring the adsorption of nitrogen at 77.35°K.

The apparent adsorption capacity of the calcined pellets was determined by measuring the gain in weight of a single pellet held at a preselected temperature and exposed to a gas stream consisting largely of nitrogen with a small amount of H_2S . As the pellet reacted with H_2S it gained weight which was measured with a sensitive electrobalance.

The results of formulating a series of core in shall pellets with different proportions of limestone and calcium aluminate cement in the shell are shown in Table 1. In each case the core was composed of 80 wt.% limestone and 20 wt.% cement. The breaking force is

indicated for both the cured pellets and the calcined pellets but not for the pellets which had been reacted with H_2S . The apparent adsorption capacity of the calcined pellets is also shown. The adsorption capacity represents the gain in weight of the pellets exposed to a gas stream containing 1.1% H₂S at 880°C for 1.0 hr. Each listed value of adsorption capacity is an average of three determinations. It can be seen that the force required to break the calcined pellets was much less than that required to break the cured but otherwise untreated pellets. Also the breaking force decreased markedly as the limestone concentration of the shell rose. On the other hand, the adsorption capacity increased noticeably as the limestone concentration increased. Shell thickness appeared to have a greater effect on adsorption capacity than on pellet strength. The percent gain in weight was noticeably larger for the pellets with the thinner shells which could have been due to their overall higher lime content, whereas the force required to break the pellets with the thinner shells was only slightly lower. Pellets with the thinner shell made with 60 wt.% limestone appeared to offer the best compromise between crushing strength and adsorption capacity. Therefore, this pellet formulation was selected for more complete characterization.

The effect of steam curing time on the compressive strength of the pellets with the apparently optimum composition was determined by subjecting different pellets to different curing times which ranged from 0 to 72 hr. The pellets were tested after curing and also after they had been cured and calcined at 1000°C. Pellets which had been steam cured but not calcined developed a maximum compressive strength after only 8 hr of curing, whereas pellets which had been subjected to both curing and calcining continued to gain strength with up to 72 hr of steam curing. However, the additional gain in strength achieved by extending the curing time from 24 to 72 hr was slight.

Property	Cured	Calcined
Breaking force, N/mm	33.2 ± 3.5	11.5 ± 1.0
Crushing pressure, kPa	8470 ± 1100	3070 ± 260
Crushing pressure, lb/in ²	1230 ± 160	445 ± 38
Pore volume, cm^3/g	0.59 ± 0.02	0.61 ± 0.02
Apparent porosity, %	48.0 ± 1.9	65.5 ± 3.1
Surface area, m^2/g	8.2 ± 2.2	6.7 ± 1.8
Theoretical maximum weight gain, %		13.5 ± 0.7
^a Actual weight gain, %		7.8 ± 0.6

Table 2. Properties of core-in-shell pellets with 80 wt.% limestone in the core and 60 wt.%limestone in the shell. Results of replicate tests with 95% confidence intervals.

^aTreated with 1.1% H_2S at 880° for 1.0 hr.

Several batches of pellets were prepared with 80 wt.% limestone in the core and 60 wt.% limestone in the shell. After the pellets had been steam cured for three days and calcined at 1000°C for 2.0 hr, they were tested by various methods and the results are indicated in Table 2. The pellets had a shall thickness of 0.4 mm and an outside diameter of 4.76 mm based on screen size and confirmed by direct measurement with a pair of calipers. The compressive strength is reported both as the breaking force per unit diameter and the crushing pressure based on the pellet cross sectional area. The average breaking force for five different batches of pellets was 33.2 N/mm after curing and 11.5 N/mm after both curing and calcining. These results are believed to be more reliable than those reported in Table 1 which were based on only a single batch of pellets for each different shell composition. Although the crushing strength of the pellets was greatly reduced by the heat treatment, the

other properties were affected much less. Calcining caused the apparent porosity to rise which was probably due to the evolution of carbon dioxide and the creation of larger pores as a result of incipient sintering. Calcining also reduced the surface area which seemed to reflect the conversion of smaller pores into larger pores by sintering.

Based on the reputed composition of the limestone and the loss in weight experienced by the cured pellets when they were heated gradually in a thermogravimetric analysis apparatus, it was possible to predict the gain in weight which the pellets could experience if all of the calcium oxide from the limestone were converted to calcium sulfide by reaction with H₂S. The predicted value was 13.46 wt.%. When several calcined pellets were treated with a gas stream containing 1.1% H₂S at 880°C for 1.0 hr, the average gain of the pellets was 7.83 wt.% or 58% of the theoretically possible gain in weight.

A series of adsorption tests was conducted to study the effects of temperature and gas concentration on the apparent rate of conversion of the prepared core in shell pellets. Again the pellets selected for these tests were prepared with 80 wt.% limestone in the core and 60 wt.% limestone in the shell. The pellets were steam cured for three days and calcined at 1000°C for 2 hr before testing. The results of two runs made under similar conditions indicate that the results were highly reproducible (see Figure 1). These results were achieved by treating 4.76 mm diameter pellets with a gas stream containing 3% H₂S at 880°C. It can be seen that the pellets gained weight rapidly at first and then more slowly. After 150 min. the pellets exceeded the theoretical maximum gain in weight which did not take into account the adsorptive capacity of the calcium aluminate cement which was significant.

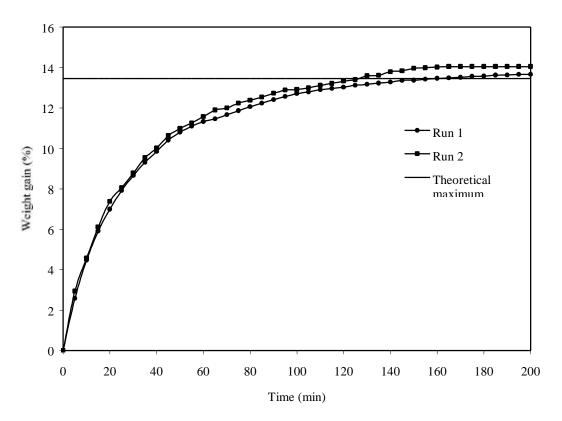


Figure 1. The gain in weight of a core-in-shell sorbent exposed to a gas stream containing 3% H₂S at 880°C.

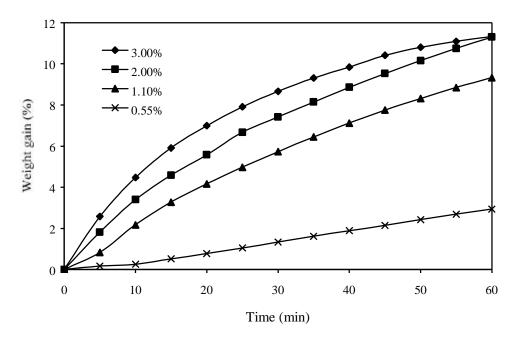


Figure 2. Effect of H_2S concentration on the rate of gain in weight of the core-in-shell sorbent at 880°C.

The results of several adsorption tests conducted to determine the effect of gas concentration are presented in Figure 2. These tests were conducted at 880°C. The increase in the slope of the curves with gas concentration indicates that the rate of adsorption of H₂S increased greatly with gas concentration. It is evident that the increase in H₂S concentration from 0.55% to 1.1% had the greatest effect on the rate of adsorption, and it also changed the shape of the adsorption curves which suggests a change in rate controlling mechanism.

The results of other adsorption tests designed to determine the effect of temperature are shown in Figure 3. These tests were conducted with 1.1% H₂S in the gas. It is apparent that the highest average rate of adsorption over 60 min. was observed at a temperature of 920°C. Either higher or lower temperatures resulted in a lower rate of adsorption.

In order to see how well the core-in-shell sorbent would withstand repeated loading and regeneration, a single pellet was subjected to 10 cycles of loading and regeneration. The composition of the pellet was similar to that used for the preceding tests. During the adsorption phase of each cycle, the pellet was treated at 880°C for 20 min. with a gas stream containing 3% H₂S. During the regeneration phase, the pellet was treated at 1100°C for 60 min. with a gas stream containing 67% CO₂ and 33% N₂. The weight gained by the pellet during each cycle is indicated in Figure 4. Although there was some variation in the weight gained from cycle to cycle and a slight downward trend overall, the results suggest that the performance of the material did not suffer significantly. This conclusion was supported further by crushing the pellet at the end of the test. It required a force of 12.4 N/mm to break the pellet which was in the range of force required to break calcined but unreacted pellets (see Table 2). Therefore, the crushing strength of the pellet did not appear to have suffered significantly.

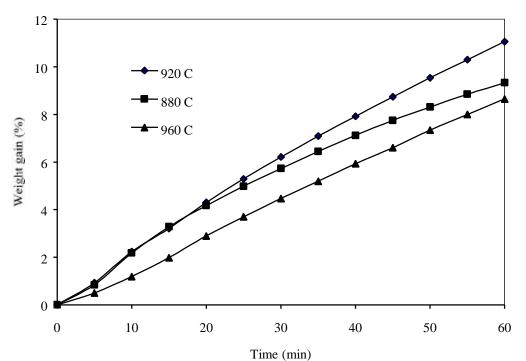


Figure 3. Effect of temperature on the rate of gain in weight of the core-in-shell sorbent treated with 1.1% H₂S.

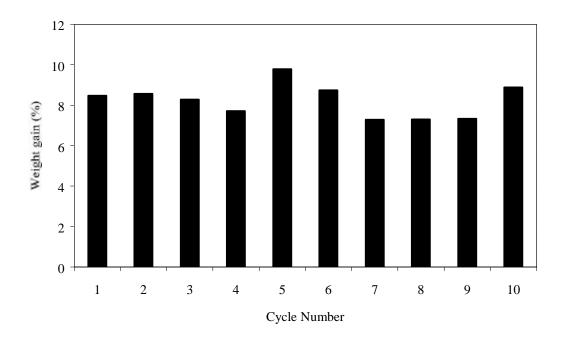


Figure 4. Results of repeated adsorption and regeneration of a single core-in-shell pellet. Adsorption was conducted at 880°C with 3% H₂S for 20 min. Regeneration was conducted at 1100°C with 67% CO₂ for 60 min.

Molded Tablets

Molded tablets composed of a mixture of calcium carbonate and either A-16SG calcined alumina or CA-14 calcium aluminate cement were prepared by first mixing the dry powders and then adding sufficient water to form a thick paste. The paste was pressed into a mold which produced tablets that were 6.35 mm (0.25 in.) thick and either 12.7 or 6.35 mm (0.50 or 0.25 in.) in diameter. The Plexiglas mold was treated with a mold release agent (PAM manufactured by International Food Corp.). The amount of water incorporated in the paste was approximately 20% by weight. The paste also contained approximately 5% by weight of a solution of Darvan 821 dispersion agent. Both the calcined alumina and calcium aluminate cement were supplied by Alcoa. The calcium carbonate was reagent grade with a particle size finer than 200 mesh. The molded tablets were allowed to air dry for 24 hr. before they were removed from the mold. Then the tablets were air dried for an additional 2 hr. before they were tested or calcined. Since the CA-14 cement is a hydraulic cement, the tablets made with this material were steam cured for three days to gain the necessary strength. The dried and/or cured tablets were subsequently heated gradually over a 12 hr. period to reach an induration temperature of either 1000 or 1100°C. After the pellets had been held at this temperature for 2.0 hr., they were cooled gradually back to room temperature over a 10 hr. period. The crushing strength and porosity of the heat-treated tablets were determined, and in some cases the adsorption capacity was also measured. For measuring the crushing strength, a gradually increasing force was applied across the tablet mounted on its circular edge between the plates of a hydraulic press. The crushing force per

Composition		Apparent	Calcined Crushing Pressu	
CA-14 wt.%	CaCO ₃ wt.%	– Porosity, - %	kPA	lb/in ²
60	40	58.1	2255	327
50	50	65.8	691	100
40	60	71.5	413	60
30	70	74.0	316	46

Table 3. Properties of the 12.7 mm diameter tablets made with CA-14 cement and CaCO₃ and calcined at 1000°C.

unit of cross-sectional area is reported based on the rectangular area perpendicular to the circular area.

A number of 12.7 mm (0.50 in.) diameter tablets were prepared with different proportions of calcium carbonate and CA-14 cement. After the tablets were steam cured, they were heat treated at 1000°C and then tested. The results are shown in Table 3 where it can be seen that the pressure required to crush the tablets ranged from 2255 kPa (327 psia) for tablets made with 60 wt.% CA-14 cement down to 316 kPa (46 psia) for tablets made with 30 wt.% CA-14 cement. Over the same range of composition the apparent porosity increased from 58% to 74%.

Another series of 12.7 mm (0.50 in.) diameter tablets was prepared using A-16SG alumina and calcium carbonate in different proportions. These tablets did not require steam curing, and even before heat treating they exhibited a significant crushing strength. The pressure required to crush the tablets before and after heat treating is shown in Table 4. It is apparent that the pressure needed to crush the tablets was considerably greater after heat

Composition		Apparent	Crushing Pressure			
Al ₂ O ₃	CaCO ₃	- Porosity,	Unca	lcined	Calc	cined
wt.%	wt.%	%	kPA	lb/in ²	kPA	lb/in ²
60	40	60.0	486	70	3144	456
50	50	67.2	438	64	3002	435
40	60	69.8	330	48	1744	253

Table 4. Properties of the 12.7 mm diameter tablets made with A-16 SG alumina and CaCO₃ and calcined at 1000°C.

treating than before. Also the pressure required to crush the tablets made with A-16SG alumina was substantially greater than that required to crush the tablets made with CA-14 cement (see Figure 5). On the other hand, the apparent porosity of the two series of tablets was similar.

Since the 12.7 mm (0.50 in.) diameter tablets were too large to be used for gas adsorption tests without first being crushed, the method was applied to making 6.35 mm (0.25 in) diameter tablets which could be used directly for adsorption testing. A number of the smaller tablets were prepared with different proportions of A-16SG alumina and calcium carbonate. One set of tablets was heat treated at 1000°C while another set was heat treated at 1100°C. The results are presented in Table 5 and Figure 6. It is apparent that the pressure required to crush the tablets heated to 1100°C was slightly greater than that required to crush the tablets heated to 1100°C. As before, the crushing strength of tablets decreased markedly as the alumina content decreased. However, for any given composition the pressure required to crush a 6.35 mm (0.25 in.) diameter tablet was appreciably greater than that required to crush a 12.7 mm (0.50 in.) diameter tablet (see Figure 5).

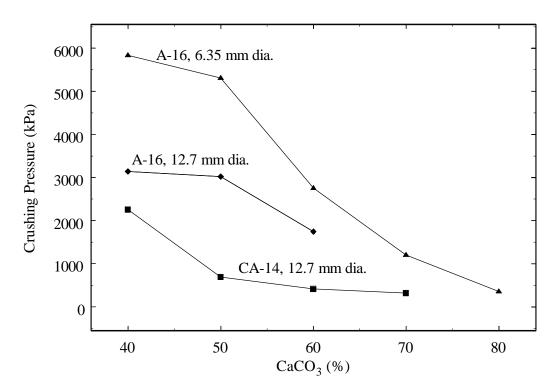


Figure 5. Pressure required to crush different diameter tablets made with different bonding agents.

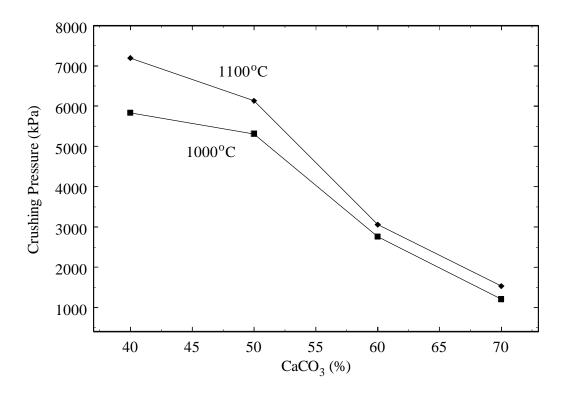


Figure 6. Pressure required to crush 6.35 mm diameter tablets treated at different temperatures.

Composition		mposition Calcin. Apparent		Crushing Pressure			
Al_2O_3 ,	CaCO ₃ ,	Temp.,	Porosity,	Uncalcined		Calcined	
wt.%	wt.%	°C	°C %	kPa	lb/in ²	kPa	lb/in ²
60	40	1000	38.8	1516	220	5837	847
50	50	1000	41.5	1364	198	5314	771
40	60	1000	47.9	1343	195	2757	400
30	70	1000	55.6	883	128	1205	175
20	80	1000	64.3	571	83	360	52
60	40	1100	62.1			7196	1044
50	50	1100	63.6			6134	890
40	60	1100	67.9			3059	444
30	70	1100	76.2			1532	222

Table 5. Properties of the 6.35 diameter molded tablets made with A-16SG alumina and $CaCO_3$

The apparent porosity of the 6.35 mm (0.25 in.) diameter tablets which had been heated to 1000°C was noticeably smaller than that of either similar size tablets heated to 1100°C or larger size tablets heated to 1000°C. While the increase in porosity and crushing strength attributable to an increase in induration temperature can be explained on the basis of an increase in particle sintering, the difference in porosity attributable to the difference in tablet diameter needs a different explanation. The lower porosity of the smaller tablets goes hand in hand with their greater strength.

The adsorption capacity of several 6.35 mm (0.25 in.) diameter tablets was determined by exposing each tablet individually to a gas stream containing 1.1 mole % H₂S. The temperature of the reaction system was maintained at 880°C, and the weight of each tablet was measured and recorded as the material reacted. The gain in weight of tablets made with different proportions of A-16SG alumina and calcium carbonate is indicated in Figures 7 and

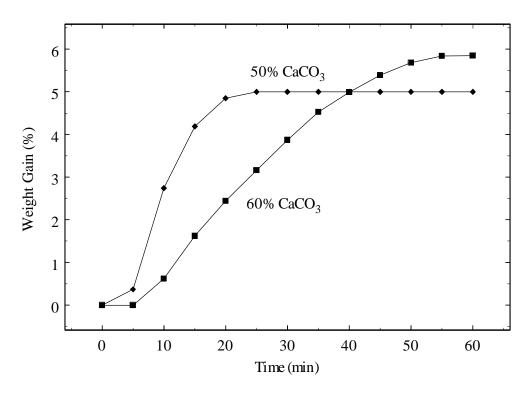


Figure 7. Gain in weight of 6.35 mm diameter tablets which had been heat treated at 1000° C and then exposed to 1.1% H₂S at 880°C.

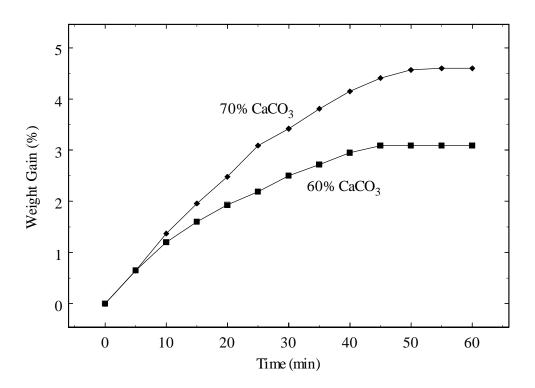


Figure 8. Gain in weight of 6.35 mm diameter tablets which had been heat trated at 1100° C and then exposed to 1.1% H₂S.

8. Tablets which had been calcined or heat-treated at 1000°C produced the results shown in Figure 7, whereas the tablets which had been calcined at 1100°C produced the results indicated by Figure 8. It can be seen that tablets with a higher initial content of calcium carbonate tended to experience a larger gain in weight. Also tablets which had been calcined at 1000°C tended to undergo a larger gain in weight than tablets which had been calcined at 1100°C. The results suggest that the amount of free calcium oxide available to react with H₂S was proportional to the initial concentration of calcium carbonate and also that more free calcium oxide was available in tablets calcined at the lower temperature. The heat treatment tended to convert the alumina and the calcia into calcium aluminate which was revealed by X-ray diffraction analysis. Apparently more of these materials were converted at the higher temperatures so less free calcium oxide was available for reaction with H₂S.

CONCLUSIONS

One of the most promising materials for removing H_2S from hot coal gas was prepared by combining powdered limestone with a calcium aluminate cement in a two step pelletization process which produced pellets with a relatively weak core encased in a much stronger shell. The best results were achieved by incorporating 20 wt.% calcium aluminate cement in the core and 40 wt.% in the shell. While a higher concentration of cement in the shell produced stronger pellets, it also reduced the adsorption capacity of the material. The pellets were steam cured and then calcined at 1000°C before testing as a sorbent. Although the crushing strength of the finished pellets continued to increase with curing time for up to three days, most of the strength was gained in the first 24 hr of curing. The rate of adsorption of H_2S was observed to reach a maximum value at 920°C, and it was observed to increase with increasing H_2S concentration. Subjecting a sample of the sorbent to 10 cycles of adsorption and regeneration had only a slight effect on the properties of the material. Therefore, the sorbent may be reusable for many more cycles.

Other promising sorbent materials were prepared by combining calcium carbonate with either calcined alumina or calcium aluminate cement. The finely divided powders were mixed with water to form a thick paste which was then molded into small tablets. The dried tablets were strengthened and hardened either by steam curing in the case of tablets made with calcium aluminate cement or by calcining in the case of tablets made with alumina. The crushing strength of the tablets made with alumina was greater than that of the tablets made with cement. In both cases, the crushing strength declined as the concentration of calcium carbonate in the tablet was raised. On the other hand, the adsorption capacity of the material tended to rise with increasing calcium carbonate content. A larger pressure was required to crush 6.35 mm diameter tablets than to crush 12.7 mm diameter tablets. In addition, alumina containing tablets which had been calcined at 1100°C were stronger than those calcined at 1000°C. However, the adsorption capacity of tablets calcined at 1000°C.

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