Fuel Rich Sulfur Capture in a Combustion Environment

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■ A refractory-lined, natural gas furnace was used to study fuel rich sulfur capture reactions of calcium sorbents under typical combustion conditions. The fuel rich sulfur species H_2S and COS were monitored in a near-continuous fashion using a gas chromatograph equipped with a flame photometric detector and an automatic sampling system which sampled every 30 s. Below the fuel rich zone, 25% excess air was added and the ultimate fuel lean capture was simultaneously measured using a continuous SO_2 monitor. Under fuel rich conditions high levels of sulfur capture were obtained, and calcium utilization increased with sulfur concentration. The ultimate lean capture was found to be weakly dependent on sulfur concentration and independent of the sulfur capture level obtained in the fuel rich zone. Thus, for the sorbents used in this study, the high captures realized in the rich zone were lost in the lean zone.

Introduction

A major concern associated with the combustion of coal for heat and electricity is the emission of acid rain precursors, NO_x and SO_2. Dry, calcium-based sorbent injection is a potential method for reducing SO_2 emissions from existing coal-fired boilers. The calcium sorbents most frequently considered are calcium carbonate and calcium hydroxide, which upon injection into a furnace environment rapidly decompose to highly reactive calcium oxide.

$$Ca(OH)_2(s) + heat \rightarrow CaO(s) + H_2O \tag{1}$$

$$CaCO_3(s) + heat \rightarrow CaO(s) + CO_2 \tag{2}$$
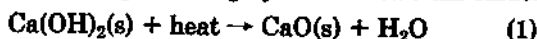
It is generally assumed that the above calcination reactions occur with little change in the particle dimensions (2). Since the molar volume of CaO (16.9 cm^3/mol) is less than the molar volume of $CaCO_3$ (36.9 cm^3/mol) or $Ca(OH)_2$ (33.1 cm^3/mol), the CaO formed is greater than 50% porous and surface areas of 90 m^2/g have been reported (3).

Considerable study has been devoted to the fuel-lean SO_2 reaction (1, 4, 5):

$$CaO + SO_2 + \frac{1}{2}O_2 \rightarrow CaSO_4 \tag{3}$$
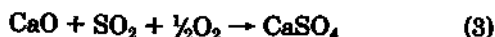
Since the molar volume of $CaSO_4$ (52.2 cm^3/mol) is greater than that of either parent material, the pore volume of the CaO will theoretically be filled before complete conversion to $CaSO_4$ is reached. Thus, there is a theoretical basis for an upper limit on the fuel lean SO_2 capture, assuming no particle expansion during the short reaction times.

Borgwardt et al. (2, 4) have shown that the important fuel rich sulfur capture reactions to be considered are

$$CaO(s) + H_2S \leftrightarrow CaS(s) + H_2O \tag{4}$$

$$CaO(s) + COS \leftrightarrow CaS(s) + CO_2 \tag{5}$$
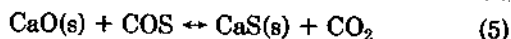
CaS is thermodynamically more stable at higher temperatures than is $CaSO_4$ and Borgwardt et al. have shown the fuel rich reactions are kinetically faster than the fuel lean. Also, the fuel rich reactions should be less limited by intraparticle resistances because the molar volume of CaS (28.9 cm^3/mol) allows for complete conversion of CaO to CaS before the initial void space is filled.

Background

The fuel rich reactions have not been as extensively studied as the fuel lean reaction, especially under combustion conditions. Most early kinetic studies of reactions 4 and 5 utilized microbalance techniques (6–9) where milligram quantities of relatively large particles were reacted in a bed. These studies indicated activation energies of 3–5 kcal/mol, which are typical of gas diffusion control and do not reflect the true chemical kinetics. The large particles that were typically used resulted in substantial intraparticle diffusion resistance, and even with small particles (8), agglomeration increases the effective particle size. Also, interparticle effects are not easily eliminated in a bed of solids because of the close proximity of adjacent particles.

Borgwardt (2) used milligram quantities of well-dispersed, 2-μm CaO particles to study the kinetics of reactions 4 and 5 as a function of the CaO surface area. He found the activation energy of both reactions to be 31 kcal/mol. Attar and Dupuis (10) used a pulsed differential reactor and measured an activation energy of 37 kcal/mol for reaction 4. Extrapolating the results of these studies to higher temperatures indicates that high levels of sulfur capture may be obtainable in the fuel rich zone of a multistage pulverized coal burner provided the sulfur capture is not lost during the transition to fuel lean conditions. However, high-temperature study is required to verify this extrapolation.

Zallen et al. (11) burned pulverized coal premixed with limestone and found staging increased sulfur capture with Utah coal, but the effect was less with western Kentucky coal. Freund and Lyon (12) found that mixing sorbent and coal produced poor results but that ion-exchangeable calcium in coal was much more effective in fuel rich sulfur capture. Freund (13) reacted dispersed 69-μm limestone and dolomite particles with H_2S (1250 and 2600 ppm) in a tubular reactor at 1065 and 1320 °C and various levels of calcium availability (Ca/S = 1–4). The sulfur reductions
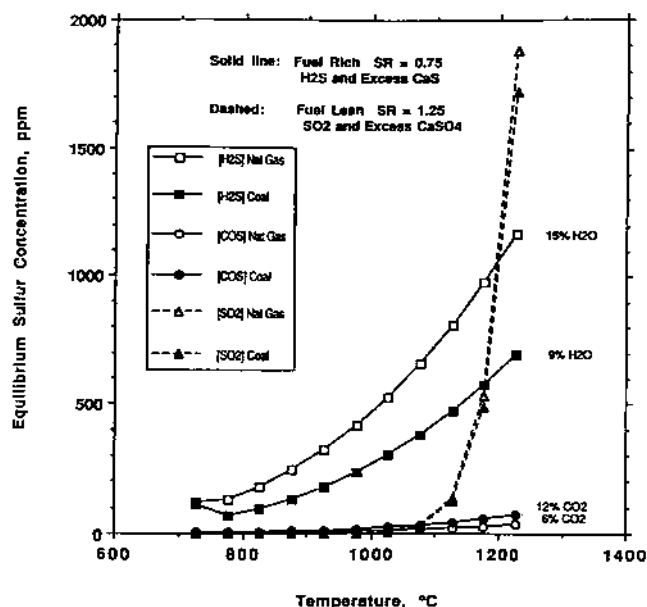
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Environ. Sci. Technol., Vol. 26, No. 7, 1992 **1427**

Figure 1. Equilibrium concentration of H_2S and COS in the presence of CaS, and SO_2 in the presence of $CaSO_4$, under typical combustion conditions.

obtained were not impressive and were greater at the lower temperature, indicating that at higher temperatures reaction 4 is slow compared to desurfacing reactions. Internal particle diffusion was determined to be the principle resistance to sulfur capture. The use of smaller particles more typical of dry sorbent injection may have resulted in much higher rates. In a recent study, Whitney et al. (14) physically mixed coal and CaO and combusted it at 1103 °C under fuel rich conditions in a semibatch experiment. They achieved high levels of sulfur retention in the ash approaching calculated equilibrium values.

In summary, some studies have shown that high levels of fuel rich capture are obtainable under combustion conditions but other studies have not. To date, no studies have simultaneously measured fuel rich and fuel lean sulfur capture. The objective of the present study is to quantify the parameters important to fuel rich sulfur capture and determine whether this capture is lost when the sorbent enters a fuel lean zone.

Thermodynamics

Thermodynamically, the fuel rich sulfur capture reactions are quite different from the fuel lean sulfur capture reaction. The gaseous products of the fuel rich reactions are also major products of combustion, H_2O and CO_2, which is not the case for the fuel lean reaction. This should make fuel rich sulfur more dependent on fuel type than fuel lean sulfur capture; i.e., coal combustion produces more CO_2 and less H_2O than natural gas combustion.

Figure 1 shows the results of thermodynamic calculations using the NASA CET86 chemical equilibrium program (15). Depicted in this figure is the temperature dependence of the equilibrium concentrations of H_2S and COS in the presence of CaO and CaS at a stoichiometric ratio (SR) of 0.75 and the equilibrium concentration of SO_2 in the presence of CaO and $CaSO_4$ at SR = 1.25. To illustrate the effect of fuel type the calculations were made for the combustion conditions of both natural gas and Illinois bituminous coal (16).

Above 1200 °C the equilibrium SO_2 concentration is very high (CaSO$_4$ decomposes) so fuel lean SO_2 reduction is thermodynamically prohibited. Below 1200 °C the fuel lean equilibrium SO_2 level drops steeply and below 1100
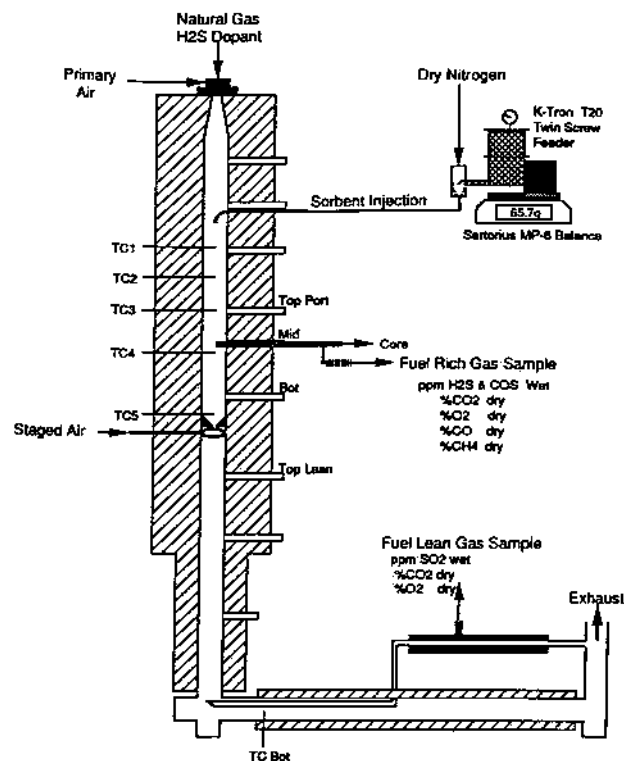

Figure 2. Diagram of the experimental furnace setup.

°C the equilibrium SO_2 level is nil, meaning no thermodynamic limitation to sulfur reduction exists. Also, it can be seen that fuel type makes little difference in equilibrium SO_2 level. The fuel rich equilibrium H_2S and COS levels are characterized by a gradual drop with decreasing temperature. Thus, thermodynamic limitation to sulfur reduction exists at all temperatures of interest, especially for H_2S. The equilibrium H_2S level for coal is lower because less water is produced; likewise, the equilibrium COS level for coal is higher because more CO_2 is produced. It is often stated that CaS is thermodynamically more stable than $CaSO_4$ at high temperatures, which is true; however, it should be further noted that fuel rich sulfur reduction is thermodynamically limited to the equilibrium sulfur level.

Experimental Section

Furnace. Figure 2 illustrates the furnace facility used in this study. The furnace was a natural gas fueled, down-fired, refractory-lined furnace 13 ft tall and 6 in. in inner diameter. The overall fire rate was 90 000 Btu/h. The H_2S dopant was accurately metered into the natural gas stream and the primary air was fed tangentially into the burner to help produce a well-mixed flame. All major flows into the furnace were metered through high-accuracy Brooks rotameters except the natural gas which was metered through a Rockwell International R-315 dry gas meter. The top portion of the furnace was operated under fuel rich conditions at a stoichiometric ratio (SR1 $\approx$ $Q_{O_2}/2Q_{CH_4}$) of either 0.65 or 0.75.

The total flow in the rich zone was adjusted to a constant value for any given set of conditions, and three horizontal, rich sample ports (top, middle, bottom) facilitated measurements at residence times of 0.30, 0.45, or 0.65 s (assuming plug flow). The fuel rich gas sample was monitored continuously for H_2 and CO_2; H_2S and COS were monitored in a nearly continuous fashion, and CO and CH_4 were measured periodically to close the material balance on the rich zone. Five type B thermocouples positioned on the gas stream center line were used to continuously monitor
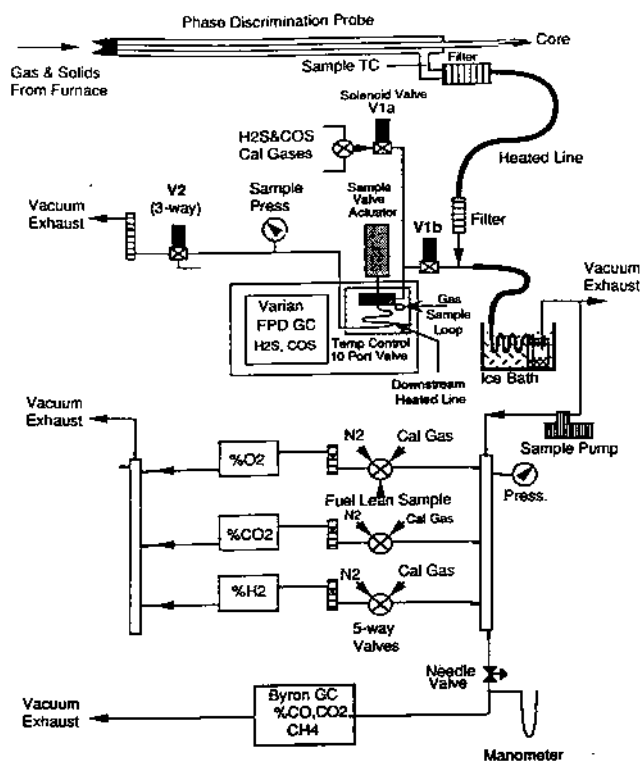
Figure 3. Schematic of the fuel rich sample and analysis system.

Figure 4. Typical GC trace for a fuel rich sulfur capture run.

Figure 5. Schematic of fuel lean sample and analysis system.

the rich zone temperature profile. Suction pyrometry and fine wire thermocouples were used periodically to check the effects of radiation on the temperature measurements.

The sorbents were injected pneumatically with dry nitrogen through a 4.5-mm-i.d. stainless steel probe which was either water cooled (for high quench rate) or refractory insulated (for lower quench rates). The sorbent feed rate was metered with a K-Tron T20 twin screw volumetric feeder. The rate of sorbent addition was measured by mass loss using a Sartorius MP-8 balance, which was accurate to ±0.1 g.

Below the rich zone, the flow was partially choked and 25% excess air was added through the staged air injection ring (SR2 = 1.25). At the bottom of the furnace the ultimate fuel lean sulfur capture was continuously monitored using an Anacon nondispersive ultraviolet analyzer (ND-UV). The lean oxygen and carbon dioxide were also monitored continuously.

All of the continuous gas concentration measurements, gas temperature measurements, and sorbent weight loss measurement were collected by a Digistrip III data logger. A data set was collected every 10 s and stored on computer disks.

Fuel Rich Sample and Analysis System. Figure 3 shows a schematic drawing of the fuel rich analytical system. The gas sampling probe and fuel rich sulfur analysis system are discussed in detail elsewhere (17, 18) and are only briefly described here. The fuel rich gas sample was taken from the center line of the furnace through a phase discrimination probe, which separates most of the solids from the gas at the probe tip. The fuel rich sulfur sample was continuously drawn through the gas sampling valve of a Varian 3700 GC equipped with a sulfur-specific flame photometric detector (FPD). GC samples were injected automatically every 30 s, resulting in near-continuous monitoring of H_2S and COS. Figure 4 shows a typical GC trace for a run. The H_2S and COS peaks were well separated with a Supelco Supelpak-S column, and the COS was completely evolved in just under 30 s. There are 11 peaks before sorbent injection, 10 peaks
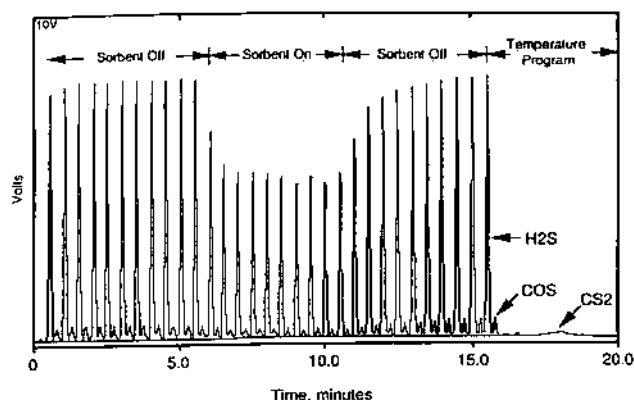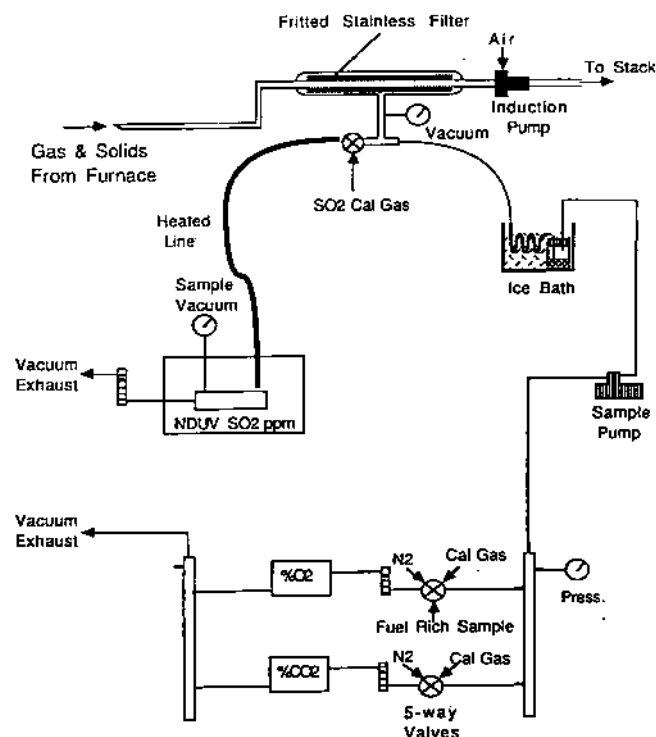
during sorbent injection, and 10 peaks after sorbent injection. The sulfur capture calculation is based on the average fuel rich sulfur level (H_2S + COS) 2 min before sorbent injection begins and the average level 2 min before sorbent injection ends.

The major portion of the rich sample continued on after the GC to an ice bath where the water was condensed out. Part of the dry stream was then pressurized and distributed to the continuous analyzers. A Beckman CO_2 analyzer and a Delphi H_2 analyzer were dedicated to the rich analytical system. The Beckman O_2 analyzer was shared between the rich and lean analytical systems. The O_2 measurement on the rich sample was used as a test for leaks into the sample. Since there should be no oxygen in the fuel rich sample, any O_2 present indicated a leak into the sample stream. An oxygen concentration of less than 0.2% was taken as leak free, and typically values were less than 0.05%.

The Byron GC was used to measure CO, CO_2, and CH_4 in the fuel rich gas sample. It was not operated when sulfur was in the sample to protect the catalyst used in this instrument. The Byron GC analysis was done prior to selected runs and whenever the stoichiometry was changed in the furnace.

Table I. Gas Analysis Summary SR = 0.65

	flows (SCFM), °C/s		flows (SCFM), °C/s
Q_{gas} [a]	1.592	Q_{air2}	9.30
Q_{inj}	1.60	Q_{tot1}	14.33
Q_{air1}	10.08	Q_{tot2}	22.61

analyte	conc, vol % wet	analyte	conc, vol % wet
Rich Zone			
CO_2	4.95	H_2	6.27
CO	6.33	H_2O	15.33
CH_4	0.42	N_2	66.70
Rich Zone S Fraction			
H_2S	0.978	CS_2	trace
COS	0.022		
Lean Zone			
O_2	3.69	H_2O	14.23
CO_2	7.30	N_2	74.78

[a] With composition $C_{1.0446}H_{4.0415}O_{0.02015}N_{0.0074}$.

Table II. Gas Analysis Summary SR = 0.75

	flows (SCFM), °C/s		flows (SCFM), °C/s
Q_{gas} [a]	1.461	Q_{air2}	7.11
Q_{inj}	1.60	Q_{tot1}	14.56
Q_{air1}	10.67	Q_{tot2}	20.88

analyte	conc, vol % wet	analyte	conc, vol % wet
Rich Zone			
CO_2	5.91	H_2	3.80
CO	4.89	H_2O	16.48
CH_4	0.00	N_2	68.92
Rich Zone S Fraction			
H_2S	0.910	CS_2	trace
COS	0.090		
Lean Zone			
O_2	3.67	H_2O	14.14
CO_2	7.24	N_2	74.95

[a] With composition $C_{1.0446}H_{4.0415}O_{0.02015}N_{0.0074}$.

Fuel Lean Sample System. Figure 5 shows a schematic drawing of the fuel lean sample system. Gas and solids were drawn from the furnace by a vacuum induction pump. A solids-free sample was obtained by pulling a small sample through a stainless steel filter. Part of the hot wet sample was conveyed via a heated Teflon line directly to a continuous Anacon NDUV SO_2 analyzer. The rest of the lean sample was dried in an ice bath, pressurized, and sent to the continuous Beckman O_2 and CO_2 analyzers.

Sorbents. Two types of sorbents were used, a Marblehead hydrate and a Fredonia carbonate. The hydrate was simply sifted to <300 μm to remove lumps, while the carbonate was aerodynamically sized to 3-10 μm using a Donaldson Acucut classifier. Fewer runs were made with the carbonate because of the limited amount of the sized material.

Results and Discussion

Overall Gas Analysis. Tables I and II give a summary of the average flows and gas analysis in the rich and lean zones for the runs with a rich zone stoichiometric ratio (SR) of 0.65 and 0.75, respectively. A number of observations can be made regarding the rich zone gas analysis at the two stoichiometric ratios. When the oxygen is in-
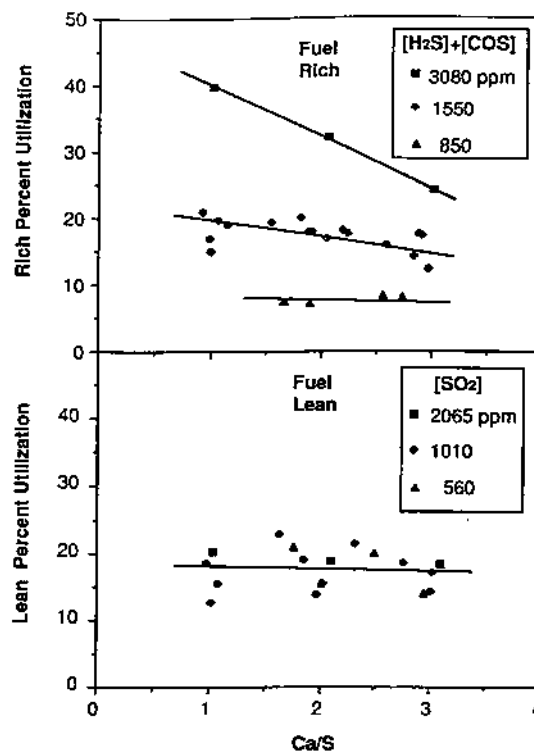

Figure 6. Effect of Ca/S for hydrate at SR = 0.75.

creased from SR = 0.65 to 0.75, the level of CO and CH_4 decreases and more CO_2 is formed and the level of H_2 decreases and more H_2O is formed. During the runs, it was visually observed that more carbon black was formed at SR = 0.65. There were also differences in the distribution of sulfur species at the two stoichiometric ratios. The major fuel rich sulfur species were H_2S and COS. More COS was formed at SR = 0.75, and at both ratios the distribution was independent of overall sulfur level and position sampled in the furnace. More CS_2 was formed at SR = 0.65; however, the level was not quantified and was considered negligible.

Residence Time Effects. Both the fuel rich capture and the fuel lean transition reactions were found to occur in less than 0.30 s. The rich capture was found to be insensitive to the residence times studied (0.30-0.65 s). This may be due to product layer diffusion resistances and low temperatures at the lower sample ports (typically 1010 °C at the top port, 955 °C at the middle port, and 900 °C at the bottom port). Although at the lower temperatures the thermodynamics are more favorable, the kinetic rate may be too slow for much additional reaction to occur. On the lean side, the lean capture a short distance below the point of staged air injection (~0.11 s) was essentially the same as that measured at the furnace bottom during limited simultaneous lean measurements.

Effects of Calcium Availability and Sulfur Concentration. Figures 6-9 show calcium utilization as a function of calcium availability and sulfur concentration for each sorbent at stoichiometric ratios of 0.75 and 0.65. Each figure shows data taken simultaneously from the rich and lean zones of the furnace. The data are reported in terms of percent utilization, which is defined as the sulfur capture divided by the calcium to sulfur molar ratio (Ca/S). The results on the lean side (lower portion of each figure) are very typical of lean data reported by others (1); i.e., the calcium utilization is weakly dependent on Ca/S and sulfur concentration.

The fuel rich data (upper portion of each figure) exhibit a strong positive dependence on sulfur concentration and
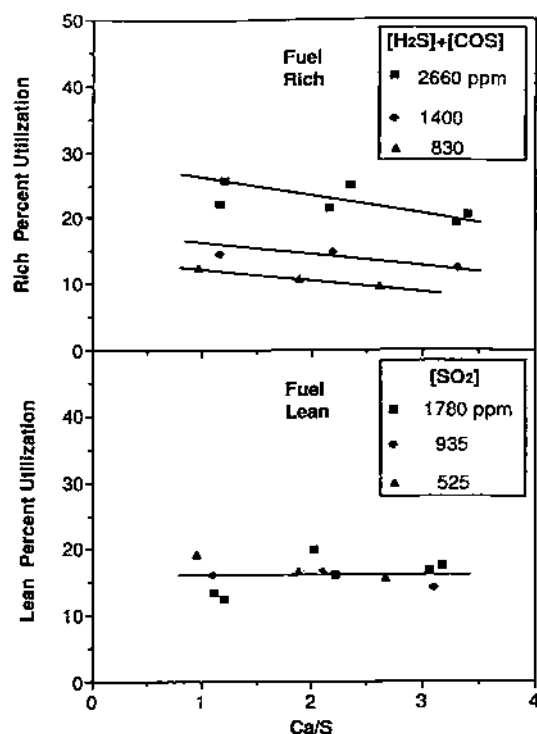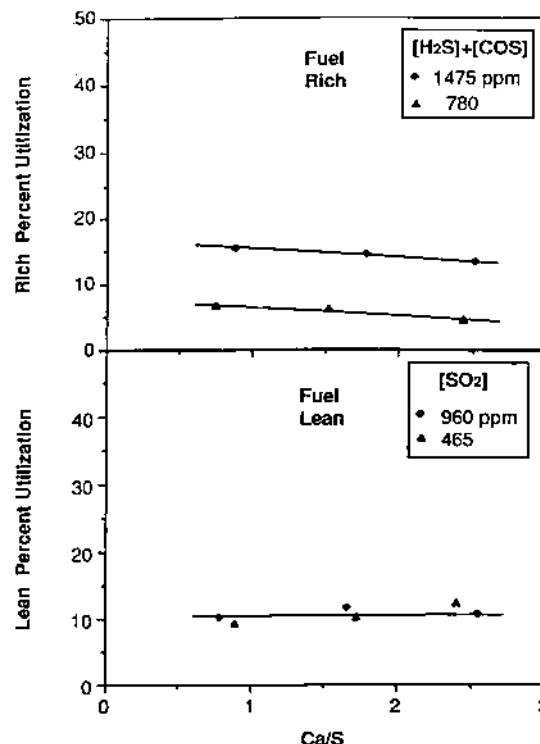
Figure 7. Effect of Ca/S for hydrate at SR = 0.65.
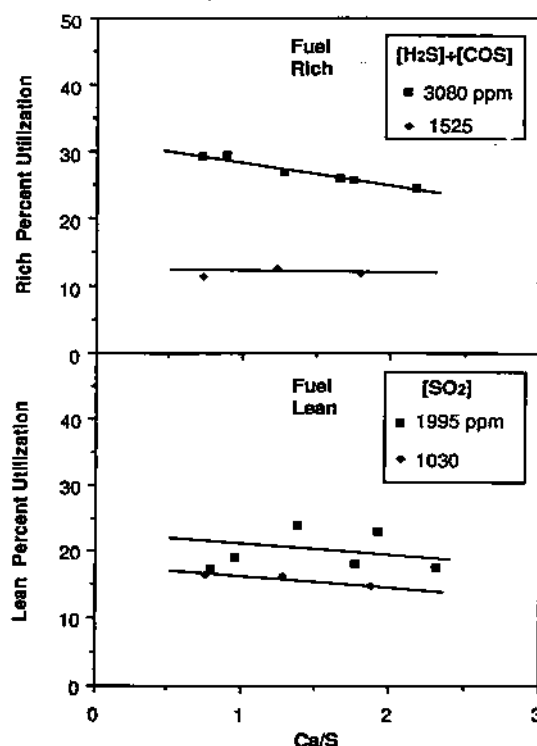

Figure 8. Effect of Ca/S for carbonate at SR = 0.75.

Figure 9. Effect of Ca/S for carbonate at SR = 0.65.

Sulfur Concentration, ppm

Figure 10. Effect of sulfur concentration at Ca/S = 2.

a negative dependence on Ca/S. The negative dependence on Ca/S is consistent with the positive dependence on sulfur concentration. As the Ca/S is increased, the sulfur concentration is reduced to lower levels (which approach equilibrium), slowing the rate of additional calcium utilization. These figures also show that the high utilizations obtained in the rich zone with high sulfur levels are partly lost when the sorbent passes into the lean zone. However, at low sulfur levels the utilization increases when the sorbent passes through the lean zone.

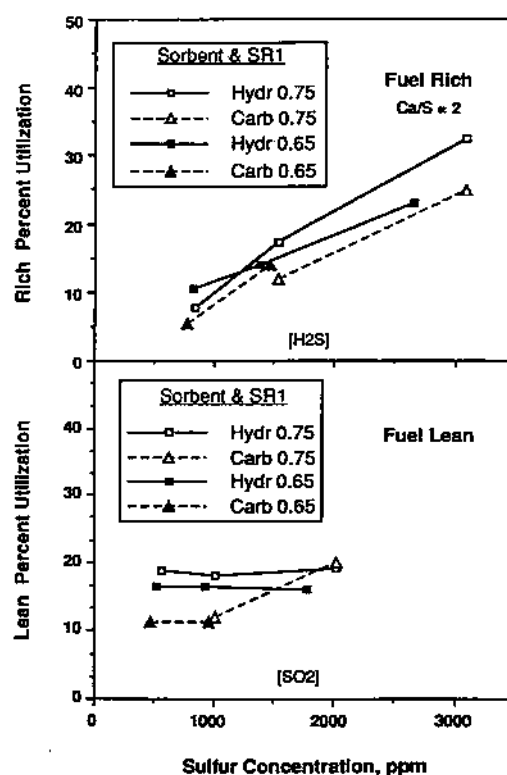The strong influence of H_2S concentration on the rich sulfur capture and the much weaker influence of SO_2 concentration on the lean sulfur capture are well illustrated in Figure 10. Calcium utilization at an average residence time of 0.44 s and Ca/S = 2 is reported as a function of sulfur concentration for both the hydrate (squares) and the carbonate (triangles) sorbents at SR = 0.75 (open symbols) and 0.65 (dark symbols). Fuel rich calcium utilization increases with increasing sulfur concentration. The slight reduction in reaction rate at higher concentrations is expected because, as the product layer thickens, diffusion resistance through the product layer will increase. It appears even higher calcium utilization could be realized
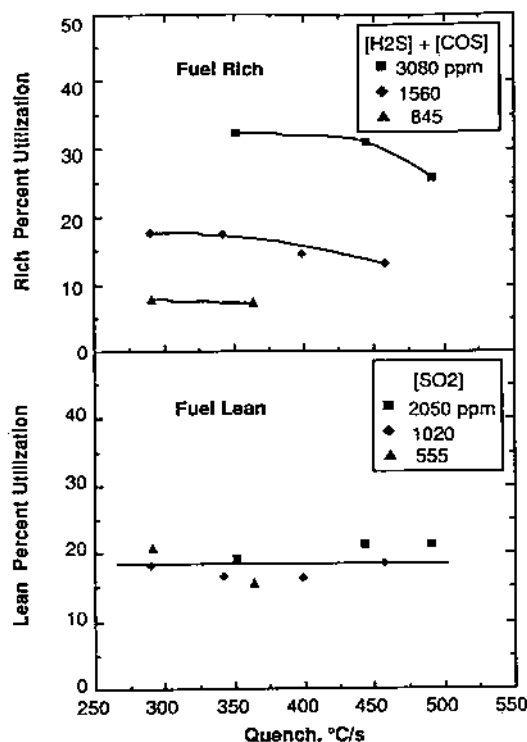
Figure 11. Effect of rich zone quench rate for hydrate at SR = 0.75 and Ca/S = 2.

Figure 12. Effect of rich zone stoichiometric ratio for the hydrate at Ca/S = 2.

at higher sulfur concentrations, indicating that no physical limits to additional utilization seem to exist (at these levels of conversion). At low concentrations, the curves seem to trend to zero utilization at a concentration of ~500 ppm. This corresponds to the equilibrium concentration of H_2S at 1040 °C, which is approximately the average temperature of these runs. With the lower moisture levels obtained with coal combustion, these curves would probably shift to the left so that higher utilization could be obtained at a given sulfur concentration. On the lean side, the utilization is insensitive to the gas-phase sulfur driving force, suggesting the existence of a physical limit on calcium availability.

Figure 10 also shows that the Marblehead hydrate was better than the Fredonia carbonate at capturing sulfur in both the fuel rich and fuel lean zones. This is consistent with the fuel lean sulfur capture results of other investigators (1).

Thermal Effects. The sorbent injection temperature for the SR1 = 0.75 and 0.65 runs was 1100 ± 20 and 1060 ± 15 °C, respectively. The rich zone temperatures were lower for the SR1 = 0.65 runs because of the lower energy input to the rich zone at the lower stoichiometry. The fuel rich capture was found to be insensitive to the injection temperature over the limited range studied at a given stoichiometric ratio; however, the quench rate in the top portion of the rich zone did have an effect when the quench was greater than 360 °C/s. The quench rate varied from 250 to 500 °C/s. Most of the data in this study fall in the 280–390 °C/s range. Figure 11 shows the effect of quench rate on rich calcium utilization for the hydrate at a stoichiometric ratio of 0.75 and Ca/S = 2. As the quench is increased above 360 °C/s, the fuel rich calcium utilization decreases. On the lean side no effect of the rich zone quench was observed.

Effects of Rich Zone Stoichiometry. Figure 12 shows calcium utilization as a function of the fuel rich stoichiometric ratio (SR1) for Marblehead hydrate at three sulfur levels (Ca/S = 2). The data from the rich zone show some
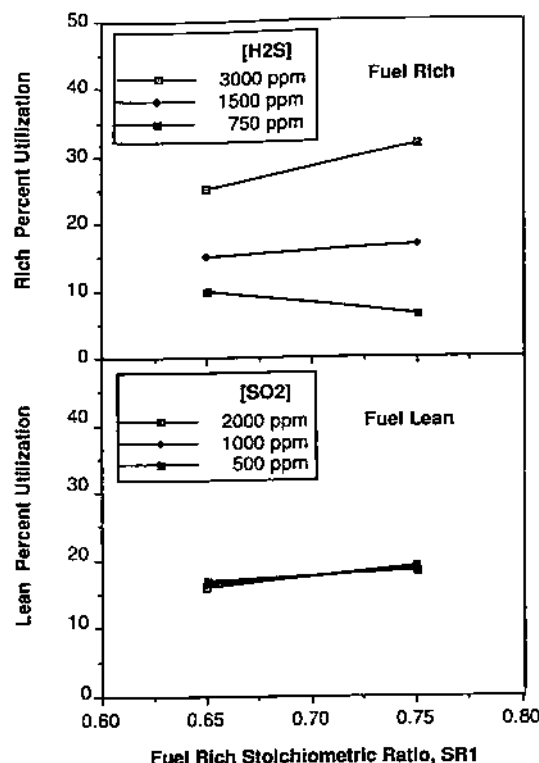
interesting results. At the low H_2S level, sulfur capture was higher at SR = 0.65, while at the high H_2S level, capture was higher at SR = 0.75. The low concentration results can be explained by thermodynamics. The moisture level at SR = 0.65 is over 1% lower than at SR = 0.75 (15.33% and 16.48%, respectively); thus, the equilibrium H_2S level is ~75 ppm lower at SR = 0.65. For the low sulfur level runs, any lowering of the thermodynamic limitation is significant. The high-concentration results can be explained by kinetics. The average sorbent injection temperature was higher for the SR = 0.75 runs (1100 versus 1060 °C at SR = 0.65); thus, faster reaction and diffusion rates and therefore higher capture would be expected at SR = 0.75. At the low sulfur level the increased temperature would not increase capture because of thermodynamic limitations at the higher temperatures. At the 1500 ppm level the two effects seem to counterbalance each other. On the lean side, no effect of the rich zone stoichiometry can be seen and again the lean capture is independent of the level obtained in the rich zone.

Conclusions

Analysis of the fuel rich results shows a strong positive dependence of calcium utilization on sulfur concentration and a negative dependence on Ca/S. The results on the lean side are very typical of lean capture data reported by others (1); i.e., the calcium utilization is weakly dependent on Ca/S and sulfur concentration. The fuel lean sulfur capture seems to be independent of the level of capture obtained in the fuel rich zone; thus, at high sulfur concentrations, the high capture obtained in the fuel rich zone is partly lost when the sorbent passes into the lean zone. However, at low sulfur levels the utilization increases when the sorbent passes through the lean zone.

Thermodynamics plays an important role in fuel rich sulfur capture because the gaseous products of the sulfur capture reactions, H_2O and CO_2, are also major products of combustion. This means that, for a given level of H_2S, higher sulfur capture is possible in coal combustion than

in natural gas combustion because the moisture level is lower. Anything that reduces the moisture level, such as lowering the stoichiometric ratio or predrying the coal, will help to increase fuel rich sulfur capture, especially at lower sulfur levels. Increasing temperature will increase fuel rich sulfur capture only if the sulfur level is significantly above the thermodynamic limit (which increases with temperature).

These results suggest that the rate-limiting step under fuel rich conditions is distinctly different than under fuel lean conditions. Theoretical calculations using a distributed pore model (19) have suggested that the relative insensitivity of calcium utilization to SO_2 concentration under fuel lean conditions is a result of pore closure effects; as the SO_2 concentration is increased, the kinetic and diffusion rates increase but this further aggravates the problem of pore closure and results in essentially no overall increase in sulfur capture. The increased calcium utilization measured with increasing sulfur species concentrations under fuel rich conditions may be the direct result of enhanced product layer diffusion; pore closure would not be expected to limit calcium utilization under fuel rich conditions because the molar volume of calcium sulfide is less than either parent sorbent.

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