

Bench-Scale Evaluation of Calcium Sorbents for Acid Gas Emission Control

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Calcium sorbents for acid gas emission control were evaluated for effectiveness in removing SO₂/HCl and SO₂/NO from simulated incinerator and boiler flue gases. All tests were conducted in a bench-scale reactor (fixed-bed) simulating fabric filter conditions in an acid gas removal process. Reagent grade Ca(OH)₂ was used to establish baseline sorbent performance. The reactivity of reagent grade Ca(OH)₂ with HCl from SO₂/HCl mixtures gradually increased with decreasing approach to saturation temperature. SO₂ reactivity toward Ca(OH)₂ was very sensitive to approach to saturation. Novel calcium silicate sorbents were tested for reactivity with both SO₂ and HCl. A "thermal window" for optimum NO removal was found at 90°C (194°F) when Ca(OH)₂ was used at SO₂/NO ratios of 1:1. Reactivity of Ca(OH)₂ toward SO₂ from SO₂/NO mixtures was very sensitive to approach to saturation, while reactivity with NO was insensitive. Several additives were subsequently tested to determine optimum sorbent combinations for SO₂/NO control. To date the most promising additives are Mg(OH)₂ and Na₂HPO₄ at 10 mol percent concentrations. As with SO₂/HCl, calcium silicate has been shown to be superior for SO₂/NO capture. Some implications for larger-scale process configurations and sorbent selection for HCl/SO₂/NO control are discussed. Future activities and limited larger-scale pilot plant results are also discussed.

INTRODUCTION

The widespread recognition of municipal solid waste incineration (MSWI) as a means of disposing of refuse prompted the interest in the control of acid gas emissions into the atmosphere. Both wet and dry processes are considered for the removal of acid gases such as HCl, SO₂, and NO. Wet scrubbers (with preceding ESP) are particularly favored for trace heavy metal control in addition to acid gas control [1]. Wet scrubbers can achieve 95 percent removal of HCl and 90 percent removal of SO₂ and can be designed for NO_x control [8]. However, they are costly and technically complex compared to dry systems. Spray drying, using a fabric filter for particulate collection, is particularly widespread.

In this process, dry powdered sorbent is injected into the duct containing flue gas that is humidified to decrease the approach to saturation temperature. The concentration of acid gases is reduced by chemical reaction occurring during evaporative cooling—achieved by the evaporation of a finely atomized Ca(OH)₂ slurry into hot flue gas. Compared with spray drying, dry sorbent injection into the duct offers the possibility of an even more economical process for the cleanup of MSWI flue gas.

The approach to saturation is known to be a very important parameter governing the reactivity of sorbent during dry sorbent duct injection. Since the dry injection offers only limited sorbent utilization, a fabric filter is commonly used as a particulate collector to allow for additional reaction of acid gas with sorbent deposited on the surface of the filter. Effective acid gas control could be achieved in such a way as in tests previously conducted by Environment Canada [10]. Post-combustion control of NO_x is generally perceived as the most costly of the three acid gases mentioned above.

The objective of this study was to investigate at the bench-scale the feasibility of dry injection processes for the removal of HCl, SO₂, and NO. A fixed-bed reactor was used to evaluate potential calcitic sorbents for the control of acid gas emissions. The approach to saturation between 10°C and 40°C (18°F and 72°F) was investigated, being representative of operational conditions encountered during the duct injection process. Based on the findings of the bench-scale experiments as well as on limited pilot-scale results discussed here, some recommendations were given concerning the operation of dry sorbent injection systems for acid gas emission control.

EXPERIMENTAL

Apparatus

The core of the experimental apparatus, shown in Figure 1, was the sand-bed reactor, where the test gas with the desired SO_2/HCl or SO_2/NO concentration reacted with the alkaline sorbent. This type of reactor has been successfully used in past screening of sorbents for SO_2 removal [4], [5]. A similar type of fixed-bed reactor system was employed for the study of adsorption of HCl on solid slaked lime [6].

The reactor was a vertical packed bed of 1 g sorbent dispersed in 40 g of inert silica sand. The entire bed was supported on a 45 mm diameter fritted glass plate in a glass cylinder. The simulated flue gas passed down through the sand-bed at a face velocity of 6 cm/s. Commercially available calibrated span gases of SO_2 in nitrogen or NO in nitrogen were used as the test gases. The desired concentration of HCl in the flue gas was obtained by mixing pure HCl with the span gas stream.

For SO_2/HCl removal testing, 500 ppm SO_2 and 1,000 ppm HCl concentrations were used; whereas, for SO_2/NO , nominal concentrations were 400 ppm of both SO_2 and NO . The SO_2/NO gas stream also contained 5 percent by volume of O_2 obtained by mixing SO_2 and NO span gases with air. The nitrogen purging system was incorporated to address the problems associated with the high water affinity of HCl and the resulting corrosion risks.

Two electrically heated reactor systems were used interchangeably during testing. The operating temperature range was 54°C – 177°C (130°F – 350°F) for SO_2/HCl testing. Initial baseline testing of SO_2/NO removal was done in the electrically heated reactor. Following sorbent screening, SO_2/NO was removed in the reactor heated by the water bath operated at 90°C (194°F).

In order to test the effect of moisture in the flue gas, water was continuously injected into the evaporation chamber by a syringe type dispensing pump, equipped with two syringes to allow continuous operation with a transient-free switchover period. Steady evaporation was achieved in an electrically heated sand reservoir, from which water vapor was injected into the process gas. The moisture was removed from the gas by the condenser/ice-cooled trap and analyzed for chloride contents. The SO_2 and NO concentration in the outlet gas was continuously monitored by Thermolectron Model 40 pulsed fluorescence analyzer and Thermolectron Model 10A chemiluminescence analyzer working in NO_x mode, re-

spectively. A 1 h period was arbitrarily selected for an experimental run as being representative of a typical fabric-filter cleaning cycle.

Analytical Procedures

The removal of SO_2 and NO was determined by integration of the 1 h concentration curve recorded by the analyzer. The removal of HCl by the sorbent in the sand-bed reactor was determined by washing the bed and analyzing the filtrate by ion chromatography for chloride. After completing the run, the reactor was cooled and the sorbent containing sand was removed to a beaker. The reactor was washed with distilled water, and the rinse, together with an additional amount of water, was poured into the beaker containing sand. The contents of the beaker were stirred at room temperature for 1 h. The sample was filtered, diluted, and analyzed by ion chromatography. The amount of chloride in the reactor wash and in the condensate was compared against the amount of HCl injected into the span gas, as shown by a flowmeter. The mass balance of HCl could usually be closed to within ± 10 percent. The reactivity of sorbent is described here as the ratio of millimoles of acid-gas pollutant removed after 1 h contacting in the sand-bed reactor per 100 millimoles of $\text{Ca}(\text{OH})_2$.

Sorbents

Reagent grade $\text{Ca}(\text{OH})_2$ (Fisher C-97) was selected to establish the baseline reactivity of lime with HCl , SO_2 , and NO . The B.E.T. specific surface area measured by nitrogen adsorption was $12.4 \text{ m}^2/\text{g}$ for this sorbent. Several additives were tested to promote the removal of NO from SO_2/NO gas. Also tested were novel silicate sorbents, described earlier as being capable of superior reactivity with SO_2 compared to dry hydrated lime when contacted under dry sorbent duct injection conditions [5]. These sorbents were produced by slurring flyash and $\text{Ca}(\text{OH})_2$, in ratios varying from 3:1 to 10:1 at 90°C (194°F) for 8 h. The above conditions were shown to produce sorbents having significantly higher B.E.T. specific surface areas and up to about 3 times the reactivity of reagent grade $\text{Ca}(\text{OH})_2$ with SO_2 [5].

RESULTS AND DISCUSSION

Reactivity with SO_2 and HCl

The first series of bench-scale experiments were directed toward establishing the baseline reactivity of reagent grade $\text{Ca}(\text{OH})_2$ as a function of the approach to saturation temperature (relative humidity) in the reactor. The removal of HCl from HCl/N_2 gas was tested first: the results are shown in Figure 2. For these tests the HCl concentration was 1,000 ppm and the absolute humidity was constant at $0.1 \text{ g H}_2\text{O/g}$ of dry gas; the temperature of the gas varied from 66 to 177°C (150 to 350°F). Relative humidity (controlled by temperature) had a positive effect on the reactivity of reagent grade lime with HCl , with reactivity increasing with the increasing relative humidity in the reactor (decreasing approach to the adiabatic saturation temperature). Under the experimental conditions (1 g of $\text{Ca}(\text{OH})_2$ and $1,000 \text{ ppm HCl}$), the reactivity of 90 percent removal of HCl from the simulated flue gas. Based on these experimental results, 30°C (54°F) approach to saturation or less would be recommended to achieve at least 80 percent removal of HCl from the flue gas.

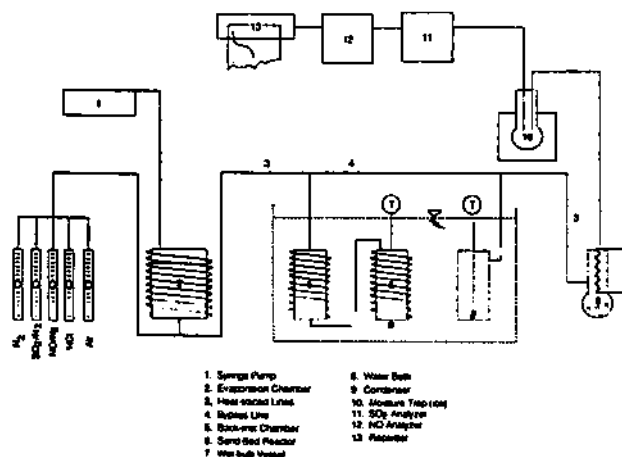


Figure 1. Experimental apparatus.

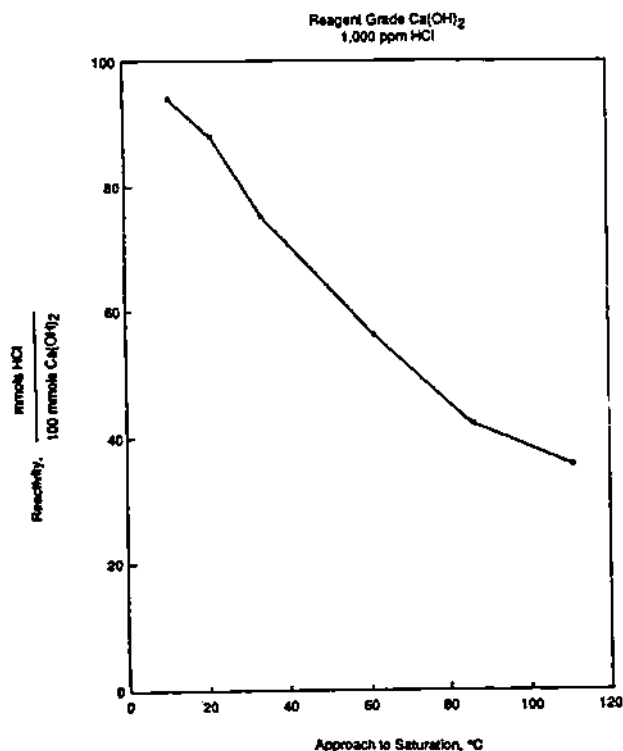


Figure 2. The effect of the approach to saturation on reactivity of $\text{Ca}(\text{OH})_2$ with HCl.

Because typical MSWI flue gas would contain SO_2 in addition to HCl, several tests were performed to evaluate the effect of the approach to saturation (relative humidity) on the reactivity of reagent grade lime with SO_2 alone.

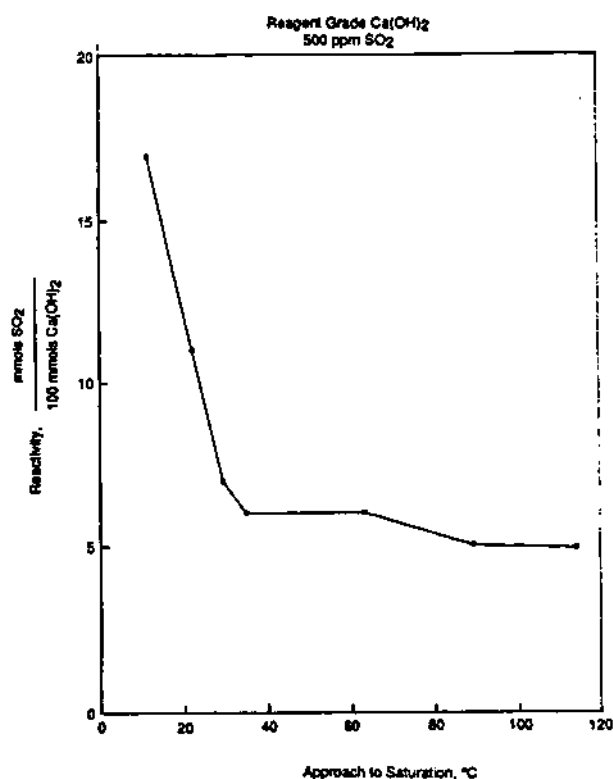


Figure 3. The effect of the approach to saturation on reactivity of $\text{Ca}(\text{OH})_2$ with SO_2 .

The results are presented in Figure 3. The approach to saturation was affected by varying the temperature from 66°C to 177°C (150°F to 350°F). Two reaction regimes could be defined based on findings presented in Figure 3. For the approach to saturation greater than about 35°C (63°F), reactivity with SO_2 was low, revealed little sensitivity toward relative humidity, and ranged from 4.9 to 6.2 mmol SO_2 /100 mmol $\text{Ca}(\text{OH})_2$. The reactivity was very sensitive toward relative humidity for the approach to saturation of less than 35°C (63°F) and was increasing with the decreasing approach to saturation temperature, to reach an experimental maximum of 17 mmol SO_2 /100 mmol $\text{Ca}(\text{OH})_2$ at 10°C (18°F) approach.

For the conditions used in the above experiments, 35°C (63°F) approach would correspond to about 18 percent relative humidity. According to data on equilibrium water vapor adsorption, about 1.2 molecular layers of water would adsorb on lime at 18 percent relative humidity [7]. Results of this study agree quite well, therefore, with others indicating that significant SO_2 capture by dry $\text{Ca}(\text{OH})_2$ requires at least a 1.0 molecular layer of adsorbed water on lime surfaces [9].

The reactivity of reagent grade $\text{Ca}(\text{OH})_2$ with combined SO_2 /HCl was also tested in the sand-bed reactor. The results of experiments when 1,000 ppm HCl and 500 ppm SO_2 were simultaneously present in the flue gas are shown in Figure 4. For comparison, the results for HCl or SO_2 alone are presented with broken lines in Figure 4. As was the case when only one acid gas was present in the flue gas, sorbent reactivity when HCl and SO_2 were present increased with decreasing approach to adiabatic saturation. The reactivity with HCl absorbed from flue gas containing HCl and SO_2 was essentially unchanged, compared to reactivity with HCl alone. The reactivity with SO_2 was somewhat enhanced by the simultaneous presence of HCl in the flue gas for approach to saturation of less than 60°C (108°F).

It is possible that the deliquescent product of the $\text{Ca}(\text{OH})_2$ /HCl reaction might have subsequently promoted reactivity of $\text{Ca}(\text{OH})_2$ with SO_2 . For the conditions tested, it appears that the reactivity of lime with HCl is about one order of magnitude higher than that with SO_2 .

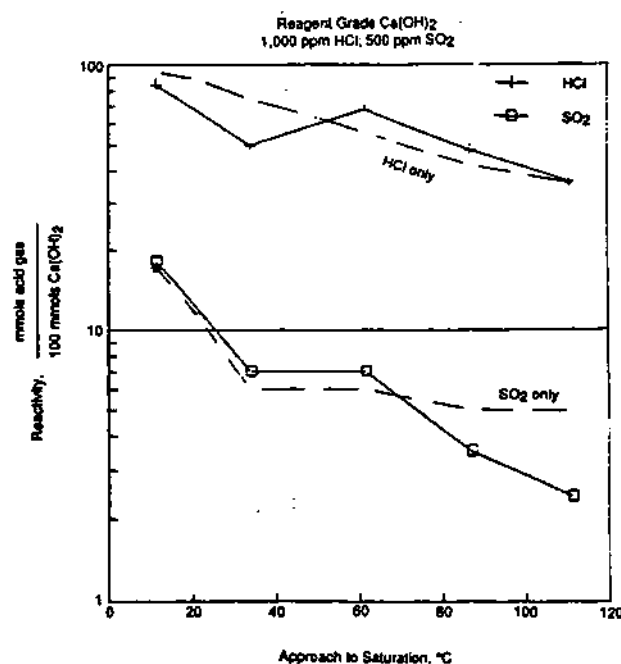


Figure 4. The effect of the approach to saturation on combined reactivity of $\text{Ca}(\text{OH})_2$ with SO_2 and HCl.

The reactivity with HCl was significantly less sensitive to the approach to saturation than with SO₂. For example, decreasing the approach to saturation from 35°C to 12°C (63°F to 22°F) increased the reactivity with HCl by 33 percent from 60 to 80 mmols HCl/100 mmols Ca(OH)₂. The same change increased the reactivity with SO₂ by 183 percent from 7 to 17 mmols SO₂/100 mmols Ca(OH)₂.

Previous work [5] has demonstrated that silica-enhanced Ca(OH)₂ was significantly more reactive toward SO₂ than Ca(OH)₂ alone. For fixed sorbent preparation conditions (90°C for 8h), the reactivity increased with the increasing weight ratio of flyash to lime [5]. Sorbents were prepared for this study using reagent grade Ca(OH)₂ and low calcium flyash from the Clinch River Plant in Carbo, VA. Tested weight ratios of flyash to Ca(OH)₂ were 10:1, 5:1, and 3:1, and the approach to saturation was varied from 20°C to 40°C (36°F to 72°F). The results are presented in Figure 5, which gives the reactivity of silica-enhanced sorbents as a function of the approach to saturation. Baseline results for reagent grade Ca(OH)₂ exposed to 1,000 ppm HCl and 500 ppm SO₂ are shown by the broken lines.

Only moderate enhancement of reactivity was observed with a flyash-to-Ca(OH)₂ weight ratio of 10:1. No enhancement was measured for the 5:1 sorbent, and 3:1 sorbent revealed lower reactivity than reagent grade Ca(OH)₂. This is in contrast to earlier findings about the reactivity of silica-enhanced sorbents for removal of SO₂ alone [5]. Apparently, the reaction product with HCl blinds the surface and precludes active sites from reacting with SO₂.

One way that blinding might have occurred was the combination of the surface moisture of the silicate sorbent with CaCl₂ reaction product. Bound moisture would probably not be available for the reaction with SO₂. The ability of silicate sorbent to release surface moisture to make it available during the reaction of Ca(OH)₂ with SO₂ is believed (aside from its high specific surface area) to be the reason for its greatly enhanced reactivity with SO₂ compared to Ca(OH)₂. Silica-enhanced sorbents captured more HCl per unit mass of Ca(OH)₂ than did reagent grade Ca(OH)₂. For example, at 40°C (72°F) approach to saturation, the reactivity of reagent grade Ca(OH)₂ was about 70 mmols HCl/100 mmols Ca(OH)₂ when exposed to 1,000 ppm HCl and 500 ppm SO₂. Sil-

ica-enhanced sorbents revealed an average reactivity of about 110 mmols HCl/100 mmols Ca(OH)₂ after exposure to acid gas pollutants at the same conditions in the sand-bed reactor. There was little dependence of calcium silicate reactivity with HCl on the flyash-to-Ca(OH)₂ weight ratio. Reactivity varied from 107 to 112 mmols HCl/100 mmols Ca(OH)₂ for the 10:1 to 3:1 weight ratio range and 40°C (72°F) approach to saturation. For the same conditions, the reactivity with SO₂ varied from 4.6 to 9.5 mmols SO₂/100 mmols Ca(OH)₂ for the flyash-to-Ca(OH)₂ weight ratio range from 3:1 to 10:1.

Reactivity with SO₂ and NO

Because NO is a major acid gas pollutant in MSWI flue gas, effort was directed to investigate potential sorbents for its removal under duct injection conditions. As in the approach discussed earlier for testing the reactivity of sorbents with HCl and SO₂, reagent grade Ca(OH)₂ was tested first. Temperature in the reactor was varied from 70°C to 180°C (158°F to 356°F), and absolute humidity was constant at 0.1g H₂O/g dry gas. The concentration of NO was 400 ppm, and the ratio of SO₂ to NO—known to affect the removal of NO from the flue gas [2]—was 1:1.

The simulated flue gas contained 5 volume percent of oxygen (initial experiments indicated no removal of NO without oxygen present in the flue gas). The results are shown in Figure 6, giving the reactivity as a function of the temperature/approach to saturation in the reactor. The highest reactivity of 0.8 mmol NO/100 mmol Ca(OH)₂ was achieved at 177°C (350°F). However, with these conditions of temperature/relative humidity, only low reactivity with SO₂ (5 mmols SO₂/100 mmols Ca(OH)₂) would be expected for simultaneous removal of SO₂/NO. The temperature of 90°C (194°F), for which somewhat higher reactivity with SO₂ was measured, seems, therefore, to be the optimum for the simultaneous removal of SO₂/NO, based on the available data presented in Figure 6. This temperature agrees with the previously reported so-called thermal window within 87°C to 107°C (189°F to 225°F) where SO₂ and NO can react simultaneously [3].

Also shown in Figure 6 is the reactivity of Ca(OH)₂ with SO₂ for simultaneous removal of SO₂ and NO. It was

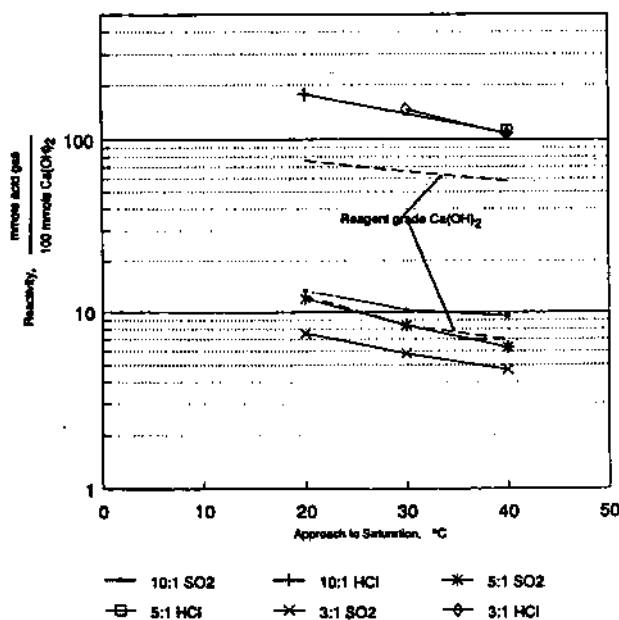


Figure 5. The reactivity of silica-enhanced sorbents with SO₂ and HCl.

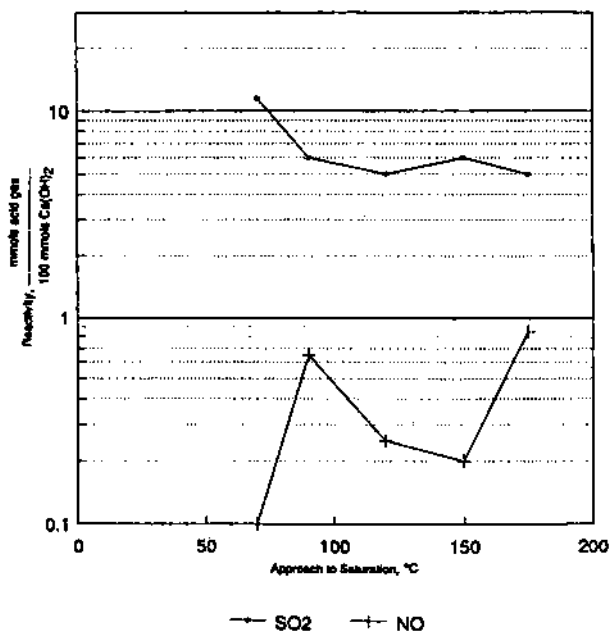


Figure 6. The temperature dependence of Ca(OH)₂ reactivity with SO₂ and NO.

a strong function of the approach to saturation (relative humidity) in the reactor, with reactivity increasing significantly for the approach to saturation less than 35°C (63°F) (relative humidity greater than 18 percent). The reactivity of $\text{Ca}(\text{OH})_2$ with NO was not a clear function of the approach to saturation. However, it has been reported [2], [3] that moisture in the flue gas is necessary for NO removal.

Another factor that has been recognized as affecting the reactivity of calcitic sorbents with NO is the SO_2/NO ratio. This has been confirmed during this study. When a mixture of NO/N_2 and 5 percent oxygen was contacted with $\text{Ca}(\text{OH})_2$, only 0.12 mmol NO/100 mmol $\text{Ca}(\text{OH})_2$ reactivity was measured at 90°C 35°C (63°F) approach to saturation. The reactivity was 0.65 mmol NO/100 mmol $\text{Ca}(\text{OH})_2$ for SO_2/NO ratio of 1:1, with other parameters unaffected. The comparison of data presented in Figure 6 with those discussed in Figure 4 indicates that the reactivity of calcitic sorbents with acid gases follows the descending order $\text{HCl} > \text{SO}_2 > \text{NO}$. $\text{Ca}(\text{OH})_2$ was measured to be about one order of magnitude less reactive with NO than it was with SO_2 .

In the next step, several additives were tested with the goal of enhancing reactivity of $\text{Ca}(\text{OH})_2$ with NO. Each additive was used at the 10 mol percent concentration level induced by slurrying $\text{Ca}(\text{OH})_2$ with a given additive for 10 min and oven drying the resulting 50 percent solids slurry overnight. The conditions in the sand-bed reactor were 400 ppm SO_2 , 400 ppm NO, 90°C (194°F) temperature, and 35°C (63°F) approach to saturation. The results are shown in Figure 7. The most promising additives selected so far were $\text{Mg}(\text{OH})_2$ and NaHPO_4 . A certain increase of reactivity with SO_2 was also observed for these two additives. More bench-scale and pilot-scale testing are needed to fully evaluate the effectiveness of additives on reactivity of $\text{Ca}(\text{OH})_2$ with NO.

Another sorbent tested for simultaneous removal of SO_2 and NO was silicate. It has been demonstrated that silicate sorbents are more reactive toward NO than reagent grade $\text{Ca}(\text{OH})_2$ when prepared with 10 mol percent NaOH [2]. The sorbents were prepared with flyash-to- $\text{Ca}(\text{OH})_2$ weight ratios of 10:1 and 3:1. Both had 10 mol

percent NaOH added [based on $\text{Ca}(\text{OH})_2$] and were slurried at 90°C (194°F) for 8h. The results of testing are presented in Figure 8 for approaches to saturation ranging from 10°C to 30°C (18°F to 54°F) and 400 ppm NO with a SO_2/NO ratio of 1:1. For a given approach to saturation, the reactivity with both SO_2 and NO was increased with increasing flyash-to- $\text{Ca}(\text{OH})_2$ weight ratio of the sorbent. Similar to baseline results discussed earlier, reactivity with NO for any given sorbent increased, and reactivity with SO_2 decreased for the increasing approach to saturation. At 30°C (54°F) approach to saturation, the reactivity of sorbent prepared by slurrying flyash with $\text{Ca}(\text{OH})_2$ at a 3:1 weight ratio was significantly higher for both SO_2 and NO than the reactivity measured for the reagent grade $\text{Ca}(\text{OH})_2$.

On the commercial scale, silicate sorbents with high flyash-to- $\text{Ca}(\text{OH})_2$ weight ratio would be achieved by increasing the recycle of flyash and spent sorbent. Calcium sulfite (CaSO_3) would also be present when recycling spent sorbent. Several experiments were performed to evaluate the effect of CaSO_3 alone on the reactivity of $\text{Ca}(\text{OH})_2$ with NO. When CaSO_3 was slurried with $\text{Ca}(\text{OH})_2$ at a 4:1 weight ratio (no flyash), oven dried, and exposed to 400 ppm NO/400 ppm SO_2 , flue gas in the sand-bed reactor operated at 35°C (63°F) approach to saturation, the reactivity was 1.52 mmol NO/100 mmol $\text{Ca}(\text{OH})_2$ and 12.41 mmol SO_2 /100 mmol $\text{Ca}(\text{OH})_2$ (compared to 0.65 mmol NO and 6.0 mmol SO_2 /100 mmol $\text{Ca}(\text{OH})_2$ for reagent grade).

Reactivity of only 1.09 mmol NO/100 mmol $\text{Ca}(\text{OH})_2$ was measured when the same sorbent was exposed to gas containing only 400 ppm NO (no SO_2). It seems, therefore, that there is some other effect besides formation of CaSO_3 on the surface of $\text{Ca}(\text{OH})_2$ prior to reactivity with NO. Possibly some catalytic effect of the combined presence of SO_2 and O_2 facilitates oxidation of NO. To better simulate commercial systems flyash, $\text{Ca}(\text{OH})_2$, and CaSO_3 were slurried at a 4:1:4 weight ratio, with 0.1 mole NaOH per mole $\text{Ca}(\text{OH})_2$ added. The resulting reactivity of this sorbent with NO increased to 2.21 mmol NO/100 mmol $\text{Ca}(\text{OH})_2$. Ultimately, 10 mol percent of $\text{Mg}(\text{OH})_2$ was added to the above flyash/ $\text{Ca}(\text{OH})_2$ / CaSO_3 /NaOH sorbent, and the reactivity with NO was 2.71 mmol NO/100 mmol $\text{Ca}(\text{OH})_2$.

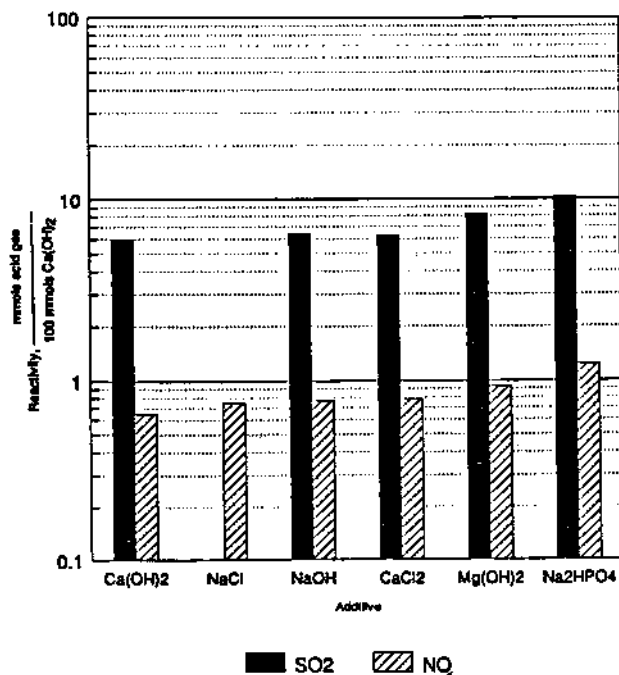


Figure 7. Screening of additives for the enhancement of $\text{Ca}(\text{OH})_2$ reactivity with SO_2 and NO.

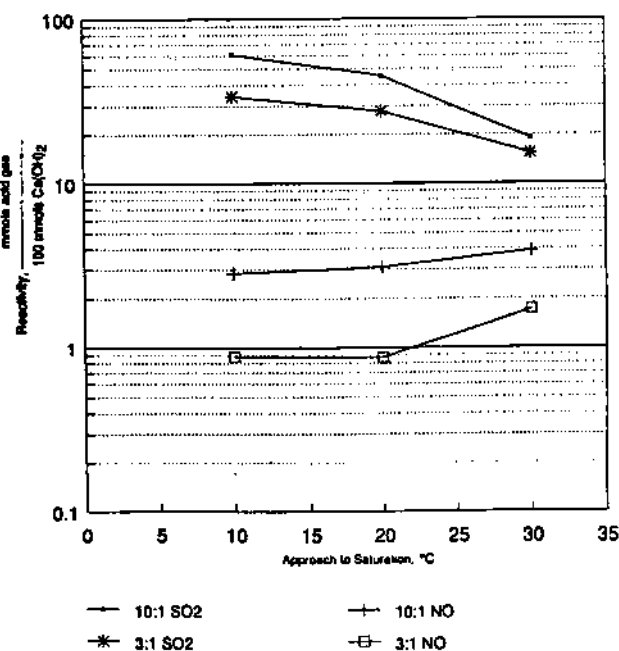


Figure 8. The reactivity of silica-enhanced sorbents with SO_2 and NO.

CONCLUSIONS AND RECOMMENDATIONS

The following conclusions and recommendations are based on the results of this study.

- The reactivity of calcitic sorbents with the three acid gases investigated during this work could be arranged in decreasing order as $\text{HCl} > \text{SO}_2 > \text{NO}$. The reactivity with NO would be about 2 orders of magnitude lower than with HCl.
- SO_2 should be removed from the SO_2/HCl or $\text{SO}_2/\text{HCl}/\text{NO}$ flue gas at an approach to saturation of 35°C (63°F) or lower to ensure the buildup of at least 1.0 monolayer of molecular water on the surface of the sorbent (relative humidity of about 20 percent).
- High reactivities can be achieved with HCl for almost all conditions tested, and this pollutant should be the easiest to remove of the three tested.
- Low removals of NO were measured across the sand-bed reactor (generally below 10 percent). The reactivity with NO was not proportional to the relative humidity in the reactor. Optimum temperature for the removal of NO was found to be 90°C (194°F).
- Calcium silicate sorbents were more reactive than dry hydrated lime for the removal of acid gas pollutants. The greatest enhancement was observed for reactivity with SO_2 and NO. Severe blinding of active sites, combined with blinding of surface moisture normally available for the reaction, occurred as a result of reaction with HCl.
- Silicate sorbents were more reactive with HCl than reagent grade $\text{Ca}(\text{OH})_2$. However, the enhancement of reactivity with SO_2 (simultaneous HCl/SO_2 removal) was measured only for flyash-to- $\text{Ca}(\text{OH})_2$ weight ratio of 10:1.
- Several additives were evaluated for the enhancement of reactivity with NO. The two most promising were $\text{Mg}(\text{OH})_2$ and Na_2HPO_4 . The former seems to be more attractive commercially because of its lower price and the potential for using dolomitic lime for calcium silicate preparation. More work is needed to fully evaluate the effect of additives on reactivity of $\text{Ca}(\text{OH})_2$ with NO.

- CaSO_3 and NaOH were found to promote the reactivity of $\text{Ca}(\text{OH})_2$ and calcium silicates with NO. CaSO_3 did not significantly affect reactivity with SO_2 .
- O_2 and SO_2 were necessary for NO removal. Reactivity with SO_2 was essentially unaffected by NO.

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