

CARBON DIOXIDE CAPTURE FROM FLUE GAS USING DRY REGENERABLE SORBENTS

QUARTERLY TECHNICAL PROGRESS REPORT

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ABSTRACT

This report describes research conducted between January 1, 2005 and March 31, 2005 on the use of dry regenerable sorbents for removal of carbon dioxide from flue gas. Engineered sorbents composed of sodium carbonate on a ceramic support were tested in a laboratory fluidized bed reactor system and found to be capable of essentially complete removal of carbon dioxide at 60°C in a short residence time. Upon breakthrough the sorbents can be thermally regenerated to recover essentially all of the absorbed carbon dioxide. An optimized supported sorbent tested in a pilot-scale entrained bed absorber retained its reactivity in multicycle tests and experienced no attrition. Removal of >90% of carbon dioxide in simulated flue gas was achieved in an entrained bed reactor.

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1.0 EXECUTIVE SUMMARY

The objective of this project is to develop a simple and inexpensive process to separate CO₂ as an essentially pure stream from a fossil fuel combustion system using a regenerable sorbent. The sorbents being investigated in this project are alkali carbonates, and particularly sodium carbonate, which is converted to bicarbonate or to an intermediate salt through reaction with carbon dioxide and water vapor. The sorbent is regenerated to carbonate when heated, producing a nearly pure CO₂ stream after condensation of water vapor.

Laboratory fluidized bed testing of supported sorbents composed of 10-15% sodium carbonate on a ceramic support showed that these materials can achieve significant carbon dioxide removal until they reach their saturation capacity. These sorbents release essentially all of the CO₂ absorbed when heated to 150 to 180°C. The “inert” support is responsible for a relatively small fraction of the absorption capacity.

Testing of an optimized sorbent in a pilot-scale entrained bed reactor system was successful in that the sorbent was demonstrated to retain its activity over multiple cycles in simulated flue gas. Regeneration of the sorbent was conducted in fluidized bed mode in nitrogen. Essentially complete thermal regeneration was successfully demonstrated.

2.0 INTRODUCTION

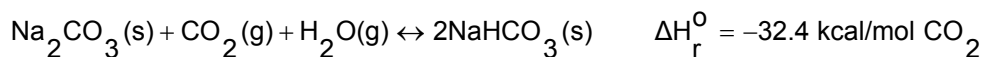
Fossil fuels used for power generation, transportation, and by non-utility sectors are the primary sources of anthropogenic CO₂ emissions. While there are many potential approaches to limiting greenhouse gas emissions, including increased energy efficiency and use of carbon-free fuels, it is becoming increasingly clear that CO₂ capture and sequestration will play an important role in mitigating the progress of global warming. In the near future, CO₂ capture efforts will likely focus on large stationary sources, such as fossil fueled power plants, since they will offer the benefits of economy of scale and the greatest benefits of carbon dioxide reduction from any one industry. It is for this reason that the United States Department of Energy's Carbon Sequestration Program administered by the Office of Fossil Energy and managed by the National Energy Technology Laboratory conducts and funds research to develop CO₂ capture and sequestration technologies should there be a need. Currently there are other drivers, such as market forces and the international sector, that are exerting a research pull for the development of these capture and separation technologies. These initial drivers present opportunities for this technology to enjoy a first major advantage over other emerging technologies.

The focus of this project is to develop a simple and inexpensive process to initially remove CO₂ from the flue gas of existing power plants using a dry, regenerable sorbent. This capture technology is based on the reversible reaction between sodium carbonate, carbon dioxide and water vapor, to form sodium bicarbonate (and/or Wegscheider's salt). Using a thermal swing cyclic process, an essentially pure carbon dioxide stream can be removed from flue gas for subsequent sequestration or reuse. This process is suitable for coal-fired power plants incorporating wet flue gas desulfurization, natural gas-fired power plants, and can be retrofitted

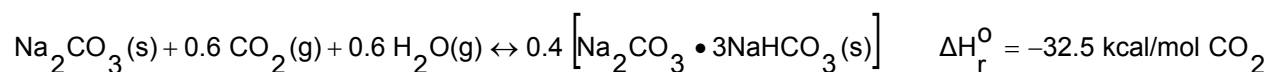
to existing plants. Process modeling suggests that a process of this type offers a lower energy requirement than existing amine-based capture processes.

Capture of CO₂ using sodium carbonate (Na₂CO₃) sorbents results in the reversible formation of sodium bicarbonate (NaHCO₃) and/or Wegscheider's salt (Na₂CO₃•3NaHCO₃):

Equation 1



Equation 2



Both forward reactions (CO₂ absorptions) are exothermic. The equivalent reverse reactions (sorbent regeneration) are endothermic and produce equal molar quantities of CO₂ and H₂O. Condensation of H₂O from the regeneration product results in a pure CO₂ stream suitable for sequestration or reuse.

This report describes activities conducted between January 1, 2005 and March 31, 2005 by RTI. Activities conducted this quarter include pilot-scale entrained bed testing and bench scale fluidized bed testing.

3.0 EXPERIMENTAL

3.1 Entrained Bed Testing

Pilot-scale, entrained-bed testing of an optimized sorbent was conducted at the CANMET Energy Technology Centre in Ottawa, Ontario. CANMET, part of Natural Resources Canada, modified their "mini" circulating fluidized bed combustor to accommodate the testing of RTI's sorbent in both absorption and regeneration mode. A schematic of the modified CANMET system is shown in Figure 1.

CANMET's "single loop" entrained-bed system consists of a 1-m high, 10-cm ID, electrically-heated "fluid bed" section; a 4-m high, 10-cm ID, stainless steel, heat traced and insulated "riser" section; a 80-cm high cyclone; and a 3.2-m high, 5.1-cm ID, "return leg" section. Simulated flue gas enters the fluid-bed section and passes through a distributor plate that evenly distributes the gas to the bed and supports the bed when slumped. Sorbent particles are added to the system through a feed port and enter the entrained bed system at the bottom of the riser. With the proper gas flow rate, the particles become entrained in the flue gas and flow up through the riser section. Instrumentation ports for thermocouples and pressure transmitters are distributed at approximately 30-cm intervals over the height of the riser section. The flue gas (along with the sorbent particles) exits tangentially at the top of the riser into the cyclone.

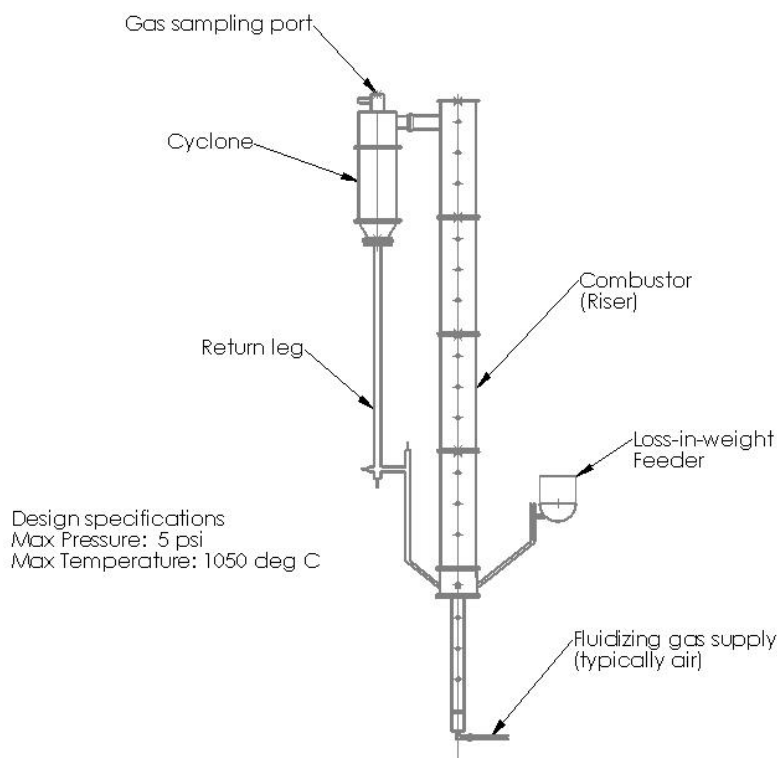


Figure 1. Pilot-scale entrained bed reactor

The cyclone is designed to remove 100% of particles larger than 40 microns from the flue gas. All particles larger than 40 microns (and most above 20 microns) are directed to the return leg section. Only the very fine particles exit with the flue gas and pass through a bag house for fine particle removal. The purpose of the return leg is to transport the captured particles from the cyclone and re-inject them into the riser section. The unit consists of a 2.1-m drop pipe from the bottom of the cyclone to the injection system. The drop pipe contains a diverter valve that allows material to be sampled when required. At the base of the drop pipe the captured solids are blown through a 90° elbow (L-valve) by nitrogen injection into the riser.

All data acquisition signals are sent to an HP3497A data acquisition module for processing. The HP3497A transmits all data to and from a PC running LabView. CO₂ concentrations in the outlet flue gas are monitored using a nondispersive infrared carbon dioxide analyzer. The main gas sampling port is located in the cyclone outlet piping.

The entrained bed testing procedure involved cycling the sorbent between absorption and regeneration. For an absorption test, the system is first stabilized at absorption conditions without the sorbent present. Absorption is conducted at 55°C (± 5°C) with 3 to 10 vol% CO₂ in a flue gas stream saturated with water vapor (balance N₂). The circulation flow rate is usually between 200 – 270 L/min. Once the system is stable at these conditions, roughly 4 to 6 kg of sorbent is introduced to the riser through the solids feed port. Changes in CO₂ concentration, pressure, and temperature are all monitored using LabView. The absorption test was ended when the CO₂ concentration in the outlet flue gas was stable at the inlet flue gas concentration.

The sorbent was then unloaded from the system and weighed. At this point, a sample was taken for particle size analysis using a Sympatec-Helos laser diffraction particle analyzer.

Regeneration was conducted in a fluidized bed mode rather than full circulation. This was done to save time and increase the number of absorption/regeneration cycles. During a regeneration test, the entire system remained at about 55°C (absorption temperature) except for the fluid-bed section (where all of the sorbent is contained when slumped) which was heated to 160 to 180°C. These temperatures were higher than typical regeneration temperatures, because a large amount of ambient temperature sorbent would be added to the fluid-bed section causing the temperature to decrease significantly. The goal was to maintain a regeneration temperature above the absorption temperatures of 60 to 80°C for the entire test. Nitrogen was passed through the fluid-bed section at a flow rate of 100 to 130 L/min. Once the system was stable at these conditions, the carbonated sorbent was introduced into the fluid-bed section through the solids feed port. The sorbent remained fluidized for the length of the regeneration cycle. CO₂ concentration in the outlet gas stream was monitored and the run was ended when the concentration fell back to 0%. The sorbent was again unloaded from the system, weighed, and a sample was taken for particle size analysis.

3.2 Fluidized Bed Testing of Supported Sorbents

Five supported sorbents were subjected to cyclic testing in the RTI 1-inch diameter quartz fluidized bed reactor system. All of the sorbents were composed of sodium carbonate on ceramic supports. Four of the sorbents were produced by a commercial catalyst manufacturer; the remaining sorbent was produced at RTI. This system was operated as described in the previous quarterly report (Green, et al., 2005), with the exception that the positions of the steam and air/nitrogen inlet tubing were changed so that steam enters the reactor above the gas feed. In previous quarters, all tests were run with the steam entered the system below the gas feed.

All absorption cycles were conducted at a nominal temperature of 60°C with a test gas containing 3% water vapor. The sorbent bed was exposed to water vapor prior to the introduction of carbon dioxide in all absorption cycles to eliminate the uncertainty over delays in pumping small volumes of water to the liquid vaporizer (steam generator). All regeneration cycles were conducted in 100% nitrogen.

4.0 RESULTS AND DISCUSSION

4.1 Entrained Bed Testing

The objective of the entrained bed testing was to evaluate the performance of RTI's optimized sorbent in a system that represented the conditions (flow rate, gas-to-solid contact, circulation, etc.) anticipated for a commercial process. The important performance measures are reactivity and attrition resistance over multiple cycles.

4.1.1 Single-cycle test

In a single-cycle absorption test, 5.4 kg of RTI supported sorbent was exposed to a simulated flue gas of 3 vol% CO₂ saturated with water vapor (balance N₂) at a flow rate of 200 L/min and nominal temperature of 60°C. The carbon dioxide removal profile and average riser temperature profile for this run are shown in Figure 2.

Figure 2 shows one of the benefits of conducting the exothermic carbonation reaction in an entrained-bed; the system temperature does not rise enough to extinguish the reaction. The temperature rise over the entire 15 minute test was limited to about 4°C in the riser section, which preserved favorable conditions for the absorption reaction. The support material helped to control the temperature rise by providing a sink for the heat of absorption.

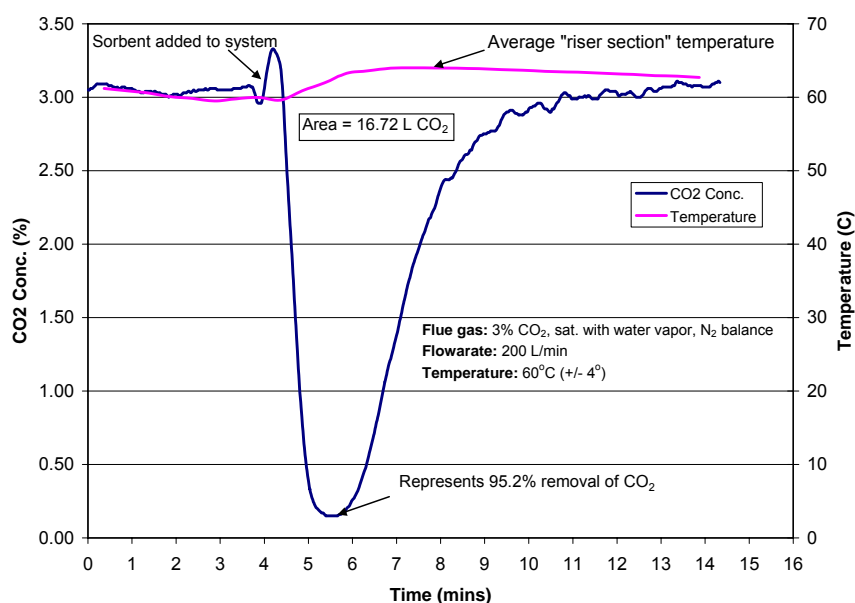


Figure 2. Carbon dioxide concentration of reactor outlet gas during first absorption cycle.

Figure 3 shows the percentage of CO₂ removed from the simulated flue gas at any given time during absorption. Ninety percent removal (and above) is achieved for over 1 minute during this run. These data suggest that the RTI sorbent should be able to achieve 90% removal over the entire residence time (5 to 20 seconds) associated with entrained-bed type systems.

The total amount of CO₂ absorbed (based on integration of the CO₂ removal plot) was calculated to be 16.72 L or roughly 0.75 mols. A quantitative measure of reactivity, based on cumulative CO₂ absorption is shown in Figure 4.

Following the absorption cycle, the sorbent was removed from the system to prepare the fluid-bed section for a regeneration test. The fluid-bed section was heated to approximately 180°C and the “room temperature” sorbent was then fed back into the system. Regeneration was carried out in pure nitrogen at a flow of 130 L/min. The release of CO₂ and change in fluid-bed temperature were monitored and are shown in Figure 5.

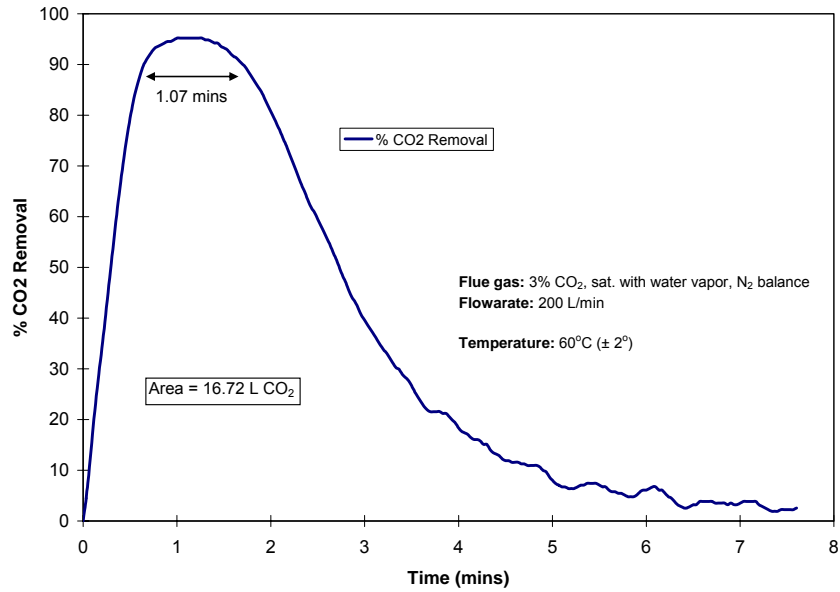


Figure 3. Carbon dioxide removal in first absorption cycle.

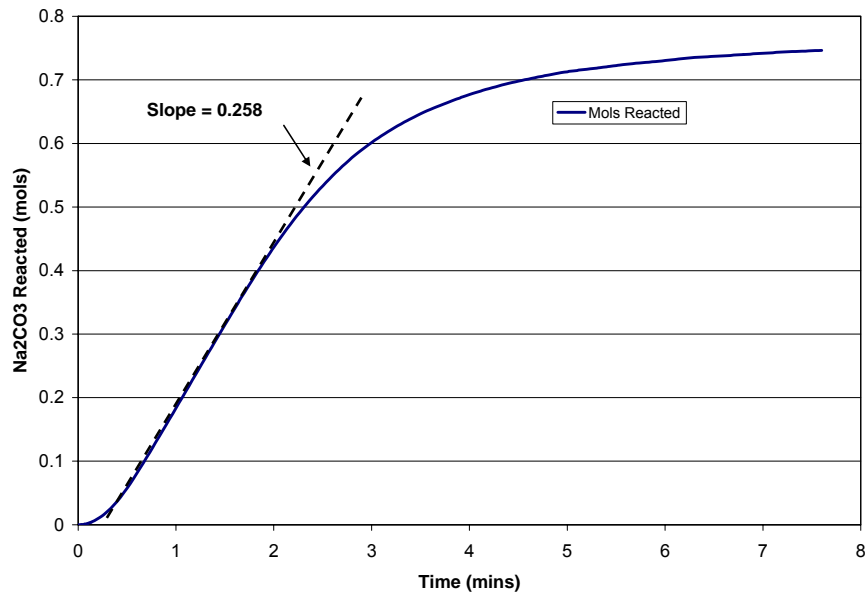


Figure 4. Cumulative sodium carbonate conversion in first absorption cycle.

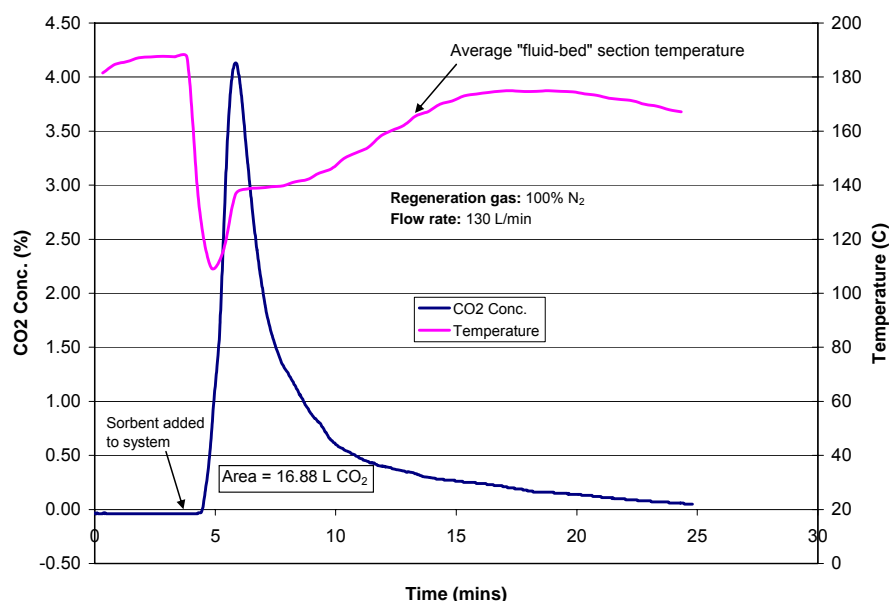


Figure 5. Carbon dioxide release during first regeneration cycle.

These data show that the average temperature of the fluid-bed decreased significantly when the sorbent was added, but never dropped into a range where absorption of CO₂ can be expected (60 to 80°C). For most of the regeneration test, the temperature remained between 140 and 170°C. Integration of the regeneration peak yields a total of 16.88 L CO₂ or roughly 0.75 mols. This represents slightly more CO₂ than was shown to be absorbed but is within expected experimental error, demonstrating a good material balance closure.

4.1.2 Multi-cycle tests

RTI's supported sorbent was used in a seven cycle test to assess its long-term performance. A summary of the test results is provided in Tables 1 and 2.

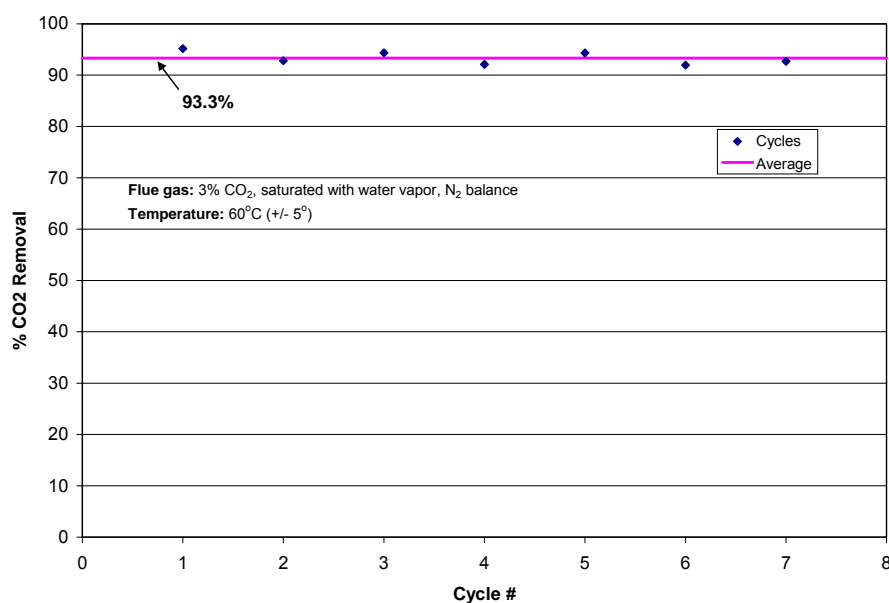
Table 1. Summary of Adsorption Results from Seven Cycle Entrained Bed Test

Cycle	1	2	3	4	5	6	7
CO ₂ removed (L)	16.72	18.30	17.67	14.11	13.24	15.89	14.97
% CO ₂ removal (max)	95	93	94	93	92	92	92
Length of 90% removal (min)	1.07	0.64	1.00	0.53	0.43	0.63	0.47
Start temperature (°C)	61	60	56	60	64	61	65
Temperature rise (°C)	4	13	11	10	12	13	11
Reaction rate (moles/min)	0.258	0.343	0.279	0.248	0.259	0.296	0.272

Table 2. Summary of Regeneration Results from Seven Cycle Test

Cycle	1	2	3	4	5	6	7
CO ₂ released (L)	16.88	15.24	18.07	10.06	13.39	12.29	NA
Start temperature (°C)	187	166	189	186	141	150	NA
Average temperature (°C)	163	154	160	158	151	156	NA

The data presented in Table 1 show the overall sorbent CO₂ removal performance during each of the seven absorption runs. In terms of maximum removal achieved and initial reactivity, the sorbent performed quite consistently over all seven cycles. RTI's sorbent is capable of greater than 90% removal of CO₂ in every cycle and shows negligible drop-off in the seven cycles were conducted as shown in Figure 6.

**Figure 6. Maximum CO₂ removal (%) comparison over seven cycles.**

Another measure of sorbent performance that stayed constant over seven cycles was the sorbent's attrition resistance. It was demonstrated, by measuring the sorbent's Davison Index, that the material was expected to be physically strong for entrained bed operation. During the entrained bed tests at CANMET it was not feasible to collect any meaningful data on weight of fines collected in the system's baghouse. To compensate for this, two relative measures of attrition were used instead: sorbent weight loss per cycle and particle size distribution of the sorbent after each cycle.

As noted earlier, 5.4 kg of the RTI sorbent was loaded into the entrained-bed system to start the multi-cycle tests. After the seventh absorption cycle, 5.95 kg of sorbent was removed from the system. Some of this excess was due to the reaction. It is also possible that some material was left in the system following the initial pretreatment of the sorbent. But if the sorbent experienced significant attrition during these tests, it would be expected that the final sorbent

weight would be less than the starting weight of 5.4 kg. These results suggest that the sorbent can withstand the mechanical stress of an entrained bed system.

Particle size analysis also suggests that the material is not producing any fines due to attrition. Table 3 presents a comparison of particle size data collected after the first five cycles.

Table 3. Comparison of Sorbents Particle Size after Absorption/Regeneration Cycles

Cycle	Fresh	1	2	3	4	5
% of particles below 30 microns	2.7%	0.8%	0.9%	1.0%	1.2%	1.5%
% of particles below 50 microns	19.5%	16.1%	14.1%	20.6%	20.6%	21.4%
Average particle size (microns)	76.38	78.16	83.87	72.02	72.64	72.30

These particle size data do not suggest that the sorbent particles are being converted into finer particles while circulating. The particle size data are consistent over each cycle. In fact, after the first cycle the average particle size actually increased, suggesting that the fines contained in the fresh sorbent batch were blown to the baghouse and no new fines were produced. The percentage of particles below 30 microns shows a negligible increase over five cycles.

4.1.3 Absorption at different CO₂ concentrations

Additional entrained bed reactor testing was conducted to evaluate sorbent performance as a function of starting CO₂ concentration in the flue gas. Table 4 summarizes the results of these tests.

Table 4. Comparison of Sorbent CO₂ Removal Performance at Different Initial CO₂ Concentrations

Starting CO₂ Concentration in Flue Gas	3%	10%	15%
CO ₂ removed (L)	16.72	23.32	24.09
% CO ₂ removal (max)	95	71	61
Time of absorption (min)	6	3.5	3
Start temperature (°C)	61	50	57
Temperature rise (°C)	4	7	14
Reaction rate (mols/min)	0.258	0.752	0.978

Higher initial concentrations of CO₂ in the flue gas have a significant effect on CO₂ removal. The fractional CO₂ removal was lower at higher initial concentrations, but both the total amount of CO₂ absorbed and the rate of CO₂ absorption increased. At a starting concentration of 15%, the sorbent removed 61% of the CO₂ passing through the entrained-bed reactor, under the conditions used.

The improved reactivity at higher CO₂ concentrations is consistent with the previous results obtained during the fixed-bed testing (Green, et al., 2004), but the extent of the increase in reactivity was not expected. The reactivity at 15% initial CO₂ concentration was nearly four

times the reactivity exhibited at 3% initial CO₂ concentration. A graphical comparison of the reactivity results is shown in Figure 7.

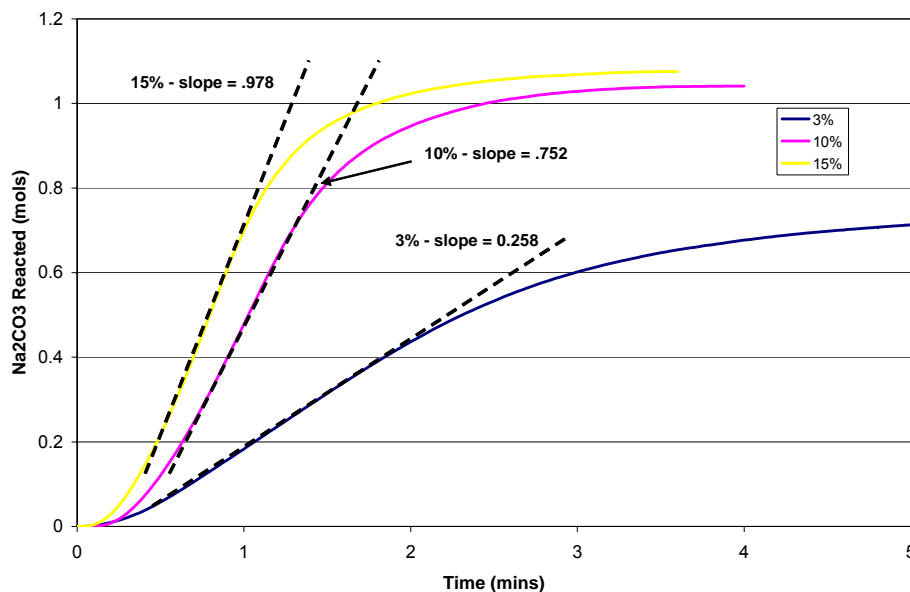


Figure 7. Initial reactivity as a function of inlet carbon dioxide concentration.

4.1.3 Comparison of RTI sorbent to unsupported materials

A single absorption/regeneration cycle at 3% initial CO₂ concentration was conducted for both calcined trona and calcined sodium bicarbonate in order to compare the extent of reaction and reactivity to the RTI sorbent. A summary of the results is shown in Table 5.

Table 5. Comparison Summary of Three Different Sorbents

Sorbent	RTI Supported Sorbent	Calcined SBC	Calcined Trona
CO ₂ removed (L) – absorption	16.2	3.14	3.24
% CO ₂ removal (max)	95	52	56
Time of absorption (min)	6	1.5	1.8
Reactivity rated (mols/min)	0.258	0.133	0.164

Table 5 shows that the RTI sorbent has much higher reactivity rate than the unsupported materials. The higher surface area of the supported material allows for more reactive sites to come in contact with CO₂ and leads to the observed performance enhancement. Although the unsupported materials have a higher overall capacity for CO₂ removal, they removed only 20% of the CO₂. As the reaction takes place in these unsupported materials, the heat generated quickly raises the temperature of the sorbent particle and extinguishes the driving force for CO₂

absorption. Despite the lower reactivity, the unsupported materials still absorbed CO₂ in the entrained-bed reactor. Therefore, if better heat removal techniques (i.e., adding water for evaporative cooling) can be employed in a reactor design, their use may be feasible.

4.2 Fluidized Bed Testing

Six materials were tested in the 1"-quartz reactor system this quarter. These materials consisted of an inert alumina support material and 5 engineered sorbents composed of sodium carbonate supported on ceramic supports. The initial sorbent mass loaded to the reactor was 35 g for all tests. The initial water vapor content of the absorption gas was held constant at 3%. Sorbent descriptions are given in Table 6.

Table 6. Sorbent Compositions Tested at RTI in 1" Fluidized Bed Reactor

Sorbent	Composition
T-2610 Alumina	0% sodium carbonate
SCI-110504-2	15% sodium carbonate
SCI-111704-1	15% sodium carbonate
SCI-011905-1	10% sodium carbonate
SCI-012705-1	15% sodium carbonate
110204-2-C	40% sodium carbonate

The reactivity and useful capacity of the sorbents were compared under various test conditions. Outlet CO₂ concentrations as a function of time are given in Figure 8, for absorption tests conducted with an initial CO₂ concentration of 0.8%. In this series of tests, the sorbent bed was exposed to a gas containing 3% water vapor (balance nitrogen), before the CO₂ was added to the absorption gas.

Carbon dioxide recoveries following regeneration in nitrogen at 150 to 180°C are given in Table 7. The four supported sorbents had very similar capacities at breakthrough. The carbon dioxide capacity of the "inert" support material was found to make a minor contribution to the total capacity of these sorbents.

The following series of tests included 4 of the five materials used in the first series, as well as an additional sorbent containing 40% sodium carbonate. These tests were conducted in a similar manner to the first series, except that the carbon dioxide concentration of the absorption gas was increased to 3%. Absorption data are shown in Figure 9.

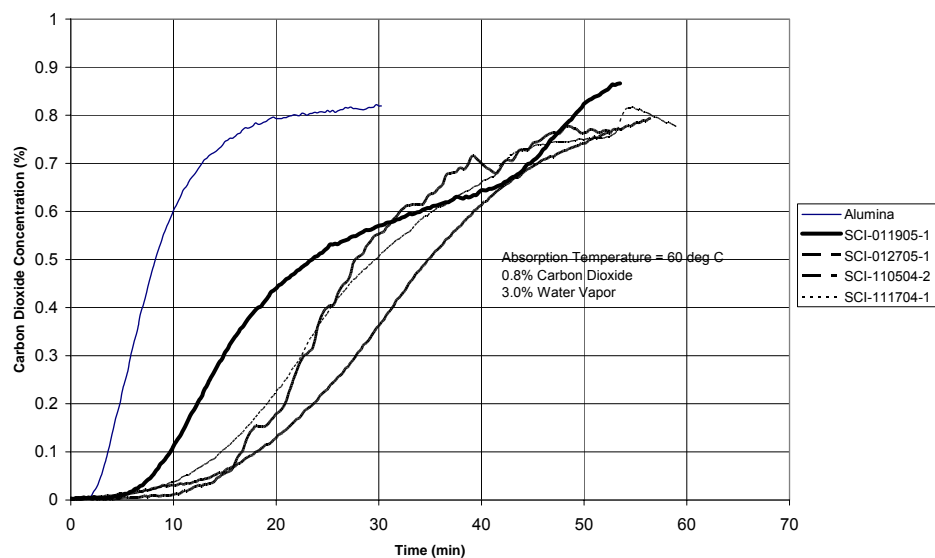


Figure 8. Reactivity of supported sorbents (0.8% carbon dioxide; bed exposed to water vapor before carbon dioxide)

Table 7. Carbon Dioxide Recoveries Following Absorption (0.8% CO₂)

Sorbent	Carbon Dioxide Recovery (mL)
T-2610 Alumina	4.99
SCI-110504-2	140
SCI-111704-1	140
SCI-011905-1	134
SCI-012705-1	126

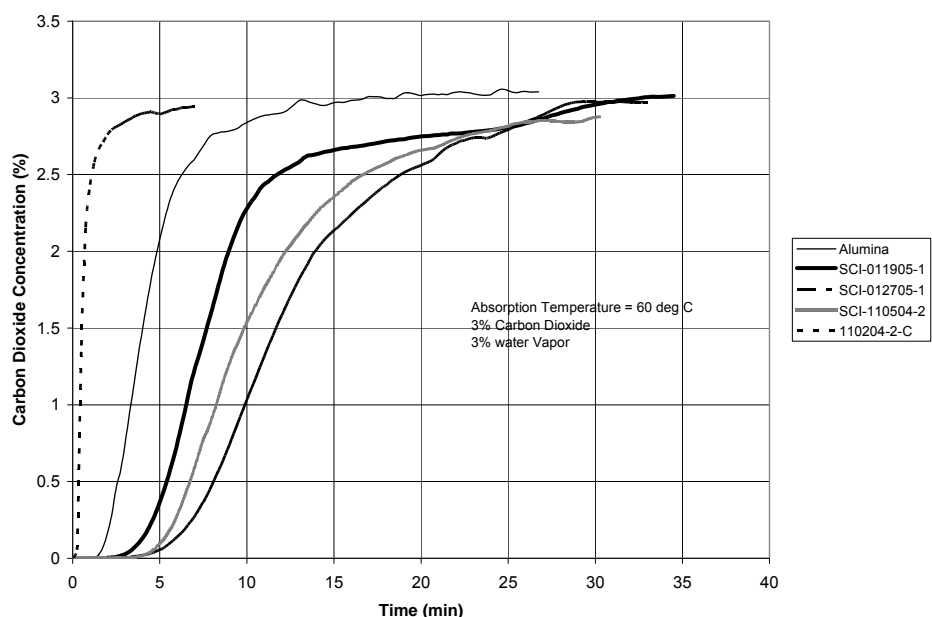


Figure 9. Reactivity of supported sorbents (3% carbon dioxide; bed exposed to water vapor before carbon dioxide)

As expected, breakthrough occurred much more rapidly in the more concentrated absorption gas. Carbon dioxide recoveries following regeneration in nitrogen at 150 to 180°C are given in Table 8.

Table 8. Carbon Dioxide Recoveries Following Absorption (3% CO₂)

Sorbent	Carbon Dioxide Recovery (mL)
T-2610 Alumina	10.1
SCI-110504-2	157
110204-2-C	30.7
SCI-011905-1	130
SCI-012705-1	125

4.3 Other Project Activities

A meeting was held with representatives of Church and Dwight to discuss their continuing participation in the project.

5.0 CONCLUSIONS

An optimized sorbent, prepared at a commercial manufacturing facility, maintained its reactivity over multiple absorption-regeneration cycles in a pilot-scale entrained bed reactor system. Over seven cycles of entrained bed absorption and seven cycles of fluidized bed regeneration, no

significant attrition of the sorbent was observed. The carbon dioxide recovered upon regeneration was roughly equivalent to the integrated carbon dioxide removal during the preceding absorption cycle. This sorbent is capable of removing >90% of the carbon dioxide in flue gas for an expected entrained bed reactor absorption residence time of 5 to 20 seconds.

Supported sorbents of 10 to 15% sodium carbonate demonstrated good initial reactivity in laboratory fluidized bed experiments, with CO₂ breakthrough times influenced by the initial CO₂ concentration of the flue gas. Some absorption of CO₂ will take place even on a ceramic support material with no active ingredient; however this was shown to be relatively minor.

6.0 FUTURE WORK

A presentation will be made at the Fourth Annual Conference on Carbon Capture and Sequestration, and a paper will be prepared for inclusion in the conference proceedings.

Preliminary design of a short residence time dilute phase reactor system will be started with the goal of reducing energy requirements for sorbent conveying.

7.0 REFERENCES

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