

# Results from Experimental Evaluations of Post-Combustion CO<sub>2</sub> Sorbents

Paper 2010-A-59-AWMA

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## ABSTRACT

Carbon capture and sequestration (CCS) can be an important component of reducing worldwide CO<sub>2</sub> emissions from stationary point sources, such as coal-fired power plants. Aqueous amines and ammonia are being demonstrated for CO<sub>2</sub> capture in a temperature swing cyclic process. Solid sorbents can also be used in a similar process, but have the potential to drastically reduce the energy required to release the CO<sub>2</sub> during material regeneration. However, the majority of solid sorbent research is being conducted on lab-scale samples without significant consideration of process and/or equipment design. ADA Environmental Solutions (ADA-ES) is examining available sorbents as well as process equipment options to assess the viability and accelerate development of solid sorbents for CO<sub>2</sub> capture. This work is being completed through a DOE Cooperative Agreement with supplemental funding from ADA-ES, EPRI, and industry. During the first phase of this program, ADA-ES screened over sixty materials using a fixed bed system with simulated flue gas and actual flue gas for multiple adsorption/regeneration cycles to determine which materials were promising for scale-up. Several hundred pounds of select sorbents were produced so they could be evaluated using a unique 1 kW pilot system installed on a slipstream of flue gas at an operating coal-fired power plant. General observations concerning solid sorbents for post-combustion CO<sub>2</sub> capture as well as experimental results from laboratory-scale and 1 kW pilot testing will be discussed.

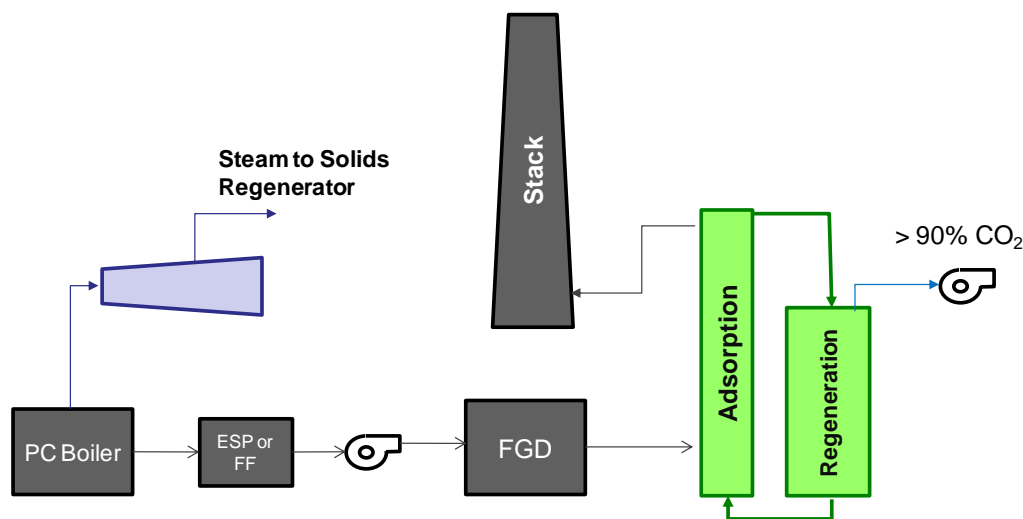
## INTRODUCTION

Approximately one-third of all anthropogenic CO<sub>2</sub> emissions are due to the generation of electricity.<sup>1</sup> The existing coal-fired power plants in the U.S., as many as 1,100, provide 325 GWs of electricity or roughly 50% of the U.S. demand, according to a recent National Coal Council report.<sup>2</sup> The DOE Energy Information Administration (EIA) estimates an additional 46 GW of new capacity will come online between 2007 and 2030.<sup>1</sup> If CO<sub>2</sub> emissions from the electric sector are to be reduced, the current fleet of conventional coal-fired plants must be addressed. Post-combustion capture is one of the few viable options to retrofit such plants.

Nearly 2.5 tons of CO<sub>2</sub> are produced per ton of coal combusted. This quantity is significantly higher than the levels produced for other pollutants such as SO<sub>2</sub> or mercury that are present in coal-combustion flue gas in parts per million (ppm) or parts per billion (ppb) levels, which are captured using chemicals that are used once and then removed from the system (i.e. not regenerated). If a sorbent-based CO<sub>2</sub> control system with a working CO<sub>2</sub> capacity of 20wt% is

employed (note that this working capacity is higher than what has been measured to date under realistic working conditions), nearly 12.5 tons of sorbent would be required per ton of incoming coal. This quantity of sorbent would not be manageable or affordable if used in a once-through process. Therefore, regenerable processes are currently being considered for CO<sub>2</sub> capture.

Currently, the most advanced post-combustion capture options are based upon contacting CO<sub>2</sub>-laden flue gas with a solvent containing amines or ammonia and regenerating the solvent via a temperature-swing adsorption (TSA) process. A generic sketch of a post-combustion TSA system is shown in Figure 1. Note that the CO<sub>2</sub> capture system shown is downstream of a flue gas desulfurization system (FGD). Any sorbents that chemically react with CO<sub>2</sub> will also react with SO<sub>2</sub> to form heat stable salts, which will result in a loss in CO<sub>2</sub> capture performance.



**Figure 1. Generic Post-Combustion TSA Process.**

The energy penalty associated with solvent-based TSA processes is relatively high. For example, recent studies have shown that aqueous monoethanolamine (MEA) for 90% CO<sub>2</sub> capture from a retrofit coal-fired power plant can reduce the thermal efficiency from approximately 35% (HHV basis) to 24.4% and cost \$80 per ton CO<sub>2</sub> removed.<sup>3</sup> Most of this cost is associated with the energy penalty incurred when releasing the purified CO<sub>2</sub> in the regeneration step shown in Figure 1. To reduce the costs associated with commercial-scale carbon capture and sequestration (CCS) technology, emissions control options must continue to be evaluated and improved. One promising option to reduce the energy penalty and cost associated with the material regeneration is to use solid sorbents; these materials require less energy to heat due to a lower specific heat. In addition, they have exhibited greater CO<sub>2</sub> working capacities, so less material will be required.

ADA-ES is currently evaluating solid-based temperature-swing adsorption as an alternative to solvent-based systems through cooperative agreement (DE-NT0005649) with the U.S. Department of Energy (DOE) National Energy Technology Laboratory (NETL) and co-funders including AEP, Ameren, EPRI, First Energy, Luminant, North American Power, Southern Company, and Xcel Energy. The test program consists of screening sorbents in a laboratory-scale fixed bed device, characterizing the performance of the most promising candidates in a 1

kW pilot-scale transport reactor, and developing a process concept and rough costs for a full-scale (500 MW) retrofit system.

## EXPERIMENTAL METHODS

### Laboratory-Scale

The laboratory-scale sorbent screening equipment was designed to be used in the laboratory on simulated flue gas as well as in the field on actual flue gas with minimal modifications. A programmable logic controller (PLC) controlling solenoid valves was employed so that the system was completely automated. The flow rate of the simulated or actual flue gas was approximately 255 scfm, and the amount of sorbent tested varied from 0.5 to 2.5 g. The sorbent and flue gas (either simulated or actual) were contacted in a fixed bed. Although the team does not expect that a fixed bed represents the final contactor design for a large-scale system, this configuration allowed for fast, efficient lab-scale sorbent screening. Figure 2 is a schematic of the sorbent screening testing unit when setup for laboratory testing (only minor modifications are necessary for field testing). The CO<sub>2</sub> analyzer was a continuous NDIR sensor with a 90% response time of 10 seconds.

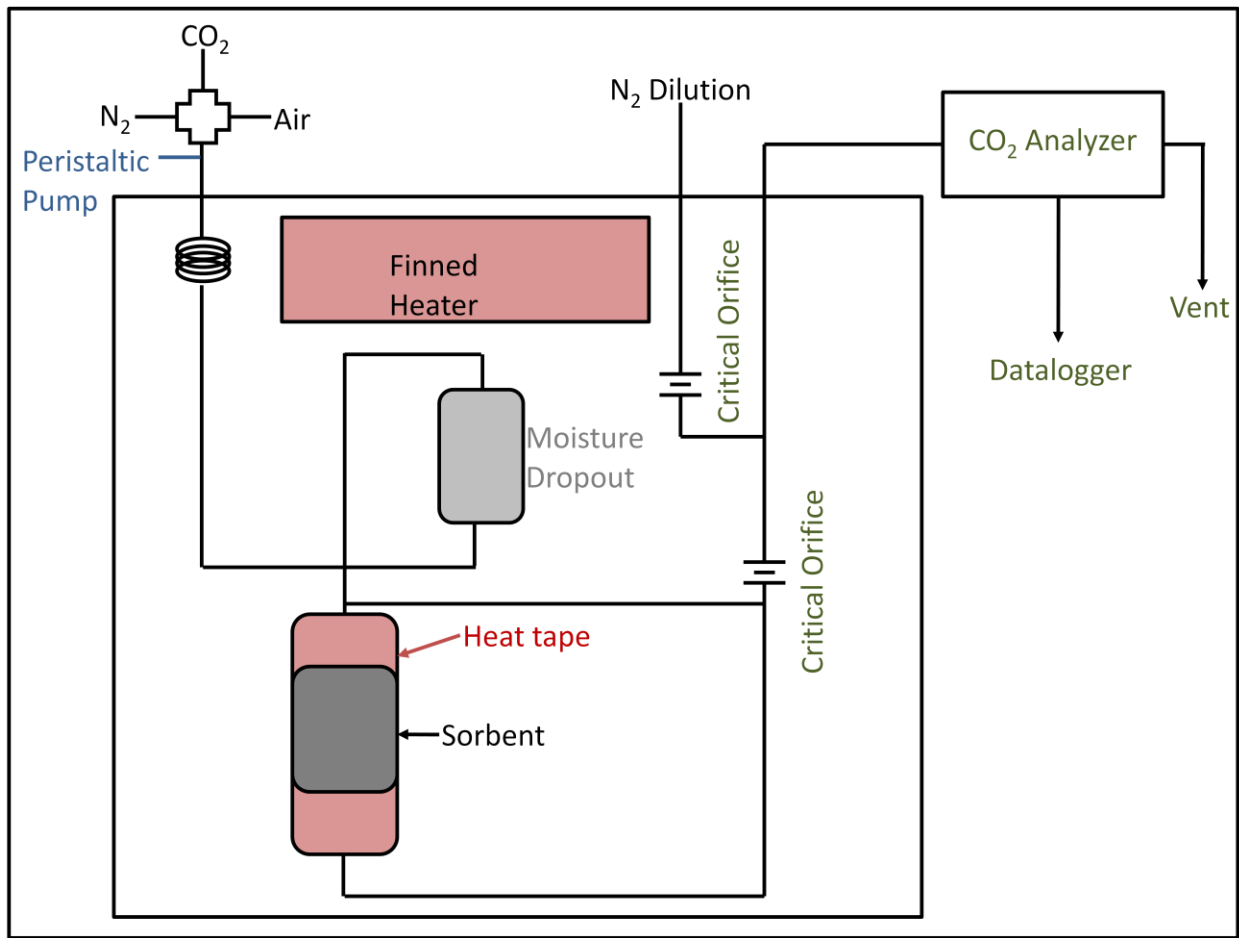


Figure 2: Schematic of Sorbent Screening Test Unit

The steps to carry out the adsorption/regeneration cycles were the same whether tests were conducted on simulated or actual flue gas. First, 2.5g sorbent was placed in the fixed bed, unless the sorbent developer provided less than 2.5g of material in which case the full sample was used. The pressure drop across the fixed bed was measured. If this pressure drop was less than 12 in H<sub>2</sub>O, then 2.5g of sorbent was the sample size. However, if the pressure drop was greater than 12 in H<sub>2</sub>O, then sorbent was removed until the pressure drop was less than or equal to this maximum limit. The pressure drop was measured because the pressure in the sorbent bed has a direct effect on the working CO<sub>2</sub> capacity. Because ADA-ES is testing many different materials with highly varied physical properties, a limit was set on pressure drop.

After the appropriate sorbent sample size was determined, it was heated to an initial flushing temperature. The initial flushing temperature was set based on the expected regeneration temperature, unless a different temperature was recommended by the sorbent developer. A thermocouple on the outside of the glass fixed bed was used to determine when the bed had reached the desired temperature. When the bed temperature matched the desired adsorption temperature, the sorbent was flushed with dry N<sub>2</sub> for 10 minutes or until no CO<sub>2</sub> was being evolved (whichever was longer). Then, the simulated flue gas, composed by mixing compressed gases from tanks, was directed through the bypass line, so that it did not contact the sorbent. The composition of the laboratory sample gas, by volume, was approximately 12% CO<sub>2</sub>, 4% O<sub>2</sub>, with a balance of N<sub>2</sub>. The relative humidity, controlled by adding water using a peristaltic pump and then evaporating, was 0% or 90% (0 or ~15% by volume, respectively). The temperature during the adsorption step was 55°C. The flue gas temperatures and concentrations were selected to be similar to those observed downstream of a wet SO<sub>2</sub> scrubber at a power plant (note that not all flue gas constituents were included in the laboratory testing). When the CO<sub>2</sub> monitor reading was stable and represented the correct concentration of CO<sub>2</sub> (i.e. baseline reading) the gas flow was directed through the sorbent. After the CO<sub>2</sub> levels returned to their original levels (i.e., the sorbent was saturated with CO<sub>2</sub>), the gas was sent through the bypass, which was the end of the adsorption step.

A temperature swing with N<sub>2</sub> purge gas was used to regenerate the sorbents and desorb the CO<sub>2</sub>. Dry N<sub>2</sub> was sent through the bed to flush out the CO<sub>2</sub> as the sorbent was heated. The regeneration temperature was usually in the range of 100 to 120°C, but was dependent on the sorbent type. Clearly, this does not represent a final desorption process, but was necessary due to the fixed-bed size restrictions and the amount of gas required by the analyzer.

Using simulated flue gas and temperature swing regeneration with a N<sub>2</sub> purge, breakthrough curves and estimates for working CO<sub>2</sub> capacity were obtained. In addition, sorbents have been tested with multiple capture/regeneration cycles.

The key sorbent performance properties under investigation in the laboratory-scale research included:

- Working CO<sub>2</sub> capacity (when reported as a weight percentage):  $\frac{100 * mass_{CO_2-removed\ from\ gas}}{mass_{sorbent}}$
- Regeneration potential: ability of a sorbent to be used repeatedly without any reduction in capacity

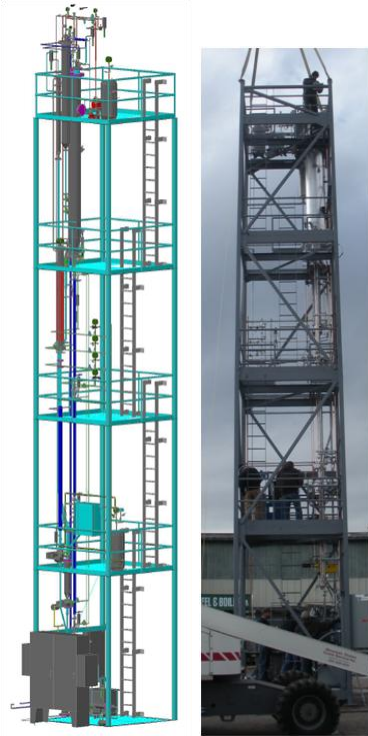
- Poisoning from other flue gas constituents: Decrease in capacity due to SO<sub>2</sub>, NO<sub>x</sub>, etc. (field tests only)
- $\Delta T_{\text{capture-regeneration}}$ : The difference between capture and regeneration temperatures
- Theoretical energy required for regeneration (summation of the sensible heat to increase the sorbent temperature as well as the heat of reaction between the CO<sub>2</sub> and the sorbent)

Three different types of sorbents were tested under the DOE NETL program: carbon-based, supported amines, and zeolites. After testing the materials on simulated flue gas, several of the best performing materials were evaluated for multiple adsorption/regeneration cycles using actual flue gas from a coal-fired power plant.

## 1 kW Pilot Scale

Much work to date regarding solid sorbents for CO<sub>2</sub> capture has been completed in fixed beds in the laboratory. However, to accelerate the development of these materials and a related CO<sub>2</sub> capture process, they must be evaluated in a more realistic system using actual flue gas. Working with researchers at Southern Company, ADA-ES has designed and fabricated a one of a kind 1 kW pilot-scale system designed specifically to test CO<sub>2</sub> sorbents. The system consists of three major components: a transport reactor for adsorption (referred to as the riser), a heated fluidized bed for regeneration (regenerator), and a cooling section. A 3D model sketch and photo of the 1 kW CO<sub>2</sub> capture system are provided in Figure 3. Nominally 5 acfm of treated flue gas is sent through the adsorption riser, where it entrains and reacts with the sorbent. The flue gas for this system is drawn from a duct downstream of a wet FGD system. Because the supported amine sorbents will be negatively affected by the presence of SO<sub>2</sub>, most of the SO<sub>2</sub> will be removed prior to the CO<sub>2</sub> capture system.

The riser temperature will be maintained at approximately 130°F (~55°C), which is the actual flue gas temperature. Although the riser for the pilot system is only 1 inch in diameter, it is 40 feet tall, the approximate required height for a commercial system. This height will provide a contact time of approximately 3 seconds. There is also an added option for sorbent recirculation if additional contact time is required. If the riser scale was increased so that it could treat a larger amount of gas flow, the diameter of would be increased (i.e. the height would not change).



**Figure 3: 3D Model Sketch and Photo of 1 kW System**

Projected regeneration temperatures are in the range of 210°F to 250°F (100°C to 120°C). The maximum residence time in the regenerator is approximately 500 seconds. An ID fan is used to extract the enriched CO<sub>2</sub> stream.

After the sorbent is heated and the CO<sub>2</sub> is removed the sorbent will be sent through a cooler that utilizes jacketed cooling to reduce the temperature of the sorbents to approximately the riser temperature. After the cooler the sorbent is re-introduced to the riser, and the recirculation is continued.

## **RESULTS AND DISCUSSION**

### **Laboratory-Scale**

Sorbents for 1 kW testing were down-selected from more than 100 screened by ADA-ES at the laboratory scale. Since one of the most important advantages of solid sorbents is the potential to reduce the regeneration energy compared to aqueous systems, the theoretical regeneration energy for each material was calculated. The theoretical regeneration energy included the heat input required to increase the temperature of the sorbent (i.e. sensible heat) as well as the energy required to overcome the endothermic reaction to release the CO<sub>2</sub>. A summary of the laboratory screening results, showing the theoretical regeneration energy for several materials compared to aqueous MEA (represented by the red dashed line)<sup>3</sup>, is presented in Figure 4. The specific heat of the materials has not been measured in several cases, but was usually assumed to be in the range of 0.8 to 1.3 kJ/kg K. The results from three different sorbent types are shown in this figure: carbon-based materials (black), supported amines (blue), and zeolites (yellow).

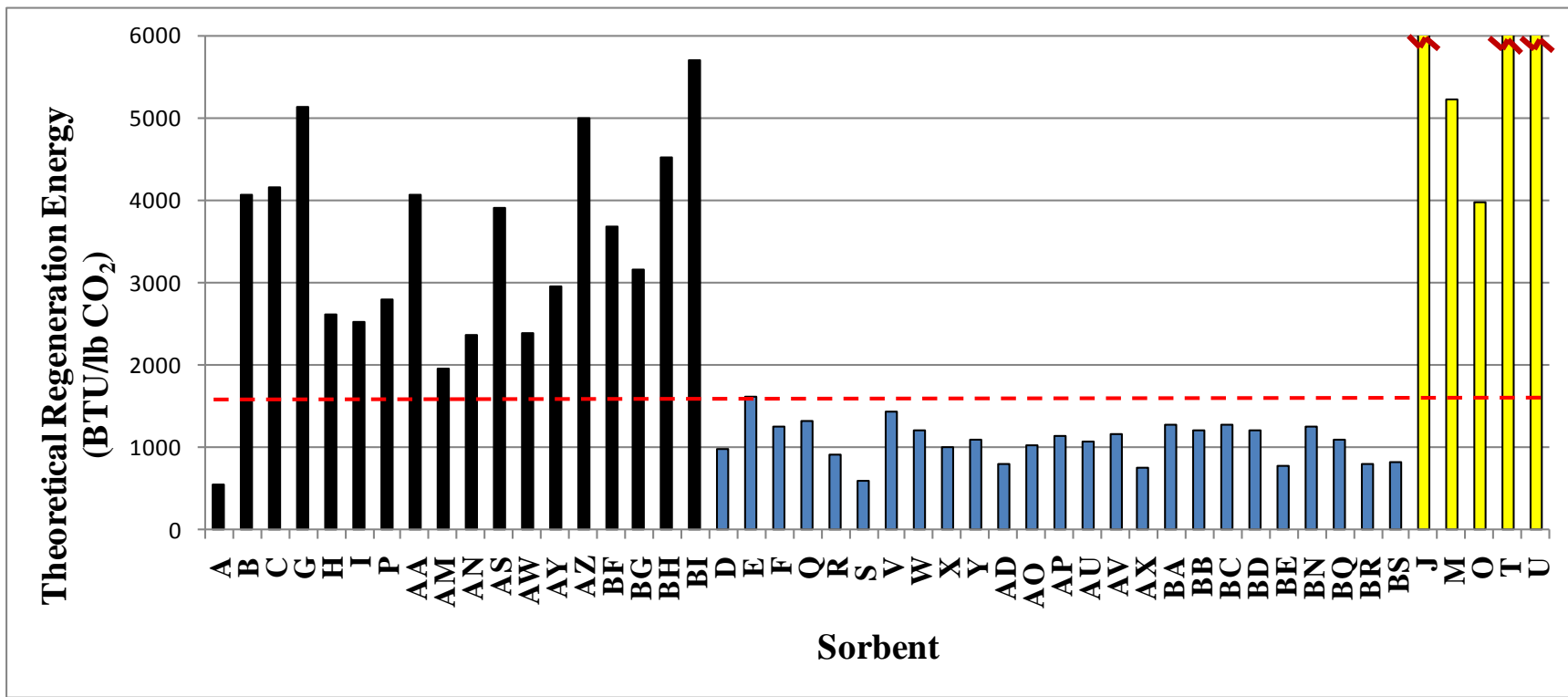
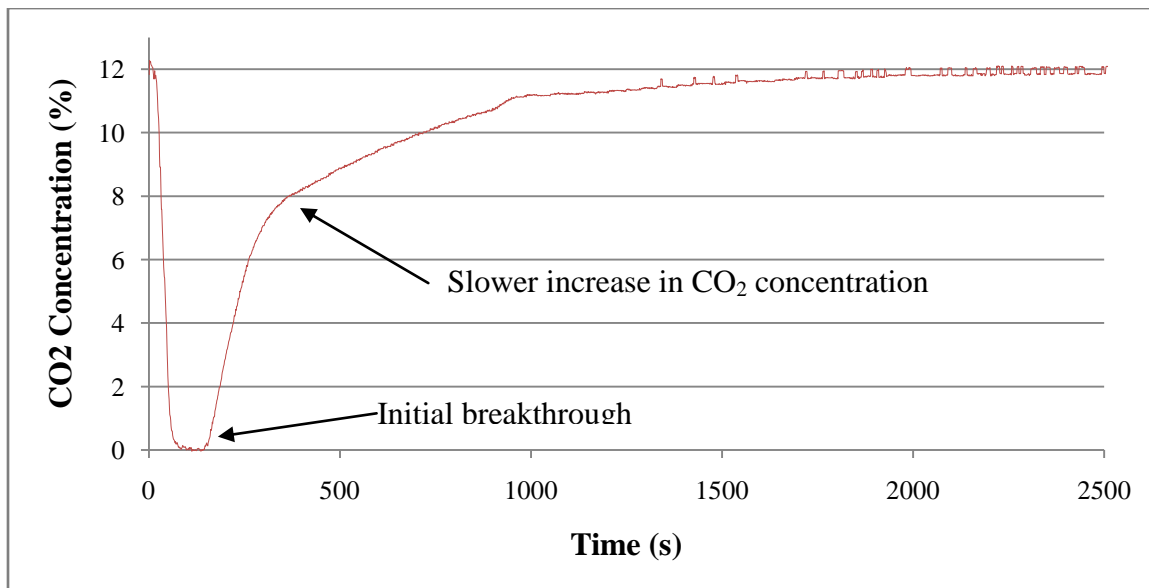


Figure 4: Summary of Sorbent Screening Results

## Supported Amines

Supported amines include any solid material that contains some level of amine functionalization that dominates the reaction with CO<sub>2</sub>. Materials in this sorbent family vary widely; the amine can be chemically bonded or physical adsorbed onto the surface of the substrate. The amine can be a primary, secondary, tertiary, or polymeric in nature. In many cases the substrate is a high surface area material, but this is not always the case. Different types of supports include mesoporous silicas, clays, resins, etc. One key characteristic shared by all substrates is that the specific heat is significantly lower than that of water, which is one of the most important reasons that less energy will be required in a solid-based CO<sub>2</sub> capture system. Based on lab-scale testing results, supported amines are promising for CO<sub>2</sub> capture. This can be attributed to high working CO<sub>2</sub> capacities (over 14 wt% has been measured) as well as low heat capacities.

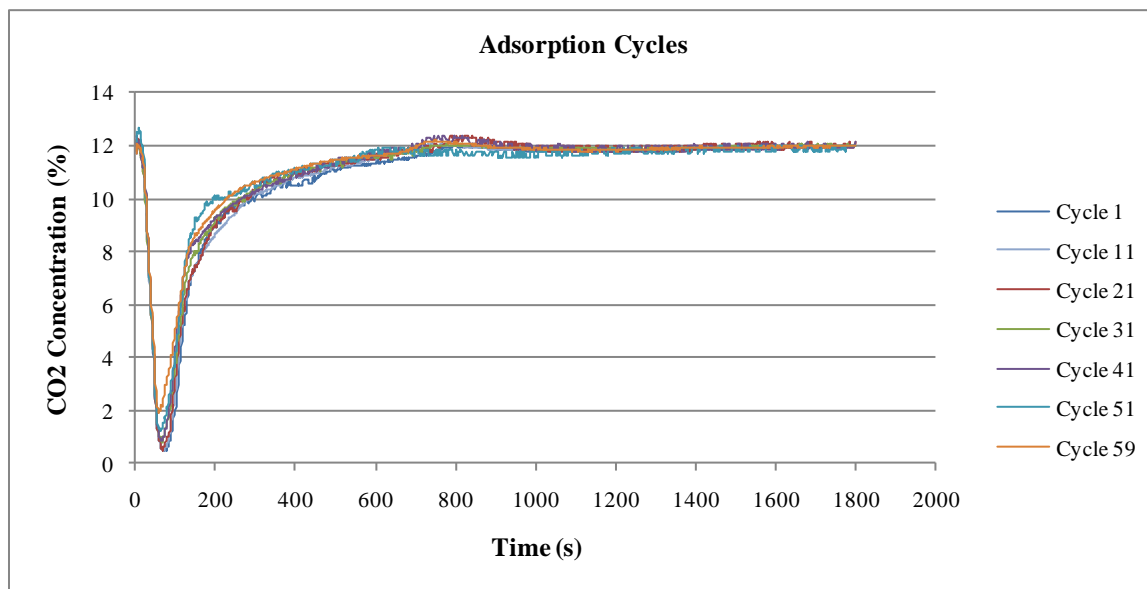
An adsorption profile for sorbent R is provided in Figure 5 to serve as an example of the shape of the breakthrough curve characteristic for supported amines. Clearly this sorbent was able to remove all the CO<sub>2</sub> from the simulated flue gas stream. The initial breakthrough resulted in a fast increase in the CO<sub>2</sub> concentration, followed by a much slower increase in the CO<sub>2</sub> concentration to achieve complete saturation. One theory to explain the slow increase in CO<sub>2</sub> concentration after the initial breakthrough is related to the temperature in the system. First, in a fixed bed system it is difficult to remove the heat from the exothermic reaction. ADA-ES measured a significant increase in the bed temperature (5°C to 10°C). Since the CO<sub>2</sub> capacity is directly related to temperature, this is likely to have some effect on the adsorption profile. However, others who used lower gas flow rates and may have had lesser increases in bed temperature have also measured similar breakthrough curves.



**Figure 5: Sorbent R Adsorption Profile**

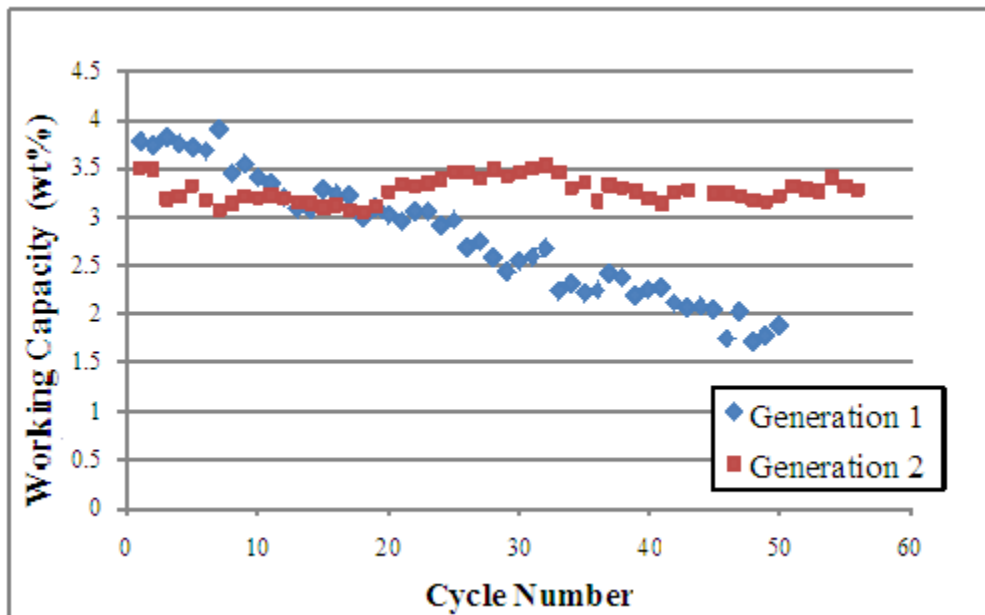
Supported amines have already demonstrated working capacities high enough to dramatically reduce the energy penalty associated with CO<sub>2</sub> capture. The most important challenges for

supported amine sorbents are related to long-term cyclic stability, poisoning by flue gas constituents, and cost. Many, but not all, of the supported amine sorbents demonstrated a loss in CO<sub>2</sub> capacity when using simulated flue gas in the laboratory. As an example, select adsorption profiles from laboratory tests for sorbent AX are provided in Figure 6. Although the testing conditions were the same for all cycles, the adsorption profiles reveal a slow, progressive decrease in the CO<sub>2</sub> capacity of the material, which is most easily recognized due to the increase in the value of the minimum CO<sub>2</sub> level achieved (i.e. maximum level of removal decreases as the sorbent performance decreases).



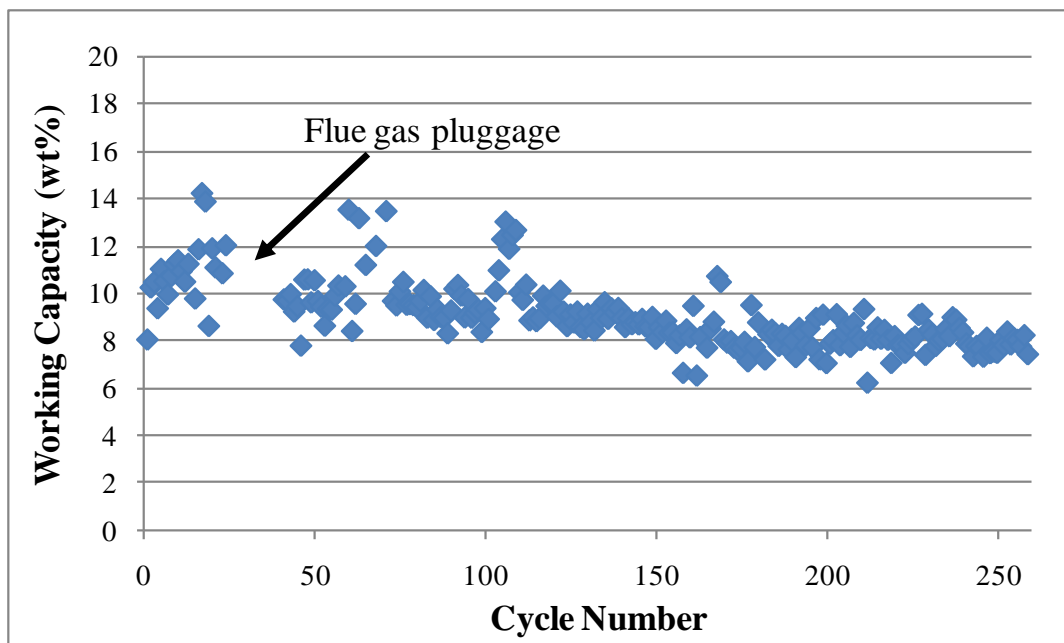
**Figure 6: Sorbent AX Laboratory Adsorption Profiles**

One important goal of this program is to work closely with sorbent developers; ADA-ES's testing results are provided to the developers in the hopes of leading to improvements in sorbent performance. In several cases, collaboration between ADA-ES and sorbent developers has led to second or third generation sorbents exhibiting significantly improved performance properties compared to their first generation counterparts. The working capacities versus cycle number for two different sorbents are shown in **Error! Reference source not found.** Both sorbents are variations of an amine supported by a clay substrate. The generation 1 sorbent was tested by ADA-ES and was found to degrade as the cycle number increased, even when using simulated flue gas in the laboratory. The degradation was attributed to evaporation of the amine from the sorbent, which was supported by the observation of viscous liquid downstream of the sorbent bed. Several months later the developer provided a similar sorbent that was also tested for multiple cycles. During the 56 cycle test, there was no sign of a decrease in capacity. Notably, the developer has measured a significantly higher CO<sub>2</sub> working capacity, which may be attributed to a very low gas residence time in the ADA-ES fixed bed system (~1 s). In the 1 kW pilot field test the gas residence time will be closer to 3 s and may lead to greater working CO<sub>2</sub> capacities. The improvement of this material is an example of how collaboration can lead to better sorbent options and thus an increase in the chance of success.



**Figure 7: Example of Improvement between First and Second Generation Sorbents**

Once acceptable cyclic stability is achieved in the laboratory, the next challenge is to determine if additional flue gas cleanup steps are required to achieve similar stability at a power plant. Sorbent BR was selected for extended field testing. Over 250 cycles were completed using actual flue gas. Because the reaction between  $\text{SO}_2$  and amines to form heat stable compounds is well-documented, ADA-ES fabricated a lab-scale  $\text{SO}_2$  scrubber that was used to reduce the  $\text{SO}_2$  concentration in the flue gas to less than 10 ppm. The working capacities measured during this field test are provided as a function of cycle number in Figure 8. Although there was some decrease in the working capacity over the 250 cycles, the stability was significantly better than other supported amine sorbents evaluated in actual flue gas. Additional modifications will be necessary to even further reduce the loss in capacity, but the improvement over the sorbents tested only one year before are noteworthy.

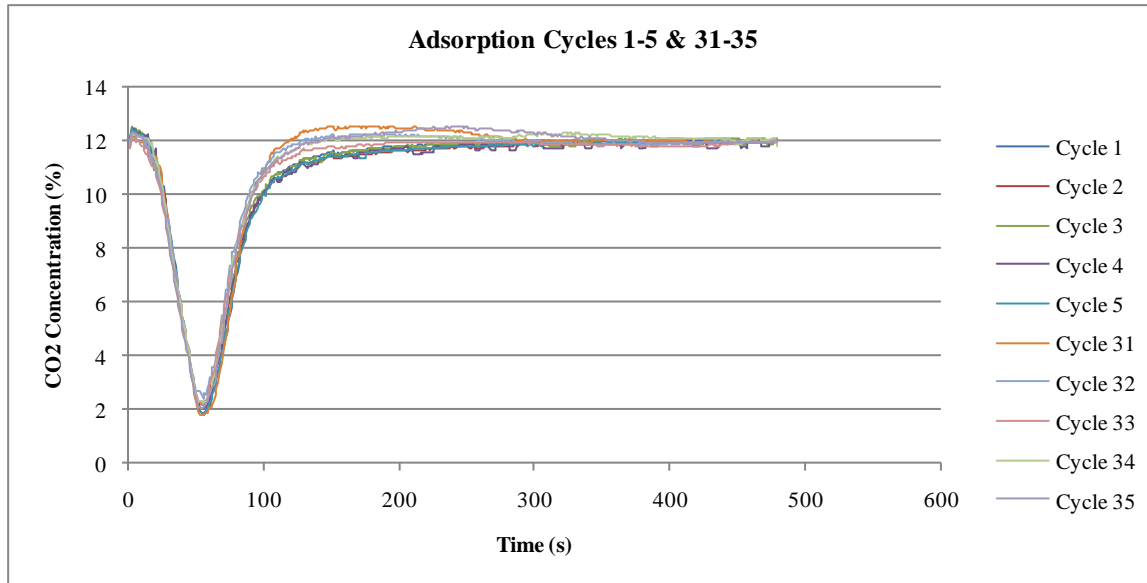


**Figure 8: Sorbent BR Field Working Capacities**

### ***Carbon Based Sorbents***

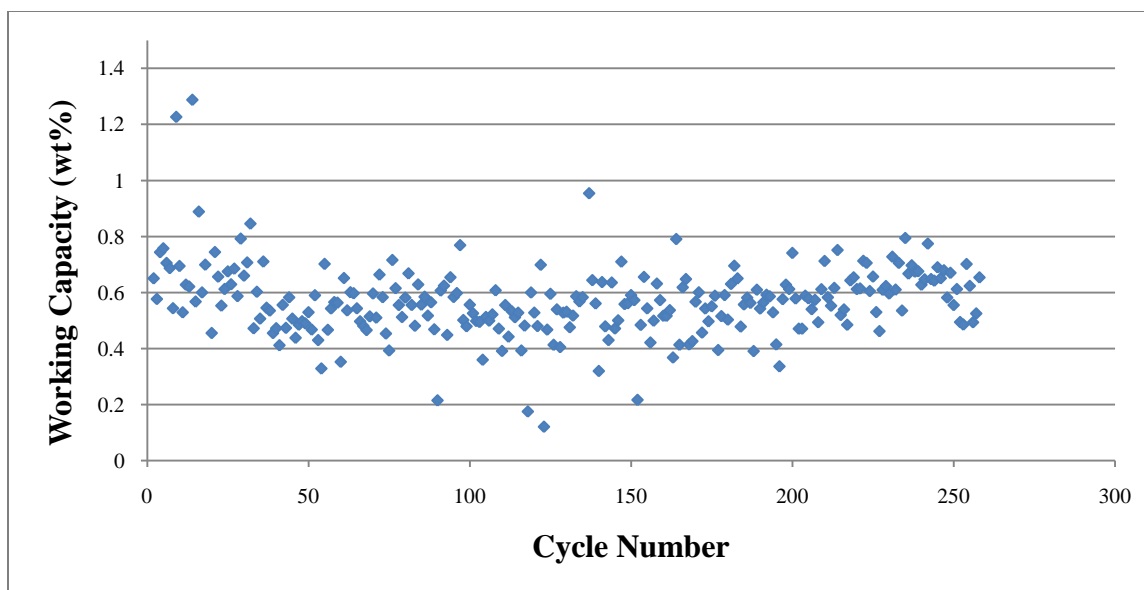
The carbon-based materials evaluated during this program have included everything from highly novel carbon nano-tubes to commercially available activated carbons. With the exception of the carbon nano-tubes (sorbent A), all the working capacities were significantly lower than those of the supported amines. In general the working capacities were lower than 1.1 wt%. In terms of a materials handling issue, to achieve 90% CO<sub>2</sub> capture using low-capacity carbons would require significant energy to convey the materials. However, there are also advantages to using carbon-based materials. Specifically, they are relatively low cost, commercially produced today, they can be burned after they are spent (i.e. less waste generated by the CO<sub>2</sub> capture process), and they are less easily poisoned by flue gas constituents compared to both amines (negatively affected by SO<sub>2</sub>) and zeolites (prohibitively affected by moisture). Some carbons have shown a decrease in CO<sub>2</sub> performance in moist versus dry flue gas, but this only decreases the capacity to some equilibrium value, it does not remove all the CO<sub>2</sub> capacity.

To serve as an example select adsorption profiles for sorbent AM are provided in Figure 9. Cycles 1-5 were run using dry simulated flue gas, while cycles 31 to 35 were run using simulated flue gas with 90% relative humidity. Although there is a noticeable difference in the profiles, the majority of the adsorption capacity is maintained regardless of moisture levels.



**Figure 9: Sorbent AM Laboratory Adsorption Profiles**

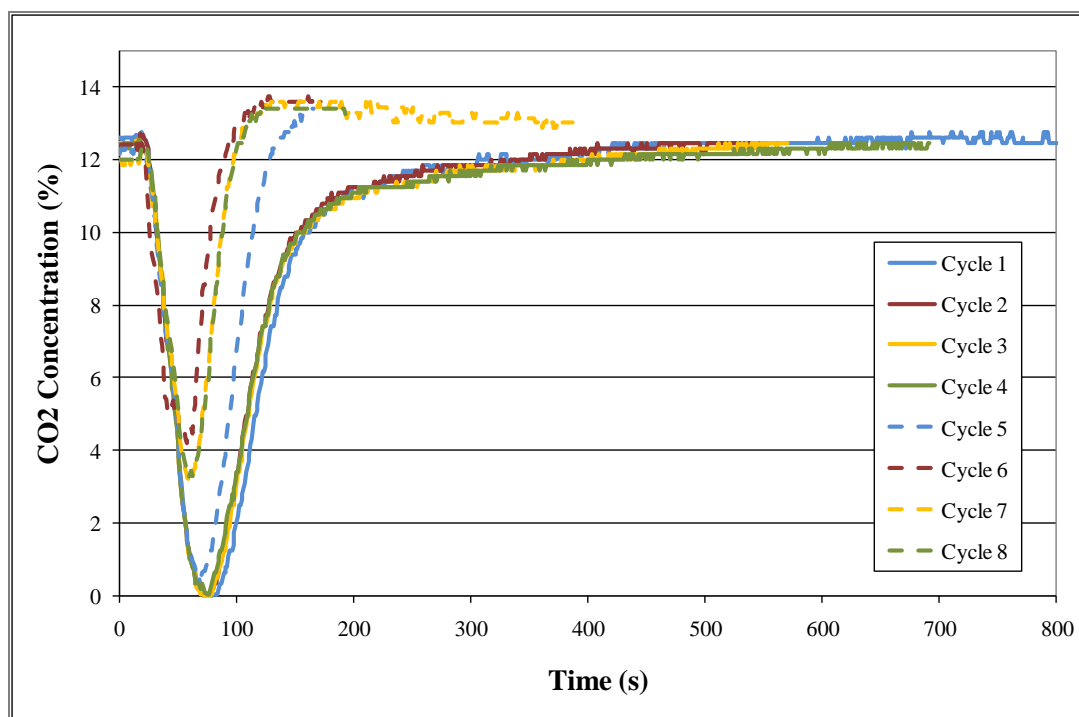
Moisture is not the only flue gas constituent that causes a concern. Chemicals that react with CO<sub>2</sub> usually react with SO<sub>2</sub> to form a heat stable salt. Over time this will result in a complete loss of CO<sub>2</sub> adsorption capacity. One potential advantage of carbon-based sorbents is that they do not chemically react with CO<sub>2</sub>. Therefore, it is theorized that they will not be permanently damaged by flue gas constituents. ADA-ES tested this theory by completing over 250 adsorption / regeneration cycles on actual flue gas with a commercial activated carbon. Note that this material was not produced specifically for CO<sub>2</sub> capture. The flue gas used for the cyclic testing was drawn from the duct of a lignite fired power plant. The SO<sub>2</sub> concentration was in the range of 50 to 250 ppm and the flue gas was saturated with moisture. The working capacities from this test are provided in Figure 10. No permanent decrease in capacity is observed for the 250+ cycles, confirming that carbon-based materials are more resistant to poisoning compared to other materials. The challenge for carbon-based materials is to improve the CO<sub>2</sub> capacity and selectivity. ADA-ES continues to work with sorbent developers to provide feedback in the hopes of improving these promising materials.



**Figure 10: Sorbent AY Field Working Capacities**

### ***Zeolites***

At first glance zeolites are an attractive option for CO<sub>2</sub> capture because several of these materials are commercially available, and may be durable for many adsorption / regeneration cycles even in the presence of flue gas constituents. During the initial sorbent screening a few zeolites exhibited the ability to remove CO<sub>2</sub> from dry simulated flue gas. However, as moisture was added to the simulated flue gas the working capacity of the materials dropped dramatically. Even at high regeneration temperatures (up to 200°C), they performed poorly in the presence of moisture. Select adsorption profiles from laboratory screening are provided in Figure 11. The first four adsorption / regeneration cycles were conducted using dry simulated flue gas while cycles 4-8 were conducted using humid flue gas. The decrease in CO<sub>2</sub> capture performance is clear. When humid simulated flue gas was used, the final CO<sub>2</sub> concentration was actually greater than the starting CO<sub>2</sub> concentration. Initially, this was a source of confusion; however, additional testing with longer adsorption times revealed that the CO<sub>2</sub> concentration would eventually return to the starting value. This can be attributed to the displacement of adsorbed CO<sub>2</sub> by H<sub>2</sub>O; the adsorption of CO<sub>2</sub> is more rapid initially, but given enough time exposure to moist simulated flue gas the zeolite will actually displace the adsorbed CO<sub>2</sub> in favor of H<sub>2</sub>O molecules.



**Figure 11: Sorbent J Laboratory Adsorption Profiles**

When a low CO<sub>2</sub> capacity and a high regeneration temperature are exhibited, the theoretical regeneration energy is exceedingly high, as is shown in Figure 4.

## 1 kW Pilot Scale

The 1 kW pilot system is a one of a kind CO<sub>2</sub> capture system designed and constructed to characterize the CO<sub>2</sub> capture effectiveness of solid sorbents on a more meaningful scale compared to laboratory-scale tests. It will be used to evaluate CO<sub>2</sub> sorbents with highly varied physical and chemical properties. Testing in the 1 kW pilot system began in March 2010 at Host Site 1, which is a lignite-fired power plant equipped with a wet FGD system. The first sorbent being tested is a supported amine. When the sorbent was first introduced to the flue gas, it was entrained as planned. In the lab-scale reactor a small temperature increase (~10°C) was observed during adsorption due to the heat of reaction. However, in the 1 kW pilot the temperature increase was over 25°C at the sorbent injection location. This measurement confirmed that the high level of mixing in the 1 kW pilot adsorption section aids the reaction by reducing the mass diffusion limitations. In addition, the CO<sub>2</sub> concentration exiting the riser dropped significantly. Although these tests are ongoing and the observations must be confirmed through additional testing, results to date have been promising.

## CONCLUSIONS

Recent evaluations of CO<sub>2</sub> capture using solid sorbents indicate that this technology has the potential to meet a 90% CO<sub>2</sub> capture target. One of the goals of the program was to evaluate a

wide range of materials from multiple developers on the laboratory-scale. General observations from initial screening tests include:

- Supported amines have exhibited high CO<sub>2</sub> capacities and low theoretical regeneration energies, but many are not cyclically stable and can be negatively affected by flue gas constituents.
- Carbon-based sorbents have a low CO<sub>2</sub> capacity, but exhibit superior cyclic stability and a resistance to poisoning by flue gas constituents.
- Zeolites have exhibited an extreme affiliation to moisture that makes their use for CO<sub>2</sub> capture unlikely.

The most important advantage of using solid sorbents for post-combustion CO<sub>2</sub> capture versus other state of the art technologies is the potential operating cost savings resulting from a lower energy penalty. Sorbents have exhibited significantly lower heat capacities and higher working CO<sub>2</sub> capacities compared to solvents such as aqueous MEA, resulting in a substantially lower regeneration energy. In addition, these same sorbent characteristics have the potential to reduce additional water requirements. However, material stability and must continue to be addressed and evaluated as technology development is continued. The 1kW pilot-scale field tests currently underway will provide valuable information based on more realistic operating conditions.

## **ACKNOWLEDGEMENTS**

The authors gratefully acknowledge the organizations that provided both financial and technical support for this project. Support for this work was provided by the US Department of Energy National Energy Technology Laboratory (DE-NT0005649) with additional cost share from American Electric Power, Ameren, EPRI, Luminant, North American Power Group, Southern Company, Xcel Energy.

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## **KEYWORDS**

Carbon capture and sequestration, CO<sub>2</sub> capture, adsorption, solid sorbents