

SOLID SORBENTS AS A RETROFIT CO₂ CAPTURE TECHNOLOGY: 1 MW PILOT DESIGN

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ABSTRACT

ADA Environmental Solutions (ADA) recently completed a multi-year project to assess the viability and accelerate development of solid sorbents as a CO₂ capture retrofit technology. This project included an in depth materials study as well as a high level evaluation of one potential 430 MW_e conceptual design. The materials study consisted of laboratory and 1 kW_e slipstream tests. From the laboratory results general conclusions were drawn regarding different sorbent types. In addition, the laboratory results were used to select the most promising sorbents, which were then produced in larger quantities and evaluated using actual flue gas in a unique 1 kW_e equivalent slipstream test device at three different power plants. A high level cost assessment for one conceptual process design was completed for ADA by Stantec Consulting to help direct future development by identifying potential cost drivers. The information collected and knowledge gained during the viability assessment is being funneled into a subsequent project where a 1 MW_e pilot will be designed, fabricated, and operated. Significant results and major conclusions from the sorbent evaluation and equipment assessment will be presented and the conceptual design for the 1 MW_e pilot will be described. These projects are being funded by the DOE National Energy Technology Laboratory’s Innovations for Existing Plants (IEP) Program.

INTRODUCTION

Post-combustion CO₂ capture (PCCC) and sequestration is one of the only feasible means to significantly reduce CO₂ emissions from existing large coal-fired power plants. Although no CO₂ capture technology is currently offered commercially, the most developed processes utilize an aqueous amine solvent, often monoethanolamine (MEA), to react with the CO₂ at low temperature and then release it in a purified form at a higher temperature. The energy penalty associated with solvent-based temperature swing processes is relatively high. For example, recent studies have shown that aqueous MEA for 90% CO₂ 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 CO₂ removed.¹ Much of this cost is associated with the energy penalty incurred when releasing the purified CO₂ in the heating/regeneration step.

Solid sorbents can also be used in a process that utilizes a temperature swing, referred to as temperature swing adsorption (TSA). However, a temperature swing process that utilizes solids has potential to significantly reduce the energy penalty associated with the regeneration step due to an appreciably lower specific heat of solid versus aqueous materials. A simplified schematic of a TSA process is provided in Figure 1.

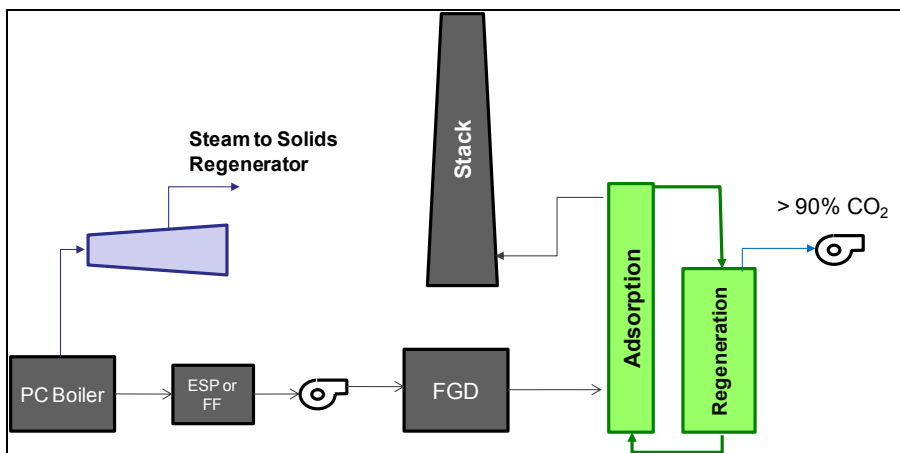


Figure 1. Generic Post-Combustion Temperature Swing Process

An extensive viability assessment was recently completed by ADA-ES under a cooperative agreement with the Department of Energy (DE-NT0005649). This viability assessment included sorbent screening at the laboratory and 1 kW scale. In addition a high level equipment evaluation and cost assessment was completed. Based on the project results, it was determined that supported amine sorbents currently offer the best potential to significantly reduce the energy penalty associated with post-combustion CO₂ capture.^{2,3} However, such sorbents must be paired with a system/process that can take advantage of their beneficial properties.

Under a separate cooperative agreement DE-FE0004343 ADA-ES is continuing development of a sorbent based CO₂ capture technology through process evaluation, 1 MW pilot testing, and a techno-economic assessment. One of the first steps of this project was to select a sorbent and design to test at the 1 MW scale. This report discusses the key selection criteria, experimental techniques, and conclusions that led to a sorbent and process selection.

EXPERIMENTAL

Laboratory Testing

A laboratory-scale fixed bed system was used to quickly and efficiently measure the CO₂ uptake of many different sorbents under simulated flue gas conditions. During adsorption the composition of the laboratory sample gas, by volume, was approximately 12% CO₂, 4% O₂, with a balance of N₂. The relative humidity was controlled by passing the simulated flue gas through a heated bubbler at a temperature of approximately 40°C, (0 or ~7% by volume). The temperature during the adsorption step was 55°C. The simulated flue gas temperatures and concentrations were selected to be similar to those observed downstream of a wet SO₂ scrubber at a coal-fired power plant (note that not all flue gas constituents were included in the laboratory testing). A temperature swing with a N₂ purge gas was used to regenerate the sorbents and desorb the CO₂. The N₂ used for regeneration was first passed through a bubbler at room temperature to ensure that the sorbents were not completely dried during regeneration, which was especially important for the supported amine sorbents.^{4,6} 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 N₂ purge, breakthrough curves were measured, which were then integrated to obtain the CO₂ capacity. To check for stability most sorbents were tested for multiple, up to 260, adsorption/regeneration

cycles. For a few of the most promising sorbents trace flue gas constituents such as SO₂, NO₂, or NO were added to the simulated flue gas.

A Perkin Elmer Pyris 1 Thermogravimetric Analyzer (TGA) was used to determine the working capacity of the materials when regenerating under increased CO₂ concentrations. The CO₂ uptake was measured at several different CO₂ partial pressures and nine different temperatures. Since the TGA was operated under atmospheric pressure, and the tests were located at the company headquarters in Littleton, CO, even with 100% CO₂ the partial pressure was approximately 0.81 bar. Note that a small amount of moisture was added to the gas during the TGA tests due to the aforementioned concern about completely drying the supported amine sorbents. Still, the moisture levels were lower than 1% by volume so that the effect on the weight change of the sorbent would be minimal. The test details are included in Table 1.

Table 1: TGA Systems and Operating Conditions

TGA	Sample Size (mg)	Gas Flow Rate (mL/min)	Temperatures (°C)	CO ₂ Partial Pressure (bar)	Dew Point (°C)
Perkin Elmer Pyris 1	1 to 5	100	40-120	0.04 to 0.81	<22

1 kW Pilot Testing

Much work to date regarding solid sorbents for CO₂ capture has been completed in the laboratory using compressed gases. However, to accelerate the development of these materials and a related CO₂ capture process, they must be evaluated in a more realistic system using actual flue gas. Working with researchers at Southern Company, ADA has designed and fabricated a one of a kind 1 kW pilot-scale system designed specifically to test CO₂ sorbents. This system was designed to be used to test several different sorbents with highly varied physical properties, and, therefore, was not intended to be a small-scale version of a final commercial technology. The system consisted 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 CO₂ capture system are provided in Figure 2. Nominally 5 acfm of treated flue gas was sent through the adsorption riser, where it entrained and reacted with the sorbent. The flue gas for this system was drawn from a duct downstream of a wet FGD system and additional SO₂ scrubbing was employed. The riser temperature was approximately 130°F (~55°C), which was the actual flue gas temperature. Although the riser for the pilot system was 1 inch in diameter, it was ~ 38 feet tall to provide a gas/solid contact time of approximately 2.5 seconds. 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).

The regeneration temperatures were varied during the tests, but were approximately 100°C. The residence time in the regenerator was approximately one hour. After the regenerator, the sorbent was sent through a cooler that utilized jacketed cooling to reduce the temperature of the sorbents to approximately the riser temperature. After the cooler the sorbent was re-introduced to the riser, and the recirculation was continued.



Figure 2. 3D Model Sketch and Photo of 1 kW System

Equipment Cost Analysis

Currently, there is no commercial process to achieve post-combustion CO₂ capture using solids. However, to assess the viability of solids for this application, both the sorbents and the commercial-scale costs must be taken into account. ADA hired the engineering firm Stantec Consulting to study the equipment, process, and costs related to using sorbents for PCCC at the commercial scale. While realizing that a commercial CO₂ capture process must be designed specifically for this application, high level estimates of capital and operating costs were determined to 1) assess the viability of the technology and 2) identify the technology cost drivers and direct future development efforts. Solid-based CO₂ capture can be divided into the following unit operations:

- Adsorption
- Heating and cooling, or heat transfer
- Conveying
- Desorption

For these four main unit operations a technology review was conducted. The review included collecting information from a wide variety of sources, including published papers, advertisements, web searches, and vendor interviews. The survey encompassed a broad range of gas-solids contactor and thermal regeneration technologies (including fluid beds, entrained flow, gravitational cross flow, moving bed, radial flow fixed bed, other), from air pollution control (APC) as well as chemical process industry (CPI) and mineral processing industry (MPI) that could be used for solid sorbent CO₂ capture will be performed. A technology survey sheet was generated for each technology.

A weighted scoring system used to objectively analyze and screen the technologies. The process detailed in “The New Rational Manager” by Kepner and Tregoe was utilized in this analysis.⁷ A list of criteria was compiled, and each was given a weight according to the perceived importance of each item. When a technology was screened, it was given a score from 1 to 10 against each criterion, and the weighted score was the product of the weight and the score. A summation of the weighted scores provided a total score, which was then

compared to all the other technologies. This method allowed each technology to be compared against others, and higher scoring technologies were then selected for further investigation. In many cases technologies were applicable to more than one of the four main categories (adsorption, heat transfer, convey, and desorption). In such cases, the technology was rated separately applicability in each category.

The technologies that scored the highest were carried forward to create a ~430 MW conceptual design, around which capital and operating costs could be developed. The purpose of this study was to identify key cost drivers and to collect an order of magnitude assessment of costs for the viability assessment.

1 MW Design

To develop concepts and compare them for the 1 MW pilot design, ADA worked with team members from Stantec Consulting Ltd. and Shaw Energy & Chemicals, Inc. Different configurations of entrained flow reactors, moving beds, and fluidized beds were compared. Key selection criteria used to evaluate various process designs included:

- Unit operations available (or can be available with further development) at the scale required
 - Gas/solids contacting (limiting mass diffusion as much as possible)
 - Heat removal/addition (as fast as possible)
 - Material handling (transfer of solids from one point to another)
- Low capital cost
 - Simple configuration
 - Less expensive materials of construction
 - Small size and/or the lowest number possible of operating trains
- Low operating costs
 - Low energy penalty
 - Maximization of sorbent working CO₂ capacity (i.e. effectively counter current gas/solids contacting)
 - Low pressure drop across the process
 - Ability to be thermally integrated with power plant
 - High operability
 - Few moving parts
 - Industry proven unit operations
 - Reliability and availability standards similar to previous Utility operations

RESULTS AND DISCUSSION

Laboratory-Scale Testing

Since October 1, 2008 over 140 potential CO₂ capture sorbents were collected and tested in the laboratory using the equipment and procedures discussed in the Experimental section. The sorbents evaluated during the fixed bed laboratory screening were divided into the following categories: supported amines, carbon-based, supported carbonates, zeolites, and hydrotalcites. Based on the results of the extensive fixed bed screening, supported amines were selected as the most promising option for post-combustion CO₂ capture. Four supported amine sorbents were selected for testing at the 1 kW scale and were then produced in 500 lb+ quantities.

The TGA was used to develop isotherms for the supported amines that demonstrated 1) the greatest CO₂ capacity under simulated flue gas conditions using the fixed bed and 2) cyclic stability. The CO₂ working capacity dictates the minimum amount of sorbent required to capture 90% of CO₂ from flue gas. The actual conditions (i.e. temperature and CO₂ partial pressure) to which the sorbent will be exposed will be determined

by the capture/regeneration process. However, for the purpose of comparing different supported amine sorbents, the general conditions of 50°C, and 0.081 bar CO₂ for adsorption and 120°C and 0.81 bar CO₂ for regeneration were used to calculate CO₂ working capacities. The CO₂ capacity at different conditions measured using the TGA for one supported amine sorbent, called sorbent BN, are provided in Figure 3.

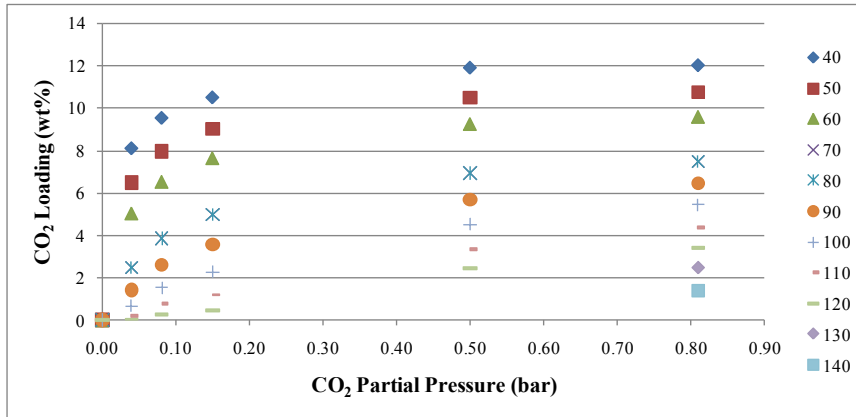


Figure 3: Sorbent BN CO₂ Capacity at Various Temperatures and CO₂ Partial Pressures

1 kW Pilot Testing

Field testing with the 1 kW pilot was completed at two different power plants: Luminant’s Martin Lake Steam Electric Station (Martin Lake) and Xcel Energy’s Sherburne County Generation Station (Sherco). Four different supported amine sorbents were evaluated in the 1 kW pilot. Sorbent R was a supported amine sorbent, originally invented by researchers at NETL. Ten batch mode adsorption cycles were measured during tests at Martin Lake. Although 90% CO₂ removal could not be maintained when operating the 1 kW pilot continuously with sorbent R; over 90% CO₂ removal was possible when the system was operated in batch mode. Sorbent AX, which was similar to sorbent R except the substrate was a different porous silica, was operated in batch mode at Sherco. The maximum CO₂ removal demonstrated by the two sorbents is provided in Figure 4. Although the removal observed in cycle 5 for sorbent AX was significantly lower than the other cycles, it is believed that this was caused by equipment issues due to extremely low temperatures, rather than a decrease in sorbent performance.

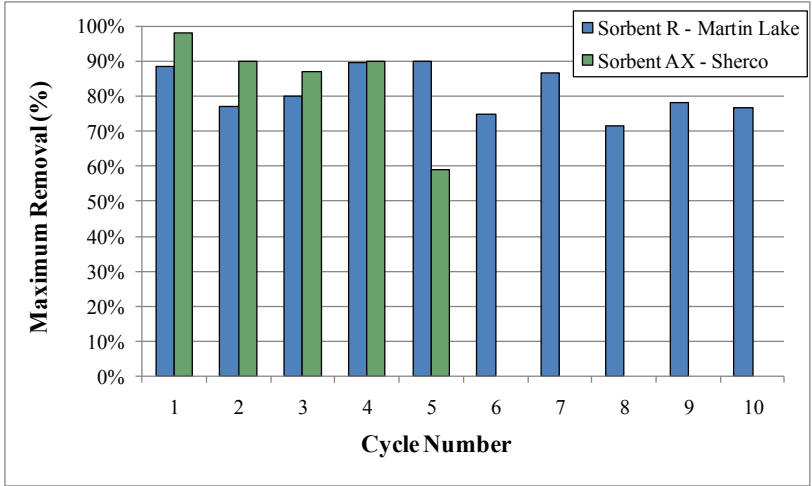


Figure 4. Maximum CO₂ Removal for Sorbent R at Martin Lake and Sorbent AX at Sherco

The 1 kW pilot was used to effectively compare different sorbents under the same operating conditions. However, it was not possible to maintain 90% continuous CO₂ capture with any of the sorbents tested to date using such a configuration. Two theoretical isotherms and the 1 kW pilot configuration are provided together in Figure 5 to facilitate explanation of why it was difficult to achieve high removal levels in the 1 kW pilot with the sorbents evaluated in the screening program.

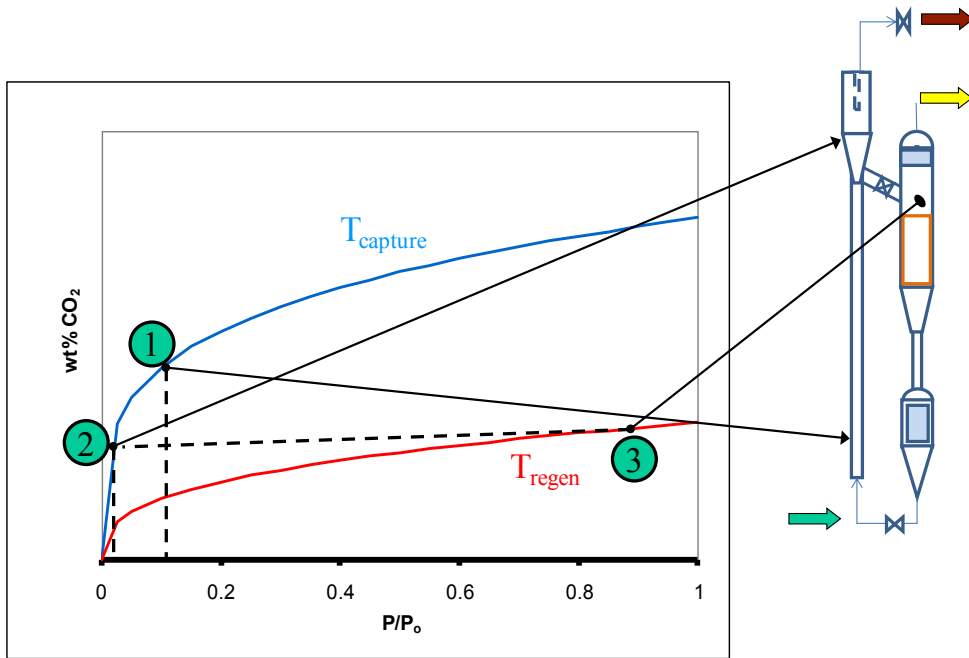


Figure 5. 1 kW Pilot with Theoretical Isotherms

The two isotherms provided in

Figure 5 are theoretical, but are similar in shape to what has been measured previously for supported amine sorbents. The y-axis corresponds to the equilibrium CO₂ capacity (in wt%) of a sorbent and the x-axis corresponds to the partial pressure of CO₂ in the gas. The blue isotherm signifies the relationship between sorbent CO₂ loading and CO₂ partial pressure in the gas at the adsorption/capture temperature, while the red isotherm represents the same relationship at the regeneration temperature. The 1 kW pilot consisted of a riser (i.e. transport reactor) for adsorption. Therefore, the maximum driving force for CO₂ uptake to the sorbent is at the bottom of the riser where the flue gas CO₂ concentration was the greatest (point 1). As the sorbent began to remove the CO₂ from the gas, the driving force for the sorbent to continue removing CO₂ decreased because the partial pressure of CO₂ in the gas was decreasing. At the top of the riser the partial pressure of CO₂ in the gas was lower than that at the bottom, thus, the equilibrium CO₂ loading of the sorbent was lower (point 2). A fluidized bed with a downcomer tube was used for the regenerator. Ideally when the sorbent moved from the riser to the regenerator it would release CO₂ due to only a temperature swing with only CO₂ as the fluidization gas. Because the regenerator was a slow bubbling fluidized bed the sorbent was not always in contact with N₂. To regenerate quickly in this type of fluidized bed the sorbent would have to regenerate in the presence of pure CO₂. Although different sorbent isotherms demonstrate that some sorbents regenerate in the presence of CO₂, this will only occur if the CO₂ loading during adsorption was high enough to create a driving force for high partial pressure regeneration. When comparing the CO₂ loading on the sorbent at the top of the riser (point 2) to the CO₂ loading in the regenerator (point 3) there is no driving force for regeneration. Therefore, the regeneration was slow as the sorbent only regenerated when in the presence of the fluidization N₂. The sorbents evaluated in the screening program cannot be used to maintain 90% CO₂ removal in this type of adsorption/regeneration system. However, the system was still effective at comparing the removal level for different materials.

Equipment Cost Analysis

Using the methods described in the Experimental section, a conceptual design for a commercial-scale solid-based CO₂ capture process was selected by Stantec. This conceptual design is not proposed as a final commercial option, but rather one potential design that can be used to identify cost drivers and direct future development work. The conceptual flow sheet provided in Figure 6 shows how the selected equipment could be arranged in a power plant to capture carbon dioxide from flue gas.

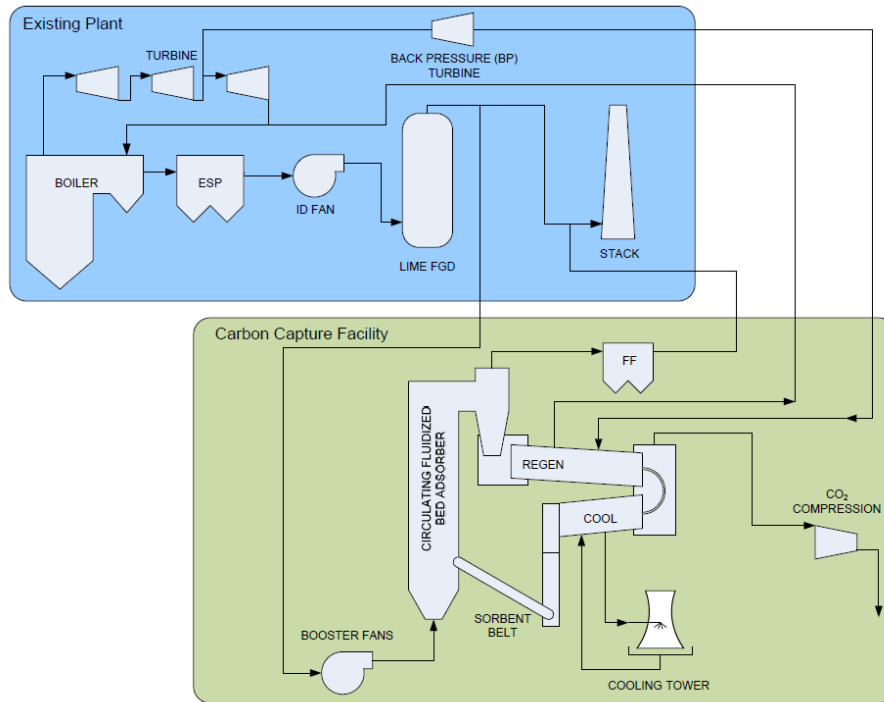


Figure 6. Conceptual Flow Sheet

In this conceptual arrangement, flue gas is sourced from the existing unit after particulate removal. It then proceeds through a heat recovery device, where the flue gas is cooled. The recovered heat is incorporated with the regeneration of the sorbent. The cooled flue gas then enters a wet FGD system for desulphurization and additional cooling. The pretreated gas then proceeds through a booster fan, then through a vertically arranged adsorber. Cooled solid sorbent is introduced to the bottom of the adsorber, and the flue gas carries it upwards in a circulating fluidized bed (i.e. transport reactor).

Carbon dioxide is adsorbed by the solid particles (either through a physical reaction, a chemical reaction, or a combination of the two), which are separated from the flue gas by a cyclone. The flue gas then proceeds back to stack, while the solids are directed towards regeneration. A rotary kiln, equipped with a jacketed shell to provide heat, conveys the solids slowly away from the adsorber, and then back. The carbon dioxide is driven off as the adsorption reaction is reversed and is withdrawn for compression. A final kiln is jacketed for cooling, which prepares the sorbent for reintroduction to the adsorber.

Stantec used the process shown in Figure 6 to evaluate the capital costs, operating costs, footprint, and other important criteria for a commercial-scale power plant that is retrofit for CO₂ capture. The power plant where the technology was implemented was AEP's Conesville plant, which was selected so that direct comparisons could be made to MEA using a DOE NETL report.³ A comparison of the contribution to the increase in the levelized cost of electricity (LCOE) is provided in Figure 7. The LCOE increases have been normalized to the NETL base case for MEA for comparison.

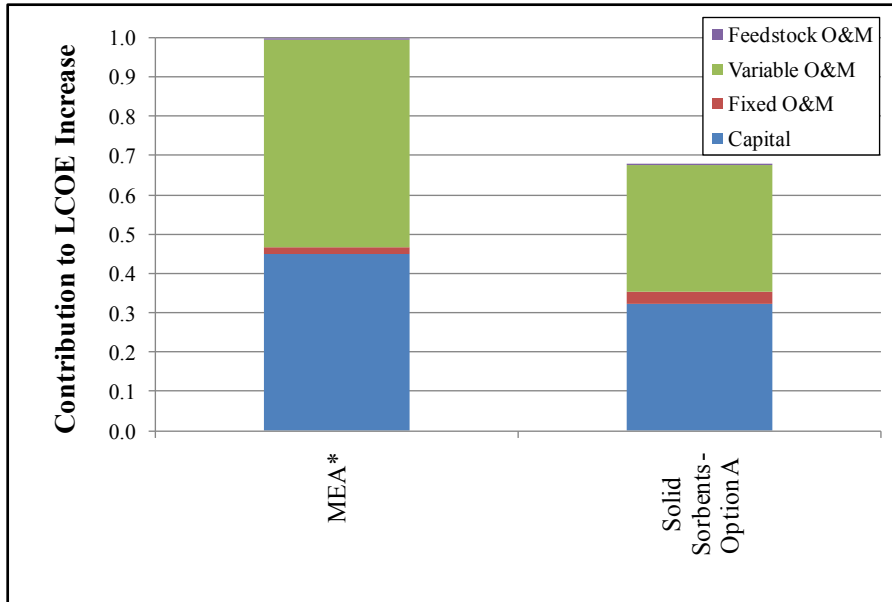


Figure 7. Contribution of LCOE Increases for CO₂ Capture Options at AEP's Conesville Power Plant (*MEA Costs³ from NETL, 2007 with costs scaled to 2010 dollars)

Both solid sorbent cases were over 30% lower than that of the MEA capture system. This can be attributed to the significant reduction of steam for the solid-based capture systems. Although reducing the energy penalty is crucial, the capital costs also play an important role in the overall increase in the LCOE. Stantec identified the cost drivers in order of importance as follows:

Capital Costs > Operating Costs >> Sorbent Replacement > Employees

The sorbent-based capture options yielded significant energy penalty and cost savings versus an aqueous amine system. In fact, for a sorbent-based CO₂ capture process without a CO₂ laden/lean sorbent heat exchanger the LCOE increase was over 30% lower than that of the MEA capture process. However, this cost savings was not currently enough to meet the DOE's target of <35% increase in LCOE. The cost drivers were also identified during this study.

1 MW Design

The laboratory results were used to select the sorbent that is the design basis and will be tested using the 1 MW pilot. The results from the 1 kW pilot were used to determine the key properties used to down select the processes considered for the 1 MW pilot. The high level equipment cost analysis identified the cost drivers for the overall sorbent based CO₂ capture process, which also fed into the key selection properties for the 1 MW pilot. In addition the 1 kW pilot provided important operational information regarding entrained flow reactors being considered for full-scale design. Using all this information collected from the viability assessment project, ADA worked with Shaw and Stantec to evaluate several different potential reactor designs for the 1 MW pilot. The different designs were developed and compared based on 1) technical feasibility and 2) total cost for CO₂ capture (includes both capital and operating costs). The three main processes that were considered for the 1 MW pilot included:

- Transport reactors (entrained flow)

- Moving beds
- Staged fluidized beds

All three options offered distinct benefits and disadvantages. Based on the technology level of development and estimated costs, staged fluidized beds (SFB) were selected for the full-scale conceptual design and subsequently the 1 MW pilot. Figure 8 is a simplified schematic of the SFB process design. While the SFB is utilized for the CO₂ adsorption and gas/solids contacting, a fluidized bed is also utilized for sorbent regeneration. Heat is generated during the adsorption phase due to the exothermic reaction between the sorbent and CO₂ in the flue gas stream. The cooling coils inside the adsorber module are used to remove the heat generated during adsorption and maintain an acceptable operating temperature. Once the sorbent particle is loaded with CO₂, the sorbent is discharged from the adsorber module and conveyed to a regeneration module where the solid sorbent particle is heated to liberate the CO₂. The gaseous CO₂ is thermally driven off the sorbent particle and then sent to a CO₂ compressor where it is compressed and sent to a pipeline for sequestration. The regeneration module in the configuration shown consists of only a single fluidized bed. The fluidization media could be either recycled CO₂, steam, or a mixture of thereof. It is possible that more than one stage could be used for the regenerator as well. The heating coils inside the regenerator module are used to maintain the desired operating temperature inside the regenerator.

The key characteristics of this process include:

- Advantages
 - Low mass transfer limitations
 - Heat transfer (near isothermal operation possible)
 - Equipment components have been demonstrated successfully on the required scale
 - Effectively counter current flow (for a high number of stages)
- Disadvantages
 - Pressure drop across the process trains
 - Multiple process trains required
 - Sorbent entrainment

Based on the simplified SFB design shown in Figure 8, a 1 MW pilot is currently being designed and will be evaluated using a supported amine sorbent. Current project schedule indicates pilot fabrication and installation will begin 2QTR2012 and pilot testing and operation will begin 4QTR2013.

While the project team has used data from the viability assessment phase as well as laboratory data from our current pilot project, there remains several critical pieces of information and field data to be collected which will provide additional information as to the technical and economic feasibility for this technology. Below is a list of focus areas for the 1 MW pilot, where process information will be collected using flue gas from a PC boiler and the test device will be of reasonable size to accurately scale process information to full-scale design (i.e., 550 MW_e)

- Sorbent attrition
 - Physical and chemical attrition
- Validate regeneration energy requirement
- Measure actual process temperatures to maintain 90% CO₂ capture
- Determine CO₂ purity in regenerator exhaust stream
- Measure the time required for sorbent regeneration
- Optimize process operating variables
 - Select process temperatures
 - Sorbent circulation rates

- Measure volatile emissions

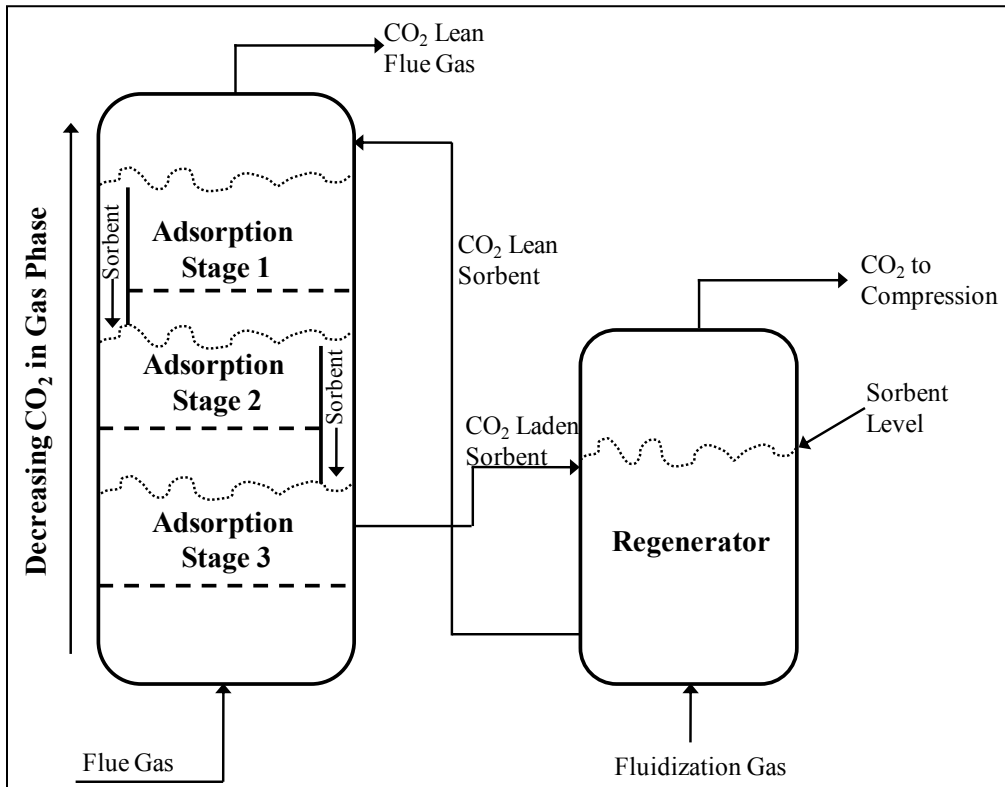


Figure 8. Process Sketch of a Potential Configuration of the Invention

CONCLUSIONS

A solid sorbent based process offers the potential to significantly reduce the energy penalty, and thus the overall cost, associated with post-combustion CO₂ capture. However, solid sorbents are still at a relatively early stage of development. ADA completed a concurrent approach to assessing the viability and accelerating development of solid sorbents for this application and used the results of this project to select a sorbent and design for a 1 MW pilot on a subsequent effort. The conclusions from the different aspects of the project are listed below.

- Viability Assessment
 - Materials Evaluation – Laboratory Scale
 - Supported amine sorbents were selected as the best sorbent type for post-combustion CO₂ capture
 - A specific supported amine sorbent was selected for the 1 MW pilot testing that demonstrated an acceptable working capacity when regenerated under high CO₂ partial pressures
 - Materials Evaluation – 1 kW Pilot
 - Over 90% CO₂ removal from actual flue gas was repeatedly achieved at the 1 kW scale in batch mode using a supported amine sorbent
 - A counter-current contactor would be a preferable configuration for testing at an increased scale in order to maximize sorbent working capacity

- Commercial-Scale Equipment Evaluation
 - Cost analysis of one potential conceptual design has shown the potential for a significant reduction in costs versus state of the art aqueous amines at the same retrofit power plant
 - Capital costs and energy penalty are both important cost drivers
 - If the DOE's goals of <35% increase in LCOE are to be achieved further cost savings will be necessary
- 1 MW Pilot Design
 - Design selection for the 1 MW pilot included a staged fluidized bed for adsorption and a fluidized bed for regeneration.

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