

Executive Summary

Carbon Capture Scientific, LLC (CCS), CONSOL Energy Inc. (CONSOL), Nexant Inc. (NEXANT), and Western Kentucky University (WKU) have been working together to develop a novel Gas Pressurized Stripping (GPS) process for post-combustion CO₂ capture. Cooperative Agreement DE-FE0007567 was awarded on October 1, 2011 and has a planned end date of September 30, 2015.

This project was designed to combine experimental and simulation data to evaluate the GPS process at the lab and bench scale. The techno-economic analysis of the GPS process follows the standards developed by the National Energy Technology Laboratory (NETL) to evaluate performance of the technology.

Before the bench-scale test at National Carbon Capture Center (NCCC), CCS has conducted computer simulation and experimental tests to evaluate the GPS technology, including 1) GPS column simulation and optimization, 2) optimization of GPS process for an existing plant, 3) simulation of alternative separation methods for replacing the second absorption cycle, 4) phase equilibrium data measurement, 5) individual lab-scale tests for absorption column, GPS column, and second absorption column, 6) solvent stability and corrosion tests at high loading and high temperature and 7) GPS process techno-economic analysis. These computer simulation and experimental tests provided all necessary design data for the further continuous system tests carried out at NCCC.

With the experimental and computer simulation data, CCS collaborated with Ascension Industries (fabrication vendor in Buffalo, NY), Allied Circuits and the National Carbon Capture Center (NCCC) to complete the design and fabrication of the 500 SLPM skid-mounted, column-based GPS bench unit, which meets the safety requirements regulated by the NCCC host site. The skid has dimensions of 10'6" length and 8' width. The absorber has 8" ID and 32' height and the GPS column has 6" ID and 30' height. The skid was equipped with PLC control system and able to run without manual intervention.

Fabrication of the skid was completed in early May 2014, followed by the preliminary factory acceptance testing conducted at Ascension site in the same month. The skid was shipped from Ascension Industries to NCCC in June, 2014. The relevant documents for the skid, including the testing reports, owner's manual and equipment data sheet, were completed accordingly by the CCS team.

The installation of the skid was completed in early August 2014, followed by commissioning and shakedown test. CCS team provided engineering support to NCCC for the installation of the skid. After the shakedown test, CCS team engaged in the parametric test and long term test.

An amine-based CCS proprietary blended solvent was used in all the parametric and long term tests. Parametric tests focused on investigating the effects of the following factors on the GPS process performance:

- total flue gas flow rate;
- gas/liquid (G/L) volume ratio;
- stripper operating pressure;
- stripping gas (N₂) flow rate.

The performance was evaluated by the CO₂ removal rate in the absorber and CO₂ product pressure and purity from the stripper. Parametric test results showed that:

- with the increase of total flue gas flow rate, the CO₂ removal rate in the absorber reduced while CO₂ purity from the stripper reduced slightly;
- with the increase of G/L volume ratio, the CO₂ removal rate reduced and CO₂ purity was not affected;
- with the increase of stripper operating pressure, both the CO₂ product purity from the stripper and CO₂ removal rate in the absorber reduced;
- with the increase of N₂ flow rate, the CO₂ product purity reduced but CO₂ removal rate increased.

These test results were expected and agreed with those obtained from the lab scale individual unit tests and the computer simulation. During the parametric tests, it was also found that the fluctuations of the absorber and stripper performances became small after the system operation reaches steady state. All the data was recorded when the system reached steady state.

After the parametric tests, the skid was transitioned to the long-term test and 24-hour operation mode. The operating conditions were maintained at which 90% CO₂ removal rate in the absorber is achieved and CO₂ product with 95% purity from the stripper is produced. These operating conditions were determined from the parametric tests. The pressure in the stripper was kept around 6 bar. During the parametric and long-term tests at NCCC, the total accumulated operating hours were 1437 hours, among which 1145 hours were conducted with coal derived flue gas.

Energy consumption test was conducted to evaluate the performance of the GPS technology as shown in Table 1. Since the sensible heat strongly depends on the selection of a heat exchanger, the tests were designed only to measure the reaction heat and the stripping heat. The sensible heat of the solvent can be estimated based on the working capacity and specific heat of the rich and lean solutions. The estimated sensible heat was in the range of 250 to 500 kJ/kg captured CO_2 .

| Solvent flow rate (GPM) | Flue gas flow rate (SCFM) | Stripper pressure (bar) | N ₂ flow rate (slpm) | The sum of reaction heat and stripping heat (kJ/ kg captured CO ₂) |
|-------------------------|---------------------------|-------------------------|---------------------------------|--|
| 0.89 | 14.5 | 6 | 2.59 | 1,600 |
| 1.10 | 17.8 | 5.8 | 2.63 | 1,562 |
| 0.57 | 11.6 | 6 | 1.67 | 1,597 |

Table E1. Energy consumption test results

During the long term tests, solvent loss was also measured. However, since the bench-scale skid system was not equipped with a solvent recovery system, the solvent loss was high.

The long-term test was terminated in early August, 2015 and followed by the decommissioning of the skid. Decommissioning of the skid was completed in early September, 2015.

Both parametric tests and long term tests at NCCC have demonstrated that GPS process is able to achieve 90% CO₂ removal from typical coal derived flue gas. GPS process is also able to

produce high pressure CO_2 product with required purity. Additionally, the energy consumption of the GPS process is much lower than that of the DOE MEA baseline case. Reviewing the qualified data acquired from the bench-scale skid test, it is reasonable to pursue pilot-scale test for GPS technology.

1. Background

With the awarded DOE funding (DE-FE0007567), Carbon Capture Scientific, LLC (CCS) took the lead to develop a breakthrough Gas Pressurized Stripping (GPS) process-based technology for post-combustion CO₂ capture. The project team includes Carbon Capture Scientific, LLC, CONSOL Energy Inc. (CONSOL), Nexant Inc. (NEXANT) and Western Kentucky University (WKU). Through a comprehensive series of computer simulations, lab-scale experimental work, and techno-economic analyses, the proposed project will acquire all of the information required to advance the technology into pilot scale.

The main objectives of the whole project include: 1) bench-scale tests of individual process units to document experimental results and obtain necessary information to progress the technology to the pilot scale; 2) computer simulations to maximize the benefit of the GPS technology for existing power plants; 3) experimental investigation of selected solvents to minimize the economic risk of the proposed technology; 4) lab-scale testing of a rotating packed bed (RPB) at anticipated absorption and stripping operating conditions to evaluate performance and the potential to reduce GPS system capital costs; and 5) design, fabrication, and operating a bench-scale unit capable of processing about 500 standard liter per minute (slpm) actual coal-derived flue gas in conventional column-based GPS system at the National Carbon Capture Center (NCCC).

For the test at NCCC, the objectives are: 1) validation of the GPS process simulation results with experiments under real flue gas; 2) investigation of the influence of the operating parameters on GPS process performance; 3) experimentally measurement of energy consumed for GPS process; 4) observation of the GPS skid performance for long-term operation.

2. Bench-scale GPS test unit

The flow diagram of the bench-scale GPS test unit is shown in Fig. 2.1. GPS process is an innovative patented technology (US Patent 8425655). It has an absorber and a stripper. Both absorber and stripper have two inter-stage heat exchangers for either cooling or heating. CO₂ is absorbed from the flue gas in the absorber and released at elevated pressure with high purity in the stripper.

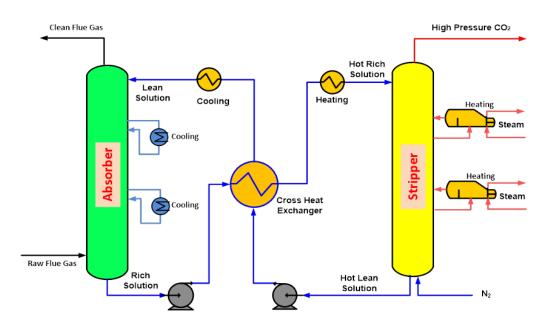


Figure 2.1. The flow diagram of the bench-scale GPS test unit

The whole GPS skid is mounted on an 8'x10'6" steel base. To protect the heat exchangers and pumps in the outdoor condition, a guard, made of large stainless metal panels, is installed to enclose them inside.

As shown in Fig. 2.2, the absorption column (blue) and stripping column (silver) are two key components in the GPS skid mounted system. Both columns are ASME certified vessels. And 3/8 inch stainless steel Pall ring packing is used for both columns. The specifications of the columns are listed in Table 2.1. There are three sections of packing in series in the absorber. The absorber has two inter-stage cooling sections connecting the three packing sections. These sections are connected by flanges. The inter-stage cooling sections have liquid collectors, heat exchangers and redistributors. The liquid from upper packing sections is collected by the liquid collectors, flows through the heat exchanger to be cooled down, and then is sprayed into the lower packing section by the redistributors. For the stripper, there are also three sections of packing in series. The stripper has two inter-stage heating sections connecting the three packing sections. The design of the inter-stage sections is the same as the absorber. The operating pressure in stripper can go up to 150 psi at temperature of 257 F.





Figure 2.2. The pictures of GPS skid

Table 2.1. Specifications of the absorber and stripper

| | Absorber | Stripper |
|--------------------------------|----------|----------|
| ID (inch) | 8 | 6 |
| Total height of packing (foot) | 23 | 21 |
| Total height of column (foot) | 32 | 30 |

The GPS skid electrical system is comprised of two panels -- PLC panel with touch screen and data logger, and the electrical panel for motor drivers and the transformer. Many operating parameters are controlled via the PLC panel, including the incoming flue gas flow rate, the solvent flow rate, the solvent levels at the inter-stage liquid collectors, the solvent temperatures in the cooling/heating loops. The

stripper pressure is controlled by a back pressure valve. The nitrogen flow rate into the stripper is controlled by a manual valve.

3. Experimental results

The experiments included parametric tests, energy consumption measurement experiments and long-term tests.

NCCC provided crucial support for the GPS skid tests. NCCC lab measured the gas concentrations at the inlet and outlet of the absorber and outlet of the stripper. The gas concentration analyses were continuous when the skid was running. The data was recorded by NCCC's computer. Additionally, NCCC lab analyzed the solvent concentrations for representative samples collected by CCS team.

3.1. Parametric test

The skid ran quite stable after it reached a steady state, which is evident by the CO₂ concentration measurements shown in Fig. 3.1.1. For parametric test, the data was recorded after the skid reached steady state for new conditions.

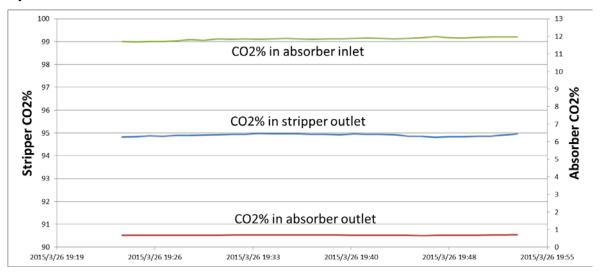


Fig. 3.1.1. CO₂ concentrations under a steady state on March 26

Parametric tests investigated the influences of total flue gas flow rate, gas/liquid volume ratio, stripper operating pressure and N_2 flow rate on the GPS system performance. The performance was characterized by the CO_2 removal rate in the absorber and CO_2 product pressure and purity from the stripper. The CO_2 removal rate was calculated based on the average CO_2 concentration at the absorber inlet and outlet and, the CO_2 purity was also based on the averaged CO_2 concentration at stripper outlet.

The influence of the total flue gas flow rate on the bench unit performance of GPS process is illustrated in Fig. 3.1.2. In these tests, the gas/liquid volume ratio was kept at 145 and the stripper operating pressure was at 6.1 bar. The nitrogen flow rate into the stripper was adjusted proportionally with the flue gas flow rate. With the increase of total flue gas flow rate, the CO₂ removal rate in the absorber reduced obviously while CO₂ purity from the stripper reduced slightly. As the flue gas and solvent flow rate increased, the residence time in absorber reduced. Less reaction time led to lower reaction conversion, so that CO₂ removal rate decreased. The height of stripper packing was more over-designed than that of absorber. Additionally, the stripping reaction was faster than absorption reaction due to its higher temperature. Thus, reduced reaction time had less influence on conversion in stripper than that in absorber, which explained why the reduction of CO₂ purity is less significant.

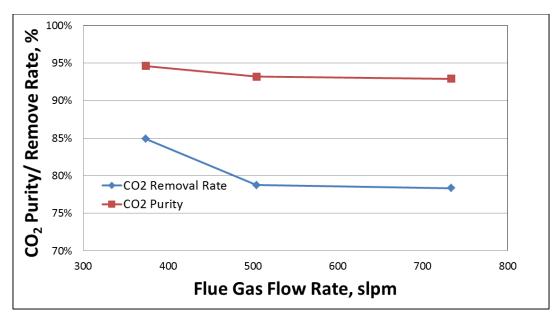


Figure 3.1.2. Influence of total flue gas flow rate on the performance of GPS technology

The effect of the gas/liquid volume ratio on the performance of GPS process is shown in Fig. 3.1.3. The stripper operating pressure, the solvent flow rate and the nitrogen flow rate into the stripper were kept at about 6 bar, 0.89 GPM and 2.35 slpm, respectively for all tested cases. The G/L volume ratio was changed by changing the flue gas flow rate and keeping the solvent flow rate constant. With increase of G/L volume ratio, the CO₂ removal rate reduced significantly but CO₂ purity was not affected. As the G/L volume ratio increased, the amount of flue gas processed per kg of solvent increased. In this case, to achieve the same the CO₂ removal rate as the low G/L volume ratio, the height of absorber packing needed to be increased, because the required number of transfer unit increased when G/L volume ratio increased. However, since the same absorber was used in these experiments, CO₂ removal rate is reduced. On the other hand, due to the fact that the operating conditions in the stripper were kept the same as G/L volume ratio increased, there was small variation in CO₂ purity from the stripper.

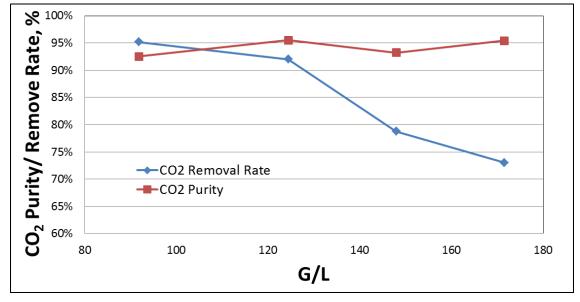


Figure 3.1.3. Influence of G/L ratio on the performance of GPS technology

The influence of the stripper operating pressure on the performance of GPS process is shown in Fig. 3.1.4. The flue gas flow rate and solvent flow rate were maintained at about 455 slpm (16 SCFM) and 0.95 GPM, respectively. During the experiments, the N₂ flow rate was increased accordingly to ensure the lean loading was still low enough to achieve over 90% removal rate in the absorber. With the increase of stripper operating pressure, both the CO₂ purity from the stripper and CO₂ removal rate in the absorber reduced. Because the CO₂ equilibrium partial pressure was a strong function of temperature and the stripper temperature was kept the same for the four tests, CO₂ purity at the top of the stripper decreased when the total pressure is increased. As the increase of stripper operating pressure, the lean loading of the solvent at the outlet of stripper increased in order to maintain a higher equilibrium partial pressure of CO₂. Higher lean loading solvent compromised the performance in the absorber and as a result CO₂ removal rate is reduced.

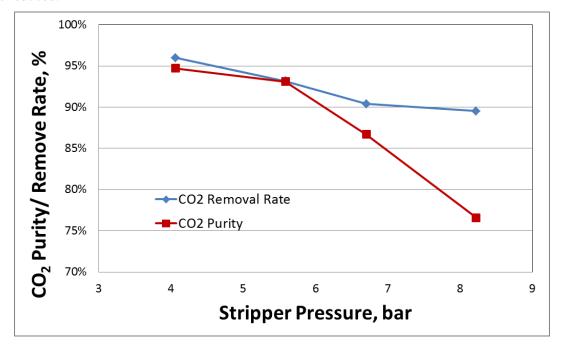


Figure 3.1.4. Influence of stripper operating pressure on the performance of GPS process

The effect of the N_2 flow rate on the performance of GPS technology is shown in Fig. 3.1.5. In the experiments, the flue gas flow rate, stripper operating pressure and the solvent flow rate were kept about 500 slpm (18 SCFM), 5.9 bar and 1.09 GPM, respectively. With the increase of N_2 flow rate, the CO_2 purity reduced but CO_2 removal rate increased. As the N_2 flow rate increased, the lean loading of the solvent at the outlet of stripper decreased due to the lower CO_2 % at the bottom of the stripper. The solvent with lower lean loading enhanced the performance in the absorber subsequently. In the meantime, owing to the dilution of N_2 , CO_2 purity from the stripper reduced.

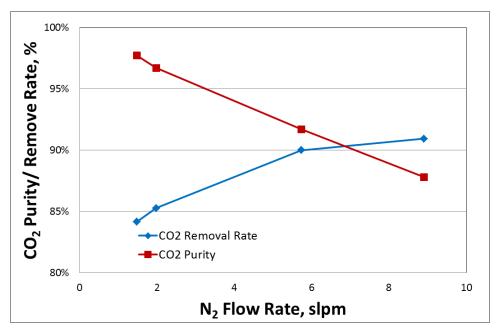


Figure 3.1.5. Influence of N₂ flow rate on the performance of GPS technology

3.2. Energy consumption tests

The simulation results showed GPS technology consumed much less energy than the DOE MEA baseline case. Energy consumption test was conducted to validate the simulation. Since the sensible heat amount strongly depends on the selection of a heat exchanger, the tests were designed only to measure the reaction heat and the stripping heat. The sensible heat of the solvent can be estimated based on the working capacity and specific heat of the rich and lean solvents. The estimated sensible heat was in the range of 250 to 500 kJ/kg captured CO₂. To measure the actual reaction and stripping heat, the steam usage data was obtained after the skid reached steady state. The net steam usage counted for sum of reaction and stripping heat was the difference between the steam usage when the skid was operated with flue gas flow and without flue gas flow. The steam usage for the skid operation without flue gas flow is actually the sum of sensible heat and heat loss. The results were listed in Table 3.2.1. The reaction and stripping heat was 1,562~1,600 kJ/kg captured CO₂. These results were very close to the result obtained from computer simulation, which was 1570 kJ/kg captured CO₂.

| | | - | - | | | |
|-------------------------|---------------------------|-----|------|--|--|--|
| Solvent flow rate (GPM) | Flue gas flow rate (SCFM) | - | | The sum of reaction heat and stripping heat (kJ/ kg captured CO ₂) | | |
| 0.89 | 14.5 | 6 | 2.59 | 1,600 | | |
| 1.10 | 17.8 | 5.8 | 2.63 | 1,562 | | |
| 0.57 | 11.6 | 6 | 1.67 | 1,597 | | |

Table 3.2.1. Energy consumption test results

3.3. Long-term tests

For the long-term test, the skid was set to run at a condition that could achieve 90% CO₂ removal rate in absorber and 95% purity CO₂ product from stripper proved in parametric test. The incoming flue gas and the solvent flow rates were 330 slpm and 0.56 GPM, respectively. That made the G/L ratio as 156. The

pressure in stripper was 6 bar. The skid was operated in 24-hour mode unless it was shut down for maintenance or trouble-shooting. The long-term test started from late May and stopped in early August. The operation was fairly stable and a representative CO₂ concentration data in a 24-hour period was shown in Fig. 3.3.1.

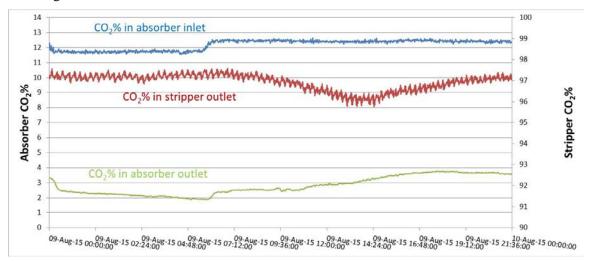


Figure 3.3.1. Gas analysis data on August 9

In the summer time, the cooling water temperature was relatively high compared to other seasons. The heat exchangers installed on the absorber could hardly cool the solvent to the target low temperature. That is why the performance was not as good as that in spring. In addition, the cooling water temperature was always low in early morning and high in the afternoon. That caused the CO_2 removal rate in early morning was higher than that in the afternoon.

The solvent loss test accounted for the loss for both water and amine. The loss rate was calculated from the historical data of the lean tank solvent level over a period of time. Based on the lean tank solvent level reading at 19:00 from July 27 to 30, when the skid continuously ran, the average solvent net loss was 2.9 kg/day. Based on the lean tank solvent level reading at 7:00 from August 7 to 10, the average solvent net loss was 3.2 kg/day. Since the bench-scale skid system was not equipped with a solvent recovery system, the solvent loss rate was high.

To investigate the influence of solvent loss on its concentration, CCS team took sample regularly and had NCCC lab to analyze the representative samples for piperazine and water concentration. Table 3.3.1 showed the test results in a period that no make-up solvent was added. The data showed that the piperazine concentration increased gradually and water concentration decreased gradually over a long period of time. Obviously, the loss rate of water was greater than that of piperazine. Thus, the water concentration reduced when the total amount of solvent reduced in the same time.

| Table 3.3.1. Test results | s of pip | erazine and | watei | concen | tration | during l | ong-term t | est |
|---------------------------|----------|-------------|-------|--------|---------|----------|------------|-----|
| | ъ. | D: . | .0/ | *** | . 0/ | | | |

| Date | Piperazine wt% | Water wt % | | |
|------|----------------|------------|--|--|
| 7/28 | 18.2% | 39.6% | | |
| 7/30 | 19.0% | 38.2% | | |
| 8/6 | 19.7% | N/A | | |
| 8/7 | 20.4% | 34.8% | | |

During the parametric and long-term tests at NCCC, the total accumulated operating hours were 1437 hours, among which 1145 hours were with coal derived flue gas.

4. Conclusions

The tests of the bench-scale GPS skid at NCCC achieved the original goals. The parametric tests validated the influences of operating parameters on GPS process performance, which were simulated before the tests. The long-term test showed the skid could run continuously for a long period of time. The results have demonstrated that GPS process is able to achieve 90% CO₂ removal from typical coal derived flue gas. GPS process is also able to produce high pressure CO₂ product with required purity. Additionally, the energy consumption of the GPS process is much lower than that of the DOE MEA baseline case.

Reviewing the qualified data acquired from the bench-scale skid test, it is reasonable to pursue pilot-scale test for GPS technology.