

Design of Bench-Scale Silicone Process for Low-Cost CO₂ Capture

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Abstract:

The major goal of the project is to design and optimize a bench-scale process for novel silicone CO₂-capture solvents and establish scalability and potential for commercialization of post-combustion capture of CO₂ from coal-fired power plants. This system should be capable of 90% capture efficiency and demonstrate that less than 35% increase in the cost of energy services can be achieved upon scale-up. Experiments were conducted to obtain data required for design of the major unit operations. The bench-scale system design has been completed, including sizing of major unit operations and the development of a detailed Process and Instrument Diagram (P&ID). The system has been designed to be able to operate over a wide range of process conditions so that the effect of various process variables on performance can be determined. To facilitate flexibility in operation, the absorption column has been designed in a modular manner, so that the height of the column can be varied. The desorber has also been designed to allow for a range of residence times, temperatures, and pressures. The system will be fabricated at Techniserv Inc.

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Executive Summary

The major goal of the project is to design and optimize a bench-scale process for novel silicone CO₂-capture solvents and establish scalability and potential for commercialization of post-combustion capture of CO₂ from coal-fired power plants. This system should be capable of 90% capture efficiency and demonstrate that less than 35% increase in the cost of energy services can be achieved upon scale-up. Experiments were conducted to obtain data required for design of the major unit operations. The bench-scale system design has been completed, including sizing of major unit operations and the development of a detailed Process and Instrument Diagram (P&ID). The system has been designed to be able to operate over a wide range of process conditions so that the effect of various process variables on performance can be determined. To facilitate flexibility in operation, the absorption column has been designed in a modular manner, so that the height of the column can be varied. The desorber has also been designed to allow for a range of residence times, temperatures, and pressures. The system will be fabricated at Techniserv Inc.

The system consists of a gasoline generator that produces exhaust gas as a proxy for the flue gas from a coal-fired power plant. The exhaust from this generator is chilled in a heat exchanger to lower the water content to that typical of the flue gas from a coal-fired power plant. The exhaust can then be mixed with a variety of gases, such as SO₂, supplied from a gas manifold, to adjust the composition to match the flue gas from coal. The mixture is then heated to 40 to 60 °C. The exhaust, with a flow rate of 50-200 SLPM, is sent to the bottom of a column, where it flows up, countercurrent to the aminosilicone solution at 1-2 L/min, and CO₂ is captured. The column is designed in a modular fashion, so that the height of the column can be adjusted, and a variety of different packing materials can be tested. The stripped exhaust exits the top of the column and is sent to a mass spec and CO₂ analyzer for compositional analysis. The rich solvent leaves the bottom of the column, and is pumped to the high pressure desorber. The required size of the absorption column was estimated to have a diameter of 0.3 meters and a height of 3 meters. Different packing materials will be investigated to determine the effect on CO₂ capture and column pressure drop.

The desorber is a jacketed, high-pressure, continuous, stirred-tank reactor with a total volume of 30 liters and a nominal working volume of 15 liters. The reactor is designed to withstand 300 psig and 300 °C. It has a recirculation loop, with a heat exchanger. The recirculation loop is designed to provide additional heat input to that provided by the jacketing on the tank. In addition, the recirculation will be used to increase mass transfer in the reactor. The lean sorbent leaves the bottom of the desorber and is cooled, dropped in pressure, and sent to a storage tank, before being sent back to the top of the column. The CO₂ produced in the desorber is throttled down in pressure and sent to gas analysis. The system will be fully automated, with detailed measurements of all important process variables, including temperatures and pressures.

The heat of absorption of CO₂ in 60/40 (wt/wt) GAP-1/TEG was required to determine the energy requirements for the absorber and desorber. The heat of absorption of 60/40 (wt/wt) GAP-1/TEG was measured to be 2265 ± 75 kJ/kg CO₂ at 40 °C.

Completion of Task 3.1: Design Bench-Scale System

During the first two quarters of 2012, the conceptual design of a bench-scale system to utilize aminosilicones for CO₂-capture has been developed. The detailed design of this system was then conducted by GE Global Research working with Techniserv, Inc., who will build the bench-scale system. In order to complete the detailed design, experiments have been conducted to obtain information, such as kinetic rates and heats of reaction, necessary for the sizing of the main unit operations. The design of the main unit operations have been completed, and the auxiliary equipment, such as control valves, tubing size and type, and process monitoring equipment have been specified.

Design of Bench-Scale System

Figure 1 shows the detailed Piping and Instrumentation Diagram (P&ID) for the bench-scale system. The system consists of a gasoline generator that produces exhaust gas as a proxy for the flue gas from a coal-fired power plant. The exhaust from this generator is chilled in a heat exchanger to lower the water content to that typical of the flue gas from a coal-fired power plant. The exhaust can then be mixed with a variety of gases, such as SO₂, supplied from a gas manifold, to adjust the composition to match the flue gas from coal. The mixture is then heated to 40 to 60 °C. The exhaust, with a flow rate of 50-200 SLPM, is sent to the bottom of a column, where it flows up, countercurrent to the aminosilicone solution at 1-2 L/min, which captures CO₂. The column is designed in a modular fashion, so that the height of the column can be adjusted, and a variety of different packing materials can be tested. The stripped exhaust exits the top of the column and is sent to a mass spec and CO₂ analyzer for compositional analysis. The rich solvent leaves the bottom of the column, and is pumped to the high pressure desorber.

The desorber is a jacketed, high-pressure, continuous, stirred-tank reactor. It has a recirculation loop, with a heat exchanger. The recirculation loop is designed to provide additional heat input to that provided by the jacketing on the tank. In addition, the recirculation will be used to increase mass transfer in the reactor. The lean sorbent leaves the bottom of the desorber and is cooled, dropped in pressure, and sent to a storage tank, before being sent back to the top of the column. The CO₂ produced in the desorber is throttled down in pressure and sent to gas analysis. The system will be fully automated, with detailed measurements of all important process variables, including temperatures and pressures.

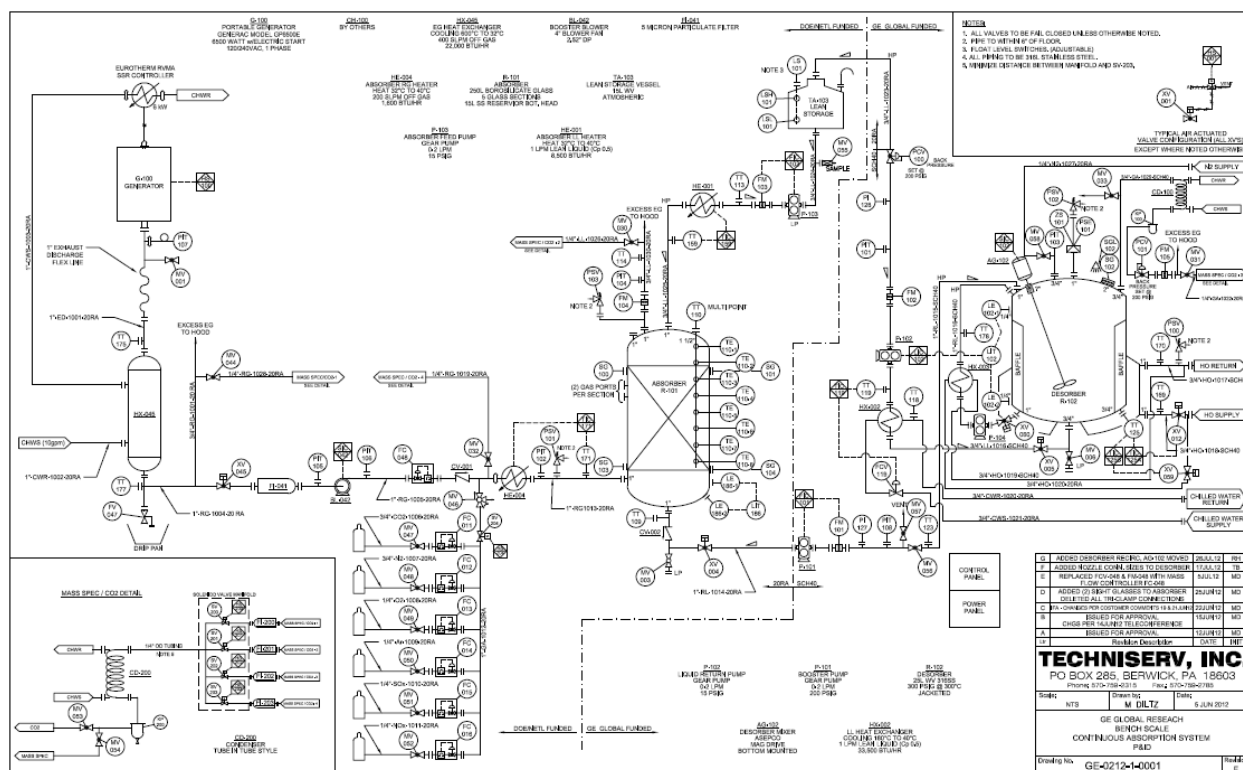


Figure 1. The detailed P&ID for the bench-scale system.

Absorber Design

Equilibrium Loading

The equilibrium loading measurements of CO₂ in a 60/40 (wt/wt) GAP-1/TEG solution were completed as a function of temperature and partial pressure of CO₂. These data were required for sizing the bench-scale absorption column. The temperatures studied ranged from 30 °C to 75 °C and the CO₂ partial pressures ranged from 0.27 psi to 2.41 psi. These temperatures and pressures were chosen to span the range of conditions expected in the absorption column. To achieve sub-atmospheric partial pressures of CO₂, the 16.44 vol% CO₂ in N₂ was mixed with 100% N₂. The results are shown in Figure 2. The error bars shown are 95% confidence intervals. Figure 2 shows that under these conditions, equilibrium loading is a strong function of both temperature and CO₂ partial pressure. At low temperatures, the CO₂ partial pressure does not significantly impact the equilibrium loading. However, at higher temperatures, the equilibrium loading in the liquid drops significantly at lower CO₂ partial pressures.

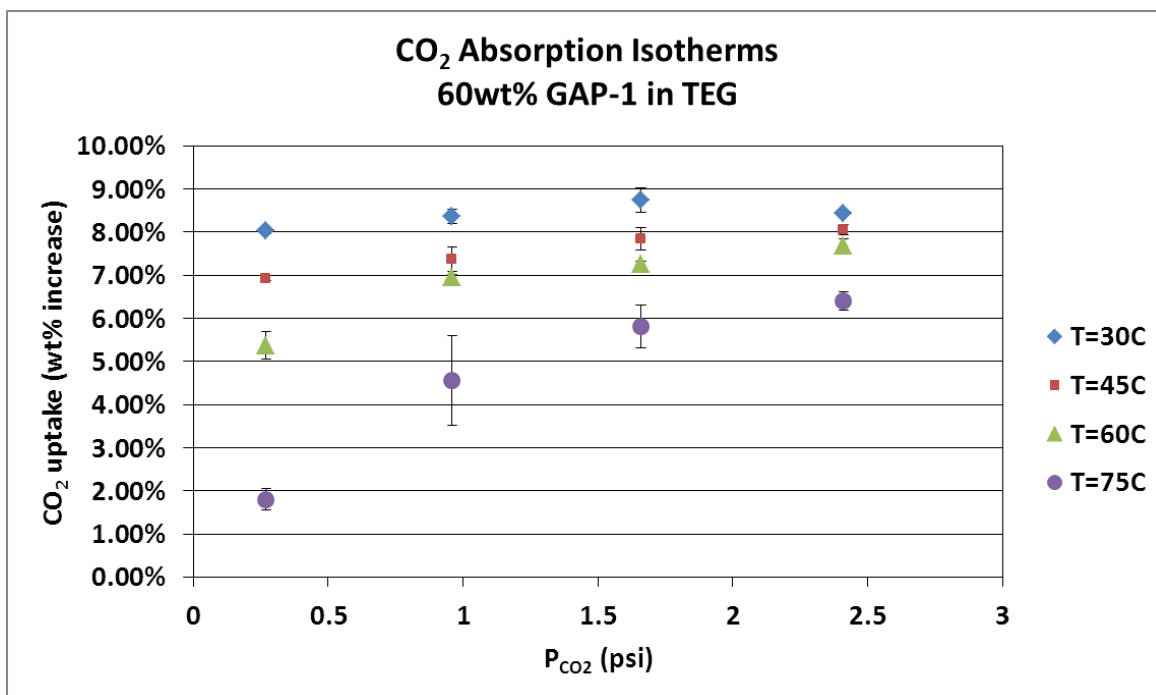


Figure 2. Equilibrium loading of CO₂ in 60/40 (wt/wt) GAP-1/TEG mixture (error bars shown are 95% confidence intervals).

Equilibrium Loading Experimental Details

A 25 mL 3-neck round bottom flask was loaded with approximately 2 g of a 60/40 (wt/wt) GAP-1/TEG. The flask was fitted in one neck with a glass stirrer bearing, a ground glass stir shaft, and a Teflon stirrer paddle. In the other two necks were a gas inlet port and a gas outlet port. The gas outlet port was then connected with Tygon tubing to a silicone bubbler containing less than 1" of silicone oil. The gas inlet port was connected to two mass flow controllers, one of which was connected to a bottle of 16.44 vol% CO₂ in N₂ and the other of which was connected to a bottle of 100% N₂. The set points for the mass flow controllers were varied for each experiment to adjust the partial pressure of CO₂ to the desired value. The total flow sent to the round bottom flask was fixed at 45 SCCM. The flask loaded with the GAP-1/TEG mixture was immersed in a silicone oil bath set at the desired temperature. The overhead stirrer was set at a fixed stir rate. A schematic of the experimental setup is shown in Figure 3. The gas outlet and gas inlet were connected to the flask and the gas flow was started. The gas flow was stopped and the flask was removed from the oil bath and weighed every 5-10 minutes to track the uptake of CO₂ in the liquid. The experiment was stopped when the reaction was determined to have reached equilibrium (the weight did not change significantly for at least 3 measurements).

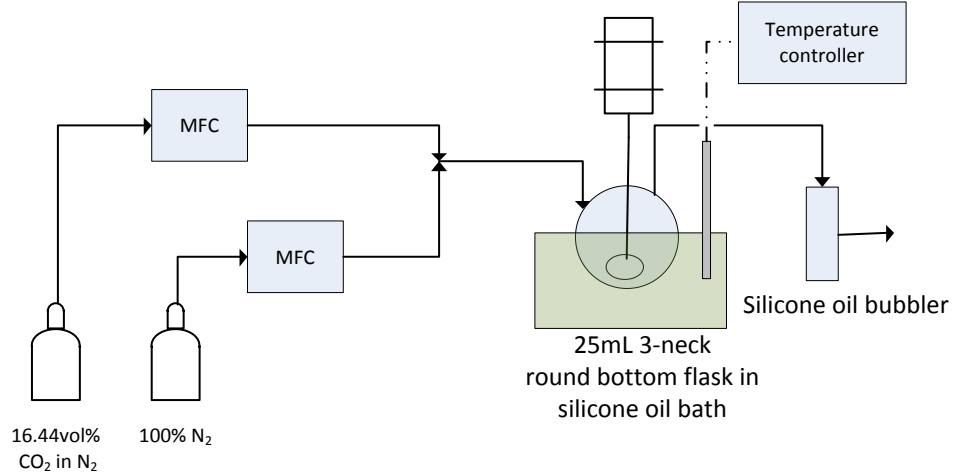


Figure 3. Schematic of equilibrium absorption test apparatus.

Mass Transfer Coefficient

To determine the size the bench scale absorption column required to achieve 90% CO₂ capture using 60/40 (wt/wt) GAP-1/TEG, the overall gas-phase mass transfer coefficient (K_{Ga}) for the system needed to be estimated. To estimate the K_{Ga} for the bench-scale system, the K_{Ga} for a laboratory scale column was determined at room temperature for 60/40 (wt/wt) GAP-1/TEG, 16.44 vol% CO₂ in N₂, and a liquid flowrate of 10mL/min. A series of experiments were performed for four packing heights and five molar gas to liquid ratios (Gm/Lm) spanning from 0.4-1.2. At each condition, K_{Ga} was calculated using Equation 1 and Equation 2. These results are shown in Figure 4. As expected, as the gas flow rate increases, so does K_{Ga} . While this K_{Ga} is specific to this column size, packing, and temperature, and this value will change with varying operating conditions and packing, this value can be used to estimate the size of the column.

$$Z = H_{OG} N_{OG}, \text{ where } Z = \text{packing height and } N_{OG} \text{ was determined numerically} \quad (1)$$

$$H_{OG} = \frac{G_m}{K_G a P}, \text{ where } P = \text{column pressure} \quad (2)$$

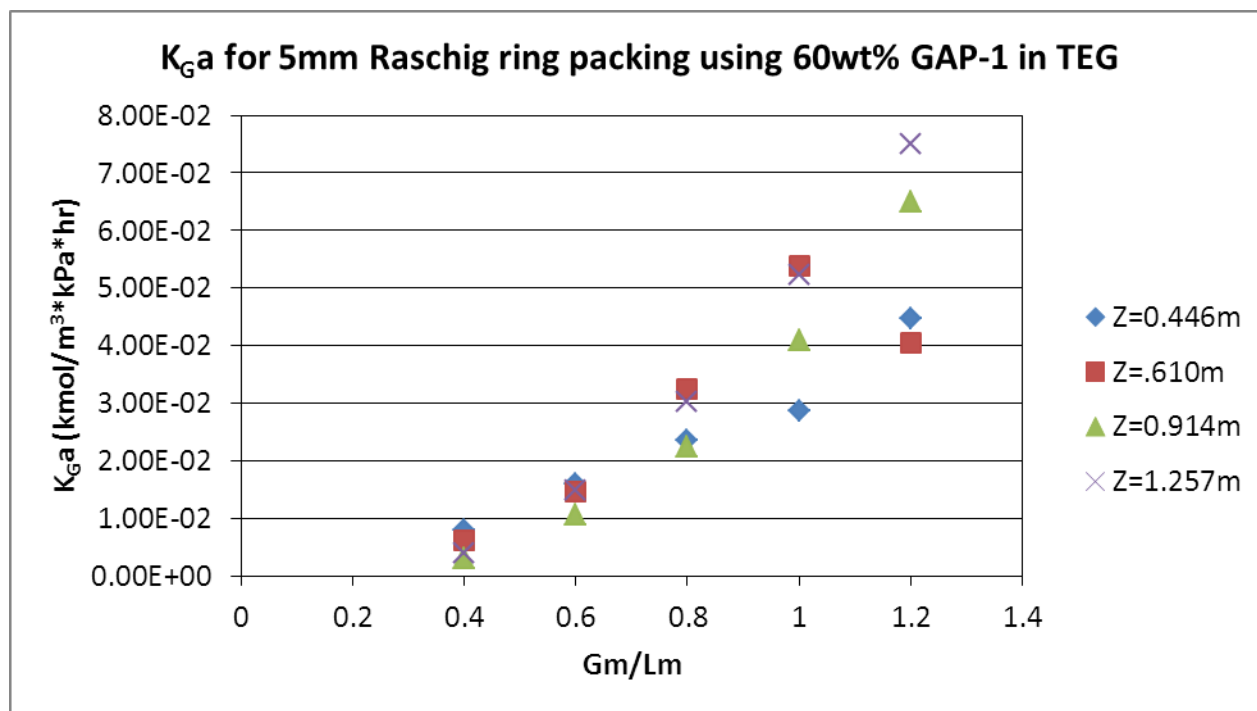


Figure 4. K_G for 50mm laboratory-scale absorption column and 5mm Raschig rings, $T = 20^\circ\text{C}$.

The temperature of the column was monitored to determine the temperature rise as a function of column height. Table 1 gives the temperature rise from the top to the bottom of the column as recorded at the highest Gm/Lm for each packing height. As expected, a temperature rise was observed in the column. The temperature rise was small, despite the large heat of absorption measured for this reaction, due to the non-adiabatic nature of the column.

Table 1. Temperature rise in 50mm laboratory scale absorption column.

Packing Height (m)	T Rise ($^\circ\text{C}$)
0.446	3.3
0.610	4.0
1.257	3.5

Mass Transfer Coefficient Experimental Details

A laboratory-scale column 50 mm in diameter and approximately 1.3 m tall was packed with Raschig rings having a length of 5.3 ± 0.5 mm, an outer diameter of 5.02 ± 0.04 mm, and an inner diameter of 3.4 ± 0.4 mm. The bulk density was measured to be 807 kg/m^3 and the surface area per unit volume (a_v) was calculated to be $921 \text{ m}^2/\text{m}^3$. The column was supplied with a 16.44% (by volume) CO_2 in N_2 mixture from a cylinder purchased from AirGas. The composition of the gas entering the column was measured using a MKS Cirrus mass spectrometer and the composition of the gas exiting the column was monitored throughout the experiments. The column was filled with 0.446 m of packing. The liquid flow rate was set to 10 mL/min and the gas flow rate was set to 0.464 SLPM ($\text{Gm/Lm} = 0.4$). The system was allowed to come to steady-state and the system pressures, temperatures, and gas compositions were recorded. The gas flowrate was increased and the procedure was repeated for $\text{Gm/Lm} = 0.4, 0.6, 0.8, 1.0$, and 1.2 and for packing heights of 0.446 m, 0.610 m, 0.914 m, and 1.257 m.

Column Sizing

For quotation purposes, the initial size of the absorption column was estimated to have a diameter of 0.3 meters and a height of 3 meters based on scaling of the liquid and gas flow rates from the laboratory scale to the bench scale. This column sizing required to achieve 90% CO_2 capture and an acceptable pressure drop was verified based on the laboratory scale data.

Pressure Drop

The expected pressure drop in the column was calculated for a column diameter of 12 inches, a liquid flow rate of 1 L/min, gas flow rates of 19-135 SLPM ($\text{Gm/Lm} = 0.2$ -1.2), and packing factors (F_p) of 200 - 1200 m^{-1} using the generalized pressure drop correlation found in Perry's Chemical Engineers' Handbook¹. The packing factors studied span the range found for conventional random packing. The maximum pressure drop calculated was 8 mm $\text{H}_2\text{O}/\text{m}$ packing at $F_p = 1200 \text{ m}^{-1}$ and $\text{Gm/Lm} = 1.2$. The recommended design values for the pressure drop in random packed column absorbers is 15-50 mm $\text{H}_2\text{O}/\text{m}$ packing². For a column 3 m high, a pressure drop of 8 mm $\text{H}_2\text{O}/\text{m}$ packing equals a total pressure drop across the column of 0.03 psi and a pressure drop of 50 mm $\text{H}_2\text{O}/\text{m}$ packing equals a total pressure drop across the column of 0.21 psi. Pressure drop of 0.21psi or less across the column is acceptable. This number is used for the design of the continuous system to determine the required delivery pressure of the rich gas to the column.

¹ Green, Don W.; Perry, Robert H. (2008). Perry's Chemical Engineers' Handbook (8th Edition). McGraw-Hill.

² Sinnott, R.K. (2005). Coulson and Richardson's Chemical Engineering Volume 6 - Chemical Engineering Design (4th Edition). Elsevier.

Packing Height

The packing height required to achieve 90% capture was calculated based on the K_{Ga} determined from the laboratory scale column and assuming a column diameter of 12 inches. The column height was calculated from Equation 1 and Equation 2 using $P = 17$ psia and an N_{OG} calculated numerically. To calculate N_{OG} , a straight operating line was assumed such that 90% CO_2 capture was achieved with a liquid working capacity that is 50% of the total CO_2 capacity of the liquid based on stoichiometry. Figure 5 shows the packing height for three different scenarios. Scenario 1 is calculated using a liquid flowrate of 1 L/min and the K_{Ga} calculated from the laboratory scale column for the 0.914 m packing height. Scenario 2 is calculated using a liquid flowrate of 0.75 L/min and the K_{Ga} calculated from the laboratory scale column for the 0.914 m packing height. Scenario 3 is calculated using a liquid flowrate of 1 L/min and a K_{Ga} an order of magnitude smaller than the value calculated from the laboratory scale column for the 0.914 m packing height.

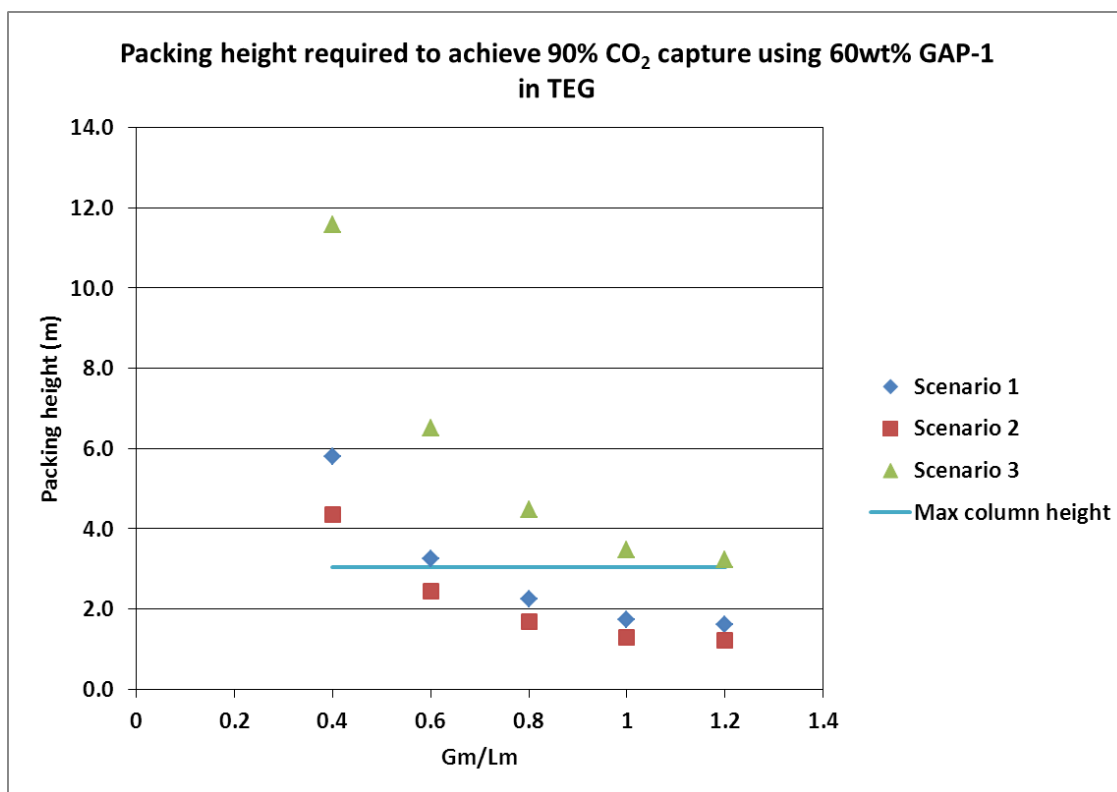


Figure 5. Packing height required for 90% CO_2 capture for a 12 inch diameter column using 60/40 (wt/wt) GAP-1/TEG.

Figure 5 shows that 90% CO₂ capture can be achieved with a column diameter of 12 inches and a packing height of 3 m. It is important, however, that the liquid and gas flow rates in the bench-scale system can be varied over a fairly wide range so that the capture efficiency can be varied and the effect of the Gm/Lm ratio can be studied.

Figure 5 uses the K_{GA} value calculated from the lab scale column using 5mm Raschig ring packing with an $a_v = 921\text{m}^2/\text{m}^3$. On a larger scale column, the K_{GA} value will change as a function of the packing type and size, as the a_v changes with packing type. To estimate the impact of changing the packing type, the packing height required to achieve 90% CO₂ capture in a random packed column having a diameter of 12 inches at a liquid flow rate of 1 L/min was calculated as a function of a_v . The column height was determined by first calculating the surface area of 5mm Rasching ring packing that would be required to achieve 90% capture at the bench-scale conditions, then dividing that number by the new a_v to get the volume of the new packing required. That value was then divided by the cross-sectional area of the column to get the height of the packing. These results are shown in Figure 6. This figure also shows that for a packing height of 3 m and a column diameter of 12 inches, 90% CO₂ capture can be achieved using 60/40 (wt/wt) GAP-1/TEG.

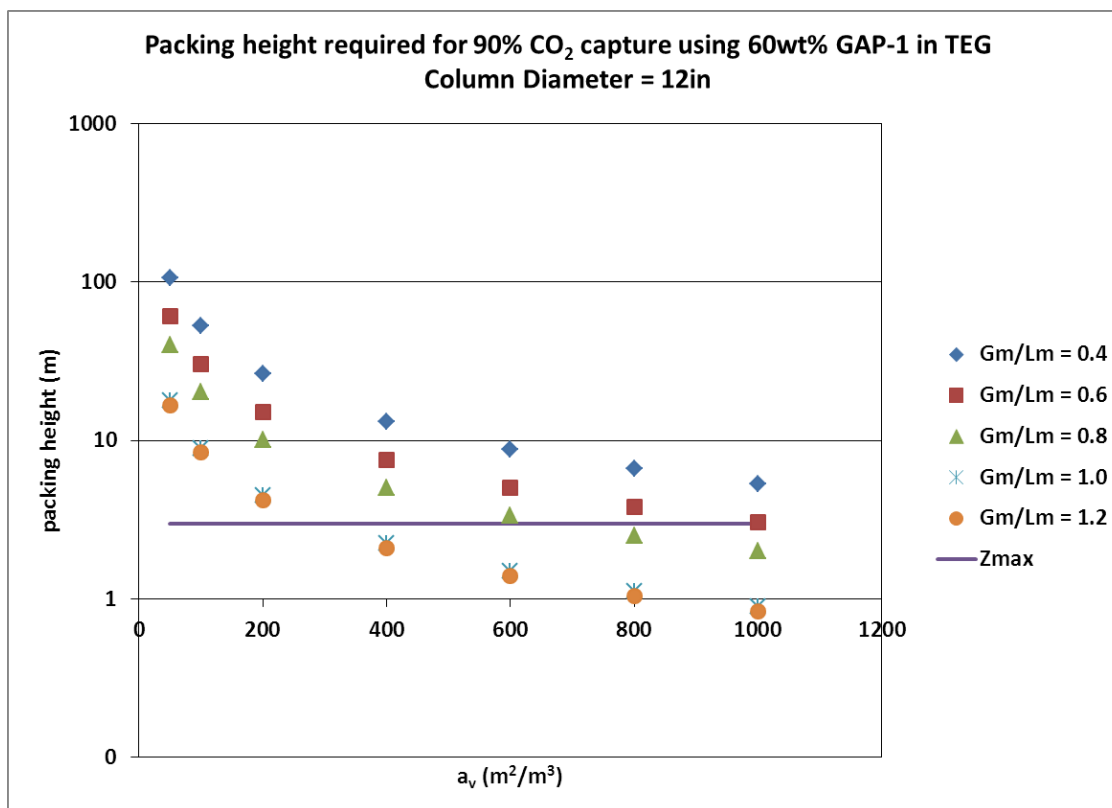


Figure 6. Packing height required for 90% CO₂ capture for a 12 inch diameter column using 60/40 (wt/wt) GAP-1/TEG for varying a_v values.

Heat of absorption

The heat of absorption of CO₂ in 60/40 (wt/wt) GAP-1/TEG was required to determine the energy requirements for the absorber and desorber. The heat of absorption of 60/40 (wt/wt) GAP-1/TEG was measured to be 2265 ± 75 kJ/kg CO₂ at 40 °C. Figure 7 shows a comparison of the heat of absorption of 60/40 (wt/wt) GAP-1/TEG with 30 wt% MEA in water measured on the same reaction calorimeter.

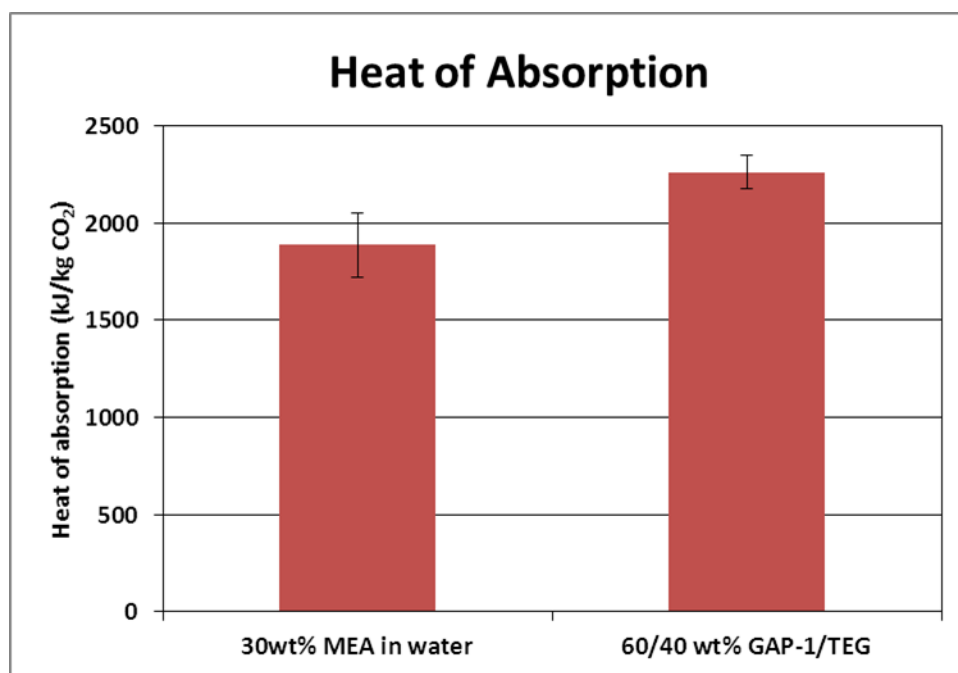


Figure 7. Heat of absorption of CO₂ at 40 °C for 30 wt% MEA in water and 60/40 (wt/wt) GAP-1/TEG (error bars shown are 95% confidence intervals).

Heat of Absorption Experimental Details

The heats of absorption of CO₂ with 60/40 (wt/wt) GAP-1/TEG were measured using an OmniCal ReactMax-Z3-UL Reaction Calorimeter. Hasteloy-C reactor vessels (25mL) supplied by the calorimeter manufacturer were used that can withstand pressures up to 34.5 bar. An additional stainless steel vessel was added adjacent to the calorimeter in order to supply heated CO₂ to the reactor vessel. This additional vessel was placed in a heated box fitted with a circulating fan. A Sierra Instruments Smart-Trak®2 Model# C100L mass flow controller was installed in-between the reactor vessel and the additional stainless steel CO₂ storage vessel to

measure the amount of CO₂ added to the reactor. This mass flow controller has an integrated totalizer to measure the total flow of a gas over a user-defined time.

The reactor vessel was filled with ~2.5 g of sample and a magnetic stir bar was added. The exact volume of the sample was calculated using the density of each sample. The reactor was sealed, placed inside the calorimeter, stirring set to between 600 and 780 RPM and the temperatures of the calorimeter and the CO₂ storage vessel were set to 40 °C. The CO₂ storage vessel was filled with CO₂ from the supply tank. The system was then allowed to come to equilibrium for 1-2 hours. When both the heat flow and the calorimeter temperature achieved steady-state, the system was considered to be at equilibrium.

The totalizer on the mass flow controller was reset to zero and the reactor was filled with ~20 SCC of CO₂. The value on the mass flow controller totalizer was recorded and the reaction was allowed to proceed for 2 hours. This procedure was repeated 13 more times, for a total CO₂ addition of ~280 SCC of CO₂. For each addition of CO₂, the baseline value for the heat flow was established and subtracted from the raw data. The baseline-subtracted heat flow was then integrated over the reaction time to determine the total reaction heat. The total amount of CO₂ remaining in the headspace of the reactor was calculated from the pressure, temperature, and headspace volume. The total amount of CO₂ absorbed by the sample was calculated by subtracting the CO₂ remaining in the headspace at the end of the reaction from the total CO₂ that was added plus the CO₂ remaining in the headspace after the previous reaction step. The heat of reaction for each step was then calculated by dividing the total reaction heat by the amount of CO₂ absorbed by the sample.

Desorber Design

Desorption isotherms

During second quarter, equilibrium isotherms of the carbamate in 60/40 (wt/wt) GAP-1/TEG at the temperatures and pressures of the desorber were determined. The experimental system is presented below on Figure 8.

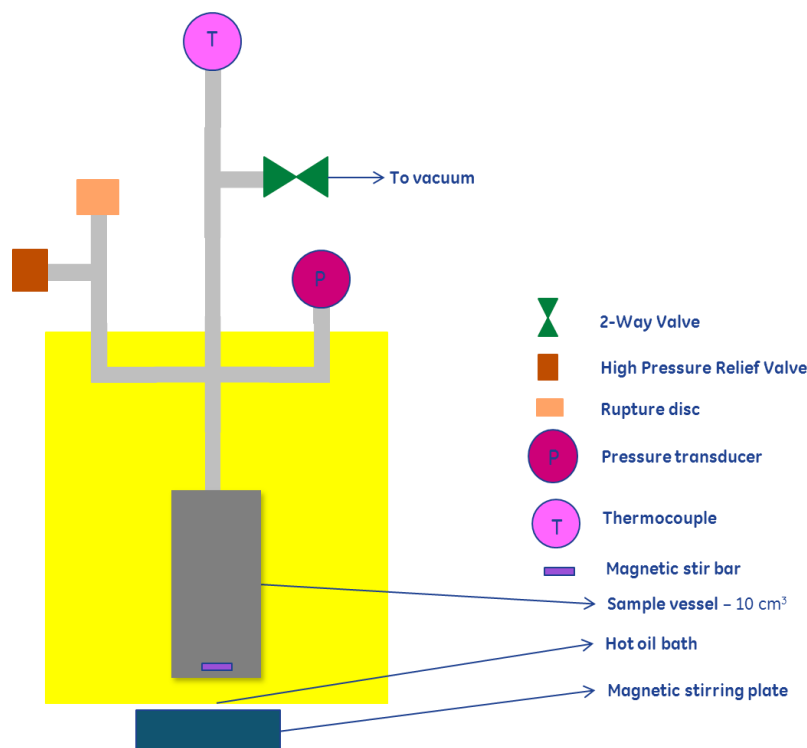


Figure 8. Desorption isotherm experimental setup.

At the beginning of each experiment, the system was cleaned with methanol and dried in a vacuum oven. The magnetic stirrer was placed into the reactor, and then ~2 g of sample were added. After the sample vessel was connected to the system, evacuation of the system was performed to remove any air from the head space. Afterwards, the system was placed into a hot-oil bath, and a temperature ramp was performed to heat up the solution to 100 °C, 120 °C, 140 °C, 160 °C, and 180 °C. The system was held at each temperature for 3 to 5 hours, in order to provide a sufficient time to reach equilibrium. Pressure readings were continuously collected and the amount of CO₂ in the gas phase and equilibrium carbamate loading in the liquid phase were determined. The experiment was run with samples with carbamate loadings of 20%, 40%, 60%, 80%, and 100%. Resulting equilibrium isotherms and 95% confidence intervals are presented in Figure 9.

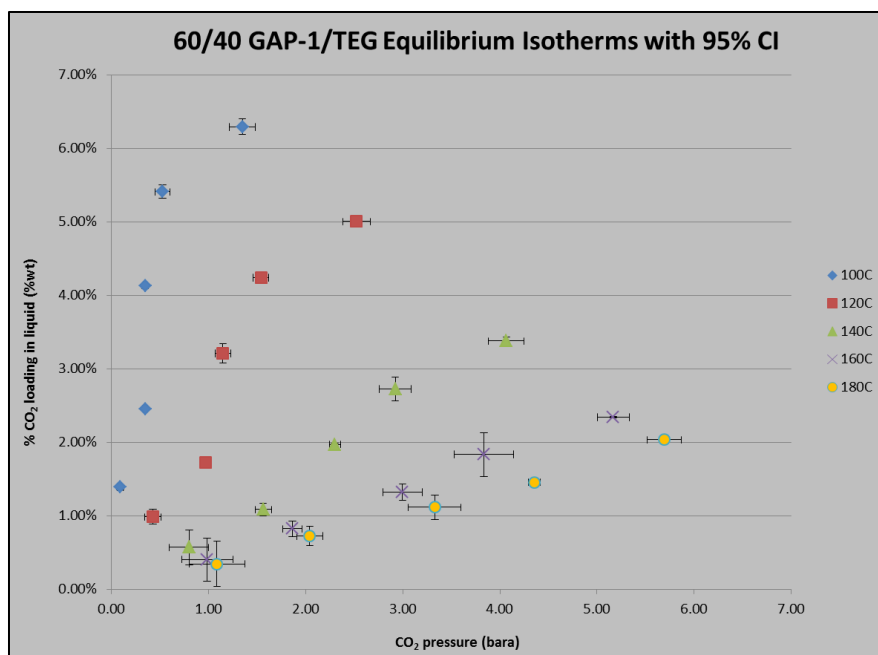


Figure 9. Equilibrium isotherms for 60/40 (wt/wt) GAP-1/TEG solution.

Desorption Kinetics

To determine the kinetic rate of desorption of CO₂ from the 60/40 (wt/wt) GAP-1/TEG mixture Thermogravimetric Analysis (TGA) was performed for samples with various carbamate loadings. For consistency sample sizes were ~8-8.5mg, and the heating rate was set to 5 °C/min. Pure 60/40 (wt/wt) GAP-1/TEG mixture was used as a background curve for the analysis, and each sample was run three times for repeatability purposes. In Figure 10 it can be observed that there is a peak which corresponds to desorption of CO₂ from the 60/40 (wt/wt) GAP-1/TEG carbamate mixture loaded to 100%. The background of GAP-1/TEG evaporation was subtracted from the carbamate sample run and the resulting CO₂ peak is presented on Figure 11.

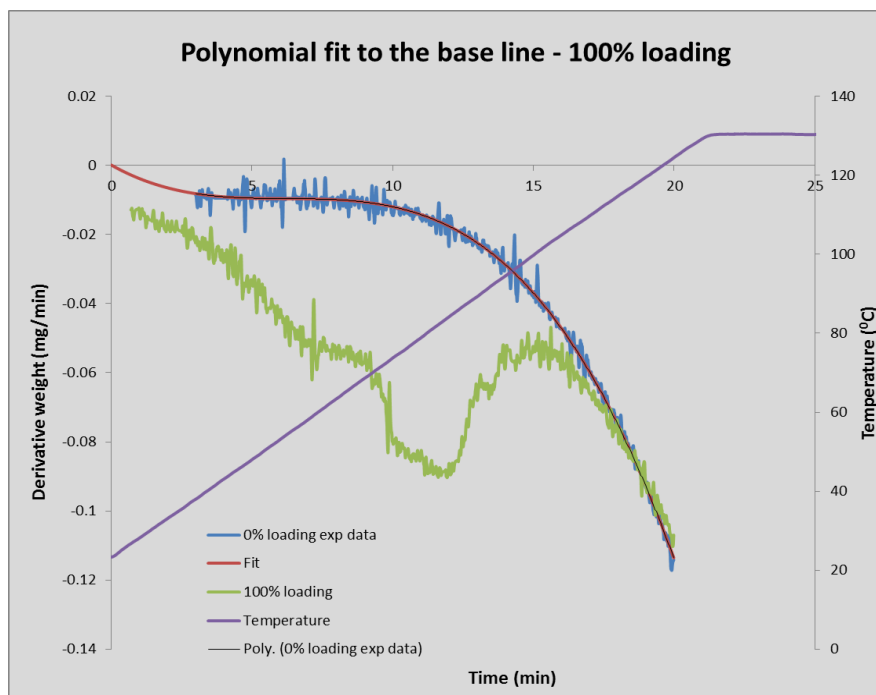


Figure 10. TGA run results for 60/40 (wt/wt) GAP-1/TEG and 100% loaded carbamate in 60/40 (wt/wt) GAP-1/TEG.

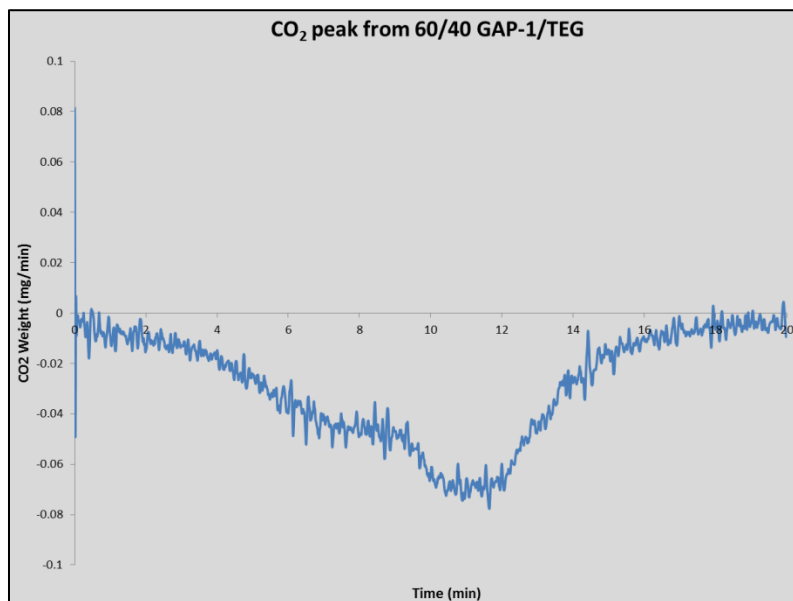


Figure 11. TGA peak corresponding to CO₂ desorption from 60/40 (wt/wt) GAP-1/TEG carbamate mixture.

To determine the area under the CO₂ peak, it was fitted to a Gaussian curve. The Gaussian was then fitted with five Lorentzian peaks using Peak Fit Software as shown in Figure 12.

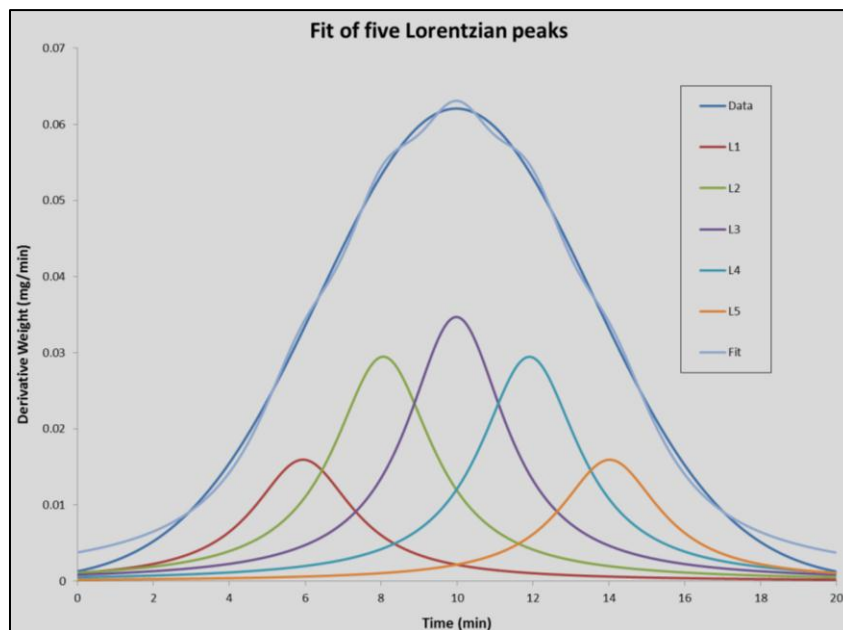


Figure 12. CO₂ peak fitted with five Lorentzian peaks.

The Lorentzian peak fitting parameters allow the integration of the area under the CO₂ desorption curve and the determination of the amount of CO₂ desorbed. Desorption reaction order was assumed to be 1, and from the available data it was possible to plot $\ln(k_2)$ vs $1/T$, where k_2 = desorption constant and T = temperature. This plot is shown in Figure 13.

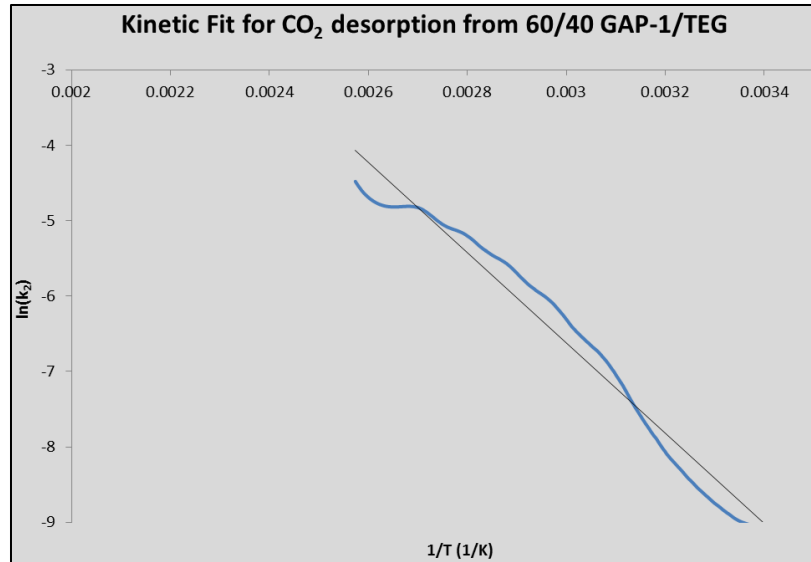


Figure 13. Dependence of desorption rate constant on temperature.

According to the Arrhenius equation:

$$\ln k = -\frac{E_a}{R} * \frac{1}{T} + \ln A_0$$

Where T = Temperature (K)

R = gas constant = 8.314 (kJ/kmol*K)

E_a = activation energy (kJ/kmol)

A_0 = frequency factor.

Therefore, using the plot from Figure 13, the activation energy and frequency factor can be determined, and their values were estimated to be $E_a = 7.69$ kcal/mol and $A_0 = 568$. These reaction parameters were used to determine the residence time required for the desired desorption in a continuous stirred-tank reactor. Figure 14 shows that small residence times are required to desorb most of the CO_2 from the solution.

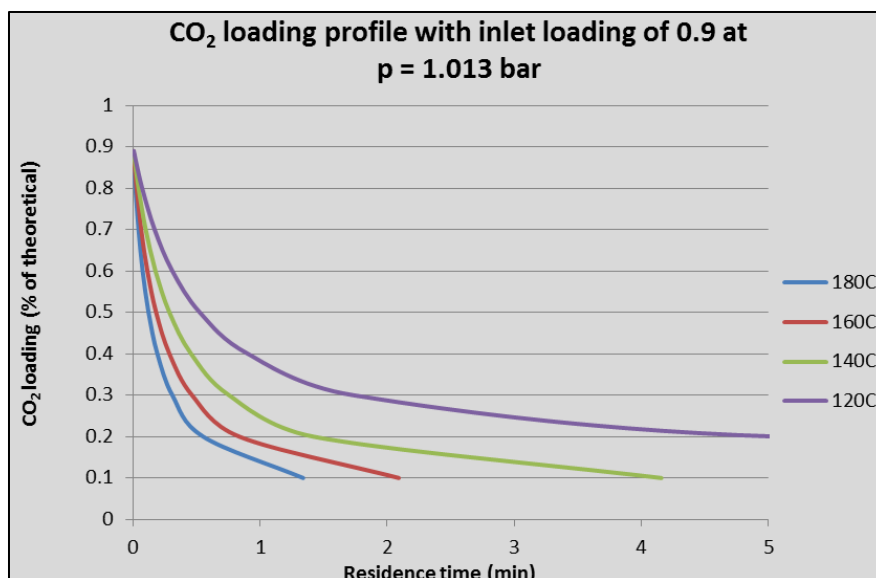


Figure 14. CO₂ loading profile with inlet loading of 0.9 at p = 1.013 bar for a CSTR.

Bench-Scale Desorber Sizing

The bench-scale desorber size was determined using the kinetic parameters determined for desorption. The bench-scale desorber is a CSTR with a working volume of 15 liters, which will provide a residence time of up to 15 minutes, and the total volume of ~30 liters. The reactor is designed to withstand 300 psig and 300 °C. A reactor jacket will be provided, and a hot oil system will be connected for heating. An impeller and three baffles are added to provide sufficient mixing to improve mass and heat transfer. Also, the recirculation loop with a high pressure gear pump and heat exchanger are added to the desorber to ensure required heating and to increase the mass transfer through sparging of the liquid back to the reactor. The level of the liquid in the desorber, temperature, and pressure will be controlled by the instrumentation and the control system.

Conclusions

A bench-scale process utilizing a novel silicone CO₂-capture solvent has been designed. This system will be used to establish scalability and potential for commercialization of post-combustion capture of CO₂ from coal-fired power plants. Due to the high level of flexibility in the process conditions under which this system operate, it should be possible to investigate a wide array of process conditions, facilitating demonstration of 90% capture efficiency and that less than 35% increase in the cost of energy services can be achieved upon scale-up.

Experiments were conducted to obtain data required for design of the major unit operations. The bench-scale system design has been completed, including sizing of major unit operations

and the development of a detailed Process and Instrument Diagram (P&ID). The system will be fabricated at Techniserv Inc.