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# Demonstration of a Concentrated Potassium Carbonate Process for CO<sub>2</sub> Capture

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**ABSTRACT:** A precipitating potassium carbonate (K<sub>2</sub>CO<sub>3</sub>)-based solvent absorption process has been developed by the Cooperative Research Centre for Greenhouse Gas Technologies (CO2CRC) for capturing carbon dioxide (CO<sub>2</sub>) from industrial sources, such as power plant flue gases. Demonstration of this process is underway using both a laboratory-based pilot plant located at The University of Melbourne and an industrial pilot plant located at the Hazelwood Power Station in Victoria, Australia. The laboratory-scale pilot plant has been designed to capture 4–10 kg/h CO<sub>2</sub> from an air/CO<sub>2</sub> feed gas rate of 30–55 kg/h. The power-station-based pilot plant has been designed to capture up to 1 tonne/day CO<sub>2</sub> from the flue gas of a brown-coal-fired power station. In this paper, results from trials using concentrated potassium carbonate (20–40 wt %) solvent are presented for both pilot plants. Performance data (including pressure drop, holdup, solvent loadings, temperature profile, and CO<sub>2</sub> removal efficiency) have been collected from each plant and presented for a range of operating conditions. Plant data for the laboratory-scale pilot plant (including temperature profiles, solvent loadings, and exit gas CO<sub>2</sub> concentrations) have been used to validate and further develop Aspen Plus simulations, in anticipation of further work involving precipitation and the industry-based pilot plant.

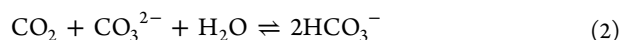
## ■ INTRODUCTION

Carbon dioxide capture and storage (CCS) is a promising process for removing carbon dioxide (CO<sub>2</sub>) from industrial sources, such as coal-fired power stations, and, thereby, limiting the effects of global climate change. The Cooperative Research Centre for Greenhouse Gas Technologies (CO2CRC) researches and develops technologies for CO<sub>2</sub> capture and geological storage of CO<sub>2</sub>. The CO2CRC solvent absorption capture group has been researching the use of potassium carbonate (K<sub>2</sub>CO<sub>3</sub>) solvent for CO<sub>2</sub> capture under both pre- and post-combustion capture conditions for a number of years.<sup>1–4</sup> More recently, the CO2CRC has been developing a novel precipitating K<sub>2</sub>CO<sub>3</sub> solvent absorption process, known as UNO MK 3,<sup>5</sup> which is designed to capture 90% of CO<sub>2</sub> emissions from large-scale emission sources, such as power stations. The UNO MK 3 process uses higher concentrations of K<sub>2</sub>CO<sub>3</sub> and precipitates potassium bicarbonate (KHCO<sub>3</sub>), which allows for lower solvent circulation rates and, hence, lower energy usage and smaller regeneration equipment size.<sup>5</sup> Some of the key benefits of this process, as compared to traditional amine-based capture processes, include lower regeneration energy, lower overall cost, low volatility and environmental impact, low toxicity, and the ability to incorporate multi-impurity capture of CO<sub>2</sub>, SO<sub>x</sub>, and NO<sub>x</sub> and production of valuable byproducts.<sup>6,7</sup>

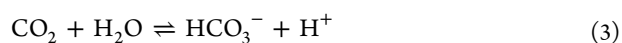
The potassium carbonate absorption process has been used to remove CO<sub>2</sub> in a range of industrial processes since it was first developed in the 1950s by Benson and Field.<sup>8–10</sup> The absorption of CO<sub>2</sub> into a K<sub>2</sub>CO<sub>3</sub> solution is described by the following overall reaction 1:<sup>10–12</sup>



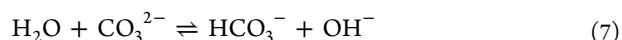
Because potassium carbonate and bicarbonate are strong electrolytes, it can be assumed that the metal is present only in the form of K<sup>+</sup> ions and eq 1 can be represented as



Reaction 2 proceeds according to the following sequence of elementary steps:



Reactions 3 and 4 are both followed by subsequent instantaneous reactions as follows:



The reaction sequence 3, 5, and 6 is known as the acidic mechanism.<sup>13</sup> The contribution of the acidic mechanism to the overall rate is negligible, unless the pH of the liquid solution is very low. Almost all cases of industrial absorption are held at

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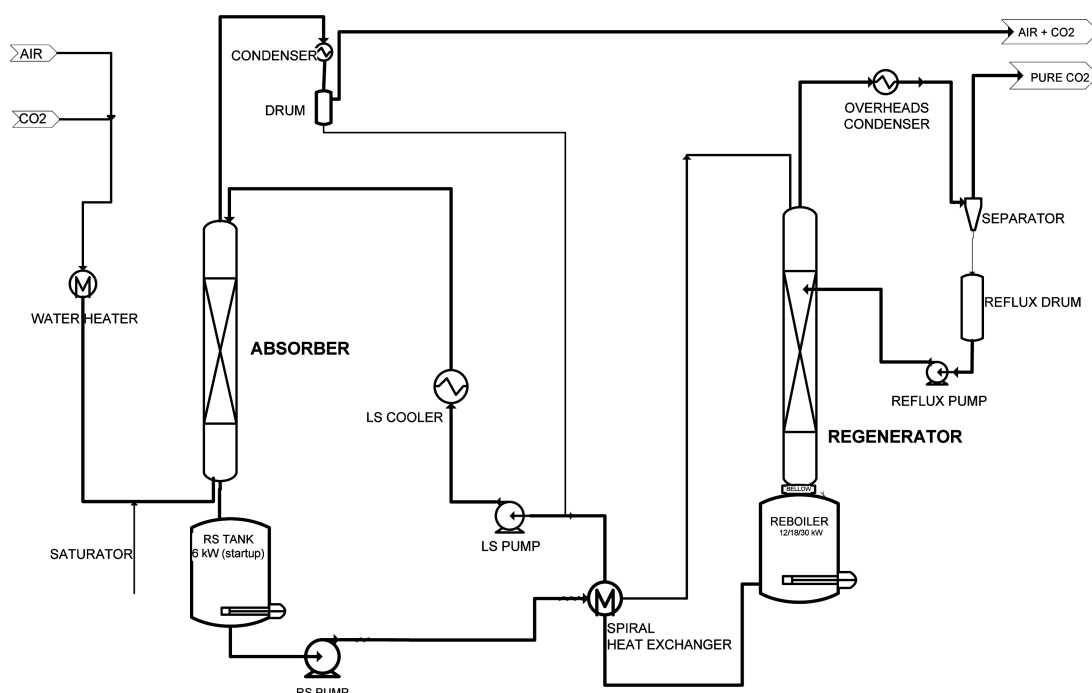


Figure 1. Process flow diagram of the laboratory-scale pilot plant.

high pH (generally,  $\text{pH} > 8$ ), and as such, the acidic mechanism can be neglected. Therefore, reaction 4 is the rate-controlling step for absorption of  $\text{CO}_2$  into hot potassium carbonate solution because reactions 5 and 7 are instantaneous reactions.

In the UNO MK 3 process, the concentration of  $\text{KHCO}_3$  solids in the rich solvent leaving the absorber is increased through cooling and separation of the supernatant liquor prior to solvent regeneration. The solids-rich solvent enters the regeneration column, where  $\text{CO}_2$  is released via heat addition, resulting in a  $\text{K}_2\text{CO}_3$  solution lean in  $\text{KHCO}_3$ , which can then be mixed with the previously separated solvent and recycled back to the absorption column.

In this work, simulation of the laboratory-scale  $\text{CO}_2$  capture plant was completed using Aspen Plus (version 7.3) software. Because Aspen Plus RateSep has not yet incorporated the ability for precipitation in its rate-based models, an equilibrium model using RadFrac was also developed. Within Aspen Plus, the electrolyte nonrandom two liquid (eNRTL) model was chosen for modeling the potassium carbonate solvent system and the binary interaction parameters were regressed using data from a wide array of literature sources.<sup>14–19</sup> The physical properties and rate constant correlations were developed at The University of Melbourne.<sup>20</sup> The liquid-phase mass-transfer coefficient was predicted using the Onda correlation,<sup>21</sup> which is relevant for a range of packing materials, including the pall rings used in the current pilot plant.

As described, RateSep has not yet incorporated the ability for precipitation in its rate-based models; therefore, an equilibrium model using RadFrac was also developed. This model relies on the use of a Murphree stage efficiency ( $\text{eff}_{\text{CO}_2,j}^{\text{M}}$ ). In the model developed, the Murphree stage efficiency of  $\text{CO}_2$  was adjusted only. In Aspen, the Murphree stage efficiency is given as

$$\text{eff}_{\text{CO}_2,j}^{\text{M}} = \frac{y_{\text{CO}_2,j} - y_{\text{CO}_2,j+1}}{K_{\text{CO}_2,j}x_{\text{CO}_2,j} - y_{\text{CO}_2,j+1}} \quad (8)$$

where  $x_{\text{CO}_2,j}$  is the liquid mole fraction of  $\text{CO}_2$  in the  $j$ th stage,  $y_{\text{CO}_2,j}$  is the vapor mole fraction of  $\text{CO}_2$  in the  $j$ th stage, and  $K_{\text{CO}_2,j}$  is the equilibrium  $K$  value for  $\text{CO}_2$  in the  $j$ th stage.

## EXPERIMENTAL SECTION

Performance data will be presented for a laboratory-scale pilot plant located at The University of Melbourne and an industrial-based pilot plant located at Hazelwood Power Station (GDF SUEZ Australian Energy, Hazelwood) in Victoria's Latrobe Valley. Aspen Plus simulation results will also be presented for performance of the laboratory-based pilot plant.

**Laboratory-Scale Pilot Plant.** A laboratory-scale pilot plant, designed to capture 4–10 kg/h  $\text{CO}_2$  from an air/ $\text{CO}_2$  feed gas rate of 30–55 kg/h, has been built in the Department of Chemical and Biomolecular Engineering at The University of Melbourne. A process flow diagram of the pilot plant has been provided in Figure 1. The plant has been designed to test the hydraulics of a precipitating potassium carbonate solvent system and to use these data to validate Aspen Plus simulations.

Feed gas containing 10–25 vol %  $\text{CO}_2$ , with the remainder compressed filtered air, is fed to a packed absorption column via Bronkhorst EL-FLOW mass flow controllers and a heated water bath. In a typical experimental run, the total gas flow rate is 30 kg/h and the gas mixture is at 50 °C. A gas saturator can also be used to obtain a feed gas with 80–95% relative humidity. The temperature and humidity of the feed gas are measured via a humidity probe located near the gas entry to the absorber. The absorber column is made of borosilicate glass and has a diameter of 100 mm and a total height of 4.25 m. The absorber has three packed bed sections, each 0.8 m in height. The packed bed sections are filled with stainless-steel pall rings with a diameter of 10 mm. The rich solvent tank at the base of the absorber is used for storage and contains a heating element for use during column startup procedures. The rich solvent leaving the absorber is sent to the regeneration column via a rich solvent tank, rich solvent pump, and the lean–rich exchanger. The lean–rich exchanger is a spiral heat exchanger, which heats the rich solvent stream via the lean solvent from the reboiler. The gas leaving the top of the absorber passes through a glass condenser to remove most of the moisture in



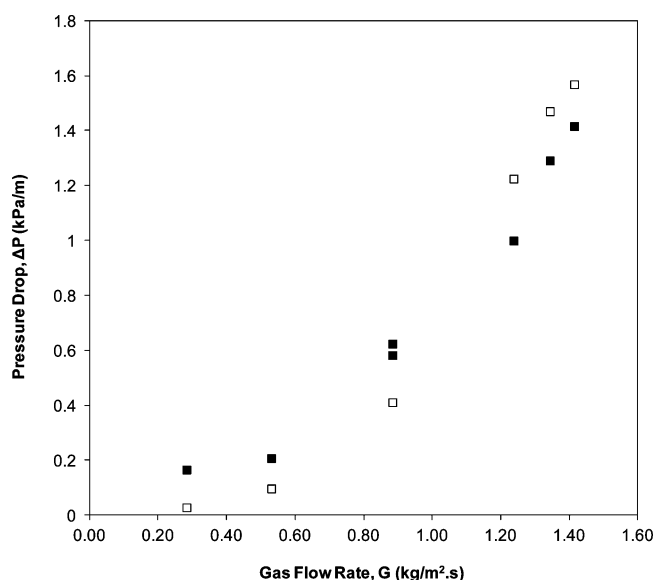
regeneration column or sent to drain to maintain the water balance of the system. The CO<sub>2</sub> gas product is then sent back to the power plant stack.

Plant operational data, including stream information, such as flow rate, pressure, and temperature, were continuously logged via a programmable logic controller (PLC). Rich and lean solvent samples were collected hourly throughout the operating period and analyzed for solvent concentration and CO<sub>2</sub> loading. Gas samples for the inlet and outlet of the absorber were analyzed using a PG250 gas analyzer (Horiba, Japan). The gas analyzer measured the concentrations of CO<sub>2</sub>, O<sub>2</sub>, CO, SO<sub>x</sub>, and NO<sub>x</sub> on a dry basis.

## RESULTS AND DISCUSSION

Hydrodynamic and mass-transfer performance data are presented for both the laboratory-scale and industrial-based pilot plants using 20–40 wt % K<sub>2</sub>CO<sub>3</sub> solvent. Aspen Plus simulations have also been developed to predict the performance of the laboratory-based pilot plant.

**Laboratory-Scale Pilot Plant Performance Data.** Gas and liquid flow rates were chosen to satisfy a range of liquid-to-gas (L/G) ratios and to maximize the pressure drop through the column. The theoretical pressure drop across the Pall ring packing was calculated using the generalized pressure drop correlation,<sup>25</sup> assuming a negligible effect on the pressure drop from the packing support plates. This assumption is justified by the choice of packing support plates with similar open area to that of the packing. Experimental and predicted pressure drop per meter of packing for a range of gas flow rates can be found in Figure 3. At low gas flow rates, the correlation tends to

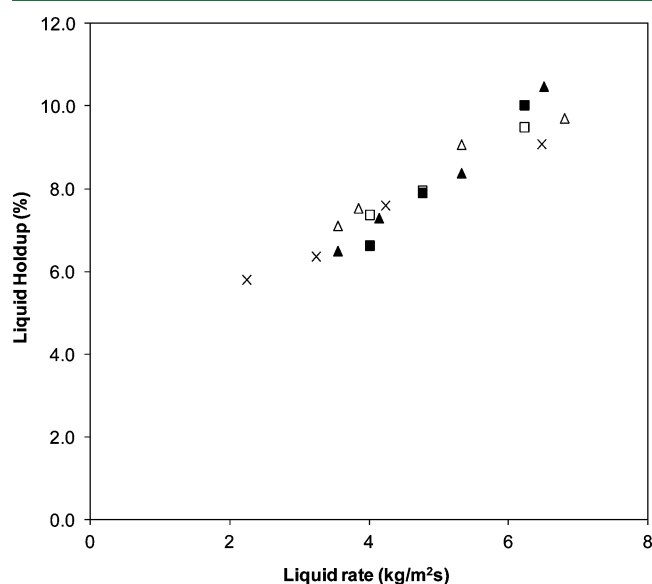


**Figure 3.** Pressure drop in the laboratory-scale pilot plant absorber column as a function of the gas flow rate for (■) experimental data and (□) predicted data. Predictions were obtained using the generalized pressure drop correlation.<sup>25</sup>

underpredict the experimental data, and this trend is reversed at higher gas flow rates. The variability in these predictions could be due to limitations of this correlation for non-industrial size packing. It has been found that the pressure drop in a packed column will vary with the column diameter if the diameter of the column is less than 0.9 m.<sup>26</sup> To ensure the column operates below flooding conditions and to provide adequate gas-to-liquid contact in the absorber at different L/G ratios, the flow

rate of gas was maintained at 1.06 kg m<sup>-2</sup> s<sup>-1</sup>. The flow rate of liquid was also maintained above the minimum wetting rate.

Liquid holdup was also measured for a range of solvent flow rates at different K<sub>2</sub>CO<sub>3</sub> concentrations, as shown in Figure 4.



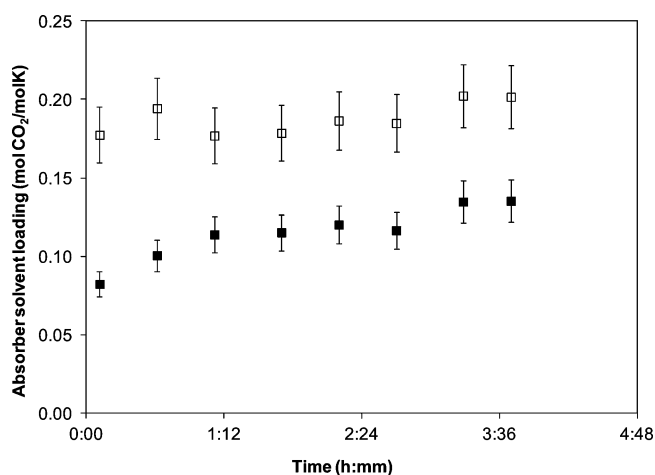
**Figure 4.** Liquid holdup in the laboratory-scale pilot plant absorber as a function of the liquid load for a range of K<sub>2</sub>CO<sub>3</sub> concentrations: (x) 20 wt % K<sub>2</sub>CO<sub>3</sub> dry gas, (□) 30 wt % K<sub>2</sub>CO<sub>3</sub> dry gas, (■) 30 wt % K<sub>2</sub>CO<sub>3</sub> wet gas, (△) 40 wt % K<sub>2</sub>CO<sub>3</sub> dry gas, and (▲) 40 wt % K<sub>2</sub>CO<sub>3</sub> wet gas.

Holdup was determined by measuring the volume of liquid that drained from the packing after simultaneously stopping the solvent and gas flows into and out of the column. Results in Figure 4 show that the liquid holdup increases linearly with the liquid flow rate. It was also found that there is negligible difference in holdup when a dry or wet gas is used with the same solvent concentration, which indicates that the mass of water transferred from the dry gas to the solvent had little effect on the holdup in the column. There was no significant change in the holdup when the K<sub>2</sub>CO<sub>3</sub> concentration was increased from 20 to 40 wt %.

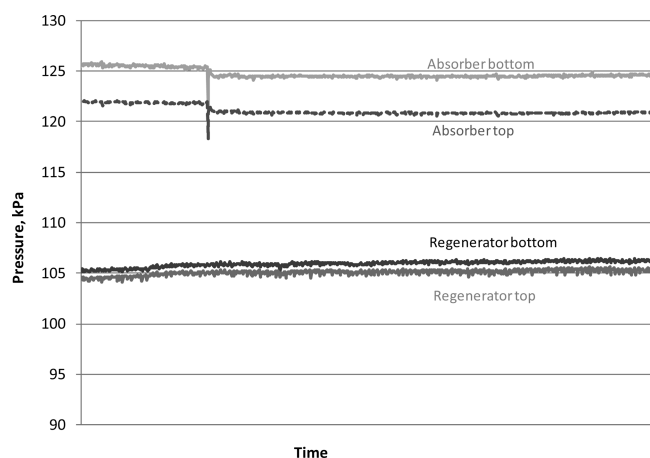
Solvent samples were collected every 30 min, and CO<sub>2</sub> loading was determined for both lean and rich solvent samples. Figure 5 shows a typical trend for lean and rich solvent loadings using 30 wt % K<sub>2</sub>CO<sub>3</sub>. It can be seen that steady-state conditions were achieved after approximately 1 h of operation with CO<sub>2</sub> gas. Temperature and pressure were logged continuously throughout the plant using temperature and pressure transmitters via Labview software. Typical experimental trends for absorber and regenerator pressure and temperature using 40 wt % K<sub>2</sub>CO<sub>3</sub> are shown in Figures 6 and 7, respectively.

The CO<sub>2</sub> gas concentration in the outlet of the absorber was measured every 15 min using a Horiba gas analyzer. Experiments 4, 12, and 13 were completed with a CO<sub>2</sub> feed concentration of 10 vol %, which is representative of power station flue gas. However, some experiments were also completed with a CO<sub>2</sub> feed concentration of 25 vol % to enhance mass-transfer performance. A comparison of the results of both the rate- and equilibrium-based simulations against the experimental data over three key parameters, CO<sub>2</sub> outlet concentration, rich solvent loading, and change in

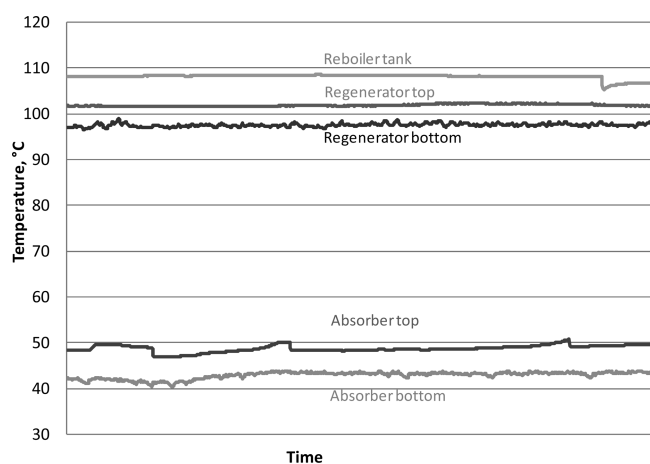




**Figure 5.** Typical solvent loading results over time for 30 wt %  $K_2CO_3$  in the laboratory-scale pilot plant: (■) lean solvent and (□) rich solvent.

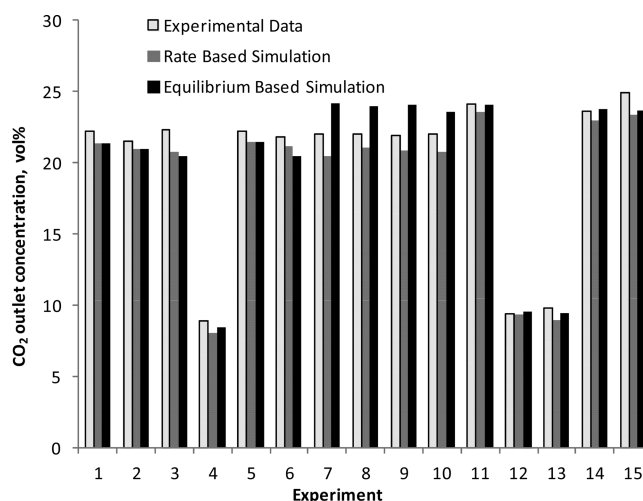


**Figure 6.** Typical absorber and regenerator pressure profiles over time for 40 wt %  $K_2CO_3$  in the laboratory-scale pilot plant.

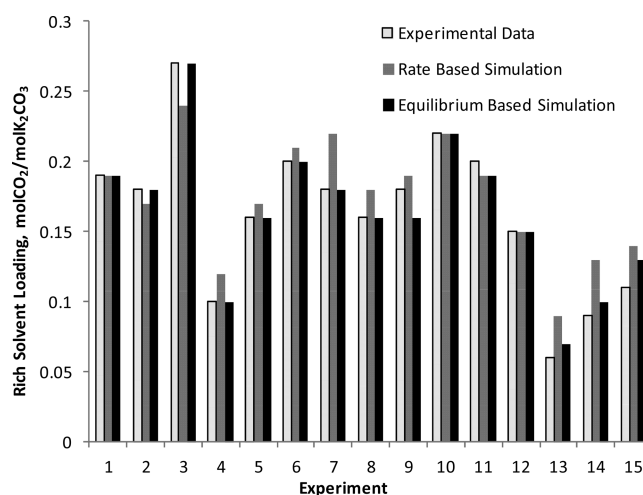


**Figure 7.** Typical absorber and regenerator temperature profiles over time for 40 wt %  $K_2CO_3$  in the laboratory-scale pilot plant.

solvent temperature, are presented in Figures 8–10, respectively. It was found that both the rate- and equilibrium-based models represent the data well with good accuracy in  $CO_2$  removal efficiency and loading. The Murphree efficiencies determined varied from 1.2 to 2%, although more than 50% of



**Figure 8.**  $CO_2$  absorber outlet gas concentration for a range of experimental conditions in the laboratory-scale pilot plant. Experimental data compare well to both rate- and equilibrium-based simulation results. All experiments were completed with a  $CO_2$  inlet concentration of 25 vol %, except for experiments 4, 12, and 13, which had a feed concentration of 10 vol %.

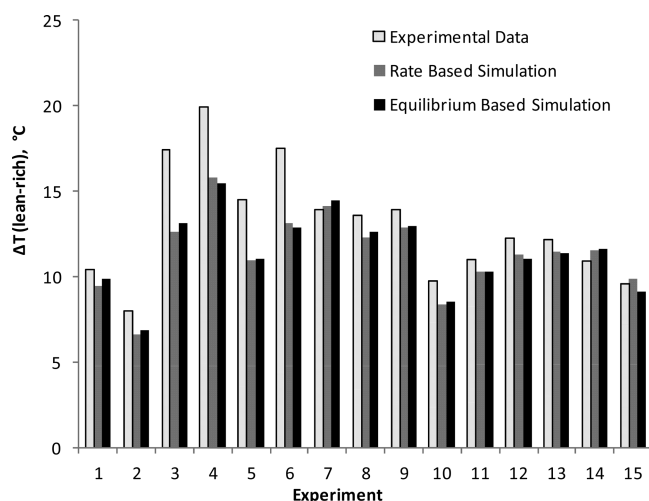


**Figure 9.** Rich solvent loading (moles of  $CO_2$  per mole of  $K_2CO_3$ ) for a range of experimental conditions in the laboratory-scale pilot plant. Experimental data compare well to both rate- and equilibrium-based simulation results.

those determined were calculated to be 1.3%. Additionally, although the variation between 1.2 and 2% appears significant, it represents less than a 10% variation in system prediction. It is expected that higher Murphree efficiencies will be obtained using a promoted potassium carbonate solvent. Temperature profiles have been predicted with a difference of  $\pm 1^\circ C$  for the majority of the experiments.

**Industrial-Scale Pilot Plant Performance Data.** The industrial-based pilot plant at Hazelwood Power Station has been operated over a range of operating conditions, including different L/G ratios, flue gas and solvent flow rates, and lean solvent temperatures. A summary of operating conditions for the experiments performed using this plant is presented in Table 3.

Liquid holdup was measured over a range of operating conditions, with results presented in Table 4. The experimental values shown in the table refer only to the liquid holdup within



**Figure 10.** Absorber temperature profile for a range of experimental conditions in the laboratory-scale pilot plant. Experimental data compare well to both rate- and equilibrium-based simulation results.

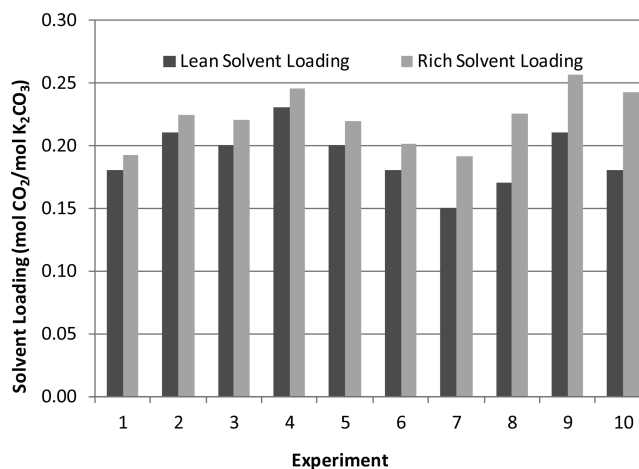
the packed bed and not the holdup within the distributors. Liquid holdup in this pilot plant was generally lower than that measured with the laboratory-scale pilot plant, which is most likely due to the use of different column packing and different solvent and gas flow rates.

Figures 11 and 12 show experimental data collected from the plant for lean and rich solvent loading measurements and  $\text{CO}_2$  removal rates, respectively. The gas-phase analysis was based on the assumption that  $\text{CO}_2$  is the only component absorbed in the solvent. Therefore, the rate of  $\text{CO}_2$  absorption is based on the ratio of  $\text{CO}_2$  in the outlet compared to  $\text{CO}_2$  in the inlet, normalized for the change in the total flow rate because of the removal of  $\text{CO}_2$ . This method is independent of the amount of water in the feed and lean gas because the gas analyzer measures dry gas concentrations. The liquid-phase  $\text{CO}_2$  removal rate was based on the difference in loading between the  $\text{CO}_2$  lean solvent and the  $\text{CO}_2$  rich solvent. The amount of  $\text{CO}_2$  in the lean and rich solvents was determined by an acid–base titration method, along with the flow rate and density of the respective streams. Figure 12 shows that most  $\text{CO}_2$  removal rates from the gas-phase analysis are higher than those from the liquid-phase analysis. The lower removal rates observed using liquid analysis are likely due to experimental error, such as the loss of  $\text{CO}_2$  from the solvent sample vials because of flashing.

$\text{CO}_2$  removal rate, which was calculated on the basis of the gas-phase analysis, was between 5 and 15% depending upon

**Table 4.** Experimental Liquid Holdup for the Industry-Based Pilot Plant

gas flow rate		lean solvent flow rate		L/G	packed bed holdup (L)	packed bed holdup (%)
(kg/h)	(kg/m <sup>2</sup> s)	(kg/h)	(m <sup>3</sup> /h)			
180	1.4	900	0.7	5.0	9.8	4.8
180	1.4	360	0.28	2.0	6.5	3.2
180	1.4	540	0.42	3.0	8.5	4.1
90	0.7	350	0.27	5.0	6.4	3.1
90	0.7	210	0.16	3.0	4.2	2.1
90	0.7	140	0.11	2.0	4.1	2.0



**Figure 11.** Lean and rich solvent loading for a range of experimental conditions in the industry-based pilot plant.

operating conditions. Although removal efficiency is limited by the height of the pilot plant packed column, removal rates are expected to improve with the addition of a rate promoter as well as higher concentrations of  $\text{K}_2\text{CO}_3$ . A range of rate promoters, including boric acid,<sup>27</sup> monoethanolamine (MEA),<sup>28</sup> and amino acids,<sup>29</sup> have been shown to improve the reaction kinetics of the absorption process using potassium carbonate solvent. With the addition of a rate promoter as well as higher concentrations of potassium carbonate to allow for bicarbonate precipitation, it is expected that higher  $\text{CO}_2$  removal rates will be achieved along with low regeneration energy.<sup>30</sup> The promoter is expected to remain in the solvent phase when precipitation occurs. Future work will involve modeling the industry-based pilot plant and incorporating the

**Table 3.** Summary of Operating Conditions for the Industry-Based Pilot Plant

test	flow rate (kg/h)		pressure (kPag)			temperature (°C)			lean solvent	
	gas	lean solvent	gas	reboiler	L/G	gas	lean solvent	reboiler	$\text{K}_2\text{CO}_3$ (wt %)	lean loading
1	178	899	5	50	5	47	43	116	29	0.18
2	183	1080	4	50	6	53	41	116	29	0.21
3	187	900	5	50	5	40	40	116	28	0.20
4	177	900	4	50	5	51	41	116	28	0.23
5	188	900	4	50	5	41	55	116	28	0.20
6	182	900	6	50	5	39	53	116	28	0.18
7	184	540	2	50	3	41	49	116	28	0.15
8	97	350	4	50	4	61	40	116	28	0.17
9	96	350	5	50	4	65	41	116	28	0.21
10	91	350	4	50	4	68	54	116	28	0.18

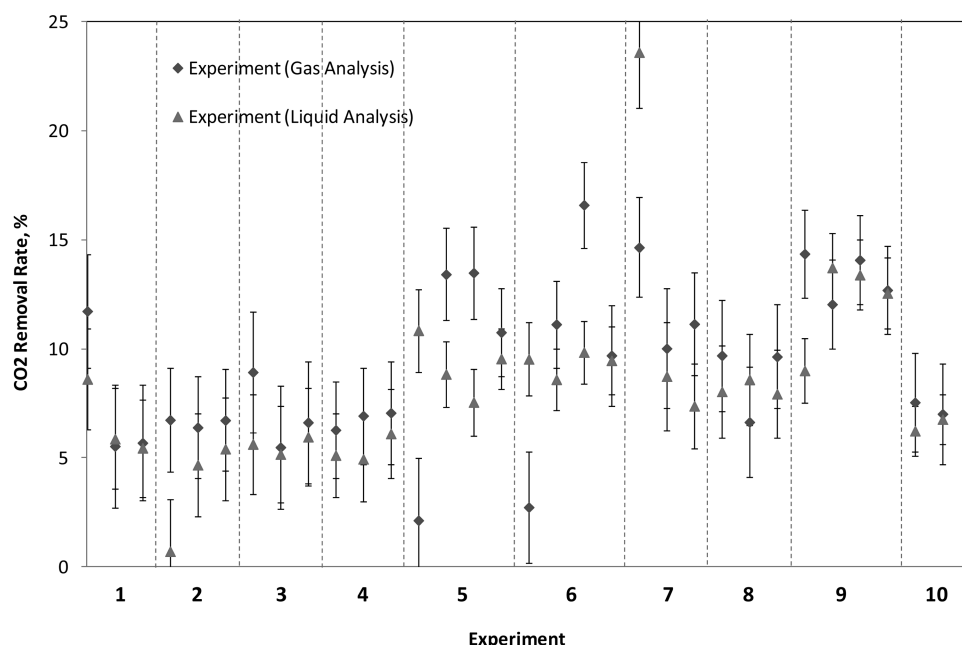


Figure 12. CO<sub>2</sub> removal rate for a range of experimental conditions in the industry-based pilot plant.

promoter and precipitation into the Aspen Plus simulation, which will then enable the prediction of equipment size and performance for higher CO<sub>2</sub> removal rates.

## CONCLUSION

Experimental data have been presented for CO<sub>2</sub> capture using both laboratory-scale and industry-based pilot plants. The solvent used in these trials was unpromoted potassium carbonate with concentrations ranging from 20 to 40 wt %. Laboratory-scale performance data have been used to validate an Aspen Plus simulation, which has been based on both rate- and equilibrium-based models using Murphree efficiencies ranging from 1.2 to 2%. The addition of a rate promoter is expected to improve these values. Further work will involve studying the performance of a range of promoted potassium carbonate solvent systems with precipitation and optimizing the performance of the regeneration process. The UNO MK 3 process is expected to have a regeneration energy of around 2–2.5 GJ/tonne of CO<sub>2</sub> compared to a state of the art amine of around 3 GJ/tonne of CO<sub>2</sub>.<sup>30</sup> Validation and optimization of these performance models are important for future work associated with the demonstration of a precipitating potassium carbonate solvent process.

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### Notes

The authors declare no competing financial interest.

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