CO2 Gas Absorption Experiment

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Abstract
Through UF’s IPPD program, our team constructed a carbon dioxide gas absorption system in the Unit Operations lab with the intent for it being used as a future experiment for the Unit Operations course. Standard operating procedures and system overview documents have already been written so future students can operate the experiment as well as complete experimental objectives that were created. Data has already been collected and analyzed to complete these objectives and serve as an example of data that future students could collect. The chemicals used in the experiment include carbon dioxide and anhydrous potassium carbonate that is used to make a 30 wt/wt% solution. A MSDS is also being provided as well as a form for students to properly record data during the experiment.

Introduction
CO2 gas emissions are becoming a greater impact on the environment, and new technology regarding absorbing this pollutant is being researched. One of these technologies is using a gas absorption column. Solvent is introduced counter-currently to an incoming gas stream where chemical and physical absorption occur in a packing material in the center of the column. This material slows the flowrate of both the gas and liquid and allows for greater absorption. The Department of Energy is currently funding different methods to improve the efficiency of CO2 absorption at numerous universities across the nation. This report goes over absorption tower technology and the different parameters that can be changed to optimize CO2 absorption.

Objectives
• Compare experimental L/V ratios with ratios found through using McCabe-Thiele analysis for low concentration system with random packing material
• Compare CO2 absorbance with changing L/V ratios
• Compare absorption of CO2 using water and solvent
• Calculate mass transfer coefficient for range of L/V ratios
Experimental Procedure

Materials

Table 1: Materials used during experiment

<table>
<thead>
<tr>
<th>Materials</th>
<th>Usage</th>
<th>Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>Wash out absorption column and collect absorption data</td>
<td>-Physical state: Liquid</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-Molecular Weight: 18.02 g/mol</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-Density: 0.9970474 g/mL at 25°C</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-Viscosity: 8.9x 10^-4 Pa.s</td>
</tr>
<tr>
<td>Potassium Carbonate (Anhydrous)</td>
<td>Create a 30 wt/wt % solution to act as solvent</td>
<td>-Physical state: solid</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-Molecular Weight: 138.21 g/mol</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-Density: 2.43 g/mL at 19°C</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-Solubility: Soluble in water</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-pH: 11.6 (10% aq solution)</td>
</tr>
<tr>
<td>Carbon Dioxide</td>
<td>Chemical that will be absorbed from the gas phase</td>
<td>-Physical state: gas</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-Molecular Weight: 44 g/mol</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-Density: 1.98 kg/m^3 at STP</td>
</tr>
</tbody>
</table>

Equipment

The system uses a combination of sensors, pumps, and tubing to transport both gas and solvent throughout the experiment. Figure 1 below shows the process flow diagram for the experiment as well as labels for all valves and equipment.
The experimental system spans all three floors of the unit operations lab and each section below shows what equipment is found on each of these floors:

**First Floor:**

*Figure 2* lays out the connections to the first story solvent storage tank. This is where solvent will be measured and added to the system and pumped up to the third story storage tank. This first story tank will
also work as a holding tank for solvent to be recirculated through the system again after passing through the column.

\[ \text{Figure 2: First floor configuration of CO2 gas absorption system} \]

**Second Floor:**

**Figure 3** shows the control panel for the experiment with each flowmeter labeled so the user can know which flow they are controlling. Two sensors are also placed on the board and monitor pH of the solvent as well as the CO2 concentrations of inlet and outlet gas streams to the column.
Third Floor:

Figure 4 displays the third story solvent storage tank. This is where solvent will be mixed through the use of a mixing pump before being sent to the flowmeter located on the control board.
**Experimental Design**

The objective of this experiment is to alter the liquid/gas ratio in the absorption column and notice how it plays a role on the absorption of CO2. The flowrate of gas will remain constant while only solvent flowrate is changed. Data will be organized using the following format for each trial ran:

<table>
<thead>
<tr>
<th>Time</th>
<th>Co2 % in</th>
<th>CO2 % out</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

Before data can be accurately collected, calibrations must also be done for the CO2 and solvent flowmeters as they are not calibrated from the manufacturer for these chemicals. Calibrations for CO2 flow rate were done using Equation 1 below and gave the following calibration curve shown in Figure 5.

\[
F_{CO2} = F_{Air} \left( \frac{SG_{Air}}{SG_{CO2}} \right)^{0.5}
\]  

1
Calibrations for the solvent flowmeter were done by opening valve SV-4 and recording the time it took for the flow to fill up a graduated cylinder. This data was collected for a range of flowrates and gave the following calibration curve shown in Figure 6:

The reason for the great difference between measured and actual flowrate is due to the difference in density between water and 30 wt% K2CO₃; potassium carbonate is 40% more dense.
Experimental Protocol

Vary Liquid/Gas ratio in the column:

- Ensure solvent is thoroughly mixed and configure valves to flow to second story control board
- Open compressed air and CO2 lines and configure their respective rotameters to achieve an inlet CO2 concentration of 13.5%
- Open valves and solvent flowmeter to flow solvent to column and down to the first story storage tank.
- Monitor pH and outlet concentration of CO2
- Allow system to reach steady state before changing to a different solvent flowrate

Safety

a) Personal Protective Equipment
   - Hard hat
   - Safety glasses
   - Long pants
   - Closed-toe shoes
   - N-95 Respirator (if handling anhydrous chemical)
   - Nitrile gloves (if handling solvent)

b) Safety Hazards and Prevention

<table>
<thead>
<tr>
<th>Safety Hazard</th>
<th>Prevention</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trip/Slip</td>
<td>- Remove trip objects&lt;br&gt;- Locate tubes and valves near floor</td>
</tr>
<tr>
<td>Chemical Spills</td>
<td>- Follow instructions outlined at <a href="http://ww2.che.ufl.edu/unit-ops-lab//safety.htm">http://ww2.che.ufl.edu/unit-ops-lab//safety.htm</a></td>
</tr>
<tr>
<td>Potassium Carbonate (Anhydrous) (Harmful lung irritant)</td>
<td>- Sign and review safety procedures for wearing N-95 respirator mask&lt;br&gt;- Wear mask whenever in direct contact with solid&lt;br&gt;- Do not wear contact lenses as the solid particles can get into eyes</td>
</tr>
</tbody>
</table>
Potassium Carbonate (30 wt/wt % solution)
- Wear nitrile gloves when handling solvent due to its corrosive properties

CO2 Heater
- Heater should not be touched while in operation but if needed, wear steam rated heat gloves when handling

Results and Discussion
During experimental runs, our team was able to observe CO2 absorption as well as trends for when Liquid/Gas ratio changes. The data collected was all compiled into one graph (Figure 7) that compares time and % CO2 absorbed through the system using both water and K2CO3:

![CO2 Absorbance Graph](image)

*Figure 7: CO2 Absorption data vs time*

As can be seen from the graph, CO2 absorption decreases as liquid/gas ratio also deceases. This was an expected trend because if you lower the solvent flowrate, the reaction between the CO2 and solvent will decrease. The graph includes linear trend lines as a way to show this decrease. The reason for the jumps between different flowrates is due to our team changing the flowrates while running the
experiment and waiting for steady state to be reached again. Once steady state was reached for each experimental run, the solvent flowrate was decreased. Water was also plotted alongside potassium carbonate and showed another trend that was predicted. Water absorbed a significantly less amount of CO2 than K2CO3. Water has a much higher Henrys constant than potassium carbonate which allows for only a small amount of CO2 to actually be absorbed.

One unexpected phenomena occurred however, which was the absorbance of CO2 without any solvent running through the column. Without solvent flow, the outlet CO2 concentration should read 13.5% but would always read on average around 9.7%. We are unsure how this absorption could occur unless there is some absorbance between the CO2 and the wet packing material. To account for this, all percent absorbance shown in Figure 7 are calculated assuming the inlet concentration is the outlet concentration with no solvent flow running.

The experimental data collected was then compared to predicted values from using a McCabe-Thiele analysis assuming gas and liquid flows have low concentrations of CO2. The equilibrium line for the McCabe-Thiele analysis was found using the following VLE data for K2CO3 and CO2. A tangent line was drawn at the center of the curve so we could assume the Henrys constant would remain constant. This VLE data is shown below in Figure 8 and gave our team a Henrys constant value of 6.13.

![Figure 8: VLE data for 30 wt% potassium carbonate solution at 40 Celsius [1]](image)

An operating line was then formulated assuming a CO2 input of 13.5% and an output concentration equal to what was experimentally measured. Figure 9 below shows the McCabe-Thiele analysis for only one of three Liquid/Gas ratios tested.
To reach the desired CO2 outlet concentration from what was experienced during the experiment, the McCabe-Thiele analysis suggests a molar flowrate Liquid/gas ratio of 3. The experimental run, however, was ran at a molar flowrate Liquid/Gas ratio of 12 (which corresponds to $L/V = 12.5$ [L/m^3]). The difference in the expected vs theoretical ratios may be due to the Henrys constant chosen. In the future, this constant could be found through benchtop experimentation in the Unit Operations lab and could show better predictions.

Regarding the calculations for the overall mass transfer coefficient, its value did not change for each of the three liquid/gas ratios tested. This could be due to how the number of equilibrium stages was found. They were approximated using drawing tools on the graph where they could accurately be calculated through the use of coding. The value for the overall mass coefficient found was 22,700 mol/(m^3*h).

**Conclusions**

Overall, the gas absorption system showed expected trends regarding the effect of CO2 absorption with respect to Liquid/Gas ratio. As the Liquid/Gas ratio was decreased, the percent CO2 absorbed decreased as well. At peak absorbance, the system was able to absorb approximately 29%. This absorbance takes into account the background absorption caused by the wet packing material with no solvent flow. Experimental data was then compared to theoretical predictions using the McCabe-
Thiele analysis for low concentration systems. There was a significant difference between the two and this could be caused by an inaccurate Henrys constant for potassium carbonate 30 wt% solution. An overall mass transfer coefficient was also calculated and was the same for all Liquid/Gas ratios tested which should not be the case. This could be caused by how the number of theoretical stages was calculated. If more precise methods could be used to find the number of stages, a more accurate coefficient could be calculated.

Acknowledgements
I would like to acknowledge Dr. Rivera and the rest of my IPPD team for the help in constructing and designing this experiment.

References