Carbon dioxide absorption in a membrane contactor

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Abstract

The objective of this laboratory experiment is to determine carbon dioxide absorption rate as a function of the liquid velocity in a hollow fiber membrane contactor. The experimentally measured absorption rate is to be compared with an absorption rate predicted by a model. The applicability of the model is then to be critically analyzed. Is the provided model a good model for describing this process?

1 Introduction

Liquid/liquid or gas/liquid interphase interactions are traditionally carried out using a column, a tower or a mixer. All these designs require that the two phases are in direct contact. This direct contact could lead to unwanted phenomena such as foaming, flooding and formation of emulsions, that will reduce the efficiency of the process (1). An alternative for overcoming these complications are the use of non-dispersive contact using a microporous membrane, which also gives a large interfacial area. This experiment introduces a membrane contracting process, which is a hybrid of gas absorption and membrane separation process (2) and use it to study the absorption of CO$_2$ from a gas stream into a liquid solvent.

2 Theoretical background

A membrane contactor is a device where the mass transfer occurs between two phases without dispersion of one phase within another. That mass transfer is diffusive transport and by assuming steady-state conditions the molar absorption rate is described by Fick’s first law, defined by eq. (1).

\[ J_A = -D_A \left( \frac{\partial \mu_A}{\partial x} \right) \] (1)

\( J_A \) is the flux of a component A across a surface, \( D_A \) the diffusion coefficient of the component, \( x \) the distance and \( \mu_A \) is the chemical potential. The absorption rate, which is the same as the flux, is then driven by the difference in chemical potential across the interface and will have a continuous profile as shown in fig. 1.

To simplify the calculation, we split the diffusion into three segments. The first segment is from the gas phase into the membrane surface (Phase \( \alpha \)), the second segment is the membrane, and the last segment is from the membrane surface at into the liquid bulk (Phase \( \beta \)). For the two first segments, the mass transport rate is fast (3). We, therefore, simplify by assuming very fast diffusion from the gas phase into the interphase at the boundary between the membrane and the liquid phase. The flux is then limited by the solubility of CO$_2$ in water. By doing the assumption of fast transport into membrane limited by the solubility, the flux can then be described by eq. (2), which will be used for comparison in this experiment.

\[ J_A = k_L (C_{iA} - C_{bA}) \] (2)

Where, \( k_L \) represents the average liquid phase mass transfer coefficient, \( C_{iA} \) is the interface concentration, \( C_{bA} \) is the interface concentration as given by Henry’s law, and the liquid bulk concentration is represented by \( C_{bA} \). The mass transfer
coefficient can be estimated using the Sherwood number, which describes the ratio between convective and diffusive mass transport, defined by eq. (3).

\[ Sh = \frac{k_L d}{D_A} \]  

(3)

\( Sh \) is the Sherwood number, \( d \) is diameter and \( D_A \) is the diffusion coefficient. The Sherwood number can be estimated using the analogy of Leveque’s solution for heat transfer (4).

For \( Gz < 10 \), \( Sh = 3.67 \)

(4)

For \( Gz > 20 \), \( Sh = 1.62 (Gz)^{\frac{1}{3}} \)

(5)

For the intermediate range of Graetz numbers the Sherwood number can be estimated using the definition defined by eq. (6).

\[ Sh = (3.67^3 + 1.62^3 Gz)^{\frac{1}{3}} \]  

(6)

With \( Gz \) representing the Graetz number defined by eq. (7).

\[ Gz = \frac{v_L d^2}{D_A L} \]  

(7)

Where, \( v_L \) is the liquid velocity and \( L \) is the length of the fiber. By assuming laminar flow of liquid and a fully developed velocity profile through the hollow fiber, the bulk concentration in the liquid at any axial distance, \( z \), is given by (8) as defined in ??.

\[ C_A^b \bigg|_{z} = C_A^i \left[ 1 - \exp \left( -4k_L \frac{L}{v_L d} z \right) \right] \]  

(8)

Where \( v \) is the liquid velocity and \( d \) is the diameter of the pipe. The average bulk concentration in the fiber, to be used in eq. (2), can be obtained from the integration of \( C_A^b \) over the length of the fiber, \( L \), as defined in eq. (9).

\[ \langle C_A^b \rangle = \frac{1}{L} \int_0^L C_A^b \, dz \]  

(9)

The model defined by eq. (2) is valid only for physical absorption of gas with constant gas-liquid interface conditions. This is not the case in this experiment. A high partial pressure would impose a high absorption rate, which will reduce the total pressure in the gas phase. This will lead to a significant drop in the partial pressure in the gas phase, and therefore a lower concentration at the gas-liquid interface across the length of the fiber. In this situation, high deviations may occur between the flux predicted by the model and experimentally measured flux, particularly at high liquid flow rates, when the absorption is higher.

3 Set-up and experimental procedure

The experimental set-up is illustrated in fig. with the corresponding symbol explanations included in table IV.

Table 1: Unit descriptions for of the membrane contactor rig in illustrated in fig. 2

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image" alt="Check valve" /></td>
<td>Check valve</td>
</tr>
<tr>
<td><img src="image" alt="Controlled valve" /></td>
<td>Controlled valve</td>
</tr>
<tr>
<td><img src="image" alt="Humidifier" /></td>
<td>Humidifier</td>
</tr>
<tr>
<td><img src="image" alt="IR-sensor" /></td>
<td>IR-sensor</td>
</tr>
<tr>
<td><img src="image" alt="Manually operated 3-way valve" /></td>
<td>Manually operated 3-way valve</td>
</tr>
<tr>
<td><img src="image" alt="Mass flow" /></td>
<td>Mass flow</td>
</tr>
<tr>
<td><img src="image" alt="Measurement of variable x" /></td>
<td>Measurement of variable x</td>
</tr>
<tr>
<td><img src="image" alt="Membrane contactor module" /></td>
<td>Membrane contactor module</td>
</tr>
<tr>
<td><img src="image" alt="Pump" /></td>
<td>Pump</td>
</tr>
<tr>
<td><img src="image" alt="Reservoir" /></td>
<td>Reservoir</td>
</tr>
<tr>
<td><img src="image" alt="Safety valve" /></td>
<td>Safety valve</td>
</tr>
</tbody>
</table>

Set-up description

The setup is designed for having a mixture of CO₂ and N₂ in contact with a solvent, which will be water for this experiment, using a membrane contactor. This facilitate for absorption of components between the contacting phases. The membrane contactor module used in this experiment consists of two membrane contactors coupled in parallel, doubling the total capacity. Each of the two membrane contactors contains thousands of small tubes densely packed together with a diameter of each tube in the scale of \( \mu m \). The result is that the relative surface area is very large compared to total volume. The walls of these tubes are the membrane fiber. The solvent will be inside the tubes and the gas will be on the outside. Physical parameters on the membrane are available either in data sheets located in the lab or measurable. The letters in the measurements are P for pressure, H for humidity, F for flow, T for temperature. The controllers also have an information bubble, and the C stands for controller. Set-points for the controllers are manipulated via the software. The raw data from the IR-sensor is a number between \( 0 \) and \( 2^{16} \), which you have to calibrate to a number that corresponds to the concentration before you can start the experiment. More information on the manipulation of the set-points for the controller and the calibration of the IR sensor is given below.
Figure 2: This figure illustrates the experimental setup for the membrane contactor rig. The units are explained in table I.

Experimental

A summary of the experimental procedure is given below.

i Fill 25 liters of distilled water into the feeding tank and add a small amount of dissolved BTB (Bromothymol blue).

ii Add the required amount of NaOH 0.01 M into the feeding tank.

iii Give the controllers the selected set-points. More information on the controllers below.

iv Calibrate the IR sensor in the region you expect to operate. More information on how to perform the calibration below.

v Start the experiment by pumping the feeding water into the lumen of the hollow fibers. Start with a low pump velocity. Before you start taking measurements, make sure that there is no air in the liquid feeding hose.

vi When the concentration of CO₂ stabilizes, determine the liquid flow rate by measuring the time necessary to collect a given volume of liquid. This have be done manually using a measuring cylinder and a stop watch. Record gas temperatures, flow rates of CO₂ and N₂ in the gas inlet, pressures in liquid and gas phases in both inlet and outlet and composition of the outlet gas stream. This is handled by the software. To start the measuring press 'Start'. Stop the measuring by pressing 'Stop Sampling'.

vii Repeat the two previous point with an increasing liquid flow rate by increasing the pump speed. Collect data for ten different flow rates. As the experiment progresses, you should be able to observe a color change from blue to yellow.

viii After the ten experiments, re-set the controllers, transfer your data and clean up.

Table 2: Controllers in the membrane contactor rig. The setpoints for the flow controllers will be decided by you. The total flow in the gas phase should not exceed 0.5 L/min. Note that the pressure in the gas phase is given in absolute pressure, while the pressure in the liquid phase uses relative pressure.

<table>
<thead>
<tr>
<th>Controller</th>
<th>Description</th>
<th>Set-point</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>CO₂</td>
<td>Flow controller for pure CO₂ in</td>
<td>-</td>
<td>$\frac{l}{min} (STP)$</td>
</tr>
<tr>
<td>N₂</td>
<td>Flow controller for pure N₂ in</td>
<td>-</td>
<td>$\frac{l}{min} (STP)$</td>
</tr>
<tr>
<td>$p_g$</td>
<td>Pressure controller for the gas outlet</td>
<td>17</td>
<td>psia</td>
</tr>
<tr>
<td>$p_l$</td>
<td>Pressure controller for the liquid outlet</td>
<td>3</td>
<td>psig</td>
</tr>
</tbody>
</table>
Setup of controllers

There are in total four controllers that require a given set-point. These controllers are listed and specified in table 2. Note that pressure controller for the gas phase operates with absolute pressure, while the pressure controller for the liquid phase uses relative pressure. The set-point for the flow controllers will be decided by you, but the total amount of gas should not exceed 0.5 \( \frac{L}{min} \). The flow rates for the flow controllers are set in SLPM\(^1\).

Calibration of the IR-sensor

The IR-sensor is used to measure content of CO\(_2\) in the retentate. It has to be calibrated before usage. The measurement read an analog signal in the range from 4mA – 20mA, which is translated 16-bit number before it is available to us. This means that we get a number between 0 and 2\(^{16}\) which we have to translate to a percentage. You have to perform a regression in the lab similar to the illustration included in fig. 3.

i Bypass the membrane unit by selecting the bypass using the 3-way valve.

ii Set the controllers to the setpoints.

iii Read the 16-bit number when the number is stable.

iv Reduce the amount of CO\(_2\) by setting a lower set-point for that flow controller and take new measurements when stable. Repeat this a couple of times.

v Make your regression and put it into the software.

vi Set the controllers back to the point before calibration and lead the gas flow into the membrane unit using the 3-way valve.

\[ y = ax + b \]

Figure 3: Example of a linear calibration curve. The \( x \) is the bit reading measured, which have to be translated to a percentage, \( y \), using regression.

\(^1\)Standard Litres Per Minute, 0°C, 14.6959 psia

4 Data treatment

You will use eq. 2 as comparison to your experimental results. The computer is not connected to the Internet, so please bring a USB-stick to transfer the measurements.

Make sure that you read the risk a assessment before you do the experiment.

Work plan

In addition to the tasks described in the guideline for work plans for the Felleslab, some more specific information for the this experiment is given here. Read the comments on what to include in the report, so that you prepare to perform the experiment in such a way that you will be able to answer the questions raised there. Based on what you shall do in the experiment, the following should be included in the work plan:

- Some background and theory:
  - Write a short theory summary that includes the equations required for the calculations. This can be included in the final report.
  - How to prepare a 25 liter solution of pH 7.5 from a 0.01 M solution of NaOH.
  - What is purpose of the first humidifier included in the experiment?
  - How can you determine the absorption rate from the available measurements?
  - Prepare how to do a linear regression for the calibration of the IR-sensor in the lab.

- Procedure:
  - A description of what you will do in the experiment and operating procedure.
  - Name the measurements you have to take and the parameters you require to do the calculations.

- HSE:
  - Name the possible risks.
  - According to your opinion, what is the largest risk with the experiment?
  - What will you do to avoid or reduce this risk?

The work plan is to be handed in no later than two working days before lab day. It has to be approved by your supervisor before you can gain access to the lab. As this might mean you need to revise it, you are advised to deliver at your earliest convenience.
The report should follow the guideline for report for the Felleslab, some more specific information for this experiment is given here. Plot the flux of CO\textsubscript{2} as a function of the liquid velocity. Plot both the measured flux and the theoretical flux you have estimated from your model eq. (2). Data for solubility and diffusivity input parameters can be found in (6) and membrane parameters is available in the lab. Compare your results with the model at different flowrates. Comment on the change in composition of the gas phase, throughout the experiment, and how this affects the results. Also comment on the applicability of the model in this experiment.

You will get a lot of data after you have carried out your experiment. Filter out the measurements you require to do your calculations and any measurements relevant in your discussion and place them in the appendix. You are also required to do sample calculations with numbers and units for one run. It is sufficient to refer to the equation in your theory section and write it out with numbers and units in the appendix. The calibration of the IR-sensor is also to be included in the appendix.

Bibliography


