



FAMU–FSU College of Engineering  
Department of Chemical and Biomedical Engineering

## Transport Phenomena Laboratory



### Experiment

### **380 – Absorption Tower**

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## Executive Summary

### Purpose

Experiment 380 consists of an absorption tower that was utilized to determine the effects of the inlet water flow rate and inlet air flow rate on the rate of CO<sub>2</sub> absorption ( $n_{abs}$ ) and the overall volumetric mass transfer coefficient in the liquid phase ( $K_x a$ ). The main trials were conducted at randomized combination of levels. The inlet air flow rate levels were 10 SLPM and 20 SLPM. The inlet mass flow rate levels were 2 kg/min and 8 kg/min. For the ODEX and SODEX trials the effects of temperature and mole fraction of CO<sub>2</sub> in the inlet gas stream were investigated, respectively.

### Procedure

The main apparatus used was a packed bed absorption tower with a countercurrent flow of liquid and gas, with the water flowing downwards and the gas flowing upwards. The inlet water flow rate was measured at the beginning of trial and after adjusting the factors we waited for the system to reach steady state before recording any data.

### Results and Predictions

With the values of the mole fraction of CO<sub>2</sub> in the inlet and outlet gas streams collected we calculated and analyzed the rate of CO<sub>2</sub> absorption and the overall volumetric mass transfer coefficient in the liquid phase.

The values of  $n_{abs}$  ranged from 0.128 mmol/s to 0.681 mmol/s with a coefficient of variation of 0.078. For  $K_x a$  the values ranged from 0.25 kmol/m<sup>3</sup>\*s to 1.82 kmol/m<sup>3</sup>\*s with a coefficient of variation of 0.135. Since both responses presented a coefficient of variability greater than the alpha value of 0.05, it was determined that the trials had poor repeatability.

When doing a least square analysis to determine the parameters of an interaction model for both responses it was found that the inlet air flow rate was statistically insignificant. This consequently means that the inlet water flow rate is the factor with the greatest effect on the system responses, as expected by the prediction models.

The ODEX showed that the temperature was statistically insignificant on  $n_{abs}$  and  $K_x a$ , contradicting the prediction. The coefficient of variation for  $n_{abs}$  was 0.103 and for  $K_x a$  0.065, indicating poor repeatability of the trials.

The SODEX showed that the mole fraction of CO<sub>2</sub> in the inlet gas stream affects  $n_{abs}$  and  $K_x a$  in a directly proportional way. The Rsquare value outputted by JMP Pro was 0.99, indicating that the experimental data fits well the model.

### Conclusions and Recommendations

We recommend that when analyzing the effect of temperature operators choose a larger temperature difference than 5°C to see if the temperature would then be statistically significant. We also recommend that future operators are well familiar with all the apparatus limitations, as well as consider the weather since that will affect the inlet water temperature, which should be kept constant.

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## Introduction

### *Background*

An absorption tower is an important equipment for mass transfer of gases and liquids, and it is widely used in industrial processes for separation, filtration, distillation, and purification. A critical aspect of absorption is to ensure that the reactant is efficiently absorbed from the gas phase into the liquid phase. If there is not an efficient absorption, then it is more likely that the reaction may produce undesirable byproducts.

An important application in the industry is the use of packed towers for air pollution control, where a countercurrent design allows for high absorption of many toxic gases. This is achieved by flowing a type of scrubbing liquid, usually water, downwards by gravity over the packing material and flowing the gas upwards, which allows the liquid to clean the gas by absorbing its toxic components before it is released to the environment.

Similar to the application mentioned above, this experiment used an absorption tower with a countercurrent design to clean the CO<sub>2</sub> present in the air by using water. The main goal of conducting this experiment was to analyze the effect of inlet water flow rate and inlet air flow rate on the mole fraction of CO<sub>2</sub> in the outlet stream, the rate of CO<sub>2</sub> absorption, and the overall mass transfer coefficient.

### *Experiment Objectives*

**1a.** Utilize the DOE tool in JMP Pro to plan for trials and complete table 1. Determine experimentally the performance of the absorption column at the conditions listed in table 1 for replication 1 (trials 1a...1d). Repeat the experimental trials using the experimental conditions listed for replication 2 (trials 2a...2d) and, if time is available, using the experimental conditions listed for replications 3, 4, and 5 (trials 3a...3d, trials 4a...4d, trials 5a...5d).

Characterize the performance by the following three criteria:

- $y_a$ , the carbon dioxide content in the outlet gas stream (in units of mole fraction)

- $n_{\text{abs}}$ , the rate of carbon dioxide absorption (in units of mmol/s)
- $K_{\text{x}a}$ , the overall volumetric liquid-phase mass-transfer coefficient [in units of kmol/(s·m<sup>3</sup>)].

**1b.** Assess the stability of the carbon dioxide analyzer in terms of its calibration constant. Evaluate whether the frequency of desiccant replacement suffices.

**1c.** Determine the repeatability of  $n_{\text{abs}}$  and the repeatability of  $K_{\text{x}a}$  in terms of pooled standard deviation.

**1d.** Apply least-squares analysis at the actual conditions of the trials to determine the parameters of an interaction model for the rate of carbon dioxide absorption.

$$\hat{y} = b_0 + b_1 X_1 + b_2 X_2 + b_{12} X_1 X_2$$

where  $\hat{y} = n_{\text{abs}}$  = the rate of carbon dioxide absorption (in units of mmol/s)

$X_1$  = inlet flow rate of air (coded)

$X_2$  = inlet flow rate of water (coded)

In addition, apply least-squares analysis at the actual conditions of the trials to determine the parameters of an interaction model for the overall volumetric liquid-phase mass-transfer coefficient [in units of kmol/(s·m<sup>3</sup>)]. That is, determine the parameters in the above equation for  $\hat{y} = K_{\text{x}a}$ .

Estimate the standard error of each parameter and test for the statistical significance of each parameter.

**1e.** Predict  $K_{\text{x}a}$ ,  $n_{\text{abs}}$ , and  $y_a$  at the same conditions as those for the experimental trials. Use a spreadsheet with empirical correlations to make the predictions. Compare these predictions to the experimental results in terms of discrepancy as defined below.

$$\text{Disc}[K_{\text{x}a}] = [K_{\text{x}a}]_{\text{pred}} - [K_{\text{x}a}]_{\text{expt}}$$

$$\text{Disc}[n_{\text{abs}}] = [n_{\text{abs}}]_{\text{pred}} - [n_{\text{abs}}]_{\text{expt}}$$

$$\text{Disc}[y_a] = [y_a]_{\text{pred}} - [y_a]_{\text{expt}}$$

Characterize overall model performance for all the trials by computing the root mean square of  $\text{Disc}[K_x a]$ ,  $\text{Disc}[n_{\text{abs}}]$ , and  $\text{Disc}[y_a]$ .

- 1f.** Predict  $n_{\text{abs}}$ , and  $y_a$  at the same conditions as those for the experimental trials. Use Aspen Plus to make the predictions. Compare these predictions to the experimental results in terms of discrepancy as defined previously. Characterize overall model performance for all the trials as explained above.
- 2.** Demonstrate that each team member has achieved proficiency in operating all aspects of the apparatus.
- 3.** (ODEX) Design and conduct an experiment to determine the effect of the liquid feed stream temperature on  $\text{CO}_2$  absorption.
- 4.** (SODEX) Design and conduct a new experiment to determine the effect of the inlet  $\text{CO}_2$  volumetric flow rate on  $\text{CO}_2$  absorption.

### ***Process Description***

Experiment 380 consists mainly of an absorption tower and four streams. The water path consists of a domestic source that is directed to a feed tank and flows to the tower using a pump. In order to obtain lower water temperature, there is a chilled water source that goes through a heat exchanger and the coils inside the feed tank. For a higher water temperature, there is a heater that connects to the feed tank. The air flow comes from the air source, which is controlled by a valve. The carbon dioxide source is from a high-pressure cylinder. These two gas lines mix and enter the absorption tower and to the silica gel filter that connects to a carbon dioxide analyzer to read the percent of carbon dioxide going into the tower. The water exiting the tower goes to a discharge tank that drains out. Most of the gas exits the tower to the room and other small part connects to the carbon dioxide analyzer, where we collect our data from.



When entering the lab we first had to ensure all members were wearing the appropriate personal protective equipment which for this experiment included safety glasses and a hard hat. Before starting the experiment we confirmed that all valves were in the correct start up position and all equipment was working properly. After start up, the first step is to set up the water flow rate by adjusting two rotameters, the inlet rotameter that controls the flow of water into the feed tank and the outlet rotameter that controls the flow from the feed tank into the absorption column. Once the rotameters have been adjusted we need to physically confirm the flow rate by allowing water to accumulate in the discharge tank for 1 min and recording the initial and final values. Our target values for the water flow rate are 2 kg/min and 8 kg/min. If the recorded flow rate was within 0.2 kg/min of the target values then we moved on to the gas flow rate setup. If not we adjusted the rotameters and rerecorded the flow until we reached the desired flow rate.

The gas flow rate, both air and carbon dioxide are controlled by two separate air mass flow controllers that are connected to a computer. Using a handout that was available in the laboratory we entered our desired values for CO<sub>2</sub> and air. The desired air flow rate values were 10 Standard Liters per minute (SLPM) and 2 SLPM. For our main trial the amount of CO<sub>2</sub> in the feed should always be approximately 8% which we confirm by sampling the inlet gas with the carbon dioxide analyzer. We can adjust the CO<sub>2</sub> if needed using the computer. After the CO<sub>2</sub> is set we can then sample the gas leaving the absorption column which allows us to determine how much CO<sub>2</sub> was absorbed into the water. When sampling the outlet gas stream it is important to make sure no water is inside the tubing and that the silica gel filter meant to absorb any moisture is not more than 20 % pink which can interfere with the results.

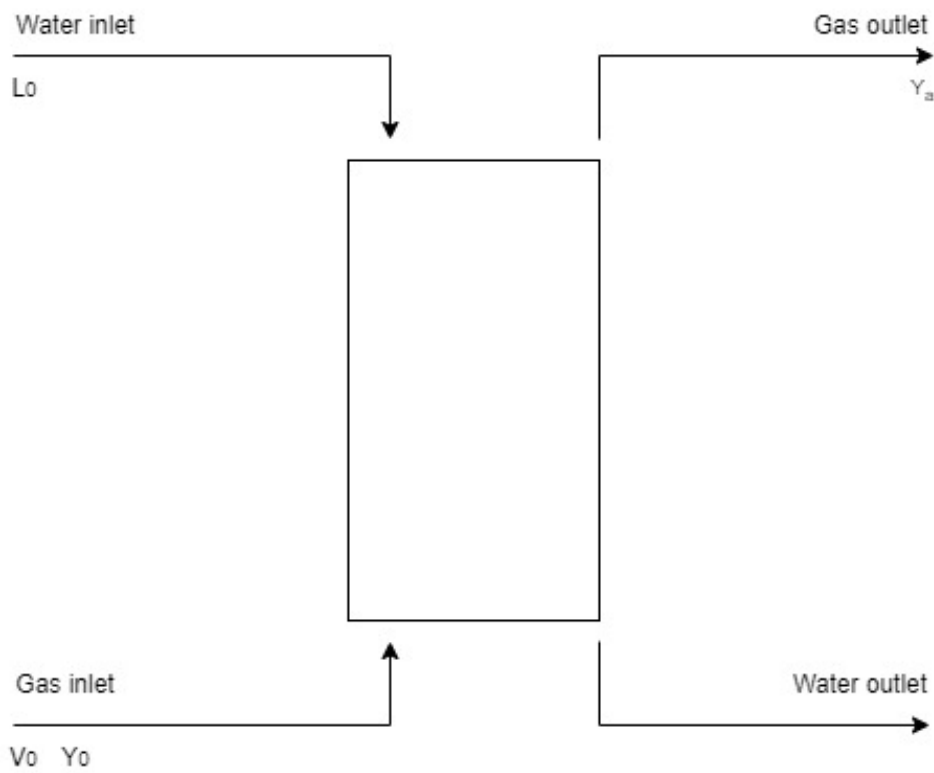
**Process Variable List**

| Primary Factors                                       |                   |                     |                     |                     |
|---|-------------------|---------------------|---------------------|---------------------|
| Variable Name   | Variable Symbol   | Units               | Target Values       |                     |
| Inlet water mass flow rate                            | $m_{La}$          | kg/min              | (-) level: 2 kg/min | (+) level: 8 kg/min |
| Inlet air volumetric flow rate                        | $v_{SA}$          | SLPM                | (-) level: 10 SLPM  | (+) level: 20 SLPM  |
| Secondary Factors                                     |                   |                     |                     |                     |
| Variable Name   | Variable Symbol   | Units               | Target Values       |                     |
| Mole fraction of CO <sub>2</sub> in supplied air      | $y_{SA}$          | Unitless            | 0.08                |                     |
| Temperature of water at top of packing                | $\Theta_{La}$     | °C                  | 25 °C               |                     |
| Inlet CO <sub>2</sub> volumetric flow rate            | $v_{SC}$          | SLPM                | (-) level: 1350 MFC | (+) level: 2544 MFC |
| Primary Responses                                     |                   |                     |                     |                     |
| Variable Name   | Variable Symbol   | Units               |                     |                     |
| CO <sub>2</sub> analyzer reading at top of packing    | $g_{araw}$        | %                   |                     |                     |
| CO <sub>2</sub> analyzer reading at bottom of packing | $g_{braw}$        | %                   |                     |                     |
| Secondary Responses                                   |                   |                     |                     |                     |
| Variable Name   | Variable Symbol   | Units               |                     |                     |
| Temperature of gas at top of packing                  | $\Theta_{Ga}$     | °C                  |                     |                     |
| Temperature of liquid at bottom of packing            | $\Theta_{Lb}$     | °C                  |                     |                     |
| Temperature of gas at bottom of packing               | $\Theta_{Gb}$     | °C                  |                     |                     |
| Pressure-head drop across the packing                 | $\Delta h_{H_2O}$ | in-H <sub>2</sub> O |                     |                     |

|   |           |                       |
|---|-----------|-----------------------|
| Rate of absorption of CO <sub>2</sub>                     | $n_{abs}$ | mmol/s                |
| Overall volumetric liquid-phase mass-transfer coefficient | $K_x a$   | kmol/s·m <sup>3</sup> |

**Table 1.** Factors and target values for the main experiment and all other variables used to analyse the results.

**Process Concept Sketch**



**Figure 1.** Process concept sketch.

## **Theoretical Background**

### *Instrumentation – Calibration of the Carbon Dioxide Analyzer*

We can collect data at different values of carbon dioxide to relate  $y_b$  to  $g_{braw}$ . The relationship is approximately linear, shown in the equation below:

$$g_{braw} = m_{calib} * y_b + b_{calib}$$

where  $g_{braw}$  = analyzer reading at a point b

$y_b$  = actual mole fraction of carbon dioxide

$m_{calib}$  = calibration slope

$b_{calib}$  = calibration intercept

We can then do the same analysis at point a to determine  $y_a$ :

$$y_a = \frac{g_{araw} - b_{calib}}{m_{calib}}$$

However, for our apparatus we found  $b_{calib}$  to be zero therefore the equations become:

$$m_{calib} = \frac{g_{braw}}{y_b}$$

$$y_a = \frac{g_{araw}}{m_{calib}}$$

### *Trial Conditions*

The cross-sectional area of the column is found by:

$$S = \frac{\pi D_i^2}{4}$$

where  $D_i$  = internal diameter of the column [m]

The volume of the packed section can be found by multiplying equation (1) by the total height of the packed section:

$$SZ_T = S * Z_T$$

Further equations require us to know the pressure at the bottom of the packing, which can be calculated by summing the pressure at the top of the packing to the pressure drop across the packing:

$$P_b = P_a + \rho_{H_2O} * g * \Delta h_{H_2O}$$

where  $P_a$  = pressure at the top of the packing [Pa]

$\Delta h_{H_2O}$  = pressure-head drop across the packing [ft]

However, the operators found that for every trial the pressure-head drop across the packing was zero, meaning that we can consider  $P_a = P_b$  for the remainder of the calculations.

One of the factors in this experiment is the inlet volumetric flow rate of air, which is measured in SLPM on the equipment. In order to proceed with the calculations for our responses we need to convert this variable to mol/s:

$$V_{SA} = \frac{P_{std} * v_{SA}}{R * T_{std}}$$

where  $P_{std}$  = pressure at standard conditions of gas [Pa]

$v_{SA}$  = standard volumetric flow rate of supplied air at the top of the packing [std m<sup>3</sup>/s]

$T_{std}$  = temperature at standard conditions of gas [K]

$R$  = molar gas constant = 8.314 J/K·mol

Since we are interested in the rate of CO<sub>2</sub> absorption, we first need to determine the mole fraction of CO<sub>2</sub> in the outlet gas stream so that we can later find how much CO<sub>2</sub> was absorbed by the water by relating it to the difference of the mole fraction of CO<sub>2</sub> in the inlet and outlet gas streams:

$$y_b = \frac{y_{SA} * v_{SA} + v_{SC}}{v_{SA} + v_{SC}}$$

where  $y_{SA}$  = mole fraction of CO<sub>2</sub> in supplied air

$v_{SC}$  = standard volumetric flow rate of supplied CO<sub>2</sub> [std m<sup>3</sup>/s]

By relating equations () and () we can find the molal flow rate of the gas stream at the bottom of the packing:

$$V_b = \frac{(1 - y_{SA}) * V_{SA}}{1 - y_b}$$

Now that we have defined the variables for the gas stream at the trial conditions we can move forward to the liquid stream. First, we need to find the molar mass of the liquid by using the molar masses of CO<sub>2</sub> and water, which were acquired from CHEMCAD:

$$M_{La} = (1 - x_a) * M_{H2O} + x_a * M_{CO2}$$

where  $M_{H2O}$  = Molar mass of water = 0.018 kg/mol

$M_{CO2}$  = molar mass of CO<sub>2</sub> = 0.044 kg/mol

$x_a$  = mole fraction of CO<sub>2</sub> in liquid at the top of the packing

One of the factors used in this experiment was the inlet mass flow rate of water, which was measured in the laboratory by the operators. This flow rate can be divided by equation () to find the molal flow rate of the liquid:

$$L_a = \frac{m_{La}}{M_{La}}$$

where  $m_{La}$  = inlet mass flow rate of water [kg/min]

*Equipment performance:  $n_{abs}$  and  $K_x a$*

Using the same concept of how we found the molal flow rate of the gas stream at the bottom of the packing through equation (), we can find the molal flow rate of the gas stream at the top of the packing by:

$$V_a = \frac{(1 - y_{SA}) * V_{SA}}{1 - y_a}$$

Finally, we can determine what is the rate of CO<sub>2</sub> absorption for each trial by using the following equation:

$$n_{abs} = y_b * V_b - y_a * V_a$$

Now that we have defined  $n_{abs}$  we can proceed to finding the overall volumetric liquid-phase mass-transfer coefficient. First, we need to calculate the molal flow rate of the liquid stream at the bottom of the packing. Since the water is absorbing the CO<sub>2</sub> that means that the molal flow rate of water at the bottom of the packing is simply the sum of the molal flow rate of water at the top of the packing and the rate of CO<sub>2</sub> absorption:

$$L_b = L_a + n_{abs}$$

The rate of CO<sub>2</sub> absorption can also be written in terms of the liquid stream by:

$$n_{abs} = x_b * L_b - x_a * L_a$$

Extracting  $x_b$  from equation () we can find an equation for the mole fraction of CO<sub>2</sub> in the liquid stream at the bottom of the packing:

$$x_b = \frac{x_a * L_a + n_{abs}}{L_b}$$

The next step into finding  $K_x a$  is to determine the equilibrium mole fraction of CO<sub>2</sub> in the liquid at the top and bottom of the packing. These variables can be acquired by dividing the partial pressure of CO<sub>2</sub> in gas by Henry's law coefficient.

$$x_a^* = \frac{y_a * P_a}{H_a}$$

where  $x_a^*$  = equilibrium mole fraction of CO<sub>2</sub> in the liquid at the top of the packing

$H_a$  = Henry's law coefficient at the top of the packing =  $1.396 \cdot 10^8$  [Pa]

$$x_b^* = \frac{y_b \cdot P_b}{H_b}$$

where  $x_b^*$  = equilibrium mole fraction of CO<sub>2</sub> in the liquid at the bottom of the packing

$H_b$  = Henry's law coefficient at the bottom of the packing =  $1.376 \cdot 10^8$  [Pa]

By subtracting the mole fraction of CO<sub>2</sub> from the equilibrium mole fraction of CO<sub>2</sub> we can find the liquid-phase driving force.

$x_a^* - x_a$  = liquid-phase driving force at the top of the packing

$x_b^* - x_b$  = liquid-phase driving force at the bottom of the packing

Then, we can determine the logarithmic mean liquid-phase driving force by:

$$(x^* - x)_{im} = \frac{(x_a^* - x_a) - (x_b^* - x_b)}{\ln \frac{x_a^* - x_a}{x_b^* - x_b}}$$

Finally, we can find an expression for  $K_x a$  by relating the rate of CO<sub>2</sub> absorption, the volume of the packing and the logarithmic mean liquid-phase driving force:

$$K_x a = \frac{n_{abs}}{SZ_T \cdot (x^* - x)_{im}}$$

*Predicted performance:  $K_x a$*

Before conducting any experiment, the operator must have a hypothesis and predict what results they expect to achieve. Prediction models can also help analyzing your experimental data based on empirical correlations. In this section we will introduce equations that are used to predict  $n_{abs}$  and  $K_x a$  for this system.

First, we will start by defining constant variables that will be used throughout the calculations. These values were taken from either Chemcad, McCabe 7<sup>th</sup> edition, or Geankoplis 4<sup>th</sup> edition.

$\mu_r$  = viscosity of water at 25°C = 0.000922 Pa·s



$(N_{Sc})_r$  = Schmidt number for oxygen in water at 25°C = 381

$\rho_r$  = density of water at 25°C = 997 kg/m<sup>3</sup>

$D_r$  = diffusivity of oxygen in water at 25°C = 2.41\*10<sup>-9</sup> m<sup>2</sup>/s

$D_{298}$  = diffusivity of CO<sub>2</sub> in water at 25°C = 2\*10<sup>-9</sup> m<sup>2</sup>/s

Calculate the molar mass of the gas,

$$M_{Gb} = (1 - y_b) * M_{air} + y_b * M_{CO2}$$

where  $M_{air}$  = molar mass of air = 0.0289 kg/mol

To find the mass velocity, G, we need to multiply the molar mass by the molal flow rate and divide by the cross-sectional area of the column. Therefore, the gas mass velocity is found by

$$G_y = \frac{V_b * M_{Gb}}{S}$$

And the liquid mass velocity is found by

$$G_x = \frac{L_b * M_{H2O}}{S}$$

For this prediction model we will use an average temperature in the liquid phase,  $T_L$ , for the remainder of the calculations. We will base this temperature on the experimental temperatures for the liquid at the top of the packing ( $\theta_{La}$ ) and for the liquid at the bottom of the packing ( $\theta_{Lb}$ ) recorded during the experiment for each trial.

$$T_L = \frac{\theta_{La} + \theta_{Lb}}{2} + 273.15$$

In order to find the diffusivity of CO<sub>2</sub> in water at  $T_L$  we can use the following expression,

$$D = \frac{T_L}{298.15} * \frac{\mu_{298}}{\mu} * D_{298}$$

Then, the Schmidt number for CO<sub>2</sub> in water at  $T_L$  is

$$N_{Sc} = \frac{\mu / \rho}{D}$$

Calculate the height of a transfer unit for CO<sub>2</sub> in water at  $T_{Lb}$ ,

$$H_{Ox} = \left(\frac{\mu_r}{\mu}\right)^{0.4} * \left(\frac{N_{Sc}}{(N_{Sc})_r}\right)^{0.5} * (H_{Ox})_r$$

Finally,  $K_x a$  predicted can be found by

$$K_x a = \frac{L_b / S}{H_{Ox}}$$

*Predicted performance:  $y_a$  and  $n_{abs}$*

This section will use the Number of Transfer Units (NTU) method to find expressions that can be used to predict the values of  $n_{abs}$  and  $K_x a$ . This derivation will also assume that the inlet and outlet conditions for both the liquid and gas streams are the same, that is,  $P_a = P_b$ ,  $H_a = H_b$ ,  $V_a = V_b$ ,  $L_a = L_b$ .

First, we will do a preliminary estimate using the NTU method to calculate the number of mass transfer units ( $[N_{Ox}]_{prim}$ ), the capacity ratio ( $[R_c]_{prim}$ ), exponent in effectiveness formula ( $[\alpha]_{prim}$ ), the absorption-tower effectiveness ( $[\hat{E}]_{prim}$ ), the rate of  $CO_2$  absorption ( $[n_{abs}]_{prim}$ ), and finally the mole fraction of  $CO_2$  in the outlet stream ( $[y_a]_{prim}$ ):

$$[N_{Ox}]_{prim} = \frac{K_x a * SZ_T}{L_a}$$

$$[R_c]_{prim} = \frac{P_b}{H_b} * \frac{L_a}{V_b}$$

$$[\alpha]_{prim} = -[N_{Ox}]_{prim} * (1 - [R_c]_{prim})$$

$$[\hat{E}]_{prim} = \frac{1 - e^{[\alpha]_{prim}}}{1 - [R_c]_{prim} * e^{[\alpha]_{prim}}}$$

$$[n_{abs}]_{prim} = [\hat{E}]_{prim} * L_a * (x_b^* - x_a)$$

$$[y_a]_{prim} = y_b - \frac{[n_{abs}]_{prim}}{V_b}$$

Calculate the molal flow rate of gas in the outlet stream,

$$[V_a]_1 = V_b - [n_{abs}]_{prim}$$

Then, calculate the mole fraction of  $CO_2$ ,

$$[y_a]_1 = \frac{y_b * V_b - [n_{abs}]_{prim}}{[V_a]_1}$$

To predict  $y_a$  we need to use the solver function on Excel. We will need to guess a value for  $y_a$  and use this function to find its true value based on the following constraints:

$$[y_a]_{max} = y_b - 0.0002$$

$$[y_a]_{min} = y_b - \frac{L_a}{V_b} * (0.98 * x_b^* - x_a)$$

Once  $y_a$  has been solved for we can find the molal flow rate of the gas by

$$V_a = \frac{(1 - y_{SA}) * V_{SA}}{1 - y_a}$$

Finally, the rate of CO<sub>2</sub> absorption can be found by using the following expression,

$$n_{abs} = y_b * V_b - y_a * V_a$$

### *Statistical Analysis*

In order to determine repeatability, outliers, and other significant results in our experiment, we followed statistical calculations.

The average of a set of values:

$$\bar{y} = \frac{\sum y_i}{n}$$

The variance of a sample in a set of values:

$$S_y^2 = \frac{\sum (y_i - \bar{y})^2}{n - 1}$$

Which will be used for the standard pooled deviation

$$S_p = \sqrt{S_y^2}$$

And finally, we can solve for coefficient of variation

$$CV = \frac{S_p}{\bar{y}}$$

When data points are suspected to be outliers, we use Grubb's test to determine if we can neglect them or not by using the following formulas:

First, we calculate the mean of the data points using the previous explained formula for average and the standard deviation as well. Then, we calculate a G value for each data point that is suspected to be an outlier:

$$G = \frac{y_i - \bar{y}}{S_p}$$

When we obtain a value for G, we want to compare it to G critical.

$$G_{critical} = \frac{n - 1}{\sqrt{n}} \sqrt{\frac{(t_{n-2}^{\alpha})^2}{n - 2 + (t_{n-2}^{\alpha})^2}}$$

If our value for G is bigger than G critical, it can be identified as an outlier.

## **Operations**

### ***Safety Performance***

All operators, including cross-trainers, were properly trained in all the safety aspects of the experiment by completing a safety quiz and Operational Readiness Check. The operators also successfully performed the experiment and used the apparatus without causing any accidents or incidents.

### ***Productivity***

During the first day the operators noticed right away that the domestic water supply was coming in at 21°C as it was a cold day outside and that heavily affects the inlet water temperature. Since the main trials needed to be performed at 25°C, the operators needed to use the heater so that the ideal temperature was reached. Not only that, but since the heater does not allow for a specific temperature to be set, the operators also needed to use the chilled water supply to balance the heat transfer by finding an optimal flow rate, where the inlet water would be maintained at 25°C and there would also be a constant overflow in the tank. In virtue to this, the operators started performing the first trial later than expected, which affected the predicted productivity of the day. Thus, instead of performing the 12 trials that were planned, the operators were only able to perform 10 trials.

During the second day in the laboratory the operators had already decided to perform all ODEX trials and had asked the lab engineer to set the inlet water temperature to 35 °C. The first trial performed was at the lower inlet flow rate of water, but once the operators started performing the second trial at the higher flow rate, they realized that the inlet water temperature dropped a few degrees Celsius and could no longer be maintained at the desired temperature. The operators then decided that they should restart all trials and this time operate at 30 °C, since

this temperature was able to be maintained at any level of the water flow rate. Even though some time was lost at the beginning, the operators wanted to ensure that the data was reliable by not having major discrepancies in the temperature. Despite of this, the operators were able to conduct 11 out of the 12 trials planned during this day.

The last day was the most challenging since the operators had to perform a total of 15 trials: 12 SODEX trials, 2 ODEX trials, and 3 main trials. Thankfully the operators had a great start and because no issues came up throughout the day, the operators were able to perform every trial in a timely manner and achieve the expected goal of the day.

### ***Operational Proficiency***

All operators were well trained before the first laboratory period by writing an operating procedure, successfully passing an Operational Readiness Check conducted by the teaching assistant, and by studying the equipment and asking questions beforehand. The operators felt well prepared when coming into the laboratory during the first day and were confident on what they had to do. The team was also organized and proficient when collecting data and had already created an Excel workbook prior to the first day with the data sheets needed to record all the data gathered from the experiment.

### ***Equipment Operability***

The only issue that the operators encountered was that during the second laboratory day the operators noticed that even though the heater was on, and the thermometer should be displaying the temperature of the inlet water as 35 °C, the thermocouple was displaying a temperature of around 24 °C. After waiting a few minutes there was no change in this temperature measurement and thus, the operators concluded that either the thermocouple or the heater was not working properly. After notifying the teaching assistant and lab engineer, they found that the thermocouple had a loose connection and was not measuring the temperature correctly. In order to not lose time the teaching assistant helped the operators measure the inlet water temperature with a thermometer, which allowed us to confirm that it was at the

desired temperature of 35 °C and the operators were able to keep working right away. Every other aspect of the equipment worked well and as expected.

### ***Operational Blunders***

When planning for the ODEX trials, the operators believed that conducting the trials at 35°C was practicable, however, during the second laboratory day they found that this temperature could only be maintained at low inlet flow rates of water since it was cold outside and thus, the heater could not provide enough heat to increase the water temperature at a higher flow rate. We advise that future experimenters look deeper into the limitations of the heater, or any other equipment that they plan on manipulating, and that these limitations are better communicated between the lab engineer, teaching assistant, and operators.

## **Results and Discussion**

### ***Data validation and reconciliation (DVR)***

During the experiment, operators collected data for the inlet water and air flow rates, the inlet and outlet mole fraction of CO<sub>2</sub>, the water and air temperatures at the top and bottom of the packing, the pressure head across the packing, and the inlet volumetric flow rate of CO<sub>2</sub>. This experimental data was then used to calculate the rate of CO<sub>2</sub> absorption and the overall volumetric liquid-phase mass-transfer coefficient.

An important aspect of the experiment was to wait for the system to reach steady state by observing the CO<sub>2</sub> analyzer readings. The operators noticed that the values shown in this equipment fluctuated around the same numbers even after waiting 15 minutes for steady state, when the recommended wait time for steady state is about 5-6 minutes. Because of this the operators were reluctant to assume that steady state had been reached but decided to record the data anyways since the values fluctuated from numbers within a certain range. The values recorded cannot be taken as the true values, as human error may have significantly affected the readings for the inlet and outlet mole fraction of CO<sub>2</sub>.

The operators were concerned about the equipment uncertainty since day one, but they decided to conduct as many replicates for each trial as time permitted because having multiple replications of experimental trials tend to average out random effects. With that being said, three replicates of each main trial, ODEX, and SODEX were conducted during the laboratory days.

For our ODEX, some data points were suspected to be outliers. To confirm this theory, a Grubb test was conducted and even though the initial guess of operators suggested that some values were meant to be disregarded because of the lack of consistency with other trials, all of our ODEX and ODEX values did not pass Grubb test, meaning that there are outliers in this experiment.

**Statistical Analysis**

Knowing that varying the operating conditions at which the trials were conducted affect the rate of CO2 absorption ( $n_{abs}$ ) and the overall volumetric liquid-phase mass-transfer coefficient ( $K_{xa}$ ), we can use the experimental data gathered during this experiment to find  $n_{abs}$  and  $K_{xa}$  for each trial, as well as their repeatability, shown in Table 2 below. These results and their significance will be analyzed in detail later in this report.

|                            | $n_{abs}$ [mmol/s] | $K_{xa}$ [kmol/s·m <sup>3</sup> ] |
|----------------------------|--------------------|-----------------------------------|
| Average                    | 0.371              | 0.898                             |
| Standard deviation         | 0.236              | 0.621                             |
| Coefficient of variability | 0.078              | 0.135                             |

**Table 2.** Repeatability of  $n_{abs}$  and  $K_{xa}$  for main trials.

**Main Trials**

**Objective 1a**

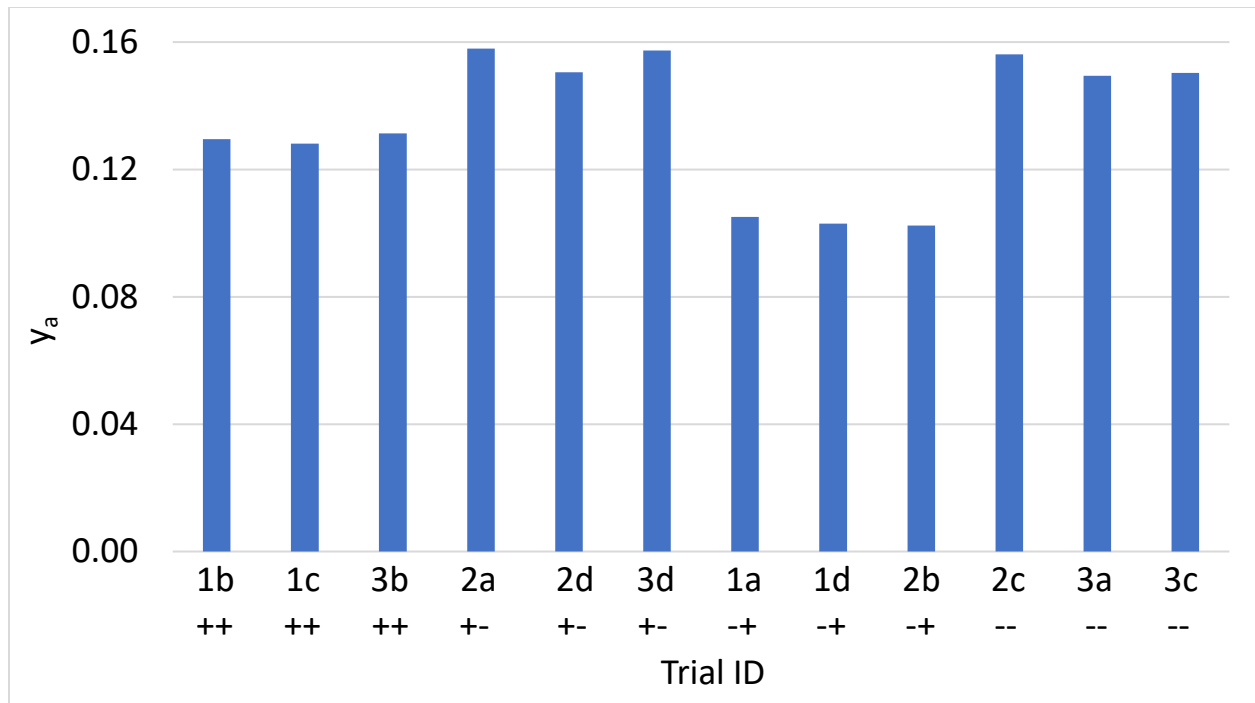
In order to ensure that the operators performed the trials in a randomized order, we utilized JMP Pro to plan for experimental trials. Based on the factors and levels, as well as the number of replications, JMP Pro provided us with the trials order and combination of factor levels as shown in Table 3 below.

| Trial Number | Factor 1<br>(Inlet air flow rate) |                     | Factor 2<br>(Inlet water flow rate) |                       |
|--------------|-----------------------------------|---------------------|-------------------------------------|-----------------------|
|              | Level                             | Target Value (SLPM) | Level                               | Target Value (kg/min) |
| 1a           | -                                 | 10                  | +                                   | 8                     |
| 1b           | +                                 | 20                  | +                                   | 8                     |
| 1c           | +                                 | 20                  | +                                   | 8                     |
| 1d           | -                                 | 10                  | +                                   | 8                     |
| 2a           | +                                 | 20                  | -                                   | 2                     |
| 2b           | -                                 | 10                  | +                                   | 8                     |
| 2c           | -                                 | 10                  | -                                   | 2                     |
| 2d           | +                                 | 20                  | -                                   | 2                     |
| 3a           | -                                 | 10                  | -                                   | 2                     |
| 3b           | +                                 | 20                  | +                                   | 8                     |
| 3c           | -                                 | 10                  | -                                   | 2                     |
| 3d           | +                                 | 20                  | -                                   | 2                     |

**Table 3.** Randomized order at which the trials were performed.

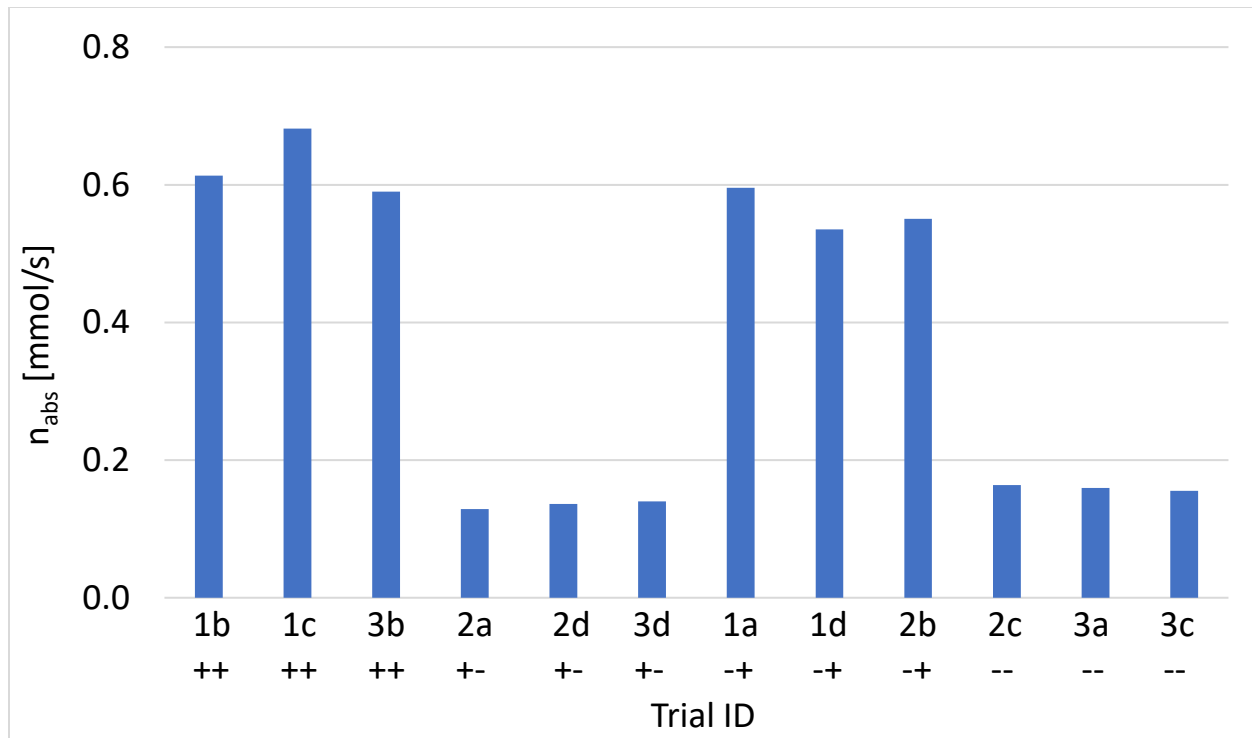
To analyze the absorption performance, we determined the CO<sub>2</sub> mole fraction in the outlet gas stream ( $y_a$ ), the rate of CO<sub>2</sub> absorption ( $n_{abs}$ ), and the overall volumetric liquid-phase mass-transfer coefficient ( $K_{x,a}$ ) for each trial.





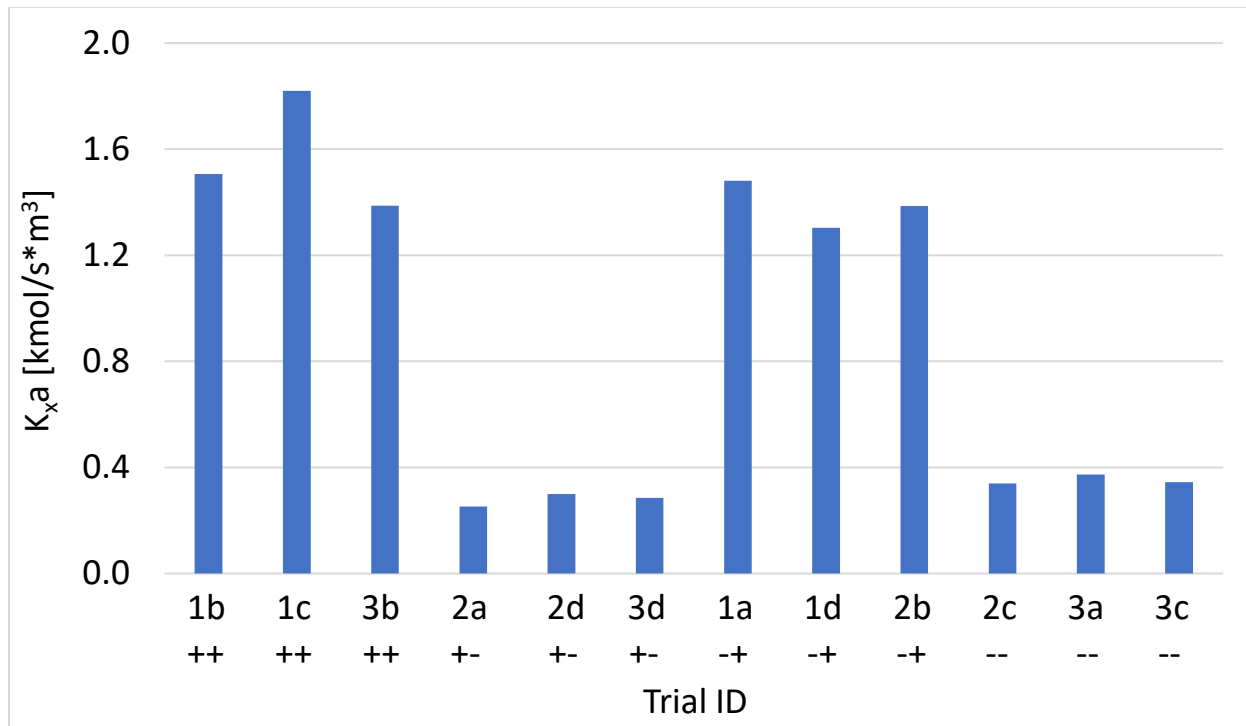
**Figure 2.** Mole fraction of CO<sub>2</sub> in the outlet gas stream for all trials. The first and second factors represent the inlet air flow rate and the inlet water flow rate, respectively.

The mole fraction of CO<sub>2</sub> in the outlet gas stream is the fraction of CO<sub>2</sub> that was not absorbed by the water. By observing Figure 2 we can notice a trend where the trials with higher inlet mass flow rate of water have a lower mole fraction of CO<sub>2</sub> in the gas outlet stream, which consequently means that the water outlet stream contains a higher mole fraction of CO<sub>2</sub>, since the water is absorbing the CO<sub>2</sub>. Likewise, the trials with the lowest level of inlet water mass flow rate shows a higher mole fraction of CO<sub>2</sub> in the outlet gas stream, that is, a lower absorption of CO<sub>2</sub> by the water.



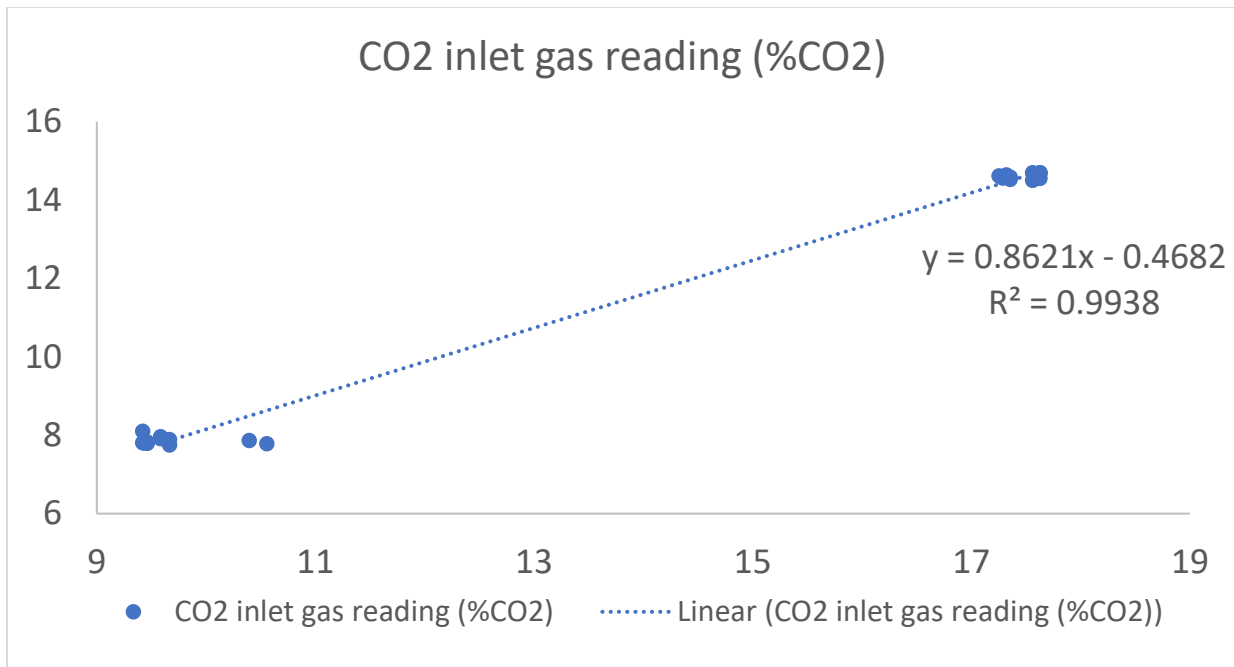
**Figure 3.** Rate of CO<sub>2</sub> absorption for all trials. The first and second factors represent the inlet air flow rate and the inlet water flow rate, respectively.

Figure 3 analyses the absorption rate of CO<sub>2</sub>, that is, how much CO<sub>2</sub> was absorbed by the water (in mmol/s). This figure is directly related to Figure 2 and is further proof that the absorption of CO<sub>2</sub> is heavily dependent on the inlet water mass flow rate. Figure 3 indicates that the higher water flow rate levels have a greater absorption of CO<sub>2</sub>, while the trials with the lowest level of water flow rate show a significantly lower absorption of CO<sub>2</sub>.



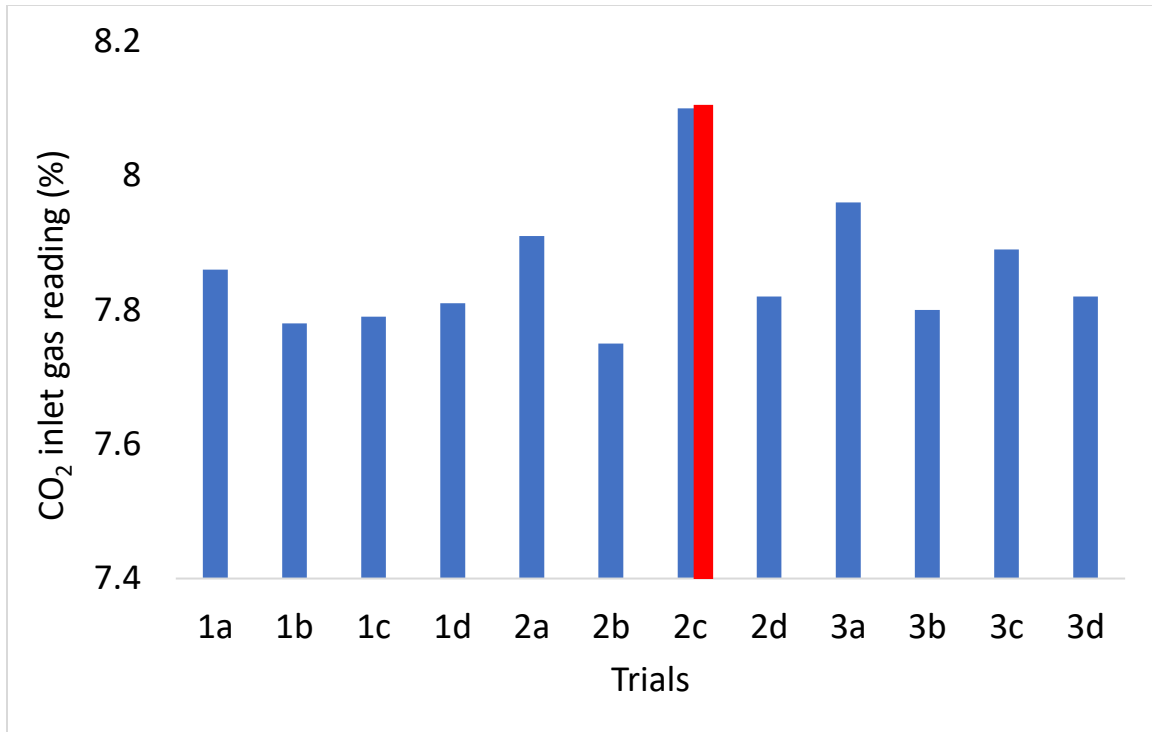
**Figure 4.** Overall volumetric liquid-phase mass-transfer coefficient for all trials. The first and second factors represent the inlet air flow rate and the inlet water flow rate, respectively.

**Objective 1b**



**Figure 5.** Calibration of CO<sub>2</sub> inlet gas reading (%CO<sub>2</sub>).

Our calibration was obtained by graphing the actual amount of carbon dioxide in the inlet versus the CO<sub>2</sub> analyzer reading in the inlet. The x axis was obtained by getting the mole fraction of the inlet with the mass controller flow rate values, while the y axis was obtained from the laboratory readings. A slope of 0.8 is close to 1, which would be ideal since we want the rise over run to be equally proportional. This leads us to the conclusion that the CO<sub>2</sub> analyzer is lacking accuracy when reading carbon dioxide values. During our laboratory period we were constantly checking if the gel filter needed a replacement, which was only the case in the first day where a 30% was pink.



**Figure 6.** CO<sub>2</sub> inlet gas reading for all trials.

The red bar observed in the graph above represents the gel filter change we made in the first day, we can see that after the change, the values returned to a similar pattern. This indicates that we should have changed the filter before performing trial 2c and that this could have interfered with our data collection in the laboratory.

### **Objective 1c**

Since each combination of levels had three replicates, summing up to a total of 12 trials, we need to find whether the operators conducted the trials with satisfactory repeatability based on the absorption rate of CO<sub>2</sub> ( $n_{abs}$ ) and the overall volumetric liquid-phase mass-transfer coefficient ( $K_L a$ ). The results are as follows:

|                            | $n_{\text{abs}}$ [mmol/s] | $K_{\text{xa}}$ [kmol/s·m <sup>3</sup> ] |
|----------------------------|---------------------------|--|
| Grand average              | 0.371                     | 0.898                                    |
| Pooled standard deviation  | 0.029                     | 0.122                                    |
| Coefficient of variability | 0.078                     | 0.135                                    |

**Table 4.** Repeatability of the rate of CO<sub>2</sub> absorption and the overall volumetric liquid-phase mass-transfer coefficient.

The coefficient of variability (C.V.) is what we can use to measure the repeatability of a sample, which in this case is all 12 trials. By determining a 95% acceptable probability of Type I error ( $\alpha = 0.05$ ), we can observe that both  $n_{\text{abs}}$  and  $K_{\text{xa}}$  have a C.V. greater than the alpha value proposed, indicating that the trials were with poor repeatability.

We believe that this low repeatability is due to the equipment uncertainty discussed in previous sections as well as the fact that not every trial had the exact same inlet water flow rate, which as shown before, this factor heavily affects the experimental data collected. We also believe that conducting more replications could have minimized random errors and provided a better repeatability for the trials, but due to time constraints the operators were only able to perform three replicates.

### **Objective 1d**

By applying least-square analysis at the trial conditions we can determine the parameters of an interaction model, as well as which factors are statistically significant based on the rate of CO<sub>2</sub> absorption. We used JMP Pro to determine the effect of the inlet air and water flow rates on  $n_{\text{abs}}$  and  $K_{\text{xa}}$ , as well as to estimate the values for the parameters in the interaction model.

First, we will analyze the effect of our factors on the rate of CO<sub>2</sub> absorption. The equation with the interaction model parameters is:

$$\hat{y} = 0.371 + 0.011X_1 + 0.223X_2 + 0.023X_1X_2$$

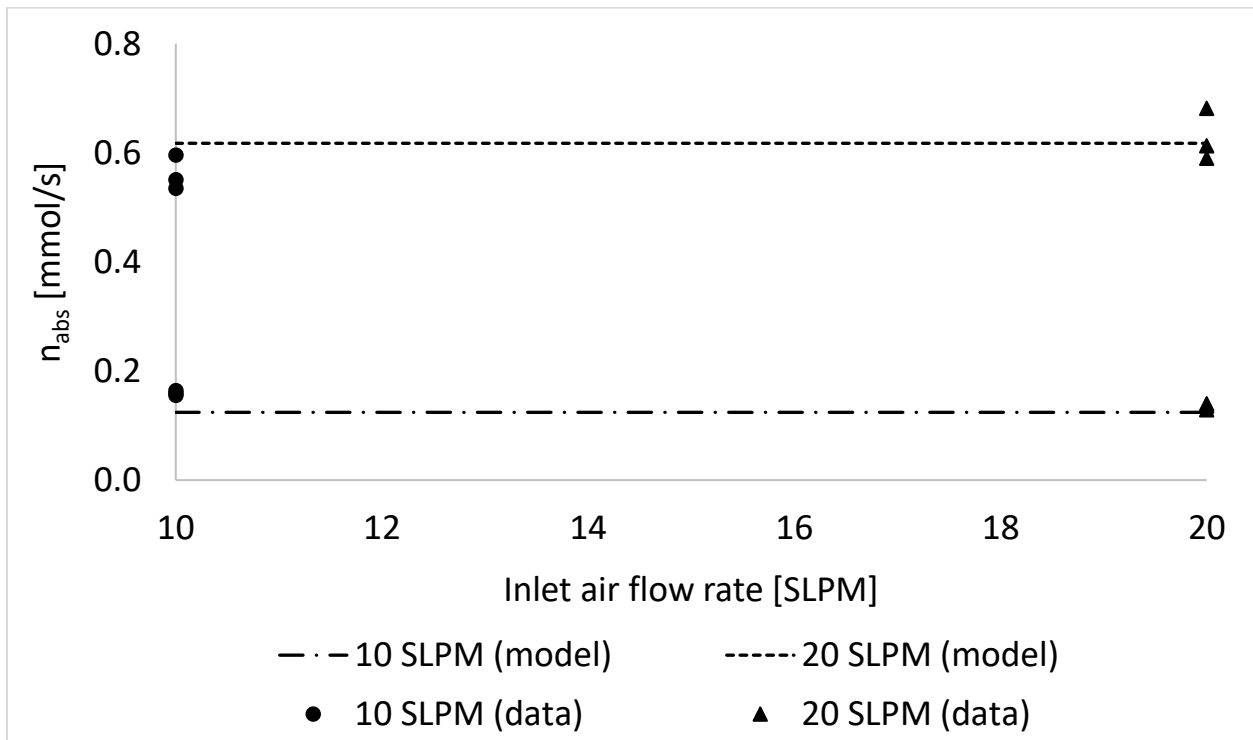
where  $\hat{y}$  = rate of CO<sub>2</sub> absorption ( $n_{\text{abs}}$ ) in mmol/s

$X_1$  = inlet air flow rate (coded)

$X_2$  = inlet water flow rate (coded)

| Parameter             | Standard Error | Prob >  t | Significance    | RSq   |
|-----------------------|----------------|-----------|-----------------|-------|
| Intercept             | 0.0083         | < .0001   | Significant     | 0.989 |
| Inlet air flow rate   | 0.0083         | 0.230     | Not significant |       |
| Inlet water flow rate | 0.0083         | < .0001   | Significant     |       |
| Interaction           | 0.0083         | 0.024     | Significant     |       |

**Table 5.** Standard error and statistical significance of each parameter of the interaction model for  $n_{abs}$ .



**Figure 7.** Effect of inlet air flow rate on  $n_{abs}$ .

The significance of each parameter was determined based on the term “Prob > |t|”, that is, if this term is less than the proposed alpha value of 0.05, the parameter can be determined as statistically significant, but if it is greater than alpha, than the parameter is statistically insignificant. As expected, and as discussed previously, the inlet flow rate of water is statistically significant, as observed in Table 5.

On the other hand, this analysis shows that the inlet flow rate of air is statistically insignificant. This means that since the molecules diffuse quicker through the gas phase, the CO2 diffuses extremely quickly from the has bulk to the interface and then it encounters some

resistance once it enters the liquid. In this case the gas phase boundary layer can be neglected, and we can assume that all the resistance to mass transfer is in the liquid phase boundary layer.

Now, we will analyze the effect of our factors on the overall volumetric liquid-phase mass-transfer coefficient. The equation with the interaction model parameters is:

$$\hat{y} = 0.898 + 0.028X_1 + 0.583X_2 + 0.063X_1X_2$$

where  $\hat{y}$  = overall volumetric liquid-phase mass-transfer coefficient ( $K_L a$ ) in  $\text{kmol/s}\cdot\text{m}^3$

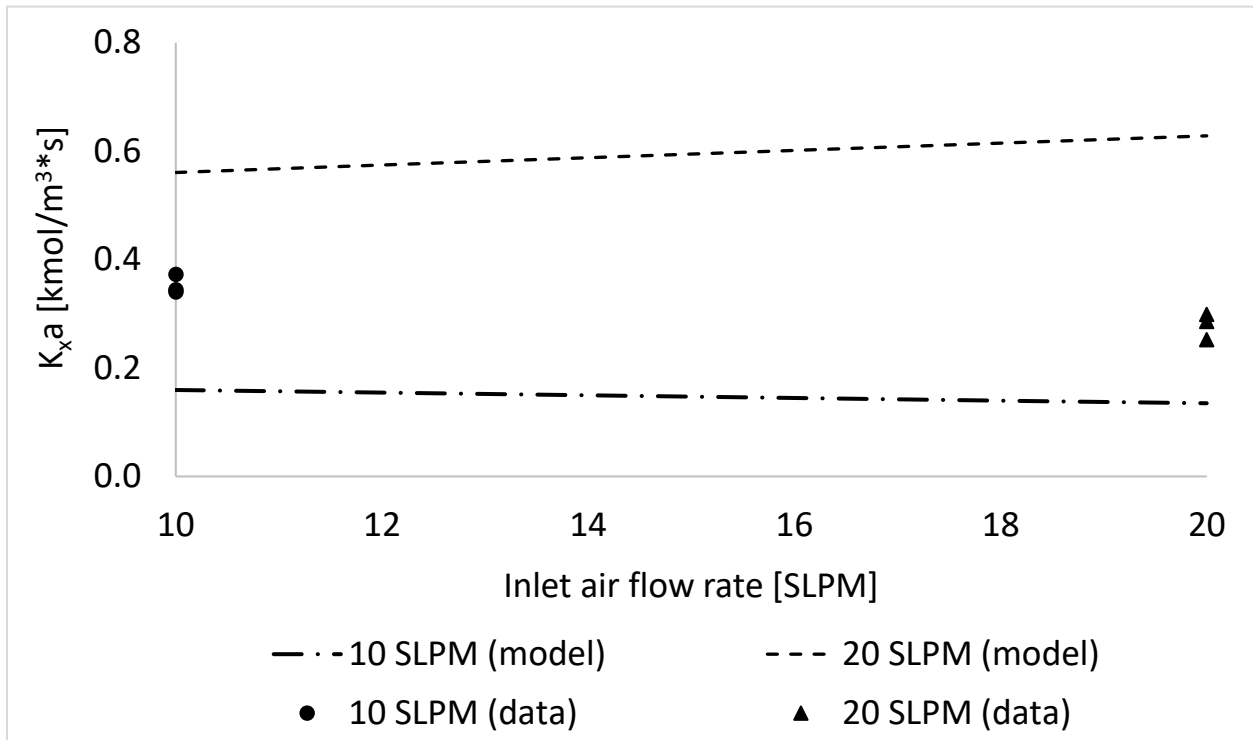
$X_1$  = inlet air flow rate (coded)

$X_2$  = inlet water flow rate (coded)

| Parameter             | Standard Error | Prob >  t | Significance    | RSq  |
|-----------------------|----------------|-----------|-----------------|------|
| Intercept             | 0.0348         | < .0001   | Significant     | 0.97 |
| Inlet air flow rate   | 0.0348         | 0.44      | Not significant |      |
| Inlet water flow rate | 0.0348         | < .0001   | Significant     |      |
| Interaction           | 0.0348         | 0.11      | Not significant |      |

**Table 6.** Standard error and statistical significance of each parameter of the interaction model for  $K_L a$ .



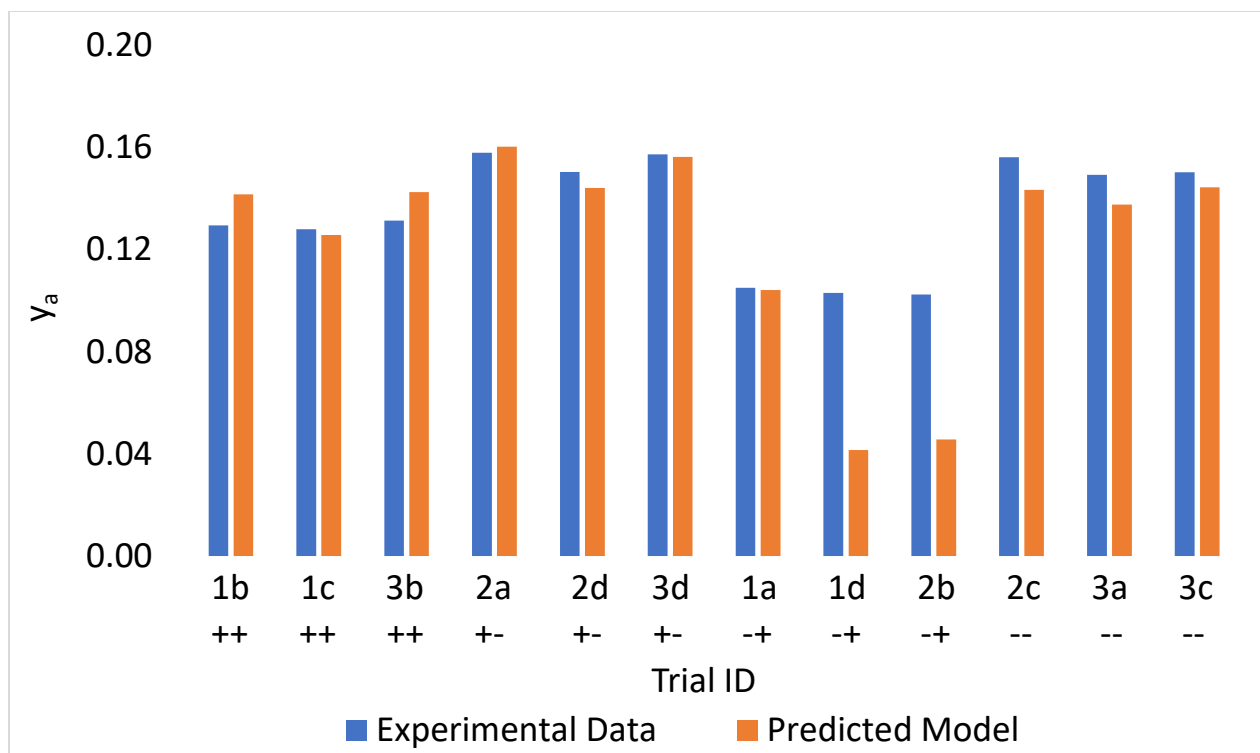


**Figure 8.** Effect of inlet air flow rate on  $K_{xa}$ .

From Table 6 we can conclude that the inlet water flow rate is statistically significant since its  $\text{Prob} > |t|$  is less than the alpha value of 0.05. However, here we can see that the inlet air flow rate is once again statistically insignificant. This is due to the same reasons discussed previously since the values of  $K_{xa}$  are based on those of  $n_{abs}$ .

### **Objective 1e**

To determine how well the experimental data fits the prediction model we needed to create empirical correlations and analyze the results in terms of discrepancy. We examined the discrepancy values of the outlet mole fraction of  $\text{CO}_2$ , the rate of  $\text{CO}_2$  absorption and the overall volumetric mass transfer coefficient in the liquid phase.



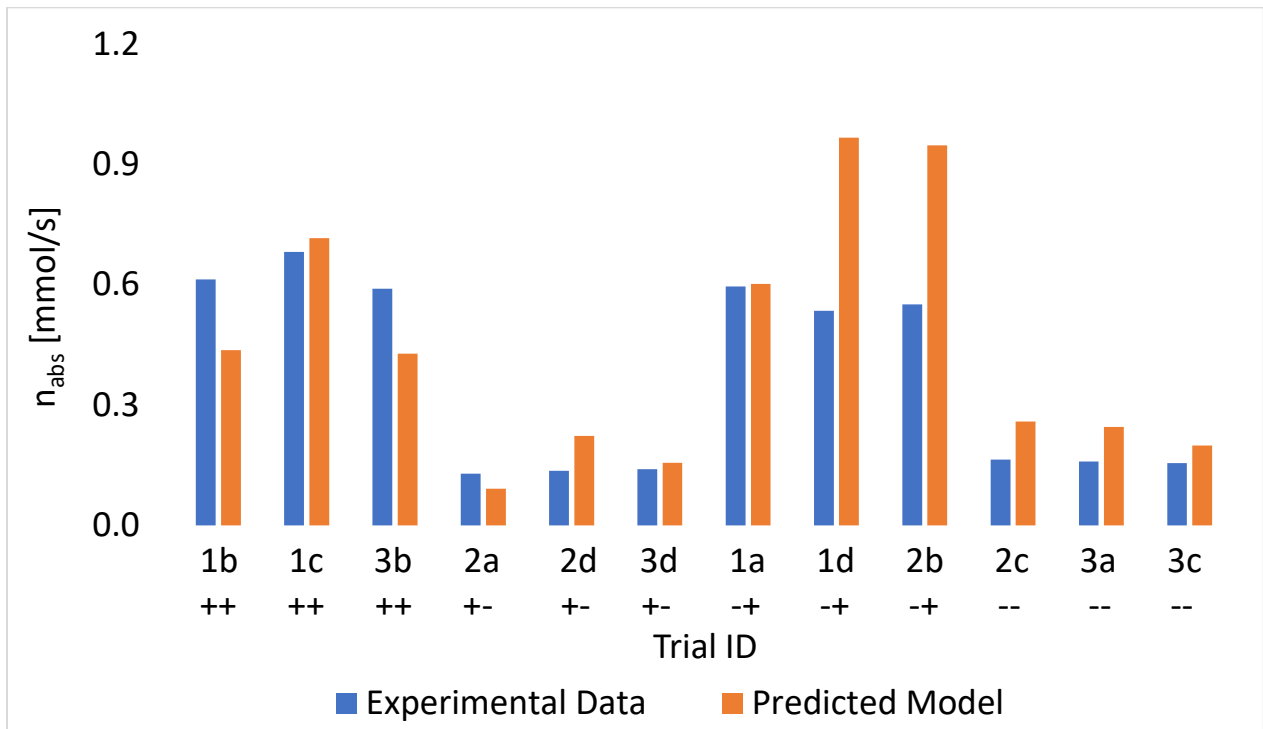
**Figure 9.** Comparison between experimental data and predicted model for the mole fraction of CO<sub>2</sub> in the outlet gas stream.

| Trial Number | Trial ID | Disc[ $y_a$ ] |
|--------------|----------|---------------|
| 1b           | ++       | 0.0122        |
| 1c           | ++       | -0.0024       |
| 3b           | ++       | 0.0112        |
| 2a           | +-       | 0.0025        |
| 2d           | +-       | -0.0063       |
| 3d           | +-       | 0.0011        |
| 1a           | -+       | -0.0009       |
| 1d           | -+       | -0.0615       |
| 2b           | -+       | -0.0567       |
| 2c           | --       | -0.0128       |
| 3a           | --       | -0.0117       |
| 3c           | --       | -0.0059       |

**Table 7.** Discrepancy of  $y_a$ .

In Figure # we can observe that our experimental data for the mole fraction of CO<sub>2</sub> in the outlet gas stream presents relatively close values to our prediction model, except for trials 1d

and 2b where the experimental values for  $y_a$  are much larger than the predicted values. The average experimental value of  $y_a$  is 0.1351 and the predicted average is 0.1239, which gives of an average discrepancy of 0.013. Table # shows the discrepancy for each trial and as we can observe there is a slight discrepancy, but since it is small, we can conclude that our experimental data is a good fit to our prediction, indicating that there is only a small error with our data.



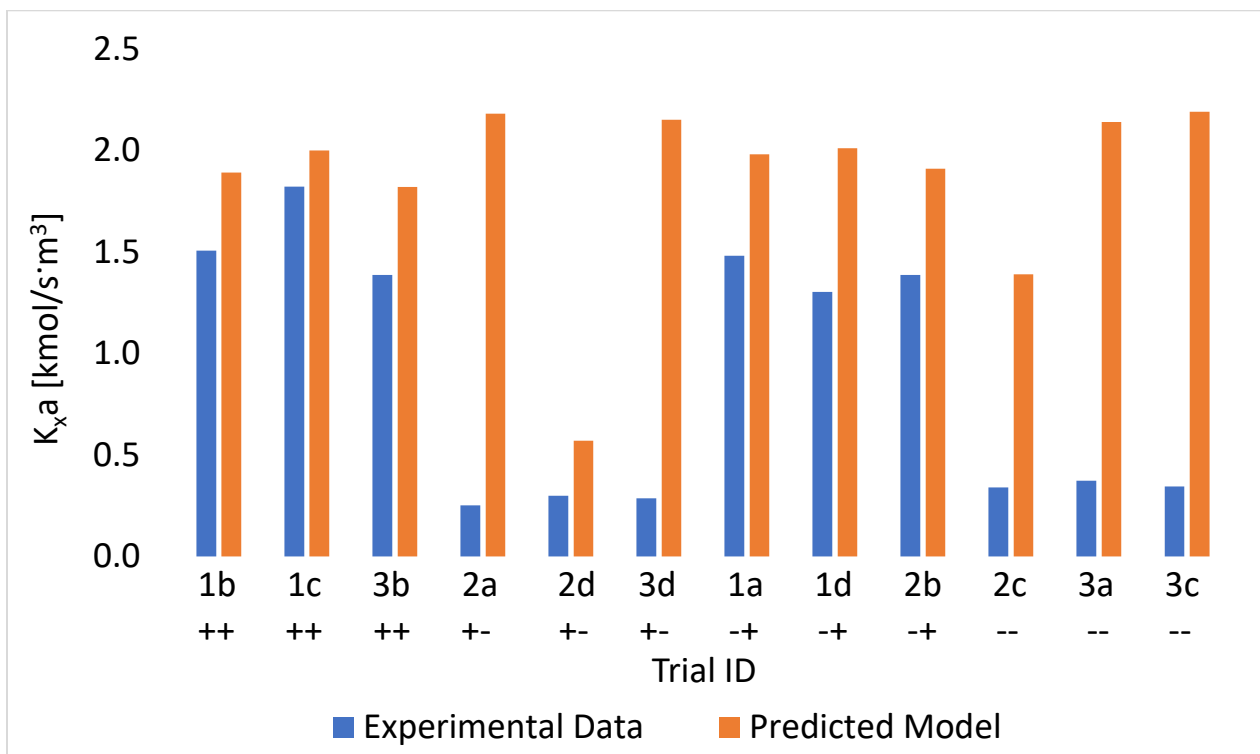
**Figure 10.** Comparison between experimental data and predicted model for the rate of CO<sub>2</sub> absorption.

| Trial Number | Trial ID | Disc[ $n_{abs}$ ] |
|--------------|----------|-------------------|
| 1b           | ++       | -0.176            |
| 1c           | ++       | 0.034             |
| 3b           | ++       | -0.162            |
| 2a           | +-       | -0.037            |
| 2d           | +-       | 0.087             |
| 3d           | +-       | 0.016             |
| 1a           | -+       | 0.006             |
| 1d           | -+       | 0.431             |
| 2b           | -+       | 0.396             |
| 2c           | --       | 0.045             |

|    |    |       |
|----|----|-------|
| 3a | -- | 0.086 |
| 3c | -- | 0.044 |

**Table 8.** Discrepancy of  $n_{abs}$ .

The empirical model for the rate of  $CO_2$  absorption is less accurate for our experimental data than the values for the mole fraction of  $CO_2$ . Similar to the evaluation of  $y_a$ , trials 1d and 2b present the highest deviation of experimental values to prediction values, as observed in Figure 9. The empirical model predictions range from 0.091 to 0.947 mmol/s while the experimental values range from 0.12 to 0.59 mmol/s, which gives us an average discrepancy of 0.068 mmol/s. By analyzing Table # we can see that some of the discrepancy values are significantly higher than expected, which makes out experimental data not be such a good fit for our prediction model.



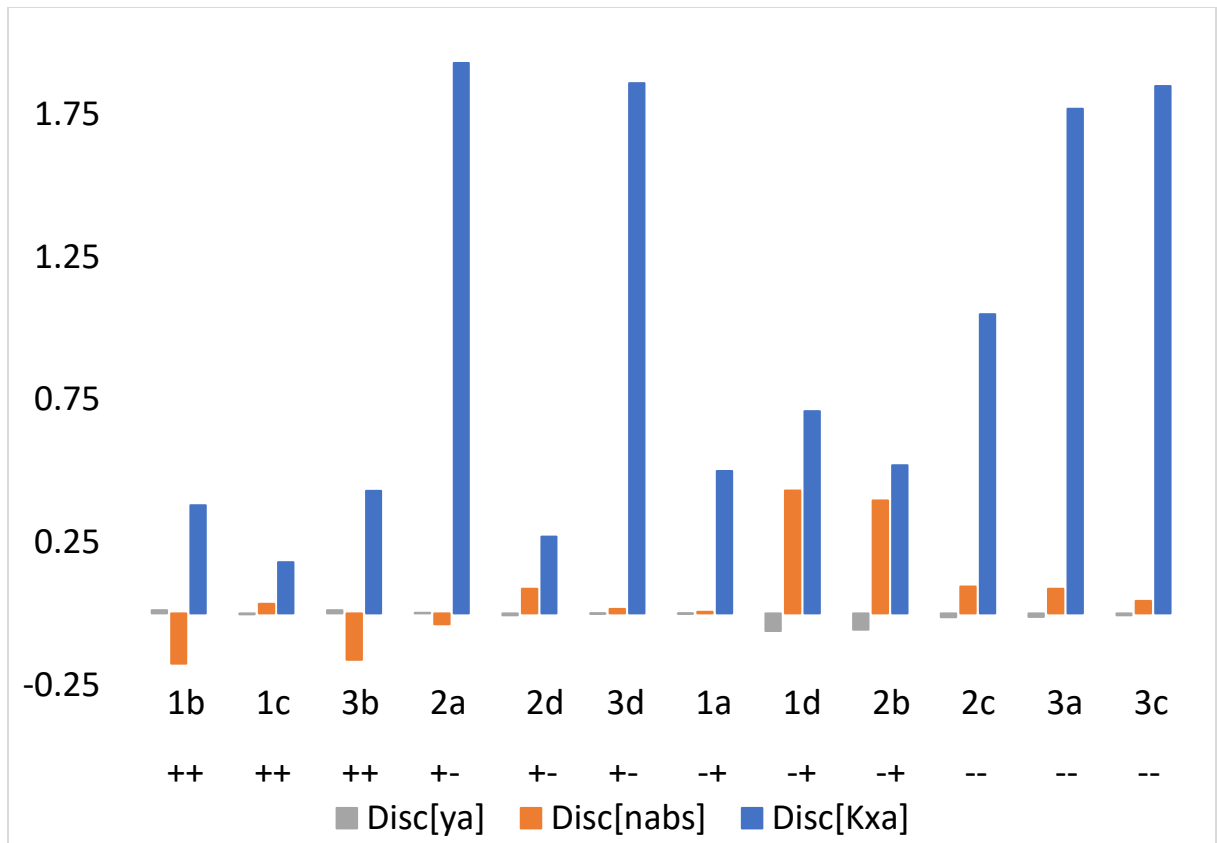
**Figure 11.** Comparison between experimental data and predicted model for the overall volumetric liquid-phase mass-transfer coefficient.

| Trial Number | Trial ID | Disc[ $K_{x,a}$ ] |
|--------------|----------|-------------------|
| 1b           | ++       | 0.38              |
| 1c           | ++       | 0.18              |

|    |    |      |
|----|----|------|
| 3b | ++ | 0.43 |
| 2a | +- | 1.93 |
| 2d | +- | 0.27 |
| 3d | +- | 1.86 |
| 1a | -+ | 0.50 |
| 1d | -+ | 0.71 |
| 2b | -+ | 0.52 |
| 2c | -- | 1.05 |
| 3a | -- | 1.77 |
| 3c | -- | 1.85 |

**Table 9.** Discrepancy of  $K_x a$ .

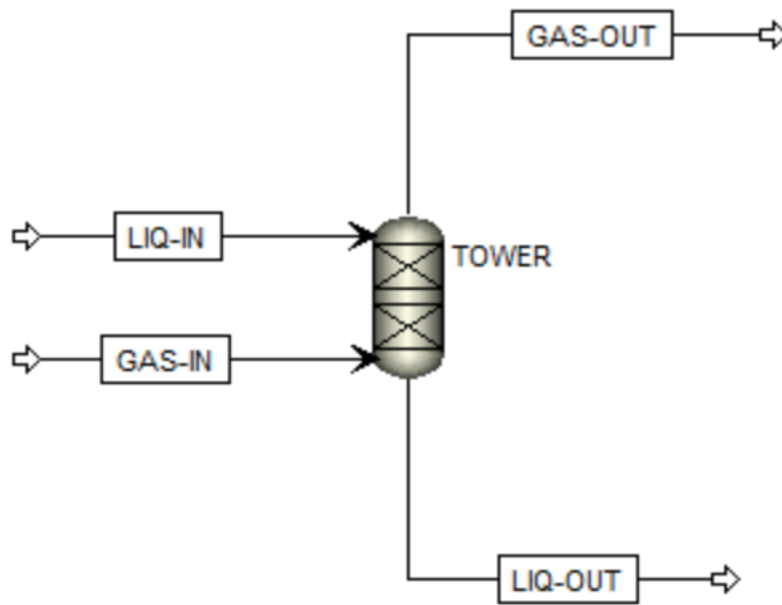
The overall volumetric mass transfer coefficient in the liquid phase is predicted to be much higher for most trials than we determined experimentally, as seen clearly in Figure 10. The average  $K_x a$  value of the empirical model is  $1.85 \text{ kmol/s}\cdot\text{m}^3$ , which is much higher than the experimental average that is only  $0.898 \text{ kmol/s}\cdot\text{m}^3$ . The discrepancy for each trial can be seen in Table # and by analyzing these values we can conclude that our experimental data is not a good fit for our model since we observe relatively large discrepancies.



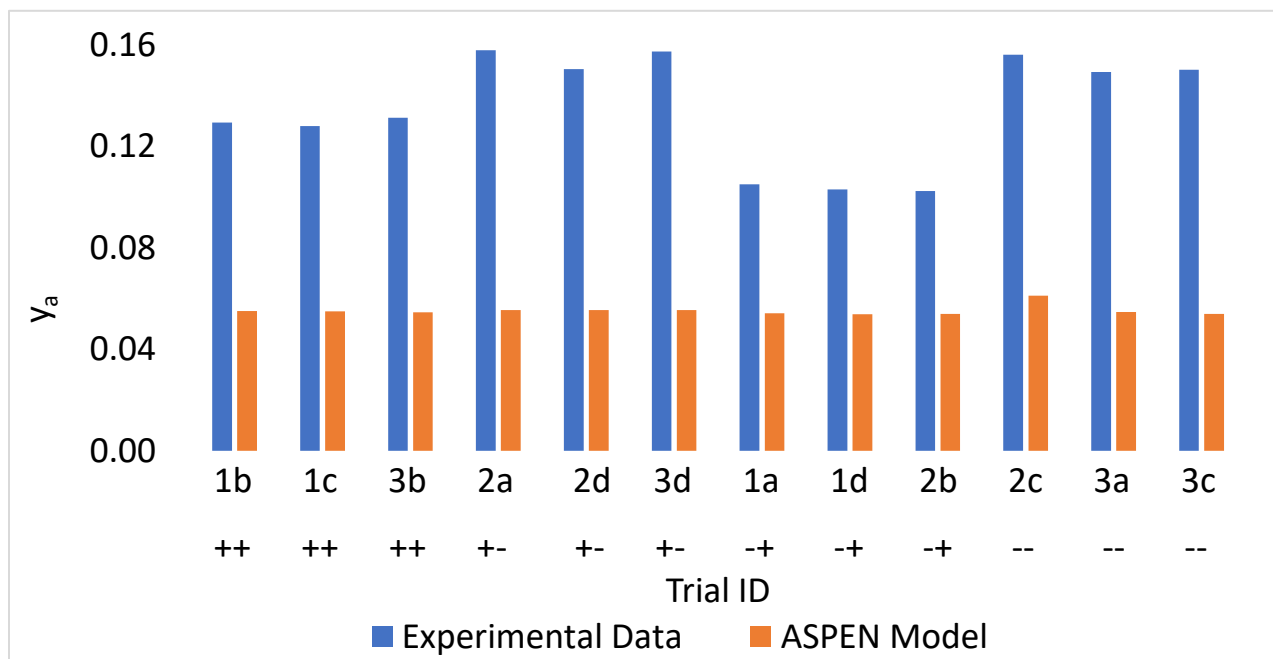
**Figure 12.** Empirical discrepancy values of  $K_{xa}$ ,  $n_{abs}$  and  $y_a$  for each trial.

**Objective 1f**

We also predicted the values for the mole fraction of  $CO_2$  in the outlet gas stream, the rate of  $CO_2$  absorption, and the overall volumetric liquid-phase mass-transfer coefficient by using the ASPEN software. We then compared these results to the experimental data. In order to get ASPEN’s predicted values, we modeled the absorption column used during our experiment as a packed absorber and input the trials conditions into the intel streams for both gas and water. The figure below depicts the ASPEN simulation.



**Figure 13:** ASPEN simulation of absorption column.



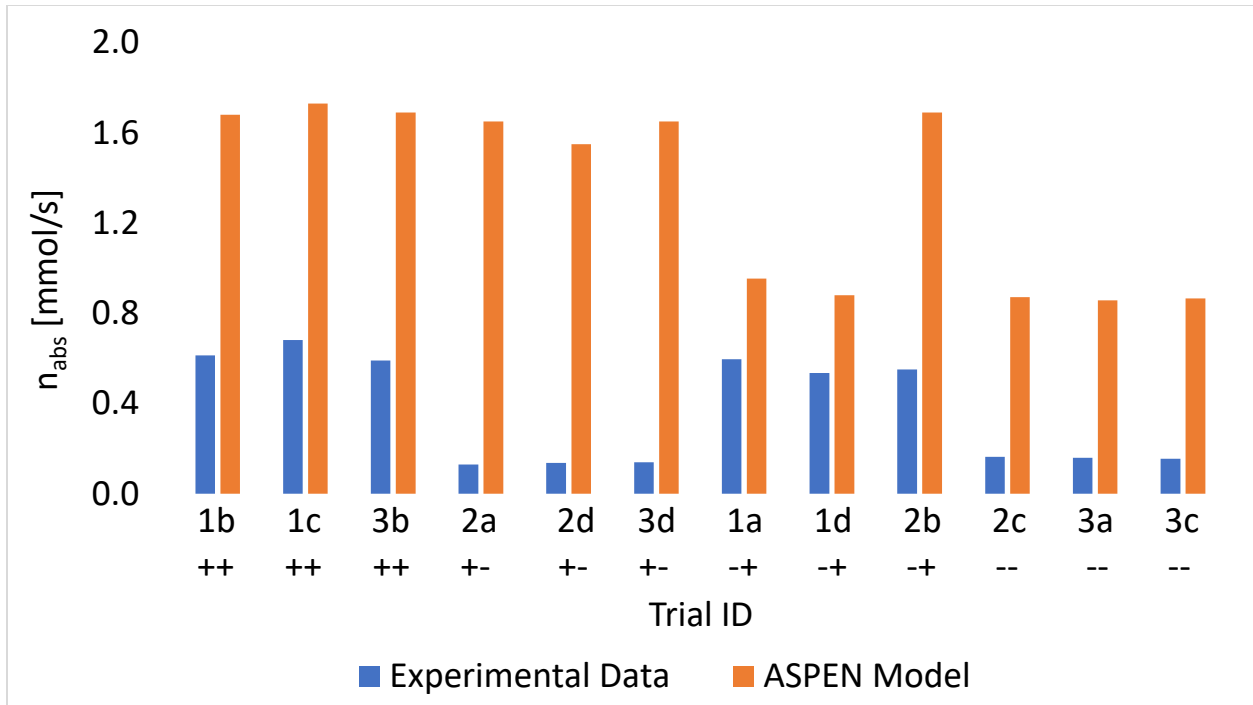
**Figure 14.** Comparison between experimental data and predicted ASPEN model for the mole fraction of CO<sub>2</sub> in the outlet gas stream.

| Trial Number | Trial ID | Disc[ $y_a$ ] |
|--------------|----------|---------------|
| 1b           | ++       | -0.0062       |
| 1c           | ++       | -0.0195       |
| 3b           | ++       | -0.0075       |
| 2a           | +-       | -0.0190       |
| 2d           | +-       | -0.0189       |
| 3d           | +-       | -0.0188       |
| 1a           | -+       | 0.0075        |
| 1d           | -+       | 0.0060        |
| 2b           | -+       | 0.0059        |
| 2c           | --       | -0.0129       |
| 3a           | --       | -0.0159       |
| 3c           | --       | -0.0173       |

**Table 10.** Discrepancy of  $y_a$  based on ASPEN prediction.

As shown in Figure 13, the ASPEN predicted values for the mole fraction of  $\text{CO}_2$  in the outlet gas stream are much lower than our experimental data. This is expected because the ASPEN simulation represents an ideal absorption column where majority of the  $\text{CO}_2$  fed to the column is absorbed into the water resulting in a very low mole fraction of  $\text{CO}_2$  in the outlet gas stream. The average value of  $y_a$  from ASPEN is approximately 0.055 while our experimental average is 0.064 giving us an average discrepancy of 0.012. Table 10 shows the discrepancy between our experimental data and the ASPEN model.





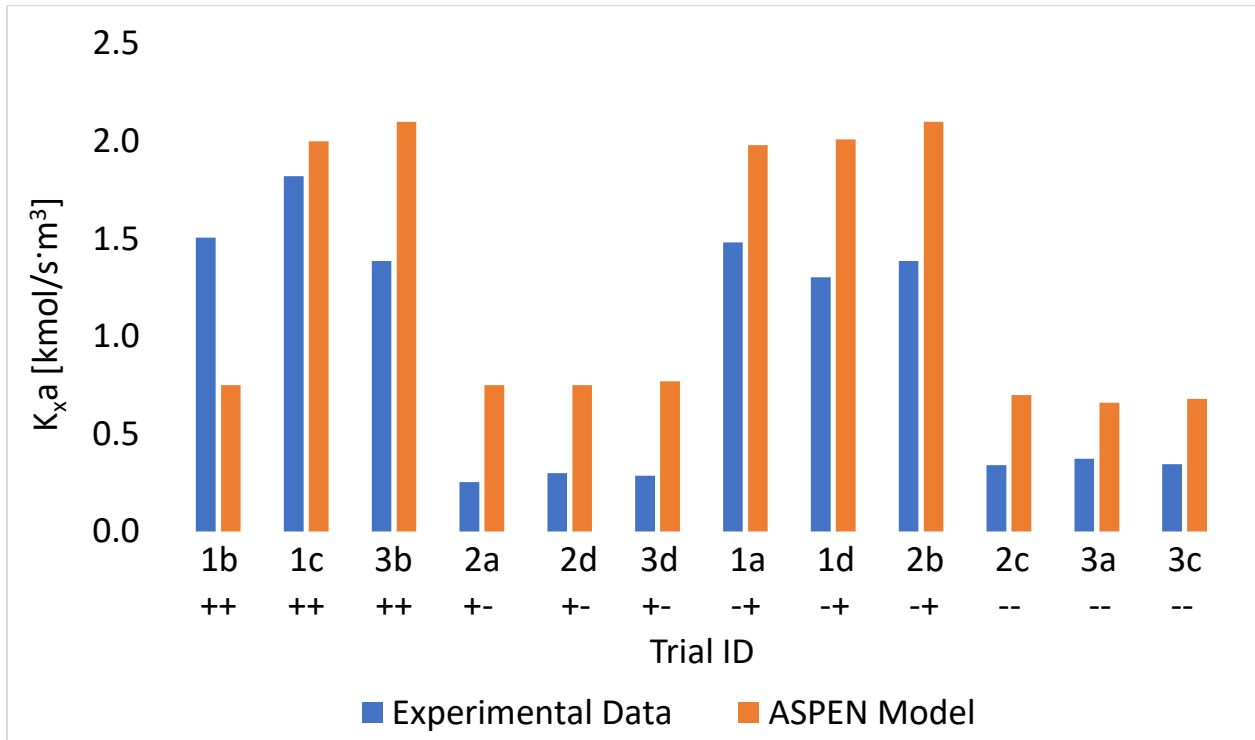
**Figure 15.** Comparison between experimental data and predicted ASPEN model for the rate of CO<sub>2</sub> absorption.

| Trial Number | Trial ID | Disc[n <sub>abs</sub> ] |
|--------------|----------|-------------------------|
| 1b           | ++       | 1.067                   |
| 1c           | ++       | 1.048                   |
| 3b           | ++       | 1.10                    |
| 2a           | +-       | 1.521                   |
| 2d           | +-       | 1.414                   |
| 3d           | +-       | 1.510                   |
| 1a           | -+       | 0.358                   |
| 1d           | -+       | 0.344                   |
| 2b           | -+       | 0.379                   |
| 2c           | --       | 0.707                   |
| 3a           | --       | 0.698                   |
| 3c           | --       | 0.711                   |

**Table 11.** Discrepancy of n<sub>abs</sub> based on ASPEN prediction.

The rate of CO<sub>2</sub> absorption predicted in ASPEN and experimentally determined are compared in Figure 14. As previously mentioned, ASPEN is a model that assumes a much higher rate of absorption than we achieve experimentally. ASPEN predicts an average rate of

approximately 1.27 mmol/s while our experimental average is only 0.371 mmol/s, resulting in an average discrepancy of 0.905. Table 11 shows the discrepancy of  $n_{abs}$  for each trial.



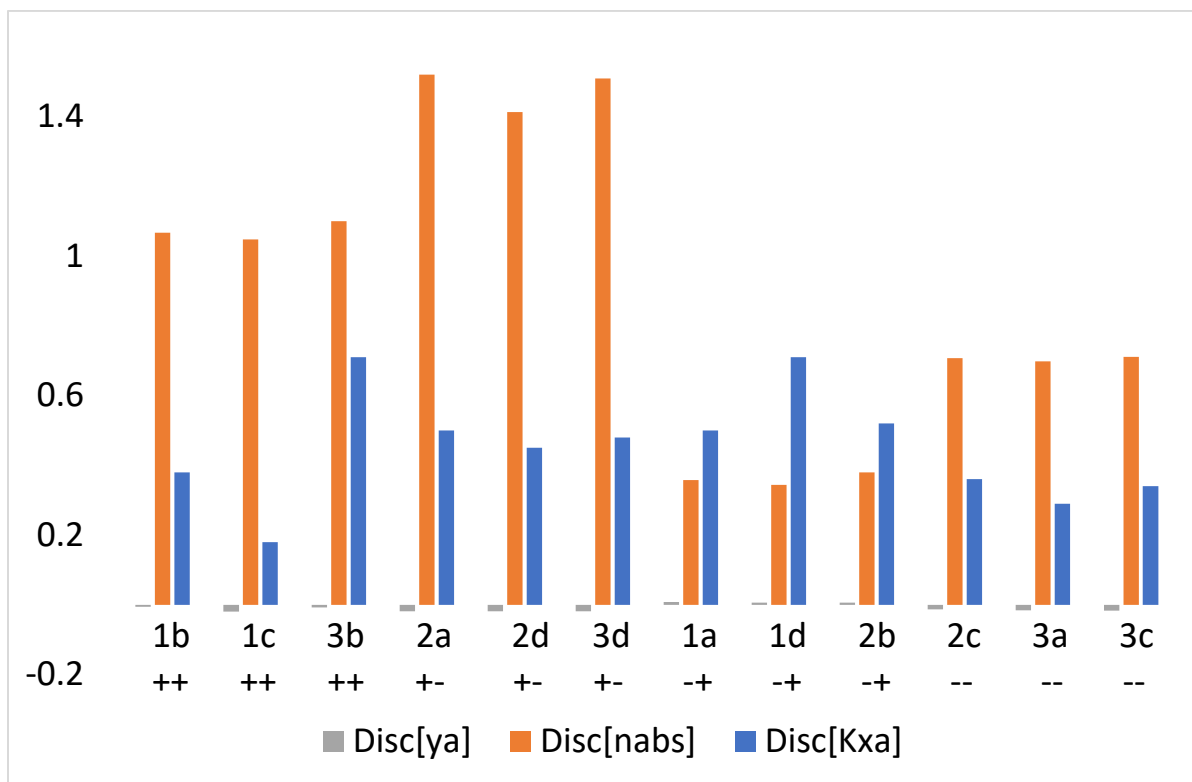
**Figure 16.** Comparison between experimental data and predicted ASPEN model for the overall volumetric liquid-phase mass-transfer coefficient.

| Trial Number | Trial ID | Disc[ $K_{x,a}$ ] |
|--------------|----------|-------------------|
| 1b           | ++       | 0.38              |
| 1c           | ++       | 0.18              |
| 3b           | ++       | 0.71              |
| 2a           | +-       | 0.50              |
| 2d           | +-       | 0.45              |
| 3d           | +-       | 0.48              |
| 1a           | -+       | 0.50              |
| 1d           | -+       | 0.71              |
| 2b           | -+       | 0.52              |
| 2c           | --       | 0.36              |

|    |    |      |
|----|----|------|
| 3a | -- | 0.29 |
| 3c | -- | 0.34 |

**Table 12.** Discrepancy of  $K_{xa}$  based on ASPEN prediction.

The overall volumetric liquid-phase mass-transfer coefficient,  $K_{xa}$  varies by trial which can be seen in Figure #. With the exception of trial 1b, the ASPEN predicted value of  $K_{xa}$  is always higher than the value determined experimentally. The discrepancy values of  $K_{xa}$ , which can be seen in Table #, are the larger than those for both the mole fraction of  $CO_2$  in the outlet gas stream and the rate of  $CO_2$  absorption. The average discrepancy is 0.452 and the overall mass transfer coefficient of the liquid phase ranges from 0.25 to 1.82 experimentally.



**Figure 17:** ASPEN discrepancy values for  $K_{xa}$ ,  $n_{abs}$  and  $y_a$ .

## ODEX

The ODEX is an experiment conducted at the conditions listed in Table 13 and will be used to compare the results to the main trials to evaluate the effect of the liquid feed stream temperature on the rate of  $CO_2$  absorption.

| Primary Factors                                  |                 |          |                     |                     |
|--|-----------------|----------|---------------------|---------------------|
| Variable Name                                    | Variable Symbol | Units    | Target Values       |                     |
| Inlet water mass flow rate                       | $m_{La}$        | kg/min   | (-) level: 2 kg/min | (+) level: 8 kg/min |
| Inlet air volumetric flow rate                   | $v_{SA}$        | SLPM     | (-) level: 10 SLPM  | (+) level: 20 SLPM  |
| Inlet water stream temperature                   | $\Theta_{La}$   | °C       | 30 °C               |                     |
| Secondary Factors                                |                 |          |                     |                     |
| Variable Name                                    | Variable Symbol | Units    | Target Values       |                     |
| Mole fraction of CO <sub>2</sub> in supplied air | $y_{SA}$        | Unitless | 0.08                |                     |
| Inlet CO <sub>2</sub> volumetric flow rate       | $v_{SC}$        | SLPM     | (-) level: 1350 MFC | (+) level: 2544 MFC |

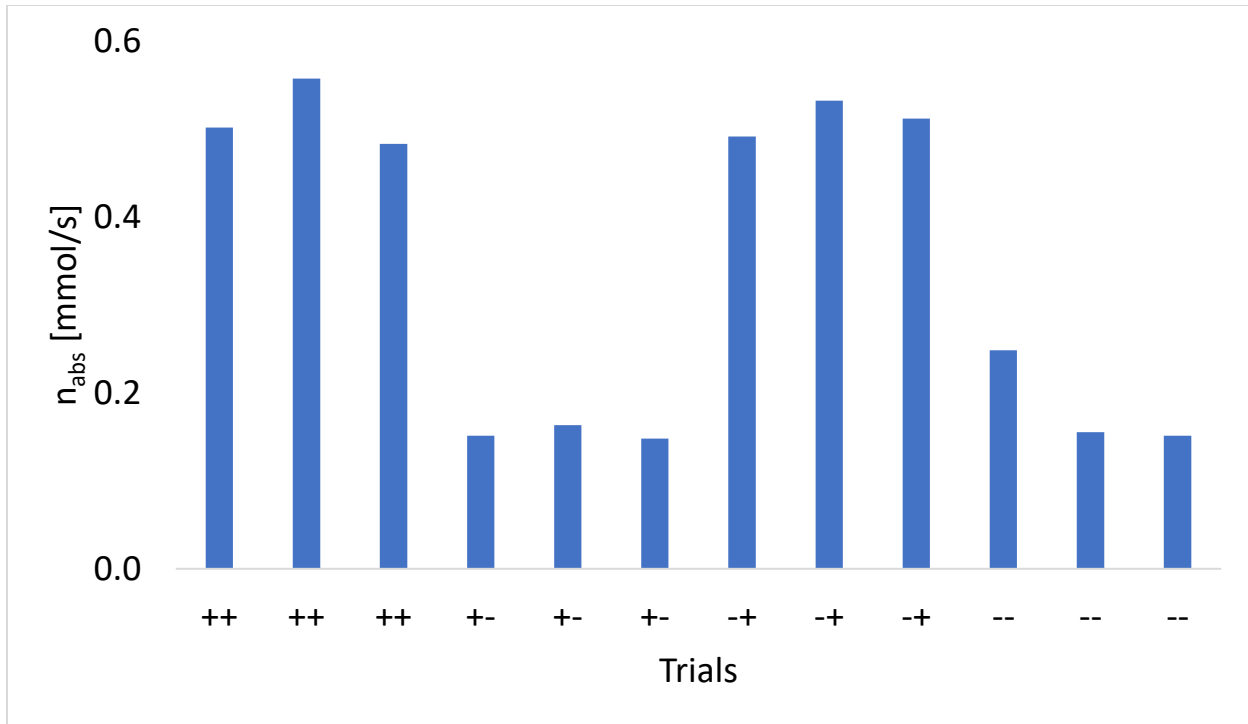
**Table 13.** Factors and target values for the ODEX experiment.

Our results for the ODEX are the following:

| Trial | Trial ID | $n_{abs}$ (mmol/s) | $K_{xa}$ ([kmol/(s·m <sup>3</sup> )]) |
|-------|----------|--------------------|---------------------------------------|
| O1    | - +      | 0.4917             | 1.13                                  |
| O2    | + +      | 0.5017             | 1.05                                  |
| O3    | + -      | 0.1514             | 0.33                                  |
| O4    | --       | 0.2484             | 0.39                                  |
| O5    | - +      | 0.5321             | 1.11                                  |
| O6    | + +      | 0.5574             | 1.10                                  |
| O7    | + -      | 0.1634             | 0.33                                  |
| O8    | --       | 0.1553             | 0.29                                  |
| O9    | - +      | 0.5119             | 1.05                                  |
| O10   | + +      | 0.4830             | 0.99                                  |
| O11   | + -      | 0.1482             | 0.32                                  |
| O12   | --       | 0.1514             | 0.29                                  |

**Table 14.** Trials and responses for ODEX.

It can be better observed in the following graph:



**Figure 18.** Rate of CO<sub>2</sub> absorption for all ODEX trials. The first and second factors represent the inlet air flow rate and the inlet water flow rate, respectively.

After analyzing our ODEX results, we obtained the following:

$$\hat{y} = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 + b_{123}X_1X_2X_3$$

where  $\hat{y}$  = rate of CO<sub>2</sub> absorption (n<sub>abs</sub>) in mmol/s

$X_1$  = Inlet water temperature (coded)

$X_2$  = inlet water flow rate (coded)

$X_3$  = inlet air flow rate (coded)

| Parameter       | Standard Error | Prob > t | Significance    | R square |
|-----------------|----------------|----------|-----------------|----------|
| Intercept       | 0.006573       | < .0001  | Significant     | 0.98     |
| Temperature     | 0.006573       | 0.0393   | Not significant |          |
| Water Flow Rate | 0.006573       | < .0001  | Significant     |          |
| Air Flow Rate   | 0.006573       | 0.7849   | Not significant |          |

|  |          |        |                 |
|--|----------|--------|-----------------|
| Temperature*Water flow rate                | 0.006573 | 0.0011 | Significant     |
| Temperature*Air flow rate                  | 0.006573 | 0.1914 | Not significant |
| Water flow rate*Air flow rate              | 0.006573 | 0.0303 | Not significant |
| Temperature* Water flow rate*Air flow rate | 0.006573 | 0.2764 | Not significant |

**Table 15.** Standard error and statistical significance of each parameter of the interaction model for the rate of CO<sub>2</sub> absorption.

Our initial hypothesis supported that temperature would affect the absorption of carbon dioxide. However, our JMP results showed that temperature was not significant. Performing the experiment at a higher temperature providing a greater gap from our main trials temperature might be able to give us a more accurate result. Also, since our R square for the JMP Model was 0.98, it means that our data fits well the model.

Also, we suspected that O1 and O2 data points were outliers due to operational blunders and that they were interfering with our data analysis, so we proceeded to do a Grubb's test with an alpha of 0.05 and obtain the following results:

|                    | Trial O1 (- +) | Trial O2 (+ +) |
|--------------------|----------------|----------------|
| Average            | 13.1           | 9.4            |
| Standard deviation | 0.2            | 0.2            |
| G                  | -1.134         | -1.126         |

**Table 16.** Grubb's test results for ODEX.

With a G critical of 1.15, none of the G values were greater than G critical, therefore, we were not able to neglect our data points that are deviating significantly from the average.

We also determined the repeatability of ODEX trials by relating the grand average to the pooled standard deviation, as seen in Table # below.

|                           | $n_{abs}$ [mmol/s] | $K_{xa}$ [kmol/m <sup>3</sup> ·s] |
|---------------------------|--------------------|-----------------------------------|
| Grand average             | 0.341              | 0.698                             |
| Pooled standard deviation | 0.035              | 0.045                             |

|                            |       |       |
|----------------------------|-------|-------|
| Coefficient of variability | 0.103 | 0.064 |
|----------------------------|-------|-------|

**Table 17.** Repeatability of  $n_{abs}$  and  $K_{xa}$  for ODEX trials.

## SODEX

The SODEX is a new experiment where the same equipment and materials of the main experiment are used, but it is conducted at the conditions listed in Table 15. This experiment will be used to analyze the effect of the inlet percentage of CO<sub>2</sub> and will be compared to the main experiment trials.

| Primary Factors                                  |                 |          |                     |                     |
|--|-----------------|----------|---------------------|---------------------|
| Variable Name                                    | Variable Symbol | Units    | Target Values       |                     |
| Inlet water mass flow rate                       | $m_{La}$        | kg/min   | (-) level: 2 kg/min | (+) level: 8 kg/min |
| Inlet air volumetric flow rate                   | $v_{SA}$        | SLPM     | (-) level: 10 SLPM  | (+) level: 20 SLPM  |
| Secondary Factors                                |                 |          |                     |                     |
| Variable Name                                    | Variable Symbol | Units    | Target Values       |                     |
| Mole fraction of CO <sub>2</sub> in supplied air | $y_{SA}$        | Unitless | 0.15                |                     |
| Temperature of water at top of packing           | $\Theta_{La}$   | °C       | 25 °C               |                     |
| Inlet CO <sub>2</sub> volumetric flow rate       | $v_{SC}$        | SLPM     | (-) level: 1350 MFC | (+) level: 2544 MFC |

**Table 18.** Factors and target values for the SODEX experiment.

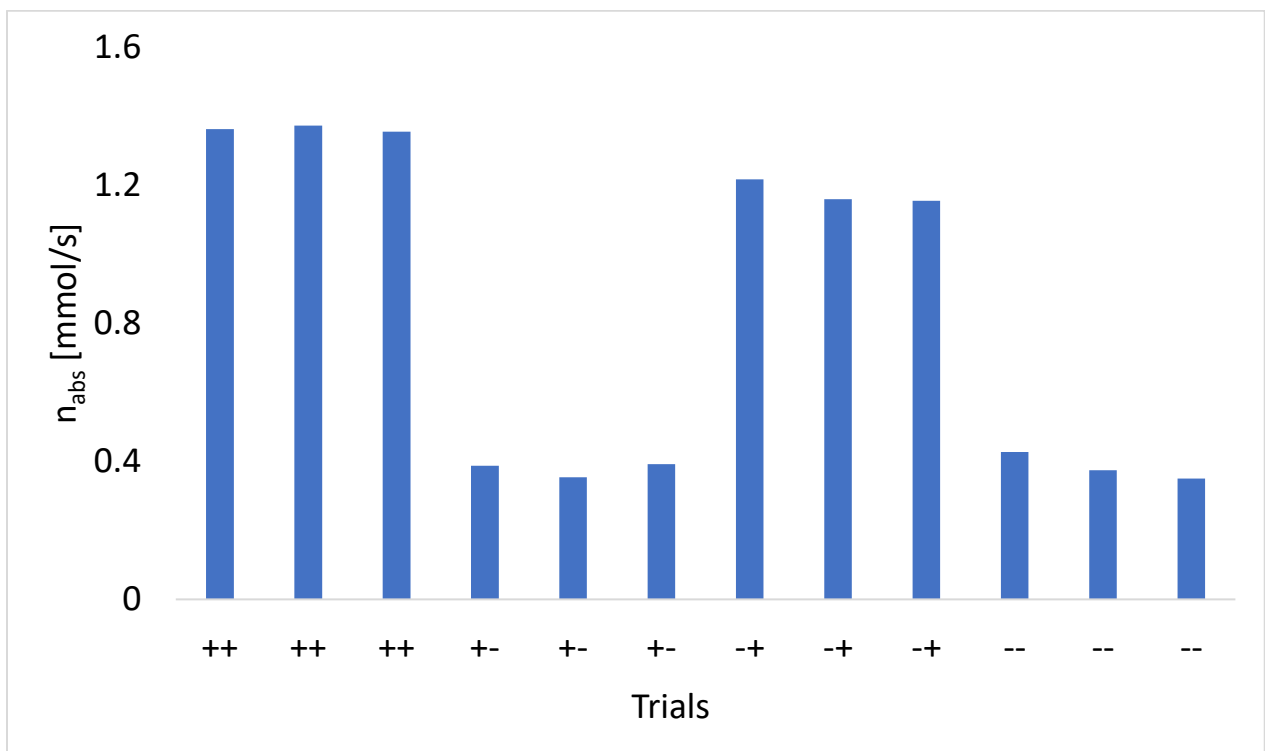
We obtained the following results:

| Trial | Trial ID | $n_{abs}$<br>(mmol/s) | $K_{xa}$<br>([kmol/(s·m <sup>3</sup> )]) |
|-------|----------|-----------------------|--|
| S3    | ++       | 1.3615                | 2.11                                     |
| S7    | ++       | 1.3716                | 2.15                                     |
| S11   | ++       | 1.3541                | 2.08                                     |
| S4    | +-       | 0.387                 | 0.7                                      |
| S8    | +-       | 0.3538                | 0.53                                     |

|     |    |        |      |
|-----|----|--------|------|
| S12 | +- | 0.3915 | 0.74 |
| S2  | -+ | 1.2165 | 1.84 |
| S6  | -+ | 1.1593 | 1.66 |
| S10 | -+ | 1.154  | 1.65 |
| S1  | -- | 0.4269 | 1.15 |
| S5  | -- | 0.374  | 0.63 |
| S9  | -- | 0.3495 | 0.51 |

**Table 19.** Overall results of Gas Composition for the SODEX experiment

In the following graph, we can appreciate better the results:



**Figure 19.** Rate of CO<sub>2</sub> absorption for all SODEX trials. The first and second factors represent the inlet air flow rate and the inlet water flow rate, respectively.

After analyzing our SODEX results, we obtained the following:

$$\hat{y} = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 + b_{123}X_1X_2X_3$$

where  $\hat{y}$  = rate of CO<sub>2</sub> absorption (n<sub>abs</sub>) in mmol/s

$X_1$  = Inlet air flow rate(coded)



$X_2$  = inlet water flow rate (coded)

$X_3$  = inlet carbon dioxide percentage (coded)

| Parameter  | Standard Error | Prob > t | Significance    | R square |
|--|----------------|----------|-----------------|----------|
| Intercept  | 0.005853       | < .0001  | Significant     | 0.99     |
| Air flow rate                                      | 0.005853       | 0.0002   | Not significant |          |
| Water Flow Rate                                    | 0.005853       | < .0001  | Significant     |          |
| CO2 inlet percentage                               | 0.005853       | < .0001  | Significant     |          |
| Water flow rate*Air flow rate                      | 0.005853       | < .0001  | Significant     |          |
| CO2 inlet percentage*Air flow rate                 | 0.005853       | 0.0101   | Not significant |          |
| Water flow rate*CO2 inlet percentage               | 0.005853       | < .0001  | Significant     |          |
| CO2 inlet percentage*Water flow rate*Air flow rate | 0.005853       | 0.0491   | Not significant |          |

**Table 20.** Standard error and statistical significance of each parameter of the interaction model for the rate of CO<sub>2</sub> absorption.

Our JMP result indicated that the inlet percentage of carbon dioxide was significant with an estimate of 0.22, meaning that in magnitude it has a strong impact on the response compared to the other factors and that it is directly proportional to the response, as we have a positive estimate. Our non-significant parameters were inlet percentage of carbon dioxide times the air flow rate, and the combination of all factors. As we doubled the percentage of carbon dioxide compared to our main trials, the difference was planned to be large enough to see a difference and our JMP result confirmed the impact on the response. Also, as our R square value was 0.99, the model is a good fit for the data.

To determine the repeatability of our SODEX, we obtained a CV value with the following values:

|                            | $n_{abs}$ [mmol/s] | $K_{xa}$ [kmol/m <sup>3</sup> ·s] |
|----------------------------|--------------------|-----------------------------------|
| Grand average              | 0.825              | 1.313                             |
| Pooled standard deviation  | 0.029              | 0.188                             |
| Coefficient of variability | 0.035              | 0.143                             |

**Table 21.** Repeatability of  $n_{abs}$  and  $K_{xa}$  for SODEX trials

The rate of absorption for carbon dioxide in our SODEX had a good repeatability since the coefficient of variability is less than 0.5, however, the overall volumetric liquid-phase mass-transfer coefficient did not show good repeatability, having a coefficient of variability higher than our alpha value.

## Conclusions

Our ODEX analysis demonstrated that temperature has no effect on rate of absorption, having a R square of 0.98, showing that the model fits well our data. Even though we suspected the analysis was failing due to outliers, after performing a Grubb's test on the doubtful data points, the test showed that all values were significant on the experiment.

With our SODEX, our objective was to determine if the effect of carbon dioxide in the inlet was significant or not. After analyzing the data obtained in the laboratory, we concluded that the effect of carbon dioxide in the inlet will affect the rate of absorption in a directly proportional way, having an estimate of 0.22. Our R square value for the JMP model was 0.99, confirming that the model fits well the data.

## Recommendations

To future teams that will perform Experiment 380, we strongly recommend taking into account the outdoor weather the same day of their experiment and prepare to accommodate the domestic water temperature to the desired 25 degrees Celsius. Arriving to the laboratory and not prioritizing how to achieve the desired water temperature can cost time that could have been

invested in performing trials. We also would like to add the importance of constantly checking the gel filter and be proactively changing the filter if needed.

## Acknowledgments

We want to thank our instructor, Dr. Hunter for always being readily available to answer any questions or concerns we had throughout the performance of the experiment. We would also like to thank our teaching assistant, Patrick Blatt, and the lab engineer, Mr. Crisler, for preparing us to conduct this experiment, as well as helping us with some issues we encountered when performing the experiment. Additionally, we would like to acknowledge team 22 for their cross-training help.

## Literature Cited

Center for Chemical Process Safety. *Guidelines for Writing Effective Operating and Maintenance Procedures*. New York: American Institute of Chemical Engineers, 1996.

McCabe, Warren L., Julian C. Smith, and Peter Harriott. *Unit Operations of Chemical Engineering*. 7th ed. New York, NY: McGraw-Hill, 2005.

The National Institute for Occupational Safety and Health. *NIOSH Pocket Guide to Chemical Hazards*, U.S. Department of Health and Human Services Publication No. 2005-149, September, 2007.<sup>1</sup>

*Available at the course site in Blackboard*

Materials© > Literature Resources

-- Koch, Jr., Howard A., Leroy F. Stutzman, Harold A. Blum, and LeROI E. Hutchings. "Liquid

Transfer Coefficients for the Carbon Dioxide – Air – Water System." *Chemical Engineering*

*Progress* 45 (11), 677-682, 1949.

-- This article presents experimental results for  $KLa$  for an absorption system similar to that in

this experiment. Enterprising students will convert these  $KLa$  values to  $Kxa$  values and compare them to their results for this experiment.

Lab Library > Data Analysis Examples

-- Expt 380 – Aspen Plus® Tutorial

A step-by-step tutorial, loaded with screen shots, walks the novice user through setting up Aspen Plus® to simulate this experiment.

-- Expt 380 – Mass-Transfer Coefficient – Framework

An Excel spreadsheet lays out the sequence of calculations for carbon dioxide absorbed into water to obtain the value of the overall volumetric liquid-phase mass-transfer coefficient.<sup>13</sup>

-- Expt 380 – Mass-Transfer Coefficient – Example

Printout of example calculation using the Excel spreadsheet.

Lab Library > Lab Procedures

-- Expt 380 Equipment Specifications

Print out this detailed list of equipment specifications and include it in the operating procedure. The file is available as a Word document to enable convenient updating. Review this list when reconnoitering.

## Appendix

### Copies of Incident/Accident reports

No accidents or incidents occurred during our experiment.

#### Prior accidents/incidents

The safety officer must check for recent accident/incident reports associated with this experiment.

These reports are located in the Unit Operations Laboratory in a loose-leaf binder near the SDS collection. A thin binder near the apparatus contains pertinent SDSs and accident/incident reports specifically for the extraction experiment.

The safety officer will update the written procedure to include these recent accident/incidents in the list below. The team will incorporate appropriate measures into the operating procedure to prevent such accidents/incidents.

#### September 24, 2003

- Accident: The valves for the inlet and outlet concentrations of CO<sub>2</sub> were set up incorrectly. This led to misreading in the CO<sub>2</sub> analyzer.

- Prevention: The correct way to set the valves for taking the measurements was shown to the two operators by the lab assistant. This was done on the same day as the incident, September 24, 2003.

#### September 26, 2003

- Accident: When the experiment began, the inlet valve for CO<sub>2</sub> was noticed to have bubbles in the tube. It was observed that the water in the manometer had been blown out into the CO<sub>2</sub> tubes for the inlet and outlet. The event could have been caused by the air and CO<sub>2</sub> rotameters being fully open during startup thus leading to a large change in pressure that forced the water out of the manometer.
- Prevention: Check the rotameter settings prior to startup as well as the water manometer. This was shown on 9/29/2003 by the lab assistant.

#### September 24, 2004

- Accident: After opening the air into the column and beginning to open the water valve, the column flooded. This flooding went into the CO<sub>2</sub> sensor lines filling the water trap and moving into the CO<sub>2</sub> sensor
- Prevention: Adding an immediate drain valve so that water drain trap can be drained promptly in case flooding occurs. Also by setting the rotameters to set values of shutdown so that at startup flooding does not occur.

#### September 29, 2004

- Accident: The liquid flow rate was 4 L/min, the gas flow rate was 20 L/min, soda ash solution were recycled in the column. And the operator and team leader tried to shut off the column following the same procedure used before in day Sept. 24 and 27, but the column flooded.
- Prevention: Set the volumetric flow rate to low before shutting it off.

#### October 25, 2006

- Accident: Air flow rate was too high and the absorption column flooded. Water entered the manometer tube, but the CO<sub>2</sub> valves were closed so no equipment was damaged.
- Prevention: When adjusting air flow rate for cooling tower objective, the water flow rate needs to be adjusted as well. As the air flow rate increases, watch carefully the column and the manometer to prevent flooding.

#### September 9, 2009

- Accident: During shutdown, the CO<sub>2</sub> analyzer was still running and foam built up at the bottom of the column. Foam started to flow up into the water trap and almost into the analyzer. As this happened, the valve leading to the analyzer was shut.

- Prevention: Making sure to shut down in a controlled manner, after flushing the column with fresh water following soda ash trials. Turn off analyzer pump, then shut both valves leading to the analyzer. Flush the column with fresh water, then shut off air.

#### September 14, 2009

- Accident: Had both blue valves (CO2 analyzer) closed while analyzer pump was on.
- Prevention: Study the safety section of the procedure more accurately

#### October 12, 2009

- Accident: Mass flow controllers were turned on but CO2 tank was closed. CO2 controller was heating up but was not damaged
- Prevention: During startup, verify that gauge reads correctly when pump is open to confirm. Operator should double check during startup. Follow your procedure.

#### September 27, 2010

- Accident: Turned on analyzer for CO2 without valve open for flow through system while the pump was turned on. The valve to or from the CO2 sensor must be opened while there is flow through the system.
- Prevention: Make sure to have at least one valve open when taking measurements from CO2 analyzer when pump is operating.

#### October 24, 2011

- Accident: Our team was unaware of the drain valve on the base of the tank and the valve was slightly open. This caused for some of the soda ash to drain on to the floor.
- Prevention: Make sure each and every team/team member knows of this valve and to check it

#### September 7, 2012

- Accident: The gas inlet valve to the bottom of the column was closed, causing the calibration line to disconnect from the CO2 analyzer due to excessive pressure in the line.
- Prevention: The team made sure to note in the procedure to not close this valve.

#### September 23, 2013

- Accident: The valve that drains the tank was open during mixing of soda ash solution for the ODEX trials. The lab engineer neutralized the remaining 0.05 mol/L solution and drained the tank.
- Prevention: All operators must confirm the proper valve configurations for the startup of the ODEX trials.

#### September 19, 2014

- Accident: Water entered the lower level of the absorption column, past the air inlet line.
- Prevention: Do not start this valve while pumping. Start with the valve closed.

## Management Task List

|            | Description | Team Role      | Student Name   |
|------------|-------------|----------------|----------------|
| Expt. No.  | 380         | Team Leader    | Wendy Juzwiak  |
| Expt. Name | Absorption  | Safety Officer | Luciana Castro |
| Team No.   | 23          | Operator       | Akilah Sanders |

### Deadlines (from Report Schedule in Lab Manual)

|                              |             |
|------------------------------|-------------|
| Draft Procedure              | October 20  |
| PAQ                          | October 21  |
| ORC                          | October 27  |
| Lab Period                   | November 1  |
| Lab Period                   | November 3  |
| Lab Period                   | November 5  |
| Draft Theory and Intro       | November 8  |
| Draft Report Results         | November 9  |
| WTR Draft to Peer Reviewer's | November 12 |
| Written Technical Report     | November 15 |

### Detailed Task List

| Target Date | Target Day | Target Time | Team Leader | Safety Officer | Operator | Task Description |
|-------------|------------|-------------|-------------|----------------|----------|------------------|
| Oct 12      | Tuesday    | 4:00 PM     | X           | X              | X        | Work on OP       |

|        |           |          |   |   |   |   |
|--------|-----------|----------|---|---|---|---|
| Oct 13 | Wednesday | 1:30 PM  | X | X | X | Work on OP; start PAQ                                     |
| Oct 14 | Thursday  | 4:00 PM  | X | X | X | Work on PAQ   |
| Oct 15 | Friday    | 1:30 PM  | X | X | X | Reconnoiter in lab; work on equipment sketch; work on PAQ |
| Oct 18 | Monday    | 1:30 PM  | X | X | X | Reconnoiter in lab; work on OP; work on PAQ               |
| Oct 19 | Tuesday   | 4:00 PM  | X | X | X | Finalize OP; work on PAQ                                  |
| Oct 20 | Wednesday | 12:00 PM | X |   |   | <b>Submit OP</b>  |
| Oct 20 | Wednesday | 2:00 PM  | X | X | X | Finalize on PAQ   |
| Oct 21 | Thursday  | 12:00 PM | X |   |   | <b>Submit PAQ</b>   |
| Oct 22 | Friday    | 1:30 PM  | X | X | X | Reconnoiter in lab; prepare for ORC                       |
| Oct 25 | Monday    | 1:30 PM  | X | X | X | Reconnoiter in lab; review for ORC                        |
| Oct 27 | Wednesday | 12:55 PM | X | X | X | <b>ORC</b>  |
| Oct 28 | Thursday  | 4:00 PM  | X | X | X | Prepare data sheets for lab day; start draft theory       |
| Nov 1  | Monday    | 2:00 PM  | X | X | X | Lab period; work draft theory                             |
| Nov 2  | Tuesday   | 4:00 PM  | X | X | X | Review data collected in lab; start draft results         |



|        |               |             |   |   |   |  |
|--------|---------------|-------------|---|---|---|--|
| Nov 3  | Wednesd<br>ay | 2:00 PM     | X | X | X | Lab period; work on draft theory                       |
| Nov 4  | Thursday      | 4:00 PM     | X | X | X | Review data collected in lab; work on draft results    |
| Nov 5  | Friday        | 2:00 PM     | X | X | X | Lab period; refine draft theory; work on draft results |
| Nov 6  | Saturday      | 3:00 PM     | X | X | X | Refine draft theory and draft results                  |
| Nov 8  | Monday        | 12:00<br>PM | X |   |   | <b>Submit draft theory</b>                             |
| Nov 9  | Tuesday       | 12:00<br>PM | X |   |   | <b>Submit draft results</b>                            |
| Nov 10 | Wednesd<br>ay | 1:30 PM     | X | X | X | Work on finalizing WTR                                 |
| Nov 12 | Friday        | 12:00<br>PM | X |   |   | <b>Send draft theory to peer reviewers</b>             |
| Nov 13 | Saturday      | 3:00 PM     | X | X | X | Work on finalizing WTR                                 |
| Nov 14 | Sunday        | 3:00 PM     | X | X | X | Work on finalizing WTR                                 |
| Nov 15 | Monday        | 12:00<br>PM | X |   |   | <b>Submit WTR</b>                                      |

## Raw Data

| Water Flow Rate Measurements |          |                   |                 |          |                  |                    |
|------------------------------|----------|-------------------|-----------------|----------|------------------|--------------------|
| Trial Number                 | Trial ID | Initial mass (kg) | Final mass (kg) | Time (s) | Flow rate (kg/s) | Flow rate (kg/min) |
| 1a                           | --       | 1.7               | 3.9             | 61.00    | 0.036065574      | 2.163934426        |

|    |    |     |      |       |             |             |
|----|----|-----|------|-------|-------------|-------------|
| 1b | ++ | 4.3 | 12.4 | 59.87 | 0.135293135 | 8.117588108 |
| 1c | +- | 1.8 | 3.8  | 60    | 0.033333333 | 2           |
| 1d | ++ | 4.3 | 12.4 | 59.87 | 0.135293135 | 8.117588108 |
| 2a | -- | 1.8 | 3.8  | 60.00 | 0.033333333 | 2           |
| 2b | ++ | 2.8 | 11.4 | 61.00 | 0.140983607 | 8.459016393 |
| 2c | +- | 2.8 | 11   | 60.00 | 0.136666667 | 8.2         |
| 2d | +- | 1.8 | 3.8  | 60.00 | 0.033333333 | 2           |
| 3a | -- | 0.4 | 2.5  | 61.35 | 0.034229829 | 2.053789731 |
| 3b | +- | 0.4 | 2.5  | 61.35 | 0.034229829 | 2.053789731 |
| 3c | ++ | 2.8 | 11   | 60.00 | 0.136666667 | 8.2         |
| 3d | +- | 1.5 | 9.8  | 60.87 | 0.136356169 | 8.181370133 |

|                     |             | Mass Flow<br>Controlllers    |                                 | Inlet and Outlet Gas Compositions        |  |   |   | Miscellaneous  |  |
|---------------------|-------------|------------------------------|---------------------------------|--|--|---|---|--|--|
| Trial<br>Num<br>ber | Trial<br>ID | Air MFC<br>reading<br>(SLPM) | CO2<br>MFC<br>reading<br>(SLPM) | CO2<br>inlet<br>gas<br>reading<br>(%CO2) | O2<br>inlet<br>gas<br>reading<br>(%O2) | CO2<br>outlet<br>gas<br>reading<br>(%CO2) | O2<br>outlet<br>gas<br>reading<br>(%O2) | Column<br>delta P<br>manomete<br>r reading<br>(in-H2O) | Barome<br>tric<br>pressur<br>e (in-<br>Hg) |
| 1a                  | --          | 10                           | 1.16                            | 7.86                                     | 15                                     | 7.04                                      | 15                                      | 0  | 30.03                                      |
| 1b                  | ++          | 10                           | 1.18                            | 7.78                                     | 15                                     | 4.67                                      | 15.1                                    | 0  | 30.2                                       |
| 1c                  | +-          | 20                           | 2.09                            | 7.79                                     | 14.9                                   | 7.45                                      | 14.9                                    | 0  | 30.1                                       |
| 1d                  | +-          | 20                           | 2.08                            | 7.81                                     | 14.9                                   | 6.13                                      | 15                                      | 0  | 30.1                                       |
| 2a                  | --          | 10                           | 1.06                            | 7.91                                     | 14.8                                   | 7.06                                      | 14.9                                    | 0  | 30.01                                      |
| 2b                  | ++          | 10                           | 1.07                            | 7.75                                     | 14.8                                   | 4.79                                      | 15                                      | 0  | 30.01                                      |
| 2c                  | +-          | 20                           | 2.08                            | 8.1                                      | 14.7                                   | 6.19                                      | 14.9                                    | 0  | 30.01                                      |
| 2d                  | +-          | 20                           | 2.09                            | 7.82                                     | 14.8                                   | 7.44                                      | 14.9                                    | 0  | 30.01                                      |
| 3a                  | --          | 10                           | 1.06                            | 7.96                                     | 14.1                                   | 7.13                                      | 14.2                                    | 0  | 29.99                                      |
| 3b                  | +-          | 20                           | 2.09                            | 7.8                                      | 14.1                                   | 7.43                                      | 14.9                                    | 0  | 29.99                                      |

|    |    |    |      |      |      |      |      |   |       |
|----|----|----|------|------|------|------|------|---|-------|
| 3c | -+ | 10 | 1.07 | 7.89 | 14.7 | 4.81 | 14.9 | 0 | 30.01 |
| 3d | ++ | 20 | 2.09 | 7.82 | 14.1 | 6.21 | 14.2 | 0 | 29.98 |

| Trial Number | Trial ID | Feed tank inlet | Thermocouple Temperatures |      |      |      | Thermistor Temperatures |                          |                        |                           |           |                                      |                              |
|--------------|----------|-----------------|---------------------------|------|------|------|-------------------------|--------------------------|------------------------|---------------------------|-----------|--------------------------------------|------------------------------|
|              |          |                 | T1                        | T2   | T3   | T4   | Channel 1 (gas inlet)   | Channel 2 (liquid inlet) | Channel 3 (gas outlet) | Channel 4 (liquid outlet) | Channel 5 | Circulating bath temperature setting | Circulating bath temperature |
| 1a           | --       | 24.7            | 23.5                      | 23.5 | 22.3 | 22.3 | 24.2                    | 25.3                     | 23.6                   | 25.1                      | 24.5      | 25                                   | 25                           |
| 1b           | +        | 24.1            | 24.1                      | 23.1 | 23.1 |      | 24.8                    | 24.6                     | 23.7                   | 24.6                      | 24.8      | 25                                   | 25                           |
| 1c           | +-       | 23.9            | 23.9                      | 23.2 | 23.1 |      | 25.8                    | 25.3                     | 23.9                   | 25.1                      | 24.8      | 25                                   | 25                           |
| 1d           | ++       | 24.1            | 24.1                      | 23.2 | 23.1 |      | 25.1                    | 23.8                     | 24.6                   | 24.8                      |           | 25                                   | 25                           |
| 2a           | --       | 23.7            | 23.7                      | 23.1 | 23   |      | 25.7                    | 25.3                     | 23.9                   | 25.2                      | 24.1      | 25                                   | 25                           |
| 2b           | +        | 23.9            | 23.9                      | 22.9 | 22.9 |      | 25.7                    | 24.6                     | 23.8                   | 24.6                      | 24.8      | 25                                   | 25                           |
| 2c           | ++       | 23.7            | 23.7                      | 22.7 | 22.8 |      | 26.0                    | 24.6                     | 23.9                   | 24.6                      | 24.9      | 25                                   | 25                           |
| 2d           | +-       | 23.5            | 23.5                      | 23   | 22.9 |      | 25.3                    | 24.0                     | 25.1                   | 24.8                      |           | 25                                   | 25                           |

|    |    |      |      |      |      |      |      |      |      |      |         |
|----|----|------|------|------|------|------|------|------|------|------|---------|
| 3a | -- |      |      |      |      | 25.7 | 23.8 | 25.6 | 23.9 |      |         |
|    |    |      |      |      |      | 25.2 | 7    | 6    | 4    | 3    | 25.5 25 |
| 3b | +- |      |      |      |      | 25.3 | 23.9 | 25.1 | 24.8 |      |         |
|    |    |      |      |      |      | 25.9 | 3    | 9    | 5    | 3    | 25.1 25 |
| 3c | -+ |      |      |      |      | 25.7 | 24.6 | 23.8 | 24.6 | 24.8 |         |
|    |    | 23.1 | 23.1 | 22.2 | 22.2 | 5    | 3    | 5    | 4    | 9    | 25.1 25 |
| 3d | ++ |      |      |      |      | 25.6 | 24.9 |      | 24.9 |      |         |
|    |    |      |      |      |      | 7    | 1    | 24   | 4    | 24   | 25.2 25 |

## Operating Procedure

| Step No.   | Operator ID | Step   |
|------------|-------------|--|
| <b>1.0</b> |             | <b>Check-in</b>  |
| 1.1        |             | INSPECT the work area.<br>CONFIRM that it is clean and orderly.<br>REPORT deficiencies immediately to the teaching assistant.  |
| 1.2        |             | INSPECT all the pipes.<br>CONFIRM that the pipes are in good condition.<br>REPORT deficiencies immediately to the teaching assistant.  |
| 1.3        |             | INSPECT all the valves.<br>CONFIRM that the valves are in good condition.<br>CONFIRM that none of the valves are leaking.<br>REPORT deficiencies immediately to the teaching assistant<br>ADJUST any valves not in the correct position  |
| 1.4        |             | INSPECT all electrical lines.<br>CONFIRM that they are all in good condition.<br>REPORT deficiencies immediately to the teaching assistant.  |
| 1.5        |             | INSPECT each piece of ancillary equipment.<br>CONFIRM that all items are present.<br>CONFIRM that all items are clean and in good condition.<br>CONFIRM that all power switches are on.<br>CONFIRM computer is on and working properly.<br>REPORT deficiencies immediately to the teaching assistant |
| 1.6        |             | TEST the stopwatches.<br>CONFIRM that they are operable.<br>REPORT deficiencies immediately to the teaching assistant.   |

|            |  |      |
|------------|--|------|
| 1.7        | CONFIRM that necessary valves are open according to valve list at startup. |      |
| Step No.   | Operator ID  | Step |
| <b>2.0</b> | <b>Plan for Trials</b>   |      |
|            | EXECUTE trials according to Table 1.                                       |      |
|            | IDENTIFY trial conditions from Table 1.                                    |      |

**Table 1. Experiment Conditions**

| Trial No. | Factor 1 | Factor 2 |
|-----------|----------|----------|
| 1a        | -        | -        |
| 1b        | -        | +        |
| 1c        | -        | -        |
| 1d        | -        | +        |
| 2a        | -        | +        |
| 2b        | +        | -        |
| 2c        | -        | +        |
| 2d        | +        | -        |
| 3a        | -        | -        |
| 3b        | +        | -        |
| 3c        | -        | -        |
| 3d        | +        | +        |
| 4a        | -        | -        |
| 4b        | +        | -        |
| 4c        | +        | -        |
| 4d        | +        | +        |
| 5a        | -        | +        |
| 5b        | +        | +        |
| 5c        | +        | +        |
| 5d        | +        | +        |
| Notes:    |          |          |



|     |  |
|-----|--|
|     | <p>If at any time there is liquid water in the inlet gas tube, in the outlet gas tube, or in the bulk water trap, shut down the liquid flow immediately and shutdown the system.</p>   |
| 3.2 | <p>OPEN VALVE VW1<br/> OPEN VALVE VW10<br/> OPEN VALVE VW2<br/> OPEN VALVE VW3<br/> ADJUST VALVE VD2 to set totalizer to about one revolution (1 gpm).</p>   |
| 3.3 | <p>TURN OFF the flow of R2.<br/> OPEN VALVE VD1<br/> ADJUST R1 so that R1 is greater than R3 by 0.1-0.2 gpm.<br/> OBSERVE feed tank to ensure there is overflow and the water level remains constant.</p>  |
| 3.4 | <p>OPEN VALVE VA2<br/> CLOSE VALVE VA3</p> <p style="text-align: center;">NOTE</p> <p style="text-align: center;">Either VA2 or VA3 must be open before the sampling pump is turned on. Once the sampling pump is on these valves cannot be closed at the same time, always open one before closing the other.</p>   |
| 3.5 | <p>TURN ON the power strip<br/> TURN ON the air dryer via the power controller<br/> TURN ON mass balance via power controller.<br/> TURN ON air mass flow controller via power controller.<br/> TURN ON the carbon dioxide mass flow controller via power controller.<br/> TURN ON the carbon dioxide heater.<br/> TURN ON thermistor thermometer.<br/> TURN ON computer via power controller.<br/> TURN ON both circuit switches.</p> |
| 3.6 | <p>CHECK the silica gel filter.</p>  |

IS THE SILICA GEL FILTER MORE THAN 25% SATURATED WITH MOISTURE?

**YES** HOLD the ring and GENTLY PULL the filter from the fitting.  
REMOVE the filter.  
GRAB a new filter.  
IDENTIFY the flow direction.  
HOLD the ring and CONNECT the new filter to the fitting.  
ENSURE the connection are tight.

**NO** PROCEED to the next step.

NOTE

The silica gel filter is “fully charged”, meaning completely void of moisture, when it is all blue. It turns pink when it starts to collect moisture.

3.7

OPEN air mass flow rate controller program on the computer.  
CLICK “enter” a few times.

NOTE

Always change the value of the air flow before changing the value of CO2 flow.

3.8

TYPE a0 and click “enter” on the computer.  
TYPE c0 and click “enter” on the computer.  
TYPE A and the flow rate of air according to the trial and the handout available.  
TYPE C and the flow rate of CO2 according to the trial and the handout available.

NOTE

If you type an incorrect value on the computer and it outputs a question mark press “enter” a few times and re-enter the correct value.



|      |  |
|------|--|
| 3.9  | <p>OBSERVE the air flow controller setting change to the desired flow rate.</p> <p>LISTEN the air flow starting.</p> <p>IS THERE A LOUD OR ABRUPT NOISE?</p> <p><b>YES</b> TYPE a0 and click “enter” on the computer to stop air flow.<br/>TYPE c0 and click “enter” on the computer to stop CO2 flow.<br/>RE-ENTER a lower air and CO2 flow rates and raise values slowly.</p> <p><b>NO</b> PROCEED to the next step.</p>                           |
| 3.10 | <p>WAIT approximately 5-6 minutes for the sampling pump setting to stabilize.</p> <p>ENSURE the sampling pump setting reads 8%.</p> <p style="text-align: center;">NOTE</p> <p style="text-align: center;">If the sampling pump reads below 7.5% or above 8.5% let the teaching assistant or lab engineer know.</p>  |
| 3.11 | <p>IS THE TEMPERATURE OF THE SUBMERGED HEATER IN THE TANK ABOUT 25°C?</p> <p><b>YES</b> PROCEED to the next step.</p> <p><b>NO IF ABOVE 25 °C</b></p> <p>INCREASE the flow rate of R1#.</p> <p>WAIT until the temperature is about 25 °C.</p> <p>PROCEED to the next step.</p> <p><b>IF BELOW 25 °C</b></p> <p>DECREASE OR TURN OFF the flow of chilled water.</p> <p>WAIT until the temperature is about 25 C.</p> <p>PROCEED to the next step.</p> |

|  |
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| Step No. | Operator ID | Step   |
|----------|-------------|--|
| 4.0      |             | <b>Measuring the water flow rate</b>   |
| 4.1      |             | <p>SET R3 to the desired flow rate for the current trial.</p> <p>CLOSE draining valve.</p> <p style="text-align: center;"><b>NOTE</b></p> <p style="text-align: center;"><b>DO NOT PRESS THE TARE BUTTON ON THE MAS BALANCE AT ANY TIME.</b></p>   |
| 4.2      |             | <p>ALLOW water to accumulate in the tank to a convenient starting point.</p> <p>RECORD the initial mass.</p> <p>START stopwatch at the starting point and time it for about 1 minute.</p> <p>STOP timing after one minute has passed.</p> <p>RECORD final mass.</p> <p>RECORD the time displayed on the stopwatch.</p> <p>OPEN draining valve.</p> |
| 4.3      |             | <p>CALCULATE the measured water flow rate.</p> <p>IS THE WATER FLOW RATE MEASURED WITHIN 0.1 KG/MIN OF THE TARGET FLOW RATE?</p> <p><b>YES</b> PROCEED to the next step.</p> <p><b>NO</b> ADJUST R3.</p> <p>CLOSE draining valve.</p> <p>REPEAT step 4.2 for “Measuring the water flow rate”.</p>  |
| Step No. | Operator ID | Step   |
| 5.0      |             | <b>Conducting Trials</b>   |
| 5.1      |             | ENSURE the sampling pump still reads 8%.   |

|          |             |  |
|----------|-------------|--|
|          |             | <p>ENSURE the temperature of the submerged heater in the tank is 25°C.</p> <p>RECORD T1 (inlet of the tube side – domestic water – of the heat exchanger) from the thermocouple thermometer.</p> <p>RECORD T2 (outlet of the tube side – domestic water – of the heat exchanger) from the thermocouple thermometer.</p> <p>RECORD T3 (inlet of the shell side – chilled water – of the heat exchanger) from the thermocouple thermometer.</p> <p>RECORD T4 (outlet of the shell side – chilled water – of the heat exchanger) from the thermocouple thermometer.</p> |
| 5.2      |             | <p>RECORD T1 (gas inlet) from the thermistor thermometer.</p> <p>RECORD T2 (liquid inlet) from the thermistor thermometer.</p> <p>RECORD T3 (gas outlet) from the thermistor thermometer.</p> <p>RECORD T4 (liquid outlet) from the thermistor thermometer.</p>  |
| 5.3      |             | <p>ENSURE VALVE VA2 is open. (inlet CO2)</p> <p>ENSURE VALVE VA3 is closed. (outlet CO2)</p> <p>RECORD the CO2 inlet gas stream.</p>   |
| 5.4      |             | <p>OPEN VALVE VA3 (outlet CO2)</p> <p>CLOSE VALVE VA2 (inlet CO2)</p> <p>WAIT about 5-6 minutes to the CO2 composition reading to stabilize.</p> <p>RECORD the CO2 outlet gas stream.</p>  |
| 5.5      |             | <p>IS THERE SUFFICIENT TIME REMAINING TO CONDUCT ANOTHER TRIAL?</p> <p><b>YES</b> PROCEED to step 6.0 (Prepare for next trial).</p> <p><b>NO</b> PROCEED to step 9.0 (Normal shutdown).</p>  |
| Step No. | Operator ID | Step   |
| 6.0      |             | <b>Prepare for Next Trial</b>  |
| 6.1      |             | IDENTIFY trial conditions according to Table 1.  |
| 6.2      |             | <p>SET R3 to the desired flow rate according to the trial conditions.</p> <p>SET R1 about 0.1-0.2 gpm greater than R3.</p>   |
| 6.3      |             | CHECK the silica gel filter.   |

|     |  |
|-----|--|
|     | <p>IS THE SILICA GEL FILTER MORE THAN 25% SATURATED WITH MOISTURE?</p> <p><b>YES</b> HOLD the ring and GENTLY PULL the filter from the fitting.<br/>REMOVE the filter.<br/>GRAB a new filter.<br/>IDENTIFY the flow direction.<br/>HOLD the ring and CONNECT the new filter to the fitting.<br/>ENSURE the connections are tight.</p> <p><b>NO</b> PROCEED to the next step.</p> |
| 6.4 | FOLLOW procedural steps 3.8-3.11 of "Startup".   |
| 6.5 | FOLLOW procedural steps 4.0-4.3 of "Measuring the water flow rate".  |
| 6.6 | FOLLOW procedural steps 5.0-5.4 of "Conducting trials".  |

| Step No.   | Operator ID | Step   |
|------------|-------------|--|
| <b>7.0</b> |             | <b>ODEX (Operating at a different temperature)</b>   |
| 7.1        |             | REQUEST to the lab engineer to preset the water heater to 35°C during the lab day before you plan to conduct the ODEX. |
| 7.2        |             | FOLLOW procedural steps 1.0-1.6 for "Check-in"   |
| 7.3        |             | EXECUTE trials according to Table 2 below.<br>IDENTIFY trial conditions in Table 2 below.                              |

Table 2

| Trial No. | Factor 1 | Factor 2 |
|-----------|----------|----------|
| 1a        | -        | -        |
| 1b        | +        | -        |
| 1c        | -        | -        |
| 1d        | +        | -        |
| 2a        | +        | -        |

|    |   |   |
|----|---|---|
| 2b | - | + |
| 2c | + | - |
| 2d | - | + |
| 3a | - | - |
| 3b | - | + |
| 3c | - | - |
| 3d | + | + |
| 4a | - | - |
| 4b | - | + |
| 4c | - | + |
| 4d | + | + |
| 5a | + | - |
| 5b | + | + |
| 5c | + | + |
| 5d | + | + |

Notes:

Conduct all trials at 35 °C

Conduct all trials at an inlet gas composition of 0.080 mole fraction carbon dioxide, based upon the mass flow rates of supplied air and carbon dioxide.

Conduct the trials in the order indicated: 1a...1b...1c...1d...2a...2b...2c...2d.... When resuming the experiment at a later lab period, start on the trial following where you left off at the prior lab period. Do not deviate from this order.

Factor 1 refers to the inlet flow rate of air. Use values for Factor 1 as follows:

-level = 10SLPM      +level = 20SLPM

Do not use other values for these levels.

Factor 2 refers to the inlet flow rate of water. Use values for Factor 2 as follows:

- level = 2.0 kg/min      + level = 8.0 kg/min

The actual flow rates should be within 0.1 kg/min of the target flow rates above.

*If an equipment malfunction or other problem prevents conducting the trials in this order or with factors at these values, then explain this issue in your report.*

7.4

FOLLOW procedural steps 3.0-3.10 for "Startup"

|            |  |
|------------|--|
| 7.5        | <p>IS THE TEMPERATURE OF THE SUBMERGED HEATER IN THE TANK ABOUT 35°C?</p> <p><b>YES</b> PROCEED to the next step.</p> <p><b>NO IF ABOVE 35 °C</b></p> <p>INCREASE the flow rate of R1#.</p> <p>WAIT until the temperature is about 35 °C.</p> <p>PROCEED to the next step.</p> <p><b>IF BELOW 35 °C</b></p> <p>DECREASE OR TURN OFF the flow of chilled water.</p> <p>WAIT until the temperature is about 35°C.</p> <p>PROCEED to the next step.</p> |
| 7.6        | FOLLOW procedural steps 4.0-4.3 for “Measuring the water flow rate”  |
| 7.7        | <p>FOLLOW procedural steps 5.0-5.5 for “Conducting trials” with the following changes:</p> <p>ENSURE the temperature of the submerged heater in the tank is 35°C.</p>  |
| Step No.   | Operator ID Step   |
| <b>8.0</b> | <b>SODEX</b>   |
| 8.1        | FOLLOW procedural steps 1.0 – 1.6 for “Check-in”   |
| 8.2        | <p>EXECUTE trials according to Table 3.</p> <p>IDENTIFY trial conditions in Table 3.</p>   |
| 8.4        | <p>FOLLOW procedural steps 3.0-3.6 for “Startup” with the following changes:</p> <p>FLIP the disconnect switch up.</p> <p>TURN ON water heater.</p> <p style="text-align: center;">NOTE</p> <p style="text-align: center;">This change should be made in between procedural steps 3.5 and 3.6</p>  |

|     |   |
|-----|---|
| 8.5 | FOLLOW procedural steps 3.7-3.11 for “Startup” with the following change during step 3.10:<br><br>ENSURE the sampling pump setting reads the inlet gas composition according to the trial conditions.       |
| 8.6 | FOLLOW procedural steps 4.0-4.3 for “Measuring the water flow rate”   |
| 8.7 | FOLLOW procedural steps 5.0-5.5 for “Conducting trials” with the following change during step 5.1:<br><br>ENSURE the sampling pump still reads the inlet gas composition according to the trial conditions. |

Table 3

| Trial No. | Factor 1 | Factor 2 |
|-----------|----------|----------|
| 1a        | -        | -        |
| 1b        | -        | +        |
| 1c        | -        | -        |
| 1d        | -        | +        |
| 2a        | +        | -        |
| 2b        | -        | +        |
| 2c        | +        | -        |
| 2d        | -        | +        |
| 3a        | -        | -        |
| 3b        | +        | -        |
| 3c        | -        | -        |
| 3d        | +        | +        |
| 4a        | -        | -        |
| 4b        | -        | +        |
| 4c        | -        | +        |
| 4d        | +        | +        |
| 5a        | -        | +        |
| 5b        | +        | +        |
| 5c        | +        | +        |
| 5d        | +        | +        |
| Notes:    |          |          |

Notes:

Conduct all trials at 35 °C

Conduct all trials at an inlet gas composition of 0.15 mole fraction carbon dioxide, based upon the mass flow rates of supplied air and carbon dioxide.

Conduct the trials in the order indicated: 1a...1b...1c...1d...2a...2b...2c...2d.... When resuming the experiment at a later lab period, start on the trial following where you left off at the prior lab period. Do not deviate from this order.

Factor 1 refers to the inlet flow rate of air. Use values for Factor 1 as follows:

-level = 10SLPM      +level = 20SLPM

Do not use other values for these levels.

Factor 2 refers to the inlet flow rate of water. Use values for Factor 2 as follows:

- level = 2.0 kg/min      + level = 8.0 kg/min

The actual flow rates should be within 0.1 kg/min of the target flow rates above.

*If an equipment malfunction or other problem prevents conducting the trials in this order or with factors at these values, then explain this issue in your report.*

| Step No.   | Operator ID | Step   |
|------------|-------------|--|
| <b>9.0</b> |             | <b>Normal Shutdown</b>   |
| 9.1        |             | STOP conducting the experiment   |
| 9.2        |             | TURN OFF the circulating pump via the power supply.  |
| 9.3        |             | TURN OFF the gas flow by entering 'a0' and 'c0' on the computer.   |
| 9.4        |             | TURN OFF the thermocouple thermometer by pressing and holding the off button.  |
| 9.5        |             | TURN OFF the sampling pump.<br>TURN OFF the carbon dioxide analyzer<br>CLOSE VALVE V <sub>A-3</sub> and V <sub>A-4</sub> (sample inlet and outlet gas streams)   |
| 9.6        |             | TURN OFF the circulating temperature controller manually.<br>TURN OFF the power controller.<br>TURN OFF mass balance via power controller.<br>TURN OFF the carbon dioxide mass flow controller via power controller.<br>TURN OFF the carbon dioxide heater via power controller. |
| 9.7        |             | TURN OFF air dryer via power controller on flowsheet<br>TURN OFF air mass flow controller via power controller<br>TURN OFF thermometer via power controller  |

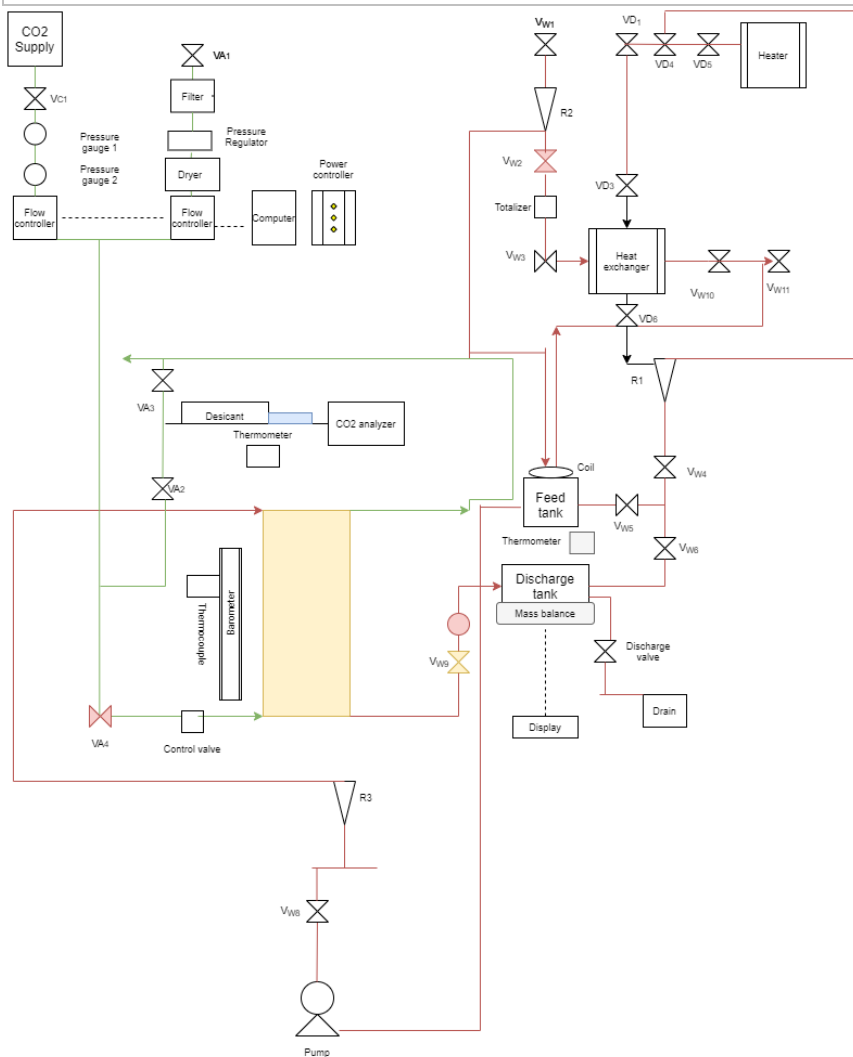


|      |   |
|------|---|
|      | TURN OFF computer via power controller<br>TURN OFF both circuit switches                                |
| 9.8  | CLOSE VALVE V <sub>A-1</sub> for air supply.<br>CLOSE VALVE V <sub>C-1</sub> for carbon dioxide supply. |
| 9.9  | CLOSE VALVE V <sub>W-1</sub> and V <sub>W-2</sub> .<br>CLOSE VALVE VD-1.                                |
| 9.10 | TURN OFF water heater.  |

| Step No.    | Operator ID | Step   |
|-------------|-------------|--|
| <b>10.0</b> |             | <b>Material Disposition and equipment decontamination</b>  |
| 10.1        |             | REFER to the Chemical Hazards and Precautions table for instructions on the disposal of all other chemicals used in this experiment. |
| 10.2        |             | ENSURE that the outside surface of all equipment is clean and dry.<br>USE paper towels to dry any equipment as required.             |

| Step No.    | Operator ID | Step   |
|-------------|-------------|--|
| <b>11.0</b> |             | <b>Cleanup and Checkout</b>  |
| 11.1        |             | NOTE<br>At the end of the cleanup section, the laboratory work area should look as it did at the beginning of the laboratory period.<br><br>REPLACE all ancillary equipment to their proper locations. |
| 11.2        |             | REMOVE all personal items from the experiment area.  |
| 11.3        |             | POSITION all chairs and stools where you found them.   |
| 11.4        |             | CONFIRM that all data entry forms are complete and legible.  |
| 11.5        |             | REPORT to the assigned teaching assistant that you are ready for inspection and checkout.  |
| 11.6        |             | WASH your hands before leaving the laboratory.   |
| Step No.    | Operator ID | Step   |
| <b>12.0</b> |             | <b>Emergency Shutdown</b>  |
| 12.1        |             | SHUT OFF Valve VD-1  |

|      |  |
|------|--|
| 12.2 | SHUT OFF Valve VA-1.<br>SHUT OFF VALVE VC-1.<br>SHUT OFF VALVE VW-1 and VW-11. |
| 12.3 | SHUT OFF circuit switches to turn down all power.                              |
| 12.4 | PLACE all ancillary equipment on laboratory table.                             |
| 12.5 | VISUALIZE the emergency exit and FOLLOW the evacuation route.                  |
| 12.6 | WAIT until you have received an “all clear” notification.                      |
| 12.7 | RETURN to the laboratory if you have received clearance.                       |



## PAQs

Prediction and Analysis Questions Expt. 380 (Absorption)

Team 23:

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- a. What is the value of  $V_{SA}$ , the molal flow rate (mol/s) of air supplied to the bottom of the tower, at the process conditions listed below? What is the value of  $y_b$ , the mole fraction of  $CO_2$  in the gas supplied to the tower? What is the value of  $V_b$ , the molal flow rate (mol/s) of gas supplied to the bottom of the tower?

| Process Variable                                     | Symbol        | Measurement             |
|--|---------------|-------------------------|
| Temperature of gas at the bottom of the tower        | $\Theta_{Gb}$ | 22.07°C                 |
| Ambient pressure                                     | $P_{amb}$     | 30.10 inHg              |
| Pressure drop across the packing                     | $P_b - P_a$   | 0.42 ftH <sub>2</sub> O |
| Std volumetric flow rate of supplied air             | $V_{SA}$      | 39.87 SLPM              |
| Std volumetric flow rate of supplied CO <sub>2</sub> | $V_{SC}$      | 3.466 SLPM              |

$$T = 22.07^\circ\text{C} + 273.15 = 295.22 \text{ K}$$

$$P = 30.10 \text{ in Hg} \times 2.54 = 76.454 \text{ mm Hg} \times \frac{14.696 \text{ psia}}{760 \text{ mm Hg}} = 1.480 \text{ psia}$$

$$F[\text{SLPM}] = F[\text{LPM}] \times \left(\frac{294.26}{T_{GAS}}\right) \times \left(\frac{P_{GAS}}{14.696}\right)$$

$$39.87 \text{ SLPM} = F[\text{LPM}] \times \left(\frac{294.26}{295.22\text{K}}\right) \times \left(\frac{1.480}{14.696}\right)$$

$$F[\text{LPM}] = 404.57 \text{ LPM} = V_{SA}$$

$$3.466 \text{ SLPM} = F[\text{LPM}] \times \left(\frac{294.26}{295.22\text{K}}\right) \times \left(\frac{1.480}{14.696}\right)$$

$$F[LPM] = 35.170 LPM = V_{SC}$$

$$P = 30.10 \text{ in Hg} \times 2.54 = 76.454 \text{ mm Hg} \times \frac{0.00131579 \text{ atm}}{1 \text{ mm Hg}} = 0.10059740866 \text{ atm}$$

$$V_{air} = 404.57 \frac{L}{min} \times \frac{1 \text{ min}}{60 \text{ sec}} = 6.743 \frac{L}{sec} V_{CO_2} = 35.170 \frac{L}{min} \times \frac{1 \text{ min}}{60 \text{ sec}} = 0.586 \frac{L}{sec}$$

$$P \times V_{air} = nRT$$

$$n = \frac{P \times V_{air}}{R \times T} = \frac{(0.100597 \text{ atm}) \times (6.743 \frac{L}{sec})}{(0.0821 \frac{atm * L}{mol * K}) \times (295.22 \text{ K})} = 0.0279 \frac{mol}{sec}$$

$$V_{sA} = 0.0279 \frac{mol}{sec}$$

$$P \times V_{CO_2} = nRT$$

$$n = \frac{P \times V_{CO_2}}{R \times T} = \frac{(0.100597 \text{ atm}) \times (0.586 \frac{L}{sec})}{(0.0821 \frac{atm * L}{mol * K}) \times (295.22 \text{ K})} = 0.0243 \frac{mol}{sec}$$

$$n_{total} = 0.027 + 0.0243 = 0.0298 \frac{mol}{sec}$$

$$y_b = \frac{0.00239}{0.02989} = 0.08022$$

$$n_{total} = V_b = 0.02989 \frac{mol}{sec}$$

- b. An energy balance for the tube side of a countercurrent double-pipe heat exchanger at steady state provides an equation to determine the rate of heat transfer in terms of measured variables and heat capacity. ( $C_p$  is assumed to be independent of temperature.)

$$Q = m_t C_{pt} (T_{at} - T_{bt})$$

Write an analogous equation using a material balance on the gas stream to determine  $n_{abs}$  (mol/s), the rate of absorption of  $CO_2$ , in terms of experimentally determined variables the molal flow rates of the gas entering and exiting the packed tower and the corresponding gas compositions.

$$\text{In} - \text{Out} - \text{Absorption} = 0$$

- c. An energy balance for the shell side of a countercurrent double-pipe heat exchanger at steady state provides an equation to determine the shell outlet temperature in terms of the shell inlet temperature, the rate of heat transfer, the shell mass flow rate, and the heat capacity.

$$T_{bs} = T_{as} - \frac{Q}{m_s C_{ps}}$$

Write an analogous equation using a material balance on the liquid stream to determine the CO<sub>2</sub> mole fraction of the liquid exiting the bottom of the tower in terms of the CO<sub>2</sub> mole fraction of the liquid entering the top of the tower, the molal flow rate of liquid, and the rate of absorption of CO<sub>2</sub>.

$$V_w * \frac{x_{iCO_2}}{1 - x_{iCO_2}} + V_{gas} * \frac{y_{CO_2}}{1 - y_{CO_2}} = V_w * \frac{x_{CO_2}}{1 - x_{CO_2}} + V_{gas} * \frac{y_{iCO_2}}{1 - y_{iCO_2}}$$

Since concentration of CO<sub>2</sub> is very small we can assume that (1-x) and (1-y) are close to 1.

$$V_w * x_{iCO_2} + V_{gas} * y_{CO_2} = V_w * x_{CO_2} + V_{gas} * y_{iCO_2}$$

$$x_{CO_2} = \frac{V_w * x_{iCO_2} + V_{gas} * y_{CO_2} - V_{gas} * y_{iCO_2}}{V_w} \Rightarrow x_{CO_2} = \frac{V_w * x_{iCO_2} + V_{gas} * (y_{CO_2} - y_{iCO_2})}{V_w}$$

w represents H<sub>2</sub>O

x<sub>iCO<sub>2</sub></sub> represents inlet liquid mole fraction

x<sub>CO<sub>2</sub></sub> represents outlet liquid mole fraction

y<sub>iCO<sub>2</sub></sub> represents inlet gas mole fraction

y<sub>CO<sub>2</sub></sub> represents outlet gas mole fraction

d. Henry's Law can be used to calculate equilibrium compositions for dilute systems such as CO<sub>2</sub> gas dissolved in water. The form of the Henry's law varies among literature sources, and may even vary within the same textbook! We choose the form used in CHEMCAD:

$$p_i = H_i x_i^*$$

where p<sub>i</sub> is the partial pressure of species i in the gas phase, H<sub>i</sub> is the Henry's-law constant, and x<sub>i</sub><sup>\*</sup> is the mole fraction of species i in the liquid phase at equilibrium. To simplify the notation, we drop the subscript referring to species i, recognizing that p, H, and x<sup>\*</sup> all refer to carbon dioxide.

Based upon the correlation in CHEMCAD, the equation for Henry's-law constant H for this system is:

$$\ln[H/(\text{atm})] = A' / (T/K) + B' \ln(T/K) + C' (T/K) + D'$$

$$A' = -773.33 \quad B' = 0.121 \quad C' = 0.026622 \quad D' = 1.3675$$

What is the value of H at the bottom of the tower, assuming that the temperature of the liquid exiting the bottom of the tower is Θ<sub>lb</sub> = 19.79 C?

$$\ln H = \frac{A'}{T} + B' \ln T + C' T + D' = \frac{-773.33}{292.79} + 0.121 * \ln(292.79) + 0.026622 * 292.79 + 1.3675$$

$$\ln H = 7.208$$

$$H = e^{7.208} = 1350.2 \text{ atm} * \frac{101325 \text{ Pa}}{1 \text{ atm}} \Rightarrow H = 1.37 * 10^8 \text{ Pa} = 137 \text{ MPa}$$

- e. What is the value of  $x_b^*$ , the equilibrium value of the CO<sub>2</sub> fraction in the liquid at the bottom of the tower, at the process conditions listed in question a and at  $\Theta_{Lb} = 19.79$  C?

We need the flow rate in mol/s:

$$X = \frac{P_{amb} * y}{H}$$

$$4 \frac{g}{min} * \frac{1}{mol} * \frac{1min}{60s} = 0.004 \frac{mol}{s}$$

Henry's Law:

$$H' = \frac{H}{P}$$

$$Y = H' * X$$

$$0.0068 \frac{mol}{s} = 0.004 \frac{mol}{s} \left( \frac{X_{CO2}}{1 - X_{CO2}} \right) + 0.03 \frac{mol}{s} \left( \frac{1420 X_{CO2}}{1 - 1420 X_{CO2}} \right)$$

Solving for X with Excel Solver:

$$X = 0.0000603 = 6.03 * 10^{-5}$$

- f. For a countercurrent double-pipe heat exchanger, the average overall driving-force for heat transfer, typically called the log-mean temperature difference, is expressed in terms of the temperatures at the shell inlet, the shell outlet, the tube inlet, and the tube outlet.

$$\Delta T_{lm} = [(T_{bs} - T_{bt}) - (T_{as} - T_{at})] / \ln[(T_{bs} - T_{bt}) / (T_{as} - T_{at})]$$

Write an analogous expression for the average overall driving-force for mass transfer in the liquid phase during absorption.

$$(x^* - x)_{LM} = \frac{(x_1^* - x_1) - (x_2^* - x_2)}{\ln \left[ \frac{(1 - x_1)}{(1 - x_1^*)} \right]}$$

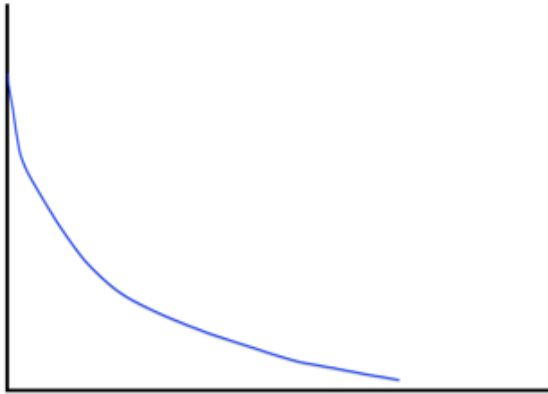
$x_1^*$  = mole fraction of solute in the liquid that is in equilibrium with the vapor of mole fraction  $y_1$

$x_2^*$  = mole fraction of solute in the liquid that is in equilibrium with the vapor of mole fraction  $y_2$

$(x_1^* - x_1)$  = concentration difference at the bottom of the column

$(x_2^* - x_2)$  = concentration difference at the top of the column

Sketch the mass fraction profile of the liquid phase for the absorption unit.



- g. For a countercurrent double-pipe heat exchanger, the overall heat-transfer coefficient may be determined experimentally in terms of the rate of heat transfer, the surface area for heat transfer, and the average overall driving-force for heat transfer.

$$K'_{xa} = \frac{L(X_1 - X_2)}{Zt * S(X^* - X)_m}$$

Write an analogous equation for the overall volumetric liquid-phase mass-transfer coefficient  $K_{x,a}$  in terms of the rate of absorption, the volume of the packing ( $SZT$ ), and the average overall driving-force for mass transfer in the liquid phase.

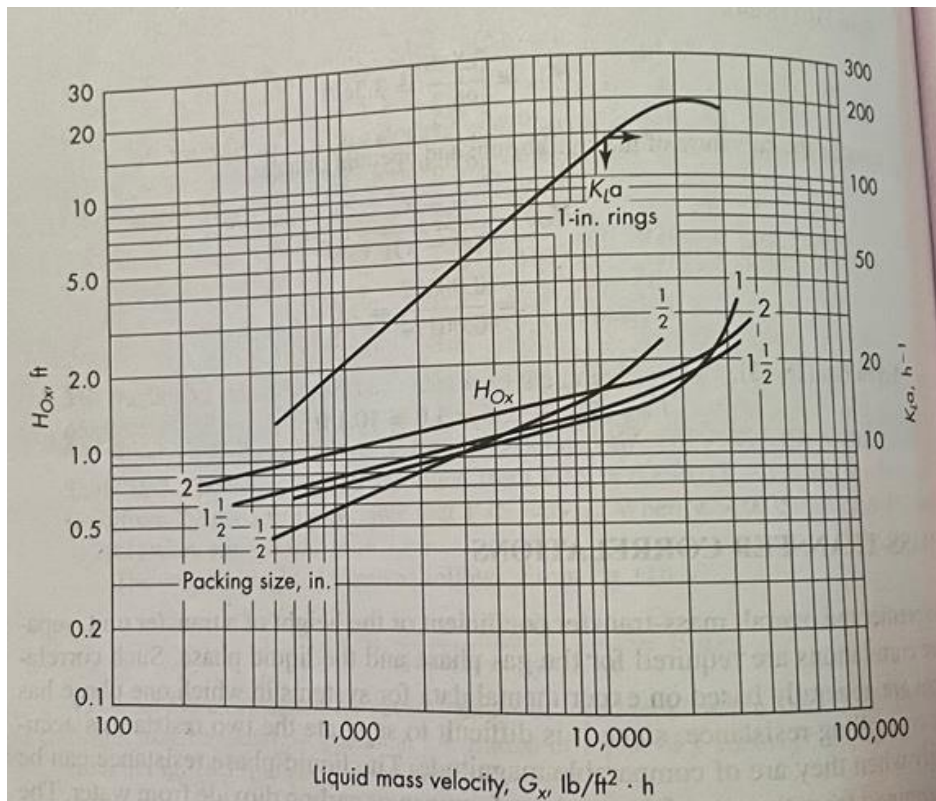
$$k'_{x,a} = \frac{L[x_1 - x_2]}{SZ_T[x^* - x]_M}$$

- h. Determine  $G_x$ , the mass velocity of liquid, and  $L/S$ , the molal load of liquid, for a water flow rate of 4.00 kg/min through our packed tower.

$$L/S = \frac{0.0037}{0.00502} = 0.737 \frac{\text{kmol}}{\text{m}^2\text{s}}$$

$$G_x = 47,770.21 \frac{\text{kg}}{\text{m}^2\text{h}} * \frac{1 \text{ h}}{3600 \text{ s}} = 13.27 \frac{\text{kg}}{\text{m}^2\text{s}}$$

- i. Determine  $(H_{O_x})_r$  the height of a transfer unit for oxygen in water at 25 °C with 1" Raschig ring packing.



**FIGURE 18.21**  
Height of a transfer unit for desorption of oxygen from water at 25°C with Raschig ring packing. (Note: In this system  $H_{Ox} \cong H_x$ .)

Figure 18.22 from the

McCabe textbook

$$G_x = 13.27 \frac{\text{kg}}{\text{m}^2 \text{s}} \times \frac{2.20462 \text{ lb}}{1 \text{ kg}} \times \left(\frac{1 \text{ m}}{3.2808 \text{ ft}}\right)^2 \times \frac{3600 \text{ sec}}{1 \text{ hr}} = 9784.70 \frac{\text{lb}}{\text{ft}^2 \text{ h}}$$

$$H_{Ox} \approx 1.5 \text{ ft} \times \frac{0.3048 \text{ m}}{1 \text{ ft}} = 0.457 \text{ m}$$

- j. The diffusivity of CO<sub>2</sub> in water is listed as 2.00 × 10<sup>-9</sup> m<sup>2</sup>/s at 25 °C. Estimate the diffusivity of CO<sub>2</sub> in water at 20 °C. Hint: Use the Wilke-Chang correlation to solve for the ratio of the diffusivities at the two temperatures.

$$D_{AB} = 7.4 \times 10^{-8} \frac{(\psi_B M_B)^2 T}{\mu V_A^{0.6}}$$

$$D_{AB} = 7.4 \times 10^{-8} \frac{(2.6 \times 18.01528 \frac{\text{g}}{\text{mol}})^2 (25 + 273)}{0.0010005 \times V_A^{0.6}}$$

$$D_{AB} = 1.75e^{-9} \frac{\text{m}^2}{\text{s}}$$

- k. Estimate the Schmidt number for a dilute solution of carbon dioxide in water at 20 °C.



$$Sc = \frac{\mu}{\rho D_{AB}}$$

$$\mu = 0.0010005$$

$$\rho = 998.23$$

$$Sc = \frac{0.0010005}{(998.23) * (1.75e^{-9})} = 572.73$$

- l. Estimate  $H_{Ox}$  and  $K_{xa}$  for the absorption of carbon dioxide into water at a liquid flow rate of 4.00 kg/min at 20 °C. Assume negligible resistance to mass transfer in the gas phase so therefore  $H_{Ox} = H_x$  and  $K_{xa} = k_{xa}$ .

$$H_{Ox} = \frac{L/S}{K'_{xa}}$$

$$\frac{1}{K'_{xa}} = \frac{1}{k_{xa}} + \frac{(1-y)_L}{mk_{ya}}$$

$$H_{Ox} = 0.51 \text{ m}$$

$$K_{xa} = 1.45 \frac{\text{kmol}}{\text{m}^3 \text{s}}$$

- m. Present a "back of the envelope" calculation comparing the magnitude of the resistance to mass transfer in the gas phase compared to that in the liquid phase for the absorption system in this experiment. Examine quantitatively the validity of the assumption  $K_{xa} = k_{xa}$ . Hint: Refer to McCabe, p. 580.

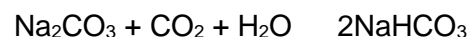
$$\frac{1}{K_G} = \frac{1}{k_G} + \frac{M}{k_L}$$

$$\frac{1}{K_L} = \frac{1}{Mk_G} + \frac{1}{k_L}$$

Since  $\text{CO}_2$  in  $\text{H}_2\text{O}$  has low solubility,  $M$  is very large the value of  $\frac{1}{Mk_G} \sim 0$  so we can neglect this term and the equation becomes:

$$\frac{1}{K_L} = \frac{1}{k_L} \Rightarrow K_L = k_L \Rightarrow K_{xa} = k_{xa}$$

- n. Write the balanced chemical reaction equation for the reaction of carbon dioxide with soda ash in water.



- o. Calculate the maximum rate of absorption of carbon dioxide from air into the soda ash solution in mol/s, assuming ideal efficiency, i.e., the rate of mass transfer and the rate of chemical reaction are instantaneous. Assume an inlet air flow rate of 20.0 L/min and an inlet liquid flow rate of 4.00 L/min at 20 °C. In addition, assume the inlet gas content of carbon

dioxide is 0.080 mole fraction and the inlet liquid concentration of soda ash is 0.050 mol/L. For this ideal case, what is the outlet gas concentration of carbon dioxide and outlet liquid concentration of soda ash?

$$G_s = G[1 - y] \quad y = \frac{y}{1-y}$$

$$L_s = L[1 - x] \quad x = \frac{x}{1-x}$$

Ideal case:  $y_2 = 0$

$$y_1 = \frac{0.08}{1 - 0.08} = 0.09$$

$$x_2 = \frac{0.05}{1 - 0.05} = 0.05$$

$$y_1 = \frac{L_s}{G_s}(x - x_2) + y_2$$

$$x = X_2 + y_1 \frac{G_s}{L_s} = 0.503$$

$$x_1 = \frac{x}{1 - x} X_1 = 0.334$$

$$c_b = c_{bo} * (1 - x_1) = 0.05 * (1 - 0.334)$$

$$c_b = 0.033 \text{ mol/L}$$