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Carbon Dioxide Absorption in a Fabricated Wetted-Wall Column Using Varying Concentrations of Aqueous Ammonia

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Due to the continued increasing levels of CO_2 emissions that is contributing to climate change, CO_2 mitigation technologies, particularly carbon capture and storage, are being developed to address the goal of abating CO_2 levels. Carbon capture technologies can be applied at the pre-combustion, oxy-fuel combustion, and post-combustion stages, the latter being the most widely used due to its flexibility. Among the several CO_2 separation processes available for carbon capture, absorption is the most widely used where amine solutions are used as absorbents. This paper highlights the use of a wetted wall column fabricated by Siy and Villanueva (2012) and simulated flue gas to determine the performance of CO_2 absorption in terms of the percentage of CO_2 absorbed, the steady state time, and the overall gas mass transfer coefficient. The concentrations used were 1, 5, 10, and 15% $NH_{3(aq)}$ at a constant temperature range of 12-17°C, solvent flow rate of 100 mL/min, and simulated flue gas flow rate of 2 L/min. It was found that increasing the solvent concentration resulted in a proportional increase both in the percentage of CO_2 absorbed and the overall gas mass transfer coefficient. The average percentage of CO_2 absorbed ranged within 52.25% to 95.29% while the overall mass transfer coefficient ranged from 0.1843 to 0.7746 $mmol/m^2 \cdot s \cdot kPa$. However, erratic behavior was seen for the time required for the system to reach steady state. Using Design Expert^{TM} for analysis, the results showed that the effect of varying the concentration had a significant effect on the percentage of CO_2 absorbed and the overall gas mass transfer coefficient. The results proved that the greater the aqueous ammonia concentration, the greater the percentage of CO_2 absorbed. The range of 5-10% aqueous ammonia is recommended because the percentage of CO_2 absorbed peaks at an average of 92% beyond the range of 5-10%.

Keywords: Carbon capture, Absorption, Wetted-wall column, Aqueous ammonia

INTRODUCTION

In the 21st century, there is a continued upward trend in CO_2 levels in the atmosphere, most of which is caused by fossil fuel combustion as the dominant form of energy utilized. Main sources of CO_2 emissions from fossil fuel burning come from large combustion units such as electricity generation and smaller distributed sources such as automobiles (Intergovernmental Panel on Climate Change [IPCC], 2005). In 1992, the United Nations Framework Convention on Climate Change (UNFCCC) had the ultimate objective of “stabilization of greenhouse gas concentrations in the atmosphere at a level that prevents dangerous anthropogenic interference with the climate system" (IPCC, 2005, p. 20). With this objective, Carbon Capture and Storage (CCS) technology has surfaced to address the international goal of abating greenhouse gas levels. It has been assessed, however, that it will be a combination of various technologies that will achieve the objective. Models of CCS systems have shown to be compatible with current energy infrastructures (IPCC, 2005).

CCS involves separating CO_2 from a gas stream, typically by scrubbing the gas with a chemical solvent (IPCC, 2005). The captured CO_2 is compressed to reduce its volume, stored into tanks, and transported to storage sites. CCS is mostly limited to power generation facilities and large industries that emit significant quantities of CO_2. The field of CCS is large, thus it is prudent to limit the system boundaries to the analysis being considered. Accordingly, CCS is divided into three main systems: capture, transport, and storage.

CO_2 capture consists of three main capture systems, namely: post-combustion, oxy-fuel combustion, and pre-combustion (IPCC, 2005; Puxty *et al.*, 2010). Among these, post-combustion has advantages over other systems. These capture systems incorporate different absorption/separation technologies, namely: absorption, adsorption, membranes, and cryogenics. Among these four technologies, absorption is the most developed by a reactive chemical absorption with an alkanolamine solvent (IPCC, 2005).

Absorption by chemical solvents is considered as a reliable and cost-efficient technology to reduce CO_2 emissions from power plants. To date, monoethanolamine (MEA) is the most widely used alkanolamine solvent. However, it is not without limitations and drawbacks, among which is its (1) low absorption capacity for CO_2 and (2) easy degradation in the presence of SO_2 and O_2, which has led to further studies on other possible solvents (Yeh and Bai, 1999). Ammonia (NH_3) is one among the alternative solvents being studied to remove CO_2 from flue gases. In addition, other factors such as the concentration of the solvent, the operating solvent temperature, and the absorption equipment used may affect the performance of the entire system. Varying concentrations of ammonia and MEA and different operating solvent temperatures influence the CO_2 removal efficiency and absorption capacity (Yeh and Bai, 1999). Lastly, the absorption equipment used may also have a bearing on the results obtained. Various equipment have been used to conduct studies on chemical absorption, namely: semi-continuous flow reactor (Yeh and Bai, 1999), wetted-wall column (Puxty *et al.*, 2010; Darde *et al.*, 2011; Siy and Villanueva, 2012), and a semi-batch reactor (Liu *et al.*, 2009).

In this study, CO_2 absorption will be done in the wetted-wall column fabricated and modified by Siy and Villanueva, whose study focused on the functionality and performance of the said equipment by testing two concentrations (1 and 3M) and temperatures (3 and 10°C). This paper will serve as an extension of their study by focusing on the effectiveness of NH_3 using different concentrations at a constant temperature range of 12-17°C, solvent flow rate of 100 mL/min, and simulated flue gas flow rate of 2 L/min. Since literature suggests that absorption be taken at relatively low concentration, experiments will be run to observe the possibility of such statement at local settings as well as to determine if the results will still be similar to those presented in literature. If the results are satisfactory, it can contribute to literature, the possibility of doing feasibility studies on the use of the wetted-wall column, and the use of NH_3 solvent in an industrial scale. The concentrations used were 1, 5, 10, and 15% $NH_{3(aq)}$.

OBJECTIVES

This study aims to determine the effectiveness of aqueous ammonia as an absorption solvent for CO_2 capture. Particularly, it aims to determine the effect of varying the concentration of aqueous NH_3 on CO_2 absorption in a wetted-wall column by measuring the percentage of CO_2 absorbed, the time required for the system to reach steady state, and the overall mass transfer coefficient of the system.

MATERIAL AND METHODS

All chemical reagents were prepared manually. A concentrated 12M hydrochloric acid (HCl) was diluted to 0.1M, and then standardized using a sample of reagent grade anhydrous sodium carbonate. Stock solutions of 25% and 28%(w/w) analytical grade ammonia from Techno Pharmchem Haryana (India) and Ajax Finechem Pty Ltd, respectively were also diluted such that it would only need a small amount of HCl for standardization. Afterwards, aqueous ammonia was diluted to the desired concentrations, which were also standardized with HCl. It was made sure that the prepared solvents were within ±10%(w/w) of the desired concentration.

The actual wetted-wall column used was fabricated by Siy and Villanueva (2012), shown in Figure 1. The boundary at which the solvent would pass through has a length of about 113 cm and an outer diameter of 25 mm. The outer water jacket occupies 71 cm of the overall length of the column and has an outer diameter approximately 5 cm about the circumference of the column. Connecting the column at both ends to the rich solution and lean solution collectors are 24/40

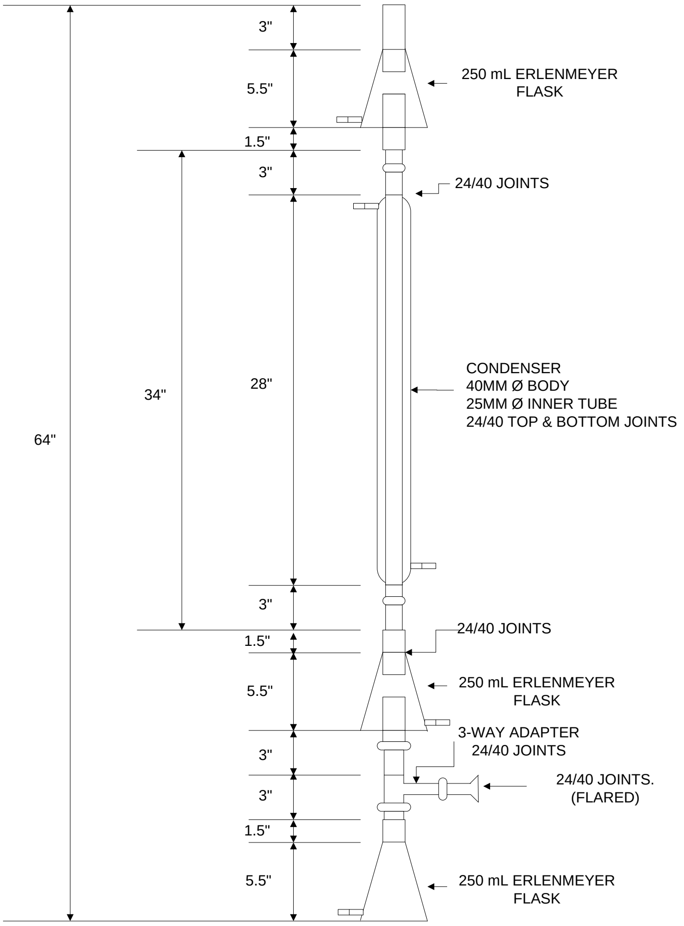


Fig. 1: Wetted-wall column

joints. Lastly, 250-mL Erlenmeyer flasks with ground joint openings served as the collectors and the surge flask.

The schematic diagram of the operation is shown in Figure 2. For every run, 5L of aqueous ammonia was placed in the overhead tank. The chiller was turned on while the three way valve was set to bypass the ammonia back to the overhead tank, allowing it to recool. A Delta A Series temperature controller was used and set to $2^{o}C$ to minimize solvent volatilization. By $3.5^{o}C$, the chilled NH_3 was re-standardized to verify its concentration. Upon operation, the flue gas was run at 1 bar and regulated to 2 L/min using a Dwyer Rate-Master Flow Meter Series RMA. Using the IMR 1050X Combustion System Analyzer, the concentrations of the entering and outgoing simulated flue gas were taken in the beginning when the valve had still not been opened for the solvent to pass to the top weir and down the column. The gas was supplied by Linde Philippines and theoretically, contains 13-18% CO_2, 3% O_2, and the rest is N_2; thus both values should be similar or in the range of 14.7 to 18 mole fraction. A container is also placed under the bottom weir to collect the rich-CO_2 solution. Afterwards, a Cole Parmer liquid flow meter was used to set the solvent's flow rate to 500 mL/min to reduce the time the solvent actually absorbs CO_2 while still in the top weir. Once the solvent starts flowing down the column, the flow rate was adjusted back to 100 mL/min where the

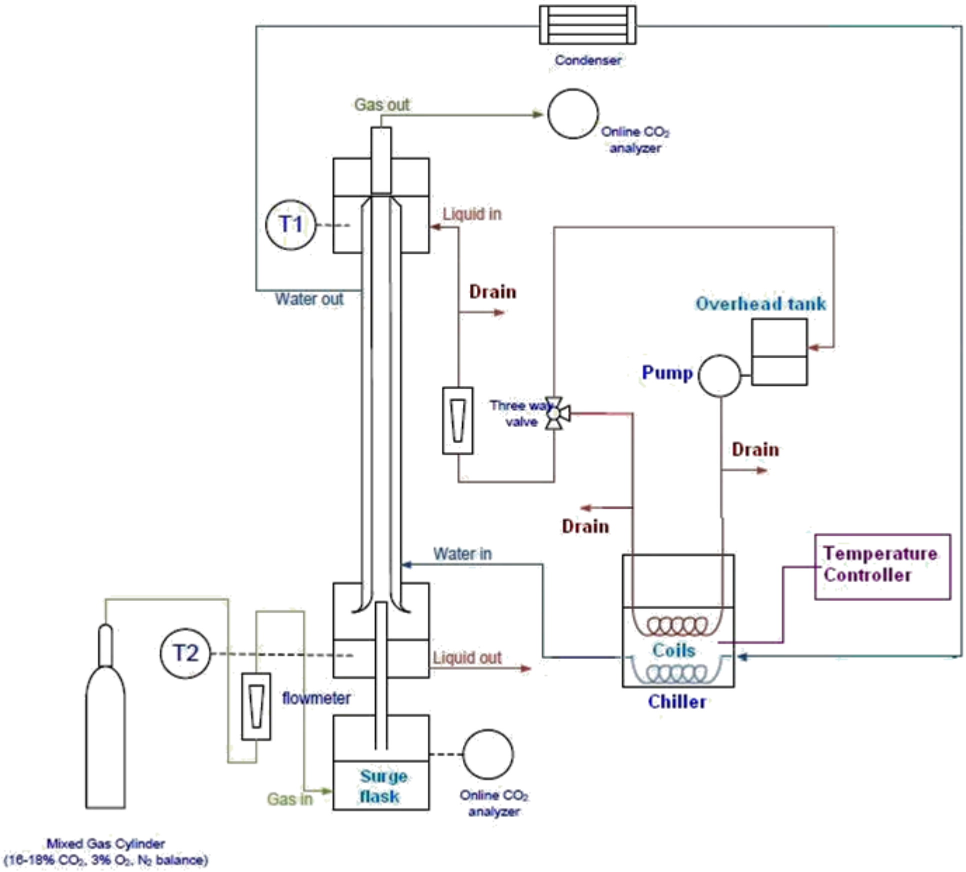


Fig. 2: Schematic process flow diagram

solution flowed uniformly along the column. For every 5 seconds until steady state time, the gas exiting the top of the column, which contains less CO_2, was analyzed. The run was ended either at this time or when the ammonia level in the overhead tank was nearing the pump level. On the other hand, the rich solution was collected and titrated with HCl to test if the NH_3 concentration decreased, indicating a reaction occurred and that NH_3 was consumed.

RESULTS AND DISCUSSION

Effect on Percent Carbon Dioxide Absorbed

Using the gas analyzer, the inlet and outlet amounts of carbon dioxide, in terms of volume percent [%(v/v)], were determined. The percent CO_2 absorbed per run as well as the average percent CO_2 absorbed per concentration were determined using Equation 1 and 2, where y_{CO_2} is the mole fraction of CO_2 in the gas.

$$\%CO_2\ absorbed = \frac{CO_{2_{in}} - CO_{2_{out}}}{CO_{2_{in}}}(100) \quad (1)$$

$$\%CO_2\ absorbed = \frac{\left(\frac{y_{CO_2}}{1-y_{CO_2}}\right)_{in} - \left(\frac{y_{CO_2}}{1-y_{CO_2}}\right)_{out}}{\left(\frac{y_{CO_2}}{1-y_{CO_2}}\right)_{in}}(100) \quad (2)$$

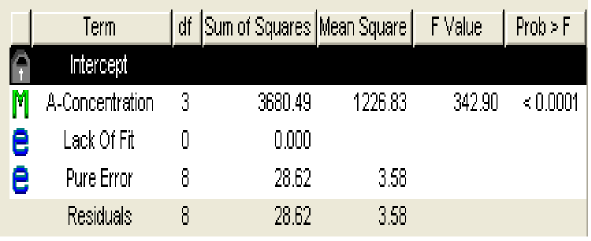
At the given operating temperature range of 12 to 17°C and varying concentrations of 1, 5, 10, and 15%(w/w), the aqueous NH_3 solution was able to absorb around 50-96% of the incoming CO_2, as summarized in Table 5.2. The lowest and highest values of $\%CO_2$ absorbed was when 1% and 15% solution was used, respectively. One of the reasons is the high solubility of ammonia in water; however, it becomes volatile when exposed to high temperature. In spite of this, it is still effective in absorbing CO_2 either as liquid or vapor. This phenomenon was observed when aqueous NH_3 was run in the system. Even when the solvent was still at the top weir and had still not flowed down the column, the outgoing CO_2 concentration was already diminishing.

From Table 1, it can be deduced that using 5% aqueous NH_3, the percentage CO_2 absorbed is 36% higher than that of 1% NH_3. However, such behavior was not followed when the solutions used have a concentration of 10% and 15% NH_3, respectively. There were still increases in the amounts of CO_2 absorbed when both solvents were used; however, the magnitude of the differences became smaller. When the concentration was changed from 5% to 10%(w/w), there was only a 6% increase in the CO_2 absorbed. On the other hand, a change of the ammonia concentration of 10% to 15%(w/w) produced an even smaller 2% difference in CO_2 absorbed. In other words, the $\%CO_2$ absorbed increased with solvent concentration; however, an insignificant difference was observed at concentrations beyond 5%(w/w).

Analysis of variance (ANOVA) using Design Expert^{TM} was used to determine the significance of the levels of concentration. With a default confidence level of 95%, the indicator of the significance is through the p-value, which should be less than 0.05. Figure 3 shows the p-value of less than 0.0001, implying that concentration has a huge effect and is a significant model on the $\%CO_2$ absorbed.

Table 1. $\%CO_2$ Absorbed at Concentrations of Aqueous NH_3 Solution

Conc. %(w/w)	Run	$\%CO_2$ Absorbed	Ave. $\%CO_2$ Absorbed
1	1	50.62	52.25
	2	55.42	
	3	50.72	
5	1	86.45	87.98
	2	87.25	
	3	90.23	
10	1	93.94	93.39
	2	92.71	
	3	93.53	
15	1	97.06	95.29
	2	94.09	
	3	94.73	



Term	df	Sum of Squares	Mean Square	F Value	Prob > F
Intercept					
A-Concentration	3	3680.49	1226.83	342.90	< 0.0001
Lack Of Fit	0	0.000			
Pure Error	8	28.62	3.58		
Residuals	8	28.62	3.58		

Fig. 3: ANOVA on $\%CO_2$ Absorbed

Figure 4 shows the actual responses in red circles, mean of the responses in black squares, and the 95% confidence interval of each concentration in vertical 'I-shaped' bars. The horizontal overlapping of the 5% and 10% bars show that their means, in terms of $\%CO_2$ absorbed, are close to one another. This however does not show which of the two is better. On the other hand, for the 1% NH_3 bar does not overlap with any of the concentrations, indicating a large gap of its mean from the others. This figure also plots the average %CO_2 absorbed with respect to the concentration used. Again, the minimum response of 52.25% absorbed CO_2 corresponded to when the NH_3 concentration used was 1% while the maximum response of 95.29% absorbed CO_2, 15 %NH_3 solution used.

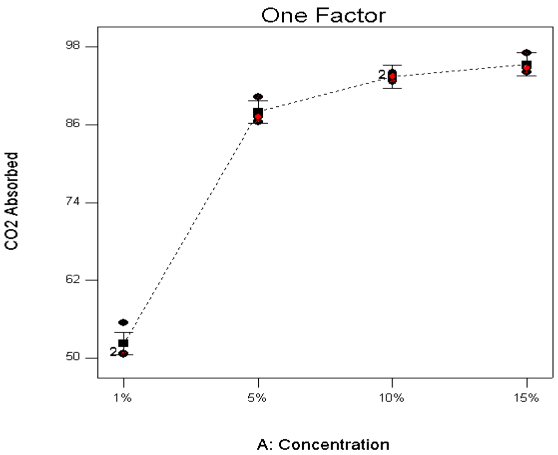


Fig. 4: Effects Graph for %CO_2 Absorbed

Effect on Steady State Time

During the runs, the time required for the system to reach steady state was recorded. This was done for each concentration, and the data are tabulated in Table 2 and shown in Figure 5. It can be seen that there is only a little difference between the steady state times for each concentration. At an aqueous NH_3 concentration of 1%, the average time for the system to achieve steady state is 91.67s. For a higher concentration of 5% NH_3, this value increased to 116.67s. However, at higher concentrations of 10 % and 15% NH_3, the average time dropped to 91.67s and 75s, respectively.

The main factors affecting the rate of chemical reaction are concentration, pressure, temperature, and nature of reactants. Despite having prepared solvent concentrations within the limit of ±10%, ambient temperature is still an uncontrollable factor. This is the reason why an operating temperature range of 12 to 17°C was set. Kinetic rate constant is primarily affected by temperature; it increases with high temperature. It is highly probable that the erratic steady state time is a result of the varying kinetic rate constants due to differences in the ambient temperature during which the experiments were done.

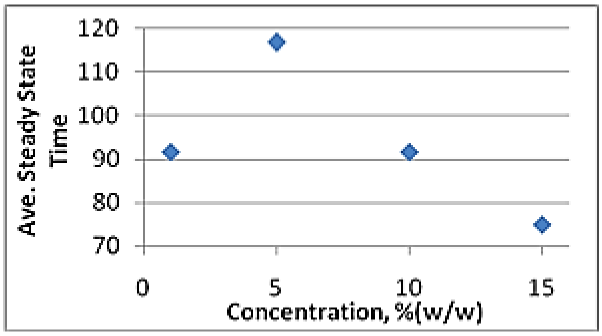


Fig. 5: Steady State Time versus Concentration

Table 2. Steady State Time at Concentrations of Aqueous NH_3 Solution

Conc. %(w/w)	Run	Steady State Time (s)	Ave. Steady State Time (s)
1	1	110	91.67
	2	90	
	3	75	
5	1	175	116.67
	2	95	
	3	80	
10	1	75	91.67
	2	85	
	3	115	
15	1	70	75.00
	2	80	
	3	75	

Effect on Overall Gas Mass Transfer Coefficient

In order to calculate for the overall mass transfer coefficient K_G, the flux must first be calculated by considering the contact area between the gas and the liquid and the amount of carbon dioxide absorbed per unit time. The following equation (Darde *et al.*, 2011) is considered:

$$N_{CO_2} = \left[\frac{\left(P_{CO_{2in}} - P_{CO_{2out}}\right)}{P}\right]\left[\frac{QV_M}{A}\right] \tag{3}$$

where:

$P_{CO2,in}$, $P_{CO2,\ out}$ = partial pressure of CO_2 in the inlet and outlet ports measured with a carbon dioxide analyzer, respectively.

Q = gas flow rate at the inlet in m^3/sec measured by a mass flow controller.

V_m = molar volume in mol/m^3.

A = contact area between the gas and the liquid in m^2.

By knowing the absorption flux at a particular partial pressure from Eq. 3 and using the logarithmic mean partial pressure of CO_2 inside the chamber as the bulk pressure, the overall mass transfer coefficient can be determined by Eq. 4, where P_{CO_2} and $P^*_{CO_2}$ are the bulk pressure and partial pressure in equilibrium with the bulk CO_2 concentration in the liquid phase, respectively, N_{CO_2} is the gaseous flux of carbon dioxide in mol/m^2·s, and K_G is expressed in mol/m^2·s·pressure unit (Darde *et al.*, 2011; Puxty *et al.*, 2010)

$$N_{CO_2} = K_G\left(P_{CO_2} - P^*{}_{CO_2}\right) \tag{4}$$

The calculated values ranged from 0.17-0.88 mmol/m^2·s·kPa on a system operating at the temperature range of 12 to 17°C and at varying concentrations of 1, 5, 10, and 15%(w/w). Equation 4 can be plotted to give N_{CO2} versus (P_{CO2}– P^*_{CO2}) to determine K_G. However, plotting only N_{CO2} versus P_{CO2} (the log mean inlet and outlet CO_2 partial pressure) yields the same K_G, thus P^*_{CO2} is not required. K_G can be determined by the linear regression of Eq. 5:

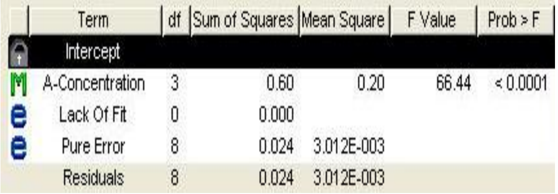
$$N_{CO_2} = K_G\left(P_{CO_2}\right) + b \tag{5}$$

Based on Table 3, increasing the concentration of the solvent resulted in a steadily increasing mass transfer coefficient. The same principles apply in that the higher the concentration of NH_3, the higher is the amount of CO_2 absorbed and thus the larger is the value of the mass transfer coefficient. Similar with the results in %CO_2 absorbed, the slope of K_G with respect to concentration is almost uniform although that of the 1%-5%(w/w) NH_3, the change is slightly higher. In particular, the K_G value for the 5% NH_3 was observed to be three times greater than the value obtained for the 1% NH_3. On the other hand, the values of K_G for aqueous ammonia concentrations between 5 to 10% only differed by 0.14 while a smaller difference of 0.10 between 10 and 15% NH_3.

Figure 6 shows a similar result with Figure 3 in that the p-value is less than 0.0001, suggesting that concentration also has a huge effect on K_G.

Table 3. Mass Transfer Coefficients at Concentrations of NH_3 Solution

Conc. %(w/w)	Run	K_G (mmol/m^2·s·kPa)	Ave. K_G (mmol/m^2·s·kPa)
1	1	0.1755	0.1843
	2	0.2018	
	3	0.1755	
5	1	0.4946	0.5311
	2	0.5165	
	3	0.5822	
10	1	0.7012	0.6758
	2	0.6480	
	3	0.6781	
15	1	0.8832	0.7746
	2	0.7068	
	3	0.7338	



Term	df	Sum of Squares	Mean Square	F Value	Prob > F
Intercept					
A-Concentration	3	0.60	0.20	66.44	< 0.0001
Lack Of Fit	0	0.000			
Pure Error	8	0.024	3.012E-003		
Residuals	8	0.024	3.012E-003		

Fig. 6: ANOVA on Mass Transfer Coefficients

Similarly, Figure 7 plots the average mass transfer coefficient with respect to the concentration used. The lowest response is obtained when the NH_3 concentration used was 1%, with a value of 0.1843 mmol/m^2·s·kPa. On the other hand, the highest observed response came from when the concentration used was 15%, with a value of 0.7746 mmol/m^2·s·kPa. The figure also illustrates that the means of 1% and 5% NH_3 are far from those of 10% and 15% NH_3 while the horizontal overlapping of the bars of 10% and 15% NH_3 indicates close values of the means. Again, the overlapping cannot determine the superiority of one concentration against the other.

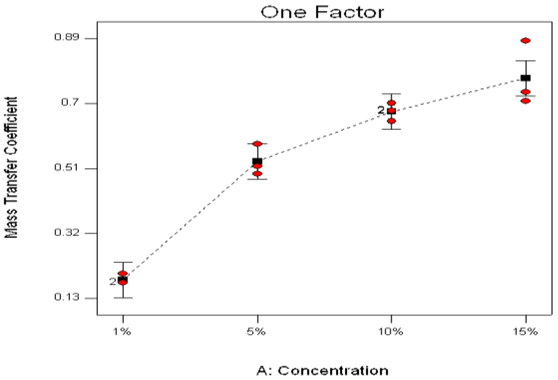


Fig. 7: Effects Graph for Overall Mass Transfer Coefficient

Comparison with Data of Siy and Villanueva (2012)

The researchers of the current study modified a few parts of the system but it is essentially the same wetted-wall column fabricated by Siy and Villanueva (2012). Siy and Villanueva tested the column using 1M and 3M aqueous NH_3 at 3°C and 10°C. The concentrations and temperatures used in this paper were quite different from those used by the past researchers. However, the 5%(w/w) solvent was computed to be 2.81M, very close to 3M, while the 12-17°C temperature range can be considered close to the 10°C temperature used by Siy and Villanueva. As seen in Table 4, the difference between the amounts of CO_2 absorbed is around 5%. This is due to the differences in the concentration as well as the temperature. Despite these differences, the K_G is almost the same because the gas flow rate used in this study was twice that of Siy and Villanueva. The data also show that even with the larger K_G value obtained, the %CO_2 absorbed was less than that of the previous study. This is due to the higher gas flow rate incorporated in this study resulting to less contact time between CO_2

and NH_3.

Table 4. Comparison of Data with Siy and Villanueva (2012)

Parameters Results	Siy and Villanueva	This Work
	3M; 10°C	5% (w/w) [2.81M]; 12-17°C
$\%CO_2$ Absorbed	97.15%	87.98%
K_G ($mmol/m^2 \cdot kPa \cdot s$)	0.4750	0.5311

Reconciliation between CO_2 and NH_3 Data

The rich solution, which is the product of the reaction between CO_2 and aqueous NH_3, was titrated to determine the amounts of NH_3 still present in the solution. Based on Table 5, there were differences in the concentration of the solvent and the rich solution. From this, it can be inferred that some amounts of the NH_3 reacted and had been consumed by CO_2 to theoretically form NH_4HCO_3.

Table 5. Concentration of the Rich Solution

Run	Solvent Conc. %(w/w)	Rich Solution Conc. %(w/w)
1	0.951	0.6904
2	0.951	0.7844
3	0.951	0.7763
1	4.647	4.6354
2	5.178	4.3367
3	4.746	4.5951
1	9.383	8.2522
2	9.383	7.9741
3	9.574	8.5211
1	15.221	12.4263
2	15.221	12.5829
3	15.221	12.4345

Based on both the entering and exiting gas and liquid streams, the results in Table 6 showed a slight discrepancy between the amount of CO_2 removed from the flue gas and the amount of CO_2 absorbed in the liquid. This may have been due to the room temperature at which the outgoing solution had been titrated. This temperature is much higher than the operating temperature of 12-17°C, which could have caused some of the NH_3 to volatilize. Furthermore, the titration of the rich solution, which used HCl as the titrant, was at best an inaccurate method in determining the concentration of NH_3 as the solution already contained other products, thereby affecting the concentration of the equivalent CO_2. Other studies such as Xu and Rochelle (2011) used total inorganic carbon (TIC) analysis to accurately determine the CO_2 content. Other factors for the discrepancy may have been that only the dominant reaction presented by Eq. 6 was considered; therefore, the by-products that were not accounted may have contributed to such error. Lastly, the temperature gradient along the column may have caused the density to vary, resulting in an increase in the flow rate of the outgoing solution and thus in the equivalent mole rate of CO_2 absorbed in the liquid.

$$CO_{2(g)} + NH_{3(g)} + H_2O_{(l)} \leftrightarrow NH_4HCO_{3(aq)} \quad (6)$$

Table 6. Discrepancy in the Amount of CO_2 Removed Expressed as the Difference between the Change in the Amount of CO_2 in the Gas Phase and that in the Liquid Phase

	Discrepancy		
Run **Conc.**	**1**	**2**	**3**
1%(w/w)	-0.00199	0.004062	0.003373
5%(w/w)	0.026929	-0.00321	0.037048
10%(w/w)	0.027115	0.005656	0.020224
15%(w/w)	-0.05239	-0.02908	-0.04312

CONCLUSIONS

This study determined that increasing the aqueous NH_3 concentration increased the CO_2 absorption in a wetted-wall column. The amount of CO_2 absorbed greatly increased from 1% to 5% aqueous NH_3 concentration, ranging from an average of 52.25% to 87.98%CO_2 absorbed, respectively. However, the same magnitude of increase was not seen beyond the range of 5% to 15% NH_3 solutions, with only an increase of 7.31% in the CO_2 absorbed; the rate of increase in %CO_2 absorbed sharply decreased at the expense of using more concentrated solvents. Thus, it is reasonable to conclude that increasing the concentration of aqueous NH_3 beyond 5% only results into diminishing returns. From 5% to 15% NH_3 solutions, the peak of the absorption has an average of 92% absorbed CO_2.

The steady state time of the system was observed to be erratic, with values increasing to 116.67s and dropping back to 91.67s and 75s at 5%, 10%, and 15% NH_3, respectively. This was mainly due to the uncontrollable ambient temperature that resulted to an operating temperature ranging from 12-17°C. It is highly probable that this resulted into a varying kinetic rate constant, leading to an erratic steady state time.

Lastly, the overall mass transfer coefficient increased with increasing aqueous NH_3 concentration as a corresponding product of an increase of the %CO_2 absorbed. Such response simply shows that the overall mass transfer coefficient is dependent on the concentration at constant temperature conditions. However, only the range of 12-17°C was maintained during the runs due to the relatively hot ambient air temperature, thus there is a possibility that the temperature may have also affected the overall mass transfer coefficient.

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