



Original Article

Effect of Column Variables on the Effective Mass Transfer Area and Efficiency in Packed Column

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Article DOI : <https://doi.org/10.37284/eaje.6.1.1625>

Publication Date: ABSTRACT

14 December 2023

Keywords:

Mass Transfer Area,
Absorption Column,
Carbon Dioxide,
Efficiency,
Solvent Superficial
Velocity

Carbon dioxide, a component gas in biogas, not only lowers its calorific value but is a greenhouse gas contributing to global warming. To increase the calorific value, the raw gas is upgraded by sequestering $[\text{CO}]_2$ in a packed absorption column using a reactive solvent. This study investigates the effect of the effective mass transfer area a_e , on the column efficiency considering a select set of column variables. These variables are the gas and solvent superficial velocities, solvent concentration and $[\text{CO}]_2$ concentration in the raw gas. The column was randomly packed using glass spheres of equal diameter. Experimental results showed that the column efficiency is a function of the effective area a_e , which is greatly influenced by the solvent superficial velocity and the solvent concentration while carbon dioxide concentration in the gas feed has least effect. From these findings, high column efficiency is attained at high ratios of solvent to gas superficial velocities due to the increased value of the effective area, created by the increase in turbulence in the liquid phase.

APA CITATION

Kemboi, M., Agullo, J. & Mbuge, D. O. (2023). Effect of Column Variables on the Effective Mass Transfer Area and Efficiency in Packed Column *East African Journal of Engineering*, 6(1), 216-227. <https://doi.org/10.37284/eaje.6.1.1625>

CHICAGO CITATION

Kemboi, Martin, Januarius Agullo and Duncan O. Mbuge. 2023. "Effect of Column Variables on the Effective Mass Transfer Area and Efficiency in Packed Column". *East African Journal of Engineering* 6 (1), 216-227. <https://doi.org/10.37284/eaje.6.1.1625>.

HARVARD CITATION

Kemboi, M., Agullo, J. & Mbuge, D. O. (2023) "Effect of Column Variables on the Effective Mass Transfer Area and Efficiency in Packed Column", *East African Journal of Engineering*, 6(1), pp. 216-227. doi: 10.37284/eaje.6.1.1625.

IEEE CITATION

M. Kemboi, J. Agullo & D. O. Mbuge "Effect of Column Variables on the Effective Mass Transfer Area and Efficiency in Packed Column" *EAJE*, vol. 6, no. 1, pp 216-227, Dec. 2023.

MLA CITATION

Kemboi, Martin, Januarius Agullo & Duncan O. Mbuge. "Effect of Column Variables on the Effective Mass Transfer Area and Efficiency in Packed Column." *East African Journal of Engineering*, Vol. 6, no. 1, Dec. 2023, pp. 216-227, doi:10.37284/eaje.6.1.1625.

INTRODUCTION

The current world insatiable energy demand is caused by the current rapid economic growth especially in the developing world with the demand not met by the dwindling fossils fuel reserves. This has given rise to the development of different energy technologies like hydropower, solar energy, wind energy, geothermal and biogas [1].

Apart from it being used as a feedstock in the production of chemicals, hydrogen and syngas, biogas is a significant source of energy which is produced from methanation of biomass through the process of anaerobic digestion [2]. The main gasses in biogas comprise of 60 – 70% combustible CH_4 and 30 – 40% noncombustible CO_2 along with traces of oxygen (0 – 1%), nitrogen (< 1%), siloxane (0 – 0.02%), halogenated hydrocarbons (< 0.6%), carbon monoxide (< 0.6%), hydrogen sulfide (0.005 – 2%) water vapor (5 – 10%) and hydrogen [3]. The raw gas is upgraded to biomethane with high methane content by sequestration of CO_2 , a recalcitrant gas that decrease the overall calorific value biogas, and all the other constituent gasses to meet the specification of the Wobbe Index [2,4]. Biomethane constitutes 95 – 99% CH_4 and 1 – 5% CO_2

Upgrading the biogas is achieved by, among others, cryogenic and membrane separations, pressure swing adsorption, carbon dioxide fixation by chemical or biological methods and CO_2 absorption in a packed column [4]. In the column, sequestration of CO_2 is by a solvent that runs co- or counter-current to the gas stream over the packing, creating turbulence as the two fluids interact. The choice of the solvent often dictates whether the absorption process is a physical or chemical an absorption based in the reactivity of the solvent and the solute molecules. The solvents include alkaline, amine, ionic solutions, or ammonia. Chemical absorption is a preferred method in upgrading biogas as it completely removes H_2S with a low operating pressure and with a high column efficiency as compared to physical absorption [1].

The use of aqueous $NaOH$ solution in absorption of CO_2 has been studied extensively since the 1940s with focus being mass transfer coefficient, the rate of absorption, film resistance and the general column performance. It is an ionic solvent with a high absorption capacity producing $NaHCO_3$ as the final product of absorption which is very soluble in aqueous solution, readily decomposing to Na_2CO_3 , H_2O and CO_2 at low temperatures. However, Na_2CO_3 is thermally stable curtailing regeneration of $NaOH$ solution [5].

In the column, transfer of solute molecules from the gas phase to the liquid phase is facilitated by the effective area for mass transfer a_e is a fraction of the total wetted area over the packing a_w [6]. The difference is made up of the dead zones whose magnitude reduces with increase in solvent superficial velocity u_x in the column. Increasing the velocity increases turbulence in the column a phenomenon that makes the effective area greater than the geometric area a_p [7,8]. Correlations have shown that the effective area is directly proportional to the density of the solvent ρ_x , and the gravitational field strength g and is inversely proportional to the surface tension of the solvent σ_x [9].

This paper investigates the influence if the hydrodynamics of the fluid and its properties of the effective area for mass transfer and its effect on CO_2 removal efficiency η .

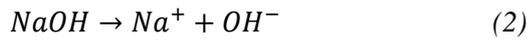
THEORETICAL FRAMEWORK

Solute-solvent reaction mechanism

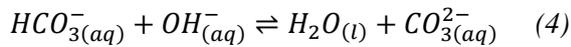
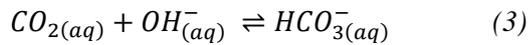
The solute molecules are partially transported from the gas phase to the liquid phase, reacting with $NaOH$ in the reaction zone, placed at a distance δx into the liquid film. It reacts with $NaOH$ by first being physically absorbed into the aqueous solution [5].



At the same time, the aqueous $NaOH$ solution in the film, being a strong alkaline solution, dissociates into sodium and hydroxide ions.

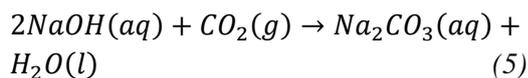


The aqueous CO_2 then reacts with the hydroxide ions to form bicarbonate and carbonate ions via Eqs. (3 and 4).



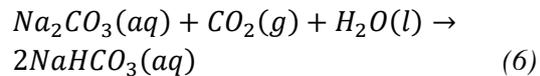
These two reactions are reversible and exothermic in the forward direction and both are characterized by high reaction rates at high pH values and since Eq (4) is instantaneous, Eq (3) is considered to be rate controlling reaction [10].

In the early stages of the absorption process, the alkalinity of the solvent makes reaction in Eq. (4) predominant increasing the concentration of the carbonate ions relative to the bicarbonate ions in the liquid phase [5]. The two reactions rapidly decrease the hydroxide concentration while increasing the concentration of carbonate ions. Initially the net irreversible reaction of the second order is given by Eq. (5) [10].

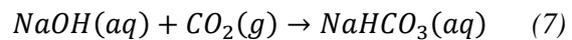


However, as the process proceeds, more carbon dioxide molecules are absorbed through the interface leading to the depletion of the hydroxyl ions thus setting the stage for the accumulation of carbonate ions via Eqs. (3 and 4). Going by the Le Chatelier's principle, an increase in carbonate

ions favours the reverse of reaction in Eq (4), leading to a favourable forward reaction in Eq. (3) [5]. The effect of this is a decrease in concentration of bicarbonate and the pH of the solution with the overall absorption process being represented by equation 6.



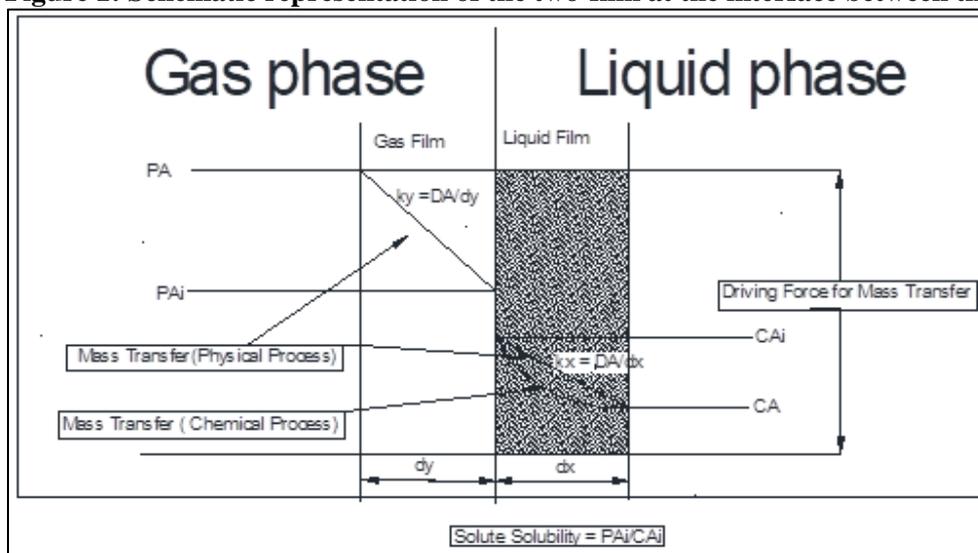
At equilibrium, some additional CO_2 may be absorbed to make up for the shortage of physically unabsorbed CO_2 in water during the reaction [5]. If aqueous sodium hydroxide solution is the limiting reactant, then the overall CO_2 absorption can be summarized as reaction in Eq. (7), which is the net reaction of Eqs. (5 and 6).



The Two-film Theory

The most widely used fundamental mass transfer model used to quantitatively describe CO_2 absorption process over a packed column is the two-film model. This model was proposed by Whitman (1923) and is the simplest theory designed for mass transfer analysis [11]. The model assumes that this gas and liquid film of equal thickness δx , exists on either side of the interface between the phases where resistance to mass transfer is localized [12]. A schematic representation of the two films is presented in *Figure 1*.

Figure 1: Schematic representation of the two-film at the interface between the phases



Due to the absence of turbulence in the two films, the flow is considered laminar and the mass transfer of the solute molecules in the films is exclusively by molecular diffusion and as the solute molecules diffuse, the experience resistance in the individual films with the overall resistance being the summation of the two resistances [13, 14, 15].

$$\frac{1}{K_y} = \frac{1}{k_y} + \frac{k_H}{\beta k_x^o} \quad (8)$$

Where, K_y is the overall mass transfer coefficient based on the gas phase, k_y is the coefficient in the gas film while k_x^o is the liquid film mass transfer coefficient for a physical absorption. k_H is the Henry's law constant for CO_2 , a constant which is the ratio of the solute's partial pressure in air to the concentration of the solute in pure water under equilibrium conditions measured at the interface.

$$k_H \left(\frac{P_A}{C_A} \right) \quad (mol/m^3.Pa) \quad (9)$$

β is the dimensionless enhancement factor for mass transfer, representing the acceleration of the process of diffusion by the chemical reaction between the solute and the solvent molecules it is the ratio of mass transfer in a chemisorption process to that of a purely physical absorption.

$$\beta = \frac{k_x}{k_x^o} \quad (10)$$

This is a measure of the contribution of the chemical reaction to the transfer of solute molecules across the interface between the phases.

In investigations done on a wetted wall column with a well-defined interfacial area, the gas phase mass transfer resistance has been found to be less than 10% of the overall resistance [14]. The overall resistance can then be assumed to be equal to the resistance in the liquid film.

$$\frac{1}{K_y} \approx \frac{k_H}{\beta k_y^o} \quad (11)$$

The reciprocal of this resistance is the overall mass transfer coefficient K_y , for the system [9, 12].

$$K_y = K_y' \approx \left(\frac{\beta k_x^o}{k_H} \right) \quad (12)$$

Mass Transfer Coefficient

The effective area for mass transfer a_e , is determined based on the chemical absorption method as proposed by Dankworth in 1970 [16]. Absorption of the solute molecules is controlled by the irreversible reaction between the solutes and solvent in Eq (7) and in the evaluation of a_e , two dimensionless numbers are considered, one is the enhancement factor β and the other is the Hatta number Ha , which is the ratio of the rate of reaction to the rate of diffusion through the interface [17].

$$Ha = \left(\frac{\sqrt{k_1[OH^-]D_{CO_2}}}{k_x} \right) \quad (13)$$

Here, k_1 and $[OH^-]$ are the reaction rate constant for the pseudo-first order reaction and the solvent concentration respectively while D_{CO_2} and k_x are the diffusivity of CO_2 molecules in the liquid film and the film's mass transfer coefficient respectively.

This parameter is used to establish the reaction regime and the position of the reaction zone within the liquid film considering the following conditions.

- If $Ha \gg 1$ and $Ha \gg \left(\frac{(D_{OH})([OH^-]^{Bulk})}{(D_{CO_2,x})(P_{CO_2}^*)} \right)$, the reaction is instantaneous and the reaction takes place at the interface.
- It $Ha \gg 1$ and $Ha \ll \left(\frac{(D_{OH})([OH^-]^{Bulk})}{(D_{CO_2,x})(P_{CO_2}^*)} \right)$, the reaction is a fast reaction and the reaction zone is at a distance δx into the liquid film.

Here, $[OH^-]^{Bulk}$ and $P_{CO_2}^*$ are the solvent concentration in the liquid bulk and carbon dioxide partial pressure at the interface.

The absorption of CO_2 molecules by aq $NaOH$ solution is a moderately fast reaction that is neither instantaneous nor intermediate but fast enough to justify exclusion of the influence of

physical absorption in the analysis of the transfer process [18]. Depending on the theoretical models used in the reaction, the Hatta number for the system lies in the region between 2 and infinity for infinite enhancement β^∞ [19, 20].

$$2 < Ha \ll \beta^\infty \tag{14}$$

It has been established that the β , is related to Ha by equation 15 [18]

$$\beta = \left(\frac{Ha}{\tanh(Ha)} \right) \tag{15}$$

The Hatta number for this system is 8 [19], making the two parameters approximately equal.

$$\beta \approx Ha \tag{16}$$

The enhancement factor is then calculated using Eq. (17).

$$\beta = \left(\frac{\sqrt{k_1[OH^-]D_{CO_2}}}{k_x^o} \right) \tag{17}$$

The apparent gas phase mass transfer coefficient K'_y , is then evaluated using Eq. (18).

$$k'_y = \left(\frac{\beta k_x^o}{k_H} \right) = \left(\frac{k_x^o}{k_H} \cdot \frac{\sqrt{k_1[OH^-]D_{CO_2,x}}}{k_x^o} \right) = \left(\frac{\sqrt{k_1[OH^-]D_{CO_2,x}}}{k_H} \right) \tag{18}$$

Evaluation of the effective area

The basis of this evaluation is the mass flux and material balance of carbon dioxide over an elemental volume in the packed section of the column [21]. If mass is conserved, the difference in mass flux in the gas stream G , entering and that leaving is equal to the mass flux of the solute molecules across the interface between the phases which is absorbed by the solvent stream L .

$$(CO_2 \text{ released by the gas stream}) = (CO_2 \text{ absorbed by the liquid stream})$$

$$G(y + dy) - Gy = LP(K_y a_e)(y - y^*)Sdz \tag{19}$$

$$-\left(\frac{G}{L}\right) dy = P(K_y a_e)(y - y^*)Sdz$$

$$dz = -\left(\frac{G}{LPS(K_y a_e)}\right) \frac{dy}{(y - y^*)} \tag{20}$$

Where, P , y , K_y and a_e are the operating pressure, CO_2 mole fraction, the overall mass transfer coefficient, and the effective area for mass transfer respectively. The equation is integrated over the following boundary conditions:

$$\text{At } z= 0, y = y_a \text{ and at } z= z_T, y = y_b$$

$$z_T = \int_0^{z_T} dz = -\left(\frac{G}{LPS(K_y a_e)}\right) \int_{y_a}^{y_b} \frac{dy}{(y - y^*)} \tag{21}$$

The absorption process is accompanied by a fast reaction with a large enhancement factor β , the reaction zone is then shifted towards the interface between the phases, making the solute concentration at the interface negligible ($y^* \approx 0$).

$$Z = \int_0^{z_T} dz = -\left(\frac{G}{LPS(K_y a_e)}\right) \int_{y_a}^{y_b} \frac{dy}{y} = -\left(\frac{G}{LPS(K_y a_e)}\right) \ln\left(\frac{y_b}{y_a}\right) \tag{22}$$

And for a unit volume of solvent operating at 1 atmosphere,

$$K_y a_e = \left(\frac{G}{ZRTS}\right) \ln\left(\frac{y_a}{y_b}\right) = \left(\frac{u_y S}{ZRTS}\right) \ln\left(\frac{y_a}{y_b}\right) \tag{23}$$

Where V, Z are the gas molar flow rate and the height of the bed respectively while y_a and y_b the mole fraction of the solute in the gas stream to and from the column respectively. On re-arrangement,

$$a_e = \left(\frac{u_y}{ZRTK_y}\right) \ln\left(\frac{y_a}{y_b}\right) = \left(\frac{u_y}{ZRTk'_y}\right) \ln\left(\frac{y_a}{y_b}\right) \tag{24}$$

Where, u_y is the gas superficial velocity. This gives the effective area for mass transfer over the packing surface. The equation is modified to evaluate the column's CO_2 removal efficiency η .

$$\eta = 1 - e^{-(a_e/A)} \tag{25}$$

Where, the constant A is the ratio $u_y/ZRTK_y$.

MATERIALS AND METHODS

Experimental Procedure

The setup is made up of an absorption column as the main unit. This column had an internal diameter of 0.23 m and was packed with glass

spheres of diameter 16 mm to a height of 0.35 m with other supporting units being a 200L solvent tank, biogas and carbon dioxide canisters, gas and liquid flow meters and a gas analyzer. The flow chart is represented in Figure 2. A solution of volume 200 L and a concentration of 0.1 gmole/L solution of aqueous sodium hydroxide was prepared by dissolving NaOH flakes into distilled water at room temperature to make 200 L of the aqueous solution This solution was pumped to the top of the column via a liquid rotameter and the flow regulated by a valve. To ensure that the column operates as a plug flow reactor, the solvent was evenly distributed over the column area S at the top of the column using a

liquid nozzle. On the other hand, the gas stream with carbon dioxide mole fraction y_a , was delivered to the bottom of the column by connecting pipes through the gas rotameter. In this stream y_a , was set by regulating the flow of biogas and pure CO_2 to the mixing chamber and confirmed by the gas analyser. The two streams interacted counter currently in the column with the solvent leaving at the bottom while the biomethane, with CO_2 mole fraction y_b , exited at the top. The process was left to run for five minutes to attain steady state operation before y_b was recorded at the sampling point 2. The operating range of the experimental factors are presented in Table 1.

Figure 2: The flow chart for biogas upgrading process used for evaluating the effective area and the column efficiency

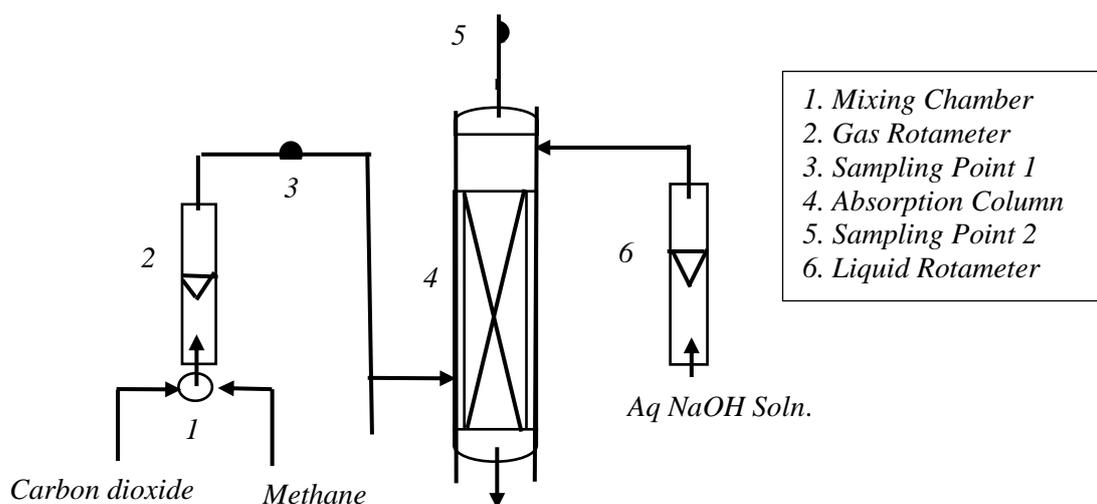


Table 1: Range of experimental factors

Variable	Low	High
$u_y (m^3/m^2 \cdot s)$	0.1	0.3
$u_x (m^3/m^2 \cdot s)$	0.1	0.3
$OH^- (gmol/L)$	0.1	1.0
$y_a (v/v)$	0.45	0.55

The influence of each of the variable were separately ascertained by varying its magnitude within the set range, keeping the other variables constant. The fractional effective area a_e/a_p , was then calculated and plotted against the variable.

RESULTS AND DISCUSSIONS

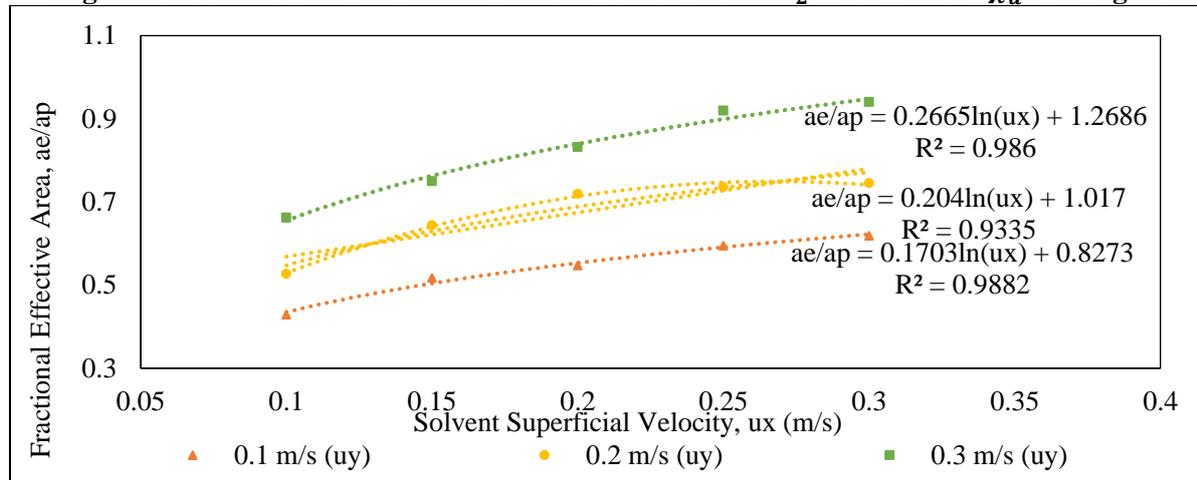
Effect of Solvent Superficial Velocity

The effect of the solvent superficial velocity u_x on the fractional effective area was determined by increasing u_x from 0.1 to 0.3 m/s. The calculated values of the fractional effective area a_e/a_p , were then plotted against u_x , at three

different gas superficial velocities u_y as shown in

Figure 3.

Figure 3: Variation of the fractional effective area a_e/a_p , with solvent superficial velocity u_x , at three gas velocities with constant solvent concentration and CO_2 mole fraction χ_a in the gas feed.



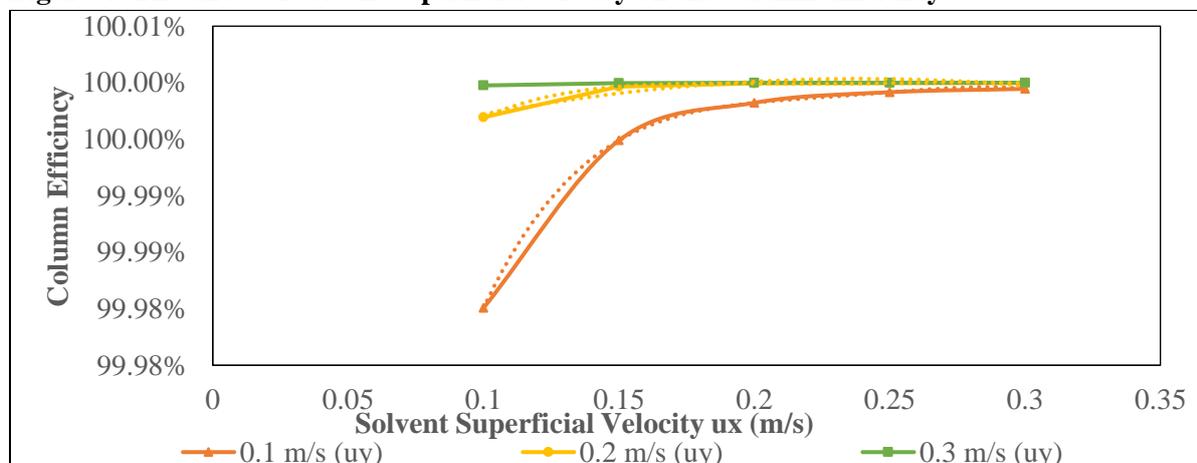
From the plots, a_e/a_p increases with increase in u_x . When u_y is maintained at 0.1 m/s, a_e/a_p increased by 44.27% and was at 41.46% and 41.91% when u_y was fixed at 0.2 and 0.3 m/s respectively. These findings suggest that the effective area a_e is greatly influenced by the solvent superficial velocity u_x .

The column efficiency η , is a function of the effective area a_e . In evaluating variation of η with u_x , the individual equations relating

a_e/a_p with u_x is substituted to Eq. (25) and the results plotted in Figure 4.

From the plots in Figure 4, η increases with u_x approaching the 100% asymptote. The increase is attributed to increased rate of surface renewal in the interface between the phases by the solvent stream thus increasing the rate of forward reaction. Higher values of the gas superficial velocity u_y , reduces the gas' residence time in the column and in the process increase the value of y_b .

Figure 4: The effect of solvent superficial velocity on the column efficiency



Effect of Gas Superficial Velocity

The effect of the gas superficial velocity u_y , on the fractional effective area and the column

efficiency η , was also investigated. While keeping all the other factors constant, u_y was increased from 0.1 to 0.3 m/s and the fractional effective

area a_e/a_p calculated over the range and plotted against u_y as in Figure 5.

The fractional effective area a_e/a_p decreases marginally with increase in u_y . The percentage reduction was 0.45, 1.02 and 1.16% when u_x was fixed at 0.1, 0.2 and 0.3 m/s respectively. These figures are kept low by the high reaction rate between the solute and solvent molecules making the effect of the reduced residence time with u_y negligible. The effect of the increasing u_y on the CO_2 removal efficiency is presented in Figure 6.

The column efficiency decreases with increase in u_y . Increasing u_y reduces the liquid-to-gas (L/G) ratio in the column, which implies that for a given amount of OH^- molecules per unit time available for the reaction, there will be an oversupply of CO_2 molecules that exits the bed unreacted increasing the value of y_b in the effluent gas. An increase in u_y reduces eddy diffusion of the solute molecules in the bulk of the gas phase making the effective area a weak function of gas superficial velocity.

Figure 5: The variation of fractional effective area with the gas superficial velocity u_y .

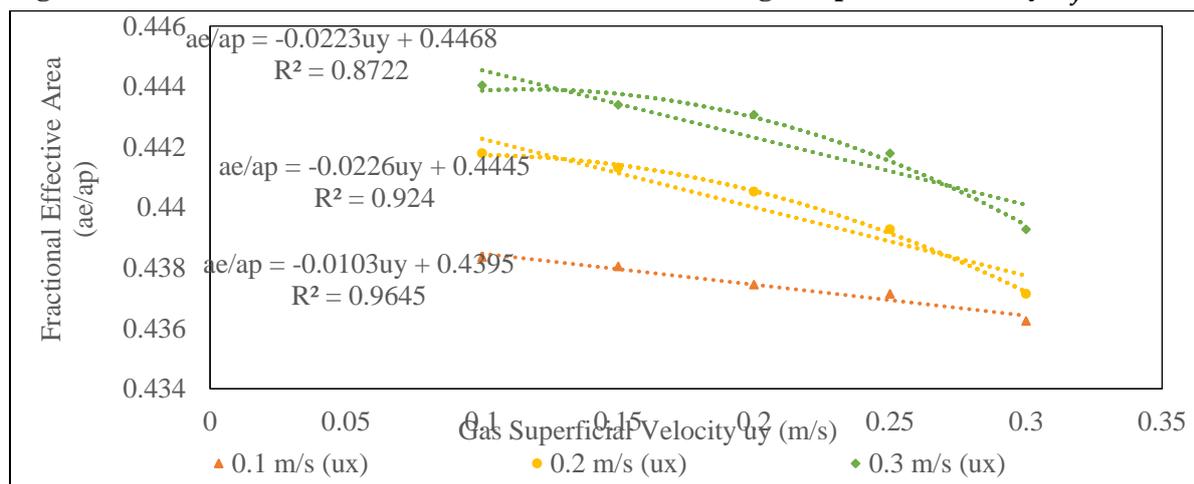
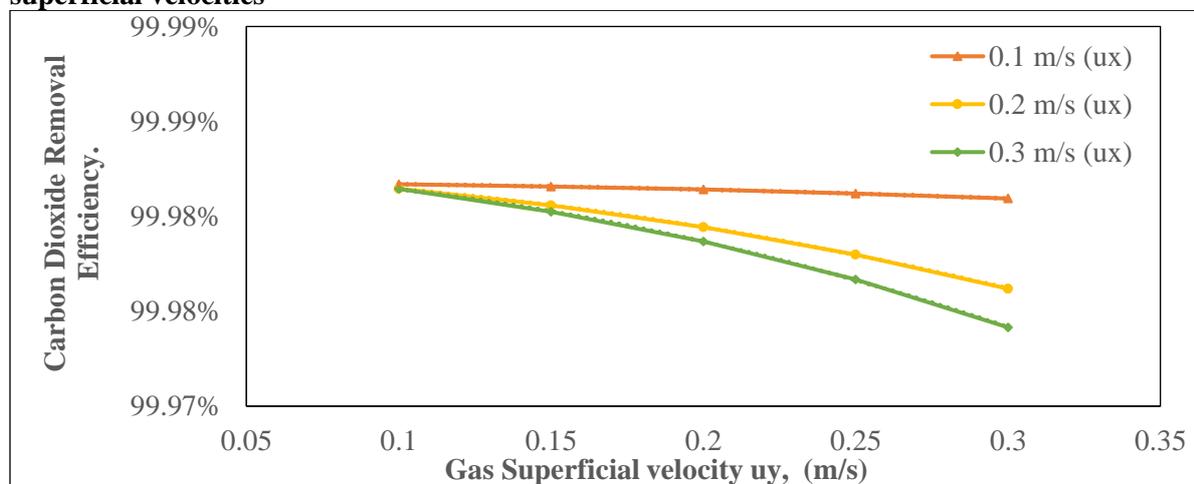


Figure 6: Variation of column efficiency with gas superficial velocity at three different solvent superficial velocities

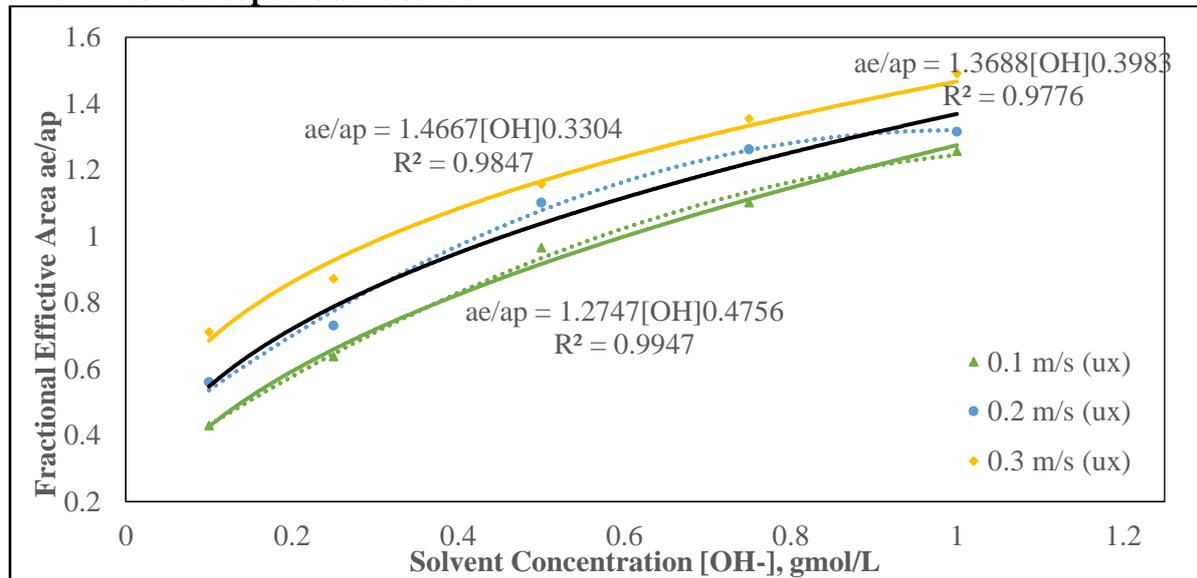


Effect of Solvent Concentration

To assess the effect of the solvent concentration of the effective area, the column was operated

with the concentration increases 0.1 to 1.0 gmol/L . while keeping the other variables constant. The effective area a_e , was then calculated over this range and plotted in Figure 7.

Figure 7: A plot of the variation of fractional effective area with the solvent concentration at different solvent superficial velocities

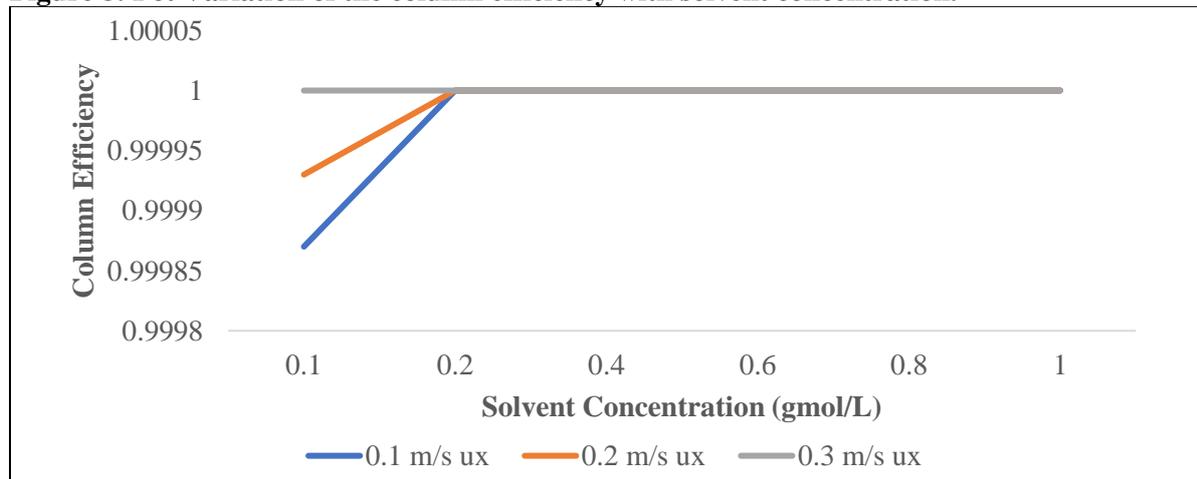


The fractional effective area a_e/a_p , increase with increase in the concentration of the solvent. The percentage increase was at 192.9%, 134.8% and 109.5% when the solvent superficial velocity is set at 0.1, 0.2 and 0.3 m/s respectively. These findings shows that the effective area is a function of the solvent concentration, however, as the concentration increases CO^{2-} molecules start to

accumulate in the reaction zone within the liquid film, slowing the reaction, and in the process slow the increase of the fractional effective area a_e/a_p . This explains the slope of the graphs in *Figure 7*.

The effect of the solvent concentration on CO_2 removal efficiency is evaluated using equation 25 and the results plotted in *Figure 8*.

Figure 8: Pot Variation of the column efficiency with solvent concentration.

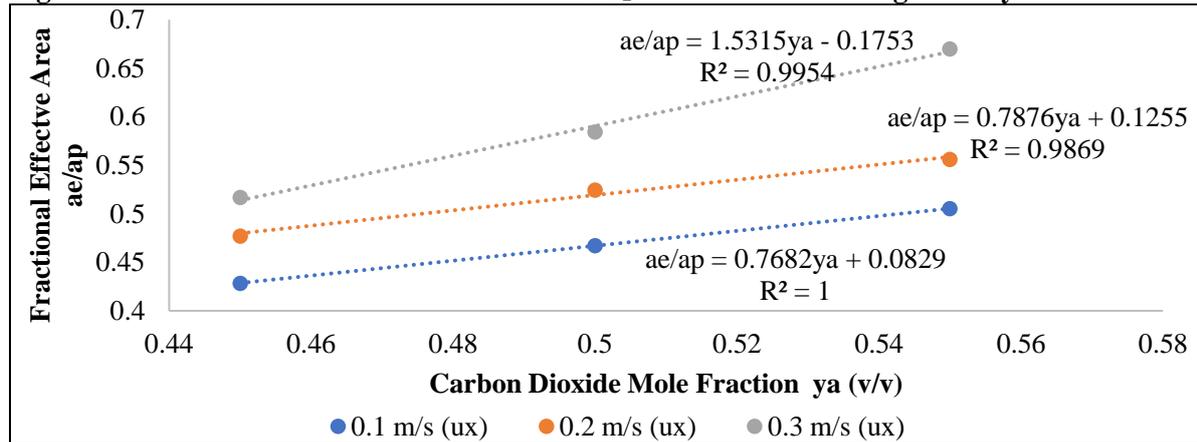


From the plots, a high solvent concentration strongly influences CO_2 removal efficiency in the column.

Effect of CO_2 molar concentration

All the variables were kept constant as CO_2 mole fraction in the gas feed y_a , was increased from 0.45 to 0.55. The fractional effective area was calculated at three solvent superficial velocities and plotted against y_a as presented in *Figure 9*.

Figure 9: Variation of the effective area with CO₂ mole fraction in the gas feed year

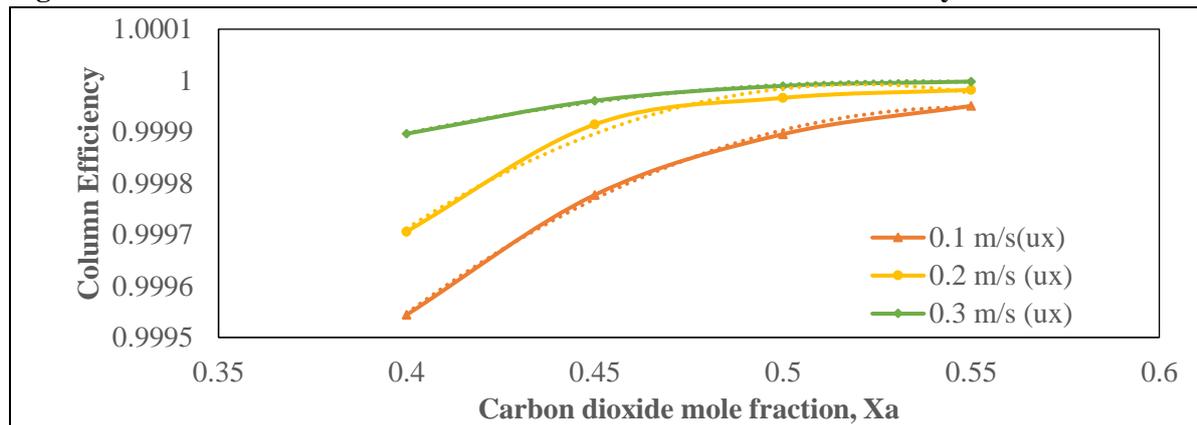


The fractional effective area increased marginally over the operating range of y_a . The increase was at 17.9, 16.5 and 29.6% when u_x was fixed at 0.1, 0.2 and 0.3 m/s respectively.

Based on the two-film model, the driving force in the gas film is the concentration gradient between the bulk of the gas and the gas-liquid interface. An increase in y_a in the gas bulk increases the concentration gradient in the gas film thus increasing the mass flux of the solute molecules across the film this has the effect of increasing the

rate of absorption. However, with a constant solvent superficial velocity, there will be an accumulation of carbonate molecules in the reaction zone within the liquid film which reduces the effective area by blocking the diffusion of the solute molecules within the liquid film to the reaction zone. At low solvent concentration, this zone is located way into the liquid film and the solute molecules have to diffuse through a liquid film of thickness δx . A plot of CO_2 removal efficiency was plotted against y_a in Figure 10.

Figure 10: The effect carbon dioxide mole fraction on the column efficiency.



Increasing CO_2 mole fraction creates molecular instability, referred as the Marangoni effects, arising from a variety of physico-chemical interactions which generates interfacial convective flows and creates forces associated with surface tension gradients. The flows thus created contributes to renewal of solute the molecules at the interface sharply increasing the effective area for mass transfer a_e and aid the chemisorption process. the column efficiency thus

increases with increase in CO_2 mole fraction in the gas feed to the column.

CONCLUSIONS AND RECOMMENDATIONS

The influence of the column parameters on the effective mass transfer coefficient and CO_2 removal efficiency was investigated in an absorption column of internal diameter 0.23 m and packed to a height of 0.35 m with glass

spheres with a diameter of $0.016m$ and operated at room temperature and pressure.

The experimental results showed that the effective area is directly proportional to the magnitude of the solvent superficial velocity u_x , the solvent concentration $[OH^-]$ and CO_2 mole fraction in the gas fed to the column, while being inversely proportional to the gas superficial velocity u_y . It is greatly influenced by the solvent to gas superficial velocities u_x/u_y and the solvent-solute concentrations $[OH^-]/y_a$ and to optimize the process of chemisorption, these ratios must be minimized.

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APPENDIX

Appendix 1: Nomenclature

k'_y	Apparent gas phase mass transfer coefficient	$moles/m^3 \cdot hr \cdot atm$
$[OH^-]$	Concentration of the solvent	$moles/m^3$
a_e/a_p	Fractional effective area for mass transfer	-
CO_2	Carbon dioxide	-
$D_{CO_2,x}$	Diffusion coefficient of CO_2 in the liquid film.	m^2/s
K_y	Overall mass transfer coefficient based on the gas film coefficient	$kg \cdot moles/m^3 \cdot hr \cdot atm$
Ha	Hatta Number	-
y_a	Carbon dioxide mole fraction in the inlet gas.	-
a_e	Effective mass transfer area	m^2/m^3
a_p	Surface area for the packing	m^2/m^3
a_w	Wetted surface area for the packing	m^2/m^3
k_1	First order reaction rate constant.	s^{-1}
k_x^o	Liquid film mass transfer coefficient for a physical process.	m/hr
k_x	Liquid film mass transfer coefficient	m/s
k_y	Gas film mass transfer coefficient.	m/s
u_x	Solvent superficial velocity	m^3/m^2s
u_y	Gas superficial velocity	m^3/m^2s
y_b	CO_2 mole fraction for the gas exiting the column	-
Z	Height of the column.	m
β^∞	Infinite enhancement factor	-
k_H	Henry's constant for the system	$mole/m^3 \cdot Pa$
P	Operating Pressure	atm
R	Universal gas constant	$J/mol \cdot K$
S	Column's base area	m^2
T	Standard temperature	K
V	Gas molar flow rate	$moles/s$
y^*	Concentration of the solute in equilibrium with the liquid	-
Subscripts		
x	Liquid phase	-
y	Gas phase	-
p	Geometric surface	m^2
w	Wetted surface	m^2
e	Effective surface	m^2