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A

Project Report

on

Experimental Investigation of CO_2 absorption in a packed bed tower

Submitted by

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In partial fulfilment of the requirements for the degree in

Bachelor of Technology in Chemical Engineering

Under the guidance of

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May, 2014

CERTIFICATE

This is certified that the work contained in the thesis entitled ***"Experimental investigation of CO_2 absorption in a packed bed tower"*** *submitted by* ***Mallikarjuna Kalika (110CH0072)****, has been carried out under my supervision and this work has not been submitted elsewhere for a degree.*

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Mallikarjuna Kalika

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ABSTRACT

The continuous and unabated emission of CO_2 from various industrial sources is a worrying sign for the future of this planet as it leads to global warming and alarmingly changing the geo-political and meteorological scenario of the world and at a faster rate. There has been an extensive research going on across various laboratories around the globe to mitigate this problem. Absorption using amine solutions, ionic liquids; adsorption on porous structural frameworks like zeolites, metal organic frameworks; natural storage (or, sequestration) under the favourable geological formations (under earth's crust, ocean floor); chemical conversion to useful products are a few of the many steps that have been taken to address the issue. Although, a few of them have overcome the technological challenges and satisfies the economic feasibility and already implemented across various industries but many of the processes are yet to come out of their infancy and still at the nascent stage (lab stage) of their development before being implemented.

In this project, CO_2 was removed from (CO_2+air) mixture using aqueous NaOH solution as the solvent in a packed bed absorption column. The column was operated counter currently using raschig ring as the packing material being randomly packed inside the column. CO_2 removal efficiency was found to satisfactory approximately varying between 50-80% under various operational conditions. The effect of solvent flow rate on removal percentage was found to be important. The height of a transfer unit (HTU) and number of transfer units (NTU) were also calculated. Finally, overall mass transfer coefficients were also calculated from the absorption data.

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NOMENCLATURE

R_O	=	Rate of absorption (moles/length^2×time)
D	=	Diffusivity of gas in solution (length^2 /time)
δ	=	Thickness of the stagnant film (length)
C	=	Concentration of gas at the liquid surface
C_O	=	Concentration of gas in the 'bulk gas'
C_A	=	Concentration of carbon dioxide at the top of the column (moles/volume)
C_{Ao}	=	Concentration of carbon dioxide at the bottom of the column (moles/volume)
P	=	Atmospheric Pressure
N_A	=	Rate of absorption of CO_2
M_A	=	Molecular weight of air
M_G	=	Molecular weight of CO_2
r	=	mass transfer rate per unit volume,
a	=	surface area per unit volume of packing
Y^o_{LM}	=	Log-mean temperature difference of CO_2.
τ	=	Average time of exposure (time)
K	=	Constant based on model (unit less)
F_g	=	Flow rate of the gas (Volume/time)
F_l	=	Flow rate of the liquid (Volume/time)
H_{OG}	=	Height of a (gas) transfer unit (HTU)
N_{OG}	=	Number of (gas) transfer units (NTU)
R'_{CO_2}	=	Amount of CO_2 absorbed in kmole/s
K'_{Ga}	=	Overall volumetric mass transfer coefficient
Q_L	=	Solvent flow rate, LPH
Q_G	=	CO_2 flow rate, LPM
Q_A	=	Air flow rate, LPM

CHAPTER 1

INTRODUCTION

Global warming which is a result of the emission of greenhouse gases has received widespread attention. Among all the greenhouse gases, Carbon Dioxide contributes more than 60% to global warming on an average because of its huge emission amount. The concentration of carbon dioxide in the atmosphere is now close to 400 ppm which is significantly greater than the pre-industrial levels of 300 ppm. To control and reduce global warming, Kyoto Protocol urged 37 industrialized nations and the European Union as well to reduce their greenhouse gas emissions to such a level that is 5.2% on average lower than that of 1990 during the period of 2008 to 2012. Copenhagen Accord also wants the global temperature increase be limited to 2°C greater than the pre-industrial level by 2100.

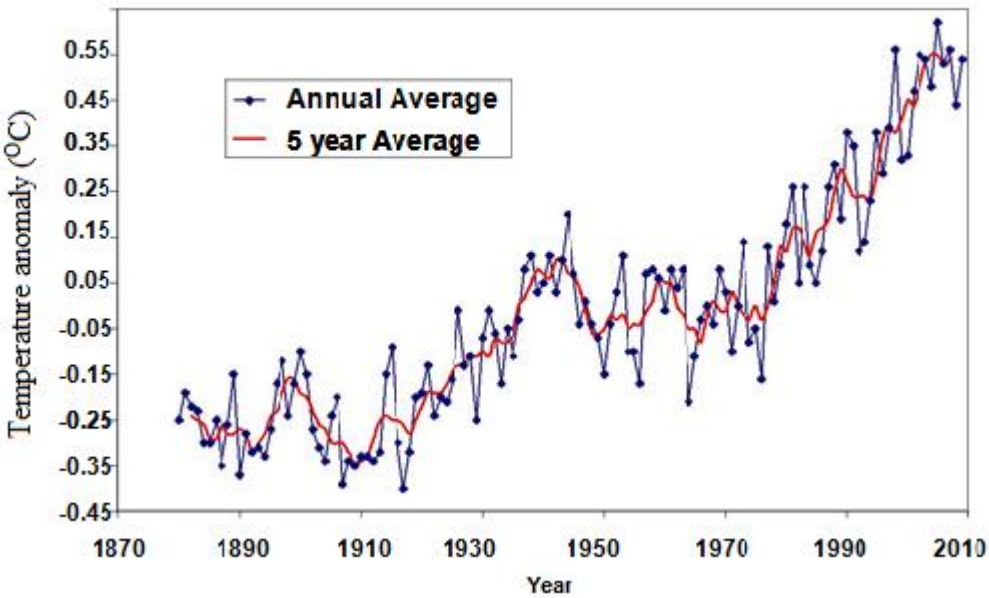
International Energy Agency (IEA) had pointed out that in order to achieve the ± 2°C goal, Carbon Dioxide capture and storage (CCS) technology is required and the contribution would be 19% by 2050. It is therefore very much essential to develop the required CCS technologies to cope with the global demand of the reduction in Carbon Dioxide emissions.

1.1 BACKGROUND OF RESEARCH

The continuous and unabated emission of CO_2 from various industrial sources is a worrying sign for the future of this planet as it leads to global warming and alarmingly changing the geo-political and meteorological scenario of the world and at a faster rate. There has been an extensive research going on across various laboratories around the globe to mitigate this problem. Absorption using amine solutions, ionic liquids; adsorption on porous structural frameworks like zeolites, metal organic frameworks; natural storage (or, sequestration) under the favourable geological formations (under earth's crust, ocean floor); chemical conversion to useful products are a few of the many steps that have been taken to address the issue. Although, a few of them have overcome the technological challenges and satisfies the economic feasibility and already implemented across various industries but many of the processes are yet to come out of their infancy and still at the nascent stage (lab stage) of their development before being implemented.

1.1.1 CARBON CAPTURE AND SEQUESTRATION

Greenhouse gas reduction and mitigation technology, particularly in relation to CO_2 is gaining increasing importance because of climate change fears. And over the last 25-30 years, there has been a rise in concern because of increasing global temperatures. Figure 1.1 depicts the plot between the increase in the difference between the global mean surface temperature and the average temperature from 1961-1990.



(Figure 1.1: Plot of global instrumental temperature anomaly (^{O}C) vs. time (year) (temperature average from 1961-1990) [1]

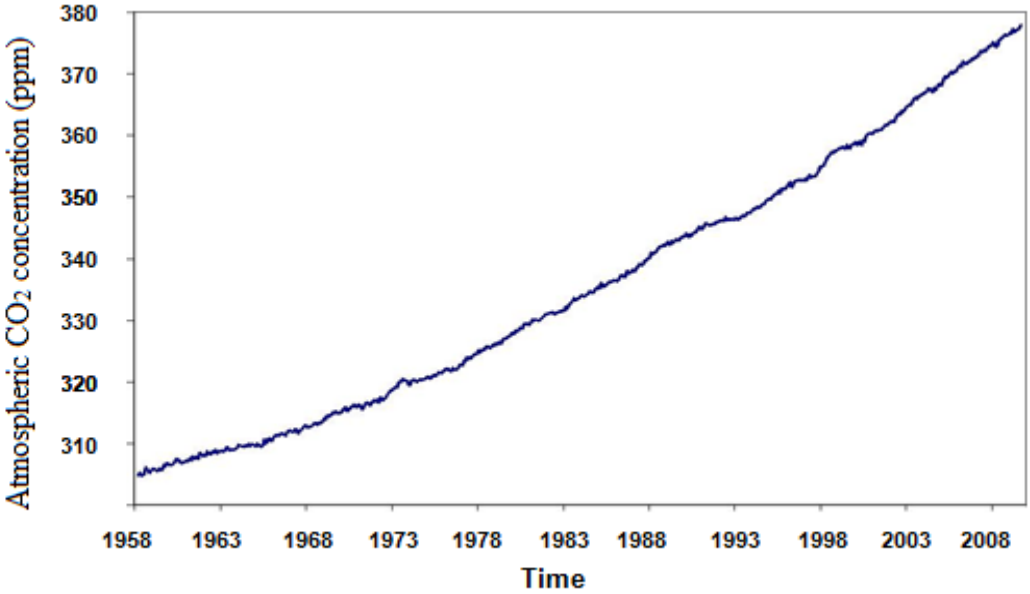


Figure 1.2: Plot of atmospheric CO_2 concentration (ppm) vs. time as measured at Mauna Loa, Hawaii [3]

Most of the increases in the global temperatures have been ascribed to the rise in CO_2 concentrations in the atmosphere due to human activities. Figure 1.2 shows the plot of atmospheric CO_2 concentration as measured at Mauna Loa Observatory, Hawaii.

The electricity producing areas – coal fired power plants as well as natural gas fired power plants - produce concentrated as well as large sources of CO_2, and on these CO_2 mitigation technologies can be tested and tried. Therefore, there is an imminent need to put into use technologies that will provide for the utilization of fossil fuels in a cleaner way and provide a route to a greener economy in the future.

Figure 1.3 depicts the expected use of different types of fuels for world electricity generation. From Figure 1.3, it is clear that coal will still thrive as the major fuel that is used for electricity generation in the coming years.

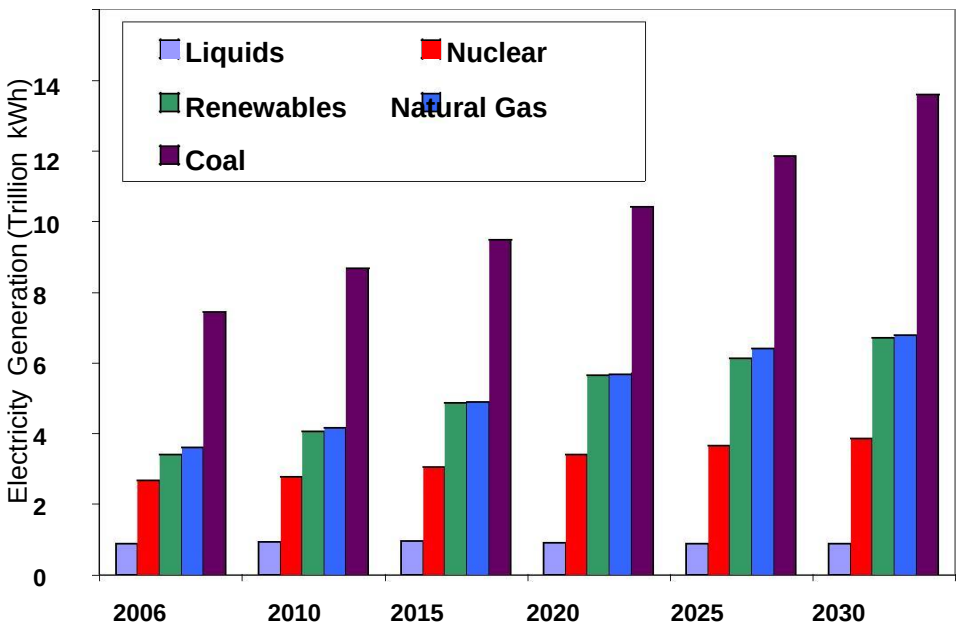


Figure 1.3: World Electricity Generation by Fuel, 2006-2030 [5-10]

1.2 BRIEF OVERVIEW OF CO_2 CAPTURE SYSTEMS

There are, broadly speaking, three types of systems for carbon dioxide capture which can be classified as

i) Post-Combustion Capture
ii) Oxy-fuel Combustion
iii) Pre-Combustion Capture

1.2.1 POST-COMBUSTION CAPTURE

Post-combustion capture is nothing but basically a downstream process which is similar to flue gas desulfurization. It largely involves the elimination of CO_2 from the flue gas which is produced as a result of the combustion of the fuel. A schematic diagram of post-combustion capture is presented in Figure 1.4.

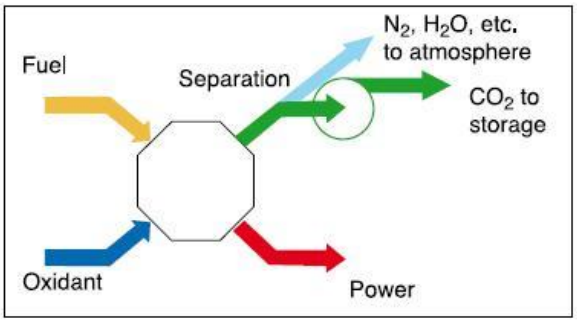


Figure 1.4: Schematic of post-combustion capture [2]

A large number of techniques exist for the post-combustion capture of CO_2 from the flue gases like physical absorption, chemical absorption, adsorption, cryogenic separation, membrane separation etc. However, among all these methods, chemical absorption is preferred.

Table 1.1: CO_2 partial pressure in flue gases of different combustion systems [2-4]

Flue Gas Source	**CO_2 concentration, %vol (dry)**	**Pressure of gas stream, MPa**	**CO_2 partial pressure, MPa**
Natural gas fired boilers	**7-10**	**0.1**	**0.007-0.01**
Gas turbines	**3-4**	**0.1**	**0.003-0.004**
Oil fired boilers	**11-13**	**0.1**	**0.011-0.013**
Coal fired boilers	**12-14**	**0.1**	**0.012-0.014**
IGCC after combustion	**12-14**	**0.1**	**0.012-0.014**
IGCC synthesis gas after gasification	**8-20**	**2-7**	**0.16-1.4 (before shift)**

1.2.1.1 CHEMICAL ABSORPTION

CO_2 is separated from the rest of the flue gas by passing the flue gas stream through a continuous scrubbing system. The continuous scrubbing system consists of an absorber and also a desorber. Absorption processes make use of the reversible chemical reaction of CO_2 with an aqueous alkaline solvent, which is generally an amine or NaOH solution. In the desorber, the CO_2 that had been absorbed is stripped from the solution and then a pure stream of CO_2 is sent for compression while at the same time the regenerated solvent is sent back to the absorber.

1.2.2 OXYFUEL COMBUSTION

The most important drawback of post-combustion capture techniques is the dilution of the flue gases due to the presence of nitrogen. This drawback can be avoided if the combustion is done/carried out in the presence of oxygen rather than air. The combustion of fossil fuels in an atmosphere of oxygen will lead to enormously high temperatures – as high as 3500°C. The temperature is controlled and moderated to a level so that the material of construction can tolerate by recycling a small fraction of the exhaust flue gases. Figure 1.5 depicts a schematic of oxy-fuel combustion.

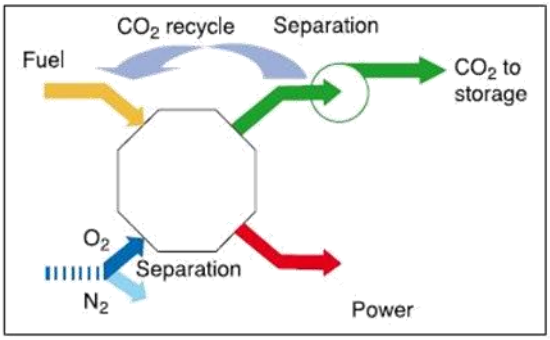


Figure 1.5: Schematic of oxy-fuel combustion [2]

1.2.3 PRE-COMBUSTION CAPTURE

In the case of pre-combustion capture, the carbon content of the fuel is somewhat reduced prior to combustion, so that when it is combusted, a stream of pure CO_2 is produced. Pre-combustion technique for de-carbonization can be used to produce hydrogen, to produce/generate electricity or both. Figure 1.6 presents a schematic of pre-combustion de-carbonization [2].

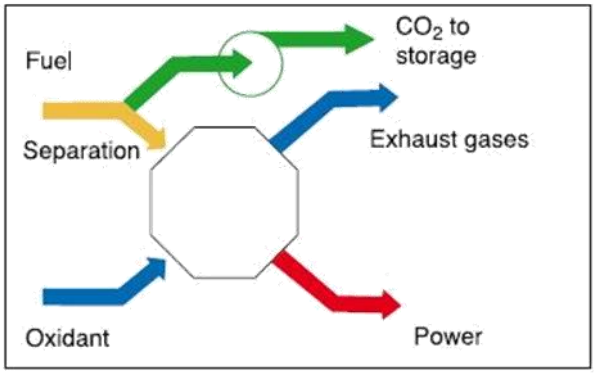


Figure 1.6: Schematic of pre-combustion de-carbonization [2]

1.3 RESEARCH OBJECTIVES

The objectives of this project work are broadly outlined below:

(a) To remove CO_2 from a mixture of (CO_2+air) using aqueous NaOH solution in a packed tower using counter current mode of operation

(b) To calculate height of a transfer unit (HTU) and number of transfer units (NTU) from the experimental data.

(c) To calculate the overall mass transfer coefficients for various operational conditions.

(d) To study the effect of solvent flow rate on % removal of CO_2.

CHAPTER 2

LITERATURE REVIEW

2.1 GAS ABSORPTION

Gas absorption takes place when a gas and a liquid come in contact and then the gas diffuses through the gas-liquid contact interface into the bulk of the contacting liquid. The driving force for such an absorption mechanism is that this absorption makes use of a concentration gradient across the gas-liquid interface. Figure 2.2 provides a schematic of the gas-liquid interface.

In this project, the absorption of carbon dioxide gas into a sodium hydroxide solution was considered. The concentration gradient will exist between the carbon dioxide gas and absorbent as long as equilibrium is not reached. Ideally, equilibrium is reached at the top of the column, utilizing the height of the column. The rate of absorption can be defined by Equation (2.1):

$$R_O = D/\delta \times (C - C_O) \qquad (2.1)$$

Where: R_O = Rate of absorption (moles/length^2 × time)
D = Diffusivity of gas in solution (length^2/time)
δ = Thickness of the stagnant film (length)
C = Concentration of gas at the liquid surface (moles/volume)
C_O = Concentration of gas in the 'bulk gas' (moles/volume)

The units of the rate of absorption (Ro) are the same as that of molar flux, and in this project, the two terms were treated synonymously. However, it is impractical to attempt to determine the stagnant film thickness since the absorption is occurring inside the packed column. This being the case, an alternate equation exists which considers the average time of exposure between the gas and the absorbent. Equation (2.3) represents this relationship:

$$R_O = K \times \sqrt{(D/\tau)} \times (C - C_O) \qquad (2.2)$$

Where: R_O = Rate of absorption (moles/length^2 × time)

D = Diffusivity of gas in solution (length^2/time)

τ = Average time of exposure (time)

C = Concentration of gas at the liquid surface (moles/volume)

C_O = Concentration of gas in the 'bulk gas' (moles/volume)

K = Constant based on model (unit less)

Although equation (2.2) represents some terms that are more easily obtained, the constant K is still difficult to determine due to its dependence on the model of the equation used. As seen in both equations (2.1) and (2.2) the rate of absorption is proportional to the concentration gradient, and both equations can be simplified to equation (2.3):

$$R_O = D/\delta \times (C - C_O) \qquad (2.3)$$

Where: k = proportionality factor (length/time)

The proportionality factor k is dependent upon the diffusivity of the gas in the liquid and the situation of the absorption, i.e. the surface area available for absorption and the flow rates of the gas and absorbent. In this project, the surface area available for absorption, the diffusivity, and the gas flow rate will remain constant, so k depends primarily on the flow rate of the absorbent.

Once equilibrium is reached between the gas and absorbent, the driving force for absorption becomes zero since the difference in concentration between the bulk gas and liquid no longer exists. The concentration gradient decreases as the gas rises in the column, constantly decreasing the driving force. This being the case, equation (2.3) can be differentiated with respect to height, z, and then integrated to determine the overall absorption within the column. Equations (2.4) and (2.5) represent the differentiation and integration, respectively.

$$R_O dz = K \times (C - C_O)\, dz \qquad (2.4)$$

$$Z = (F_g/K) \times (1/(C - C_O))\, dC \text{ integrated from } C_{A_O} \text{ to } C_A \qquad (2.5)$$

Where: F_g = Flow rate of the gas (Volume/time)

C_A = Concentration of carbon dioxide at the top of the column (moles/volume)

C_{A_O} = Concentration of carbon dioxide at the bottom of the column (moles/volume)

K = proportionality constant (area/time)

And $R_O \times dz = F_g \times dC$

Equation (2.5) assumes a constant volumetric flow rate of gas. In order for this assumption to be valid, the liquid flow must have a constant density or negligible change in density. It will be assumed in this project that the absorption of the carbon dioxide will have a negligible effect on the liquid density, so equations (2.6) and (2.7) will be applied:

F_g, entering = F_g, exiting (2.6)

F_l, entering = F_l, exiting (2.7)

Where: F_g and F_l represent the volumetric flow rates of the gas and liquid, respectively.

2.2 CHEMICAL REACTION ANALYSIS

In this project a chemical reaction between the carbon dioxide and sodium hydroxide is also considered in the absorption. The reaction is as follows:

$$CO_2\,(g) + 2NaOH\,(aq) \rightarrow Na_2CO_3\,(aq) + H_2O\,(liq)$$

The concentrations of both carbon dioxide and sodium hydroxide can be controlled in this project. The concentration of carbon dioxide will be determined using the carbon dioxide analyser, or using titration techniques.

Molarity = (Mass of NaOH)/ (MW NaOH)/ (Volume of water) (2.8)

Where: MW NaOH = 40 g/mol

Assuming this reaction takes place instantaneously and completely, the concentration of carbon dioxide at the surface of the absorbent can be assumed negligible. This being the case, C in equation (2.5) can be assumed zero, which results in the following equation (2.9):

$Z = (F_g/K) \times (1/(-C_O)\ dC$ integrated from C_{Ao} to C_A (2.9)

Using equation (2.9), K can be determined since the carbon dioxide concentrations at the top and bottom of the column are known as well as the gas flow rate and column height.

2.3 PACKED TOWERS

Packed towers are used for continuous contact of liquid and gas in both counter current and co-current flow, and are vertical columns which have been filled with packing materials of large surfaces.

The liquid is distributed over, and trickles down through the packed bed exposing a large surface to contact the gas.

Packed columns are equipment commonly found in absorption, distillation, stripping, heat exchangers and other operations, like removal of dust, mist and odours and for other purposes. Mass transfer between phases is promoted by their intimate contact through all the extent of the packed bed. The main factors involving the design of packed columns are mechanics and equipment efficiency. Among the mechanical factors one could mention liquid distributors, supports, pressure drop and capacity of the column. The factors related to column efficiency are liquid distribution and redistribution, in order to obtain the maximum area possible for liquid and vapour contact.

These columns are useful devices in the mass transfer and are available in various construction materials such as metal, plastic, porcelain, ceramic and so on. They also have good efficiency and capacity, moreover, are usually cheaper than other devices of mass transfer.

The main desirable requirements for the packing of distillation columns are: to promote a uniform distribution of gas and liquid, have large surface area (for greater contact between the liquid and vapour phase) and have an open structure, providing a low resistance to the gas flow. Packed columns are manufactured so they are able to gather, leaving small gaps without covering each other. Many types and shapes of packing can satisfactorily meet these requirements.

There are some rules which should be followed when designing a packed column:

a. The column should operate in the loading region (40 to 80% flooding), which will assure the best surface area for the maximum mass transfer efficiency;

b. The packing size (random) should not be greater than 1/8 the column diameter;

c. The packing bed is limited to 6D (Raschig rings or sells) or 12D for Pall rings. It is not recommended bed sections greater than 10m;

d. Liquid initial distribution and its redistribution at the top of each section are very important to correct liquid migration to the column walls.

A preliminary design of a packed column involves the following steps:

1. Choice of packing;
2. Column diameter estimation;
3. Mass transfer coefficients determination;
4. Pressure drop estimation;
5. Internals design.

The packing are divided in random – randomly distributed in the interior of the column – and structured – distributed in a regular geometry.

There are a variety of packings that differ in shape, size and performance that are available nowadays. These may be classified into three categories: (1) random packings/ dumped packings, (2) structured packings, and (3) grid packings.

Random packings are dumped into the shell to give the material of the packing a random orientation. On the other hand, structured packings are properly stacked and placed in the shell of the packing column so that it takes up the shape of a packed bed.

Characteristics of tower packing

Apart from the fact that the cost should be as low as possible, the packings should also preferably possess the following characteristics:

(1) A large surface area
(2) Uniform flow, both with respect to the gas and the liquid
(3) High fractional voidage
(4) The packing material should possess sufficiently high mechanical strength
(5) Fouling resistance

Types of tower packings

Tower packings are generally made up of metals, ceramics, sometimes even plastics. Focussing on the random packing materials, three generations of random packing materials have been identified:

(1) First generation random packings: Raschig ring, Berl saddle, Spiral ring
(2) Second generation random packings: Pall rings, intalox saddle
(3) Third generation random packings: Nutter ring, Norpac etc

First generation random packings: These basically include three kinds of packing: Lessing rings, rashig rings and modifications/variations of the berl saddles and raschig rings. These are, as the name suggests, packed randomly.

As far as raschig rings go, they fall under the first generation random packing category. It is the oldest packing material for packed towers that was put forth by the German chemist F. Raschig. It is nothing but a hollow cylinder with its length being equal to its diameter, their size ranging from 0.25 to 4 inches. The raschig rings are generally made of metals, plastics (HDPE) or ceramic materials (unglazed porcelain). It is probably the most rugged packing and can be used even when a severe bumping or vibrating condition exists.

Other packings that fall under this category are:

(1) Lessing ring : similar to raschig ring, but has a partition along its axis which increases its surface area
(2) Cross partition ring : two partitions as opposed to the one partition in raschig ring
(3) Ceramic spiral ring: has an internal helix that causes a whirling motion of the gas and the liquid and increases the rate of mass transfer significantly.

Berl saddles: It is actually the first modern packing that was developed in the late 1930s. It is named so because it resembles the shape of a saddle. A packed bed that has been filled with Berl saddles has a greater specific surface area (surface area per unit packed volume) and a relatively lesser voidage than the raschig rings. The pressure drop is much less in the case of Berl saddles as a result of its aerodynamic structure when compared to raschig rings. To prevent the overlapping of the surfaces of two adjacent saddles, it is provided with a rib on one surface. Berl saddles, though they provide greater capacity and performance, are more expensive, hence less popular than raschig rings.

Second generation random packing includes Intalox saddles, Pall rings, dumped intalox saddles, Metal 'flexiring', Norton 'Hy-Pak' ring, Plastic Pall ring etc.

Many industrial absorption processes are accompanied by chemical reactions. Reaction in the liquid of the absorbent component with a reagent in the absorbing liquid is especially common.

In case of a packed bed column, for the calculation of overall mass transfer coefficient, following formulae are used:

$$K_g a = N_A / (h \times P \times A \times Y^o_{LM}) \qquad (2.10)$$

Where, N_A = rate of absorption of CO_2;

h,A,P are height of the column, cross-sectional area and atmospheric pressure respectively.

Y^o_{LM} = Log-mean temperature difference of CO_2.

$$Y^o_{LM} = [(Y-Y^o)_1 - (Y-Y^o)_2] / [\ln (Y-Y^o)_1/(Y-Y^o)_2] \qquad (2.11)$$

Where Y = mole-fraction of CO_2, suffix 1 and 2 indicate the concentration at the inlet and outlet of the column.

Y^o = mole-fraction of CO_2 at equilibrium.

In case of absorption with chemical reaction (say, CO_2 absorption in NaOH solution),

Y^o = equilibrium concentration = 0 (when reaction is irreversible).

$$Y^o_{LM} = (Y_1 - Y_2) / \ln (Y_1/Y_2) \qquad (2.12)$$

2.4 ANALYSIS OPTIONS

Option 1: graphical techniques

• HETP is known

‣ HETP = (height) / (number of theoretical equilibrium stages)

‣ solve for height given number of stages

• HETP is typically found empirically & supplied by packing vendors.

Option 2: rate-based techniques

• Use mass transfer coefficients (and a few hefty assumptions)

Operating Lines

Packed absorber operating line

Solute mole balance:

$X_{in}L_{in} + yV_l = xL_l + y_{out}V_{out}$

For dilute solutions, V and Lare approximately constant:

$$y = x\,(L/V) + y_{out} - x_{in}\,(L/V) \qquad (2.13)$$

Packed stripper operating line

Solute mole balance:$xL_l + y_{in}V_{in} = x_{out}L_{out} + yV`$

For dilute solutions, V and Lare approximately constant:

$$y = x\,(L/V) + y_{in} - x_{out}(L/V) \qquad (2.14)$$

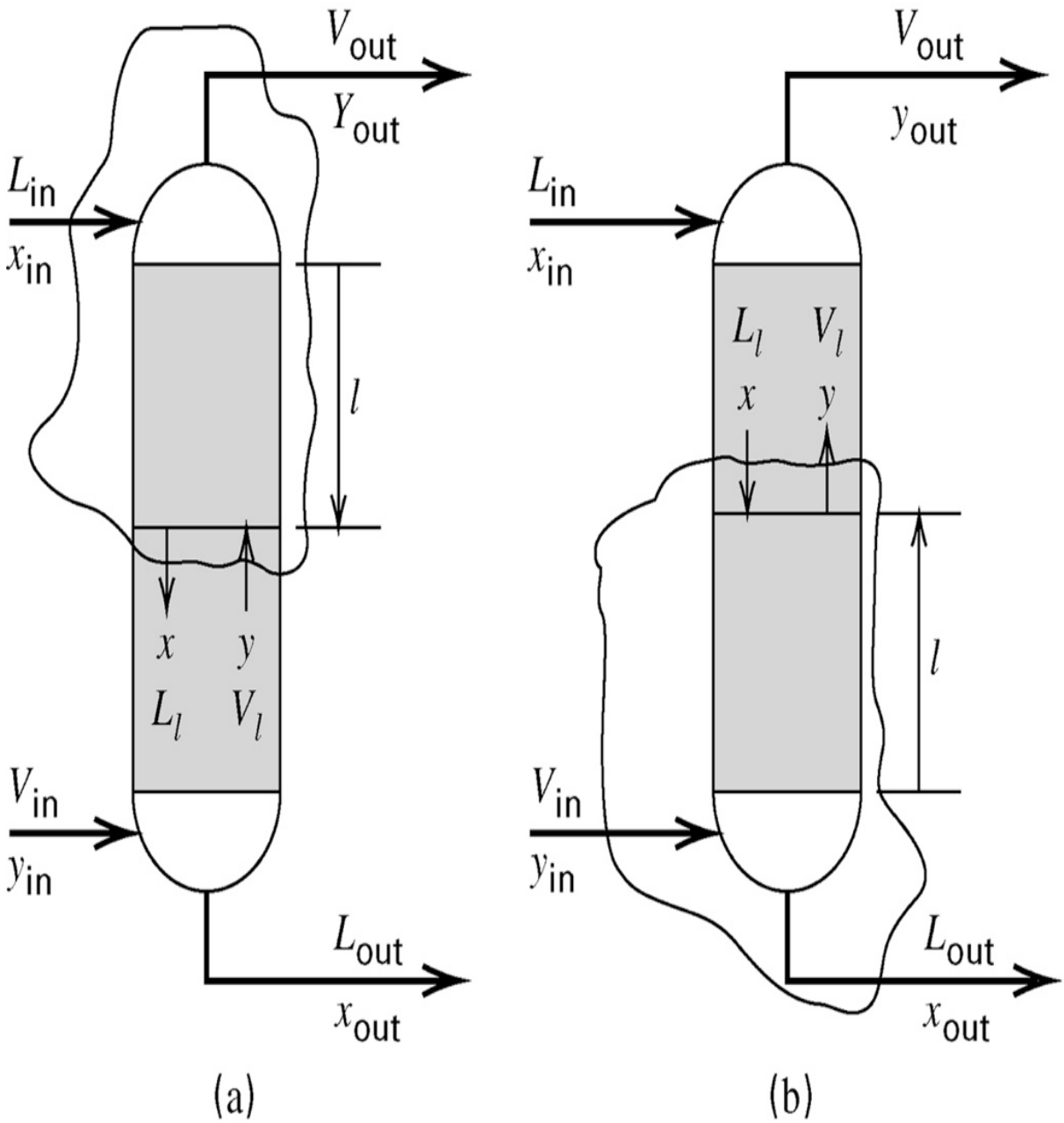


Figure 2.1: (a) Packed absorber operating line (b) Packed stripper operating line [9]

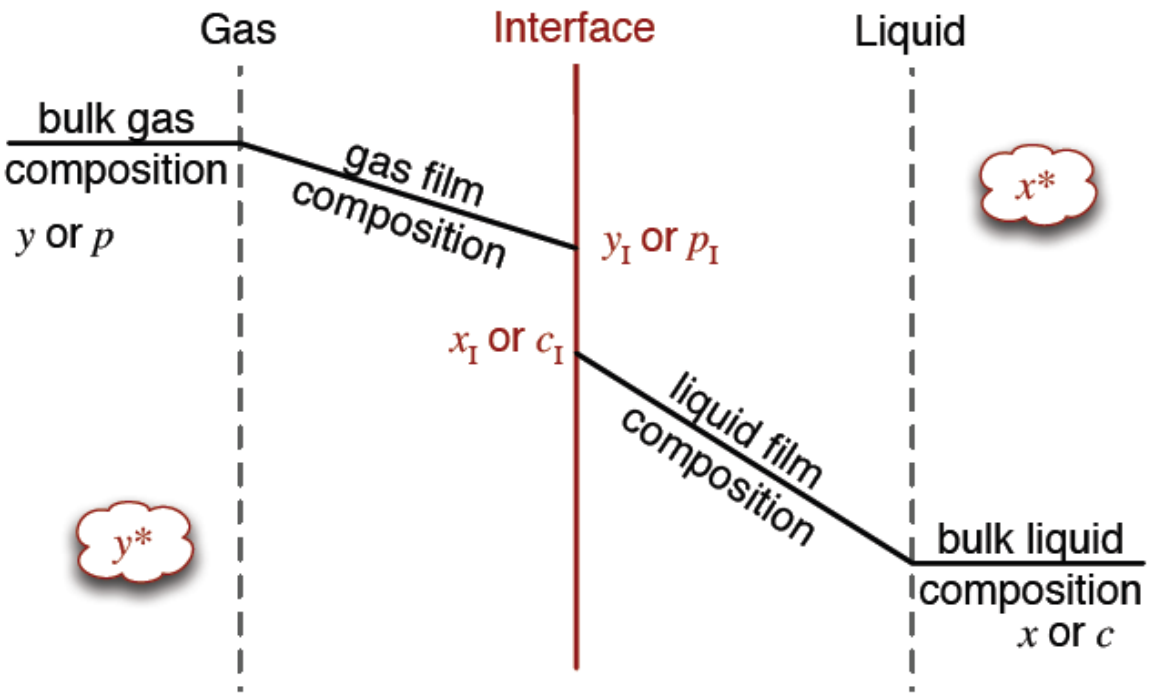


Figure 2.2: Two-film theory [9]

$J = k_y \times (y - y_1)$

Often we don't know the surface area for mass transfer from all of the packing.

$r = J_a = k_y a\,(y - y_1) = k_x a\,(x_1 - x)$

r = mass transfer rate per unit volume,

a = surface area per unit volume of packing, mol/(m^3·s)

So, $y = y_1 - (k_x a/k_y a)\,(x - x_1)$

$(k_x a/k_y a)$ = relative resistance to mass transfer between the two phases.

Overall mass transfer coefficient approach:

$r = K_y a\,(y - y^*) = K_x a\,(x^* - x)$

$1/K_y a = (1/k_y a) + (K/k_x a)$ (2.15)

$1/K_x a = (1/k_x a) + (1/K \times k_y a)$ (2.16)

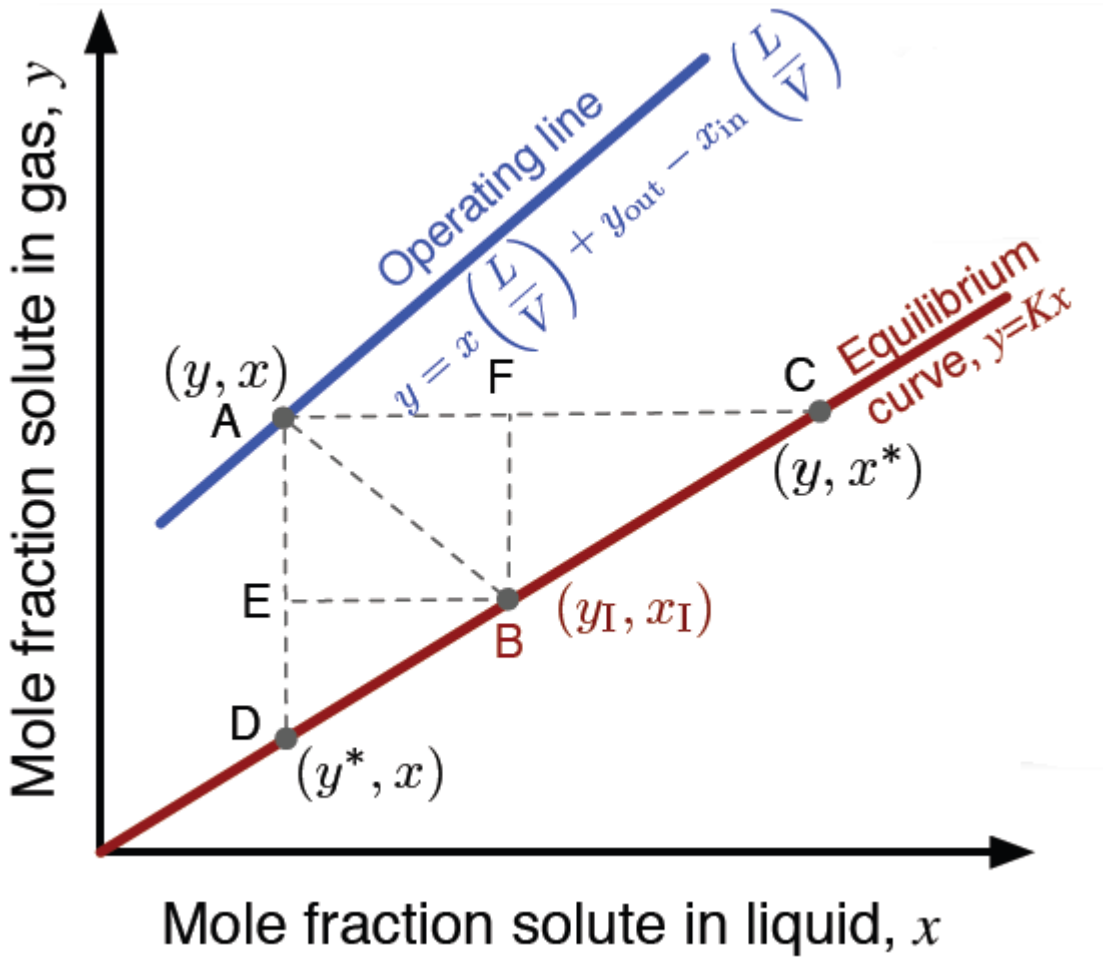


Figure 2.3: Plot showing operating line and equilibrium curve [9]

AB line: $y = y_1 - (k_x a/k_y a)(x - x_1)$

Operating line: $y = x\,(L/V) + y_{out} - x_{in}(L/V)$

From mass transfer notes on two film theory,

Liquid mole fraction, $N_A = K_x\,(x_A^* - x_{Ab})$

$x_A^* = y_{ab}/K_A$

$1/K_x = (1/k_x) + (1/K_A \times k_y)$ (2.17)

Gas mole fraction, $N_A = K_y\,(y_{Ab} - y_A^*)$

$y_A^* = x_{Ab} \times K_A$

$1/K_y = (1/k_y) + (K_A/k_x)$ (2.18)

H_{OG}-N_{OG}

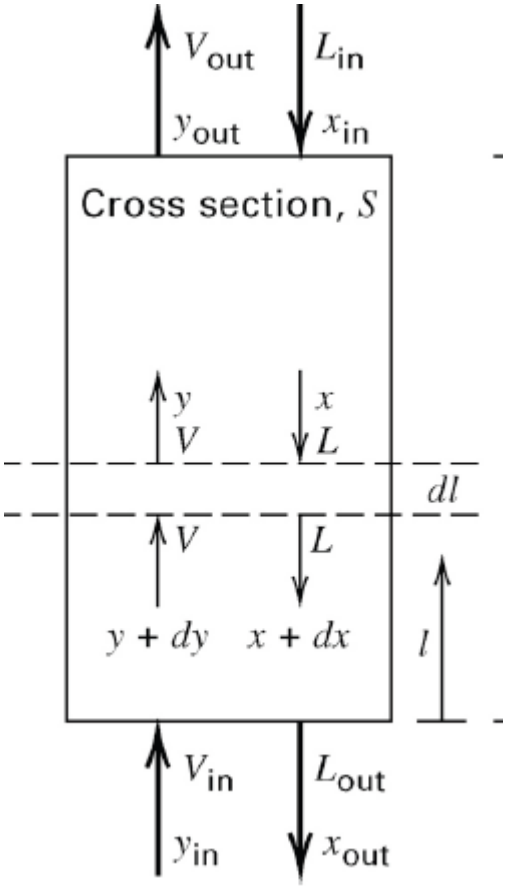


Fig 2.4: Calculation of H_{OG} and N_{OG} [9]

Material balance over dl:

Change in gas phase: V (y + dy) – Vy

Transfer to liquid phase: K_y a (y - y*) S dl

$-Vdy = K_y a (y - y^*) S\, dl$

$$\frac{K_y a S}{V}\int_0^{l_T} dl = \frac{K_y a S l_T}{V} = \int_{y_{out}}^{y_{in}} \frac{dy}{y - y_*} \quad (2.19)$$

$$l_T = \frac{V}{K_y aS} \int_{y_{out}}^{y_{in}} \frac{dy}{y - y*} \quad (2.20)$$

H_{OG} = Height of a (gas) transfer unit (HTU)

N_{OG} = Number of (gas) transfer units (NTU)

For y* = K x (constant K), and linear operating line (dilute solute),

$$N_{OG} = \frac{A}{1-A} \ln\left(\frac{(A-1)(y_{in} - K\, x_{in})}{A\,(y_{out} - K\, x_{in})} + {}^{1}/_{A} \right) \quad (2.21)$$

$$A = {}^{L}/_{KV}$$

H_{OG}, N_{OG}, N_t and HETP

H_{OG}: Height of a (gas) transfer unit (HTU)

N_{OG}: Number of (gas) transfer units (NTU) A = (L/V)

Nt: Number of theoretical stages

HETP: Height-equivalent of a theoretical plate

$$\text{HETP} = H_{OG}\, X\, \frac{A}{(1-A)}\, X \ln({}^{1}/_{A}) \quad (2.22)$$

$$N_t = N_{OG}\, X\, \frac{A}{(1-A)}\, X \ln({}^{1}/_{A}) \quad (2.23)$$

CHAPTER 3

DESCRIPTION OF MODEL/EXPERIMENTAL SET-UP

3.1 EXPERIMENTALSET-UP

The set-up basically consists of a glass column that is packed with raschig rings. Liquid feed is fed from the top of the column through a distributor. The solute gas as well as air is measured separately, followed by which they are mixed in a mixing chamber and then passed through the packing column vertically upwards and consequently absorbed in the liquid.

Rotameters are provided to measure the flow rate of each feed. Feed tank and collecting tank are given. Pressure gauge and pressure regulator are given to measure the pressure and control the pressure.

In absorption, a gas mixture is contacted with a liquid for preferential dissolution of one or more components of the gas in liquid. Such operation requires transfer of mass from gas stream to the liquid. In many industrial applications, the absorption process is accompanied by chemical reaction usually reaction in the liquid of the absorbed component with a reagent in the absorbing liquid takes place. The reagent and the product of the reaction may be soluble e.g. absorption of CO_2 into an aqueous solution of ethanol amines or other alkaline solutions.

Reaction of the absorbed solute provides higher absorption rates due to:

(1) Destruction of the absorbed solute as it forms a compound reduces the equilibrium partial pressure of the solute that in turn increases the concentration difference between the bulk phase and the interface.
(2) Increase in the liquid phase mass transfer coefficient.

3.2 UTILITIES REQUIRED

(1) Compressed air supply at 2 bar
(2) Floor drain required
(3) Laboratory glassware:

Conical flask (250ml)	=	1 no
Measuring cylinder	=	1 no
Burette (50 ml)	=	1 no

(4) Chemicals :

$BaCl_2$	=	50 gm
HCl (1 N)	=	250 ml
NaOH pellets	=	1 kg
CO_2	=	1 cylinder
Distilled water	=	10 liters
Indicator (phenolphthalein)	=	few drops

3.3 PROCEDURE

3.3.1 Starting procedure

- 2 N NaOH solution was prepared by dissolving 800 gm NaOH in 10 L distilled water.
- One burette was filled with 1 N HCl solution.
- 25 ml $BaCl_2$ was taken in a conical flask.
- All the valves were closed.
- The prepared solution was transferred into the feed tank by opening the valve V_4 and air vent valve V_5.
- Valves V_4 and V_5 were closed.
- Compressed air supply was connected by opening the valve V_6.
- Electric supply was connected to the set-up.
- The supply was started and the flow rate of NaOH solution was fixed by adjusting the valve V_1.
- The flow rate of NaOH solution was noted down.
- After overflow, the supply of CO_2 and compressed air were initiated.
- The flow rate of CO_2 was fixed by opening the valve V_3 and that of compressed air by adjusting the valve V_2.
- Flow rates of CO_2 and compressed air were noted down.
- After 10 minutes, 10 ml sample was collected in the conical flask containing 25 ml $BaCl_2$.

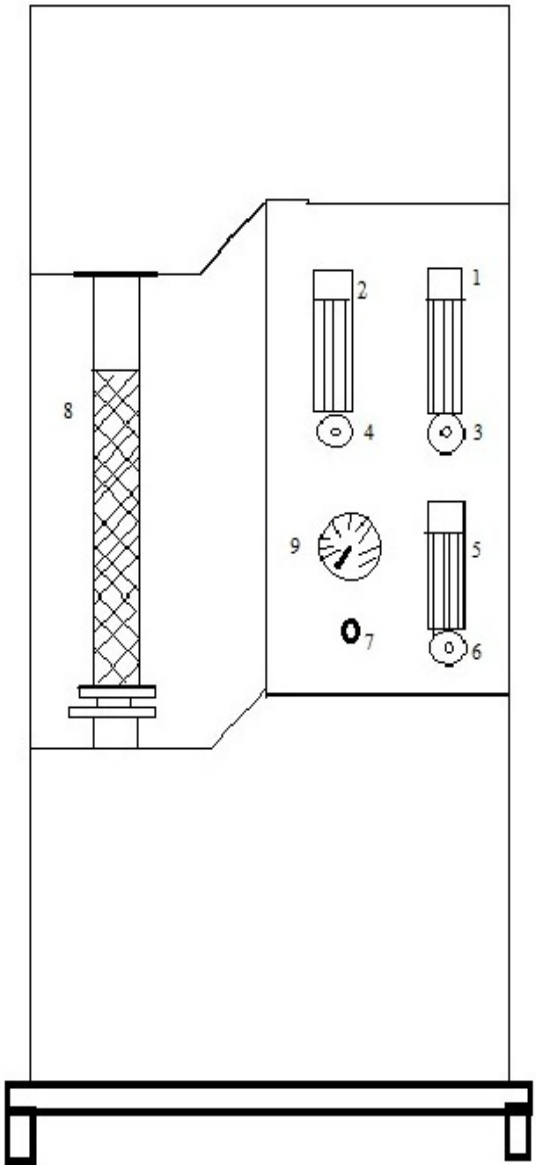
- The sample solution was titrated against 1 N HCl solution using phenolphthalein indicator.
- The experiment was repeated for different flow rates of compressed air, CO_2 and NaOH solution.

3.3.2 Closing procedure

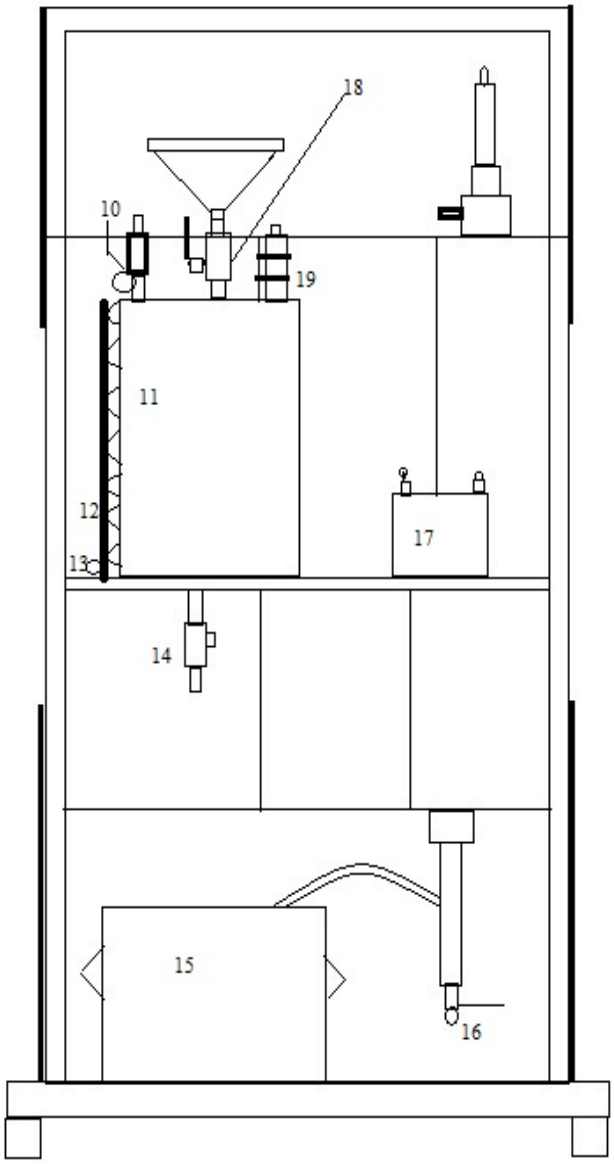
- Once the experiment was over, the supply of CO_2 and compressed air was stopped.
- The main power supply was switched OFF.
- The feed tank was drained by opening the valve V_7.
- The column was drained by opening the valve V_8.

3.4 STANDARD DATA

Column	: Borosilicate glass diameter 48 mm, length 750 mm
Packing	: Borosilicate glass Raschig rings size 8-10 mm
Feed circulation	: By compressed air
Pressure regulator	: 0-2 kg/cm^2
Pressure gauge	: Bourdon type, 0-2 kg/cm^2
Feed Tank	: Material – stainless steel 304 grade, Capacity – 20 litres
Flow measurement	: Rotameters (one each for feed, air and CO_2)
Collecting tank	: Material -Stainless steel 304 grade, Capacity 10 litres



Front View



Back View

(Fig 3.1: Block diagram of the experimental setup)

Table 3.1: Block diagram labelling

Sl No	Label
1	Rotameter for air
2	Rotameter for feed
3	Flow control valve of air
4	Flow control valve of feed
5	Rotameter for carbon dioxide
6	Flow control valve for carbon dioxide
7	Pressure regulator
8	Packed column
9	Pressure gauge
10	Air vent valve of feed tank
11	Feed tank
12	Level
13	Valve for air supply
14	Drain valve of feed tank
15	Collecting tank
16	Drain valve of packed column
17	Mixer chamber for air and carbon dioxide
18	Valve of funnel for feed inlet
19	Safety valve

(Fig 3.2: Experimental set-up)

3.5 OBSERVATIONS

Atmospheric Pressure, P	=	1 atm
Diameter of the column, d_c	=	0.048 m
Height of the column, h	=	0.750 m
Initial concentration of NaOH, C_1	=	2 mole/L
Normality of HCl used for titration, N_2	=	1gm eq /L
Molecular weight of CO_2, M_G	=	44 gm /mole
Molecular weight of air, M_A	=	29 gm /mole
Density of CO_2	=	1.977 kg/m^3
Density of air	=	1.205 kg/m^3

Table 3.2: OBSERVATION TABLE

Sl No	**Q_L (LPH)**	**Q_G (LPM)**	**Q_A (LPM)**	**V_1 (ml)**	**V_2 (ml)**
1	10	2.5	40	10	13.9
2	12.5	4	40	10	9.4
3	15	4	40	10	8.7

3.6 CALCULATIONS

For sample run 1,

$$N_1 = \frac{N_2V_2}{V_1} = \frac{1 \times 13.9}{10} = 1.39\text{geq/L}$$

$C_O = N_1$ (mole/L) = 1.39 mole/L

$$A = \frac{\pi}{4} \times {d_c}^2 = \frac{\pi}{4} \times 0.048^2 = 0.0018\, m^2$$

$$N_{abs} = \frac{(C_1 - C_0)Q_L}{2 \times 3600} = \frac{(2 - 1.39) \times 10}{2 \times 3600} = 0.000847\, mole/s$$

$$N_G = \frac{Q_G \times \rho_G}{M_G \times 60} = \frac{2.5 \times 1.997}{44 \times 60} = 0.0019\, mole/s$$

$$N_A = \frac{40 \times 1.205}{29 \times 60} = 0.028\, mole/s$$

$$y_1 = \frac{N_G}{N_G + N_A} = \frac{0.0019}{0.0019 + 0.028} = 0.0635$$

$$y_2 = \frac{N_G - N_{abs}}{N_G + N_A} = \frac{0.0019 - 0.000847}{0.0015 + 0.028} = 0.035$$

$$\%\, CO_2\, removal = \frac{N_{abs}}{N_G} \times 100 = \frac{0.000847}{0.0019} \times 100 = 44.5$$

$$\Delta\, y_{LM} = \frac{y_1 - y_2}{\ln({}^{y_1}/_{y_2})} = \frac{0.0635 - 0.035}{\ln({}^{0.0635}/_{0.035})} = 0.048$$

$$NTU = \ln({}^{y_1}/_{y_2}) = 0.63$$

$$HTU = {}^{h}/_{NTU} = \frac{0.75\, m}{0.63} = 1.19m = 3.9\, ft$$

Amount of CO2 absorbed in kmoles/m^3 of liquid $= {}^{1}/_{2} \times [(NaOH)_i - (NaOH)_O]$

$= ½ \times [2 – 1.39] = 0.305$ kmole/m^3

Liquid flow rate = 10 LPH = 2.8×10^{-6} m^3/s

Amount of CO2 absorbed in kmoles/sec =

$$R'_{CO_2} = 0.305 \times 2.8 \times 10^{-6} = 0.854 \times 10^{-6}\, kmole/s$$

Volume of packed tower = 1.357×10^{-3} m^3

Rate of CO_2 absorption per unit volume of packed tower =

$$\frac{R'_{CO_2}}{volume\ of\ packed\ tower} = 0.63 \times 10^{-3}\ {}^{kmole}/_{m^3 s}$$

Molar volume at 27° C = 24.63 L/gmole = 24.63 m^3/kmole

Volume of CO_2 absorbed = $0.854 \times 10^{-6}\ kmole/s$ ×24.63 m^3/kmole = 2.11×10^{-5} m^3/s

CO_2 flow rate at inlet = 2.5 LPM = 4.167×10^{-5} m^3/s

CO_2 flow rate at outlet = 4.167×10^{-5} m^3/s – 2.11×10^{-5} m^3/s = 2.057×10^{-5} m^3/s

Partial pressure of CO_2 at outlet,

$$P_O = \frac{2.057 \times 10^{-5}}{2.057 \times 10^{-5} + 6.67 \times 10^{-4}}\ X\ 1\ atm = 0.030\ atm$$

Partial pressure of CO_2 at inlet,

$$P_I = \frac{4.167 \times 10^{-5}}{4.167 \times 10^{-5} + 6.67 \times 10^{-4}} = 0.06$$

$$\Delta P_L = \frac{P_1 - P_0}{\ln({}^{P_1}/_{P_0})} = 0.043\ atm$$

Overall volumetric mass transfer coefficient:

$$K'_{Ga} = \frac{N_A}{Z\ A_C P_T \Delta y} = \frac{N_A}{Z\ A_C\ \Delta P}$$

$$K'_{Ga} = 0.63\ X\ 10^{-3} \times 3600 \times \frac{1}{0.043}$$

$$K'_{Ga} = 52.7\ {}^{kmole}/_{hr - m^3}$$

For sample run 2,

$$N_1 = \frac{N_2 V_2}{V_1} = \frac{1 \times 9.4}{10} = 0.94 \text{ geq/L}$$

$C_O = N_1$ (mole/L) = 0.94 mole/L

$$A = \frac{\pi}{4} \times {d_c}^2 = \frac{\pi}{4} \times 0.048^2 = 0.0018\, m^2$$

$$N_{abs} = \frac{(C_1 - C_0)Q_L}{2 \times 3600} = \frac{(2 - 0.94) \times 12.5}{2 \times 3600} = 0.00184\, mole/s$$

$$N_G = \frac{Q_G \times \rho_G}{M_G \times 60} = \frac{4 \times 1.997}{44 \times 60} = 0.003\, mole/s$$

$$N_A = \frac{40 \times 1.205}{29 \times 60} = 0.028\, mole/s$$

$$y_1 = \frac{N_G}{N_G + N_A} = \frac{0.003}{0.003 + 0.028} = 0.0968$$

$$y_2 = \frac{N_G - N_{abs}}{N_G + N_A} = \frac{0.003 - 0.00184}{0.003 + 0.028} = 0.0374$$

$$\%\, CO_2\, removal = \frac{N_{abs}}{N_G} \times 100 = \frac{0.00184}{0.003} \times 100 = 61.33$$

$$\Delta\, y_{LM} = \frac{y_1 - y_2}{\ln({}^{y_1}/_{y_2})} = \frac{0.0968 - 0.0374}{\ln({}^{0.0968}/_{0.0374})} = 0.062$$

$$NTU = \ln\left({}^{y_1}/_{y_2}\right) = 0.95$$

$$HTU = {}^{h}/_{NTU} = \frac{0.75\, m}{0.95} = 0.79m = 2.6\, ft$$

Amount of CO2 absorbed in kmoles/m^3 of liquid = ${}^{1}/_{2} \times [(NaOH)_i - (NaOH)_O]$

= ½ × [2 - 0.94] = 0.53

Liquid flow rate = 12.5 LPH = 3.47 × 10^{-6} m^3/s

Amount of CO2 absorbed in kmoles/sec =

$$R'_{CO_2} = 0.53\, X\, 3.47 \times 10^{-6} = 1.839 \times 10^{-6}\, kmole/s$$

Volume of packed tower = 1.357 × 10^{-3} m^3

Rate of CO_2 absorption per unit volume of packed tower =

$$\frac{R'_{CO_2}}{volume\ of\ packed\ tower} = 1.355 \times 10^{-3}\ {}^{kmole}/_{m^3 s}$$

Molar volume at 27° C = 24.63 L/gmole = 24.63 m^3/kmole

Volume of CO_2 absorbed = $1.839 \times 10^{-6}\ kmole/s$ ×24.63 m^3/kmole = 4.53×10^{-5} m^3/s

CO_2 flow rate at inlet = 4 LPM = 6.67×10^{-5} m^3/s

CO_2 flow rate at outlet = 6.67×10^{-5} m^3/s - 4.53×10^{-5} m^3/s = 2.14×10^{-5} m^3/s

Partial pressure of CO_2 at outlet,

$$P_O = \frac{2.14 \times 10^{-5}}{2.14 \times 10^{-5} + 6.67 \times 10^{-4}} \times 1\ atm = 0.031\ atm$$

Partial pressure of CO_2 at inlet,

$$P_I = \frac{6.67 \times 10^{-5}}{6.67 \times 10^{-5} + 6.67 \times 10^{-4}} = 0.0912$$

$$\Delta P_L = \frac{P_1 - P_0}{\ln({}^{P_1}/_{P_0})} = 0.0558\ atm$$

Overall volumetric mass transfer coefficient:

$$K'_{Ga} = \frac{N_A}{Z\ A_C P_T \Delta y} = \frac{N_A}{Z\ A_C\ \Delta P}$$

$$K'_{Ga} = 1.355 \times 10^{-3} \times 3600 \times \frac{1}{0.0558}$$

$$K'_{Ga} = 87.4\ {}^{kmole}/_{hr - m^3}$$

For sample run 3,

$$N_1 = \frac{N_2 V_2}{V_1} = \frac{1\ X\ 8.7}{10} = 0.87 \text{ geq/L}$$

$C_O = N_1$ (mole/L) = 0.87 mole/L

$$A = \frac{\pi}{4} {d_c}^2 = \frac{\pi}{4} \times 0.048^2 = 0.0018\ m^2$$

$$N_{abs} = \frac{(C_1 - C_0) Q_L}{2 \times 3600} = \frac{(2 - 0.87) \times 15}{2 \times 3600} = 0.00235\ mole/s$$

$$N_G = \frac{Q_G \times \rho_G}{M_G \times 60} = \frac{4 \times 1.997}{44 \times 60} = 0.003\ mole/s$$

$$N_A = \frac{40 \times 1.205}{29 \times 60} = 0.028\, mole/s$$

$$y_1 = \frac{N_G}{N_G + N_A} = \frac{0.003}{0.003 + 0.028} = 0.0968$$

$$y_2 = \frac{N_G - N_{abs}}{N_G + N_A} = \frac{0.003 - 0.00235}{0.003 + 0.028} = 0.021$$

$$\%\, CO_2\, removal = \frac{N_{abs}}{N_G} \times 100 = \frac{0.00235}{0.003} \times 100 = 78.33$$

$$\Delta\, y_{LM} = \frac{y_1 - y_2}{\ln(y_1/y_2)} = \frac{0.0968 - 0.021}{\ln(0.0968/0.021)} = 0.05$$

$$NTU = \ln(y_1/y_2) = 1.4$$

$$HTU = h/NTU = \frac{0.75\, m}{1.4} = 0.53m = 1.75\, ft$$

Amount of CO2 absorbed in kmoles/m^3 of liquid $= 1/2 \times [(NaOH)_i - (NaOH)_O]$

= ½ × [2 - 0.87] = 0.565

Liquid flow rate = 15 LPH = 4.167×10^{-6} m^3/s

Amount of CO2 absorbed in kmoles/sec =

$$R'_{CO_2} = 0.565 \times 4.167 \times 10^{-6} = 2.35 \times 10^{-6}\, kmole/s$$

Volume of packed tower = 1.357×10^{-3} m^3

Rate of CO_2 absorption per unit volume of packed tower =

$$\frac{R'_{CO_2}}{volume\ of\ packed\ tower} = 1.735 \times 10^{-3}\, kmole/m^3 s$$

Molar volume at 27° C = 24.63 L/gmole = 24.63 m^3/kmole

Volume of CO_2 absorbed = $2.35 \times 10^{-6}\, kmole/s$ ×24.63 m^3/kmole = 5.788×10^{-5} m^3/s

CO_2 flow rate at inlet = 4 LPM = 6.67×10^{-5} m^3/s

CO_2 flow rate at outlet = 6.67×10^{-5} m^3/s – 5.788×10^{-5} m^3/s = 0.882×10^{-5} m^3/s

Partial pressure of CO_2 at outlet,

$$P_O = \frac{0.882 \times 10^{-5}}{0.882 \times 10^{-5} + 6.67 \times 10^{-4}} \times 1\ atm = 0.013\ atm$$

Partial pressure of CO_2 at inlet,

$$P_I = \frac{6.67 \times 10^{-5}}{6.67 \times 10^{-5} + 6.67 \times 10^{-4}} = 0.0912$$

$$\Delta P_L = \frac{P_1 - P_0}{\ln({}^{P_1}/_{P_0})} = 0.04\ atm$$

Overall volumetric mass transfer coefficient:

$$K'_{Ga} = \frac{N_A}{Z\ A_C P_T \Delta y} = \frac{N_A}{Z\ A_C\ \Delta P}$$

$$K'_{Ga} = 1.735\ X\ 10^{-3} \times 3600 \times \frac{1}{0.04}$$

$$K'_{Ga} = 153.15\ {}^{kmole}/_{hr - m^3}$$

CHAPTER 4

RESULTS AND DISCUSSIONS

The experimental work was carried out as per the given standard procedure, and the observations were collected and tabulated. The final results, after all the calculations were done, have been tabulated below.

Table 4.1: Results

Sl No	Q_L (LPH)	Q_G (LPM)	Q_A (LPM)	HTU (ft)	NTU	% CO_2 removal	K'_{Ga} (kmole/ h-m^3)
1	10	2.5	40	3.9	0.63	44.5	52.7
2	12.5	4	40	2.6	0.95	61.33	87.4
3	15	4	40	1.75	1.4	78.33	153.15

Graphs showing the variation of % CO_2 removal, HTU, NTU and overall mass transfer coefficient were plotted against the flow rate of the liquid feed i.e. NaOH solution, since it can be easily seen from Table 4.1 that the flow rate of NaOH solution has a greater impact on all the aforesaid parameters.

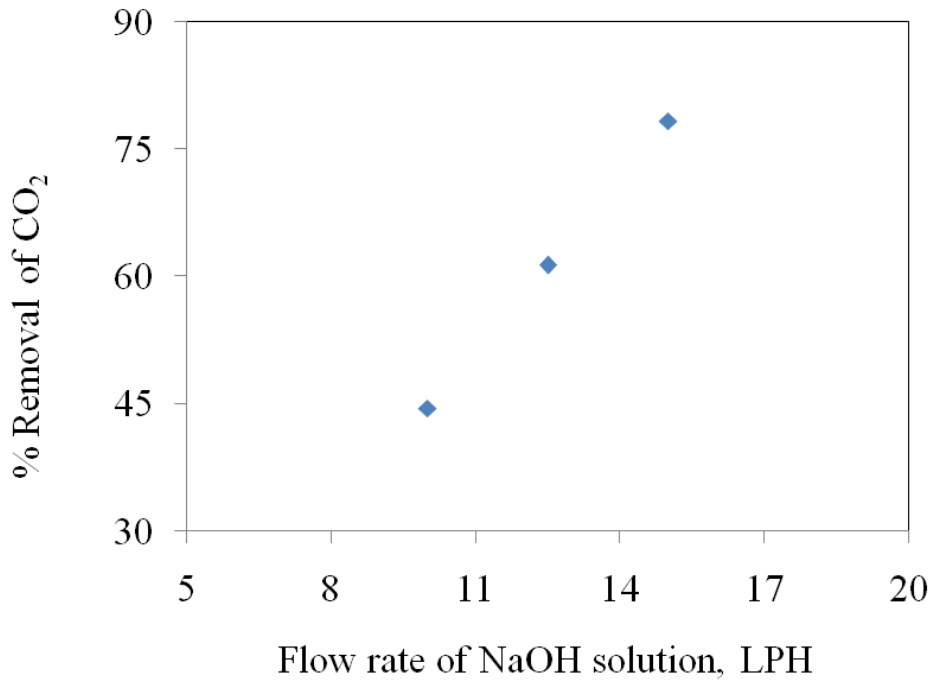


Fig 3.3: Plot showing variation of % CO_2 removal with the flow rate of NaOH solution

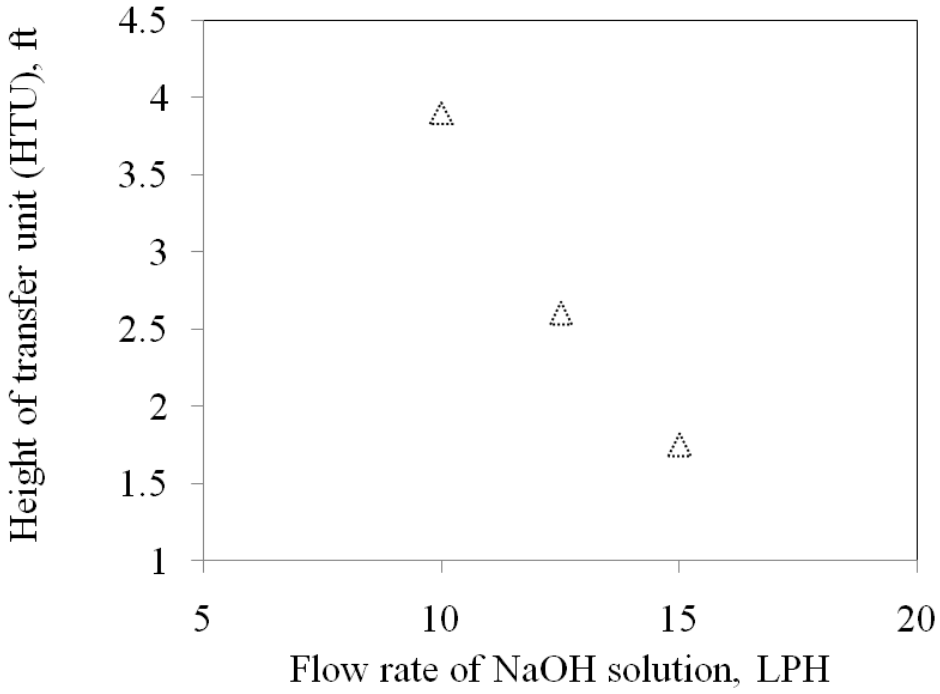


Fig 3.4: Plot showing variation of HTU with the flow rate of NaOH solution

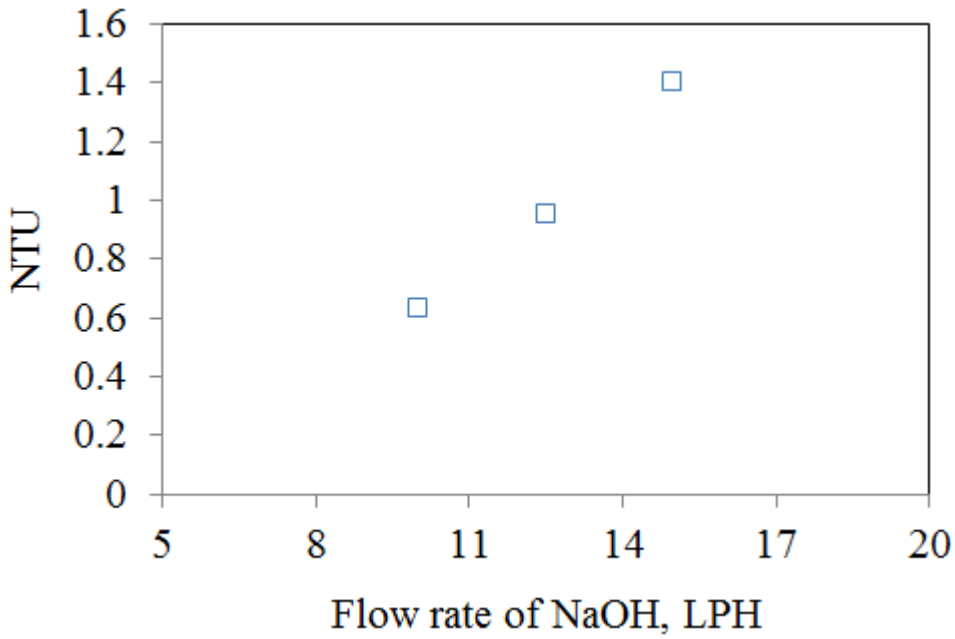


Fig 3.5: Plot showing variation of NTU with the flow rate of NaOH solution

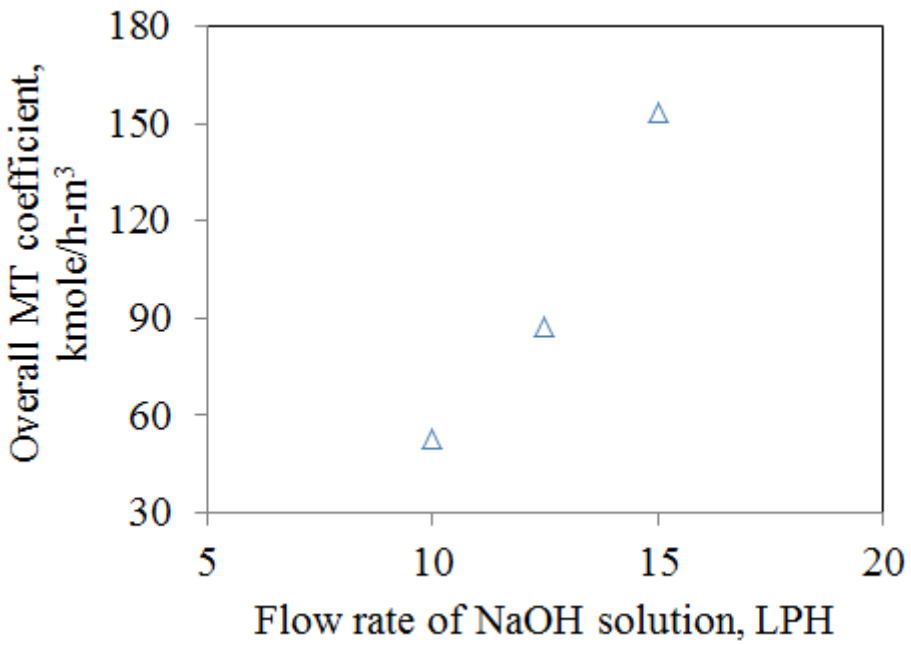


Fig 3.6: Plot showing variation of overall mass transfer coefficient with the flow rate of NaOH solution

Clearly, the solvent flow rate significantly affects the parameters under study and the general trend is that increasing the solvent flow rate results in an increase in the overall mass transfer coefficient which indicates greater efficiency as far as CO_2 removal is concerned.

Since the effect of solvent flow rate is evidently greater than the effect of CO_2 flow rate (as is evident from comparing the second/third and the first/second observations), the variations in HTU, NTU, % CO_2 removal and overall mass transfer coefficients were studied with respect to the change in solvent flow rate.

The solvent flow rate is observed to be influencing the HTU and NTU of the packed tower among other parameters. Increasing the flow rate of the solvent increases the overall mass transfer coefficient, meaning easy removal of CO_2 and hence an increase in NTU and reduction in HTU. This also explains the increase in % CO_2 removal as can be seen from the graph.

However, too high a flow rate would lead to smaller contact time for the mass transfer to take place, whereas a very low flow rate would mean improper and incomplete wetting of the packing material, leading to dry pockets that would hinder the mass transfer process. Hence, an optimum flow rate can be determined for which the CO_2 removal process is most viable.

CHAPTER 5

CONCLUSIONS AND FUTURE WORKS

In this absorptive mass transfer operation involving removal of CO_2 from (CO_2+air) mixture using aqueous NaOH solution as the solvent, following conclusions are drawn:

(a) The CO_2 removal is satisfactory and percentage removal varied considerably from 50-75%, when the packed column is operated counter-currently.

(b) Solvent flow rate affects the % removal of CO_2. An increase in the flow rate of the solvent increases the % removal of CO_2 significantly.

(c) Overall mass transfer coefficients calculated from the experimental data also corroborated the findings on high removal of CO_2, and the mass transfer coefficients show an increase with the increase in the solvent flow rate.

The present work can be extended to carry out further research in the following areas:

(a) The effect of various other types of packing materials *viz*. pall rings, saddles can also be investigated in the set up.

(b) Various other solvents like amines and/or ionic liquids can also be tested on the set up to compare the removal efficiency.

(c) The effect of gas flow rate and hence flooding velocity can also be optimized for various solvents before being effectively used.

(d) A comparative assessment with similar tray towers can also be studied under similar conditions.

(e) Simulation studies can also be done to design and scale-up of the system for higher throughput and optimizing the process parameters.

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