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OPTIMIZATION OF FLOODING IN AN ABSORPTION-DESORPTION UNIT.

Thesis

CENTRAL OSTROBOTHNIA UNIVERSITY OF APPLIED SCIENCES

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Thesis Abstract

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In absorption-desorption packed columns, if any or certain combinations of liquid or gas flow in combination with the reboiler heat pressure and stripper feed power is too high, the column becomes flooded with the liquid. Flooding is accompanied by a dramatic increase in pressure, resulting in inefficient operation and potential damage to equipment. Three alternative hypotheses were tested to guide this work. To optimize efficiency, however, it is desirable to operate close to flooding as possible. This thesis researched the optimum condition at which the packed column will operate close to 80% flooding.

Four parameters were considered for the operation, this includes the CO_2 load; liquid flow; steam heat pressure, and stripper feed power. The Taguchi experimental method was used for the experimental design and analysis for condition ranging from flooding to non-flooding as well as analysis of variance (ANOVA). Also, Katmar software for packed column calculation was used for analysis of pressure drop and flooding.

The analysis obtained an optimum combination of the four parameters under review at 80% approach to flooding. CO_2 absorbed was also taken into consideration during this work. The result from this research sets a standard optimized operation for the pilot-scale absorption-desorption packed column of Central Ostrobothnia University of Applied Sciences and application of optimization for improved efficacy of packed columns in various applicable industries. Recommendations are made based on these findings.

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DEDICATION

This thesis work is dedicated to my mother, Alhaja Khadijat Balogun of blessed memory. May her placid soul, rest in pleasant abode. Amin.

1 INTRODUCTION

In recent times, the global environmental anxiety for the reduction of greenhouse gas emissions majorly from industries, especially thermal power plants and oil processes has brought about technological development to curb global warming from Carbon Dioxide (CO_2). By and large, global warming arouse from the emission of methane, carbon dioxide, nitrous oxide and chlorofluorocarbons. Most imminent in the depletion of the ozone layer is Carbon Dioxide (CO_2). Sequel to the 1997 Kyoto protocol, the European Commission prescribed drastic CO_2 reduction of 20 percent by 2020. Consequently, the bye-product CO_2 reduction from industrial processes such as thermal power flue gases and Oil processes becomes imperative for removal.

Several separation technologies exist for the removal of CO_2 ranging from absorption into liquid solvents, adsorption on solids, cryogenic capture, permeating via membrane. However, literature and researchers opine that absorption remains the most efficient and viable approach. Cryogenic capture involves multiple compressions and cooling stages used directly to liquefy high purity carbon dioxide stream. However, this method requires enormous amount of energy though with high purity rate. Membrane capture increases CO_2 purity as a multistage formation but decreases the final recovery. The constrain that warrants the rare use in the industry is that it reduces the efficiency of the plant by eight to fourteen percent due to the compression of gas stream. Physical absorption is a process that utilizes solvent that cannot react with carbon dioxide such as water but is also rarely used because of its inefficiency. However, the most

famous and popular method in CO_2 purification is chemical absorption because it absorbs and reacts with the gas.

Gas absorption, also known as scrubbing, is a unit operation in chemical engineering whereby a soluble component of a gas mixture is dissolved in a liquid. There is mass transfer of the component of gas from the gas phase to the liquid phase and the solute transferred is absorbed by the liquid. Gas absorption is an integral part of many chemical processes, many of which have been described in literatures. It is worth noting that gas absorption is not limited only to the process industries alone but also in the biological process whereby oxygen from air is absorbed in the blood when we breathe. However, gas absorption will only be restricted to industrial applications in this work. Absorption is widely used in the removal of CO_2 and H_2S from natural gas or gas synthesis in amine solution or alkaline salt. Absorption comes either in physical or chemical process and the rate of absorption of gas is enhanced in the later than the former. Desorption or stripping is the reverse process of absorption when the dissolved gas is recovered from a solution for reuse and regenerates the solvent.

A key aspect in an absorption system is the contact between the gas and liquid phase. Although other contactor exist such as tray column, spray column, bubble column and membrane contactor, this research work focuses more on packed column which operates with either random or structured packing. A packed column like many other industrial processes encounters certain problems, of which flooding is a major concern. Flooding is a phenomenon by which gas moving in one direction in the packed column entrains liquid moving in the opposite direction in the packed column. Flooding is undesirable because it can cause a large pressure drop across the packed column as well as other effects that

are detrimental to the performance and stability of the absorption process. Hence, in the design of the absorption packed column, many parameters need to be considered for efficiency to be attained and also avoid flooding problem which will be preceded with setting the limits for this experiment.

The aim of this research is to resolve the problem of flooding in the pilot scale absorption-desorption unit of the Central Ostrobothnia University of Applied Sciences chemplant laboratory, Kokkola. The study is carried out to investigate the cause(s) of flooding in the stripper and also establish the significant effect of various factors and its interactive effect in the general packed column system to the problem of flooding. The following research questions guided this study: What are the factors that influence flooding in absorption-desorption packed column? How do the factors interact with each other to influence the flooding problem? What is the relationship between stripper pressure drop and flooding; and how do the flooding of the stripper affects the efficiency of the entire packed column in terms of CO_2 absorption? Four alternative hypotheses were formulated and tested at 0.05 level of confidence to facilitate the investigation based on set limit.

2 GAS ABSORPTION-DESORPTION PROCESS

Absorption-desorption is a complete system that consist of both an absorption column and a desorption column interrelated by different other devices and units.

2.1 Gas Absorption

A key aspect in an absorption system, also known as scrubbing, is the contact between gas and liquid phase. Gas absorption is a unit operation where one or more components in a gas mixture are dissolved in a liquid (solvent). The absorption may either be a purely physical phenomenon or involve a chemical reaction, such as the reaction between CO_2 and monoethanolamine (MEA). Also, Gas absorption is a mass transfer operation in which one or more species (solute) is removed from a gaseous stream by dissolution in a liquid (solvent). The insoluble component(s) present in the gas stream not absorbed is called the carrier gas. (Dutta 2007, 257; Falk-Pederson, Dannstrom & Witzko 1999)

Two methods of operation in gas absorption exist, which are counter-current and co-current operations. In co-current absorption, there is no minimum liquid-to-gas ratio and it is less efficient than counter-current operation. The advantage of concurrent operation is the lack of flooding limitation, and high gas flow rate can be used which reduces the required column diameter. For counter-current

operation, the gas which leaves the column or tower via the top enters from below while the liquid flows in through the top and exits via the bottom in an opposite direction. This type of operation is rarely used application and will be discussed further in Chapter 3.1.2 under contactor selection. Graph 1 and 2 depict Counter-current and Co-current operation.
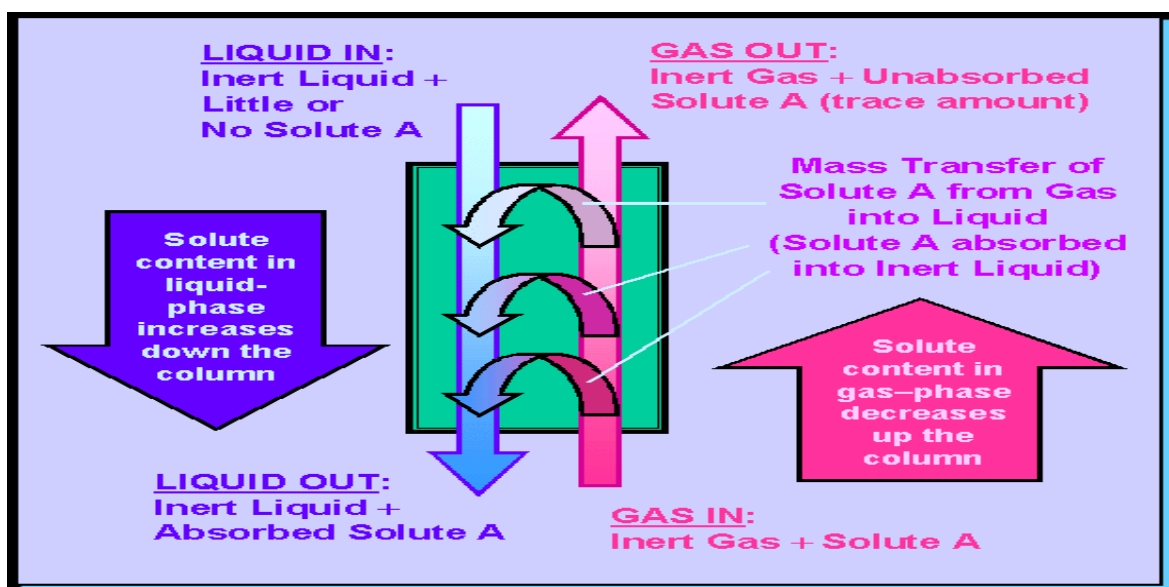
GRAPH 1. Counter-current operation (SM`S e-Home 2010)
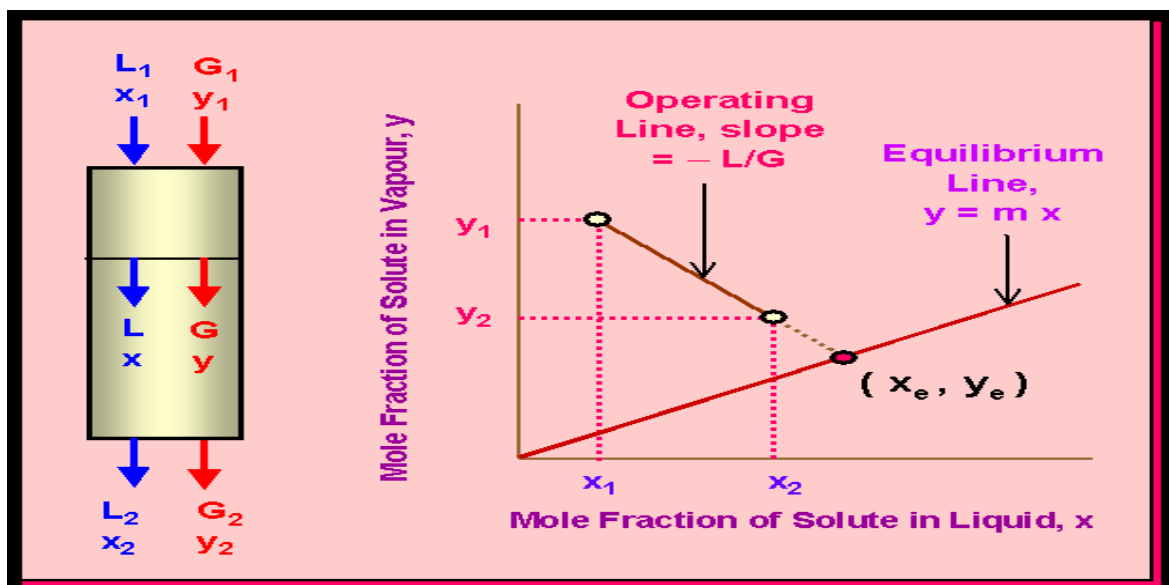
GRAPH 2. Co-current operation (SM`S e-Home 2010)

Kohl and Nielson, 1997 classified absorption in relation to gas purification into three, with respect to the nature of interaction between absorbate and the absorbent.

Physical Solution: In this type of process, the component being absorbed is more soluble in the liquid absorbent than other components of the gas stream, but does not react chemically with the absorbent. The equilibrium concentration of the absorbate in the liquid phase is strongly dependent on the partial pressure in the gas phase. An example is the absorption of hydrogen sulphide and carbon dioxide in the dimethyl ether of polyethylene glycol (SelexoI Process). Relatively simple analytical techniques have been developed for designing systems of this type (Kohl & Nielsen 1997)

Reversible Reaction: This type of absorption involves a chemical reaction between the gaseous component being absorbed and a component of the liquid phase to form a loosely bonded reaction product. The product compound exhibits a finite vapour pressure of the absorbate which increases with temperature. An example is the absorption of carbon dioxide into monoethanolamine solution. Analysis of this type of system is complicated by the nonlinear shape of the equilibrium curve and the effect of reaction rate on the absorption coefficient (Kohl & Nielsen 1997)

Irreversible Reaction: In this type of absorption the component being absorbed reacts with a component of the liquid phase to form reaction products that can not readily be decomposed to release the absorbate. An example is the absorption of hydrogen sulphide in iron chelate solution to form slurry of elemental sulphur particles. The analysis of systems involving irreversible reactions is simplified by the absence of an equilibrium vapour pressure of

absorbate over the solution, but becomes more complex if the irreversible reaction is not instantaneous or involves several steps. (Kohl & Nielsen 1997)

2.1.1 Absorption in Chemical Reaction

Absorption occurs in two reaction processes which are physical and chemical absorption. Physical absorption occurs in situation where there is no chemical reaction between the solute and the solvent (absorbent) such as the use of water and hydrocarbon oils as solvent. However, in situation where aqueous solutions are used as absorbent, absorption are accompanied by chemical reaction in the liquid phase. Absorption with chemical reaction involves the reaction in the liquid phase to vehemently remove a solute from a mixture of gas. It has the capacity to increase the absorption coefficient of the liquid-film compared to an ordinary physical absorption. Reaction in the liquid phase greatly increases the driving force for mass transfer since there is reduction in the equilibrium partial pressure of the solute over the solution. Hence, the equilibrium partial pressure will be zero if the absorption reaction is irreversible. A good instance is the absorption of NH_3 from a gas stream with the use of a dilute acidic solution. Also is the removal of CO_2 from solutions as well as other acid gases. (Kohl & Nielson 1997, 26; McCabe, Smith & Harriottet 2005, 607).

An effective comparison of chemical reaction was given by McCabe et al (2005) in the absorption of NH_3 in dilute HCl in gas concentration (ln 0.02 / 6) and the absorption of NH_3 in water which gives 5.7 and 12 respectively for the same change in concentration. Also, an experiment carried out by Bubulka, Kelly, Schruender & Wallace (2008) at the Rose- Hulman Institute of Technology Indiana

shows that chemical solvent MEA amongst other solvents used, absorbed carbon dioxide at the highest rate, collecting 1.4849 grams of carbon dioxide. The highest physical solvent used was Methanol which absorbed 1.021 grams of carbon dioxide. The result is shown in the graph 3.
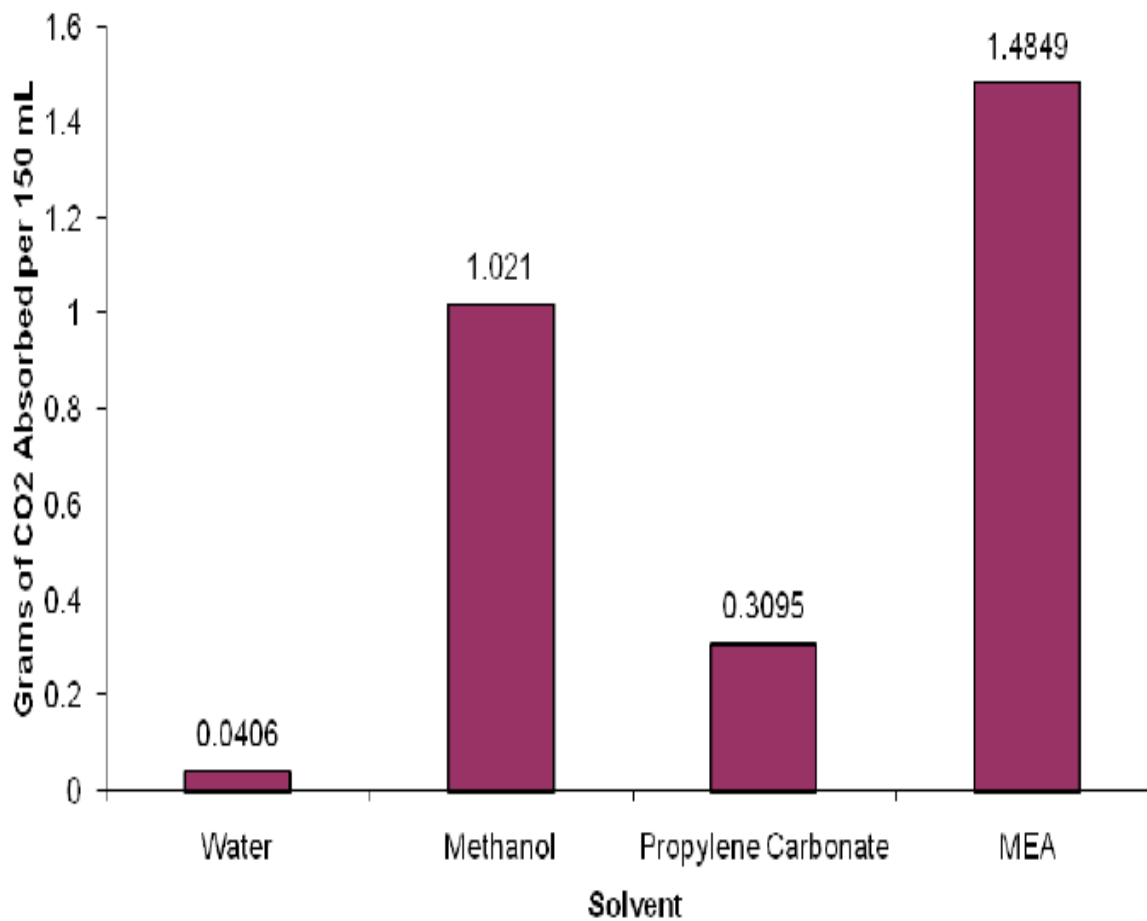

GRAPH 3. Solvent VS. Amount of CO2 Absorbed per 150 mL

2.2 Desorption Process

The advantageous characteristic of absorption is that it can be reversed by sending the CO2-rich absorbent to the desorber (stripper). Desorption involves the removal of solutes component from a liquid stream into a gas stream when in contact with an inert gas or steam but since steam condense, it is preferred in solute recovery than inert gas. However, the high cost of steam is a concern but better still, desorption process is stripped using little possible steam. The solute is desorbed from a liquid, which in much case is absorbed from a mixture of gas to regenerate the absorbing solution by recovering the solute in a more concentrated state. In an exceptional case, air is used besides steam or inert gas to desorb certain amount of compound such as organic solvents or ammonia gas from water and it cost less to provide air especially when the need for the recovery of concentrated solute is unnecessary. The height of the column is also reduced in the use of air stripping. Desorption, alternatively called stripping due to the nature of its operation requires certain factors for operational efficiency which includes reduction, in total pressure or temperature increase or otherwise. If absorption is carried out under high pressure, a large fraction of the solute can sometimes be removed simply by flashing to atmospheric pressure (McCabe et al. 2005, 590.)

2.3 Absorption-Desorption Packed Column

Packed columns are often employed in the removal of impurities from gas streams and also the removal of volatile components from liquid streams. Packed columns are also applicable in distillation processes where the boiling point of components

is very close and makes separation very tedious. It is important to explain the combined function of the absorption-desorption column with description from the model in the graph 4 below. The lines in blue contains solution, rich in CO_2, moving from the absorption column to the stripper/desorption column while the line in green is the regeneration of CO_2 free solution back to the absorber from the stripper. The absorption column operates at low temperature while the stripper operates at high temperature. The actual function of the desorber is to produce a free CO_2 solution and regenerates the washing liquid at high temperature and recycled back to the absorber for re-use with little or no CO_2 in the solution.
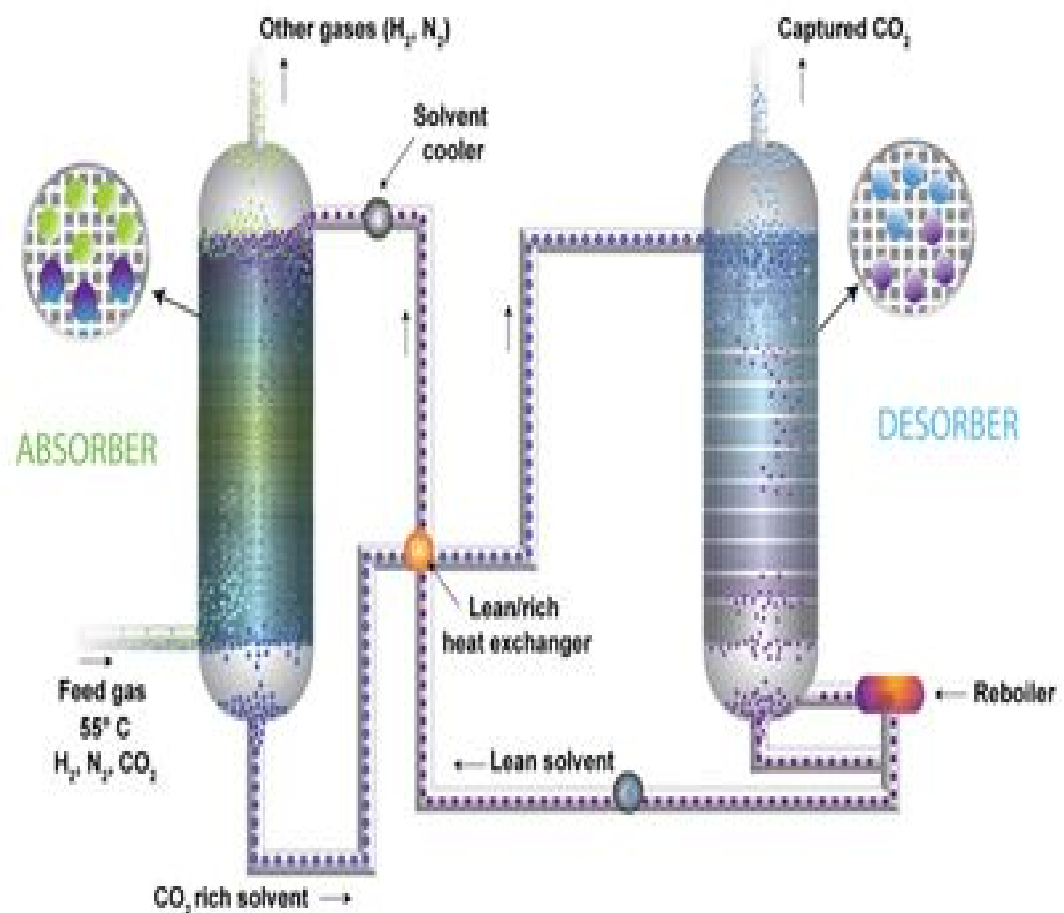
GRAPH 4. Absorption Desorption Column (CRC 2010)

2.4 Parameters for the design of Absorption-Desorption Packed Column

In the design of an absorption column, the amount of gas to be purified and the extent of purification which determines the height of the column are two basic factors which determine the size of an absorption column. The same condition in the design of absorption column size applies to the desorption column. In more specific terms, the amount of gas to be stripped is often determined by the diameter of the desorption column. The purity to be attained in the solvent decides the height. Furthermore, for the flue gas to be absorbed, the liquid surface must meet the gas. The absorption column is designed for the gas to ascend in contact with the solvent descending. Hence, the intensity of the liquid purification area desired determines the height of the column. Design in contrast to the stated guideline can lead to irregularity or functionality of absorption-desorption column. If the rate of the gas flow upwards in the column becomes too high due to a too small tower diameter, it will bring with it, the solvent which is intended to run downwards in the column, resulting in flooding. (Falk-Pederson et al. 1999.)

The solvent is consumed basically due to solvent evaporation in the absorption and desorption column as well as solvent degradation. These are due to the huge surface of temperature that transfers the heat to the solvent and chemical degrading due to the system contamination. Adequate care must be taken with packing of column in uniform manner to avoid creating a route for gas to move with low pressure loss. This aversion is eminent to both absorption and desorption column to avoid a partial treatment of the gas. In addition to flooding, corrosion is a problem of scrubbing and stripping in the absorption column, desorption column and boiler/reboiler especially in the processes where amines are used. To avoid problems in the process, the corrosion must be removed by filter means.

Other problems associated with the processes of absorption/desorption columns is foaming and could be caused by corrosion particles or other particles in the solvent could be a factor. Filters, adjustment in column operation or chemical means can be used to solve the problem. (Heibel et al., 2003.)

2.4.1 Equilibrium in Gas-Liquid System and Solubility Selection

Solubility of a solute in a solvent occurs in equilibrium distribution between two phases. This is necessary in mass transfer determination of the driving force given by Raoult's law and seldom, Henry's law for low solute concentration. The absorption of gas is done under low temperature, recall that the solubility of gases in liquid decreases with temperature and hence, there is always a larger driving force for the transfer of solute from the gas to the solvent. In contrast, desorption operates conversely because, stripping is carried out under high temperature to enable the direction of the force to be reversed. In the selection of solvent for absorption process, the solvent has to be highly soluble to absorb the solute. Several other factors determine the selection of solvent. These include volatility, viscosity, corrosiveness and toxicity. That is, an effective solvent should have low volatility which in turn, reduces the extent of solvent loss. Contrary to that will lead to high solvent loss and in turn, will create an environmental pollution as well as loss of resources. A low viscosity allows a solvent to flow easily which extensively combats the problem of flooding. (Kohl & Nielsen 2007, 37.)

2.4.2 Contactor Selection

Functionally, the purpose of gas absorption contactor is to create a broad area in contact between the liquid and the gas phase with the view to satisfying mass transfer conditions. Kohl & Richard (1997) point out that "Contactor normally employ at least one of the following mechanisms: (1) dividing the gas into small bubbles in a continuous liquid phase (e.g., bubble cap trays), (2) spreading the liquid into thin films that flow through a continuous gas phase (e.g., packed columns), and (3) forming the liquid into small drops in a continuous gas phase (e.g., spray chambers)". All the afore-mentioned contactor is applicable to gas absorption but certain condition may favour one to another. However, this work will focus mainly on packed column type of contactor.

Packed column is a common apparatus exploited in gas absorption operation and are becoming popular due to its packing performance and dependable design techniques. It is made up of a cylindrical column composed of two inlets and distributing room: A liquid inlet and a distributor at the top; gas inlet and distributing space below the column; gas outlet at the top and that of liquid at the bottom. The column is supported by firm fashioned packing. Packing element comes in either dumped, which are random packing or structured packing that are non-random. While the former is predominantly used, the later were primarily designed for complex separations in small scale distillation column. Stacked packing is less commonly used compared to the previous but specially used when high mass transfer efficiency of pressure drop is exceptionally low, very high flow rates. Dumped column packing are seldom made of steel or aluminium rings but frequently made of inexpensive, inert material such as plastics. They are made of 6 to 75 mm in dimension while packing smaller than 25 mm are used in laboratory

or pilot scale column. However, stacked packing unit are 50 to 200mm sized. Typical dumped packing used in packed columns is Raschig ring, Lessing ring, berl saddle, intalox saddle, tellerette and pall ring. "The corrugated metal sheet of structured are arranged in a crisscross relationship to create flow channels for the phase. The intersections of the corrugated sheets create mixing points for the liquid and vapour phases. Structured packing offer excellent capacity and low pressure drop per theoretical stage" (Koch-Glitsch). Dumped Random and Structured packing are illustrated in graph 6 and 7 respectively. (McCabe et all 2005, 565 - 567; Kohl & Nielsen 2007, 23.)
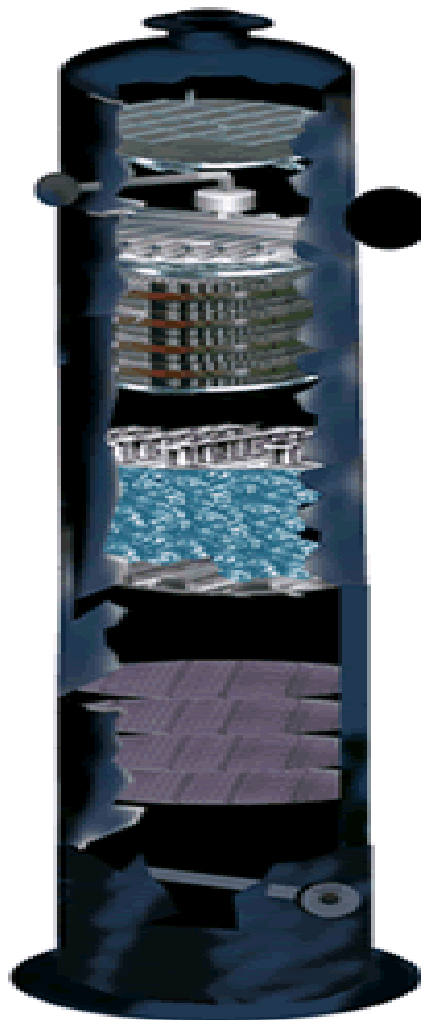
GRAPH 5. A Typical Packed Column Structure

(a)   (b)   (c)

(d)
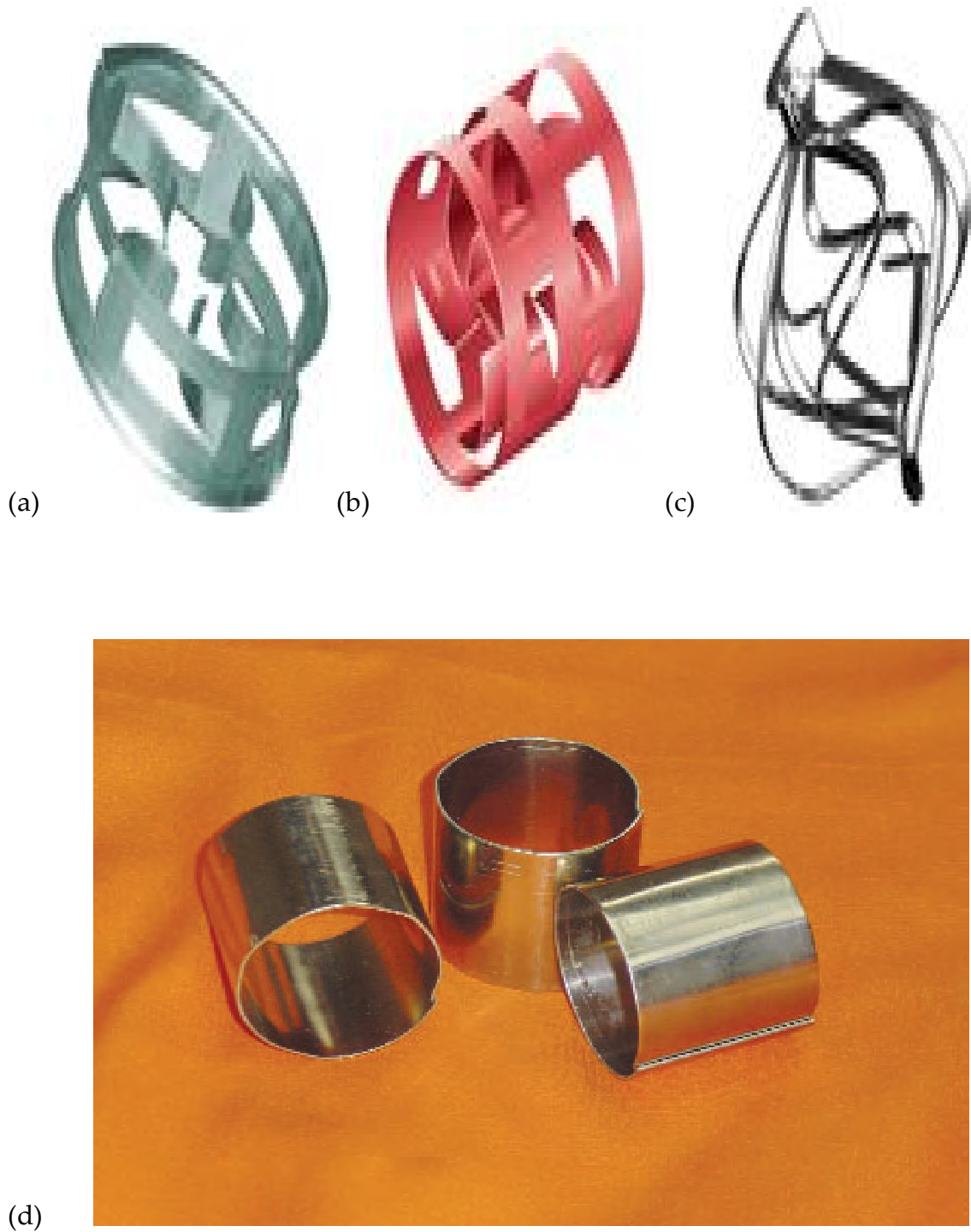
GRAPH 6. Random Packing Types: (a) Mini-Pall Ring (b) Flexi Pall Ring (c) Metal

Intalox Saddle (d) Metal Raschig (kochglitsch 2010.)
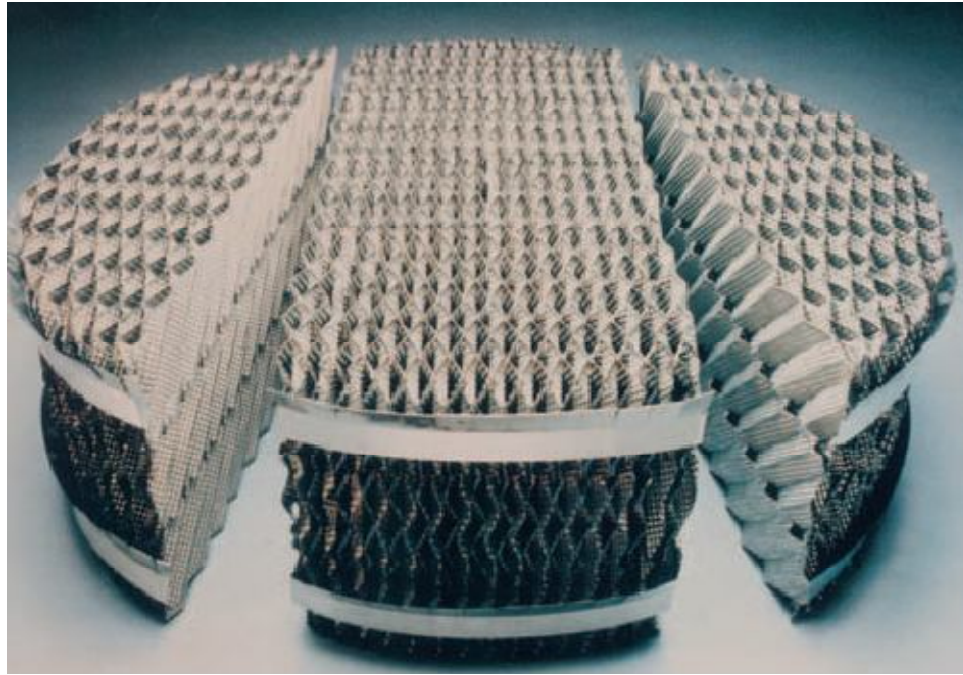
(a)
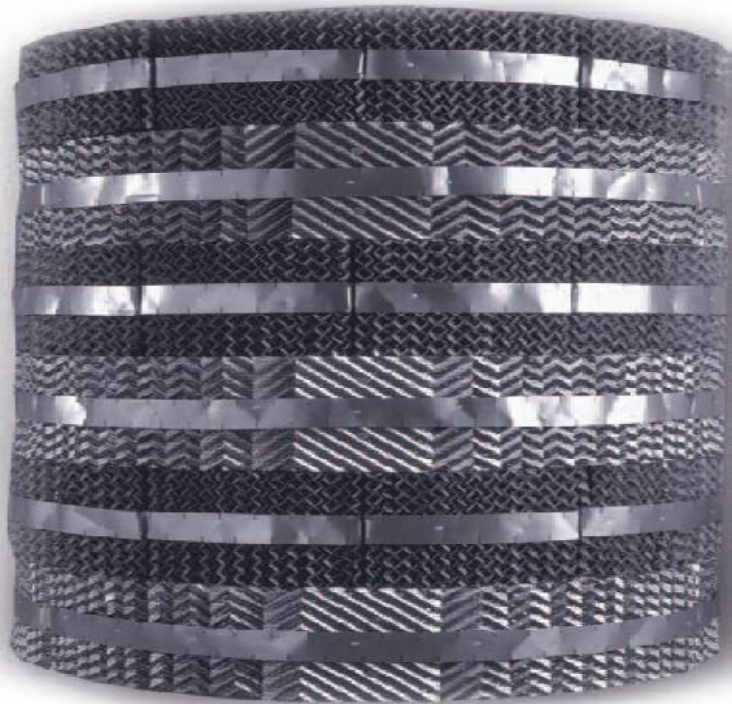
(b)

GRAPH 7. (a) Intalox Structure Packing (b) Stack of Structured Packing (kochglitsch 2010.)

Packed column are seldom used in cross flow arrangement with gas flowing horizontally while the liquid flow descending through the bed. However, counter current flow are mostly implored. "Co-current contactors using structured packing elements similar to in-line mixers are used for gas purification applications when a single contact stage is sufficient; for example, when an irreversible reaction occurs. They have the advantage of operating with much higher gas velocities than counter current designs without being subject to flooding problems". (Kohl & Nielsen 2007, 23.)

2.4.3 Pressure Drop and Flow Rates

At constant gas flow, an increase in liquid to the column will result to increase in pressure drop until the liquid flooding is attained. At this point, any excess liquid that cannot proceed through will remain at the top of the packing, causing the entire column to be filled with liquid and further intensifying the pressure drop. Moreover, at constant liquid flow downwards, increase in gas flow will lead to rise in pressure drop until flooding rate is attained and any further increase will not permit the flow of liquid and consequently, leading to accumulation of liquid at the top of the column and continuous increase in the pressure drop. (Zent 1980, 290.)

According to McCabe et al 2005, in the analysis of pressure drop in a packed tower for air-water system with 1-in. Intalox saddles, when packing is dry, the line obtained in the graph of change in pressure drop (ΔP) versus Gas flow rate (G_y) is straight and has a slope of 1.8. The pressure drop with liquid flow is greater than

the dry packing because the space available for gas flow is hindered by the liquid flow. With high increase in gas velocity, the pressure drop rises in such a way that the line almost becomes perpendicular when the pressure drop is about 250 mm of water per meter.

2.4.4 Flooding

Flooding arise when gas or liquid flow is increased beyond the capacity of a column. This limit the quantity of liquid and gas flow through a column and in counter current flow, it places an upper limit on the liquid and gas flow rates. "Flooding in a counter current flow is the flow condition in which a normally down-flowing liquid reverse course and begins to flow upward due to the interaction between the two phases". An accumulation of liquid at the top of the packing is an indication of flooding. Flooding adversely impacts negative on absorption packed column and other packed column applications such as Distillation unit. (Adusei & Heibel 2003, 1; Zent 1980, 302.)

With reference to chapter 3.3.1, Flooding effect rapidly on increase in pressure drop and affect the column operation. The accumulation of liquid on the column packing cause the pressure below the flooding point to increase. Flooding can be identified by changes in pressure difference of the column or temperature. Consequently, a differential pressure can be observed and the reboiled vapour blocked from rising, which the bottom column's temperature may increase. Many a common method is employed in combating flooding which, decreasing both or either the feed rate and reflux rate is significant. Moreover, the reduction in the

amount of liquid in the column and decreasing vapour in the column by reduction of the reboiler steam is noteworthy. (SM`S e-Home 2010)

2.5 Correlation between Pressure drop and flooding velocity in Packed Column

Several common correlations exist for pressure drop and flooding velocity in packed column which most depends on the use of log-log plot with $(G_x/G_y)(\varrho_y/\varrho_x)^{0.5}$ on the abscissa and a function containing G^2_y on the ordinate. Sherwood and Hollaway base their correlation of the flooding condition for various packing on the equation.

$$\frac{U2a}{gFd3} \frac{\varrho v}{\varrho L} \mu 0.2 = f\left(\frac{L}{G} \frac{\varrho v}{\varrho L}\right) {}^{\wedge 1/2}.$$

Where U = Superficial gas velocity at flooding. ft. /sec.

a = Total area of packing, sq. ft. /cu. ft. Packing

F_d = Fractional voids in dry packing, cu. ft. /cu. ft. Tower volume

G = gas rate, lb. / (hr.) (sq. ft.)

g = 32.2 (lb. Mass) (ft.) / (lb. Force) (sec.^{2})

L = Liquid rate, lb. / (hr.) (sq. ft.)

ϱ_v and ϱ_L = gas and liquid densities, lb./cu. ft.

μ = viscosity of liquid, centipoises

However, there is no perfect correlation for pressure drop that is applicable to all packing. (McCabe et all 2005, 572; Teller, Miller & Scheibel 1963, 18- 26).

McCabe et al (2005, 372-373) gave a commonly used generalized correlation for pressure drop estimation in dumped packing indicated in graph 8 below with recent studies showing flooding line at pressure drops of 0.7 to 1.5 in. H_2O/ft of packing for 2- or 3-in. Packings. $\Delta P_{flood} = 0.115 F_p^{0.7}$ is given as the empirical equation for limiting pressure drop.

ΔP_{flood} = Pressure drop at flooding, in. H_2O/ft of packing.

F_p= Packing factor

Strigle proposed an alternative correlation for pressure drop in packed column but with a capacity factor on the ordinate equal $\mu_o\sqrt{\rho y/(\rho x - \rho y)}$ where μ_o and v are the superficial velocity in ft/sec and kinematic viscosity of liquid in centistokes respectively.
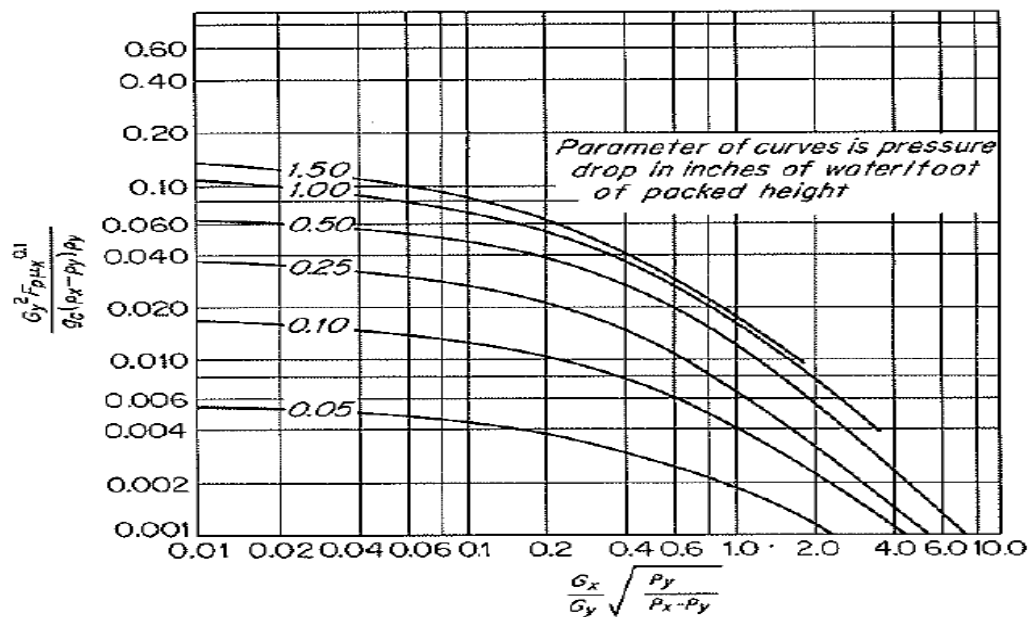
GRAPH 8. Generalized correlation for pressure drop in packed Columns (Adapted from McCabe et al.,2005. Page 694.)

Gx and Gy are given in 1b/ft2 .s, ϱ_x and ϱ_y in 1b/ft3 while μ_x and g_c are given in cP and 32.1741 lb·ft/1bf·s² respectively.
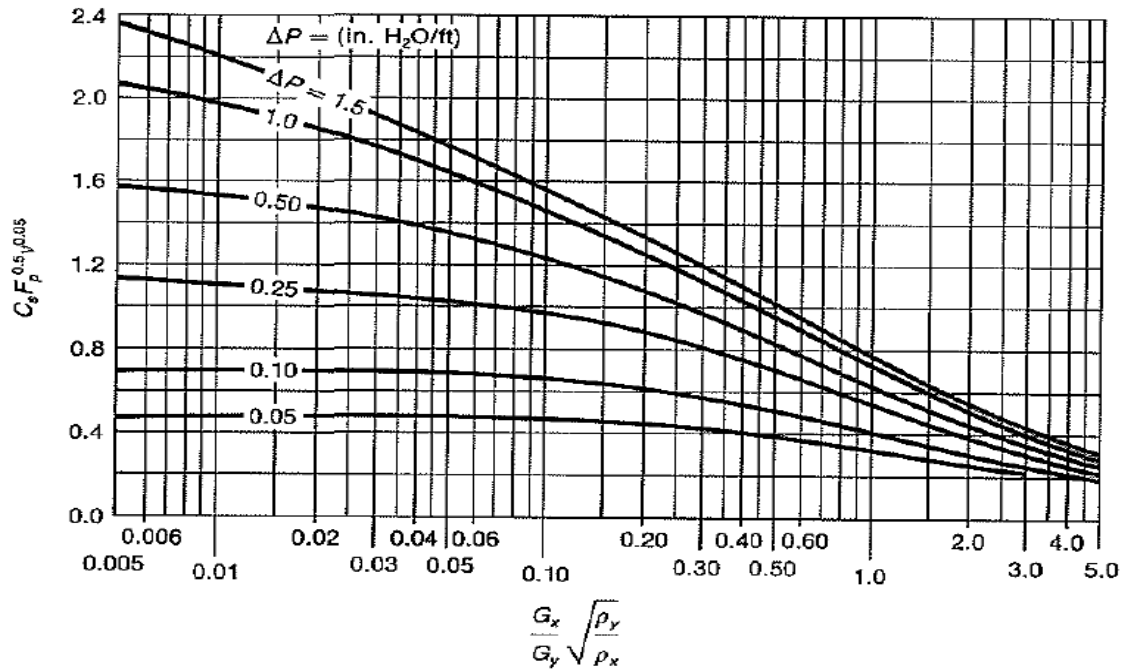
GRAPH 9. Alternative generalized pressure drop correlation   (Adapted from McCabe et al., 2005. Page 695)

2.6 Design Approach for Absorption Desorption Packed Column

The design of absorption packed column involves the combination of science and art but the first stage is more scientific than art. In absorption/desorption design, the equilibrium line is constructed subject to the contaminant steam being considered dilute or concentrated. For non-dilute stream, the mass transfer coefficient is evaluated in terms of gas and liquid flow but when the contaminant is less than 10 mole percent of the stream, it is rather ideal to consider the stream as dilute. The later allows the column designer to attain constant mass transfer and construct an operating line in terms of a simple mass balance as follows. (CER 2008.)
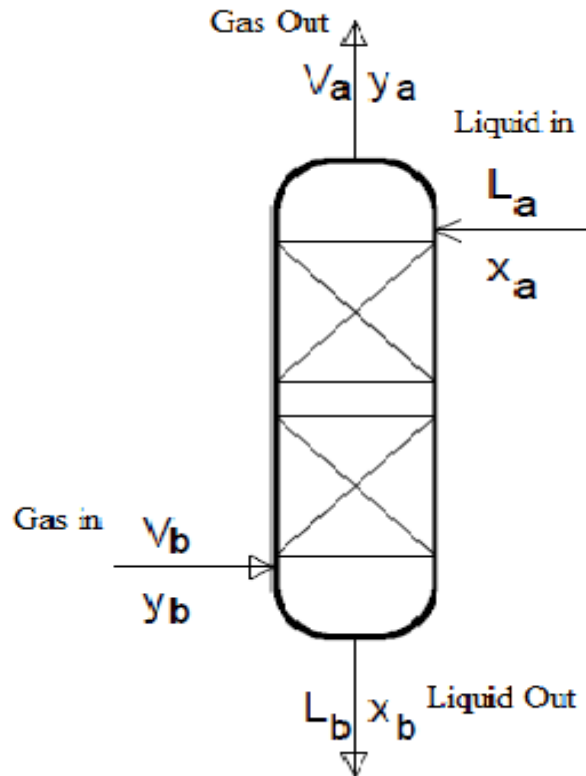
GRAPH 10. Material Balance diagram

Mass Balance: L out X out + G out Y out = L in X in + G in Y in.

The design of an absorption column usually follows the steps of contact selector sequel to the requirements and condition for its design, calculation of heat and material balances, required column height estimation subject to mass transfer analysis as well as the calculation of required column diameter or packing parameters based on hydraulic considerations and liquid and gas flow rates. Finally is the mechanical design of the hardware but although the steps are not necessarily to be conducted in the sequential order. However, the principal task in concurrent operation is the contact selector and sizing of the mixing element. Reaction rate data is required for a chemical reaction absorber and stripper but otherwise, the physical, thermal and transport properties of the gases and liquid are necessary. (Kohl & Nielsen 2007, 12.)

2.6.1 Packing Type and Size

The art of economy is somewhat adopted in this regards with respect to pressure drop constraints. Structured packing is used for very low pressure drop and the increasing the capacity of existing column. However, in the design of a new column, a more economic random packing is considered since there is no serious pressure drop constraints. In Raschig, the column diameter to packing size should be more than 30, 15 and 10 for Raschig rings, Ceramic saddles and Plastic saddles respectively. (CER 2008.)

TABLE 1. Packing factors (Adapted from Kohl & Richard 2007)

Packing	Material	1/4	3/8	½	5/8	3/4	1	11/4	11/2	2	3	31/2
Super Interlox Sadle	C						60			30		
Super Interlox Sadle	P						42			28	18	
Interlox Sadle	C	725	330	200		145	92		52	40	22	
Interlox Sadle	P						33			21	16	
Rashig Rings	C	1600	100	580	380	255	179	125	93	65	37	
Rashig Rings 1/32"	M	700	390	300	170	155	115		65	45		
Rashig Rings 1/16"	M			410	300	220	144	110	83	57	32	
Berl Saddles	C	900		240		170	110		65	45		
Pall Rings	P				95		55		40	26		17
Pall Rings	M				81		56		40	27		18
Telleretes	P						35			24		17
Maspac	P						32					21
IMPT Packing	M				51		41		24	18	12	12
Interlox Snowflake	P	Diameter 3.7 in, height 1.2 in, F= 13										
Hy-Pak Packing	M						43		26	18		15
Jaeger Tri-Packs	P						28			16		
Jaeger VSP	M						32			21		12

2.6.2 Material and Energy Balance

Absorption design can be carried out with the aid of analytical correlation and software applications and in counter current flow; its efficiency can is best analyzed by the aid of Operating line-equilibrium curve diagram, both the operating and the equilibrium line are plotted on the X and Y axis. For absorption to take place, the operating line must be lie above the equilibrium line. Hence, a positive driving force, y – y* is given. The operating line equation, $y = \frac{L}{V}x + \frac{Vaya - LaXa}{V}$ is derived from the relationship between x and y at any point in the column by rearranging a component of the material balance of the column above a random segment given by $L_aX_a + V\,y = Lx + V_ay_a$ where V and L are the molar flow rate of gas phase and liquid phase respectively. As the gas passes through the column, the total gas rate, V decreases while the liquid flow, L increases due to the absorption of soluble component from a gas mixture. Consequently, the operating line is somewhat curved as depicted in the graph below. (McCabe et all 2005, 576 - 577; Kohl & Richard 2007, 14)
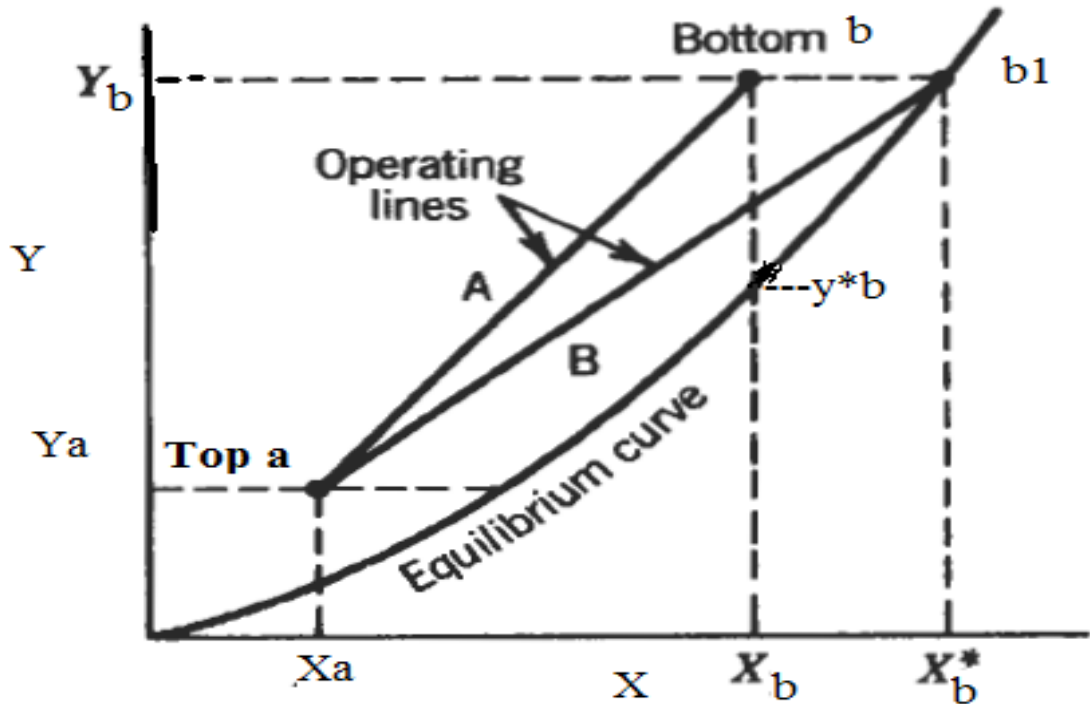
GRAPH 11. Operating Line-Equilibrium Curve (Adapted from Kohl & Richard 2007)

Two possible operating lines A and B corresponds a typical absorption design and theoretical minimum liquid flow rate respectively with the distance between the operating line and equilibrium curve represents the mass transfer driving force at any point in the column. Provided the line B touches the equilibrium curve at the bottom of the column indicates an indefinite tall column in required. The effect of changes is often ignored for dilute mixtures containing less than 10 percent of solute gas and the operating lines is straight. Heat is often released during absorption process which leads to increase in temperature of the column that warrants modification of the equilibrium curve to correspond to the actual condition at each point in the column; with respect to this, Stocker & Wilke (1977) developed a computer programme for packed columns which operates by dividing the column into arbitrary numbers of segments and also a shortcut

method that does not require a computer programme. (McCabe et all 2005, 577; Kohl & Richard 2007, 14 – 15.)

2.6.3 Determination of Column Height

The rate of absorption per unit volume of packed column where x and y are fraction of the absorbed component is given by:

$r = k_y a\, (y - y^*)$

$r = k_x a\, (x_i - x)$

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

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

The determination of the number of transfer unit, Noy from y-x diagram multiply by Height of transfer unit, Hoy correspond to Z_T = Hoy x Noy. In a situation where the operating and equilibrium line are straight and parallel, the number of transfer units at gas stage is the change in concentration divided by the logarithmic mean driving force: Noy = $\frac{yb - ya}{\Delta yL}$ and for liquid phase, Noy = $\frac{Xb - Xa}{\Delta yL}$ where Δy_L is the logarithmic means of $y_b - y^*_b$ and $y_a - y^*_a$. But when the operating line is straight but vertical than the equilibrium line which is the case in absorption, the number of transfer unit is greater than the ideal stages unlike the previous which the number of transfer unit is neo-like the number of ideal stages. (McCabe et al 2005, 579 – 582.)

Absorption coefficient is the most convenient design method for packed column based on two film theory proposed by Whitman (1923) Two absorption coefficient Kx defined as the quantity of material transferred through the liquid film per unit time, per unit area, per unit of driving force in terms of liquid concentration; and Ky, the quantity transferred through the gas film per unit time, per unit area, per unit driving force in terms of mole-fraction difference. The overall height of transfer unit is the height of packed section needed to achieve a change in concentration equal to the average driving force in that section. Similar to the mass transfer coefficient, four kinds of transfer units based on overall driving forces for gas and liquid phases exist as follows:

Gas film: $\quad \text{Hy} = \frac{V/S}{Kya} \qquad \text{Ny} = \int \frac{dy}{y - yi}$

Liquid film: $\quad \text{Hx} = \frac{L/S}{Kxa} \qquad \text{Ny} = \int \frac{dx}{xi - x}$

Overall gas: $\quad \text{Hoy} = \frac{V/S}{Kya} \qquad \text{Noy} = \int \frac{dy}{y - y*}$

Overall liquid: $\quad \text{Hox} = \frac{L/S}{Kxa} \qquad \text{Nox} = \int \frac{dx}{x* - x}$

(McCabe et al 2005, 579 – 582.)

Mass transfer coefficients are alternatively based on a partial pressure driving force for gas-film coefficient instead of the mole-fraction differences written as k_ga or K_ga. Their relationships to the coefficient are $k_ga = k_ya/P$ and $K_ga = K_ya/P$ and there unit given as mol/ft3·h·atm where P is the total pressure. In a relative case, liquid-film coefficient may be given as k_La or K_La.

Hence, $H_y = \dfrac{GM}{kgaP}$ and $H_{oy} = \dfrac{GM}{KgaP}$

$H_x = \dfrac{Gx/\rho x}{kLa}$ and $H_{ox} = \dfrac{Gx/\rho x}{KLa}$

These simplified equations are more readily used and amply accurate for most engineering-design equations. (McCabe et all 2005, 583-584; Kohl & Richard 2007, 15-19)

These equations assume that the following conditions hold:

1. The equilibrium curve is linear over the range of concentration encountered
2. The partial pressure of the inert gas is essentially constant over the length of the column.
3. The solute contents of gaseous and liquid phases are sufficiently low that the partial pressure and liquid concentration may be assumed proportional to the corresponding values when expressed in terms of moles of solute per mole of inert gas or solvent. (Kohl & Richard 2007, 17)

McCabe et al (2005) gives the Height of Transfer Unit is given as:

$H_{oy} = H_y + m\dfrac{GM}{LM}\,H_x$

$H_{ox} = H_x + \dfrac{LM}{mGM}\,H_y$

2.6.4 Determination of Column Diameter

The preliminary step in packed column sizing is predicting the shell diameter necessary to pass the required gas and liquid rates in other to avoid flooding. Liquid viscosity; density, gas density, packing size; type; surface and free space are the determining variables. Most methods in the determination of random packed columns is usually based on the flooding correlations derived from the likes of Sherwood et al. (1938), Elgin and Weiss (1939), Lobo et al. (1945), Ecket (1970A, 1975), Kister and Gill (1919), Robbins (1991) and Leva (1954). The correlation derived by Sherwood et al. (1938) encompasses all the variables which lead Lobo et al. (1945) to research intensively more on the variables and the experimental procedures. Other researchers incorporate pressure drop to the study of flow through packing in reference to data plotted by Sherwood et al and Lobo et al that fall short of flooding.

The Ecket (1975) version which is the foundation for Strigle (1994) discussed in chapter 3.2 is given in the graph below. The Y axis is the Flow Capacity Factor which includes a packing factor, F, a characteristic of the packing configuration and the X axis which is the Relative Flow Capacity.
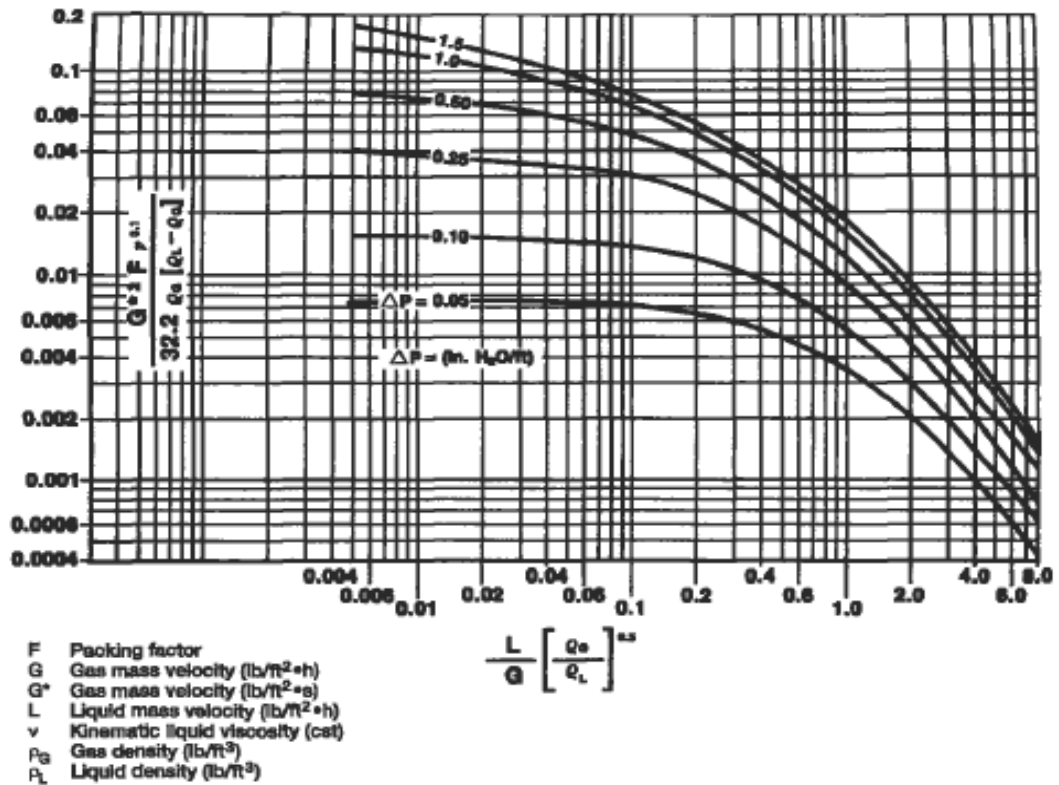
GRAPH 12. Generalized pressure drop correlation for packed column (Strigle 1994)

In a general, considering the ratio of the diameter of the column to the packing should be at least 15:1 and the liquid redistribution is required at every 5 to 10 column diameter for rings and about 20 feet for dumped packing. The liquid stream number provided by feed distributor should be 3 to 5 ft² in column larger than three feet in diameter. (Kohl & Richard 2007, 31)

3 EXPERIMENTAL DESIGN

Quality is a major concern in the industry and it is a subject to the foundation of its design which may not necessarily be achieved by cost, time or material used, but by design is being planning and implementation. Experimental design is a systematic approach for the investigation of an engineering system or industrial process, which is an efficient methodology for the experimental planning. Experimental design is also defined as "the simultaneous evaluation of two or more factors (parameters) for their ability to affect the resultant average or variability of particular product or process characteristics" (Philips 1996, 24). Hence, data obtained can be properly analysed to arrive at accurate and valid conclusions. In industries, the main objective is to utilize the maximum unbiased information of various factors affecting the process observed and have been widely used in the manufacturing and process industries for production optimization. Experimental design is widely used in the industries today and is effectively applied to optimization of many processes in the chemical and other Engineering fields. Box and Draper (1987, 115) gave an analysis of an experiment for the manufacturing of dyestuff in relation to the desired quality of brightness and fabric strength. The experiment was aimed at identifying the factors that affects the brightness, hue, and strength of the final product.

Generally, experimental design begins with defining the experimental objectives which, determines the type of design to adopt and adequate selection of the process variables/factors that may affect it and followed by the carrying out the experiment and alternating one or more variables with the aim to identifying the change effect on one or more of the variables. Thereafter, the application of

analysis of variance (ANOVA) to determine the obvious relationship between the variables subjected to the experiment.

3.1 Experimental Designs

Several experimental designs exist that are practically used in the industry, which are subject to the objective of the experiment or the process of the experiment. According to statsoft, the major classes of experimental designs typically used in the industries include: Two-level design, multi-factor design, screening design for large numbers of factors, three-level, multi-factor designs (mixed designs with 2 and 3 level factors inclusive), central composite designs, Latin square design, Taguchi robust design analysis, and mixture designs. The choice of experimental design is subject to the objective and the number of factors to the investigated and the design types are listed as follows. Completely randomized design, randomized block design, full factorial design, fractional factorial design, Plackett-Burman design, response surface design, response surface design, adding centre point runs, improving fractional design resolution, three-level factorial designs, three-level, mixed factorial and fractional designs. However, many industrial experimental situation calls for standard and advance experimental design for the purpose of precision and optimal properties. In relation to this, Taguchi experimental method is one of the common advanced methods used today in the industry and has been adopted for this research work (Statsoft 2010)

3.2 The Taguchi Experimental Design

The Taguchi method was developed by Dr. Taguchi, a Japanese scholar. The method is aimed at high quality production and low cost in favour of the manufacturer. This process involves designing an experiment to examine the effect of certain parameters on mean and variance of a process in relation to its function ability. Rather than evaluating all possible combinations, the Taguchi method combines all necessary data with few experiments to determine the factors that affect the process. The number of parameters is selected based on the variables available and the number of levels subject to the different states of the experiment. Thereafter, the test of significance is carried out by appropriate analysis such as ANOVA, Fisher's exact test or Chi-Squared test (Bredeweg, Morga, Sekol & Wong 2007.)

3.2.1 Taguchi's Philosophy

Taguchi's design was an instrumentation of the research facility sequel to the poor state of the communication system of Japan after the world war. As the head of the Japan Electrical Communication Laboratories (ECL), Dr. Taguchi observed that colossal amount of money and time was been exhausted on experimentation. This was the limelight in Taguchi's experimental design which focuses on optimization of engineering experimentation and quality control. Taguchi's approach has been productively adopted in numerous industrialized nations such as the united state. Taguchi's philosophy is a principle aimed at delivering quality engineers which

industries such as Ford motors is a beneficiary. The concept of Taguchi's philosophy proposes that:

1- Quality should be design into minimizing the deviation from a target
2- Quality is best achieved in minimizing the deviation from a target.
3- The cost of quality should be measured as a function of deviation from the standard and the losses should be measured system-wide.

According to Taguchi, building into the product is the key to improving quality because 85 percent of poor quality is attributed to manufacturing process and 15% to workers and poor quality cannot be resolved by mere inspection, screening and retrieval. Hence, to achieve efficient design product quality, Taguchi proposed three stage processes which include: System design; parameter design; and tolerance design. These three processes focus on the suitable worker's level, the design factor and conscience of the tolerance factor. (Roy 1990)

3.2.2 The Design Process

Several literatures exist for the analysis of Taguchi's experimental design. Bredeweg et al 2007 and Roy 1990, gave a four-phase explanation to it. The pictorial diagram of the phases is shown in graph 14. Though, Philip 1996 divided the experimental design into three main stages of (1) The planning phase, (2) The conductive phase and (3) The analysis phase but the process is similar to that of the former.
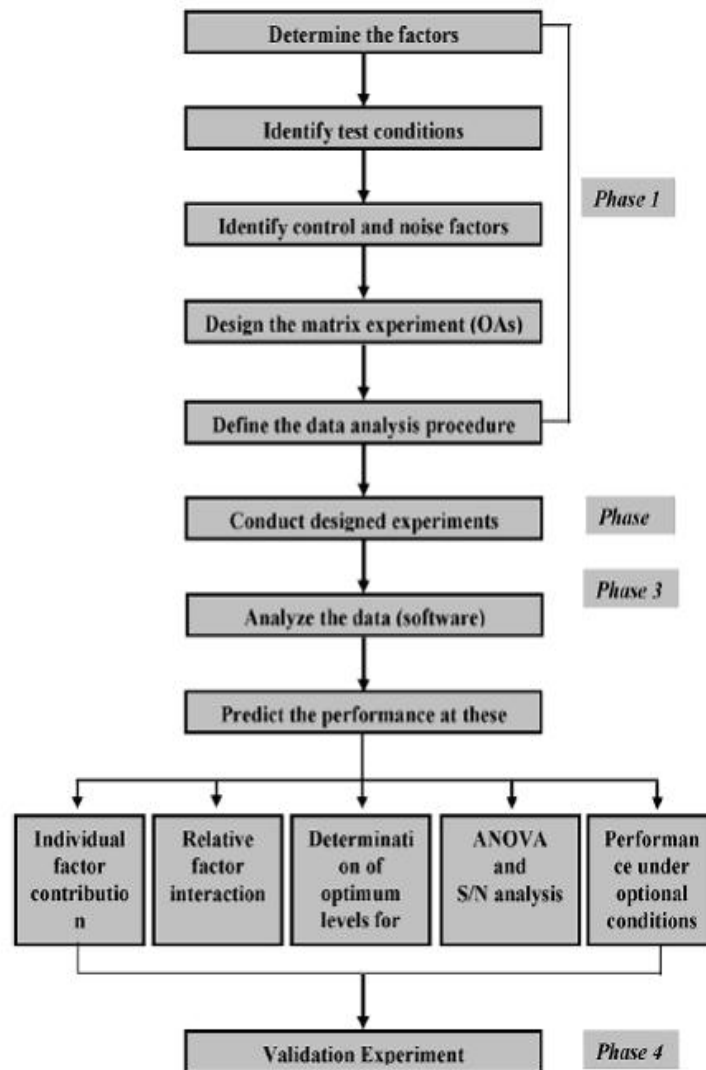
GRAPH 13. Pictorial depiction of 4- phase step analysis of Taguchi Method

The planning or brainstorming phase is the most important since it is the bedrock of the whole experiment and the stage where the parameters and levels are selected. Taguchi experimental design utilizes sets of orthogonal arrays (OA) which accommodates many experimental situations that are subject to the level of factors which could run from two to four levels. However, the number of parameters could vary from three to fifty. Hence, the experimental design will be selected (L4 to L50) which requisite the experimental phase. Noise factors are also considered in this phase which is the factors that that influence the response of a

process. Taguchi's orthogonal arrays provide an alternative to standard factorial designs. (Roy 1990, 29-30.)

GRAPH 14. Orthogonal Array Selection Table (Adapted from Michigan Chemical Process Dynamics and Controls Open Textbooks)

Experiments are conducted in the second phase and yield data based on the design of the first stage. The Taguchi method requires very few experiments compared to a conventional method such as factorial design. 4 factors at 3 levels will require a factorial experiment of 84 test runs which Taguchi accomplishes with just 9 test runs. Hence, the experiment must be well planned and implemented. The third phase which is the analysis phase is more statistical and the least understood due to its complexity. The effect of each variable on the process output is determined using the signal-to-noise (SN) ratio which is calculated for each of the experiment conducted. The equation is given below. (Bredeweg et al. 2007; Roy 1990.)

$$SN_i = 10 \log \frac{\bar{y}_i{}^2}{s_i{}^2}$$

$\bar{y}_i$ is the mean value which is the performance characteristic for a particular experiment.

$$\bar{y}_i = \frac{1}{N_i} \sum_{u=1}^{N_i} y_i, u$$

S_i is the variance and is given as

$$s_i^2 = \frac{1}{N_i - 1} \sum_{u=1}^{N_i} (y_{i,u} - y_i)$$

Where   i = Experimental Number

u = Trial Number

N_i = Number of trials for experiment i

However, SN ratios are calculated in the case of minimizing and maximizing the performance characteristic respectively as follows:

$$SN_i = -10 \log \left(\sum_{u=1}^{N_i} \frac{y_u^2}{N_i} \right)$$

$$SN_i = -10 \, log \left(\frac{1}{N_i} \sum_{u=1}^{N_i} \frac{1}{y_u^2} \right)$$

The above equation of SN ratio can be better be expressed in terms of mean square deviation (MSD) below.

$$SN = -10 \, Log_{10} \, (\mathbf{MSD})$$

Where MSD for minimizing and maximizing performance characteristics are respectively defined as follows.

$$\mathbf{MSD} = (\mathbf{y_1^2} + \mathbf{y_2^2} + \mathbf{y_3^2} + \, ... \,)/ \, \mathbf{n}$$

$$\mathbf{MSD} = (\mathbf{1/y_1^2} + \mathbf{1/y_2^2} + \mathbf{1/y_3^2} + \, ... \,)/ \, \mathbf{n}$$

However, for a nominal value whereby a target value is set to be achieved, the MSD equation is defined as:

$$\mathbf{MSD = ((y_1 - m)^2 + (y_2 - m)^2 + ...) / n}$$

Hence, y1, y2… = Result of experiments, observations, or quality characteristics such as length, weight….

m = Target value of above results

n = Number of repetitions (y_i)

Signal-to-noise ratio is calculated for each experiment and the average for each or the target factor and level are derived which is followed by calculating the SN Ratio range for each of the parameter that are carefully tabulated. The range equation is given as

$$R = (\textbf{High SN} - \textbf{Low SN})$$

The effect of the variables on the process is proportional to the R value of a parameter which indicates that, the larger the R values for a parameter, the larger the effect of the process variables. The tables below depict the step in calculating the SN-ratio for each experiment.

TABLE 2. Computation of SN-Ratio

Experimental Number	Parameter 1	Parameter 2	Parameter 3	Parameter 4	SN-Ratio Y
1	1	1	1	1	SN1
2	2	2	1	3	SN2
3	3	3	1	2	SN3
4	2	1	2	2	SN4
5	3	2	2	2	SN5
6	1	3	2	1	SN6
7	3	1	3	2	SN7
8	1	2	3	3	SN8
9	2	3	3	1	SN9

Following the calculation of the SN-Ratio for each experiment, an average performance of each factor at each level is computed which is represented as A_1, A_2…, B_1, B_2…, C_1, C_2…, and so on, depending on the number of parameters and levels used. For a L_9 array with 4 parameters and 3 levels, the computation follows the sequence of A_1 to A_3, B_1 to B_3, C_1 to C_3 and D_1 to D_3. The letters A to D represents the parameters while the alphabet 1 to 3 represents the level with respect to L_9 with four parameter and three levels. Hence, the average effect of A_1 is computed as $A_1 = (Y_1 + Y_2 + Y_3) / N$. Thereafter, the average effect is graphically represented as the main effect. The optimum combination is selected from the

graph derived. For bigger the better, the optimum combination is A_1, B_2, C_2, D_2, E_1, F_2 and G_2 while for the smaller the better, the opposite is the case.
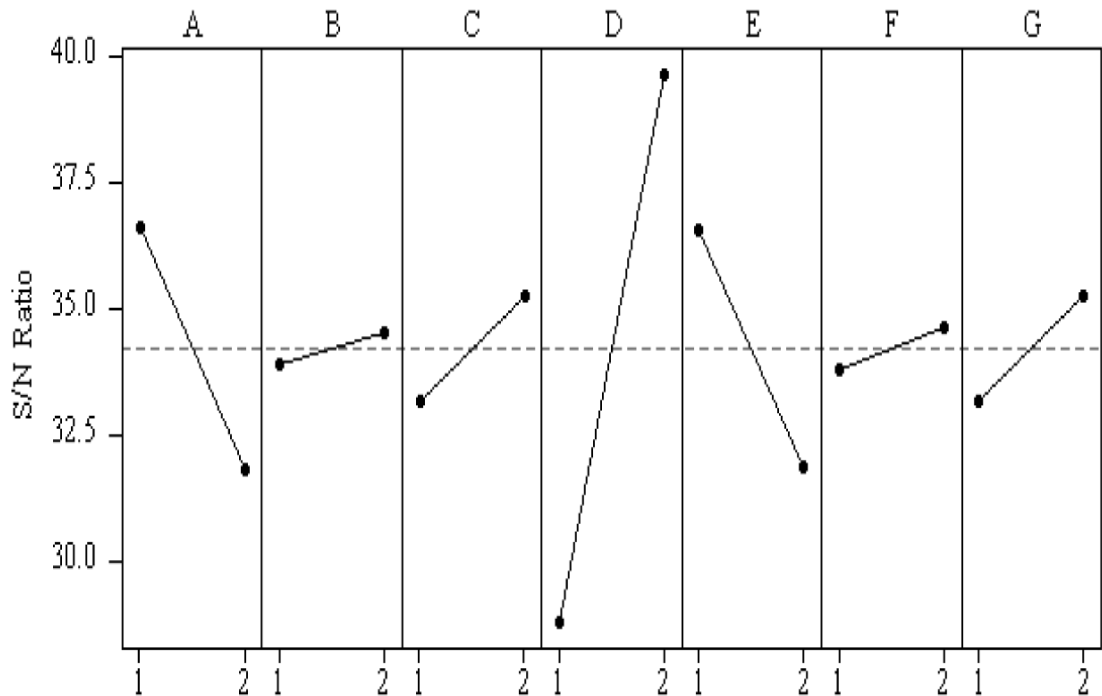
GRAPH 15. Main effect of Taguchi Method (Adapted from Sain Malaysiana 38 (3) (2009))

In order to provide a measure of confidence with respect to the third phase without directly analyzing the data, a standard statistical technique called Analysis of Variance (ANOVA) is used. The technique examines the variance of the data from which confidence is measured. When one dimensional experimental data are analyzed, it is regarded as one way ANOVA while a two way ANOVA involves an experimental data with two or more levels. Certain quantities are interrelated and defined mathematically which are computed in a standard tabular format. The quantities are defined below.

Total number of trials (n): The total number of trial which is the sum of the number of trials which is the sum of the number of trials at each level and defined mathematically as $n = n_1 + n_2 + \ldots + n_L$

Degree of freedom (DOF): This concept is the amount of information possibly determined from a data which is a factor that is equal to one less than the number of level. Experimental trial and number of runs indicated by n and r respectively are taking into consideration. DOF is indicated as $f_T = n \times r - 1$ while degree of freedom for sum of squares and error is given as $f_e = f_T - f_A - f_B - f_C$

Sum of squares (S): The total deviation of experimental data is determined by addition of each squared deviation and the sum of the deviation is denoted as $S_T = \sum_{i=1}^{n}(Yi - \hat{Y})^2$ by which $\hat{Y}$ is the average value of Y_i and where target value Y_o is involved, sum of square is given as $S_T = \sum_{i=1}^{n}(Yi - \hat{Y})^2 + n(\hat{Y} - Y_o)^2$. From the analysis of sum of squares of deviation, variance is determined by division of the sum of squares by degree of freedom denoted as $V = S_T/f$. Hence, variance is the measure of the distribution of data about the mean of the data. However, the general variance $\sigma^2 = \frac{1}{n}\sum_{i=1}^{n}(Yi - \hat{Y})^2$. More so, pure sum of squares for a given factor A is given as $S'_A = S_A - F_a \times V_e$

Variance Ratio (F): Variance ratio is the result due to the effect of factor and variance computed, subject to the DOF and variance error (S_e) to be zero. In such case, the interaction between factor A and B is not feasible and it is termed indeterminate. However, to get a reasonable result, a new error (V_e) can be calculated to by pooling which is the process of eliminating a factor and the model can be refitted. In that case, the sums of squares and degrees of freedom

corresponding to the eliminated terms are added into the residual sum of squares and degrees of freedom. F value is calculated as $F_e = V_e / V_e$

Above all, the percentage contribution for each factor is determined by the division of pure sum of squares (s) by total sum of squares (S) multiplied by 100, the percentage error can be determined from the sum of the percentage contributions less than 100.

4 EXPERIMENTAL PROCEDURE

This chapter discuss the experimental modulus operandi in-line with the Taguchi method. Recall that the experimental procedure is the second stage in the Taguchi procedure referred to as the conductive phase which trails the planning phase according to Roy 1990.

4.1 Description of the Pilot Scale Absorption-Desorption Packed Column

The absorption-Desorption column of Central Ostrobothnia University of Applied Sciences pilot scale, which is the case study in this research was designed relatively the same and functions plausibly as well as other contemporary packed column. The absorption packed column is made up of 100mm diameter sized and packed with Raschig packing. The diameter of packing was 10mm with the height of packing as 1700mm. The porosity of absorption column was 63%. The column was operated in a counter-current flow configuration with liquid solution entering at the top and the gas entering at the bottom. The desorption column otherwise known as the stripper has a diameter of 80mm while the diameter and height of the packing was 10mm and 850mm respectively. The stripper column was wrapped with foam of insulation for adiabatic operation. Two pumps and vessels attached are connected to the absorber and stripper that pump the washing liquid H_2O-MEA solution) to the columns. In between the absorption column and the stripper is the shell and tube reboiler which is a heat exchanger that allows all the

overhead vapour to return to the column. The source of heat is a centralized steam generator and the reboiler heat is pressured and regulated from a unit source at which it passes through a plate heat exchanger to the reboiler. Also connected to the absorber line was a CO_2 gas cylinder and infrared CO_2 gas analyser. The absorption packed column is automated and connected to a computer where certain control and information of the process can be accessed on the screen.

GRAPH 16. Absorption-Desorption packed column of COU

In the general analysis of the absorption-desorption packed column, flue gas is taken from CO_2 gas cylinder which interact in due proportion with air. The content of the CO_2 in the flue gas or feed was measured by carbon dioxide gas analyser. Monoethanolamine (MEA) is reacted with the flue gas containing CO_2 for chemical reaction phenomena. At the absorption column, scrubbing occurs at low temperature which releases CO_2 free gas at the top of the column while the CO_2-loaded amine solution leaving the absorption column stripped off the dissolved CO_2 with the aid of an open steam in the desorbing column. The stripped CO_2 is collected from the top of the stripper while the washing liquid regenerated; free of CO_2 is recycled back and cooled down to the absorber for re-use. This is also an economical means of avoiding the waste of the amine solution.
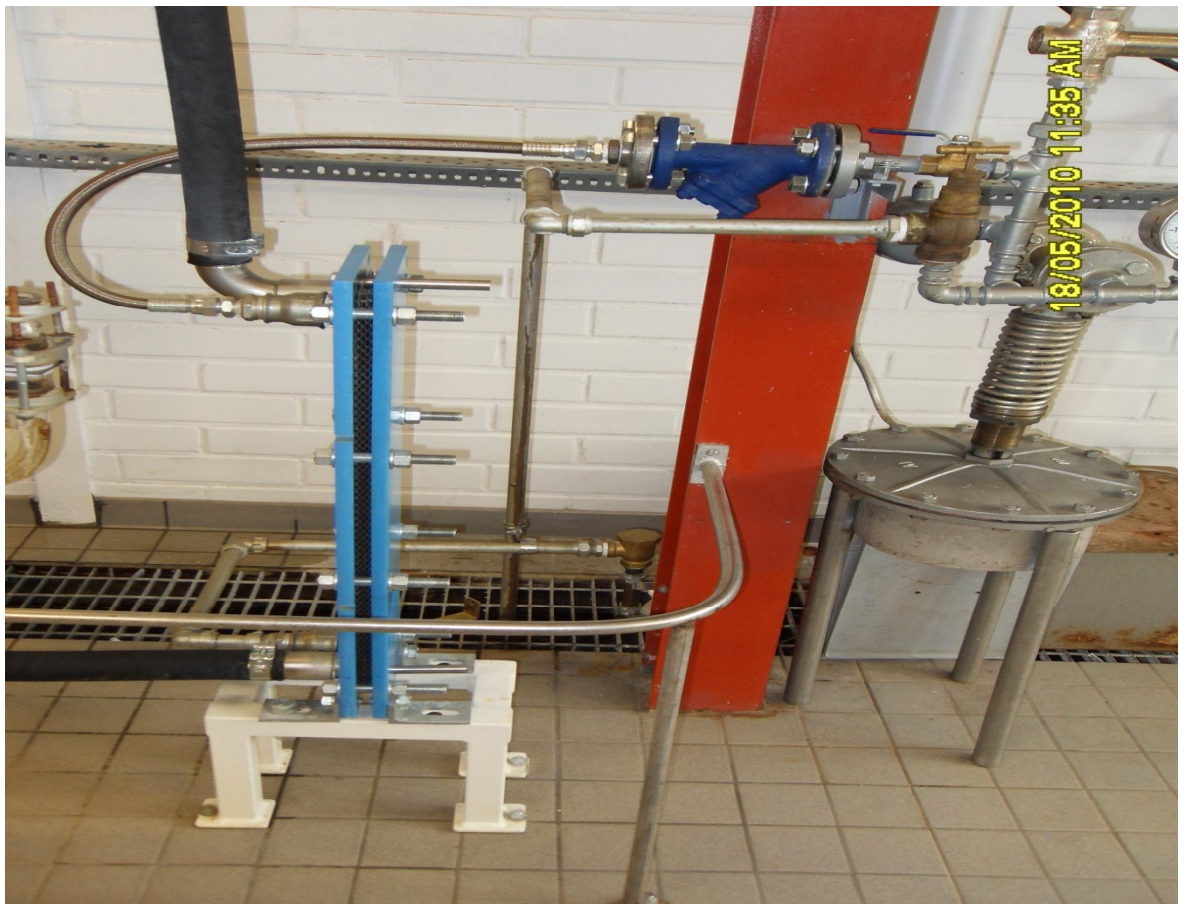
GRAPH 17. Reboiler heat gauge and plate heat exchanger
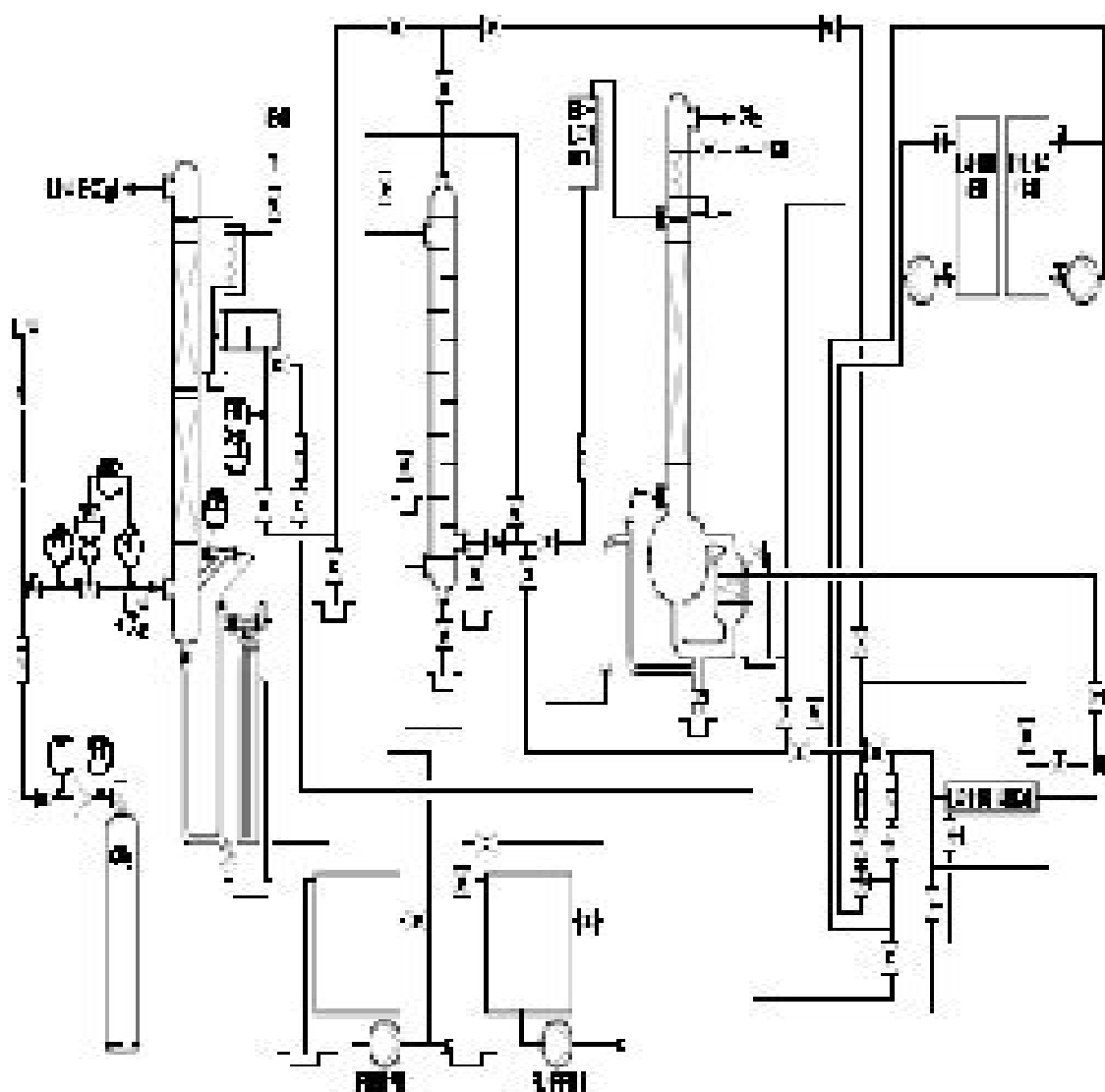
GRAPH 18. Model of Absorption/Desorption Packed Column of COU

4.2 Composition and Preparation of Amine Solution

Amine solution was prepared to a concentration of 1M of a volume that could fill the two vessels midway after stabilization of the solution around the packed columns. The amine, monoethanolamine (MEA) composition was as follows:

Molecular formula: $NH_2CH_2CH_2OH$

Molar Mass (M):   61,08G/mol

Percentage by Volume: 99, 5%

I liter of MEA was given as 1,02kg.

4.3 Set-up for the Determination of CO_2 in the Amine-Solution and Free MEA

An apparatus was setup for the determination of CO_2 in amine-solution which requires data from incoming and outgoing amine-solution by collecting the sample in a burette and measuring the pressure and temperature. The apparatus consist of a burette, a two sided glass tube and two pipettes of 2ml. 50% vol/vol phosphoric acid (H_3PO_4) set the reagent used and a seal liquid. The seal liquid was prepared from the solution of 800ml distilled water, 200g Na_2SO_4 and 40ml concentrated sulfuric acid (H_2SO_4). Furthermore, a titration apparatus was setup for the determination of Free MEA which will be titrated with 0, 1-N sulfuric acid using methyl red as indicator.
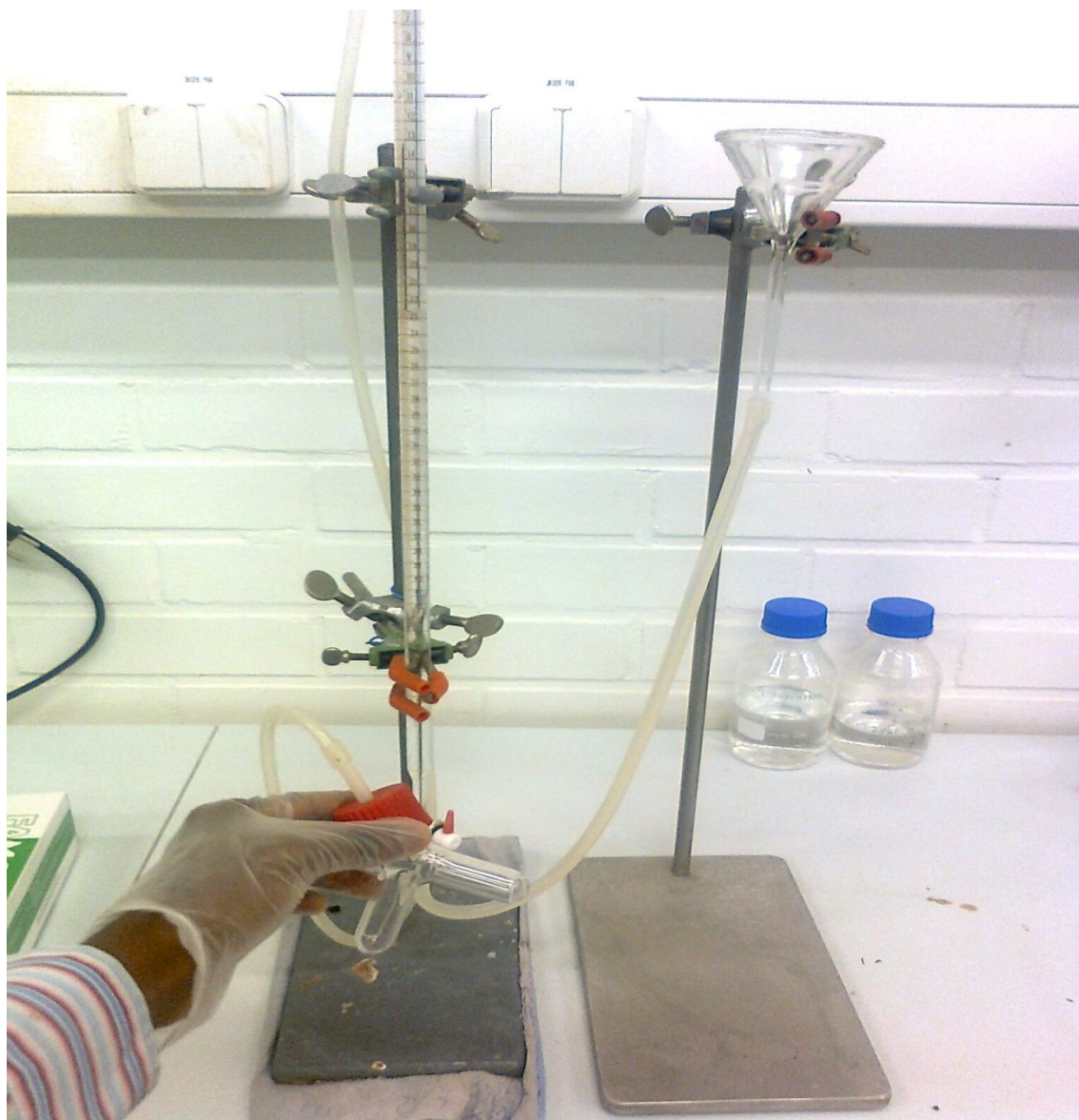
GRAPH 19. CO₂ gas analyzing equipment

4.4 Experimental Runs

The experiment was preceded by replenishing the packed column with new MEA-solution. The entire column was first drained via the tanks outlet and clean water was run through the column to rinse out previous left over MEA-solution. To

ascertain the purity and non-contamination of the column, the system was run with distilled water during the third cleansing. Thereafter, the column was filled with the fresh MEA-solution with the two columns half filled after stable circulation of the washing liquid. It should be recalled that the experimental design for this research work was based on Taguchi method with the adoption of the L9 orthogonal array subject to four (4) variables and three (3) levels. The four variables are the CO_2 load, Liquid flow, stripper feed power and reboiler heat pressure. All four variables were fluctuated based on the limit set for the experiment.

TABLE 3. Experimental Design based on Taguchi L9 orthogonal array.

Experiment Number	CO_2 Load (%)	Liquid Flow (L/min)	Reboiler Heat Pressure (Bar)	Stripper Feed Power (KW)
1	10	2.5	0.2	2
2	10	3	0.25	4
3	10	3.5	0.3	6
4	15	2.5	0.25	6
5	15	3	0.3	2
6	15	3.5	0.2	4
7	20	2.5	0.3	4
8	20	3	0.2	6
9	20	3.5	0.25	2

At the start of individual runs, the air and CO_2 gas flow was introduced at the desired rate into the system. The infrared gas analyzer verifies the desired

concentration of CO_2 present in the feed gas stream. The liquid flow was set and controlled from the computer screen while the reboiler heat pressure was adjusted from a pressure valve by turning anticlockwise and clockwise for increase and decrease pressure respectively. Lastly, the stripper feed power was adjusted analogically from the gauge measured in kilowatts. In relation to the Taguchi method, the four variables were varied at four levels which are presented in the table below and Taguchi proposed nine (9) experiments for that. Each experiment was repeated trice but one was eliminated for error reduction. All the sample of both the inlet and outlet liquid flow was taken and analyzed for the amount of CO_2 present in it with the aid of the gas analyzer with the values recorded in milliliters (ml). A confirmatory experiment was carried after the Taguchi calculations to establish the optimum value. As principled, the CO_2 absorbed was also investigated to determine the change in the absorption capacity at the optimal level for flooding.

5 DATA ANALYSIS AND CALCULATIONS

Sequel to the completion of all nine experimental runs as presented in Table 1, data from the runs are presented as follows in table 4, 5 and 6 for the first, second and third runs respectively.

TABLE 4. Experimental Data (Test run 1)

Exp Number	Gas Temp in	Gas Temp out	Liq Temp in	Liq Temp out	Stripper Liquid Temp in	Stripper Pressure Drop	Absorber Pressure Drop	CO_2 Load in	CO_2 Load out	CO_2 in	CO_2 out
Unit →	^oC	^oC	^oC	^oC	^oC	mbar	mbar	%	%	ml	ml
1	16.0	12.6	10.7	9.5	55.1	1.8	3.6	10.05	6.70	20.0	17.7
2	16.7	10.0	10.0	10.1	64.9	2.2	4.0	10.18	7.30	19.9	18.3
3	16.5	10.4	11.0	11.2	71.9	2.7	4.3	9.48	7.53	19.6	18.5
4	16.6	12.2	9.6	11.1	85.7	50.2	4.9	14.90	9.12	20.1	12.1
5	16.7	10.8	9.8	11.6	54.9	2.7	5.4	14.26	11.0	21.0	19.1
6	14.7	10.9	10.9	11.0	62.5	3.1	7.2	15.60	11.9	22.9	20.5
7	7.9	9.2	10.5	11.8	70.7	3.2	5.2	20.66	9.06	23.7	18.7
8	11.8	10.2	12.1	13.9	80.3		5.9	19.6	12.6	22.1	17.7
9	11.9	10.6	13.2	13.7	54.7		7.9	19.80	14.3	23.3	22.0

TABLE 5. Experimental Data (Test run 2)

Exp Number	Gas Temp in	Gas Temp out	Liq Temp in	Liq Temp out	Stripper Liquid Temp in	Stripper Pressure Drop	Absorber Pressure Drop	CO_2 Load in	CO_2 Load out	CO_2 in	CO_2 out
Unit →	°C	°C	°C	°C	°C	mbar	mbar	%	%	ml	ml
1	17.9	9.8	12.0	11.2	56.1	1.1	4.0	10.60	6.17	24.5	22.2
2	18.8	10.4	13.4	12.7	68.1	1.4	5.35	9.80	7.31	24.8	21.7
3	19.3	10.8	13.4	13.9	75.2	55	6.1	10.70	7.95	24	20.0
4	16.6	9.8	10.8	12.8	85.9	46	5.4	16.00	9.71	23.4	17.5
5	15.8	10.5	12.2	12.8	59.4	-2.4	6.4	15.4	11.3	22.9	21.6
6	16.6	11.3	13.6	13.9	64.2	-0.2	8.0	15.2	12.2	26.0	23.5
7	12.5	9.3	11.8	13.5	74.9	52.6	6.5	20.5	14.6	25.8	18.2
8	13.3	10.7	14.5	13.8	81.4	20	8.2	19.6	15.6	25.3	18.2
9	14.5	10.5	13.9	13.8	56.1	-2.7	9.0	19.6	16.2	25.5	23.6

TABLE 6. Experimental Data (Test run 3)

Exp Number	Gas Temp in	Gas Temp out	Liq Temp in	Liq Temp out	Stripper Liquid Temp in	Stripper Pressure Drop	Absorber Pressure Drop	CO_2 Load in	CO_2 Load out	CO_2 in	CO_2 out
Unit →	$°C$	$°C$	$°C$	$°C$	$°C$	mbar	mbar	%	%	ml	ml
1	15.5	8.7	12.0	11.9	45.31	2.1	3.9	10.0	6.04	25.5	23.3
2	15.9	8.6	12.9	13.3	67.8	1.4	4.9	10.2	7.03	25.5	22.8
3	17.7	10.2	12.9	13.3	67.8	1.4	4.9	10.2	8.8	25.5	22.8
4	15.1	9.2	11.2	13.2	86.4	50.2	6.1	15.04	10.64	25.9	18.3
5	12.9	10.1	13.8	12.8	55.7	1.8	5.9	15.5	9.81	28.0	25.0
6	13.8	11.2	14.8	14.2	62.2	1.6	7.4	15.2	11.4	28.4	26.2
7	10.9	9.7	12.2	13.1	70.2	55.2	7.3	20.2	15.2	30.1	20.8
8	10.8	10.3	13.8	14.7	79.2	57.7	8.2	20.1	16.04	28..2	22.5
9	9.7	10.9	15.4	14.5	51.7	1.2	9.3	20.50	15.06	29.9	27.8

5.1 Determination of pressure drop at approach to pressure Drop

In relation to the flooding and the stripper pressure drop and subject to the Taguchi analysis using the Signal-to-noise ratio, the analysis is carried out based on excel calculations for the parameters and pressure drop results from table 1 and 2 respectively. Recall that absorption in packed column is at its best efficiency at when flooding is near (70% to 80%). Also, considering the efficiency of CO_2

absorption to be elevated, the pressure drop was determined at 80% flooding with the use of Packed Column Calculator, a product of Katmar Software.

TABLE 7. Approach to 80% flooding using Robbins correlation (1991)

TABLE 8. Detailed Calculation for the approach to 80% flooding using Robbins correlation (1991)

Detailed Calculations		
Minimum Liquid Rate for Wetting	2.44	kg/s
Actual Liquid Flow as % of Minimum	1.64	%
Column Cross Sectional Area	3.134	m2
Gas Superficial Velocity	0.437	m/s
Liquid Superficial Velocity	1.28e-5	m/s
Flooding Pressure Drop	2450.0	Pa/m
Total Pressure Drop	858.2	Pa
Bed Volume	2.664	m3
Bed Mass - Dry	2557.1	kg
Bed Mass - Full of Liquid	4208.5	kg
Bed Mass - Full of Water	4208.5	kg
Total Bed Surface Area	2075.0	m2
Total Number of Packing Pieces	80520	#

Close

With the pressure drop per unit length through the packed bed given as pa/m, the bed height for the Desorber was 0.85m and 1pa = 0.01mbar; the pressure drop was converted to pa and then, to mbar unit corresponding to the unit of the COU packed column pressure drop.

TABLE 9. Conversion of pressure drops from pa/m to mbar unit

Percentage Flooding >	80 %	Unit	80 %	Unit	80 %	Unit
Pressure Drop	1009.6	pa/m	1187.8	pa	11.87765	mbar
Total Pressure Drop	858.2	pa	858.2	pa	8.582	mbar
Flooding pressure Drop	2450	pa/m	2882.4	pa	28.82353	mbar

5.2 Optimum Result using SN-Ratio based on Nominal the Best

Sequel to the Taguchi design, the nominal value for the pressure drop is 11.88mbar at 80% flooding. This nominal value is used to compute the Taguchi analysis which is to determine the best combinations for the Factors that will result to the pressure drop 11.88mbar at 80% flooding.

TABLE 10. L9 Experimental Data using Signal to Noise Ratio (Nominal the Best)

Exp Number	Factor 1	Factor 2	Factor 3	Factor 4	Number of Repitition			Signal to Noise Ratio Y
			Reboiler Heat Pressure	Stripper Feed Power	Stripper Pressure Drop (mbar)			
	CO_2 Load	Liquid Flow						
Unit >	%		Bar	(KW)	1	2	3	
		(L/min)						
1	10	2,5	0,2	2	1.8		2.1	-19.94
2	10	3	0,25	4		1.4	1.4	-20.99
3	10	3,5	0,3	6		55	58.9	-30.70
4	15	2,5	0,25	6	50.2		50.2	-31.67
5	15	3	0,3	2	2.7		1.8	-19.68
6	15	3,5	0,2	4	3.1		1.6	-19.61

7	20	2,5	0,3	4		52.6	55.2	-30.04
8	20	3	0,2	6		20	57.7	-30.49
9	20	3,5	0,25	2		1.2	1.2	-21.06

Target Value = 11.88   **Average SN-Ratio** = -24.91

For unit 1.

$$\text{MSD} = \frac{[(1.8 - 11.88)^2 + (2.1 - 11.88)^2]}{2} \qquad = 98.63$$

Therefore, S/N Ratio = - 10 $\log_{10}$ (98.63)   = -19.94

The same calculation procedure is repeated for unit 1 to unit 9 above.

The average performance of each factor at each level was computed and tabulated below with letter A to D representing Factor 1 to 4 respectively and alphabet 1 to 3 representing the three levels in that order. The plot of the graph of the factor against its response on each level is referred to as the main effect in Graph 1.

Table 11. Computation of Average Performance

	A		B		C		D
Â1	-23.65	**B1**	-27.22	**C1**	-23.35	**D1**	-20.23
Â2	-23.88	**B2**	-23.72	**C2**	-24.57	**D2**	-23.54
Â3	-27.20	**B3**	-23.79	**C3**	-26.81	**D3**	-30.96
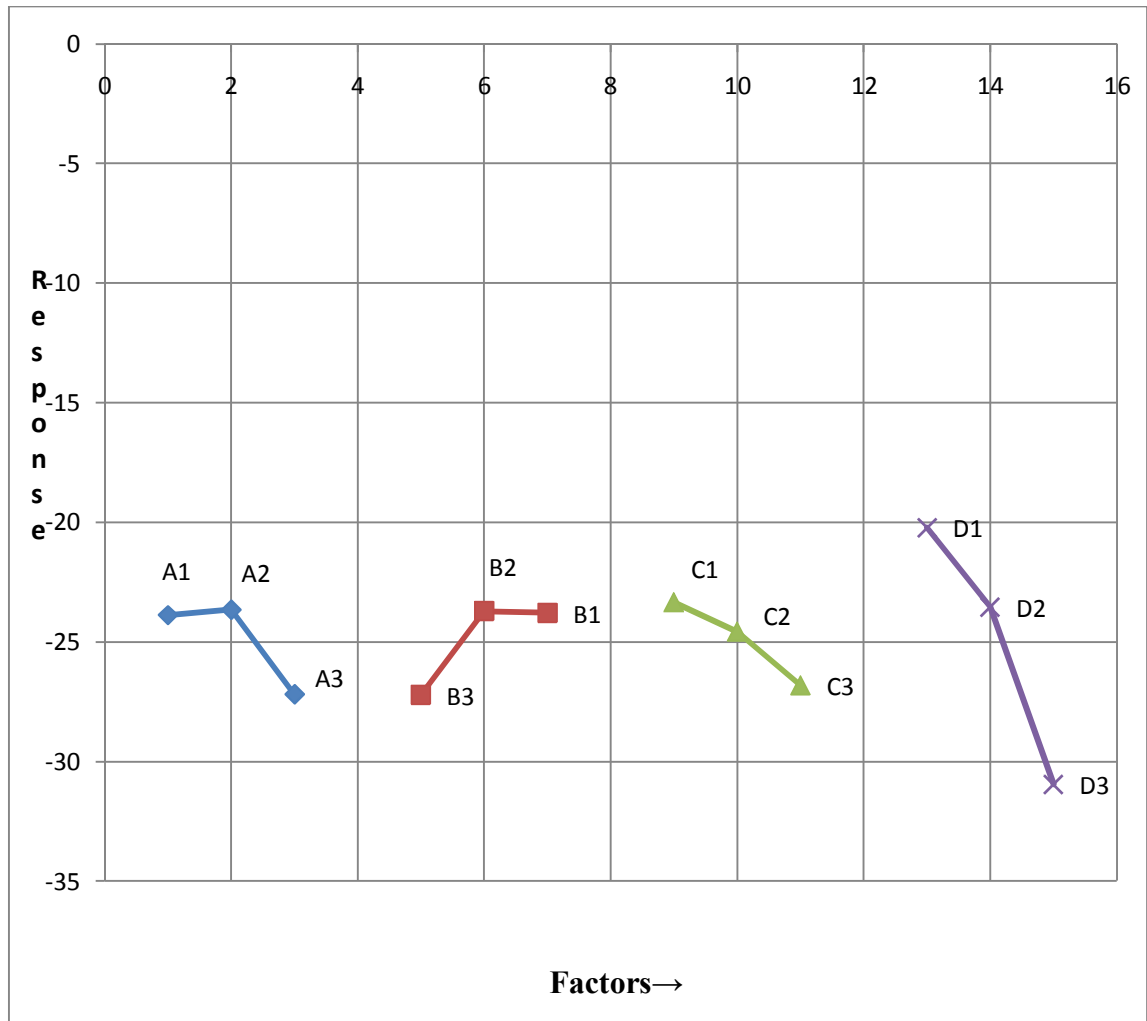
GRAPH 20. Plot of Parameters` Main Effect

From table 11 and Graph 20 considering the average signal to noise ratio, the optimum combinations are A_1, B_3, C_2, and D_2. Hence, the optimal signal to noise ratio is calculated from the addition of average performance and combination of A_1, B_3, C_2, and D_2 above average performance thus:

Y_{opt} = -24.91 + [-23.88 − (-24.91)] + [-23.79 - (-24.91)] + [-24.57 - (-24.91)]

   + [-23.54 - (-24.91)]

   = -21. 06.

Since S/N ratio was used, the Y_{opt} is converted back to the scale of units of the pressure drop.

$MSD = 10^{(-21.06)/10} = 127.52$

Then, $Y_{esp} = (127.52)^{1/2} = 11.29 mbar$

Consequently, the optimum expected pressure drop at 80 % flooding is 11.29mbar which is comparable to be nominal value of 11.88mbar.

Table 12. Optimal Combination and expected yield

Exp Number	Factor 1	Factor 2	Factor 3	Factor 4	Expected Yield	Signal to Noise Ratio Y
		Liquid Flow	Reboiler Heat Pressure	Stripper Feed Temp	Stripper Pressure Drop	
	CO_2 Load					
Unit >	%	(L/min)	Bar	(KW)	mbar	
1	15	3.5	0.25	4	11.29	-21.06

5.3 Analysis of Variance (ANOVA)

The analysis of variance is relatively the third stage of the Taguchi method which deals with the experimental results and its statistical analysis. The ANOVA, as

abruptly called, was used to determine the relative contribution of the factors by comparing their variances. However, the result in Table 13 was pooled to obtain the final ANOVA result in Table 14.

Table 13. ANOVA Table for Flooding Experiment

Factors	f	S	v	F	s	P (%)
	DOF	Sum of Squares	Variance	Variance Ratio	Sum of Squares	% Contribution
A	2	23.63	11.82			9.56
B	2	23.95	11.98			9.69
C	2	18.48	9.24			7.48
D	2	181.03	90.52			73.26
Error (e)	9	0	0			0
Totals	17	247.09				100

Table 14. ANOVA Table for Flooding Experiment with pooling

Factors	f	S	v	F	s	P (%)
	DOF	Sum of Squares	Variance	Variance Ratio	Sum of Squares	% Contribution
A	2	23.63	11.82	5.5	19.33	9.82
B	2	23.95	11.98	5.58	19.66	9.96
C	Pooled			Pooled		Pooled
D	2	181.03	90.52	42.13	176.73	73.52
Error (e) /Others	11	23.63	2.15			6.7
Totals	17	247.09				100

5.4 Confirmatory Run

As required by Taguchi analysis, a confirmatory experiment was conducted to verify the optimal result of the Taguchi Analysis. The result presented was limited to the key data needed.

Table 15. Confirmatory Run

Exp Number	Stripper		Absorber Pressure Drop	CO₂ Load in	CO₂ Load out	CO₂ removed
	Liq Temp in	Pressure Drop				
Units →	^oC	mbar	mbar	%	%	ml
1	88.5	11.7	8.5	15.98	10.61	5.4
2	89.1	11.8	9.3	15.53	11.34	4.4
3	91.6	11.0	9.7	15.09	11.24	5.1
Average	89.7	11.5	9.2	15.5	11.1	4.97

6 DISCUSSION AND CONCLUSIONS

In this thesis work, investigation was carried out to unravel the flooding problem in a pilot scale absorption desorption packed column which had a set limit. Relevant literature was reviewed and Katmar Software for calculations in packed columns was used during the research work. The preliminary experimental data was designed within the set limit and Taguchi method was introduced to simplify the research process with the view to optimizing the packed column efficiency. Though, the aim of this research work is to resolve the problem of flooding in the absorption-desorption packed column, it should be noted that the purpose of absorption column is the removal of CO_2, thus, this work also lay emphasis on it and in accordance to literatures referenced in previous chapters and evidenced in result. ANOVA analysis was used in determining the variance in the results.

This novel method helped reduce the experimental runs to nine. The result gathered from the nine experimental runs show a great deal of flooding in four of the nine experiments which are Experiment 3, 4, 8 and 9. It was observed that the flooding occurred at the maximum stripper feed power of all the six kilowatts (6 KW) combinations. Furthermore, CO_2 was observed to be better stripped in the experiments where flooding occurred, more than 100% better than the experiment where flooding did not occur. This justifies the reason for operating the columns close to flooding. The absorption column was designed to operate at 80% flooding for the dual purpose of optimal CO_2 removal. Theoretically, the pressure drop was determined to be 11.88mbar. This hypothetical pressure drop value was used in the Taguchi analysis as the target value for the pressure drop as required for the

Nominal-the-Best in the Taguchi analysis. Practically, the combination of the factors derived from the Taguchi analysis gave a pressure drop not exceeding the target value which is at 80% flooding and was confirmed experimentally.

A suitable result of standard analysis presents the signal to noise ratio of the two repetitions for each experimental run at the extreme right hand column of Table 10. Signal to Noise Ratio was used in the Taguchi analysis because it provides a selection of the optimum level based on the least variation around the target pressure drop referred to the nominal value and also the average value closest to it. With the target value of 11.88mbar at 80% flooding, the optimum combination was analyzed to be A_2, B_3, C_2 and D_2. This is interpreted to be 15% CO_2 load, 3.5 l/min liquid flow, 0.25bar reboiler heat and stripper feed temperature of 4KW. According to the Taguchi analysis, this combination will give a pressure drop of 11.29mbar after the conversion of the signal to noise ratio back to the unit scale, which is just a slight variation from the nominal value. With this combination, a confirmatory experiment was conducted to establish the validity of the result which was obtained to be 11.5mbar on average of the three repetitions. A slight variation of the confirmatory run by 3.2% and 1.8% from the nominal value and empirical value may have been due to error from instability of the CO_2 load.

An analysis of variance was conducted to provide a measure of variance to the results of the experiment without a direct influence on it as presented in Table 13. The ANOVA analysis was pooled in Table 14, because the pure sum of squares (s) cannot be calculated since the variance of error term (Ve) was zero. The percentage contribution which was first calculated from sum of squares (S) was recalculated in the pooled ANOVA from the pure sum of squares (s). Consequently, the

percentage contribution was determined to be 9.82% for the CO_2 load (A), 9.96% Liquid Flow rate (B) and, 73.52% for the stripper feed Power (D). The high percentage contribution of the stripper feed power, confirms the reason for flooding in all its maximum levels.

Conclusively, and in a way of answering the research questions in chapter 1, all the four factors have fractional effect on flooding in the pilot scale absorption-desorption packed column. However, Stripper Feed Power has the most influence on flooding at the stripper over the Liquid Flow and CO_2 load in the percentage ratio of 73.5% to about 10% and 9.8% respectively. Thereby, the optimum combination of the factors to interact and obtain 80% flooding is 15% CO_2 Load, 3.5 L/min Liquid flow, 0.25 bar reboiler heat Pressure, and 4 KW Stripper Feed Power with a pressure drop of 11.5mbar with the same condition of the packed column during the time under review. Though, types of packing definitely influence flooding rate in packed columns but raschig packing was maintained throughout the whole experiment. Any introduction of other type of packing will bring about alteration in the pressure drop at the Stripper. With respect to this, recommendations were proffered in the next sub-chapter.

The result of this research work shows a great correlation between pressure drop and flooding that stripper pressure drop increases with flooding and vice versa. Thus, flooding is accompanied by a dramatic increase in pressure, resulting in inefficient operation. It has been reported in literature that CO_2 absorbs better at near flooding and this is apparent in the result of this research where experimental runs with flooding absorbs quite a large amount of CO_2. At optimum condition, CO_2 absorbed was improved, which indicates a great improvement in the column

efficiency. Succinctly, the general implication of this research work is the optimization of the absorption-desorption packed column to the state of approach to 80% flooding for optimal efficiency of the process with the combination of the four factors under considerations using Taguchi experimental method. This optimization at this pilot scale would prove valuable in industry, allowing them to more precisely scrutinize operations and enabling such processes closer to flooding to maximizing mass transfer efficiency. Nonetheless, recommendations for further research were given.

6.1 Recommendations

Taguchi analysis helped to facilitate the simplicity of time management, cost and loss functions. However, error during the analysis will be best reduced drastically as recommended by Dr. Taguchi himself by adequate standardization and upgrade of control device. Furthermore, in a bid to increase efficiency of the Packed columns to achieve better CO_2 absorption at reduced pressure drop and flooding, literatures recommends in line with this research work, the upgrade of the packed column from the Raschig to a structured packing, which operates at lower pressure drop. However, a new optimum combination will be required to determine the best possible result at the condition.

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Model of the COU Chemplant Absorption-Desorption Unit
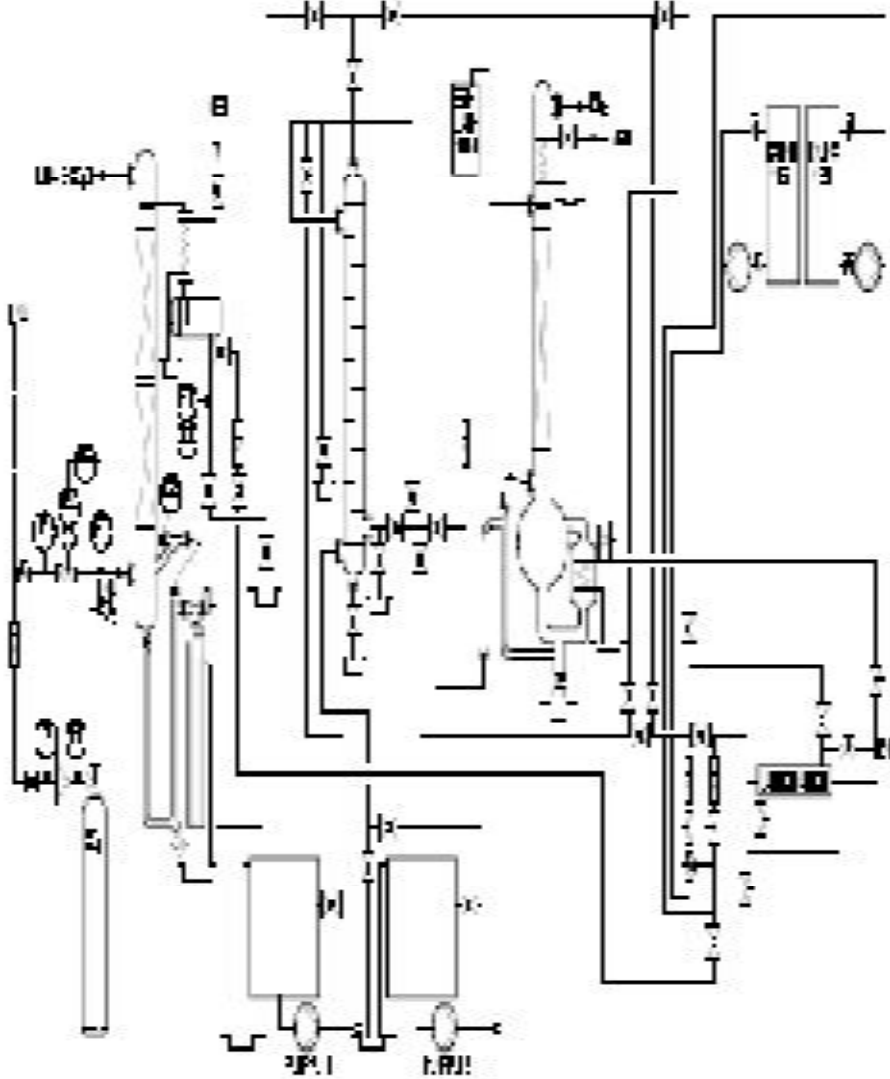
Experimental Design based on L9 Orthogonal Array

Experiment Number	CO$_2$ Load (%)	Liquid Flow (L/min)	Reboiler Heat Pressure (Bar)	Stripper Feed Power (KW)
1	10	2.5	0.2	2
2	10	3	0.25	4
3	10	3.5	0.3	6
4	15	2.5	0.25	6
5	15	3	0.3	2
6	15	3.5	0.2	4
7	20	2.5	0.3	4
8	20	3	0.2	6
9	20	3.5	0.25	2

Experimental Data (First Run)

Exp Number	Gas Temp in	Gas Temp out	Liq Temp in	Liq Temp out	Stripper Liquid Temp in	Stripper Pressure Drop	Absorber Pressure Drop	CO_2 Load in	CO_2 Load out	CO_2 in	CO_2 out
Unit →	°C	°C	°C	°C	°C	mbar	mbar	%	%	ml	ml
1	16.0	12.6	10.7	9.5	55.1	1.8	3.6	10.05	6.70	20.0	17.7
2	16.7	10.0	10.0	10.1	64.9	2.2	4.0	10.18	7.30	19.9	18.3
3	16.5	10.4	11.0	11.2	71.9	2.7	4.3	9.48	7.53	19.6	18.5
4	16.6	12.2	9.6	11.1	85.7	50.2	4.9	14.90	9.12	20.1	12.1
5	16.7	10.8	9.8	11.6	54.9	2.7	5.4	14.26	11.0	21.0	19.1
6	14.7	10.9	10.9	11.0	62.5	3.1	7.2	15.60	11.9	22.9	20.5
7	7.9	9.2	10.5	11.8	70.7	3.2	5.2	20.66	9.06	23.7	18.7
8	11.8	10.2	12.1	13.9	80.3		5.9	19.6	12.6	22.1	17.7
9	11.9	10.6	13.2	13.7	54.7		7.9	19.80	14.3	23.3	22.0

Experimental Data (Second Run)

Exp Number	Gas Temp in	Gas Temp out	Liq Temp in	Liq Temp out	Stripper Liquid Temp in	Stripper Pressure Drop	Absorber Pressure Drop	CO_2 Load in	CO_2 Load out	CO_2 in	CO_2 out
Unit →	°C	°C	°C	°C	°C	mbar	mbar	%	%	ml	ml
1	17.9	9.8	12.0	11.2	56.1	1.1	4.0	10.60	6.17	24.5	22.2
2	18.8	10.4	13.4	12.7	68.1	1.4	5.35	9.80	7.31	24.8	21.7
3	19.3	10.8	13.4	13.9	75.2	55	6.1	10.70	7.95	24	20.0
4	16.6	9.8	10.8	12.8	85.9	46	5.4	16.00	9.71	23.4	17.5
5	15.8	10.5	12.2	12.8	59.4	-2.4	6.4	15.4	11.3	22.9	21.6
6	16.6	11.3	13.6	13.9	64.2	-0.2	8.0	15.2	12.2	26.0	23.5
7	12.5	9.3	11.8	13.5	74.9	52.6	6.5	20.5	14.6	25.8	18.2
8	13.3	10.7	14.5	13.8	81.4	20	8.2	19.6	15.6	25.3	18.2
9	14.5	10.5	13.9	13.8	56.1	-2.7	9.0	19.6	16.2	25.5	23.6

Experimental Data (Third Run)

Exp Number	Gas Temp in	Gas Temp out	Liq Temp in	Liq Temp out	Stripper Liquid Temp in	Stripper Pressure Drop	Absorber Pressure Drop	CO_2 Load in	CO_2 Load out	CO_2 in	CO_2 out
Unit →	°C	°C	°C	°C	°C	mbar	mbar	%	%	ml	ml
1	15.5	8.7	12.0	11.9	45.31	2.1	3.9	10.0	6.04	25.5	23.3
2	15.9	8.6	12.9	13.3	67.8	1.4	4.9	10.2	7.03	25.5	22.8
3	17.7	10.2	12.9	13.3	67.8	1.4	4.9	10.2	8.8	25.5	22.8
4	15.1	9.2	11.2	13.2	86.4	50.2	6.1	15.04	10.64	25.9	18.3
5	12.9	10.1	13.8	12.8	55.7	1.8	5.9	15.5	9.81	28.0	25.0
6	13.8	11.2	14.8	14.2	62.2	1.6	7.4	15.2	11.4	28.4	26.2
7	10.9	9.7	12.2	13.1	70.2	55.2	7.3	20.2	15.2	30.1	20.8
8	10.8	10.3	13.8	14.7	79.2	57.7	8.2	20.1	16.04	28..2	22.5
9	9.7	10.9	15.4	14.5	51.7	1.2	9.3	20.50	15.06	29.9	27.8

ANOVA Flooding Analysis with pooling

Factors	f	S	v	F	s	P (%)
	DOF	Sum of Squares	Variance	Variance Ratio	Sum of Squares	% Contribution
A	2	23.63	11.82	5.5	19.33	9.82
B	2	23.95	11.98	5.58	19.66	9.96
C		Pooled		Pooled		Pooled
D	2	181.03	90.52	42.13	176.73	73.52
Error (e) /Others	11	23.63	2.15			6.7
Totals	17	247.09				100