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Research paper

Selection of amine combination for CO_2 capture in a packed bed scrubber

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Abstract

This investigation was to test different blends of tertiary amine; triethanolamine (TEA) into primary amine; Monoethanolamine (MEA) used to capture CO_2 in packed bed scrubber with recycle stream. Four different operating parameters: Amine Combination (A), Dilution Water (B), Liquid Flow rate (C), and Gas Flow rate (D) were varied to study the behavior of the system. Moreover, Taguchi method was employed to establish the order of importance of different parameters in the process. A 4 factor and 3 level was chosen for the study and it was explored using L9 (3^4) orthogonal array design. According to 3-level design 0%, 20% and 30% were chosen for A, 10%, 20% and 30% for B, 1 $Lmin^{-1}$, 1.5 $Lmin^{-1}$ and 2 $Lmin^{-1}$ for C, 8 $Lmin^{-1}$, 16 $Lmin^{-1}$ and 20 $Lmin^{-1}$ for D. To understand the effectiveness order of different operating parameters, three factors namely Absorption efficiency (E), Absorption Rate (RA), and Scrubbing Factor (E) were calculated upon which the order was compared. The highest efficiency of 92.2% was achieved with 20% TEA. However, with 30% of TEA and 20% solvent mix maximum scrubbing factor (E) of 0.63 mol-CO_2/mol-Solvent was achieved. As per Taguchi analysis the significance sequence for absorption efficiency (ϕ) was B > C > D > A; for absorption rate C > B > D > A and for scrubbing factor it was C > B > D > A. The blending of tertiary amine seemed advantageous for carbon dioxide capture process.

Keywords: Absorption efficiency; Absorption rate; Monoethanolamine (MEA); Scrubbing factor; Taguchi method; Triethanolamine (TEA)

1. Introduction

Global warming issue is at its alarming level worldwide nowadays. Catastrophic global warming is an intense form of greenhouse effect. Greenhouse gases are responsible for such effects. In an adequate amount such effect is necessary to keep the average temperature of earth's surface enough to sustain life but addition of more and more greenhouse gases into the atmosphere is intensifying the process and altering the conditions of earth in untoward manner. CO_2, CH_4, N_2O, and chlorofluorocarbons (CFCs) are different greenhouse gases listed by IPCC. Roughly one-third of the solar energy that reaches the top of Earth's atmosphere is reflected directly back to space. The remaining two-thirds is absorbed by the surface and, to a lesser extent, by the greenhouse gases in the atmosphere [1]. The more the greenhouse gases in the atmosphere the more radiation they absorb leading to the higher temperature of the earth's surface. It has become renowned as one of the gravest environmental issues to catch the attention of the globe in recent decades. CO_2, more than any other greenhouse gases, has contributed the most to climate change. As CO_2 carries the highest radiation, it contributes significantly to the climate change. Moreover, it has a remarkably long lifespan with half-life of 100 years in the atmosphere [2]. There are multiple sources like agriculture processing, industry activities, electricity production, mining etc. from where CO_2 is added directly into the atmosphere. Most of the sources of CO_2 use fossil fuels as the energy source directly or indirectly. According to the global carbon budget 87% of the CO_2 comes from the burning of the fossil fuels [3]. Considering the harm done by CO_2, capturing such greenhouse gas before it is vented out into atmosphere was taken as a challenge to solve. Furthermore, due to the high costs usually ascribed to separation and purification operations in process industries, the search for low-cost, non-conventional and alternatives has generated much academic research worldwide [4].

The most natural solution to the CO_2 dumping problem is reducing the use of fossil fuels but practicality is a major issue. Renewable energy and nuclear energy are at rise but according to international energy outlook 2014, 80% of the world energy

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through 2040 will be provided by fossil fuels. By then, it is necessary to mitigate the condition raised by the use of fossil fuel by reducing the amount of CO_2 being dumped in the atmosphere. A conventional method of absorbing CO_2 by CaO is studied by using ASPEN Plus process simulator to understand-steady state model of air–steam gasification of biomass [5]. The model will analyze the effect of CaO sorbent for in-situ CO_2 capture.

There are many methods adopted for CO_2 capturing like using physical adsorbent [6], catalytic conversion [7] and capturing CO_2 using different solvents like Monoethanol Amine (MEA), Ammonia, tetrahydrofuran, and tetra-n-butyl ammonium bromide [8–10]. Among all, the solvent scrubbing technique is considered to be the most advanced post-combustion capture technology [6].

Focusing on solvent based CO_2 capturing, MEA is one of the predominant solvents due to its commercial availability, relatively low cost, fast absorption rate and rich experience in industrial applications [11]. However, there are limitations of using MEA solution alone, i.e. degradation in the presence of O_2, SO_x and NO_x [12], corrosive nature of MEA [13] and high regeneration energy requirements [11]. To mitigate such limitations MEA is blended with other solvents like Triethanolamine (TEA) [14], 2-amino-2-methyl-1-propanol (AMP), benzylamine (BZA) [15], and methyldiethanolamine (MDEA) [16]. There are several advantages of blending these amines: viz.,

- Improved thermodynamic efficiency [17].
- Reduction in issues relating to degradation and operation of the solvent caused by corrosion [18].
- Flexibility in the range of amines available to tailor and optimize the composition of the solvent to achieve the highest absorption efficiency.
- High absorption rates observed in single amine solvents can often be maintained in blends of the individual components.
- Energy requirement for solvent regeneration can be reduced [19].

With significant CO_2 loading capacity TEA shows great regeneration efficiency. Additionally, with limited literature available on TEA blended MEA, there is wide scope of investigating the potential of TEA as a blended amine. Moreover, MEA + TEA blended amines system was suggested in order to capitalize the performance of TEA (tertiary) and MEA (primary) amines. The low energy requirement for regeneration and high absorption capacity of tertiary amine coupled with the fast reaction kinetics of primary amine are an ideal combination for CO_2 sequestration.

The present research was commenced primarily for: (1) Investigating the potential of TEA as a blend for MEA solution in the scrubbing of CO_2 by a continuous process; (2) Incorporating Recycle stream of solvent in the continuous scrubbing and observing equilibrium of the process; and (3) Evaluating the influence of different parameters on the process with the help of Taguchi analysis.

2. Materials and methods

MEA and TEA of laboratory grade were used as solvents. A CO_2 (99.99% pure) cylinder was used as the source of the inlet gas. Deionized water was used during every cycle of the experiment.

2.1. Experimental set-up

A Pyrex glass column of 1.15 m height and internal diameter of 0.285 m served as the scrubbing column as given in Fig. 1.

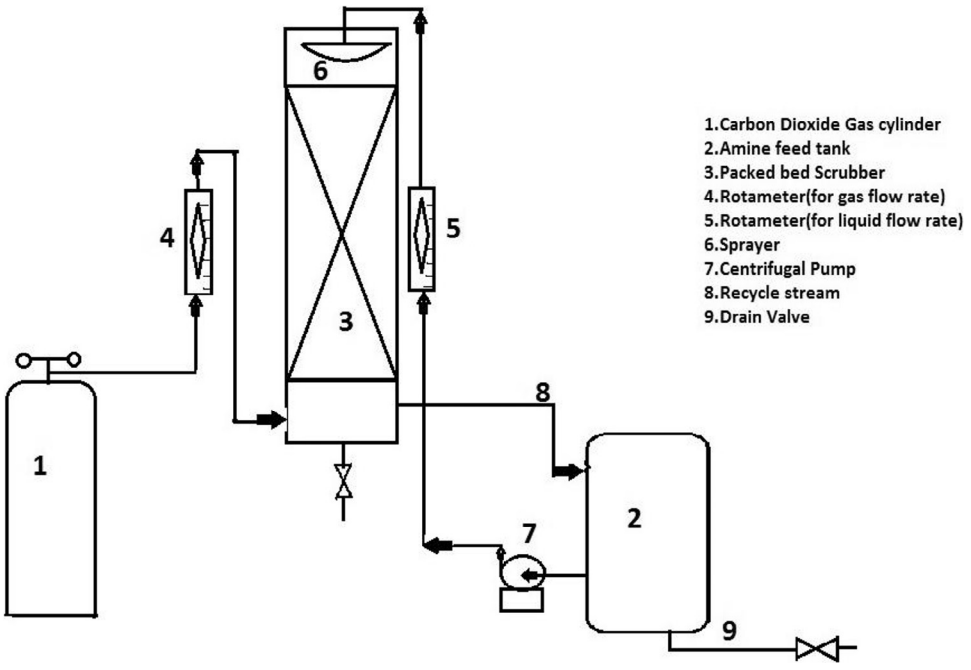

Fig. 1. Experimental set-up for CO_2 capture using amines.

The column was supported by the metal structure. An inlet for gas was set at the bottom of the column through which the gas travels upstream. For better dispersion of gas throughout the column perforated sprayer was used at the gas inlet. Similar mechanism was introduced at the top of the column at the liquid inlet for proper distribution of liquid. Raschig ring made of inert material with length 10 mm and diameter 2 mm was used as random packing inside the column. The packing used enhanced surface area of mass transfer as well as increased contact time between gas and liquid. Raschig ring is normally recommended for the liquid-gas operations. An emergency valve was introduced at the top of the column as a safety precaution. In case of increased pressure, the gas could be released from the column. A drain valve was introduced at the bottom of the column for emptying the column after the process and collecting the sample liquid. CO_2 sample for analysis could be taken from the gas outlet at the top.

Monoethanolamine (MEA) is corrosive in nature and hence plastic container was used as feed tank. All the piping and fittings used in the set-up are of Polyvinyl chloride material. Aqueous solution of amines (MEA or MEA + TEA) was pumped to the top of the column. A by-pass stream was arranged with the pump to decrease the load on the pump. CO_2 cylinder was used as a source of CO_2 to pump CO_2 at 2.5 kg/cm^2 pressure from the bottom of the column. A recycle stream was arranged at the bottom of the column leading to the feed tank. Rotameters of the unit Litre per minute (Lmin^{-1}) were installed for controlling the liquid and gas flow rates. For gas, the range of rotameter was 2–20 Lmin^{-1} and for liquid it was 1–10 Lmin^{-1}. For collecting CO_2 samples at regular time interval a stream was set at the top of the column.

Sample of CO_2 was collected in the flask containing NaOH for 3 s at regular intervals. The flask containing NaOH and CO_2 was covered with rubber balloon for better contact time without leakage of any CO_2. The absorption cycle was continued till the equilibrium was achieved. Equilibrium was assumed to have been established when a constant titer value was achieved. After every operation, the set up was cleansed with water to remove any contaminants and carbamates produced during the reaction in the column.

2.2. Estimation of CO_2

Standard known normality of HCl and NaOH solutions is prepared. NaOH solution is taken in conical flasks of required numbers based on the number of CO_2 samples to be analyzed. The flasks containing NaOH are contacted with CO_2 for 3 s at regular intervals and are allowed to react, the reaction forms sodium carbonate according to Eq. (1). The solution containing Sodium carbonate and excess NaOH is titrated against HCl using Phenolphthalein indicator. Here NaOH is neutralized and Sodium carbonate is converted to Sodium Bicarbonate. Again the solution was titrated against HCl using Methyl orange indicator (yellow to light orange). The Difference in the first and the second endpoints is used to calculate the mass of CO_2.

Volume of the titrant (L) × normality of acid × molecular weight of CO_2 = Mass of CO_2 (g)

$$\mathrm{NaOH\,(aq) + CO_2\,(g) \rightarrow Na_2CO_3\,(aq) + H_2O} \quad (1)$$

2.3. Absorption parameters

2.3.1. Absorption efficiency (E)

The absorption efficiency of the packed column can be expressed as follows: C_t and C_0 are the estimated mass of CO_2 at outlet and inlet, respectively (Table 2)

$$E = \left(1 - \frac{Ct}{Co}\right)100\% \quad (2)$$

2.3.2. Absorption rate (A_R)

The absorption rate was calculated from the inlet–outlet concentration difference or different positions at the steady-state condition, where G is the molar flux of gas,

$$RA = G(y_1 - y_2) \quad (3)$$

2.3.3. Scrubbing factor (ϕ)

The scrubbing factor is defined as shown below:

$$\Phi = A\left(\frac{E}{V}\right)y_1 \quad \text{and} \quad A = F_g / F_l \quad (4) \text{ and } (5)$$

where V is the volume of the scrubber.

F_g and F_l are gas molar flow rate and liquid molar flow rate respectively.

The scrubbing factor can be evaluated when E, V, and y_1 are available.

2.3.4. Pressure drop in a packed bed

For calculating pressure drop in a packed bed Ergun equation is used. The general form of Ergun equation is

$$\frac{\Delta P}{H} = \frac{150\mu U(1-\varepsilon)^2}{x^2\epsilon^3} + \frac{1.75\rho U^2(1-\epsilon)}{x\epsilon^3} \quad (6)$$

where

ΔP – Pressure drop in a packed bed (Pa), H – Height of the packed bed (m), μ – Viscosity of the fluid flowing through the packed bed (Pa·s), U – Superficial Fluid Velocity (m/s), ϵ – Bed voidage, ρ – density of fluid flowing through packed bed (kg/m^3), x – equivalent diameter (m).

2.3.5. Taguchi method of analysis

Genichi Taguchi, an engineer, developed a methodology for applying statistics to improve the quality of manufactured goods and the use of standard orthogonal arrays for designing experiments. It provides a simple, efficient and systematic approach to optimize the designs for performance, quality, and cost. 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 evaluated using ANOVA, Fisher's exact test or χ^2-test.

The design parameters are variables in the process that affect the performance of the process such as temperatures, flow rates etc. that can be easily controlled and determined. The number of levels that the parameters should be varied is quantified. For example, temperature may be varied between 40 °C (low) and 80 °C (high) with one or more intermediate levels. Further, increasing the number of levels a parameter is varied increases the number of experiments to be conducted. The standard orthogonal array is tabulated for a given set of experimental conditions extracted from a whole list of the conditions offered by the traditional factorial approach to experimental design. Orthogonal arrays are carefully constructed to yield unique design to the same situation by different experimenter; the selection of orthogonal arrays is based on the available parameters of the process. The results obtained are further subjected to statistical analysis to verify the outcome of the decision [20]. The experiments indicated in the array are conducted to collect data to study the effect on the performance measure [20].

The number in the names of arrays like L8, L9, L18 etc., given to the standard orthogonal arrays, denotes the number of test runs required for the specific situation.

The following orthogonal arrays are commonly used to design experiments:

2-level Arrays: L-4 L-8 L-12 L-16 L-32 L-64
3-level Arrays: L-9 L-18 L-27 (L-18 has one 2-Level column)
4-level Arrays: L-16 and L-32 Modified.

With the right array at hand, experiments are designed by mimicking the array. Once the design is completed, the experiments are planned accordingly. The execution of the experiments is usually process dependent, for example, the order in which the runs are made depends on the ease of tuning the levels of the parameters [21].

The analysis process can either make use of the average or signal to noise ratio values. Apart from the calculation of the averages or the signal to noise ratios, the analysis procedure is basically the same irrespective of the quantity adopted. When repetitions of runs are carried out, Taguchi recommends the use of his signal to noise ratio formulae for the analysis of the results. Otherwise, the conventional averages are used. The transformation of the results to signal to noise (S/N) ratio is estimated by the formula given below:

$S/N = -10 \times \log_{10} MSD$ MSD – Mean Squared Deviation from the target.

Mathematical expressions for the computation of the MSD for the quality characteristics are

Smaller the better: $MSD = (y_1^2 + y_2^2 + y_3^2 + \text{----})/n$
Nominal is best: $MSD = ((y_1 - m)^2 + (y_2 - m)^2 + \text{----})/n$
Bigger the better: $MSD = (1/y_1^2 + 1/y_2^2 + 1/y_3^2 + \text{----})/n$

In these equations, y_1, y_2, y_3 are the results for a particular run at the first, second and third repetitions respectively; m – the target value and n – the number of repetitions.

With the aid of the appropriate formula, the signal to noise ratios for all the trial conditions in an experiment can be calculated. If the averages were employed, however, the average value of the n repetitions for every trial condition would be computed instead. Whichever is the case, either with the signal to noise ratios or the average values, the average effect of all the studied parameters is calculated at their respective levels. The average effect of a parameter at a specified level is accounted for by averaging the results of all the tests containing the parameter at the level in question [21].

Table 1
Experimental design based on L_9 (3^4) orthogonal array.

Experiment No.	A (amine combination)	B (water dilution)	C (liquid flow) $Lmin^{-1}$	D (gas flow) $Lmin^{-1}$
1	MEA	10%	1	8
2	MEA	15%	1.5	16
3	MEA	20%	2	20
4	MEA + TEA (20%)	10%	2	16
5	MEA + TEA (20%)	15%	1	20
6	MEA + TEA (20%)	20%	1.5	8
7	MEA + TEA (30%)	10%	1.5	20
8	MEA + TEA (30%)	15%	2	8
9	MEA + TEA (30%)	20%	1	16

For the present study, $L_9(3^4)$ array was selected (Tables 1 and 2).

Performance parameter: CO_2 absorption. Quality characteristics: Bigger the better

3. Results and discussion

3.1. Maximum absorption of CO_2

The inlet Carbon dioxide pressure during the operation was maintained at 2.4 atm. The bed voidage was considered 0.4. The pressure drop as calculated using Eq. (4) for 1.15 m of packed bed was between 0.674 and 1.685 atm depending on the inlet flow rates. This pressure drop observed was due to the presence of Raschig ring packing randomly packed in the packed bed column. The variation of the measured CO_2 outlet concentration with time for different experimental conditions is shown in Fig. 2. As the solvent is recycled in the column, the outlet CO_2 concentration initially decreases (i.e. the ratio of C_t/C_0 decreases) with time to reach maximum point (maximum efficiency) and again starts increasing till no further absorption takes place. From the results it was observed that the ratio of outlet Carbon dioxide concentration to inlet concentration

Table 2
Effect of blending of MEA and TEA.

Ex. No.	Efficiency (E)	Absorption rate (A_R) (mol/m^2 s)	Scrubbing factor (ϕ) (mol-CO_2/mol solvent)
1	57.78	0.05	0.17
2	68.33	0.13	0.27
3	78.67	0.18	0.29
4	51.67	0.10	0.20
5	47.56	0.11	0.45
6	92.22	0.09	0.23
7	51.11	0.12	0.36
8	87.11	0.08	0.19
9	74.44	0.14	0.63

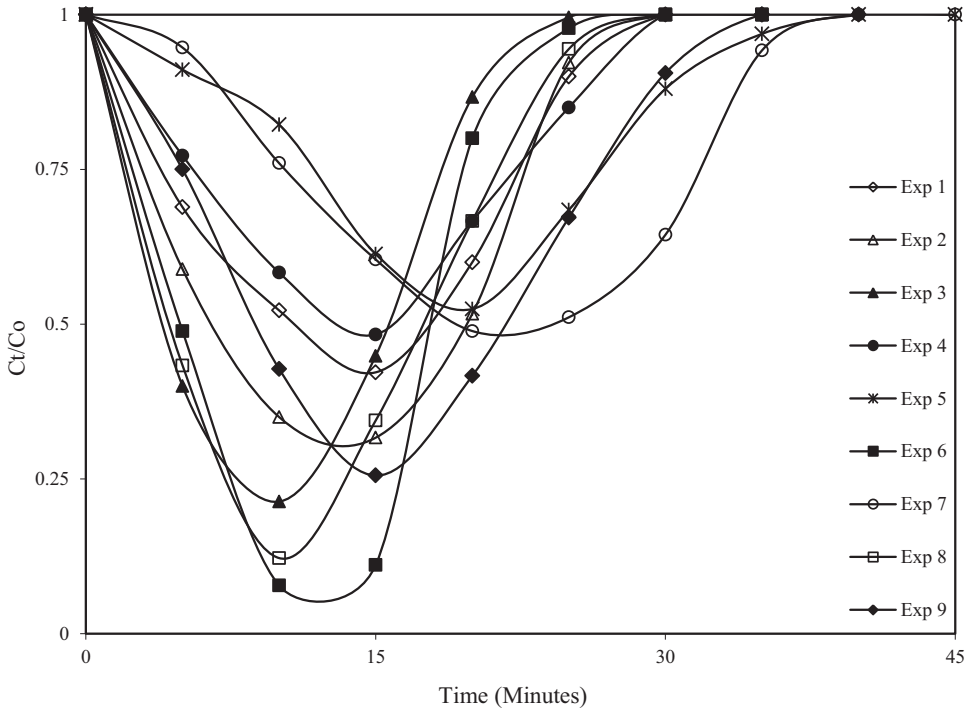

Fig. 2. Comparison of C_t/C_o v/s time for CO_2 absorption.

reaches a constant value of 1 after 35–40 minutes of operation. The absorption efficiency (E), absorption rate (RA) and scrubbing factor (ϕ) were calculated separately using Eqs. (2)–(4). The data of the 9 groups from the Taguchi experimental design are as shown in table 6.10, including E, RA and ϕ. All data were found to be in the range of 47.56–92.22%, 0.05–0.18 mol/s m^2 and 0.17–0.63 CO_2-mol/Solvent-mol L for E, RA and ϕ respectively.

3.2. Effect on absorption efficiency

The Absorption efficiency obtained in these experiments was in the range of 51.11–92.22%, depending on the operating conditions. The S/N ratios for the Absorption efficiency, as shown in Table 3.1 for factors A, B, C, and D, were 1.12, 3.66, 2.59 and 1.61, respectively. The major factors in the absorption efficiency in the order of importance were B (water dilution) > C (liquid flow rate) > D (gas flow rate) > A (blended amine Combination), meaning that the water dilution is the most important factor affecting absorption efficiency. In table 6.10, absorption efficiency for 15% and 20% dilutions was higher than that for 10%, indicating that the water dilution affected absorption efficiency. On the other hand, the optimal conditions according to the S/N ratio were found to be A3B3C1D3, i.e. type of blended amine (MEA + TEA (30%)), water dilution (20%), QL = 1 L/min, and Q_g = 20 L/min.

3.3. Analysis of absorption rate (R_A)

The absorption rate obtained here was in the range of 0.05–0.18 mol/m^2 s. The major factors influencing the absorption rate in the sequence of importance are C (Liquid Flow rate) > B (Water Dilution) > D (Gas Flow rate) > A (Amine Combination), meaning the liquid flow rate has the most important effect on the absorption rate, followed by the water dilution. Table 3.1 shows that the maximum RA was 0.18 (Table 3.2), and the corresponding gas flow rate and liquid flow rate were 20 L/min and 2 L/min, respectively. On the other hand, the second maximum value was 0.14 (No. 9), and the corresponding gas flow rate and liquid flow rate were 16 $Lmin^{-1}$ and 1 $Lmin^{-1}$, respectively. This indicated that gas flow rate and liquid flow rate had important influence on the absorption rate. From the two film models, higher liquid and gas flow rate attenuated the individual mass transfer resistance, and hence increased the absorption rate [22]. According to the S/N ratio factor, the optimal conditions are found to be A3B3C3D3, i.e. type of

Table 3.1
S/N ratio for absorption efficiency.

Level	A	B	C	D
1	36.61	34.56	37.8	35.41
2	35.70	36.37	36.13	36.72
3	36.82	38.22	35.21	37.02
Delta	1.12	3.66	2.59	1.61
Rank	4	1	2	3

Table 3.2
S/N ratio for absorption rate.

Level	A	B	C	D
1	−19.55	−21.48	−22.96	−20.76
2	−20.03	−19.61	−18.27	−19.02
3	−19.14	−17.63	−17.49	−18.94
Delta	0.89	3.85	5.46	1.81
Rank	4	2	1	3

Table 3.3
S/N ratio for scrubbing factor.

Level	A	B	C	D
1	−12.505	−12.748	−14.194	−8.780
2	−11.227	−10.911	−9.788	−11.004
3	−9.104	−9.177	−8.854	−13.052
Delta	3.401	3.571	5.340	4.272
Rank	4	3	1	2

blended amine (MEA + TEA (30%)), water dilution (20%), $Q_L = 2$ Lmin^{-1}, and $Q_g = 20$ Lmin^{-1}.

3.4. Analysis of scrubbing factor (ϕ)

The scrubbing factor obtained here was within the range of 0.17–0.63 mol-CO2/mol-MEA L. The S/N ratios for scrubbing factors, as estimated in Table 3.3, show that the major factors influencing the scrubbing factor in the sequence of importance are C (liquid flow rate) > D (gas flow rate) > B (water dilution) > A (amine combination), meaning the liquid flow rate is the major factor, followed by the gas flow rate. The results in table 6.10 found that ϕ increased with the Q_g/Q_L ratio, which was similar to that observed in the absorption rate. On the other hand, the optimum conditions were found to be A3B3C3D1, i.e. type of blended amine (MEA + TEA (30%)), water dilution (30%), $Q_L = 2$ Lmin^{-1}, and $Q_g = 8$ Lmin^{-1}.

4. Conclusions

The potential of TEA as a blend with MEA for Carbon dioxide absorption packed bed column was evaluated using Taguchi Analysis. Also the factors affecting the Carbon dioxide absorption in packed bed column like water dilution, gas flow rate and liquid flow rate were successfully evaluated using Taguchi method. The absorption efficiency, Absorption rate of Carbon Dioxide and the scrubbing factor were determined according to S/N ratio of Taguchi Analysis and optimum parameters, the important sequence of factors, are obtained. In Larger the better mode, MEA was better for absorption rate, while MEA + TEA (30%) was better for efficiency and scrubbing factor. The best suitable operating conditions were obtained for the carbon dioxide absorption in a packed bed column. By incorporating recycle stream, the amount of amine solution required was reduced. Further the work can be extended for other amine blends of MDEA, AMP with MEA and for varied types of packing in the column under the influence of different process temperatures.

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