

## IMPROVEMENT OF BIOGAS UPGRADING PROCESS USING CHEMICAL ABSORPTION AT AMBIENT CONDITIONS

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### Graphical abstract

Comparison between 3 solvents



Improving absorption using the selected solvent



Producing upgraded biogas containing more than 95 % methane

### Abstract

Biogas major components are methane, carbon dioxide and traces of hydrogen sulfide, ammonia and nitrogen. Biogas upgrading process is the process by which carbon dioxide (composing 40 % of the biogas) is removed. In this study chemical absorption process using three different solvents (10 – 30 % monoethanolamine, 4 – 12 % sodium hydroxide and 5 – 15 % aqueous ammonia) was performed to produce methane-enriched biogas. A laboratory-scale packed-column apparatus containing efficient and cheap packing material (plastic bioball) was used to perform the experimental work in this study. Initial absorption runs were performed to select the best solvent type and concentration. Monoethanolamine (MEA) was proven to have the highest ability in producing upgraded biogas using a single absorption column apparatus at ambient conditions. The liquid to gas flow ratio was investigated using 30 % MEA solution. Optimum liquid to gas flow ratio for biogas upgrading process was determined to be about 18 (on mass basis). Biogas with methane content up to 96.1 v/v% was produced with CO<sub>2</sub> loading capacity up to 0.24 mole-CO<sub>2</sub> per mole-MEA.

Keywords: CO<sub>2</sub> Removal, Biogas Upgrading, Chemical Scrubbing, Chemical Absorption, Alkaline Scrubbing

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## 1.0 INTRODUCTION

Biogas is a product of anaerobic digestion that is composed of 50 – 65 % methane, 35 – 50% carbon dioxide and traces of hydrogen sulfide, ammonia and nitrogen [1]. Biogas production and upgrading processes are considered as waste treatment processes that yield energy with a low environmental pollution impact. The two main components to be removed prior to biogas utilization are CO<sub>2</sub> and H<sub>2</sub>S [2]. Although it is flammable, H<sub>2</sub>S is usually removed from biogas prior to any utilization as a fuel due to its toxicity and corrosive nature. The process by which H<sub>2</sub>S is usually removed is called biogas purification. CO<sub>2</sub> removal is considered the most important step in

increasing the methane content in biogas and therefore increasing its heating value [3, 4]. The process of CO<sub>2</sub> removal is usually called biogas upgrading.

Several methods have been employed for the purpose of purifying and upgrading biogas including physical solvent scrubbing (such as water scrubbing) [5], pressure swing adsorption [6], biological treatment [7], chemical absorption [8–12] and cryogenic separations [9]. However, chemical absorption is believed to have a great potential in upgrading biogas since the absorption process can be applied at ambient or near ambient temperature and pressure [13]. Recent researches have shown that biogas upgrading by removing the combustion-inert

carbon dioxide is an important step for the safe and efficient use of the biogas as a fuel [14].

Chemical absorption of carbon dioxide can be performed using different types of contactors. The selection between packing, plate and membrane contactor is usually performed based on the scale and budget. While structured packing is believed to be more suitable for small scale due to its high cost, random packing are reported to be suitable for small and large column sizes if the packing material is chosen carefully [15].

When chemical absorption is performed using packed column apparatus, the most important features to be decided and selected are:

- The solvent to be used as an absorber for CO<sub>2</sub>.
- The packed column properties including: height, diameter and packing material.
- The different process conditions including temperature, pressure, solvent concentration, gas flow rate and solvent flow rate.

The best solvent for the carbon dioxide removal from biogas has to be selected based on a number of considerations such as; the minimum required concentration, low consumption of absorbing material (i.e. high load, easy regeneration, chemical and thermal stability), no environmental impact and availability and low price [1]. Several solvents were proven to comply with the aforementioned considerations including sodium and potassium hydroxides [9, 10], amines, such as momoethanolamine and diethanolamine [8, 16], and aqueous ammonia [12, 17].

Several methods were followed in similar previous studies for choosing the packed column geometrical features. However, in this study a simplified model of packed column apparatus is used as a CO<sub>2</sub> scrubber. The apparatus was designed in accordance with the considerations reported in our previous work [18].

The main objective of this study is to produce upgraded biogas with methane content above 95 % using feed biogas that contains 60 % methane and 40 % CO<sub>2</sub>. A packed column absorber apparatus was used in order to facilitate varying the different parameters of the absorption process. Three different solutions (MEA, sodium hydroxide, and aqueous ammonia) are prepared and verified for their ability to absorb carbon dioxide. The absorption process is performed to upgrade biogas at ambient temperature and pressure which is important for energy-cost reduction.

## 2.0 METHODOLOGY

### 2.1 Apparatus and Materials Preparation

A packed column apparatus was used in this study to verify the chemical absorption of CO<sub>2</sub> for biogas upgrading in the lab scale. The key characteristics of the packed column, including column height, diameter and packing material properties are shown

in Table 1. The different properties were decided based on several recommendations from literature which was discussed in our previous study [18]. As listed in Table 1, the apparatus used in this study contained a 0.1 m in diameter and 2 m in height packed column. The column was packed with the commercially available packing material called plastic bioballs which was verified to be efficient as packing material for biogas upgrading [19].

**Table 1** Characteristics of the packed column

Parameters	Values
Column diameter	0.1 m
Column Height	2 m
Packing surface	350 m <sup>2</sup> m <sup>-3</sup>
Void fraction $\epsilon_p$	0.85
Pressure drop at 70 - 80 % flooding	2 mbar/m
Minimum liquid load	0.2 m <sup>3</sup> /m <sup>2</sup> h
Maximum liquid load	200 m <sup>3</sup> /m <sup>2</sup> h

The method used for determining the packing height is independent of the mass transfer coefficient and rather depends on the initial and final solute gas concentration. The method is explained by the following equations as reported by [20].

$$Z = N_{OG} \times H_{OG} \times SF \quad (1)$$

$$N_{OG} = \ln \frac{Y_1}{Y_2} \quad (2)$$

where,  $N_{OG}$  is the number of theoretical plates,  $H_{OG}$  is the height of transfer unit, and  $SF$  is the safety factor. While  $N_{OG}$  is calculated as shown by the above equation,  $H_{OG}$  is determined from a table based on the packing type and size. Table 2 shows the height of transfer units in feet based on the packing size and type as was recommended by [20].

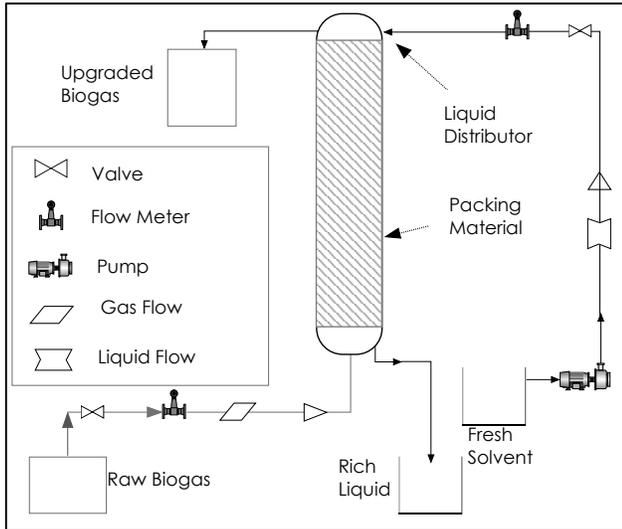
The  $H_{OG}$  corresponding to the plastic packing with 1 inch diameter (25 mm) is 1 feet, as listed in Table 2. The composition of biogas used as feed in this absorption process is 60 % methane and 40 % CO<sub>2</sub>. The objective of this study is to produce biogas with CO<sub>2</sub> content less than 5 mol/mol % (in the range 0.5 – 5 %). Therefore, the values of the mole fractions  $y_1$  and  $y_2$  of equation 2 are 0.4 and 0.005 respectively. Considering a safety factor of 1.5, the height of the column is calculated to be 2 m.

**Table 2**  $H_{OG}$  based on packing size and type [20]

Packing Diameter (in)	Plastic Packing $H_{OG}$ (ft)
1.0	1.0
1.5	1.25
2.0	1.5
3.0	2.25
3.5	2.75

A schematic diagram of the experimental apparatus is shown in Figure 1. The figure shows the different items of the apparatus including the gas

liquid containers, the packed column, pumps, valves and flow meters. The packed column and all the piping and fittings were fabricated from PVC. The packing material were the aforementioned plastic bioball. Gas cylinders holding gas slightly above atmospheric pressure were used to supply the feed biogas to the absorption column. The top of the absorption column is made of a liquid distributor with a drip-point density of 2000 points/m<sup>2</sup>.



**Figure 1** Schematic diagram of absorber column apparatus

The biogas used in the experiments was obtained from a biogas plant (Cenergi SEA Sdn Bhd) that uses palm oil mill effluent as the feedstock. The biogas was previously treated for the removal of some impurities such as H<sub>2</sub>S. The biogas characterization shown that it is composed of 40.1 v/v% CO<sub>2</sub>, 59.8 v/v% CH<sub>4</sub> and traces of H<sub>2</sub>S, NH<sub>3</sub>, N<sub>2</sub> and O<sub>2</sub>. The solutions of the three different solvents, monoethanol amine, sodium hydroxide and aqueous ammonia, were prepared in the concentrations listed in Tables 4 – 6 shown in the results and discussion section.

## 2.2 Mass Balance and Flow Rates Calculation

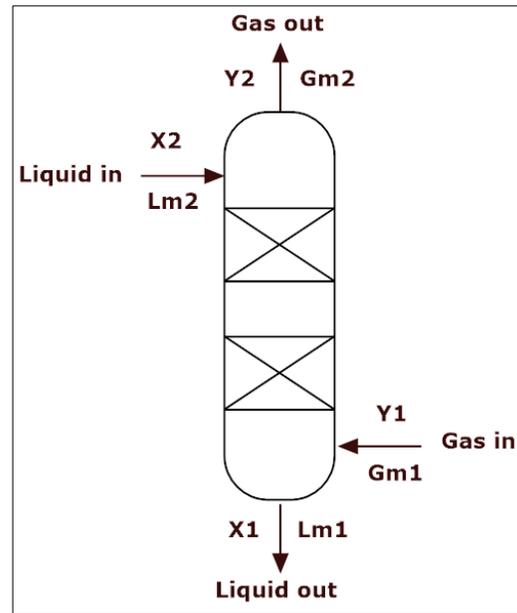
Typical material balance analysis for a counter current absorption column can be performed as shown in Figure 2. As shown in the figure, the biogas enters the packed column at molar flow of  $G_{m1}$  and exiting at  $G_{m2}$  while the scrubbing liquid is entering the packed column at  $L_{m2}$  and exiting at  $L_{m1}$ .

From Figure 2, for a component  $i$ , the mole balance is

$$G_{m1} \times y_{i1} + L_{m2} \times x_{i2} = G_{m2} \times y_{i2} + L_{m1} \times x_{i1} \quad (3)$$

$$L_{m2} \times x_{i2} - L_{m1} \times x_{i1} = G_{m2} \times y_{i2} - G_{m1} \times y_{i1} \quad (4)$$

where  $G_m$  is gas molar flow,  $L_m$  is liquid molar flow,  $x_i$  is mole fraction of solute in liquid and  $y_i$  is mole fraction of solute in gas.



**Figure 2** Material balance diagram for a counter current absorption column

A modified form of Equation 4 can be formed as shown by Equation 5.

$$G_m' (Y_i - Y_{i2}) = L_m' (X_i - X_{i2}) \quad (5)$$

$$\frac{L_m'}{G_m' \min} = \frac{(Y_i - Y_{i2})}{(X_i - X_{i2})} \quad (6)$$

where,  $G_m'$  is the solute-free gas flow rate,  $L_m'$  is the solute-free liquid flow rate,  $X_i$  is the mole ratio of solute  $i$  in liquid such that  $X_i = x/(1-x)$ , and  $Y_i$  is the mole ratio of solute  $i$  in gas such that  $Y_i = y/(1-y)$ .

In the current study, biogas containing 40.1 % CO<sub>2</sub> is to be upgraded to a methane-rich gas containing less than 5 % CO<sub>2</sub>. The upgrading process is performed using chemical absorption by a fresh solvent. However, the following assumptions are made to simplify the calculation process.

1. The upgraded biogas exits the column with CO<sub>2</sub> content in the range 0.5 – 5 mol/mol %. Hence, the CO<sub>2</sub> mole fraction in the exiting biogas ( $y_{i2}$ ) is in the range 0.005 – 0.05.
2. The fresh solvent used is free from CO<sub>2</sub>. Therefore, CO<sub>2</sub> mole fraction in entering liquid ( $x_{i2}$ ) is zero.
3. The exiting liquid reaches equilibrium composition and therefore the CO<sub>2</sub> mole fraction is equal to the maximum loading capacity.
4. 30 % MEA solution is used as a basis for flow rates calculations.

A solution containing 30 w/w% MEA was reported to have a maximum capacity of 2.9 mole of CO<sub>2</sub> per litre of the solution [16]. Using this number, the maximum CO<sub>2</sub> mole fraction in exiting 30 % MEA solution is 0.0612. The liquid flow rate range is determined first by

calculating the minimum liquid flow rate in the method explained by equations 3 – 6.

Using equation 6, the ratio of the liquid molar flow rate to the gas molar flow rate ( $G_m'$ ) can be calculated. To calculate the actual minimum flow rate ratio it is a common practice to multiply the result obtained using ( $L_m'$ ) equation 6 by a factor of 1.2 [20]. The mole fractions and mole ratio involved in equation 6 are shown in Table 3

**Table 3** CO<sub>2</sub> mole fraction and mole ration in entering and exiting streams

Stream	CO <sub>2</sub> mole fraction	CO <sub>2</sub> mole ratio
Gas in	0.4	0.6667
Gas out	0.005	0.005
Liquid in	0.0	0.0
Liquid out	0.0612	0.0652

Substituting the values of Table 3 in equation 6 gives a value for the solute-free liquid to gas molar flow ratio ( $L_m' / G_m'$ ) of 10.1. Assuming that  $L_m' = L_m$  and that  $G_m' = 0.6 \times G_m$ , the liquid to gas molar flow ratio ( $L_m / G_m$ ) is calculated as 6.1, where  $L_m$  and  $G_m$  are the molar flow of liquid and gas, respectively. Multiplying the ratio by the factor of 1.2 the ratio will be equal to 7.3. Using this value, the recommended liquid to gas mass flow ratio when using 30 % MEA will be approximately 6.

Considering minimum liquid to gas molar flow ratio is 7.5, the liquid to gas mass flow ratio should be approximately 6. In fact, the liquid to gas flow rate ratio calculated above is based on the 30 % MEA solution absorption capacity of CO<sub>2</sub>. However, in this study, aqueous solutions of 10 – 30 % MEA, 4 – 12 % NaOH and 5 – 15 % ammonia were used. Thus, the liquid to gas flow rate ratio is varied in a higher value to allow for the opportunity for full removal of carbon dioxide using all solvents used and at different concentrations.

Following the minimum liquid to gas molar or mass flow determination, the operating liquid and gas mass flow rates are calculated so as to avoid flooding at the pressure drop of the designed column. The method explained by [15] for flooding percentage calculation is followed. Using the values of density and viscosity of the various solutions used in this study, the percentage flooding was calculated for a liquid flow of 72 kg/h and gas flow of 6 kg/h (ratio of liquid to gas mass flow is equal to 12). The calculated percentage flooding using the different solutions did not exceed 40 %.

### 2.3 Experimental Procedure

The absorption process was performed in a counter current mode by feeding the gas and liquid to the column at ambient temperature of approximately 25 °C. Each experimental run started by preparing the solvent in the required concentration. The liquid flow rate was controlled using the peristaltic pump speed. The gas flow rate was regulated using a gauge pressure regulator. Manual verification of the biogas

flow was performed by collecting the gas flowing using intermediate container and measuring its volume for a period of time to calculate its volumetric flow rate in natural cubic meter per hour (Nm<sup>3</sup>/h). The volumetric gas flow is then converted to mass flow (kg/h) based on the gas composition and its anticipated density at the working conditions. Each absorption run started by feeding the raw biogas to the bottom of the packed column and spraying the absorbing solvent from top of the column, in a countercurrent flow mode. The upgraded biogas is collected from top of the packed column while the rich solvent leaves at the bottom. All experiments were performed at least three times and average values were considered as the verified results. Each run was performed until the upgraded biogas approaches constant composition. Infrared gas analyzer (Combimass GA-m) manufactured by Binder Group (Germany) was used to measure the gas composition before and after the absorption process is performed. Experiments performed were divided to two phases. In the first phase, the three different solvents were used at three different concentration while keeping the liquid to gas flow ratio constant for all of them ( $L_m' / G_m' = 12$ ). Then in the second phase, the solvent that yielded highest biogas purity (lowest CO<sub>2</sub> Mole fraction) was used to perform several absorption runs for the purpose of finding the optimum liquid to gas flow ratio.

## 3.0 RESULTS AND DISCUSSION

Several runs were initially performed to compare between MEA, sodium hydroxide and aqueous ammonia as scrubbing solvents for the biogas upgrading process. The comparison was made based on the molar composition for the upgraded biogas. All runs were performed using the packed column apparatus shown in Figure 1, at 25 °C, atmospheric pressure and at liquid flow of 72 kg/h and gas flow of 6 kg/h. The results obtained are shown in Tables 4, 5 and 6.

Table 4 shows the molar percentage of each component in the biogas after scrubbing with 10 %, 20 % and 30 % MEA solution. The table shows that using 30 % MEA solution, the scrubbed gas was composed of 92.3 % methane, 4.7 % carbon dioxide and 3.0 % water. If the gas is dehydrated and the water is fully removed, biogas of methane content higher than 95 % can be obtained.

**Table 4** Gas molar composition after scrubbing with MEA

Concentration	CH <sub>4</sub>	CO <sub>2</sub>	H <sub>2</sub> O
10	77.8	18.6	3.6
20	88.3	8.4	3.3
30	92.3	4.7	3.0

The results listed in Table 4 show that the change in CO<sub>2</sub> concentration decreases at higher

concentrations of MEA. That is clear when comparing the decrease of 9.8 % in CO<sub>2</sub> content when increasing MEA concentration from 10 to 20 % and the decrease of 3.7 % when increasing MEA concentration from 20 to 30 %. Therefore, it is anticipated that increasing MEA concentration above 30 % will lead to slight decrease in CO<sub>2</sub> content. This is explained by the fast instantaneous equilibrium reached between CO<sub>2</sub> molecules and amine molecules at higher concentrations when compared to the slow equilibrium obtained at low concentration of MEA. For this, it is believed that the increase in the corrosive nature of MEA solvent at concentration higher than 30 % outweigh the corresponding slight increase in biogas quality. This conclusion is in line with results concluded by previous studies [14].

Table 5 shows the molar composition of the biogas after scrubbing with 4 %, 8 % and 12 % sodium hydroxide. The minimum carbon dioxide content was obtained when using 12 % sodium hydroxide. This relatively high percentage makes it difficult to obtain a biogas of higher than 95 % methane content at the ambient or near ambient conditions.

**Table 5** Gas molar composition after scrubbing with NaOH

Concentration	CH <sub>4</sub>	CO <sub>2</sub>	H <sub>2</sub> O
4	80.8	15.5	3.7
8	83.3	13.2	3.5
12	85.9	10.8	3.3

The difference in the performance of MEA and sodium hydroxide can be explained by the fact that, for the solvents used in this study, sodium hydroxide solutions have less molar concentrations when compared to MEA solutions. The mole fraction range corresponding to 10 – 30 w/w% MEA solutions is 0.032 – 0.112, whereas the mole fraction range for 4 – 12 % NaOH solutions is 0.018 – 0.058. However, the aforementioned concentration ranges of the solvents were based on recommendations from literature [14] that has taken the corrosiveness of solvent in consideration. Therefore, MEA-based solvents are considered to be more suitable for biogas upgrading than the sodium hydroxide solvent due to the possibility of using more mole-concentrated solvent.

Table 6 shows the molar composition of biogas scrubbed using 5 %, 10 % and 15 % aqueous ammonia. Due to the high vapour pressure of aqueous ammonia the scrubbed gas contained a high fraction of ammonia gas, which is increasing at higher concentrations of the solvent. Aqueous ammonia have shown the ability to absorb carbon dioxide and reduce its content to 8.9 %. However, a major complication of the process when performed at ambient conditions is the large fraction of ammonia present in the upgraded gas. Thus, to upgrade biogas using aqueous ammonia, either the operating conditions have to be changed or a second column should be used to absorb ammonia using water as a

scrubbing liquid, since ammonia is highly soluble in water.

**Table 6** Gas molar composition after scrubbing with NH<sub>3</sub>

Concentration	CH <sub>4</sub>	CO <sub>2</sub>	H <sub>2</sub> O	NH <sub>3</sub>
5	76.0	13.4	3.4	7.2
10	66.2	11.6	3.4	18.8
15	51.2	8.9	3.4	36.5

As portrayed in Tables 4, 5 and 6, the results show that the packed column apparatus has been efficiently used for the absorption of CO<sub>2</sub> from raw biogas. The three solvents have shown different abilities for upgrading biogas at ambient conditions. However, using a single absorption column apparatus, MEA solution of concentration about 30 w/w% is considered the most suitable solvent.

The previous results also show that sodium hydroxide was the second effective solvent in terms of methane concentration since the scrubbed gas contained 85.9 % methane. However, the suitability of the solvent is also dependent on the CO<sub>2</sub> content which is still very high when using sodium hydroxide. In addition, the regeneration of the sodium hydroxide is considered more energy extensive when compared to MEA.

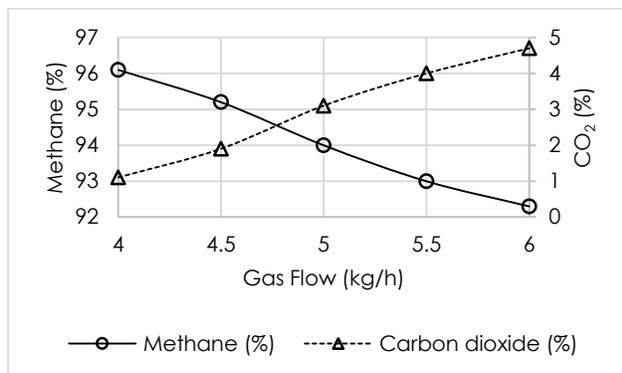
Aqueous ammonia, although produced biogas with high fraction of ammonia it is considered relatively efficient in terms of methane enriching to a certain extent. It is expected that using water scrubbing subsequent to ammonia scrubbing, biogas with methane content up to 85 % can be obtained at ambient conditions.

Subsequent to the initial absorption runs, attempts were made to produce biogas composed of more than 95 % methane using MEA as a scrubbing solvent. Few runs were performed using 30 % MEA for scrubbing biogas using the fabricated apparatus at ambient conditions and at a liquid flow rate of 72 kg/h but with a new range of biogas mass flow from 4 – 6 kg/h with an increment of 0.5 kg/h. Figure 3 shows the methane and CO<sub>2</sub> percentage in the scrubbed biogas.

Plots of methane and carbon dioxide v/v% against gas mass flow, as illustrated in Figure 3, show that upgraded biogas with methane content up to 96.1 % was obtained. It can be concluded from this figure also that the continuous decrease in gas flow below 4 kg/h is possible to lead to the production of higher purity biogas. This is a clear indication that with the current column environment and dimensions, the retention time for the mass transfer reaction did not reach the optimum value. The retention time of the current absorption process can be increased by increasing the column height. Therefore, it is expected that if the effect of column height and gas flow are studied as process factors, a significant interaction would be recorded between both factors. This is due to the fact that at higher gas flow values, more CO<sub>2</sub> molecules are flowing into the column per unit time and thus more time is required for the reaction to

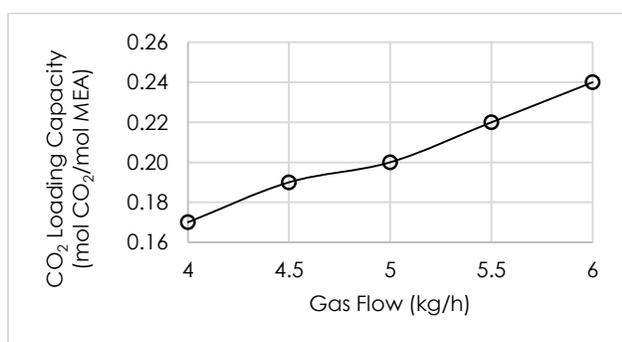
reach equilibrium. Overall, this result shows that MEA is a potential solvent to be used for CO<sub>2</sub> scrubbing and biogas upgrading at ambient conditions since biogas of more than 95 % content is produced.

However, besides methane percentage, CO<sub>2</sub> loading capacity is another important factor to be used as a basis for evaluating the suitability of the scrubbing solvent. Previous studies performed for chemical absorption of carbon dioxide using MEA have shown that the CO<sub>2</sub> loading capacity of the optimized absorption process can range between 0.17 – 0.22 mole CO<sub>2</sub> per mole MEA [19, 21].



**Figure 3** Methane and CO<sub>2</sub> percentage in the scrubbed biogas using 72 kg/h 30 % MEA solution

The CO<sub>2</sub> loading capacity values for the 30 % MEA solution used in the experiments represented in Figure 3 were calculated and represented by the plot shown in Figure 4. The plot shows that the experiments have resulted in CO<sub>2</sub> loading capacity between 0.17 – 0.24 mole CO<sub>2</sub> per mole MEA.



**Figure 4** CO<sub>2</sub> loading capacity for scrubbing biogas with 72 kg/h 30 % MEA solution

The results shown in Figure 4 indicate that increasing gas flow has great effect on increasing CO<sub>2</sub> loading capacity. This is logical since the higher the gas flow the more the CO<sub>2</sub> molecules present at the liquid-gas interphase, therefore, the more the CO<sub>2</sub>-MEA equilibrium reaction is shifted towards the product side. Hence, increasing gas flow is believed to

decrease the time the reaction requires to obtain equilibrium. For this, increasing retention time or alternatively increasing column height is anticipated to supplement the requirement of increasing gas flow for the purpose of enhancing CO<sub>2</sub> loading capacity.

Overall, the plots shown in Figures 3 and 4 show that biogas containing methane in the range of 92.3 – 96.1 % was produced using 30 % MEA at CO<sub>2</sub> loading capacity in the range 0.17– 0.24. The plots also show that the feed biogas flow had a significant effect on both methane percentage and CO<sub>2</sub> loading capacity of the MEA solution. Hence, it is expected that the process of biogas upgrading using MEA as a solvent can be further optimized by varying the gas flow rate and the liquid flow rate at wider ranges.

## 4.0 CONCLUSION

The three different chemical solvents used in this study were proven able to remove CO<sub>2</sub> from biogas with different efficiencies. MEA is proven to be the only solvent that can produce a gas of less than 5 % CO<sub>2</sub> content at ambient conditions and using a single column absorber apparatus. Sodium hydroxide did not show great potential in upgrading biogas, at the current conditions, as it produced biogas with 85.9 % methane only. Aqueous ammonia has decreased carbon dioxide percentage to 8.9 %, however, the scrubbed gas contained a big fraction of ammonia gas. Therefore, it is believed that aqueous ammonia scrubbing can be improved if the process is followed by water scrubbing to remove ammonia gas from the scrubbed gas. Several experiments were performed using 30 % MEA solvent to investigate the effect of the liquid to gas flow ratio on biogas purity and CO<sub>2</sub> loading capacity. The aforementioned experiments have shown that optimum biogas purity was obtained when performing the experiment at a relatively high value of liquid to gas flow ratio of about 18. Investigation of liquid to gas ratio effect has also led to the conclusion that other process factors, such as column height, has to be involved in a more comprehensive analysis to obtain a biogas with higher purity and at relatively higher values of CO<sub>2</sub> loading capacity. At the end of this study, upgraded biogas containing 92.3 – 96.1 % methane was produced using 30 % MEA solution. The CO<sub>2</sub> loading capacity of the 30 % MEA solution have ranged between 0.17 – 0.24 mole CO<sub>2</sub> per mole MEA.

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