

Selective H₂S Absorption Using the Mixture of NaOH-NaHCO₃-Na₂CO₃ Buffer Solvent Solution

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Highlights:

- The addition of NaHCO₃ and Na₂CO₃ buffer at a specific mass ratio to NaOH solvent could hinder CO₂ absorption, thus increasing the absorption selectivity towards H₂S that is required for acid gas enrichment unit application.
- A small L/G ratio of around 0.006 allows a reduction of CO₂ absorption activity because there is limited solvent to absorb CO₂ after absorbing H₂S close to equilibrium.
- A large L/G ratio of around 0.6 does not guarantee that the reduction of CO₂ absorption activity can be controlled precisely because of the low contact time.
- The highest H₂S selectivity at 23.1 was achieved at an L/G ratio of 0.006 using a solvent mixture of 5%-mass NaOH, 15%-mass NaHCO₃, and 18.9%-mass Na₂CO₃.

Abstract. Acid gas enrichment unit (AGEU) involves selective separation of H_2S from acid gas mixture, for example using absorption with an NaOH solvent solution. Sodium carbonate (Na₂CO₃) and sodium bicarbonate (NaHCO₃) buffer addition to NaOH solution suppresses CO₂ absorption, thereby increasing the selectivity of H_2S absorption. This study evaluated the effect of buffer addition to increase H_2S absorption selectivity using an NaOH solution. It was shown that both buffer addition and L/G ratio decrease could increase H_2S selectivity by limiting CO₂ absorption. Based on the simulation results, in the 0.006 to 0.030 L/G ratio range and NaOH solvent concentration greater than 2%-mass, the addition of NaHCO₃ with mass ratio greater than 1.5:1 to NaOH and the addition of Na₂CO₃ at 1.26 times NaHCO₃'s mass increased H_2S absorption selectivity up to 17.3%. The combination of an L/G ratio of 0.006 and solvent with a composition of 5%-mass NaOH, 15%-mass NaHCO₃, and 18.9%-mass Na₂CO₃ produced the highest H_2S selectivity of 23.1 (379.7% H_2S selectivity increase).

Keywords: acid gas; Na₂CO₃; NaHCO₃; NaOH; selective absorption; simulation.

1 Introduction

Natural gas is one of the major energy sources in Indonesia [1]. It typically contains acid gas impurities, such as H_2S and CO_2 , that need to be removed, which is commonly carried out in an acid gas removal unit (AGRU) using

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alkanolamine solvents such as MEA (ethanolamine), DEA (diethanolamine), and activated MDEA (methyl-diethanolamine) [2]. This process produces a stream of acid gas rich in the H_2S and CO_2 . The acid gas cannot be released directly into the atmosphere since it must meet the sulphur content separation requirements, for example regulated in Regulation (Permen LH) No. 13 of 2009 of the Ministry for the Environment, Indonesia [3]. Therefore, an additional process is required to reduce the H_2S content in the flue gas to meet the regulation.

The corresponding processing units that are capable of separating H₂S selectively from the acid gas stream are acid gas enrichment unit (AGEU) and sulphur recovery unit (SRU).

AGEU commonly use membrane, adsorption, and absorption technologies. The membrane technology has emerged recently, but it requires high capital and operational costs to apply it, especially due to the low feed pressure in this particular AGEU system [4]. Another method is to use adsorption in a fixed bed reactor using metal ions with a certain support substance as adsorbent of H_2S [5,6].

Absorption is the most commonly used method due to its cheapness, operation flexibility, and relatively low waste production compared to membrane and adsorption technologies. In addition, the waste that is generated by absorption, especially when using an alkaline hydroxide solvent, has high economic value [7].

In the absorption method, the acid gas feed is contacted with a solvent that can selectively absorb H_2S . Many different solvents can be used, such as metal sulphates [8,9], alkanolamines (MEA, DEA, DIPA, TEA, MDEA, etc.) [10], alkali-hydroxides (KOH and NaOH) [11], water, ammonia, and ionic liquids [12,13]. Among these options, alkanolamine solvents are the most widely used because of their high capacity and fast reaction, but they are prone to degradation [14]. Alternatively, absorption using alkali-hydroxide solvents, which is categorized as a chemisorption process, has been reported to be efficient for this application, especially due to its operation flexibility and ability to absorb trace amounts of H_2S and CO_2 [7,15].

A possible approach to achieve high H_2S absorption selectivity is to limit the CO_2 absorption activity. In general, the absorption process of a gas stream containing H_2S and CO_2 using an NaOH solvent takes place based on the reaction equations R.1 to R.4. The provided equilibrium constants (at 25 °C) were taken from [16].

$$H_2S_{(aq)} + NaOH_{(aq)} \rightleftharpoons NaHS_{(aq)} + H_2O, K_{eq} = 2.91 \times 10^{-14}$$
 (R.1)

$$NaHS_{(aq)} + NaOH_{(aq)} \rightleftharpoons Na_2S_{(aq)} + H_2O, K_{eq} = 1.84 \times 10^{-17}$$
 (R.2)

$$CO_{2(aq)} + NaOH_{(aq)} \rightleftharpoons NaHCO_{3(aq)}, K_{eq} = 1.20 \times 10^{-1}$$
(R.3)

 $NaHCO_{3(aq)} + NaOH_{(aq)} \rightleftharpoons Na_2CO_{3(aq)} + H_2O, K_{eq} = 8.38 \times 10^{-13}$ (R.4)

One method to limit CO_2 absorption activity is by limiting the reactants or by adding products based on Le Chatelier's principle [17]. The reactants (NaOH) can be limited based on the difference in reaction rates of reactions R.1 and R.3. If the available NaOH in the solution runs out first due to reaction R.1, then only a small amount of NaOH will undergo reaction R.3, thus selectivity can be achieved. Another method is to add the products of the unwanted reactions, i.e., reactions R.3 and R.4, from the very beginning of the absorption process.

The addition of NaHCO₃ (sodium bicarbonate) and Na₂CO₃ (sodium carbonate) will shift the equilibrium of reactions R.3 and R.4 and reduce the rate of these two reactions to the right, thus achieving the desired H_2S selectivity.

Previous research has shown that buffer addition to a NaOH solvent could shift absorption selectivity towards H_2S [18], especially at a small L/G ratio of 0.020 [19]. It was also concluded that the addition of NaHCO₃ only to the solvent is not sufficient to withstand the CO₂ absorption activity [19]. Therefore, the present study was conducted to figure out the effect of the NaOH concentration in the solvent and buffer addition to the solvent (NaHCO₃ and Na₂CO₃) on the reduction of the CO₂ absorption activity, which ultimately leads to high H_2S selectivity.

2 Methodology

In broad outline, the sequences of the research were a bubbling experiment, followed by a continuous experiment (once-through process), and completed with a simulation on the Aspen Plus v10 software. The optimum buffer to ratio and the optimum L/G ratio obtained from the experiment were then used to predict the selectivity of H₂S absorption at optimum conditions using Aspen Plus V10 simulation. In addition, a sensitivity test for the H₂S absorption selectivity towards changes in the concentration of NaOH and buffer of NaHCO₃ and Na₂CO₃ in the solvent was also carried out.

The contactor column, its dimensions, and other related materials used in this study are shown in Figure 1.



Figure 1 Schematic of apparatus, column specifications, and materials used.

2.1 Bubbling Experiment

This experiment was conducted to obtain the ratio of buffer to NaOH that minimizes CO_2 absorption. The obtained ratio was used in the selective H_2S absorption simulation. Bubbling was carried out in a column using 119 mL of solvent. The concentrations of NaOH in the solvent were 1%, 2%, 3%, 4%, and 5% (mass basis). NaHCO₃ was added to the solvent at mass ratios of 0:1, 1:1, 2:1, and 3:1 to NaOH, while Na₂CO₃ was added to the solvent to the added NaHCO₃ at a mass ratio of 1.26:1 due to the stoichiometric requirement of the R.4 reaction. The feed gas used was a mixture of 2,000 mL/min CO₂ and 1,000 mL/min N₂.

2.2 Continuous Experiment (Once-Through Process)

This experiment was conducted to obtain the L/G ratio that optimally minimizes CO_2 absorption. The obtained ratio was used in the selective H₂S absorption simulation. In this experiment, the concentration of NaOH in the solvent was kept constant at 5%-mass without the addition of Na₂CO₃ or NaHCO₃. The solvent flowrates were in the ranges of 18 to 21 mL/min, 900 to 1,200 mL/min, and 1,800

1050

mL/min, which respectively represented L/G ratio ranges of 0.006 to 0.007, 0.300 to 0.400, and 0.600.

2.3 Aspen Plus v10 Simulation

The simulation was carried out using the rating-based mode in a 4-cm diameter packing column. It was packed with Norton Raschig rings with a diameter of 40 cm and a height of 6 mm. The packing sections represented the two stages of the absorption process. The solvent was fed into the column at a flowrate of 18 mL/min at 25 °C and 1.2 bar. The concentration of NaOH in the solvent was varied at 1%, 2%, 3%, 4%, and 5% (mass basis). NaHCO₃ was added to the solvent at mass ratios of 0:1, 1:1, 2:1, and 3:1 to NaOH, while Na₂CO₃ was added to the solvent at a mass ratio of 1.26:1 to the added NaHCO₃. The feed gas was flowed into the column at a flowrate of 3,000 mL/min at 25 °C and 1.2 bar. The corresponding composition of the gas was CO₂, CH₄, and H₂S with molar concentrations of 66.67%, 33.30%, and 0.03%, respectively, representing the typical AGEU feed gas composition. The absorption reactions and other reactions of Aspen Plus v10.

Raw data in the form of H_2S and CO_2 concentration (volume basis) and time that had been collected from experimental variations were collected and calculated. The research data interpretation was obtained in the form of absorbed CO_2 percentage, absorbed H_2S percentage, and H_2S selectivity according to Eqs. (1) to (3).

$$%CO_{2abs} = \left(1 - \frac{C_{CO_2,out}}{C_{CO_2,in}}\right) \times 100\%$$
 (1)

$$\% H_2 S_{abs} = \left(1 - \frac{C_{H_2 S,out}}{C_{H_2 S,in}}\right) \times 100\%$$
 (2)

$$S_{H_2S} = \frac{(\%H_2S_{abs})^2}{\%CO_{2abs}}$$
(3)

3 Experimental Result and Discussion

3.1 Preliminary Bubbling Experiment

This experiment was conducted as validation of this batch experiment with the benchmark conducted in previous research [19]. The reactions involved are R.5 to R.8, which take place simultaneously. The provided equilibrium constants (at 25 °C) were taken from [16].

$$\mathrm{CO}_{2\,(\mathrm{g})} \longrightarrow \mathrm{CO}_{2\,(aq)} \tag{R.5}$$

$$CO_2 + OH^- \rightleftharpoons HCO_3^-, K_{eq} = 1.20 \times 10^{-16}$$
 (R.6)

$$HCO_3^- + OH^- \Leftrightarrow CO_3^{-2} + H_2O, K_{eq} = 8.38 \times 10^{-13}$$
 (R.7)

$$CO_{3(aq)}^{2-} + CO_{2(aq)} + H_2O_{(l)} \rightleftharpoons 2HCO_{3(aq)}^{-}, K_{eq} = 1.43 \times 10^{-4} \quad (R.8)$$

The results of the current experiment with a similar system and solvent concentration are shown in Figure 2. It was observed that the absorption activity followed reactions R.5 to R.7, which occurred continuously until no OH⁻ ions remained in the solvent, which is consistent with the benchmark [19,20].



Figure 2 Absorbed CO_2 percentage profile for CO_2 pre-run bubbling in 200 mL of 5%-mass NaOH solution.

This phenomenon is represented by the peak of the curve that occurs up until the 20^{th} minute. The exhaustion of OH⁻ ions causes the solvent to saturate with CO₃²⁻ ions so that the R.7 reaction equilibrium is shifted to the left (inversion). The combined reaction of reaction R.6 and the inversion of reaction R.7 is reaction R.8. This reaction continues until the system reaches equilibrium, where the solvent is dominated by HCO₃⁻ ions. This phenomenon is represented by the curve in Figure 2 from the 20th minute to the 120th minute. The flat trend at the

end of the curve shows the maximum physical CO_2 absorption capacity where the solvent cannot absorb CO_2 any further.

3.2 CO₂ Absorption at Varied Concentrations of NaOH, NaHCO₃, and Na₂CO₃

Figure 3 shows the CO₂ absorbed percentage at varied concentrations of NaOH (1-5 %-wt), NaHCO₃, and Na₂CO₃. It can be observed that the effect of absorption time did not have a significant effect on the CO₂ absorption percentage since the peak points of all lines are at relatively the same time as each other. At the highest point of the CO₂ absorption percentage from each line, the addition of NaHCO₃ and Na₂CO₃ buffer could reduce the CO₂ absorption activity. In addition, the equilibrium concentration (CO₂ concentration at the end of the time) depends on the amount of added NaHCO₃ and Na₂CO₃ buffer. This is thought to occur because of a further equilibrium reaction that acts on the system when the NaOH runs out, as presented in reaction R.8 [20,21].



Figure 3 Absorbed CO₂ percentage profile from CO₂ bubbling experiment.

As shown in Figure 3, the variation of NaHCO₃ to NaOH mass ratio of 1:1 and Na₂CO₃ to NaHCO₃ mass ratio of 1.26:1 addition was able to absorb CO₂ after its maximum absorption curve, which is indicated by the CO₂ absorption percentage value excessing 0%. This indicates that there were still more carbonate ions than bicarbonate ions, which consequently shifted the equilibrium of reaction R.8 to the right. This mechanism explains the trend that after the maximum absorption curve peak, a small amount of CO₂ gas was still absorbed.

The variation of NaHCO₃ to NaOH mass ratio of 2:1 and Na₂CO₃ to NaHCO₃ mass ratio of 1.26:1 addition proved that there was no further CO₂ absorption after the maximum absorption curve, which is indicated by the CO₂ absorption percentage value of approximately 0%. This indicates that the R.8 reaction shifted to the left and reached equilibrium faster because reactions R.3 and R.4 had already reached equilibrium due to the addition of NaHCO₃ and Na₂CO₃ buffer

into the solvent at the beginning of the experiment. Aside from accelerating reaction R.8 equilibrium to occur, the addition of NaHCO₃ and Na₂CO₃ simultaneously withstood CO₂ absorption by suppressing the forward reaction of reactions R.3, R.4, and R.8 consecutively. Although at the beginning more Na₂CO₃ was added than NaHCO₃, the CO₃²⁻ initiated the acceleration of HCO₃⁻ species accumulation over time so that the number of HCO₃⁻ ions could exceed the number of CO₃²⁻ ions. This mechanism explains why after the maximum absorption curve peak, no more CO₂ gas was absorbed.

The variation of NaHCO₃ to NaOH mass ratio of 3:1 and Na₂CO₃ to NaHCO₃ mass ratio of 1.26:1 addition resulted in a higher concentration of CO₂ gas than the initial value in the column gas output stream, which is indicated by the negative value of the CO₂ absorption percentage. It is estimated that there were more HCO_3^- ions than $CO_3^{2^-}$ ions since there were already more HCO_3^- ions in the solvent due to the addition of a higher amount of NaHCO₃ at the beginning, so that reaction R.8 equilibrium shifted to the left. This mechanism explains the trend that after the maximum absorption curve peak, the CO₂ concentration appeared to increase. A negative CO₂ absorption percentage should be avoided because in practice it could increase the amount of CO₂ emitted in the AGEU, which can be done by limiting the buffer addition to solvent below this ratio.

It can be concluded that the solvent containing NaOH and NaHCO₃ with a mass ratio of NaHCO₃:NaOH 2:1 and Na₂CO₃ with a mass ratio of 1.26:1 to NaHCO₃ is able to hinder CO₂ absorption at equilibrium condition. This condition can be used to achieve the H₂S absorption selectivity of a mixture of H₂S, CO₂, and CH₄ gases using NaOH. By minimizing the NaOH used to absorb CO₂, more NaOH will be available to absorb H₂S. The effect of NaHCO₃ and Na₂CO₃ addition is aligned with previous studies that suggested that reactions in the liquid solvent play a prominent role in achieving H₂S absorption selectivity [22,23].

3.3 CO₂ Absorption Experiment at Varied L/G Ratio (Once-Through Process)

The hydrodynamic constraint of the absorber column resulted in an acceptable L/G ratio from 0.006 to 0.600. The lowest L/G ratio obtained was 0.006 since the liquid flowrate would be unstable and tend to stop at lower L/G, while the highest L/G ratio of 0.600 was due to column hold-up flooding.

The peak point shown in Figure 4 indicates the saturation of the column hold-up following reactions R.6 and R.7. A difference occurs when the curves decrease after having reached their peak, where in L/G variation from 0.200 to 0.600 the curves do not return to their initial point. The large L/G ratio allows the column to be filled quickly with fresh solvent so that the liquid hold-up is not completely

saturated and limits reaction R.8. Meanwhile, at the lowest L/G of 0.006, the hold-up is saturated until it starts reaction R.8. After the peak, the curves tend to be stable, which represents system equilibrium. The contact time is the key factor, which distinguishes the equilibrium position of the respective L/G ranges.



Figure 4 Absorbed CO_2 percentage profile for the continuous once-through experiment for various L/G ranges with 5%-mass NaOH solvent solution without the addition of Na2CO₃ and NaHCO₃ buffer.

The L/G variants of 0.200 to 0.600 indicate a constant activity of CO_2 absorption caused by a short contact time, since the L/G ratios are high, so that the supplied OH⁻ is sufficient to carry out the continuous absorption of CO_2 . Meanwhile at an L/G ratio of 0.006, the low fresh solvent rate causes a high contact time and allows the hold-up to saturate with HCO₃⁻, which consequently shifts the R.8 reaction equilibrium to the left. This explains the negative curve position, which indicates an increase in CO₂ content in the treated gas. It can be concluded that a small L/G ratio allows a reduction of CO₂ absorption activity due to limited solvent to absorb CO₂ after absorbing H₂S close to equilibrium. On the other hand, a large L/G ratio does not guarantee that the reduction of the CO₂ absorption activity can be controlled precisely, because the low contact time reduces the buffer performance.

3.4 Prediction of H₂S Absorption Selectivity using Aspen Plus v10

Validation was carried out to test the simulation blocks built by comparing the simulation results with the results from previous research [18]. The validation simulation was carried out in a 0.15-m diameter and 2.35-m high column with Flexipac KOCH 500Y Metal type internal to match the benchmark. The feed solvent was 0.5 g/L caustic. The feed gas compositions were 60% CH₄, 60 ppm H_2S , and balanced CO₂ with a flowrate of 90 kg/h in 1 atm and 9 °C process

condition. Figure 5 shows the simulation validation results. The modeling approach developed in this study provides great accuracy, as it differed only 0.95% from the benchmark.



Figure 5 Simulation validation results compared to previous research [18].



Figure 6 Absorbed CO_2 and H_2S percentage at various NaOH concentrations with Na_2CO_3 to $NaHCO_3$ mass ratio of 1.26:1 and L/G ratio of 0.006. Left: at various concentrations of $NaHCO_3$. Right: at $NaHCO_3$:NaOH mass ratio of 2:1.

A follow-up simulation was conducted at two different L/G ratios (0.006 and 0.030) based on the result discussed in Section 3.3. The L/G ratio of 0.006 was chosen to minimize the solvent required in consideration of economic feasibility, while the L/G ratio of 0.030 was chosen due to the column flooding limitation.

Based on the simulation at various L/G ratios, an L/G value of 0.250 corresponds to the 80% flooding condition limit, hence the simulation was carried out with an L/G value of less than 0.250.

Figure 6 shows the results of the CO₂-CH₄-H₂S gas system absorption simulation at the optimum buffer concentration, i.e., at an NaHCO₃ to NaOH mass ratio of 2:1, an Na₂CO₃ to NaHCO₃ mass ratio of 1.26:1, and an L/G ratio of 0.006. It can be seen that the addition of NaHCO₃ and Na₂CO₃ buffer system at the designated ratio could reduce the CO₂ absorption activity, thereby increasing the selectivity of H₂S absorption. In the absorption area of solvent with an NaOH concentration below 2.3%-mass, the CO₂ and H₂S absorption percentage decreased and increased, respectively. This occurred because the added buffer underwent the R.4 equilibrium reaction, so that the number of OH⁻ ions increased.

The OH ions produced reacted with H_2S and CO_2 based on reactions R.1 and R.3, so that an increase in the absorption percentage profile of the two components could be observed. This phenomenon differed from the absorption area of solvent with an NaOH concentration above 2.3%-mass. The decrease in the H_2S absorption percentage and the brief increase in the CO_2 absorption percentage was likely affected by the reduced solubility of the gas at higher solvent component concentrations. In particular, the decrease in the CO_2 absorption percentage was also affected by the increased amount of Na_2CO_3 and $NaHCO_3$ added.

Figure 6 also shows that the concentration of NaOH in the solvent is directly proportional to the activity of CO_2 absorption at no-buffer condition (mass ratio of 0:1). In addition, it can also be seen that there is a peak point on each of the NaOH concentration lines for the absorption of CO_2 . These peak points ultimately divide the CO_2 absorption percentage profile area into three regions, namely before the peak point, after the peak point, and after the NaHCO₃ mass ratio of 1.5:1 to NaOH. Before the peak point, the addition of buffered Na₂CO₃ and NaHCO₃ was not sufficient to reduce the CO_2 absorption activity. However, in this area, selectivity values greater than 2 were still obtained due to the inversion of the R.4 reaction, which produced OH ions.

The OH ions produced reacted with the H_2S and CO_2 based on reactions R.1 and R.3, so that an increase in the percentage profile of the two components could be observed. After the peak point, high H_2S selectivity values (3 to 6) occurred because the absorption of CO_2 was reduced due to the addition of buffer into the solvent, whose concentration was sufficient to reduce the CO_2 absorption activity. In the region after the NaHCO₃ to NaOH mass ratio of 1.5:1, a maximum selectivity value greater than 7 could be observed. The higher NaOH concentration could reduce the CO_2 absorption activity because the buffer was

added proportionally to the amount of NaOH added to the solvent at the same mass ratio.

4 Conclusion

This research concludes that the addition of NaHCO₃-Na₂CO₃ buffer is inversely proportional to the CO₂ absorption percentage and hence it can inhibit CO₂ absorption. The L/G ratio is directly proportional to the CO₂ absorption percentage. Based on the simulation results, the combination of an L/G ratio of 0.006 using a solvent mixture of 5%-mass NaOH, 15%-mass NaHCO₃, and 18.9%-mass Na₂CO₃ resulted in the highest H₂S selectivity at 23.1 (increase in H₂S selectivity by 379.7%).

The combination of an L/G ratio of 0.006 using a solvent mixture of 5%-mass NaOH, 10%-mass NaHCO₃, and 12.6%-mass Na₂CO₃ resulted in the optimum H_2S selectivity at 9.6 (increase in H_2S selectivity by 123.1%). The proposed combination satisfies the SO₂ emission threshold limit of 2,600 mg/Nm³, thus it complies with Indonesian regulations. A future experiment to validate the provided selective absorption prediction is suggested, specifically for the proposed solvent composition.

Nomenclature

C _{CO2,in}	=	CO ₂ input concentration (%-vol.)
C _{CO2,out}	=	CO ₂ output concentration (%-vol.)
C _{H2S,in}	=	H ₂ S input concentration (%-vol.)
C _{H2S,out}	=	H ₂ S output concentration (%-vol.)
L/G	=	Ratio of solvent volumetric flowrate to gas volumetric flowrate
$S_{\rm H2S}$	=	H ₂ S absorption selectivity to CO ₂ absorption (Equation 3)
%CO _{2abs}	=	CO ₂ absorption percentage (Equation 1)
H_2S_{abs}	=	H ₂ S absorption percentage (Equation 2)

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