

## 8 Scientific articles

### 8.1 The effect of 1,4-butanediol on the efficiency of CO<sub>2</sub> absorption and inhibition of NH<sub>3</sub> escape during the mineral carbonation of gypsum

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#### Abstract

Effectively and affordably advancing methodology for selectively capturing and storing CO<sub>2</sub> from diverse sources is one of the central challenges in addressing climate change. Ammonia solutions, highly valued for advantages like improved absorption efficiency and reduced regeneration energy needs, face a problem due to ammonia's high volatility, causing desorption during gas flow. Nevertheless, ammonia solutions as the CO<sub>2</sub> absorption solvent are proposed for reasons of cost-effectiveness, high capacity, and versatility. Various studies employ inorganic, organic, and nanoparticles as additives to tackle this issue. In this particular study, different concentrations of 1,4-butanediol were utilized to counteract ammonia escape and enhance CO<sub>2</sub> absorption.

This study explored the optimal concentration of 1,4-butanediol for maximizing CO<sub>2</sub> absorption and minimizing ammonia desorption. Mineral carbonation of gypsum slurry was carried out in a bubble reactor with a magnetic stirrer maintaining constant speed for 1 hour. The gas introduced to the reactor was a mixture of CO<sub>2</sub> and air. The gas flow was controlled by the mass controllers. Gas sensors were used to determine the concentration of CO<sub>2</sub> and NH<sub>3</sub> in the outlet gas. The initial and final pH values were measured. The liquid phase was ammonia solutions at a concentration of 1.69 mol·dm<sup>-3</sup> with the addition of varying concentrations of 1,4-butanediol (ranging from 0.1 to 0.5 mol·dm<sup>-3</sup>). The ratio of gypsum to liquid phase was maintained as 1:14 (kg·dm<sup>-3</sup>). Based on measured CO<sub>2</sub> and NH<sub>3</sub> concentrations, the efficiency of carbon dioxide absorption and the efficiency of ammonia escape inhibition were calculated.

Results demonstrated a direct correlation between increased additive weight, enhanced CO<sub>2</sub> absorption, and effective inhibition of ammonia escape. The addition of 1,4-butanediol enhances CO<sub>2</sub> dispersion within the solution, expanding the gas-solvent contact area and promoting a more effective absorption process. Therefore, it effectively inhibits the escape of ammonia during reactions by temporarily binding free ammonia in the solution. The application of 1,4-butanediol emerges as a promising strategy for optimizing the overall efficiency and performance of the studied processes.

#### Introduction

CO<sub>2</sub>, recognized as a heat-trapping and greenhouse gas, experiences a notable increase in concentration primarily attributed to the extraction and combustion of fossil fuels such as coal, natural gas, and oil (Soeder, 2021). Over the span from the first industrial revolution in the 18th century to the present day, the concentration of CO<sub>2</sub> has surged by 50% (Simmer et al., 2023). According to the 2022 Our World in Data Report, the primary contributor to atmospheric CO<sub>2</sub> concentration, illustrated in (Figure 8.1.1 (a)), is presently the Asian continent, particularly China and India, collectively accounting for approximately 53% of total emissions. The United States contributes 18% to the total emissions released into the atmosphere, while the European Union (EU) contributes 17%, Africa, with a focus on South Africa, South America, Brazil, and other unlisted regions each contributes 4% to the total emissions (Hannah Ritchie, 2023). As depicted in (Figure 8.1.1(b)). When expressed as a percentage of the total cumulative emissions starting from 1750 to 2022, the United States accounts for approximately 25%, the EU for around 22%, China for about 18%, the United Kingdom (UK) for 8%, India for 6% and other countries collectively contributing a smaller share compared to the mentioned nations. The increasing CO<sub>2</sub> concentration in the atmosphere can cause climate change, ocean acidification that impacts living things (i.e. plants and animals), drought, etc (Hunter, 2007). Therefore, exploring different methods of permanently capturing CO<sub>2</sub> from the atmosphere is necessary to sequester the huge amount of CO<sub>2</sub> released by human activities.

There are different methods and techniques are employed to capture CO<sub>2</sub> from the gas mixture such as absorption, adsorption, and membrane methods. In absorption amine-based solvents, ammonia, and others are used. Adsorption utilizes solid sorbent and liquid sorbent. Membrane

uses different materials as a membrane to separate it from other components (Wu et al., 2020). These are also the well-known methods to capture CO<sub>2</sub> from the atmosphere, flue, and waste gases like biological processes, mineral carbonation and cryogenic processes (Bui et al., 2018; Castro-Muñoz et al., 2022; Cheng et al., 2023; Galina et al., 2023; Goli et al., 2016; Kim et al., 2023; Zajac et al., 2023). Gypsum is utilized in the mineral carbonation process to capture CO<sub>2</sub> from its source. It is the process where divalent metal ions react chemically with carbon dioxide,

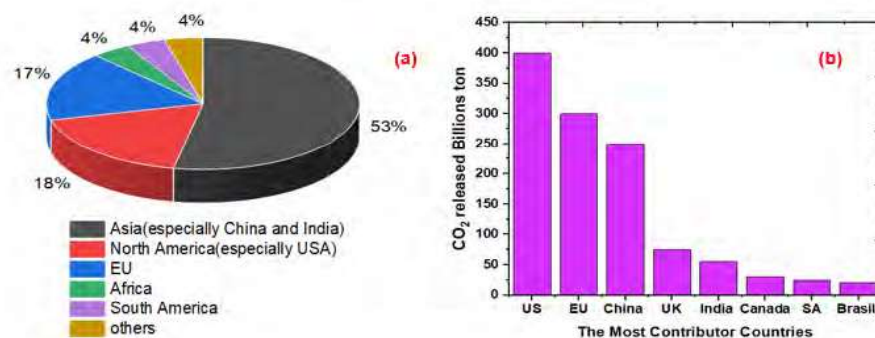


Figure 8.1.1 (a) World CO<sub>2</sub> concentration emission: (b) cumulative CO<sub>2</sub> emission. Source: Global Carbon Project (2022) published on: Our World in Data

forming carbonates. The mineral carbonation can be categorized into direct and indirect forms. Indirect mineral carbonation involves a sequential extraction and carbonation process to produce metal carbonate products. On the other hand, direct mineral carbonation consists of a single step of carbonation to yield carbonated products.

For the CO<sub>2</sub> absorption process, different solvents such as monoethanolamine (MEA), diethanolamine (DEA), methyl diethanolamine (MDEA), and ammonia are particularly effective for absorbing carbon dioxide due to their cost-efficiency, and stability. It is established that monoethanolamine (MEA) and diethanolamine (DEA) exhibit optimal performance at lower temperatures compared to methyl diethanolamine (MDEA) (de Ávila et al., 2015).

Currently, ammonia attracts attention over other amine-based solvents due to its affordable, having a high CO<sub>2</sub> capture capability, and not degrading in the presence of O<sub>2</sub> and SO<sub>2</sub> (Amibo & Konopacka-Lyskawa, 2024). Aqueous ammonia solutions also have the potential to absorb other acid gases than carbon dioxide, such as NO<sub>x</sub> and SO<sub>x</sub> found in exhaust gases. Additionally, processes that use NH<sub>3</sub> solutions have a low demand for energy (Liu et al., 2009; Wang et al., 2018). The main problem employing ammonia during CO<sub>2</sub> absorption is NH<sub>3</sub> desorption during the gas flow. To prevent the escape of ammonia, varying amounts of organic additives are used.

Various studies have shown the positive effect of various organic additives to increase the efficiency of CO<sub>2</sub> absorption and reduce NH<sub>3</sub> desorption. Seo et al. (Seo et al., 2012) found that the inhibition of ammonia escaping in the absorption system by the addition of organic additives such as glycine, ethylene glycol, and glycerol was 5.1%, 38.4%, and 59.91%, respectively. In addition to this, another study reported that glycerine and isoamyl alcohol has a high capacity to inhibit ammonia escaping by 46.38% and 42.87%, respectively (Shuangchen et al., 2013). In the study presented by Czaplicka et al. (Czaplicka et al., 2022), the effects of organic additives on CO<sub>2</sub> absorption and CaCO<sub>3</sub> precipitation during mineral carbonation were presented. In this investigation, neopentyl glycol (NPG) had a greater tendency to inhibit ammonia escaping in both the absorption and the precipitation system. Improvement of CO<sub>2</sub> capture was 39.1%, and 83.1% was for NH<sub>3</sub> desorption. The experimental finding reports remarkable results that were obtained by using NPG as an organic additive, and the high amounts of CO<sub>2</sub> absorbed in both absorption and precipitation systems were 85.4% and 86.1%, respectively.

Carbon dioxide can be efficiently captured and managed through the incorporation of gypsum in the presence of a CO<sub>2</sub>-absorbing solvent like ammonia and organic additives. In this research gypsum (CaSO<sub>4</sub>·2H<sub>2</sub>O) was used as a source of divalent metal ions (Ca<sup>2+</sup> ions). The introduced CO<sub>2</sub> reacted with calcium ions and formed calcium carbonate whereas ammonia reacted with sulfate ions and formed ammonium sulfate, which is a valuable fertilizer. In this work, the gypsum carbonation process was performed in the presence of an organic additive 1,4-butanediol. The main objective of this research was to determine the influence of 1,4-butanediol on CO<sub>2</sub> absorption efficiency in the aqueous gypsum suspension containing ammonia. The second objective was to determine the effects of the organic additive on ammonia desorption during the carbonation process.

## Methods

During the experiments, the following chemicals were utilized: ammonia solution of 25% ( $\geq 96.0\%$ ; POCH, Poland), gypsum (CaSO<sub>4</sub>·2H<sub>2</sub>O) ((Chempur, Poland)), 1,4-butanediol (99.0%, Acros-Organics, Taiwan), water from reversed osmosis, CO<sub>2</sub> (Oxygen s.c., Poland), hydrochloric acid (35-38%, POCH, Poland), and sodium hydroxide (POCH, Poland).

In this research, a liquid-gas carbonation method was employed. The glass bubble reactor with capacity of 0.5 dm<sup>3</sup> equipped with the fritted glass as a gas distributor and the magnetic stirrer was used for the batch carbonation experiments. The reactor was filled with gypsum, ammonia solution, and organic additives. The mass of gypsum was 7% wt./wt. of the solution, the concentration of ammonia used was 1.68 mol·dm<sup>-3</sup>, and 1,4-butanediol having the concentration of 0.1 mol·dm<sup>-3</sup> to 0.5 mol·dm<sup>-3</sup> were utilized. The total volume of liquid was 0.3 dm<sup>3</sup>. Before and after the reaction of carbonation the pH of the solution was measured. The inlet gas was a mixture of air and CO<sub>2</sub> and its flow was regulated at constant flow rates by employing a mass flow controller. The air and CO<sub>2</sub> flow rates were maintained at 0.007083 dm<sup>3</sup>·s<sup>-1</sup> and 0.00125 dm<sup>3</sup>·s<sup>-1</sup>, respectively, to obtain the volume fraction of CO<sub>2</sub> in the air mixture 15%. The air from the source was passed through the adsorption column with NaOH to remove the air humidity. A magnetic stirrer was adjusted at 300 rpm. The outlet gas was diluted with the air controlled by the mass controller and the unreacted CO<sub>2</sub> and ammonia were analysed by the specific sensors. The detailed scheme of used experimental setup was described by Czaplicka et al. (Czaplicka et al., 2022). The reaction was performed at room temperature and atmospheric pressure for 1 hour. After the carbonation process, the suspension was filtered using a vacuum filter and the obtained precipitate was dried overnight at a temperature of 105 °C.

## Results and Discussion

### pH of the reaction mixtures

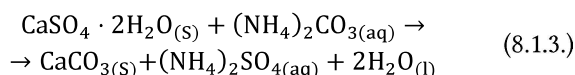
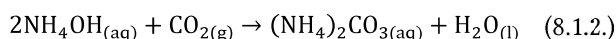
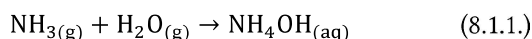
As shown in (Table 8.1.1), the initial pH values before the carbonation of gypsum slurry containing ammonia and organic additive varied from 12.28 for the control process (without 1,4-butanediol) to 11.71 for the highest tested concentration of the used diol. After carbonation the pH value of the reaction mixture decreased and the recorded values were from a range of 9.31 to 9.98. The other study on mineral carbonation supports that the pH of the solution decreased after the carbonation experiment (Huang et al., 2022; Mattila & Zevenhoven, 2015). The reason for lowering the pH is the absorption of CO<sub>2</sub> and the subsequent formation of carbonic acid in the solution. When hydrolysed, increases the concentration of hydrogen ions and increases the dissociation of ammonia. Moreover, during the precipitation of calcium carbonate in the solution, the concentration of sulfate ions increases, which also contributes to lowering the pH.

**Table 8.1.1** pH values of reaction mixture before and after the carbonation experiment

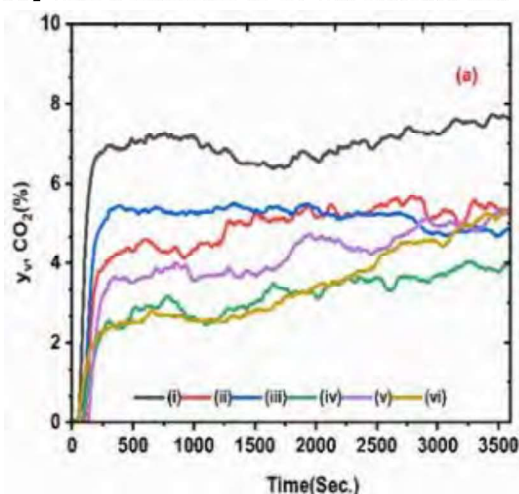
Additive concentration, mol·dm <sup>-3</sup>	Initial pH	Final pH
0.0	12.28	9.98
0.1	12.24	9.89
0.2	11.99	9.49
0.3	11.89	9.42
0.4	11.87	9.42
0.5	11.71	9.31

### Carbon dioxide absorption

During the carbonation of gypsum slurry containing ammonia, several reactions corresponding to the Mersburg process occur (Avşar et al., 2022). The first reaction described by Equation (8.1.1.) shows the conversion of ammonia into ammonium hydroxide. The NH<sub>4</sub>OH formation makes the solution alkaline medium. Equation (8.1.2.) implies CO<sub>2</sub> introduced reacted with ammonium hydroxide and then form ammonium carbonate. Equation (8.1.3.) is an overall reaction between the gypsum and ammonium carbonate and forms CaCO<sub>3</sub>, (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, and H<sub>2</sub>O. Unreacted CO<sub>2</sub> and desorbed NH<sub>3</sub> leave the solution in the outlet gas stream.



The concentration of carbon dioxide in the exhaust gas is recorded in percent of volume fraction. (Figure 8.1.2(a)) shows changes in CO<sub>2</sub> concentration in the exhaust gas over time for randomly selected experiments. When the concentration of organic additive 1,4-butanediol increased the outlet percentage volume fraction of CO<sub>2</sub> was decreased. In the case of the control experiment (*without organic additive*), the volume fraction of CO<sub>2</sub> ranged between 6-8% means from the total introduced CO<sub>2</sub> from the source more than 42% left the reactor without reaction. However, when 0.5 M concentration of 1,4-butanediol was added, a percentage volume fraction of CO<sub>2</sub> with a maximum of 4% was recorded and

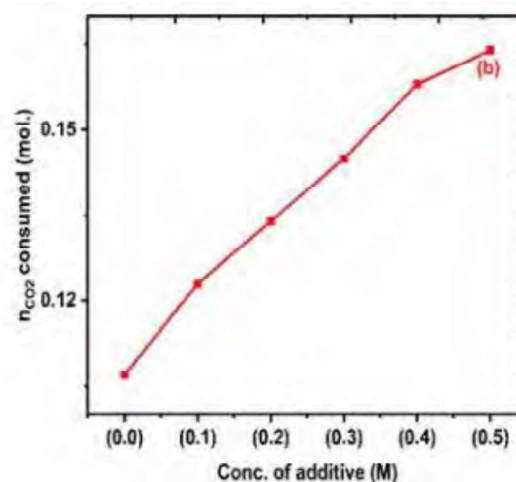


unreacted CO<sub>2</sub> left the reactor at an average of around 12%. The percentage volume fraction of CO<sub>2</sub> vs. time diagrams are similar in shape and they have four main stages such as in stage I, stage II, stage III, and stage IV. Stage I shows the complete consumption of introduced CO<sub>2</sub> within the reactor due to the high alkalinity solution favoring the consumption of CO<sub>2</sub>. Next in stage II, the outlet CO<sub>2</sub> concentration sharply rises until it reaches to stable point. This substantial increase makes a high amount of CO<sub>2</sub> released from the solution. Stage III is an almost stable stage but the CO<sub>2</sub> concentration slightly fluctuates and its near-constant values. This stage continues for a long time from around 500 to 2500. In stage IV, a slight increase in outlet percentage volume fraction of CO<sub>2</sub>. Based on the recorded CO<sub>2</sub> concentration in the inlet and outlet gas, the number of moles of carbon dioxide captured during the process was calculated, and the results are presented in (Figure 8.1.2(b)). It can be seen, that the number of moles consumed increased when the concentration of additives increased. Previous results also indicated that an organic compound could promote CO<sub>2</sub> absorption, but the effect of additive concentration was not investigated (Czaplicka et al., 2022; Konopacka-Łyskawa, Amibo, et al., 2023).

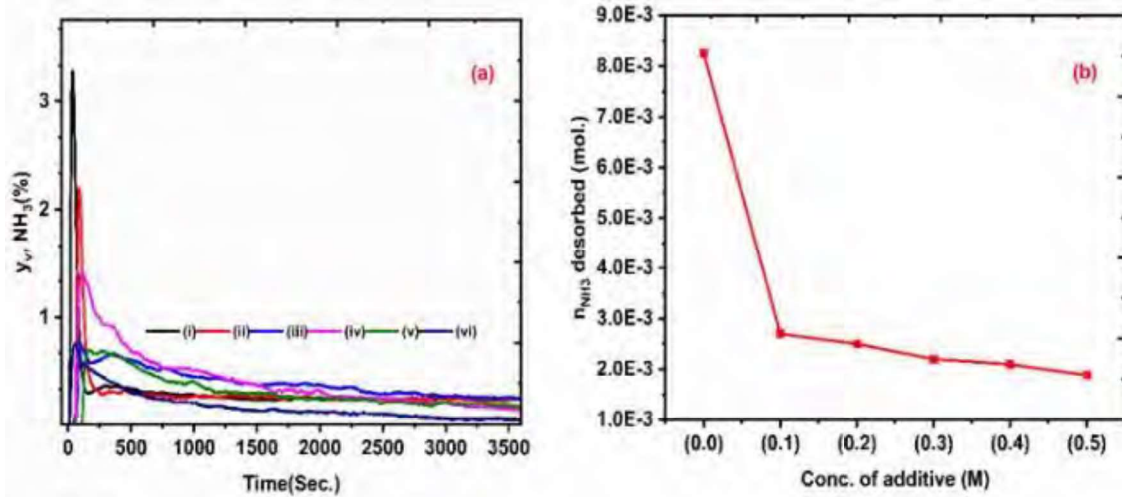
### Ammonia escaping

In the percent volume fraction of ammonia recorded over the time of reaction is presented. Generally, the course of ammonia desorption can be divided into four stages. In stage I, there is no recorded ammonia at outlet streams but as time goes a small amount of ammonia escape from the reactor this stage is termed stage II. In stage II, the ammonia concentration initiates an upward trend and reaches the maximum point.

Then, the ammonia concentration starts to decline. Beginning from this spot until it becomes stable this stage is termed stage III. Next, the final stage is in a quasi-stable state, i.e., stable with small changes and it starts around 3000 to the end of the reaction. Based on the results of the NH<sub>3</sub> concentration in the exhaust gas, the total number of moles of desorbed ammonia was calculated and is shown in (Figure 8.1.3 (b)). It can be seen that the amount of escaped ammonia significant decrease when the concentration of additives increase.



**Figure 8.1.2** (a) Percent volume fraction of CO<sub>2</sub> (i) control; (ii) Concentration of 0.1 M additive; (iii) Concentration of 0.2 M additive; (iv) Concentration of 0.3 M additive; (v) Concentration of 0.4 M additive and (vi) Concentration of 0.5 M additive; (b) the amount of average mole CO<sub>2</sub> consumed at different additive concentration.



**Figure 8.1.3** (a) Percent volume fraction of NH<sub>3</sub> (i) control; (ii) Concentration of 0.1 M additive; (iii) Concentration of 0.2M additive; (iv) Concentration of 0.3M additive; (v) Concentration of 0.4M additive and (vi) Concentration of 0.5 M additive; (b) the amount of average mole NH<sub>3</sub> escaped from the reactor at different additive concentration.

### CO<sub>2</sub> absorption efficiency

The efficiency of CO<sub>2</sub> absorption was calculated using Equation (8.1.4.):

$$\eta_{\text{CO}_2} = \frac{n_{\text{Con.}}}{n_{\text{in.}}} \cdot 100 \% \quad (8.1.4.)$$

where  $\eta_{\text{CO}_2}$  denotes the efficiency of CO<sub>2</sub> absorbed during the mineral carbonation process,  $n_{\text{Cons.}}$  and  $n_{\text{init.}}$  denotes the mole of CO<sub>2</sub> consumed and the initial mole of CO<sub>2</sub>, respectively.

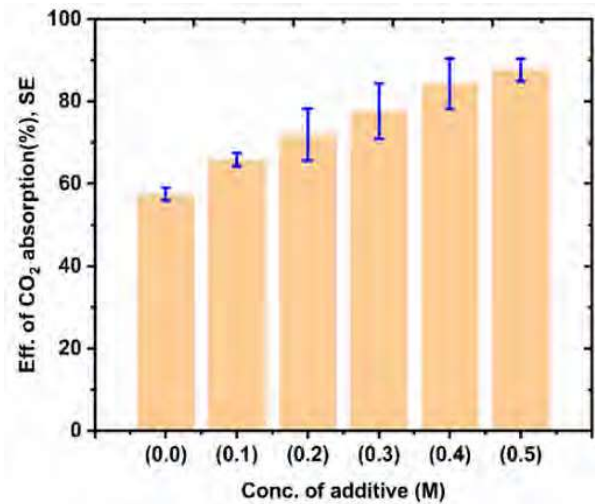
The mole CO<sub>2</sub> introduced into the system was calculated. The known values of CO<sub>2</sub> flow rate, the time of the reaction, and the amount of CO<sub>2</sub> consumed was calculated based on the CO<sub>2</sub> concentration in the outlet gas stream. From Figure 8.1.4, small amounts of additive enhance CO<sub>2</sub> absorption. As illustrated in the diagram, the efficiency for control CO<sub>2</sub> absorption was 57.47%, for 0.1 M, 0.2 M, 0.3 M, 0.4 M, and 0.5 M were 65.87%, 71.91%, 77.56%, 84.27%, and 87.65% respectively. From the study findings of Czaplicka et al. (Czaplicka et al., 2022), various organic additives such as glycerol, ethylene glycol, methanol, isopropanol, pentaerythritol, and neopentyl glycol were utilized. The calcium carbonate precipitation process with organic additives showed greater capability to absorb CO<sub>2</sub>. In this study, the highest CO<sub>2</sub> absorption efficiency was 86.1% by employing neopentyl glycol, and this result is comparable with the current finding of CO<sub>2</sub> absorption efficiency of gypsum carbonation.

### NH<sub>3</sub> escaping inhibition

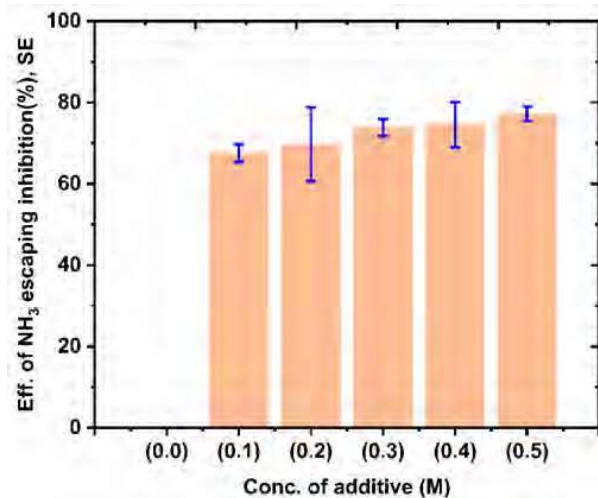
Due to its high vapor pressure, ammonia is volatile, evaporates, and escapes from the solution even at lower temperatures (Ma et al., 2016; Valera-Medina et al., 2021). In a similar manner, ammonia has a great tendency to escape from the solution during mineral carbonation. This makes it difficult to utilize ammonia during the CO<sub>2</sub> absorption process in the large-scale CO<sub>2</sub> absorption and mineral carbonation process. The amount of ammonia escape inhibited during mineral carbonation reaction can be calculated by using Equation (8.1.5).

$$\alpha_{\text{NH}_3} = \frac{n_{\text{C}} - n_{\text{A}}}{n_{\text{C}}} \cdot 100\% \quad (8.1.5.)$$

where  $\alpha_{\text{NH}_3}$ ,  $n_{\text{C}}$ , and  $n_{\text{A}}$  denotes for ammonia escaping inhibition, mole of ammonia escape inhibited without organic additive (*Control*) and mole of ammonia escape inhibited with different concentrations of 1,4-butanediol.



**Figure 8.1.4** Efficiency of CO<sub>2</sub> absorption with standard error.



**Figure 8.1.5** Efficiency of ammonia escaping inhibition with standard error.

In Figure 8.1.4, the efficiency of ammonia escaping inhibition and efficiency of ammonia escaping inhibition with standard error demonstrated. When the concentration of

organic additive increases the ammonia desorption decreases. At different concentrations of 1,4-butanediol i.e., 0.1 M, 0.2 M, 0.3 M, 0.4 M, and 0.5 M the ammonia escaping inhibition was 65.57%, 69.69%, 73.89%, 74.54, and 77.27% respectively. The highest amount of ammonia escaping inhibition was attained at a concentration of 0.5M of 1,4-butanediol. The organic additive reduces ammonia escape due to the ability of organic additive to make temporary bonds with ammonia and inhibit evaporation of ammonia during mineral carbonation reaction. Most organic additives have reactive functional groups like -OH and -NH<sub>2</sub> boosting their capacity to inhibit the evaporation through its bond (Czaplicka et al., 2020).

## Conclusion

This work presents the effect of 1,4-butanediol on CO<sub>2</sub> absorption and ammonia desorption during carbonation of a gypsum suspension in the presence of ammonia. It has been shown that even a small amount of diol (0.1M, i.e. 0.9% by weight) causes significant improvements in CO<sub>2</sub> capture and limits the escape of ammonia from the reaction mixture. The increase in the concentration of the organic additive both increased the amount of CO<sub>2</sub> absorbed in the mineral carbonation process and reduced the amount of desorbed ammonia. Therefore, the best results were obtained for the highest tested concentration of 1,4-butanediol, with the CO<sub>2</sub> absorption efficiency of 87.65% and the effectiveness of inhibiting ammonia escape of 77.27%. In conclusion, the addition of 1,4-butanediol into the reactive mixture during mineral carbonation of gypsum is an encouraging approach for optimizing CO<sub>2</sub> uptake capacity and minimizing ammonia desorption in this process.

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