



Experimental study on capture of carbon dioxide and production of sodium bicarbonate from sodium hydroxide

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ABSTRACT

Global warming due to greenhouse gases is an issue of great concern today. Fossil fuel power plants, especially coal-fired thermal power plants, are a major source of carbon dioxide emission. In this work, carbon capture and utilization using sodium hydroxide was studied experimentally. Application for flue gas of a coal-fired power plant is considered. Carbon dioxide, reacting with an aqueous solution of sodium hydroxide, could be converted to sodium bicarbonate (NaHCO_3). A bench-scale unit of a reactor system was designed for this experiment. The capture scale of the reactor system was 2 kg of carbon dioxide per day. The detailed operational condition could be determined. The purity of produced sodium bicarbonate was above 97% and the absorption rate of CO_2 was above 95% through the experiment using this reactor system. The results obtained in this experiment contain useful information for the construction and operation of a commercial-scale plant. Through this experiment, the possibility of carbon capture for coal power plants using sodium hydroxide could be confirmed.

Keywords: Carbon Capture, Carbon Utilization, Sodium Bicarbonate

1. Introduction

Over the past several decades, there has been growing international interest in limiting the emission of carbon dioxide and reducing greenhouse gases in the atmosphere. Excessive greenhouse gases in the atmosphere are responsible for various environmental problems, such as the increasing number of ocean storms and rising sea levels [1]. Carbon dioxide (CO_2) is the major contributor to global warming. According to a report by the Intergovernmental Panel on Climate Change (IPCC), the concentration of CO_2 in the atmosphere may be up to 570 ppm; it has caused a rise in the mean global temperature of 1.9°C and an increase in the mean sea level of 3.8 m [1]. One of the important parts of increasing CO_2 emission belongs to enhancing modern agriculture and irrigation systems [2, 3]. For increase in agricultural production and expanding cultivated area, water management system should be carefully planned and the climate change due to global warming should be considered [4, 5].

Carbon capture and storage (CCS) has been widely recognized as an effective strategy for reducing emissions of greenhouse gases and mitigating global warming [6, 7]. In the CCS process, carbon dioxide emitted from a point source, such as a coal-fired power plant, is captured and transported to storage sites. One of the most promising technologies among carbon capture processes is

the post-combustion amine process [8-11]. The amine process is has been a proven method for a relatively long time, and it is close to commercialization. However, it requires large-scale storage sites and steel is needed to reduce the energy penalty for re-generation of the amine absorbent [12].

Recently, utilization of carbon dioxide (carbon capture and utilization, CCU) has attracted attention [13]. CCU does not require storage sites, unlike CCS. CCU could be a cost effective option for the reduction of greenhouse gas because the capture costs could be offset by the products. CO_2 could be converted to valuable materials through chemical reaction with a reduction agent [14]. CO_2 could be sequestered as a carbonate or converted to usable materials, such as construction materials, through mineralization by suitable minerals [15].

This research studied the capture of carbon dioxide emissions from coal-fired power plants using sodium hydroxide (NaOH) experimentally. Carbon absorption in NaOH solution has been studied since the 1940 [16, 17]. Recently, Researchers have focused on capture of carbon dioxide in ambient air using the rapid reactivity of NaOH [18, 19]. CO_2 could be converted to sodium bicarbonate (NaHCO_3) through reaction with aqueous NaOH . Carbon capture capacity of NaOH has been investigated in mild concentration (1-5% NaOH solution) [20]. Sodium bicarbonate has a very wide range of uses. It can be used as baking soda, a cleaning agent,



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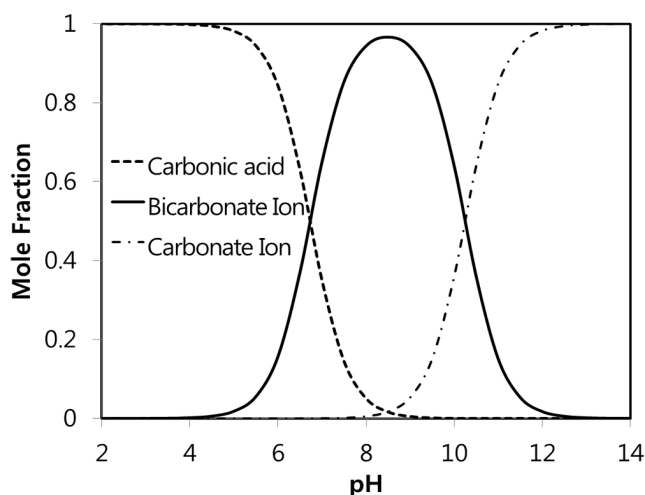


Fig. 1. pH vs. the mole fraction of carbonate species (Bjerrum plot).

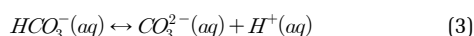
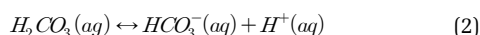
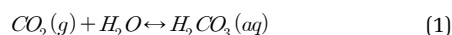
or a pH buffer in the chemical industry or the medical/ pharmaceutical industry. It can also be used as a desulfurization or denitrification agent in flue gas treatment. The sodium hydroxide required for carbon capture can be produced by a chlor-alkali process (a process for electrolysis of NaCl) [21].

Economic evaluation of a commercial-scale (100 tCO₂/d) plant using this process was already performed using the internal rate of return (IRR) and net present value (NPV) method [22]. Based on this economic evaluation, it could be concluded that this process is a cost-effective technology option for the reduction of greenhouse gas. The former works was theoretical study or lab scale experiment [16, 20]. A bench-scale reactor system (2 kgCO₂/d) was designed for this experiment. Through this experiment, the optimum operation conditions of the reactor system could be determined.

2. Theory

2.1. Reaction and Thermodynamics

Reaction chemistry and thermodynamics are major considerations in reaction engineering design. Carbon dioxide is a typical acid gas. First, gaseous CO₂ is dissolved in water, and it forms carbonic acid (H₂CO₃). The carbonic acid donates a proton (hydrogen ion, H⁺) and forms a bicarbonate ion (HCO₃⁻). The bicarbonate ion donates a proton and generates a carbonate ion (CO₃²⁻).

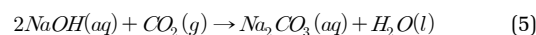


The equilibrium constant [23] at room temperature for Eq. (2) and (3) could be written as

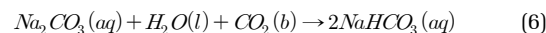
$$K_1 = \frac{[\text{HCO}_3^-][\text{H}^+]}{[\text{H}_2\text{CO}_3]} = 10^{-6.73}, \quad K_2 = \frac{[\text{CO}_3^{2-}][\text{H}^+]}{[\text{HCO}_3^-]} = 10^{-10.26} \quad (4)$$

If we introduce the pseudo-steady state condition (in this condition, $C_{\text{total}} \equiv [\text{H}_2\text{CO}_3] + [\text{HCO}_3^-] + [\text{CO}_3^{2-}] = \text{constant}$), the mole fraction of each carbonate species could be expressed as a function of pH. The mole fraction of carbonate species is plotted in Fig. 1, which is called Bjerrum plot [24].

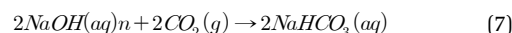
Reaction 1



Reaction 2



Overall reaction



CO₂ reacts with aqueous NaOH and forms sodium carbonate (Na₂CO₃) and sodium bicarbonate (NaHCO₃) in turn. These CO₂ absorption reactions are expressed by Eq. (5), (6) and (7). The thermodynamic parameters of each reaction are listed in Table 1.

Reactions 1 and 2 are exothermic (enthalpy of the reaction $H < 0$) and spontaneous (Gibbs free energy of the reaction $G < 0$), respectively. However, the Gibbs free energy value of reaction 2 is much smaller than that of reaction 1, and the reaction momentum of reaction 2 is also small. Therefore, the reaction rate of reaction 2 is very slow, in comparison with that of reaction 1.

Table 1. Thermodynamic Parameters of Each Reaction at Room Temperature [25]

	H (kJ/mol)	S (kJ/molK)	G (kJ/mol) = $H + S$
Reaction 1	-169.8	-0.137	-128.97
Reaction 2	-129.1	-0.334	-29.56

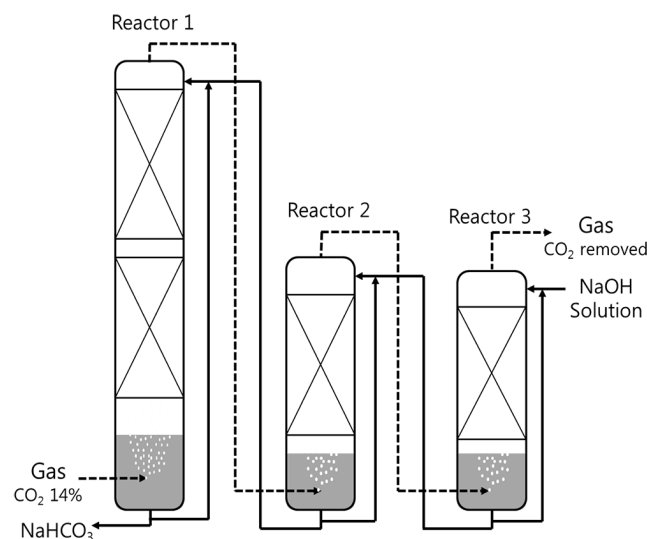


Fig. 2. Schematic diagram of the reactor system. Solid line represents line for liquid transfer and dashed line represents line for gas transfer.

2.2. Design of Reactor

The reaction rate and reaction conditions, such as pH, of reactions 1 and 2 are totally different. Therefore, the two reactions needed to be carried out in separate reactors for efficient reaction. Two or more reactors connected in a series were required to provide separate reaction conditions. The Hatta number (Ha) [26] is a dimensionless number that represents the ratio of the rate of reaction in the liquid film to the rate of transport through the film:

$$Ha = \frac{\sqrt{k_n C_{B,bulk} D_{CO_2}}}{k_L} \quad (8)$$

k_n and $C_{B,bulk}$ represent the rate constant of each reaction and concentration of the reactant (NaOH or Na_2CO_3), respectively. D_{CO_2} and k_L represent diffusion coefficient of CO_2 and mass transfer coefficient, respectively. If the reaction rate is small and Ha is smaller than 0.3, a bubble column reactor should be introduced. As mentioned previously, the reaction rate of reaction 2 is very slow compared to that of reaction 1. Ha for reaction 1 is 10^2 - 10^3 , while that of reaction 2 is 10^{-3} - 10^{-2} [27]. Therefore, a bubble column reactor should be used for efficient reaction.

The micro-sized holes of the spargers for making bubbles, which were small enough to have sufficient interface, were always clogged due to the precipitation of sodium carbonate. Bubbles could not be made small enough to secure a sufficient contact interface area. In addition, reaction 2 is very slow and requires additional space time. Thus, we combined a bubble column and a packed bed column into one reactor.

Fig. 2 is a schematic diagram of the reactor system. The inner diameter of the column was 8 cm, and a structured packing was used in the packed column. The height of the packing tower was 300-700 mm and 0.635 cm stainless steel tubing was used for liquid transport, and Teflon tubing was used for gas transport. Each reactor had a recirculation pump (Tuthill PGS 68) and a feeding pump (Masterflex L/S easy load). A pH meter, a thermometer (Orion Star A211), and pressure gauge were mounted on each reactor. At the upper end of each reactor, a CO_2 analyzer (KONICS KN-2000W) was installed.

3. Experimental methods

NaOH aqueous solution was prepared by dissolving NaOH powder (SAMCHUN Chemical, 98%) in distilled water. The concentration of the absorbent solution used in this experiment was 15 wt.%. The solubility of Na_2CO_3 (the intermediate product of this process) is less than 20 wt.% at room temperature [28]. Therefore, if the concentration of the absorbent is higher than 15 wt.%, the tubing system and gas sparger of the reactor could be clogged by precipitation of Na_2CO_3 . On the other hand, if the concentration is not high enough, the absorption rate is slow. The flow rate of the NaOH solution was 10-15 mL/min. For the feed gas, 14 volume % carbon dioxide gas balanced with nitrogen gas was used. (The flue gas of a coal-fired power plant contains 14 volume % of CO_2 .) The flow rate of the feeding gas was 5 L/min, which is equivalent to 2 kg CO_2 /d.

The pH of the liquid at the bottom of the reactors and the CO_2 concentration in the gas after each reactor ($C_{out,n}$) were measured every 5 min. As shown in Fig 1, pH is a major indicator of the progress of the reaction. The pH value of the first reactor was maintained at pH 8-9, while that of the second reactor was maintained at pH 9-10, and that of the third reactor was higher than pH 12. Therefore, the concentration of bicarbonate ions in reactor 1 and the CO_2 absorption rate in reactor 3 could be maximized. CO_2 absorption rate (γ_n) is defined as

$$\gamma_n = 100 \times \left(1 - \frac{C_{out,n} (100 - C_0)}{C_0 (100 - C_{out,n})} \right) \quad (9)$$

Here, C_0 is the volume percent of CO_2 in the inlet gas of the reactor system, and $C_{out,n}$ is the volume percent of CO_2 in the outlet gas of the n'th reactor. For the purity analysis of the product (sodium bicarbonate), quantitative X-ray diffraction was used.

To achieve optimum condition of the bench scale reactor system, three variables were selected.

- height of each packed column
- gas flow rate and gas injection point
- injection point of mother liquid

The height of each packed column is an important variable for economic design of the reactor. To avoid over-spec design or under-spec design, the height of packed column should be determined. The gas flow rate and gas injection point are necessarily determined to optimize the reactor system. The injection point of mother liquid should also be determined for stable and economic operation of the reactor system. Therefore the three variables were determined to optimize the bench scale reactor system.

Even though this reactor system is a bench scale unit, the experimental results could be influenced by various disturbances or uncontrollable conditions. The ambient temperature could influence on the reaction rate and the unremoved moisture after demister in the gas could affect measurement of CO_2 concentration. To reduce the influence of these disturbances or uncontrollable conditions, all experiments were conducted for about 2 h and results were used as averaged values. The results during the initial 30 min, when the reactor system is not yet in the steady state, were excluded.

4. Results and Discussion

The default operating results were found before testing was carried out to determine the optimal operation parameters. In default operation, the gas flow rate is 5 L/min, and the mixed gas was injected only through reactor 1. The mother liquid was not returned to the reactor system. The heights of the packing towers were 700 mm, 500 mm and 500 mm, respectively. The liquid level of the bubble column part at the bottom of each reactor was 15 cm.

Fig. 3 shows the default operating results. The respective average pH levels of the three reactors were 9.0, 10.2, and 12.5. The total CO_2 absorption ratio (γ_3) was about 99%. Generally, in post-combustion carbon capture, 90% of capture rate is the required absorption rate.

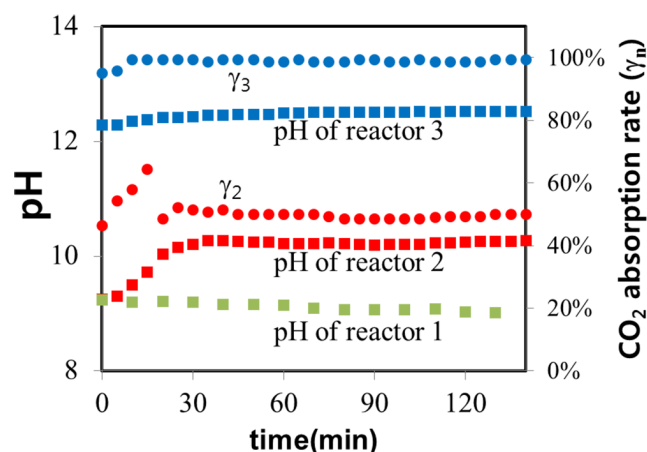


Fig. 3. Experimental results obtained with the base operation conditions.

The CO₂ absorption rate (γ_3) of the default operation was more than sufficient. We could also confirm that operation was very stable after the initial 20 min. The purity of the sodium bicarbonate powder produced in this operation was measured by XRD quantitative analysis, and it was 97.3%. The purity of the sodium bicarbonate chemical (Sigma-Aldrich) was also measured for comparison and it was 97.8%. The purity of the product was comparable to that of the commercial chemical for laboratory experiments.

4.1. Height of Packed Column in Each Reactor

The height of packed column was varied to find the optimal column height. The heights of the column modules we used in this study were 300 mm, 400 mm and 500 mm; the column height could be varied in the combinations described in Table 2.

The pH of reactor 1 was critical to the high purity of the product. In evaluating the performance of reactors 1 and 2, the pH of reactor 1 could be an important indicator. Figure 4 shows the pH of reactor 1 in relation to the height of the column in reactor 1 or reactor 2. As shown in Fig. 1, the pH of reactor 1 should be 8-9. If the height of the column in reactor 1 was shorter than 700 mm, the pH of reactor 1 was higher than 9. However, the pH of reactor 1 did not go below 9, although the height of the column in reactor 1 higher than 700 mm. Therefore, it can be concluded that the proper heights for the columns in reactors 1 is 700 mm, and, for the same reason, the proper heights for the columns in reactors 2 is 400 mm.

The height of the column in reactor 3 had almost no effect on the pH of reactor 1. The primary role of reactor 3 was absorbing CO₂ under a high pH condition. More than 60% of CO₂ was

Table 2. Height of Column in Each Reactor

	Reactor 1	Reactor 2	Reactor 3
Case 1	500 mm	400 mm	400 mm
Case 2	1000 mm	400 mm	400 mm
Case 3	700 mm	300 mm	300 mm
Case 4	700 mm	400 mm	300 mm
Case 5	700 mm	500 mm	300 mm
Case 6	500 mm	400 mm	700 mm
Case 7	700 mm	400 mm	500 mm

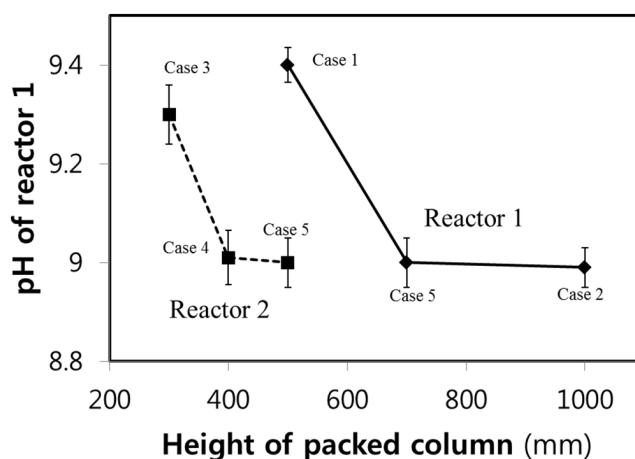


Fig. 4. Height of packed column in reactor 1 or reactor 2 vs. pH of reactor 1.

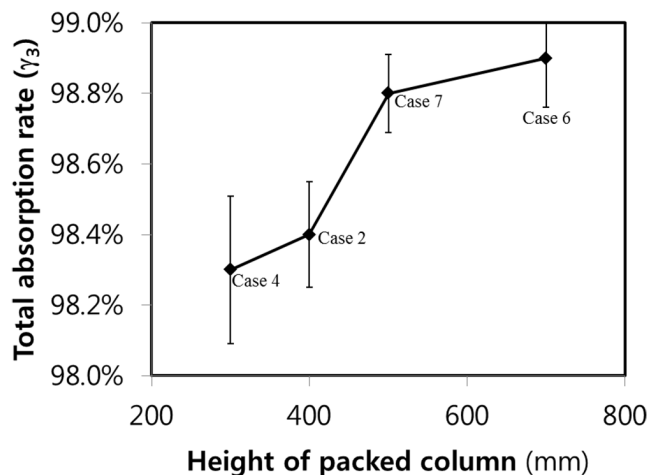


Fig. 5. Height of packed column in reactor 3 vs. total carbon absorption rate.

absorbed in reactor 3. Therefore, the total absorption rate (γ_3) could be a good indicator for evaluation of the performance of reactor 3. Fig. 5 shows the total absorption rate according to the height of the column in reactor 3. Even though the shortest column (300 mm) was used, the absorption rate was high enough. The total absorption rate of CO₂ was more than 97% regardless of the height of the column in reactor 3. The reaction rate of reaction 1 (carbonation reaction, Eq. (1)) was fast, and the bubble column part at the bottom of the reactor could absorb a large amount of CO₂.

4.2. Gas Flow Rate and Gas Injection Point

The gas flow rate was increased to determine the capacity of the reactor system. The gas injection point in the experiment was the bottom of reactor 1, as shown in Fig. 2. The gas flow rate was increased from 5 L/min to 10 L/min, in the other words, from 2 kgCO₂/d to 10 kgCO₂/d. When the flow rate was increased to 4 kgCO₂/d or higher, the reactor could not withstand the pressure and the experiment could not be conducted due to leakage. The

results are shown in Fig. 6. If the gas flow rate was increased, the total absorption rate (γ_3) decreased, and the operation of the reactor system was not stable. In particular, for the case of 10 L/min (4 kgCO₂/d) stabilization of the reactor system took longer than in other cases. In this case, the pH of reactor 3 needed to be maintained above 13 to maintain the absorption rate. Because the high pH solution was transported to reactor 2, the pH of reactor 2 was also high, and stabilization took a long time.

By injecting additional gas into reactor 2, it was considered more utilization of the absorption capacity of the reactor 2. A schematic diagram is shown in Fig. 7. We compared the results with the case of one injection point. As shown in Fig. 8, if the total gas flow rate was not changed (5 L/min) and 50% of the gas was injected into reactor 2, 3 was 98.7%, and the absorption rate was the same as the case in which all of the gas was injected into reactor 1. However, if the gas flow rate into reactor 1 was 5 L/min and the additional gas was injected into reactor 2, 3 was lower than in the case in which all of the gas was injected into reactor 1. The gas injected into reactor 2 did not pass reactor 1;

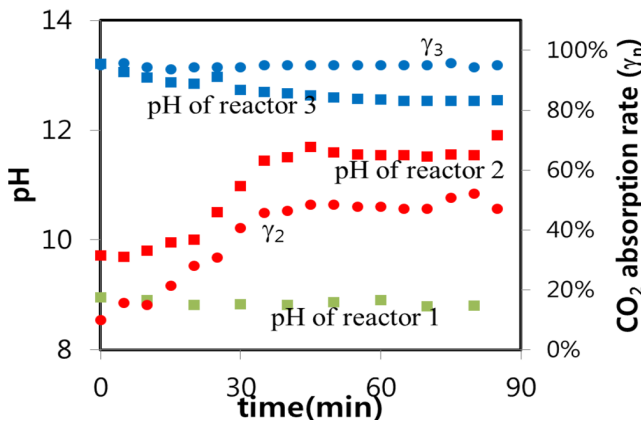


Fig. 6. Experimental results of double gas flow rate (10 L/min) condition. The gas was injected only into reactor 1.

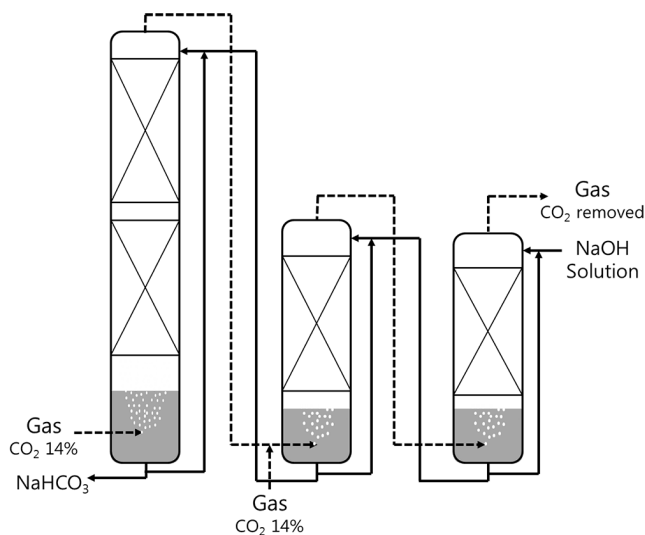


Fig. 7. Schematic diagram of additional gas injection into reactor 2.

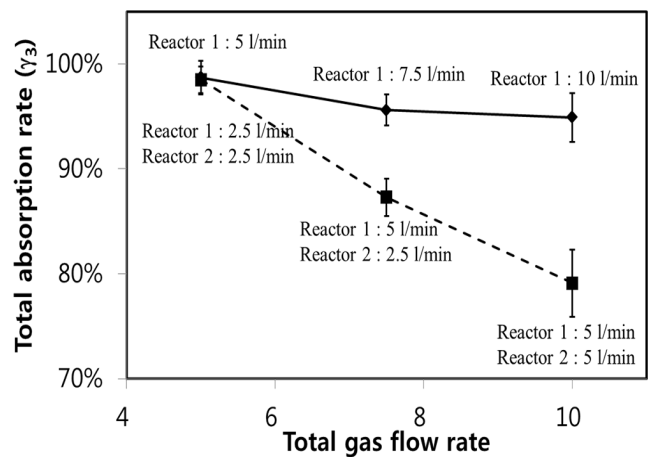


Fig. 8. Total gas flow rate vs. absorption rate. If additional gas was injected into reactor 2, not into reactor 1, the absorption rate decreased.

therefore, less CO₂ was absorbed in comparison with the case in which all of the gas was injected into reactor 1. Additional absorption of CO₂ in reactor 2 was expected but could not be observed.

4.3. Return of the Mother Liquid

In the precipitation tank, sodium bicarbonate was separated from the mother liquid because the solubility of sodium bicarbonate is much lower than that of sodium carbonate. The mother liquid separated from the precipitate is a solution of sodium carbonate and sodium bicarbonate. For a commercial plant, because the yield is important from an economic perspective, recovery of the mother liquid is essential. The operation state of the reactor system was examined when the mother liquid was injected. It was also examined to determine which reactor would be proper for injection of the mother liquid.

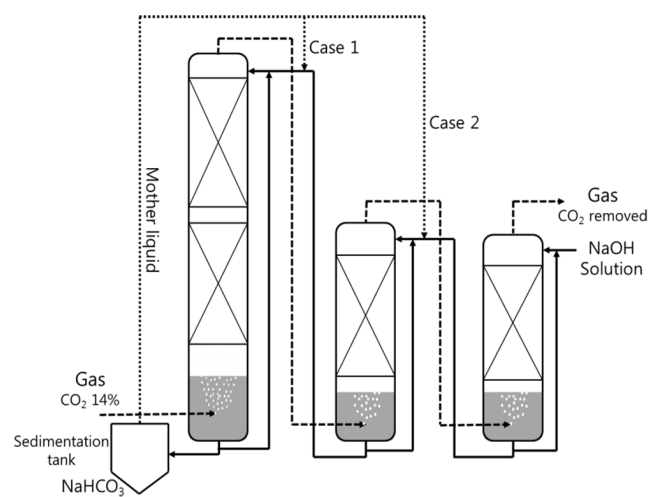


Fig. 9. Schematic diagram of the reactor system for return of the mother liquid. The dotted line represents the line for transfer of the mother liquid. Case 1 is return of the mother liquid into reactor 1, and case 2 is return of the mother liquid into reactor 2.

The pH of the mother liquid was around 9, and it was equal to the pH of the reactor 1. Injection flow rate of the mother liquid was 30 mL/min. Mother liquid injection into reactor 3 was excluded because the mother liquid could reduce the pH of reactor 3 and the absorption rate could be reduced. The operation results for return of the mother liquid are shown in Fig. 10. Of course, if the mother liquid was returned to reactor 1, the pH in reactor 2 and γ_2 did not change, as shown in Fig. 10(a). The pH of reactor 1 also was not affected because the mother liquid had almost the same pH as reactor 1. The γ_3 value was also less affected than in the case of the mother liquid returning to reactor 2. The operation status was very stable and the average γ_3 was barely affected during the entire operation time. The average γ_3 was 97.2%. The purity of the product in this case was 97.1%. Yield of the reaction in this system was about 80%.

If the mother liquid was returned to reactor 2 (Fig. 10(b)), the pH in reactor 2 and the absorption rate in reactor 2 (γ_2) decreased. However, the operation status was stable, and the average γ_3 was not reduced during the entire period of operation. The average γ_3 was 95.4%. The purity of the product (i.e. sodium bicarbonate) in this case was 95.0%. Comparing the two cases, we can conclude that returning the mother liquid to reactor 1 is better than returning it to reactor 2 in terms of both product purity and CO₂ absorption rate.

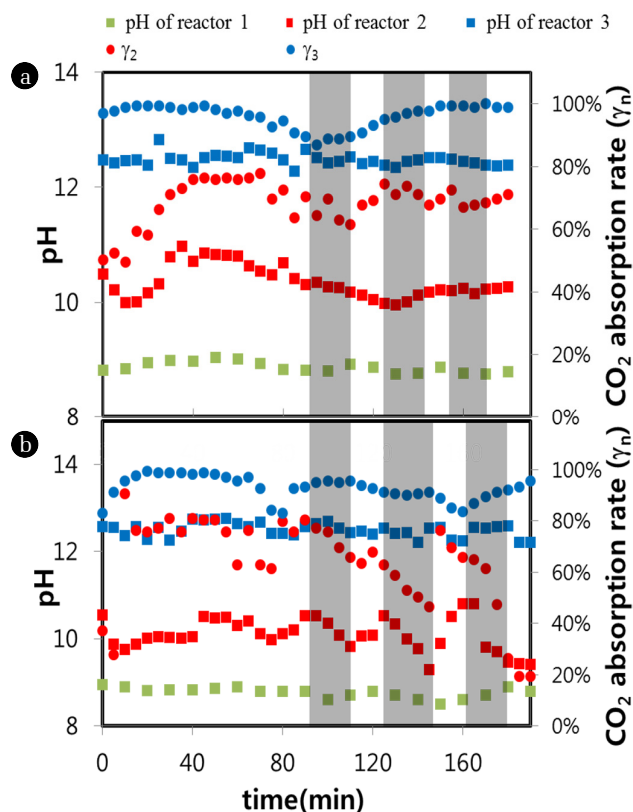


Fig. 10. Experimental results for mother liquid return: (a) mother liquid return to reactor 1 and (b) mother liquid return to reactor 2. The gray shaded area represents the time of mother liquid return.

5. Conclusions

In this study, a carbon capture and utilization method using sodium hydroxide was investigated experimentally. For this experiment, a unique series reactor system was designed. The height of each reactor, gas flow rate or injection point, and the return of mother liquid were investigated. The results can be summarized as follows.

- For our reactor system, the proper heights of the columns in reactors 1, 2 and 3 are 700 mm, 400 mm, and 300 mm, respectively.
- The gas flow rate can be increased to 4 kgCO₂/d (twice of designed capacitor), and gas injection into reactor 1 is better than gas injection into reactor 2.
- Through return of the mother liquid, the reaction yield was confirmed to be about 80%, and the best injection point of the mother liquid was determined to be reactor 1.

Even though these results were obtained on a bench-scale reactor system, the results contain useful information for the construction and operation of a commercial-scale plant. Through this study, this process was confirmed to be a feasible option for the reduction of greenhouse gas.

Acknowledgments

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