

LABORATORY TESTS ON THE EFFICIENCY OF CARBON DIOXIDE CAPTURE FROM GASES IN NaOH SOLUTIONS

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ABSTRACT

The lab-scale experiments were carried out in order to determine the effectiveness of CO₂ capture from gases in the sodium hydroxide solutions under approximately standard conditions. The flow rate of the carrier gas was 140 l/min, and the CO₂ content was 15%. The absorber was Dreschel washer with NaOH solution. The efficiency of CO₂ capture of 85% was obtained for 50% of NaOH. It was proved that the increase of temperature in NaOH solution improves CO₂ capture efficiency. The efficiency of capturing CO₂ in 1 mol/l sodium carbonate was defined as 4–5%. The products of CO₂ reactions in sorption solutions were marked.

Keywords: carbon dioxide, sodium hydroxide, capture efficiency.

INTRODUCTION

Electric energy consumption per capita in the world is increasing. The satisfaction of increasing demand for energy requires higher consumption of fossil fuels, whose combustion emits CO₂, thus stimulates the development of greenhouse effect. Numerous and diversified undertakings aimed at hindering the increase of CO₂ emission are made. They include the use of renewable sources of energy, energy saving and the increase of energy production efficiency as well as CO₂ capture and sequestration.

Numerous technologies of CO₂ capturing from flue gases are being developed (e.g. absorption, adsorption and cryogenic methods), however, it is considered that in professional carbon power industry absorption methods of gas cleaning will be used [1], one example being a well developed method of capturing CO₂ from natural gas with monoethanolamines (MEA) [2]. Due to the costs of CO₂ capture and sequestration in this method (approx. 15–35% of energy produced in a power plant [1]) other, more economic methods are being developed.

Apart from logistical problems, carbon dioxide storage as liquid or gas encounters the resistance from local communities in the sites of potential stockpiles. Ideal solution of CO₂ sequestration is a form of solid compounds, which would not pose danger to natural environment. For this purpose scientists conduct numerous works using natural processes of rock carbonisation, resulting in producing carbonates or using existing methods of removing CO₂ in other industries than energy production [3, 4].

Capture of carbon dioxide from flue gases with sodium hydroxide (NaOH) is interesting from commercial point of view because the obtained products, sodium carbonate and sodium bicarbonate, are produced in hundreds million tons per year. On the other hand, sodium hydroxide is a waste product in some chemical technologies (e.g. in chlorine production). Consequently, its use might lower the costs of CO₂ sequestration from flue gases [3].

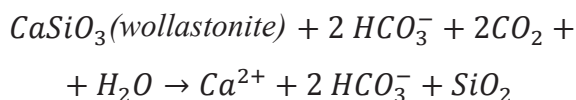
The capturing of carbon dioxide from flue gases with sodium hydroxide (NaOH) has already been a subject of a few publications, however, the results they presented did not provide



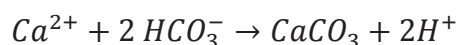
conclusive answers concerning the productivity of the method [1, 5, 6]. The present paper presents the results of the efficiency of capturing carbon dioxide with water solution of sodium hydroxide on a laboratory scale. The tests were used to evaluate the feasibility of utilising sodium hydroxide to capture CO₂ in a form of sodium carbonate with the recapturing CO₂ by producing sodium bicarbonate.

SELECTED MECHANISMS OF CAPTURING CO₂ IN THE PRODUCTION OF CARBONATES

The use of CO₂ capturing methods utilising the compound in the solid state are promising. The basis of these methods is mineral carbonisation, which is a spontaneous process in the nature. The process products include mainly carbonates, which are the products of reaction between CO₂ and minerals and can be used directly or stored safely for a long period of time. Carbon dioxide is present in the nature in such mineral compounds as serpentine and wollastonite [7]:

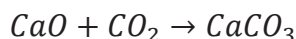


The products of the above reaction generate calcium carbonate, whose waterless forms give calcite and, less frequently, aragonite [7, 8]:

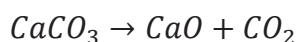


The above processes are slow in natural conditions, however, there have been attempts to accelerate them to the use on industrial scale [7].

One of very well recognised processes of binding CO₂ is the process of calcium oxide carbonisation at the temperature 650–850 °C [9]:



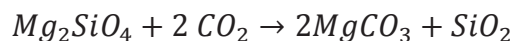
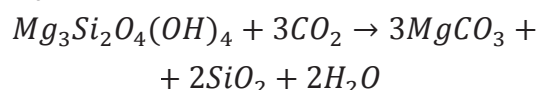
The product is calcium carbonate, which is very common and naturally present in such minerals as calcite and aragonite [8]. It can be stored in a pure form, yet it is frequently calcinated:



Calcination takes place in high temperature (900–1000 °C); as an effect carbon dioxide is released. The gas can be then compressed and transported to the storage site. The produced calcium oxide CaO(s) is returned to the process of CO₂ absorption [9].

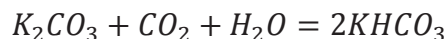
One advantage of using calcium oxide as a sorbent is its high capacity to bind carbon dioxide – 1 kg of CaO can bind as much as 0.786 CO₂ with full conversion [9].

Another promising method of capturing CO₂ is the use of magnesium ore in the process of mineral carbonation by binding carbon dioxide in magnesium carbonate – magnesite (MgCO₃). The minerals used in the process are serpentine (Mg₃Si₂O₄) and olivine (Mg₂SiO₄) [7, 8]:



In the reactor where mineral carbonation processes with magnesium ores are conducted, high pressure (115–150 atm) and temperature (c.a. 185 °C) are required. Apart from magnesite, H₄SiO₂ and SiO₂ are obtained in the process and can be used in construction industry and agriculture [9].

Potassium carbonate is considered a promising alternative to MEA in removing CO₂ from flue gases in power plants, as its solution is less corrosive and the costs of method are lower [4]. The overall reaction of the process is the following:



One disadvantage of this method is a relatively low efficiency of CO₂ absorption in calcium carbonate solutions. It can be improved by adding catalytic additives, usually piperazine.

EXPERIMENTAL METHOD

Preparing absorption solutions

The research covered the efficiency of CO₂ capturing with NaOH solution at different concentrations. For this purpose, solid CZDA form of sodium hydroxide produced by POCH Gliwice was added to demineralised water produced by POCH S.A. (maximum electrical conductivity at 20 °C 5 µS/cm) to obtain appropriate concentration which is understood as:

$$C_{\text{NaOH}} = \frac{m_{\text{NaOH}}}{m_{\text{NaOH}} + m_{\text{H}_2\text{O}}} \cdot 100\% \quad (1)$$

The mass concentrations of NaOH in sorption solutions used in the tests were 5, 10, 20, 30, 40 and 50%.

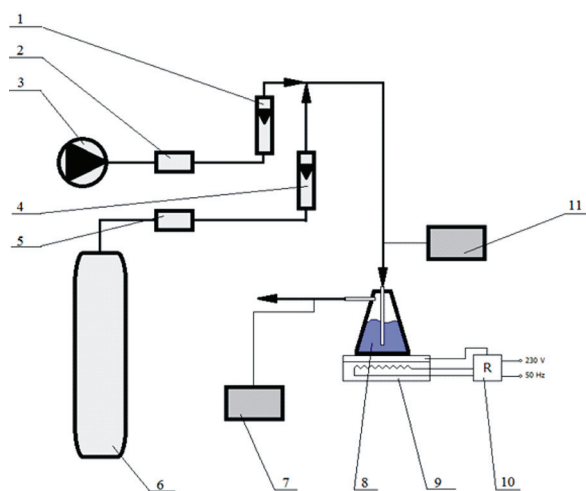


Fig. 1. The scheme of laboratory set-up used for removing CO_2 from gas with NaOH solution: 1 – air rotameter, 2 – air flow regulator, 3 – compressor, 4 – CO_2 rotameter, 5 – CO_2 flow meter, bottle with CO_2 , 7 and 11 – analyser Testo 350-xl, 8 – Dreschel washer, 9 – electric heater, 10 – temperature regulator

Description of test bench

Figure 1 presents the scheme of the test bench. It consists of Dreschel washer (8) with sorption solution, a system of carbon dioxide dosage to the carrier gas flow and gas analyser (7,9) to measure CO_2 contents. Washer (8) contained a solution of sodium hydroxide (NaOH) which was the absorbent. The carrier gas used in the washer (8) was the air from the compressor (3) with addition of carbon dioxide CO_2 . Carbon dioxide was dozed from the steel cylinder of compressed CO_2 (6) so that such volumetric flow in the carrier gas was c.a. 15%.

The flow rates of air and CO_2 volume were regulated with electronic flow regulators (2, 5) by AALBORG, model GFC17. Additionally, air and CO_2 volumetric flows were measured with rotameters (1, 4) produced by Zakłady Automatyki Rotametr Sp. z o. o. (for air) and by Kobold (for carbon dioxide). The flow of carrier gas volume and carbon dioxide was c.a. 140 l/h. In the washer intake CO_2 content was measured in infrared with analyser Testo 350-xl (7, 11). The level of pH in the solution was recorded with pH-meter CP-505 made by Elmetron. The volume of Dreschel washer (8) was 250 ml and the volume of solution inside was 100 ml. The temperature of sorption solution was maintained with an electric heater (9) with temperature controller. electric heater (9) with temperature controller.

Sodium carbonate determination in the solution with NaOH

The determination of sodium carbonate in the solution with sodium hydroxide was made with Warder's method [10]. A series of test measurements were made for different concentrations of Na_2CO_3 in 1 mol/l solution of sodium hydroxide. Specific amounts of crystal sodium carbonate $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ were added to NaOH solution in order to obtain the desired molar concentrations. The analysis was conducted for the following solutions of Na_2CO_3 : 0.10, 0.25, 0.40, 0.50, 0.60 and 0.70 mol/l. 0.2 mol/l solution of titrant was made with 35–38% hydrochloric acid produced by POCH Gliwice and diluted with appropriate amount of demineralised water. 0.2% of phenolphthalein made by Chempur and methyl orange were used as indicators. For each concentration of Na_2CO_3 the measurement was conducted twice. The comparison of titration results of $\text{Na}_2\text{CO}_{3\text{pom}}$ and the reference values of $\text{Na}_2\text{CO}_{3\text{zad}}$ are presented in Table 1. High level of titration results' agreement with the set values was obtained.

Table 1. Uncertainty of measurement in determining sodium carbonate in sodium hydroxide solution

| $\text{Na}_2\text{CO}_{3\text{pom}}$ | | | $\text{Na}_2\text{CO}_{3\text{zad}}$ | Uncertainty | |
|--------------------------------------|------------|---------|--------------------------------------|-------------|------|
| Test no. 1 | Test no. 2 | Average | | | |
| mol/l | mol/l | mol/l | mol/l | mol/l | % |
| 0.07 | 0.09 | 0.08 | 0.1 | 0.0228 | 22.8 |
| 0.22 | 0.22 | 0.22 | 0.25 | 0.0300 | 12.0 |
| 0.47 | 0.5 | 0.49 | 0.5 | 0.0184 | 3.7 |
| 0.54 | 0.52 | 0.53 | 0.6 | 0.0707 | 11.8 |
| 0.78 | 0.65 | 0.72 | 0.7 | 0.0667 | 9.5 |
| 0.36 | 0.35 | 0.36 | 0.4 | 0.0436 | 10.9 |

Methodology of testing CO_2 absorption efficiency in sodium hydroxide solution

CO_2 absorption efficiency in sodium hydroxide solution was tested by running the flow of gas with 15% share of CO_2 , which is equivalent to CO_2 content in furnace flues [13] through the washer 8 (Fig. 1). The content of CO_2 in input ($\text{CO}_{2\text{wlot}}$) and outlet carrier gas ($\text{CO}_{2\text{wyl}}$) was measured. In this way CO_2 capture efficiency was determined from the following formula:

$$\eta_{\text{CO}_2} = \left(1 - \frac{\text{CO}_{2\text{wyl}}}{\text{CO}_{2\text{wlot}}} \right) \cdot 100\% \quad (2)$$

The correctness of determining CO_2 absorption efficiency was tested by determining sodium carbonate (Na_2CO_3) content in NaOH solution according to Warder method and by comparing it with the amount that results from the chemical reaction:



The verification of result correctness was limited to determining sodium carbonate, as due to highly alkaline character of the solution (pH c.a. 13) Na_2CO_3 was the only product of the absorption [11]. The results presented in table 2 prove the correctness of the procedure.

Table 2. The amount of sodium carbonate generated in the process of CO_2 absorption in 1 mol/l solution of NaOH

| Time | Test no. 1 | Test no. 2 | Average | Measurement uncertainty | | CO_2 absorbed in solution |
|------|--|--|---------|-------------------------|-----|------------------------------------|
| | Na_2CO_3 concentration in solution | Na_2CO_3 concentration in solution | | | | |
| min | mol/l | mol/l | mol/l | mol/l | % | mol/l |
| 3 | 0.124 | 0.108 | 0.116 | 0.008 | 0.3 | 0.12 |
| 5 | 0.200 | 0.216 | 0.208 | 0.008 | 0.2 | 0.21 |
| 8 | 0.252 | 0.280 | 0.266 | 0.014 | 0.2 | 0.27 |
| 13 | 0.356 | 0.380 | 0.368 | 0.012 | 0.1 | 0.37 |
| 25 | 0.434 | 0.425 | 0.430 | 0.005 | 1.0 | 0.43 |

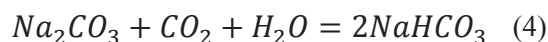
RESULTS OF EXPERIMENT

Reaction of carbon dioxide in NaOH solution

In order to determine the character of carbon dioxide reaction in sodium hydroxide solution the tests of CO_2 absorption in 1M solution of NaOH

were made. The carrier gas with 15% share of CO_2 run through washer (8) with 100 ml of the solution. CO_2 content in the intake and outlet were determined. In this way the amount of absorbed CO_2 was determined and the amount of NaOH was calculated from equation (2) (Fig. 2). It was assumed that as long as NaOH is present in the solution, carbon dioxide reacts with the hydroxide producing sodium carbonate Na_2CO_3 [11].

Figure 2 implies that after 25 minutes of carrier gas flow through the washer, NaOH was depleted and until that moment carbon dioxide reacted with sodium hydroxide generating sodium carbonate. For longer measurements it is evident that carbon dioxide was also bonded in other compound than sodium carbonate. After further input of carbon dioxide to the saturated solution of Na_2CO_3 , bicarbonate was produced according to the reaction:



The influence of NaOH concentration in the efficiency of CO_2 capturing

The tests on efficiency of CO_2 absorption in sodium hydroxide were conducted for NaOH weight concentrations of 5, 10, 20, 30, 40 and 50%. The content of CO_2 was c.a. 15%. The temperature of solution was kept at constant level of 25 °C. The flow rate of gas volume of c.a. 145 l/h was input to the washer with sodium hydroxide solution. The flow time was 5 minutes. The concentration of carbon oxide in the input and outlet were measured. The results are presented in Figure 3.

It was stated that for NaOH concentrations in the range 20–40% a fall of CO_2 capture efficien-

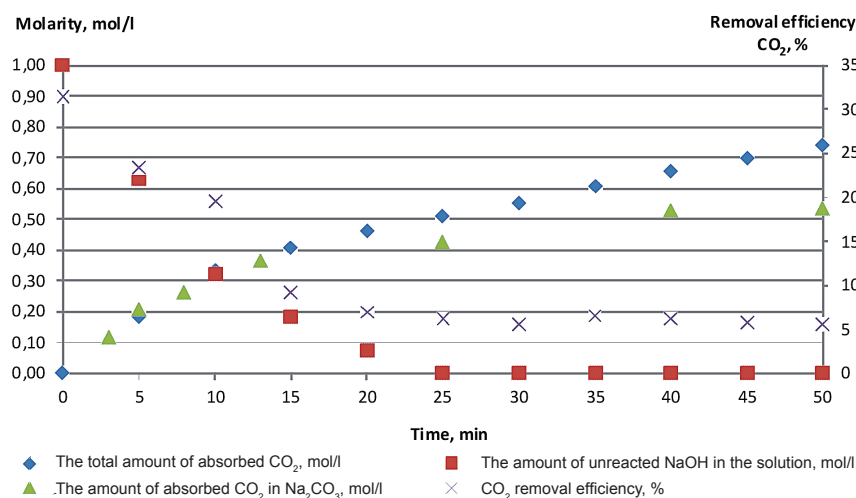


Fig. 2. The amount of absorbed CO_2 and produced sodium carbonate in comparison to the remaining amount of NaOH in the solution

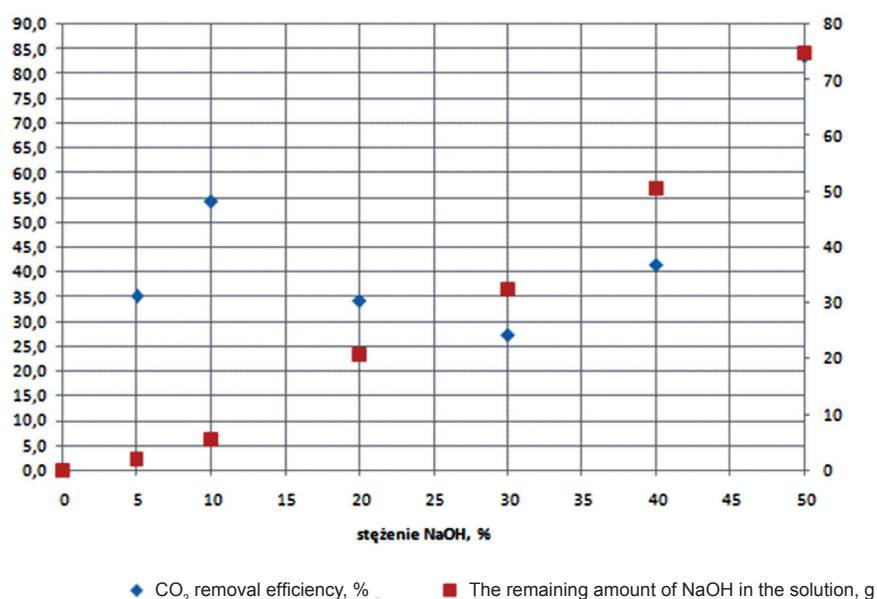


Fig. 3. The relation of CO₂ removing efficiency η_{CO_2} and non-reagent NaOH in the solution in the function of NaOH concentration (flow time = 5 min)

cy was observed. High level of efficiency η_{CO_2} (83,5%) was recorded again for NaOH concentration of 50%. Satisfying effectiveness of CO₂ sequestration, which was 54.3%, was obtained for 10% NaOH solution.

In the conducted experiment unreacted sodium hydroxide was present in the solution, therefore, the only product of the reaction was sodium carbonate (Fig. 3). This resulted from a relatively short time of gas flow through the washer.

The influence of flow time on the efficiency of CO₂ capture

The influence of longer CO₂ absorption time in the solution for NaOH concentrations equal to 5, 10 and 50% on capture efficiency was examined. Total flow time through the washer was 15 min. Other experimental conditions were the same as in previous points. CO₂ capture efficiency changes are presented in Figure 4.

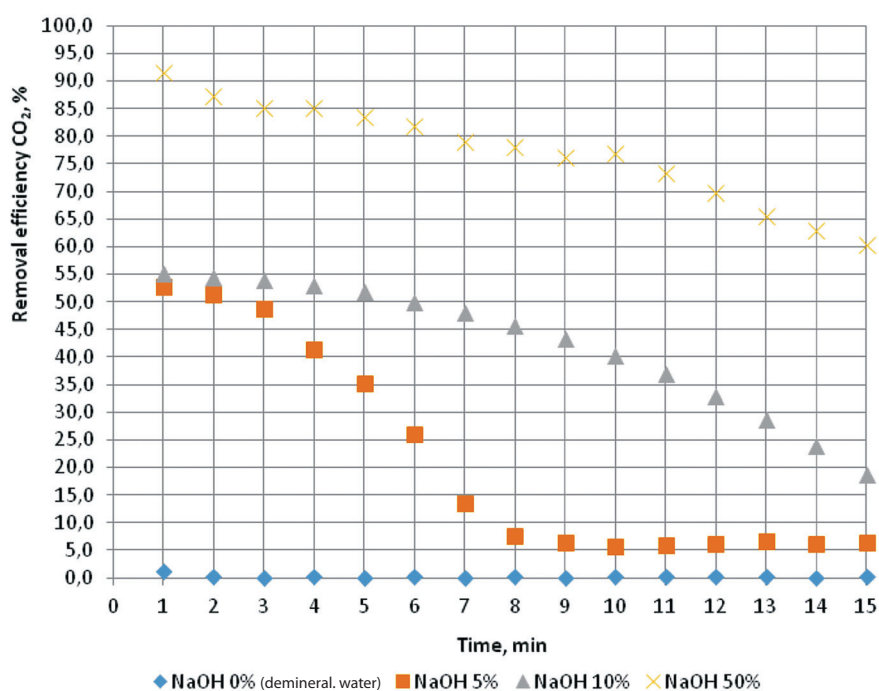


Fig. 4. Efficiency of CO₂ capture from gas in the function of time



In case of using demineralised water the efficiency of removing CO_2 from the carrier gas reach the values approximating zero as soon as after the first minute, what is caused by low CO_2 solubility [10]. For initial concentration of NaOH equal to 5%, after 7 minutes there was an abrupt fall of CO_2 capture efficiency, caused by the exhaustion of NaOH in the solution. For 10% and 50% NaOH concentrations in sorption solution a decrease of CO_2 capture efficiency was observed with time and the exhaustion of NaOH resources. The highest efficiency of CO_2 capture in the washer reaching 90% in the first few minutes was observed for 50% NaOH concentration.

Figure 4 shows that solutions with higher concentrations are more stable than the ones with lower concentrations – in a 15-minute measurement, no abrupt decrease of CO_2 sequestration efficiency was observed. It is related to the presence of unreacted sorbent that remains in the solution and can react with carbon dioxide.

Influence of solution temperature on CO_2 capture efficiency

The influence of temperature on the efficiency of CO_2 absorption was examined on the basis of the solution including 10% of NaOH. The temperature in washer (8) was maintained at a defined level with an electric heater with temperature regulator (Fig. 1). Figure 5 presents the results of CO_2 sequestration efficiency measure-

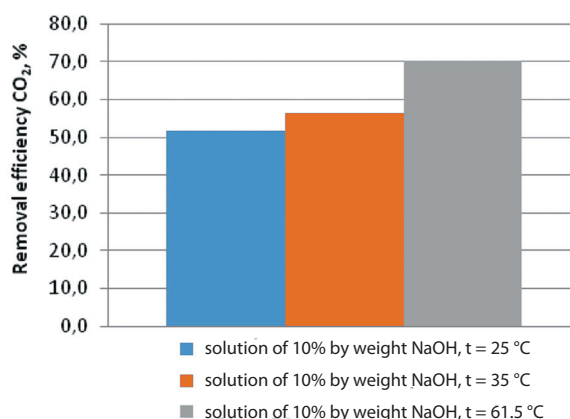


Fig. 5. CO_2 sequestration efficiency in the initial period of absorption

ment in the initial period of absorption (after 5 minutes) for three levels of temperature.

In a longer period of time (15 min), when significant decrease of CO_2 sequestration efficiency was observed, the results for two levels of temperature, 25 °C and 61.5 °C, are presented (Fig. 6).

Increasing the solution temperature intensifies CO_2 absorption, what causes higher efficiency of CO_2 sequestration and accelerates reactions of NaOH in the solution. With time, the differences in CO_2 absorption between the temperatures decreased until after c.a. 13 minutes when the solution at lower temperature reached higher absorption efficiency.

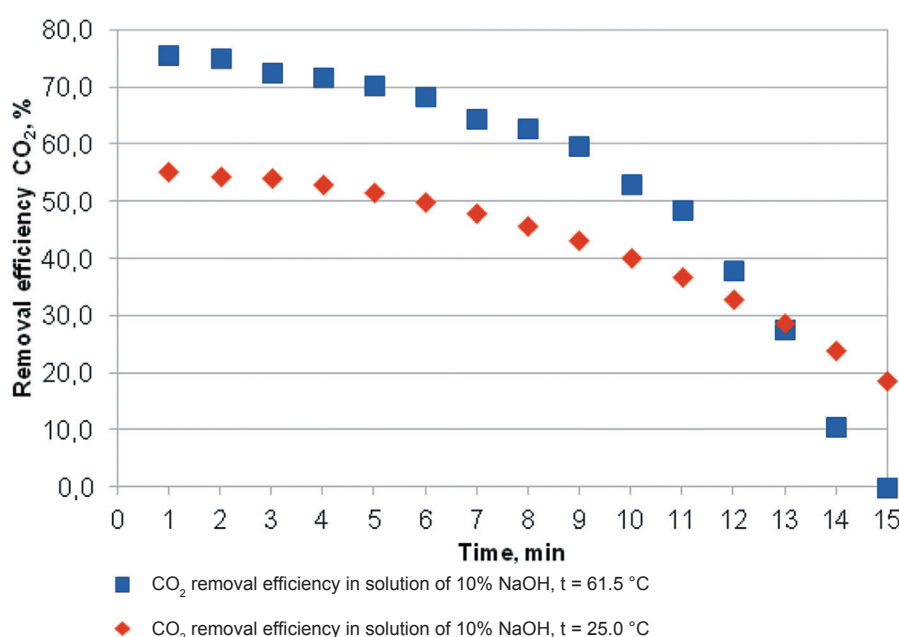


Fig. 6. Efficiency of CO_2 capture from gas in the function of time, depending on NaOH solution temperature

CO₂ sequestration in reaction with sodium carbonate

In point 4.1 it was mentioned that after the exhaustion of NaOH in the solution the sequestration process still takes place with sodium carbonate. CO₂ sequestration in reaction with sodium carbonate is significant because the release of CO₂ from decomposition of the bicarbonate (4) at temperature over 60 °C [8] and the return of sodium carbonate to re-absorption of CO₂. Two 60-minute experiments were conducted. The carrier gas containing 15% of CO₂ flew through the washer with 1 mol/l solution of Na₂CO₃ and then 1 mol/l solution of NaOH, in order to compare the efficiency of carbon oxide sequestration. In both experiments pH of the solution was measured in order to trace individual stages of transformations in the sorption solution. Sorption solution for the

first experiment (1 mol/l of sodium carbonate) was prepared from the crystal sodium carbonate Na₂CO₃ · 10H₂O (CZDA made by POCH Gliwice).

The efficiency of CO₂ sequestration in the reaction with sodium carbonate (4) in the conditions similar to standard conditions turned out to be very low (4–5%), similar to the efficiency observed while removing CO₂ in NaOH solution after its exhaustion (Fig. 2). During the test, pH slowly lowered with the reaction of carbonate to sodium bicarbonate (Fig. 7).

Figure 8 presents changes in the efficiency of CO₂ sequestration from the carrier gas in 1 mol/l NaOH solution and pH of the solution. After c.a. 20 minutes NaOH reacted completely giving Na₂CO₃, what is evident in pH change from 13 to 11. The efficiency of CO₂ sequestration from the gas stabilised at a low level of about 5%, therefore, it was approximately at the same level as in

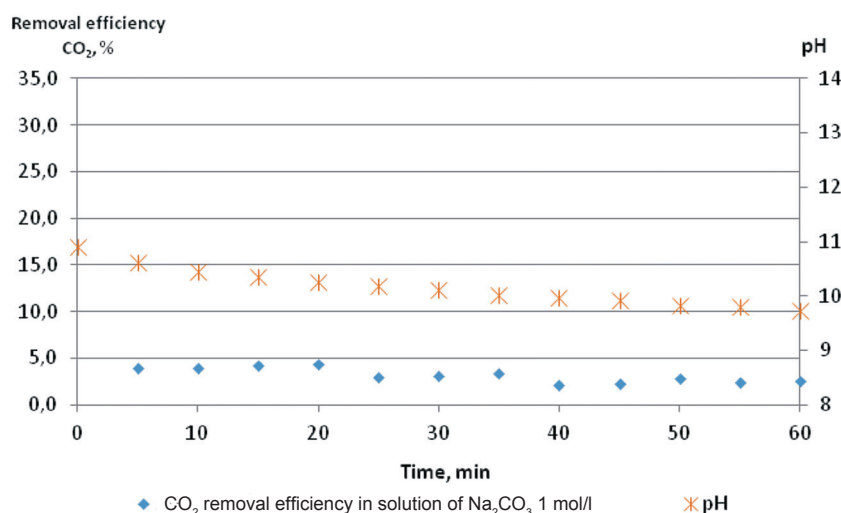


Fig. 7. pH change and the efficiency of CO₂ sequestration during flow through 1 mol/l Na₂CO₃ solution

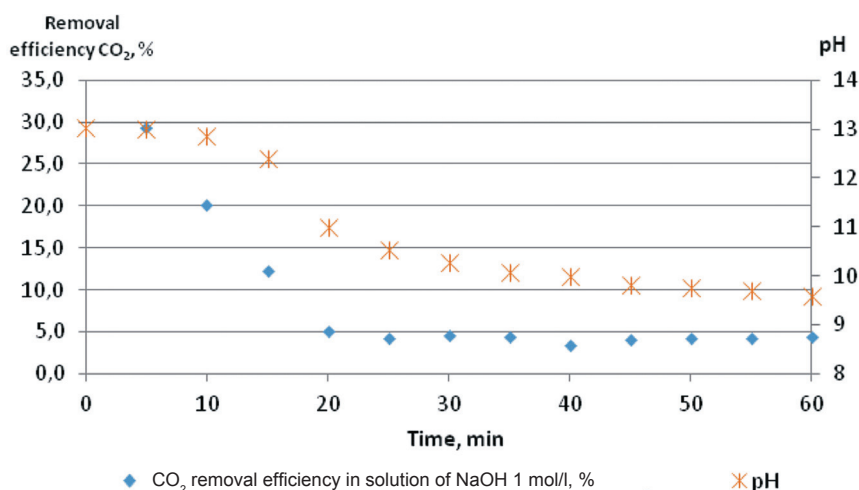


Fig. 8. pH and CO₂ sequestration efficiency change during the flow of 1 mol/l NaOH solution



the previous experiment (Fig. 7). This meant that CO_2 reacted with sodium carbonate and gave the bicarbonate. Then, pH decreased due to the generation of sodium bicarbonate.

Having finished both experiments sodium carbonate and bicarbonate in sorption solution were defined in potentiometric titration method with hydrochloric acid (Table 3).

Table 3. The content of carbonate and acidulous sodium carbonate in the solutions

| Ions | Solution Na_2CO_3 1 mol/l | Initial solution NaOH 1 mol/l |
|--------------------|--|----------------------------------|
| CO_3^{2-} | 0.585 mmol | 0.275 mmol |
| HCO_3^- | 0.345 mmol | 0.305 mmol |

The sum of carbonate ions in the solution after the first experiment was higher than after the other one because according to equation (3) Na_2CO_3 concentration might have been only 0.5 mol/l after complete reaction of NaOH. In both experiments calcium carbonate reacted only in part with CO_2 due to low CO_2 sequestration efficiency. The number of produced bicarbonate moles was similar in both experiments.

The analysis of precipitated residue

From NaOH concentration $\geq 20\%$ precipitation of white residue during measurement was observed. After one of experiments with half of NaOH solution from the washer was taken to be analysed (Fig. 9).



Fig. 9. NaOH solution with the precipitated residue

The precipitated residue underwent a two-stage cooling in order to separate solid and liq-

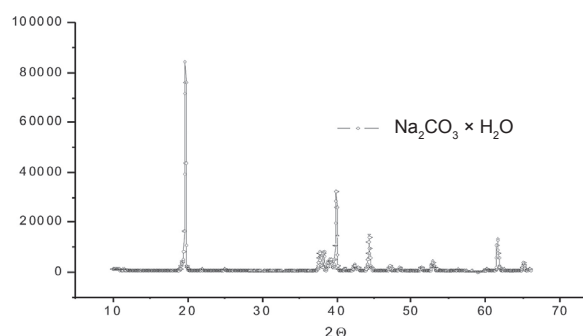


Fig. 10. Diffraction pattern in the precipitated residue after crystallisation

uid fractions. After each cooling the liquid layer was removed. The solution was cooled down to $15\text{ }^{\circ}\text{C}$ in the first stage and down to $0\text{ }^{\circ}\text{C}$ in the second one. Cooling was not continued when no further separation of liquid and solid phases was observed. The obtained residue was then dried in room temperature and underwent XRD (X-Ray Diffraction) analysis, which showed that it contains sodium carbonate ($\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$) (Fig. 10).

SUMMARY

The conducted experiments on the efficiency of carbon dioxide absorption in sodium bicarbonate solution on laboratory scale gave insight into the mechanism of the process and allowed evaluating the feasibility of its use to capture CO_2 from flue gases. The experiments with small amount of NaOH (1 mol/l) showed rapid exhaustion of NaOH and further reaction of CO_2 with sodium carbonate, according to reaction (3). The second stage was realised at low CO_2 capture efficiency (4–5%), what was proved in additional experiment with calcium carbonate.

It was stated that CO_2 capture efficiency depends primarily on NaOH concentration in the solution, which should be at least 50% for practical reasons. During the experiment with high NaOH concentration the falls in the efficiency of CO_2 sequestration from carrier gas were observed, what was caused by NaOH shortages due to reaction of significant amounts of CO_2 . In such case Na_2CO_3 solvency limits are easily breached, what resulted in precipitation of residue. Practically, this requires constant supply of NaOH to the absorber.

The analysis of the precipitated residue in the washer proved that if there is unreacted sodium

hydroxide in the solution, the only product of the reaction is sodium carbonate. If CO_2 absorption continued after the exhaustion of NaOH , carbonate is converted to sodium bicarbonate in reaction (4). However, the efficiency of this process is low (4–5%). Individual stages of this process were accompanied by characteristic pH changes. It was stated that the increase of absorption temperature improves the efficiency of CO_2 sequestration in sodium hydroxide solution.

The feasibility of CO_2 sequestration from gases in NaOH solutions was confirmed, however, the efficiency of the method in the experiment was not competitive in relation to the method using MEA [2]. It seems that the method may function in small CO_2 emitters because in the conditions of coal furnace mass flows of NaOH that are necessary for CO_2 sequestration are comparable to the flows of coal. Initial attempts of CO_2 absorption in Na_2CO_3 solution are not prospective for use with sodium bicarbonate in carbonation-decarbonation to capture, and then release of CO_2 .

CONCLUSION

The results of laboratory tests on the efficiency of CO_2 absorption with the use of NaOH as a sorbent allow formulating the following conclusions:

1. As a result of carrier gas flow including 15% of CO_2 through NaOH solution sodium carbonate Na_2CO_3 is generated until the exhaustion of sodium hydroxide.
2. After the exhaustion of sodium hydroxide CO_2 reacts with sodium carbonate giving sodium bicarbonate.
3. The efficiency of CO_2 capture from the carrier gas depends primarily on sodium hydroxide concentration in the solution: 85% efficiency requires the use of 50% NaOH solution.
4. The efficiency of CO_2 capture from calcium carbonate is ten times lower than in sodium hydroxide.
5. The increase of sorption solution in the range 20–60°C improves the efficiency of CO_2 capture.

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