

Jaruwan Charoenchaipet; Pornpote Piumsomboon; Benjapon Chalermssinsuwan

Article

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Development of CO₂ capture capacity by using impregnation method in base condition for K₂CO₃/Al₂O₃

Jaruwan Charoenchai^a, Pornpote Piumsomboon^{a,b}, Benjapon Chalermnsinsuwan^{a,b,*}

^a Department of Chemical Technology, Faculty of Science, Chulalongkorn University, 254 Phayathai Road, Wangmai,
Pathumwan, Bangkok 10330, Thailand

^b Center of Excellence on Petrochemical and Materials Technology, Chulalongkorn University, 254 Phayathai Road, Wangmai,
Pathumwan, Bangkok 10330, Thailand

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Abstract

Carbon dioxide adsorbent is an important factor in the adsorption process of carbon dioxide. Therefore, this study interested in the adsorption of potassium carbonate solid sorbents on gamma-alumina to adsorb carbon dioxide. The added basic solution was used instead of the de-ionized water by assuming that it would give higher support surface area. The various preparation parameters were explored by impregnation method as follows: the different base types (NaOH, Ca(OH)₂, and Na₂CO₃), the basicity of the solution (8, 10 and 12) and the duration of shaking (12, 18 and 24 h). From the results, all parameters affected on the carbon dioxide capture capacity. The sodium hydroxide gave the highest carbon dioxide capture capacity. An increasing in basicity of the solution resulted in higher carbon dioxide capture capacities, since the reactant was acidic. For the effect of the time spent on shaking the adsorbent, the ability to capture carbon dioxide increased with preparation time. The highest adsorption capacity of 4.34 mmol CO₂ / g sorbent was obtained from potassium carbonate adsorbent, prepared by using sodium hydroxide with a pH value of 12 and a duration of 24 h while, in normal preparation condition, potassium carbonate adsorbent had a capacity of 2.27 mmol CO₂ / g sorbent.

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Keywords: CO₂ capture capacity; Impregnation; Base condition; K₂CO₃/Al₂O₃

1. Introduction

Nowadays, the quantity of greenhouse gases in atmosphere increases, as a result, the global average temperature is raised leading to more serious the global warming problem. The global warming problem is observing from natural disasters such as El Nino, La Nina, drought and flash floods. To decrease the global warming problem, reducing

* Corresponding author at: Department of Chemical Technology, Faculty of Science, Chulalongkorn University, 254 Phayathai Road, Wangmai, Pathumwan, Bangkok 10330, Thailand.

E-mail address: benjapon.c@chula.ac.th (B. Chalermnsinsuwan).

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the amount of carbon dioxide in the atmosphere is interested in the last few years. Carbon dioxide capture process can be classified into 3 types: oxy-combustion, pre-combustion, and post-combustion carbon capture processes. The post-combustion carbon dioxide capture plays as add-on methods gives the more flexibility in maintenance and not necessary to shut the plant down while production. As a result, this process becomes more popular than other carbon dioxide capture processes [1]. Consequently, this study focuses on increasing the capture capacity of post-combustion carbon capture process. The efficiency of the post-combustion carbon capture process depends on type of sorbent. Nowadays, commercial sorbent is liquid sorbent or liquid amine. The liquid amine is widely used because their capture capacity is high even through the absorption rate is low. However, this method requires higher absorber column, causing higher operating cost. In addition, the liquid sorbent that lost from the process, increases the chances of human cancer. Therefore, solid sorbent is received attention as a sorbent for post-combustion process from many researchers because solid sorbent can reduce operating cost and toxicity [2]. The solid sorbent consists of support and active metal material. Alkaline carbonate compounds (e.g. Na_2CO_3 and K_2CO_3) is active metal that commonly used in the process because alkaline carbonate can adsorb and desorb carbon dioxide at low temperature. Moreover, alkaline carbonate is stronger and more stability than other low temperature solid sorbent [3]. Recently, Iruretagoyena et al. [4] compared the capture efficiency of Na, K, Cs in carbonate compounds. The K_2CO_3 was the best active metal because it had suitable basicity and atom size. Guo et al. [2] compared the capture capacity of K_2CO_3 on the different support type which was activated carbon, alumina, titanium, magnesium oxide, zeolites, and potassium feldspar. They found that alumina had the best support because it gave the highest CO_2 capture capacity. In addition to the type of active metal, the preparation method and preparation condition were affected to the physical properties and capture capacity of solid sorbent. Lee and Joen [5] studied the effect of pH-controlled synthesis to physical properties of solid sorbent. From the result, the high basicity preparation was the cause of the high surface area and small active site particle that affected to the high CO_2 capture capacity. Despite, Lee and Joen [5] could improve solid sorbent efficiency. The precipitation method was difficult and took a long time for solid sorbent preparation.

The purpose of this research was to develop the solid sorbent with high CO_2 capture capacity by using impregnation method in base condition for $\text{K}_2\text{CO}_3/\gamma\text{-Al}_2\text{O}_3$. The impregnation method had less complicated and took less time than the other preparation methods. This method was commonly used in many industries process for catalyst and adsorbent preparation. The impregnation in base condition, therefore, would be the new alternative to increases carbon dioxide capture capacity.

2. Experiment

This study used full factorial experimental design for design the solid sorbent preparation. This method gave the minimum number of experiments. But, the results covered the effects of all the main variables. This study interested in 3 variables which were the different base types (NaOH , $\text{Ca}(\text{OH})_2$ and Na_2CO_3), the basicity of the solution (pH = 8, 10 and 12) and the duration of shaking (12, 18 and 24 h).

2.1. Sorbent preparation

The solid sorbent was prepared by impregnation method in base condition. In the first step, pH meter was used to prepared base solution with different pH. Next, the mixture of the potassium carbonate (AR grade, Qrec) 5 g and gamma-alumina (Aluminum oxide 90 active neutral, Merck) 5 g was poured into base solution or Deionization water (DI water for normal condition). After that, the mixture was stirred with difference time (12, 18 and 24 h). Then, this mixture suctioned and washed by DI water to remove base solution from wet solid sorbent. Later, it was dried overnight at 105 °C and calcined for 4 h under atmosphere condition.

2.2. CO_2 adsorption

The CO_2 capture instrument as shown in Fig. 1 was divided into 3 sections. The first section was the gas preparation that consisted of N_2 of 99.99%, CO_2 of 12.00% balanced with N_2 and steam generator tank. The second section was the adsorption glass column and heater. And, the last section was the CO_2 sensor. This system was controlled by LabVIEW commercial program. Also, gas flow rate, temperature and pressure in the CO_2 capture instrument were controlled by accurated computer system.

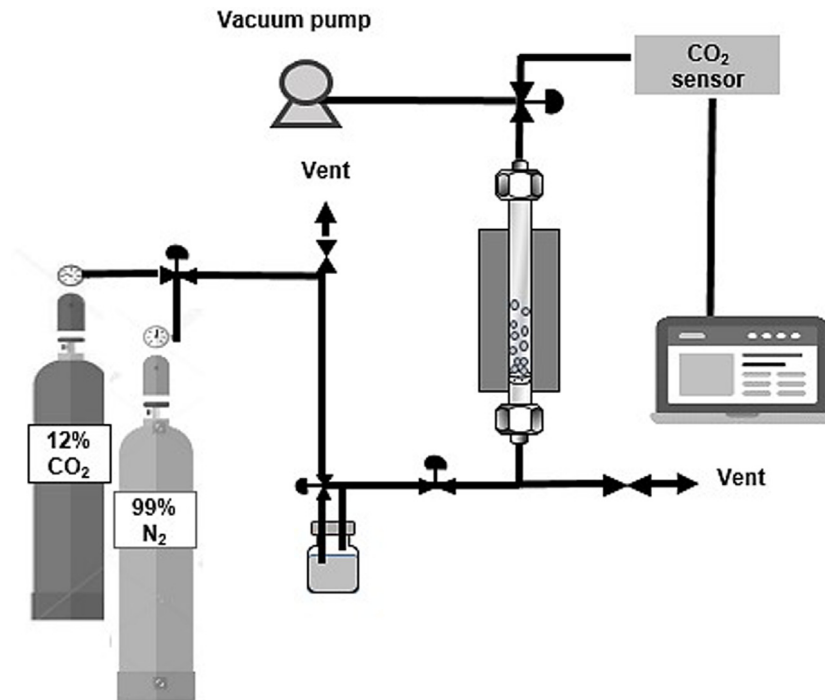


Fig. 1. CO₂ capture instrument.

Before adsorbing CO₂, N₂ was fed to clean pipe network and glass column. Next, solid sorbent was packed in the glass column. The N₂ in the glass column was suctioned out of the column by using the vacuum pump. Then, moist N₂ was fed for preparing moist solid sorbent because the amount of water vapor in the system had an effect on the efficiency of carbon dioxide adsorption as shown in Eq. (1). If the amount of steam was not enough, it would significantly reduce the efficiency of carbon dioxide adsorption.



After that, moist mixed gas was fed to the glass column for testing solid sorbent capture capacity. The CO₂ capture experiment operated under the condition: CO₂ flow rate of 5 L/min, water vapor content of 11 wt%, adsorption temperature of 60 °C and adsorption pressure of 1 atm. Later, the difference CO₂ concentration during the difference time was recorded by CO₂ sensor. The experimental result was used to calculate CO₂ capture capacity as shown in Eq. (2). The symbol nomenclature was similar to the one used in Boonprasop's study [6]. This study focused on the capture CO₂ from coal power plant which released about 10%–15% by volume CO₂. So, the CO₂ concentration in laboratory was the averaged concentration equaled to 12% by volume. For the operation condition, this system operated in turbulent fluidized bed regime because this regime gave the higher capture capacity [6].

$$q = \frac{1}{m} \int_0^t Q (C_{in} - C_{out}) dt \quad (2)$$

2.3. Characterization

Scanning electron microscope (SEM) using JSM-IT300 InTouchScope™ was used to obtain the textural property. This method displayed the distribution and shape of K₂CO₃ on γ-Al₂O₃. TPD Micromeritics AutoChem II 2920 was used to analysis the basicity of solid sorbent surface. The measured basicity was indicated the amount of active metal (K₂CO₃) on support material (Al₂O₃). N₂ adsorption–desorption technique using a Micromeritics ASAP 2020 was used for find the BET surface area, pore size, and pore volume of solid sorbent.

3. Results and discussion

Form the full factorial experimental design, this study interested in 3 preparation parameters: the different base types (NaOH, Ca(OH)₂ and Na₂CO₃), the basicity of the solution (pH = 8, 10 and 12) and the duration of shaking (12, 18 and 24 h). Table 1 shows the CO₂ capture capacity of solid sorbent by using difference preparation conditions. From the result, the solid sorbent prepared in base condition gave the higher CO₂ capture capacity than the ones prepared in DI water. For all type of base solution (NaOH, Ca(OH)₂ and Na₂CO₃), the best condition was the sorbent which prepared at pH of 12 and preparation time of 24 h. Run 9 which used sodium hydroxide (NaOH) gave the highest CO₂ capture capacity. However, the best condition was the highest duration of shaking. The experiment was then extended to find the optimum preparation time for the best solid sorbent. Therefore, this study increased the duration of shaking or preparation time in the operating condition as shown in run 28 and run 29 of Table 1.

Table 1. Preparation parameters and their CO₂ capture capacities.

Run	Base type	pH (-)	Time (h)	Capture capacity (mmol CO ₂ /g sorbent)	Run	Base type	pH (-)	Time (h)	Capture capacity (mmol CO ₂ /g sorbent)
0	DI water	7	24	2.27	15	Ca(OH) ₂	10	24	3.67
1	NaOH	8	12	2.40	16	Ca(OH) ₂	12	12	2.87
2	NaOH	8	18	2.50	17	Ca(OH) ₂	12	18	3.47
3	NaOH	8	24	3.66	18	Ca(OH) ₂	12	24	4.05
4	NaOH	10	12	2.64	19	Na ₂ CO ₃	8	12	2.23
5	NaOH	10	18	2.69	20	Na ₂ CO ₃	8	18	2.83
6	NaOH	10	24	3.97	21	Na ₂ CO ₃	8	24	3.29
7	NaOH	12	12	2.68	22	Na ₂ CO ₃	10	12	2.69
8	NaOH	12	18	3.13	23	Na ₂ CO ₃	10	18	3.30
9	NaOH	12	24	4.34	24	Na ₂ CO ₃	10	24	3.52
10	Ca(OH) ₂	8	12	2.02	25	Na ₂ CO ₃	12	12	3.24
11	Ca(OH) ₂	8	18	2.39	26	Na ₂ CO ₃	12	18	3.49
12	Ca(OH) ₂	8	24	3.24	27	Na ₂ CO ₃	12	24	3.90
13	Ca(OH) ₂	10	12	2.50	28	NaOH	12	30	3.79
14	Ca(OH) ₂	10	18	3.10	29	NaOH	12	36	2.74

3.1. The effect of base type

From the results, the preparation in base condition gave the small size of K₂CO₃ than the preparation in DI water. However, the difference base type provided the difference size and shape of K₂CO₃ as shown in Fig. 2. The best base additive for solid sorbent preparation was sodium hydroxide (NaOH). The comparison between Fig. 2b that showed the K₂CO₃ on γ -Al₂O₃ by using DI water in preparation process and Fig. 2c that showed K₂CO₃ on γ -Al₂O₃ by using sodium hydroxide solution for solid sorbent preparation, provided difference results. The shape of K₂CO₃ in Fig. 2c was found to be rod shape with smaller size than the ones in Fig. 2b. The suitable size of K₂CO₃ brought to the increases in the mass transfer occasion between CO₂ and K₂CO₃. In addition, the rod shape of K₂CO₃ was an appropriate shape for CO₂ capture reaction because this shape reduced the agglomerates of K₂CO₃ on γ -Al₂O₃ in the impregnation process [7]. The BET surface area with different base type was quite similar for all of base types as shown in Table 2. Thus, it could not summarize the obtained trend values.

However, the different base type affected to basicity of solid sorbent. The basicity value from Temperature Program Desorption (TPD) refers to amount of active site or amount of K₂CO₃ on γ -Al₂O₃ as also shown in Table 2. The solid sorbent that prepared in strong base (NaOH and Ca(OH)₂) had higher basicity than preparation the one prepared in weak base (Na₂CO₃). Thus, the amount of K₂CO₃ in strong base condition was higher than the weak base condition. The solid sorbent with strong base preparation then should had higher CO₂ capture capacity.

3.2. The effect of pH

Lee and Joen [5] explained the effect of pH-controlled synthesis to physical properties of sorbent that the increasing of pH brought to small particle size of active metal. The result in this study had the same trend with Lee's

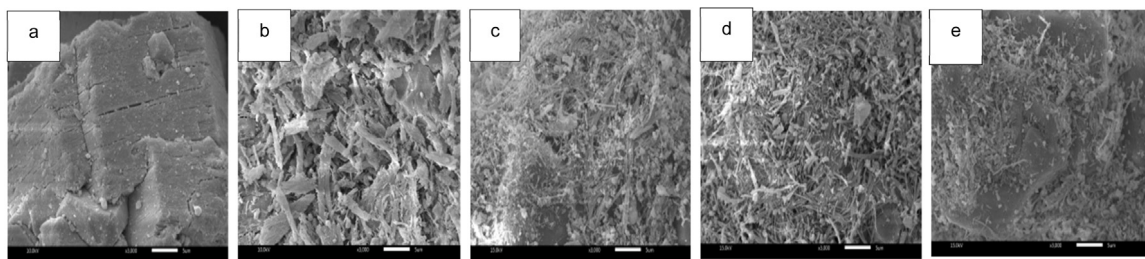


Fig. 2. SEM image (a) γ -Al₂O₃ (b) K₂CO₃/ γ -Al₂O₃ (c) K₂CO₃/ γ -Al₂O₃ (NaOH) (d) K₂CO₃/ γ -Al₂O₃ (Ca(OH)₂) and (e) K₂CO₃/ γ -Al₂O₃ (Na₂CO₃).

Table 2. BET surface area, total pore volume, pore size and basicity of prepared solid sorbent.

Sample name	S_{BET} (m ² /g)	V_{total} (cm ³ /g)	Pore size (Å)	Basicity (mmol/g)
DI-pH7-24h	102.0044	0.8944	78.79	0.253
Ca(OH) ₂ -pH12-24h	118.4901	0.8880	69.96	0.883
Na ₂ CO ₃ -pH12-24h	119.4551	0.8893	69.67	0.862
NaOH-pH8-24h	107.6508	0.8965	77.31	0.628
NaOH-pH10-24h	111.2235	0.8888	67.83	0.872
NaOH-pH12-12h	114.0394	0.8929	77.14	0.409
NaOH-pH12-24h	118.3001	0.8887	71.99	0.935
NaOH-pH12-36h	125.3583	0.8881	68.65	0.415

study [5]. Fig. 3 showed the SEM image with different pH values. The increasing of pH led to the high density of rod shape and small size of K₂CO₃ on γ -Al₂O₃. From Jiao research [8], the high basicity made the active site distribution on support material to be more homogeneous. In addition, the high basicity gave high BET surface area. Zhang et al. [9] synthesized high surface area supports material under base condition. The highest BET surface area of Al₂O₃ was found by preparing the support with pH equalled to 10. Therefore, the solid sorbent which prepared in high pH had high efficiency for capture CO₂. The obtained results in this study had similarly trend with the literature experimental result. The preparation with high pH or basicity had high CO₂ capture capacity.

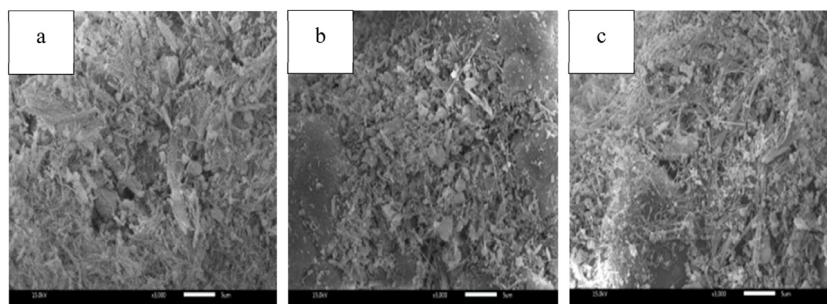


Fig. 3. SEM image (a) K₂CO₃/ γ -Al₂O₃ (pH = 8) (b) K₂CO₃/ γ -Al₂O₃ (pH = 10) and (c) K₂CO₃/ γ -Al₂O₃ (pH = 12).

3.3. The effect of shaking time

From Table 1, the increasing of shaking time increased the opportunity for active site (K₂CO₃) to contact to support (γ -Al₂O₃). Therefore, the longest shaking time (24 h) gave the best solid sorbent for each base type. The best condition for preparing solid sorbent in this study was run 9 (NaOH-pH12-24h). Because the best preparation condition was the highest duration of shaking, so this study performed more experiments to find the optimum duration of shaking. From runs 28 and 29 in Table 1, the increasing of duration time caused the capture capacity to decrease. Thus, the optimum shaking time was 24 h. Fig. 4 illustrated the comparison of SEM picture with difference shaking times. Fig. 4a shows the bulk shape and the aggregation of K₂CO₃. Both of these were not suitable for CO₂

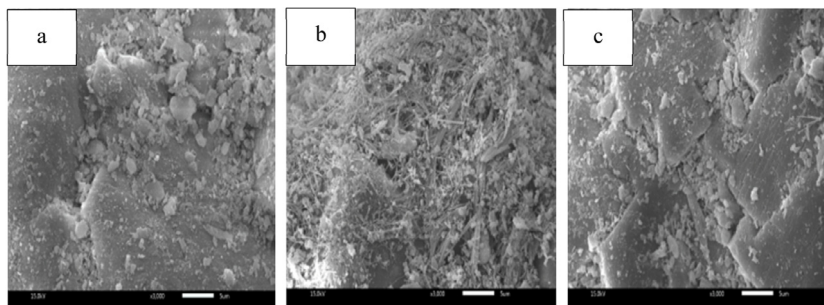


Fig. 4. SEM image (a) $\text{K}_2\text{CO}_3/\gamma\text{-Al}_2\text{O}_3$ (12 h) (b) $\text{K}_2\text{CO}_3/\gamma\text{-Al}_2\text{O}_3$ (24 h) and (c) $\text{K}_2\text{CO}_3/\gamma\text{-Al}_2\text{O}_3$ (36 h).

capture. When increasing the shaking time, SEM image appeared to be the rod shape and to be the homogeneous distribution of K_2CO_3 as shown in Fig. 4b. For the highest shaking time, the rod shape disappeared as shown in Fig. 4c. The capture capacity of the highest shaking time then decreased although this sorbent had highest BET surface area. Also, this sorbent had low basicity. The main factor that affected to CO_2 capture capacity was then structure and basicity of solid sorbent.

Table 3 shows the comparison capture capacity of K_2CO_3 with difference type of support materials. The $\gamma\text{-Al}_2\text{O}_3$ was the best support material. In addition, Table 3 clearly displays that this study was successful in increasing the carbon dioxide capture performance of $\text{K}_2\text{CO}_3/\gamma\text{-Al}_2\text{O}_3$ solid sorbent by using the impregnation method in base condition with high carbon dioxide concentration, high water vapor and low adsorption temperature.

Table 3. Capture capacity of K_2CO_3 with difference support materials from this study and literature studies.

Sorbent	Capture condition	Capture capacity (mmol CO_2/g sorbent)	References
$\text{K}_2\text{CO}_3/\text{PF}$	5% CO_2 , 10% H_2O , 60 °C	1.74	[2]
$\text{K}_2\text{CO}_3/\text{Al}_2\text{O}_3$	10% CO_2 , 14% H_2O , 60 °C	2.43	[10]
$\text{K}_2\text{CO}_3/\text{Ti}$	1% CO_2 , 1% H_2O , 60 °C	1.28	[11]
$\text{K}_2\text{CO}_3/\text{AC}$	1% CO_2 , 9% H_2O , 60 °C	2.09	[12]
$\text{K}_2\text{CO}_3/\text{MgO}$	1% CO_2 , 1% H_2O , 60 °C	2.84	[13]
$\text{K}_2\text{CO}_3/\text{Z}$	1% CO_2 , 9% H_2O , 20 °C	0.35–0.52	[14]
$\text{K}_2\text{CO}_3/\gamma\text{-Al}_2\text{O}_3$	12% CO_2 , 11% H_2O , 60 °C	4.34	This study

AC: Activated carbon, Ti: Titanium, PF: Potassium feldspar, Z: Zeolites.

4. Conclusion

This study tried to increase conventional CO_2 capture capacity of solid sorbent. K_2CO_3 and $\gamma\text{-Al}_2\text{O}_3$ was chosen as active metal and support material, respectively. The preparation method was impregnation method in base condition because it used appropriate time and it was widely used in catalyst preparation or adsorbent synthesis in the industrial. This study focused on three preparation parameters which were the different base types (NaOH, $\text{Ca}(\text{OH})_2$, and Na_2CO_3), the basicity of the solution ($\text{pH} = 8, 10$ and 12) and the duration of shaking (12, 18 and 24 h). From the result, all of base types gave similarly high BET surface area. But, the strong base gave high basicity than the weak base. Also, the increasing of BET surface area brought to the homogeneous distribution of K_2CO_3 on $\gamma\text{-Al}_2\text{O}_3$. The best base solution was sodium hydroxide (NaOH) because it provided the rod shape and small particle size of K_2CO_3 on $\gamma\text{-Al}_2\text{O}_3$. For the effect of difference basicities, CO_2 capture capacity, rod shape density, BET surface area was directly varied with pH. So, the highest pH gave the highest CO_2 capture capacity. The increasing of duration or shaking time affected to the increasing of rod shape until the duration time was more than 24 h in which the rod shape was destroyed. Thus, the K_2CO_3 shape was not suitable for CO_2 capture at that condition. In addition, the increasing of shaking time decreased the amount of K_2CO_3 as can be seen from the obtained basicity value. However, the high shaking time gave the high BET surface area.

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