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### Novel regenerable potassium-based dry sorbents for CO<sub>2</sub> capture at low temperatures

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### ABSTRACT

A novel potassium-based dry sorbent (KZrI) was developed for CO<sub>2</sub> capture at a low temperature range between 50°C and 200°C. The CO<sub>2</sub> absorption and regeneration properties of this novel regenerable potassium-based dry sorbent were measured in a fixed-bed reactor during multiple absorption/regeneration cycles at low temperature conditions (CO<sub>2</sub> absorption at 50-100 °C and regeneration at 130-200 °C). The total CO<sub>2</sub> capture capacity of the KZrI sorbent was maintained during the multiple CO2 absorption/regeneration cycles. The XRD patterns and FTIR analyses of the sorbents after CO2 absorption showed the KHCO<sub>3</sub> phase only except for the ZrO<sub>2</sub> phase used as support. Even after 10 cycles, any other new structures resulting from the by-product during CO<sub>2</sub> absorption were not observed. This phase could be easily converted into the original phase during regeneration, even at a low temperature (130 °C). The KZrI sorbent developed in this study showed excellent characteristics in CO<sub>2</sub> absorption and regeneration in that it satisfies the requirements of a large amount of CO<sub>2</sub> absorption (91.6 mg CO<sub>2</sub>/g sorbent) and the complete regeneration at a low temperature condition (1 atm, 150 °C) without deactivation.

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### 1. Introduction

Carbon dioxide (CO<sub>2</sub>) is a major greenhouse gas that is released into the air due to the use of fossil fuels. As a consequence, CO<sub>2</sub> causes global warming, which may be disastrous to the environment. It can be removed from flue gas and waste gas streams by various methods such as membrane separation, absorption with a solvent, and adsorption using molecular sieves [1-6]. These methods, however, are costly and consume a lot of energy.

One of the improved techniques for the removal of CO<sub>2</sub> is the chemical absorption of CO<sub>2</sub> with dry regenerable sorbents. The use of dry sorbents can be highly cost-effective and an energy efficient way to remove  $CO_2$  [7–24].

Alkali metal carbonates such as Na2CO3 and K2CO3 react with CO<sub>2</sub> and H<sub>2</sub>O and transform to alkali metal hydrogen carbonates after CO<sub>2</sub> absorption by the following reaction:  $M_2CO_3 + CO_2 + H_2O \rightleftharpoons 2MHCO_3$  (M = Na, K) [7–20]. Water vapor is always necessary in forming potassium hydrogen carbonate in all reactions as shown in the absorption mechanism. Alkali metal-based sorbents are employed in CO<sub>2</sub> absorption at low temperatures (50-70 °C) with thermal regeneration easily occurring at low temperatures below 200 °C. Hirano et al. have proposed a modified chemical-absorption method capable of cyclic fixed-bed operations for the recovery of carbon dioxide from flue gases over  $K_2CO_3$ -on-carbon [8]. The sorption of  $CO_2$  on the  $K_2CO_3$ -Al<sub>2</sub>O<sub>3</sub> composite sorbent, in the presence of water vapor, was studied by in-situ IR spectroscopy and X-ray diffraction analysis [11]. Several studies regarding an efficient chemical absorption over K<sub>2</sub>CO<sub>3</sub>, which was supported either on activated carbon [7-9,17,18] or on other porous matrices such as silica gel. Al<sub>2</sub>O<sub>3</sub> and vermiculite [11–13], were also reported using cyclic fixed-bed operations under moist conditions. Lee et al. reported that the K<sub>2</sub>CO<sub>3</sub>/TiO<sub>2</sub> sorbent showed excellent CO<sub>2</sub> absorption and regeneration properties [17]. In particular, the formation of the KHCO<sub>3</sub> crystal structure only after CO<sub>2</sub> absorption is reported to be an important factor for regeneration, even at low temperatures, unlike K<sub>2</sub>CO<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> which formed a new alloy structure like  $KAl(CO_3)_2(OH)_2$ . Several additives or supports such as SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, CaO, MgO, zeolites and others have been used in alkali metal-based sorbents to absorb CO2. Nonetheless, these sorbents except for  $K_2CO_3/TiO_2$  and  $K_2CO_3/activated$ carbon have a disadvantage in that there is a decrease of reactivity during multiple absorptions/regenerations at low temperatures

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#### Table 1

#### Experimental conditions of potassium-based dry sorbents

	CO <sub>2</sub> absorption	Regeneratior
Temperature (°C)	50-100	130-200
Pressure (atm)	1	1
Flow rate (ml/min)	40	40
Gas composition (vol.%)	CO <sub>2</sub> : 1, H <sub>2</sub> O: 9, N <sub>2</sub> : balance	$N_2$ : balance

(40–200 °C). An ideal dry sorbent must have a high CO<sub>2</sub> capture capacity, high absorption rate, excellent regeneration property, and high attrition resistance (physical and chemical strength) to remove CO<sub>2</sub> from flue gas in a fluidized/transport-bed reactor or fixed-bed reactor. In particular, the ideal alkali metal-based sorbent must have a complete regeneration property at a low temperature range between 130 °C and 200 °C. To design an excellent regenerable dry sorbent, it is very important to find new additives or supports to be used in the alkali metal-based sorbent.

One of the objectives of this study was to develop a new regenerable sorbent for  $CO_2$  capture at low temperature range between 50 °C and 200 °C. In addition, the change in the physical property of the sorbent before/after  $CO_2$  absorption was investigated with the aid of Power X-ray diffraction (XRD; Philips, X'PERT), Fourier transform infrared (FTIR; Mattson Instruments Inc.) and TPD.

#### 2. Experimental

The alkali metal-based sorbent used in this study was prepared by the impregnation method. A typical preparation procedure for the sorbent supported on the zirconium oxide (ZrO<sub>2</sub>, Aldrich 99%) or Al<sub>2</sub>O<sub>3</sub> (Aldrich 99%) was as follows: 5.0 g of supports were added to an aqueous solution containing 2.5 g of anhydrous alkali metal carbonate (K<sub>2</sub>CO<sub>3</sub>, Aldrich) in 25 ml of de-ionized water. Then, the content was mixed with a magnetic stirrer for 24 h at room temperature [7,8,17]. After stirring, the mixture was dried in a rotary evaporator at 60 °C. The dried samples were calcined in a furnace with N<sub>2</sub> flow (100 ml/min) for 4 h at 300 °C. The ramping rate of the temperature was maintained at 3 °C/min. We denoted the sorbent as KZrI30 or KAlI30, where K represents K<sub>2</sub>CO<sub>3</sub>, I represents the impregnation method, and 30 represents the loading amount of K<sub>2</sub>CO<sub>3</sub>. The amount of alkali metal impregnated was identified by using inductively coupled plasma-atomic emission spectrometry (ICP-AES; GBC Scientific, Integra KL). Power X-ray diffraction (XRD; Philips, X'PERT) at the Korea Basic Science Institute (Daegu) was also measured in order to confirm the structure using Cu K $\alpha$ radiation.

100 80 Fotal CO<sub>2</sub> capture capacity (mg CO<sub>2</sub>/g sorbent) 60 40 KZrI30 20 KAII30 0 1 2 3 4 5 6 7 8 9 10

**Fig. 1.** The total CO<sub>2</sub> capture capacities of the KZrl30 and KAll30 sorbents as a function of cycle number in the presence of 1 vol.% CO<sub>2</sub> and 9 vol.% H<sub>2</sub>O at  $60 \degree$ C.

Cycle number



#### 3. Results and discussion

#### 3.1. CO<sub>2</sub> absorption properties

#### 3.1.1. Comparison of the total CO<sub>2</sub> capture capacity

The CO<sub>2</sub> capture capacity of sorbent was evaluated by the breakthrough curve for  $CO_2$  absorption. In this study, the total  $CO_2$ capture capacity of sorbent was calculated from its breakthrough curve during CO<sub>2</sub> absorption in the presence of 9.0 vol.% H<sub>2</sub>O and 1 vol.% CO<sub>2</sub>. The total CO<sub>2</sub> capture capacity describes the amount of CO<sub>2</sub> absorbed until the output concentration of CO<sub>2</sub> reached 1 vol.%, which is the same value as that of the inlet. Fig. 1 shows the total CO<sub>2</sub> capture capacities of the KZrI30 and KAII30 sorbents as a function of cycle number. When both CO<sub>2</sub> absorption and regeneration are considered as a one-cycle process, the horizontal axis indicates the number of cycles repeated and the vertical axis indicates the total amount of CO<sub>2</sub> absorbed per 1 g sorbent. CO<sub>2</sub> absorption and regeneration processes were carried out at 60 °C and 200 °C, respectively. As shown in Fig. 1, the total CO<sub>2</sub> capture capacity of the KZrI30 sorbent was about 65.4 mg CO<sub>2</sub>/g sorbent at the first cyclic number. The total CO<sub>2</sub> capture capacity of the sorbent increased up to  $81.3 \text{ mg CO}_2/\text{g}$  sorbent at 2 cycles and was maintained during multiple CO<sub>2</sub> absorption/regeneration cycles. However, the total CO<sub>2</sub> capture capacity of the KAII30 sorbent gradually decreased with the cycle number, regardless of the regeneration temperature of 200 °C. These results indicate that the new KZrI30 sorbent developed in this study shows a relatively high CO<sub>2</sub> capture capacity and is completely regenerated at 200 °C, unlike the KAII30 sorbent.

Fig. 2 shows the breakthrough curves of the KZrI30 (a) and KAII30 (b) sorbents during CO<sub>2</sub> absorption in the presence of 9.0 vol.% H<sub>2</sub>O and 1 vol.% CO<sub>2</sub> at 60 °C. It was observed that the



Fig. 2. The breakthrough curves of the KZrI30 (a) and KAII30 (b) sorbents during CO<sub>2</sub> absorption in the presence of 9.0 vol.% H<sub>2</sub>O and 1 vol.% CO<sub>2</sub> at 60 °C.



Fig. 3. The XRD patterns of the KZrl30 sorbent before (a) /after  $CO_2$  absorption at 1 cycle (b) and 10 cycles (c).

breakthrough times of the KZrI30 (a) for 1st and 2nd cycle were 4 min and 16 min, respectively. After 3rd cycle, the breakthrough was not changed. The both absorption rates of the KZrI30 and KAII30 sorbents estimated from the initial slope of the transition ranges of the breakthrough curves were similar even through the deactivation phenomenon was clearly different, as shown in Fig. 1. Further study is required to enhance the CO<sub>2</sub> absorption rate, but the important point to note is that the new KZrI30 sorbent developed in this study is completely regenerated at 200 °C.

## 3.1.2. Identification of the structure of the KZrI30 sorbent before/after $CO_2$ absorption

Fig. 3 shows the XRD patterns of the KZrI30 sorbent, which was calcined at  $300 \circ C$  under nitrogen, before (a) /after CO<sub>2</sub> absorption at 1 cycle (b) and 10 cycles (c). As shown in the XRD pattern of Fig. 3(a), the fresh KZrI30 sorbent showed a K<sub>2</sub>CO<sub>3</sub> phase (JCPDS No. 71-1466) and a ZrO<sub>2</sub> phase (JCPDS No. 86-1451). As shown in Fig. 3(b) and (c), the XRD patterns of the sorbent after CO<sub>2</sub> absorp-



Fig. 4. The FTIR results of the KZrI30 sorbent at 1 cycle (a) and 10 cycles (b),  $ZrO_2$  (c), and a pure  $KHCO_3$  (d).

tion showed the only KHCO<sub>3</sub> phase except for the ZrO<sub>2</sub> phase used as a support. In particular, it must be noted that even after 10 cycles, no other new structures resulting from the by-product during CO<sub>2</sub> absorption were observed except for KHCO<sub>3</sub> in the XRD results. To precisely identify the formation of the only KHCO<sub>3</sub> phase during CO<sub>2</sub> absorption, the FTIR experiments for the KZrI30 sorbent at 1 cycle (a) and 10 cycles (b), ZrO<sub>2</sub> (c), and a pure KHCO<sub>3</sub> (d), were carried out in the range from 4000 to 400 cm<sup>-1</sup>. These results are shown in Fig. 4. The peak appearing at 3422.96 cm<sup>-1</sup> was assigned to that by  $ZrO_2$  as shown in Fig. 4(c). Table 2 summarizes the characteristic absorption peaks except for the peaks representing ZrO<sub>2</sub> and its corresponding position [25,26]. These results are in agreement with those obtained not only by Abouelhassan et al. and Nakamoto et al., but also by a pure KHCO<sub>3</sub>. The important point to note here is that no other peaks were observed from the FTIR experiments, as well as from the XRD results. From these results, it was confirmed that the only KHCO<sub>3</sub> phase was formed during CO<sub>2</sub> absorption in the presence of 9 vol.% H<sub>2</sub>O and 1 vol.% CO<sub>2</sub>. In our previous paper, we suggested that the formation of the KHCO<sub>3</sub> phase during CO<sub>2</sub> absorption was an important factor regarding the maintenance of the CO<sub>2</sub> capture capacity at low regeneration temperatures below 200 °C [17]. Also, it has previously been shown that decreases in the  $CO_2$  capture capacities of  $K_2CO_3/Al_2O_3$ 

 Table 2

 The characteristic of absorption peaks and the corresponding position

Peak position of our study (cm <sup>-1</sup> )		Expected bond [25]	Peak positions of references	
At 1 cycle	At 10 cycles		Ref. [25]	Ref. [26]
2949	2949	0−H····O + C==0	2949	2920
2627	2627	O-H	2627	2620
1631	1630	C=0	1633	1618
1398	1401	O–H···O	1409	1405
1367	1371	C···O	1368	1367
1007	1007	C−O + C· · · O	1007	1001
981	981	O–H···O	979	988
832	833	CO <sub>3</sub>	832	830
702	702	$C=0+0\cdots H$	703	698
662	663	O <sub>1</sub> CO <sub>2</sub>	662	655



Fig. 5. The loading amount of  $K_2CO_3$  as a function of the amount of  $K_2CO_3$  added in the preparation of sorbent.

and  $K_2CO_3/MgO$  sorbents after regeneration, at temperatures lower than 200 °C, were due to the formation of new structures such as KAl(CO<sub>3</sub>)<sub>2</sub>(OH)<sub>2</sub>,  $K_2Mg(CO_3)_2$  and  $K_2Mg(CO_3)_2$ ·4(H<sub>2</sub>O), except for the KHCO<sub>3</sub> phase. From these results, it was concluded that a complete regeneration during multiple CO<sub>2</sub> absorption/regeneration cycles was due to the formation of the only KHCO<sub>3</sub> without the formation of new structures during CO<sub>2</sub> absorption.



**Fig. 6.** The total CO<sub>2</sub> capture capacity (a) and the amount of the CO<sub>2</sub> absorption per 1 g of  $K_2CO_3$  and (b) of the KZrI sorbent for 1 cycle (I) and 2 cycles (II) as a function of the loading amount of  $K_2CO_3$  in the presence of 1 vol.% CO<sub>2</sub> and 9 vol.% H<sub>2</sub>O at 60 °C.



Fig. 7. The total CO<sub>2</sub> capture capacity of the KZr130 sorbent in the range of temperature between 50 °C and 100 °C in the presence of 9 vol.%  $H_2O$ .

# 3.1.3. Effects of the loading amount of $K_2CO_3$ on the $CO_2$ absorption

The effect of the loading amount of K<sub>2</sub>CO<sub>3</sub> on the total CO<sub>2</sub> capture capacity of sorbent in the presence of 9.0 vol.% H<sub>2</sub>O and 1 vol.% CO<sub>2</sub> at 60 °C was investigated. Fig. 5 shows the loading amount of K<sub>2</sub>CO<sub>3</sub> as investigated by ICP-AES analysis of the fresh sorbents. As shown in Fig. 5, the loading amount of K<sub>2</sub>CO<sub>3</sub> was almost proportional to the amount of K<sub>2</sub>CO<sub>3</sub> added in preparation of the sorbent. Fig. 6(a) shows the amount of the CO<sub>2</sub> capture per 1 g of sorbent for 1 cycle (I) and 2 cycles (II) as a function of the loading amount of K<sub>2</sub>CO<sub>3</sub>. In both cases, the total CO<sub>2</sub> capture capacity of the sorbent increased with the loading amount of K<sub>2</sub>CO<sub>3</sub>. In order to investigate effects of the loadings of K<sub>2</sub>CO<sub>3</sub> on the total CO<sub>2</sub> capture capacity of sorbent in detail, the amount of CO<sub>2</sub> absorption per 1 g of K<sub>2</sub>CO<sub>3</sub> was calculated from Fig. 6(a). These results are shown in Fig. 6(b). The theoretical value of the sorbent, which was calculated from moles of K<sub>2</sub>CO<sub>3</sub> involved in the sorbent, was 318.3 mg  $CO_2/g K_2CO_3$  as shown in Fig. 6(b-III). The  $CO_2$  capture capacities of the sorbents with 30 wt.% and 40 wt.% K<sub>2</sub>CO<sub>3</sub> were higher than those of the other sorbents for 2 cycles as shown in Fig. 6(b-II) and these values were equivalent to about 90 percent of their theoretical value. These results indicated that the KZrI30 and KZrI40 sorbents were suitable for use as the sorbent for CO<sub>2</sub> capture in that the sorbents with 30 wt.% and 40 wt.% K<sub>2</sub>CO<sub>3</sub> showed the high CO<sub>2</sub> capture capacity comparable to its theoretical value. Another interesting fact observed in Fig. 6 was that the CO<sub>2</sub> capture capaci-



Fig. 8. The TPD results of the KZrI30 sorbent after CO<sub>2</sub> absorption and a pure KHCO<sub>3</sub>.



Fig. 9. The XRD patterns of the sorbents after regeneration at various temperatures such as  $130 \degree C$  (a),  $150 \degree C$  (b), and  $200 \degree C$  (c).

ties of the sorbents for 2 cycles were much higher than those for 1 cycle. The reason for the increase in the  $CO_2$  capture capacity of the sorbent at 2 cycles is not clear, but it is thought to result from the re-dispersion of  $K_2CO_3$  supported on  $ZrO_2$  owing to regeneration at 200 °C.

#### 3.1.4. Effects of the absorption temperature on the CO<sub>2</sub> absorption

Fig. 7 shows the total CO<sub>2</sub> capture capacity of the KZrI30 sorbent at a temperature range between 50 °C and 100 °C in the presence of 9 vol.% H<sub>2</sub>O. The KZrI30 sorbent showed very low CO<sub>2</sub> capture capacity above 80 °C, while at 50 °C the sorbent showed 91.6 mg CO<sub>2</sub>/g sorbent. This value was about 96% of the theoretical value of the sorbent. These results were thought to be due to an increase in relative humidity resulting from a decrease in the absorption temperature.

#### 3.2. Regeneration properties

In addition to the CO<sub>2</sub> capture capacity, regeneration property is one of the most important factors to be considered. To investigate the regeneration property of the KZrI30 sorbent, TPD (temperature programmed desorption) and XRD analyses were performed. Fig. 8 shows the TPD results of the KZrI30 sorbent after CO<sub>2</sub> absorption and a pure KHCO<sub>3</sub>. TPD tests were carried out by measuring the concentration of CO<sub>2</sub> desorbed when the temperature ramping rate was 1 °C/min. In the case of the KZrI30 sorbent, most CO<sub>2</sub> was desorbed in the temperature range between 100 °C and 150 °C. The CO<sub>2</sub> desorption peak of the sorbent was almost consistent with that of a pure KHCO<sub>3</sub> which was at around 130 °C. Lee et al. reported that the CO<sub>2</sub> desorption peaks in the TPD results of the K<sub>2</sub>CO<sub>3</sub>/AC and K<sub>2</sub>CO<sub>3</sub>/TiO<sub>2</sub> sorbents, which formed the only KHCO<sub>3</sub> after CO<sub>2</sub> absorption, were observed at around 130 °C [23]. These results indicated that the desorbed CO<sub>2</sub> of the KZrI30 sorbent at around 130 °C was due to the decomposition of KHCO<sub>3</sub> as the K<sub>2</sub>CO<sub>3</sub>/AC and K<sub>2</sub>CO<sub>3</sub>/TiO<sub>2</sub> sorbents. Also, it was known that it was possible to regenerate the KZrI30 sorbent after CO<sub>2</sub> absorption, even at 130 °C. To identify this regeneration property of the KZrI30 sorbent in detail, the XRD patterns of the sorbents after regeneration at various temperatures, such as at 130 °C, 150 °C, and 200 °C, were observed. These results are shown in Fig. 9. The separated K<sub>2</sub>CO<sub>3</sub> crystal structure was observed in the XRD patterns at all temperatures. These results indicated that the carbonated sorbent after CO<sub>2</sub> absorption was completely regenerated even at 130 °C. This fact agreed well with the TPD result shown in Fig. 8. It was concluded that the formation of the only KHCO<sub>3</sub> crystal structure, through the CO<sub>2</sub> absorption of the KZrI sorbent in the presence of water, plays an important role in complete regeneration at low temperature range between 130 °C and 200 °C. It was also concluded that ZrO<sub>2</sub> could be used as a support and additive to prepare a sorbent having the potential for CO<sub>2</sub> capture and regeneration at low temperatures below 150°C.

#### 4. Conclusions

The potassium-based dry sorbent (KZrI30) developed in this study showed 91.6 mg CO<sub>2</sub>/g sorbent, which was about 96% of the theoretical value of the sorbent, in the presence of 9 vol.% H<sub>2</sub>O at 50 °C. In particular, the total CO<sub>2</sub> capture capacity of the KZrI30 sorbent was maintained during the multiple CO<sub>2</sub> absorption/regeneration cycles at low temperature conditions (CO<sub>2</sub> absorption at 60 °C and regeneration at 200 °C). The KZrI30 sorbent after CO<sub>2</sub> absorption formed the only KHCO<sub>3</sub> phase except for the ZrO<sub>2</sub> phase used as support. Even after 10 cycles, it was confirmed from the XRD, FTIR, and TPD results that no other new structures resulting from the by-product during CO<sub>2</sub> absorption were formed except for KHCO<sub>3</sub>. The carbonated sorbent after CO<sub>2</sub> absorption, could be completely regenerated, even at 130 °C. The formation of the only KHCO<sub>3</sub> crystal structure plays an important role in a complete regeneration at a low temperature range between 130 °C and 200 °C. Considering that the regeneration capacity was an important factor in addition to the CO<sub>2</sub> capture capacity, the new regenerable potassium-based dry sorbent (KZrI) developed in this study could be used as a sorbent having the potential for  $CO_2$ capture at low temperature ranges.

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