

The effect of relative humidity on CO₂ capture capacity of potassium-based sorbents

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Abstract—Potassium-based sorbent was prepared by impregnation with potassium carbonate on activated carbon. The role of water and its effects on pretreatment and CO₂ absorption was investigated in a fixed bed reactor. K₂CO₃ could be easily converted into K₂CO₃·1.5H₂O working as an active species by the absorption of water vapor as the following reaction: $K_2CO_3 + 3/2 H_2O \rightarrow K_2CO_3 \cdot 1.5H_2O$. One mole of K₂CO₃·1.5H₂O absorbed one mole of CO₂ as the following reaction: $K_2CO_3 \cdot 1.5H_2O + CO_2 \rightleftharpoons 2KHCO_3 + 0.5 H_2O$. The K₂CO₃·1.5H₂O phase, however, was easily transformed to the K₂CO₃ phase by thermal desorption even at low temperature under low relative humidity. To enhance CO₂ capture capacity and CO₂ absorption rate, it is very important to maintain the K₂CO₃·1.5H₂O phase worked as an active species, as well as to convert the entire K₂CO₃ to the K₂CO₃·1.5H₂O phase during CO₂ absorption at a temperature range between 50 °C and 70 °C. As a result, the relative humidity plays a very important role in preventing the transformation from K₂CO₃·1.5H₂O to the original phase (K₂CO₃) as well as in producing the K₂CO₃·1.5H₂O from K₂CO₃, during CO₂ absorption between 50 °C and 70 °C.

Key words: CO₂, Absorption, Pretreatment, K₂CO₃, K₂CO₃·1.5H₂O

INTRODUCTION

Carbon dioxide (CO₂) is a major greenhouse gas that is released into the air due to the use of fossil fuels. As a consequence, CO₂ causes global climate warming. 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 [1-5]. These methods, however, are costly and consume a great deal of energy.

One of the improved techniques for the removal of CO₂ is the chemical absorption of CO₂ with solid regenerable sorbents. The use of solid sorbents for CO₂ absorption has been reported previously [6-27]. Alkali metal carbonate was utilized in CO₂ absorption at low temperatures (50-60 °C) with thermal regeneration easily occurring at low temperatures. Calcium oxide was utilized in CO₂ absorption at higher temperatures (less than 860 °C) [15]. Several studies regarding the efficient chemical absorption over K₂CO₃, which was supported either on carbon⁴ or on other porous matrices such as silica gel, Al₂O₃, and vermiculite [17], were also reported using cyclic fixed-bed operations under moist conditions. Water content was essential in forming potassium hydrogencarbonate in all absorption reactions, while moisture contained in the flue gases, as high as 8-17 vol%, negatively affected the adsorption capacity of molecular sieves [8]. It has been reported that K₂CO₃·1.5H₂O is directly related to the CO₂ capture capacity [8,10]. Active species, however, were easily transformed to K₂CO₃ by thermal desorption even at low temperature under low concentration of water vapor due to the heat released from the exothermic reaction of the sorbent during

CO₂ absorption [10]. The new active species (K₂CO₃·1.5H₂O) could also be formed by drying of the K₄H₂(CO₃)₃·1.5H₂O phase which was formed after pre-treatment with excess water [11]. The K₂CO₃-based sorbent, which was prepared by impregnation of activated carbon with K₂CO₃, showed very low CO₂ capture capacity of about 40 mg CO₂/g sorbent in the case of 1.3 vol% H₂O and 1 vol% CO₂, although the ratio of CO₂ to water was 1 : 1.3. This result could not be explained by the fact that one mole of K₂CO₃ could absorb a stoichiometric amount of one mole of CO₂ and one mole of H₂O according to the following reaction: $K_2CO_3 + H_2O + CO_2 \rightleftharpoons 2KHCO_3$ [8-14,17,18]. Even though it is well known that water vapor plays an important role in CO₂ absorption over alkali metal-based sorbents, the role of water vapor in CO₂ absorption has not been clearly defined. In particular, there have been very few reports regarding the effects and roles of water vapor on CO₂ absorption over the alkali metal-based sorbents.

One of the main objectives of this work was to investigate the role of water vapor and its effects on CO₂ absorption. For this purpose, the CO₂ capture capacity of the potassium-based sorbent was studied in the presence of various concentrations of water vapor. In addition, changes in the physical properties of the sorbent before and after the reaction, the identification of a new active species, and a mechanism were investigated by X-ray diffraction and temperature programmed desorption (TPD).

EXPERIMENTAL

1. Preparation of Sorbents

The alkali metal-based sorbent used in this study was prepared by an impregnation method. A typical preparation procedure for the sorbent supported on the activated carbon (KACI30) was as fol-

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lows: Five (5.0) g of activated carbon (AC) was added to an aqueous solution containing 2.5 g of anhydrous alkali metal carbonate (K₂CO₃, Aldrich) in 25 ml of de-ionized water. Then, the content was mixed with a magnetic stirrer for 24 h at room temperature [8,11-13]. After stirring, the mixture was dried in a rotary evaporator at 60 °C. The dried samples were calcined in a furnace with N₂ flow (100 ml/min) for 4 h at 300 °C. The ramping rate of the temperature was maintained at 3 °C/min. The surface area of the sorbent was measured at 77 K using BET (Quantachrome, AUTOSORB-1). The surface area of activated carbon (AC, 1,987 m²/g) was reduced to 471.6 m²/g after calcination at 300 °C. Power X-ray diffraction (XRD; Philips, X'PERT) was also measured in order to confirm the structure using Cu K α radiation.

2. Absorption Apparatus and Procedures

CO₂ absorption and regeneration processes were performed in a fixed-bed quartz reactor with a diameter of 1 cm, which was placed in an electric furnace under atmospheric pressure. One-half (0.5) g of the sorbent was packed into the reactor, and space velocity (SV) was maintained at 3,000 h⁻¹ to minimize severe pressure drops and channeling phenomena. All volumetric gas flows were measured under standard temperature and pressure (STP) conditions. The column used in the analysis was a 1/8 inch stainless tube packed with Porapak Q. The conditions for pre-treatment, CO₂ absorption, regeneration, and water desorption are shown in Table 1. Before CO₂ absorption, moist nitrogen gas with 1.3-11 vol% H₂O (6.5-56% relative humidity) at 60 °C was supplied to sorbents for 120 min using a saturator. When the CO₂ concentration of the outlet gases reached the same level as the inlet gas (1 vol%) during the CO₂ absorption process, nitrogen was introduced to regenerate the spent sorbent. The regeneration process was terminated when the CO₂ concentration reached 200 ppm. Water desorption tests during the CO₂ absorption were carried out under the respective water concentration (0, 1.3, 5.0, 9.0 vol% H₂O) after pre-treatment with 9 vol% H₂O for 2 h. TPD tests of the sorbent were performed under conditions with/without 9 vol% H₂O after pre-treatment with 9 vol% H₂O, when the temperature ramping rate was 1 °C/min. The outlet CO₂ and H₂O gases from the reactor were automatically analyzed every 4 min by a thermal conductivity detector (TCD; Donam Systems Inc.), which was equipped with an auto sampler (Valco Instruments CO. Inc.). Hydrogen gas was used as a carrier gas.

RESULTS AND DISCUSSION

1. A Comparison of CO₂ Capture Capacity of a Sorbent at Various Concentrations of Water Vapor

The CO₂ capture capacity of a sorbent was evaluated by the break-

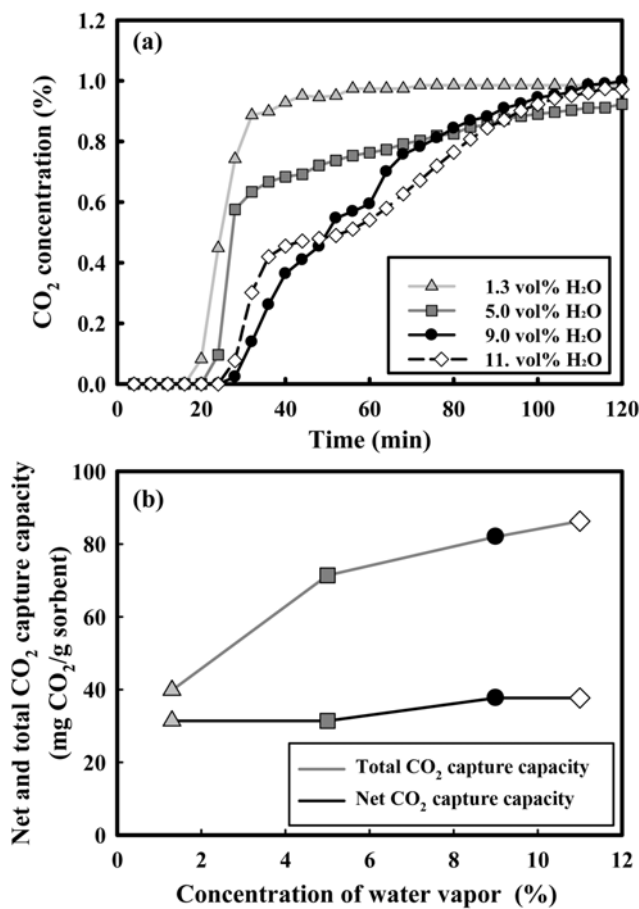


Fig. 1. The breakthrough curves (a) and the CO₂ capture capacity (b) of KACI30 in the presence of various concentrations of water vapor and 1 vol% CO₂ at 60 °C.

through curve for CO₂ absorption. In this study, the net CO₂ capture capacity and the total CO₂ capture capacity were used to evaluate CO₂ capture capacity. Total CO₂ capture capacity describes the amount of CO₂ absorbed until the output concentration of CO₂ reaches 1 vol%, which is the same value as that of the inlet. The net CO₂ capture capacity is defined as the amount of CO₂ absorbed per 1 g of sorbent until the CO₂ concentration is less than 200 ppm. Fig. 1(a) shows the breakthrough curves of a KACI30 sorbent at various concentrations of water vapor in the presence of 1 vol% CO₂ at 60 °C. It was observed that the slope of the breakthrough curve gradually decreased with an increase in the concentration of water vapor. Fig. 1(b) shows the CO₂ capture capacity, which was calculated from breakthrough curves, as a function of concentration of water vapor.

Table 1. Experimental conditions of alkali metal-based sorbent

	Pretreatment	CO ₂ absorption	Regeneration	Water desorption
Temperature (°C)	60	50-100	150	50-100
Pressure (atm)	1	1	1	1
Flow rate (ml/min)	40	40	40	40
Gas composition (vol%)	H ₂ O: 9.0 N ₂ : Balance	CO ₂ : 1.0, H ₂ O: 1.3-11.0 N ₂ : Balance	N ₂	H ₂ O: 0-9.0 N ₂ : Balance

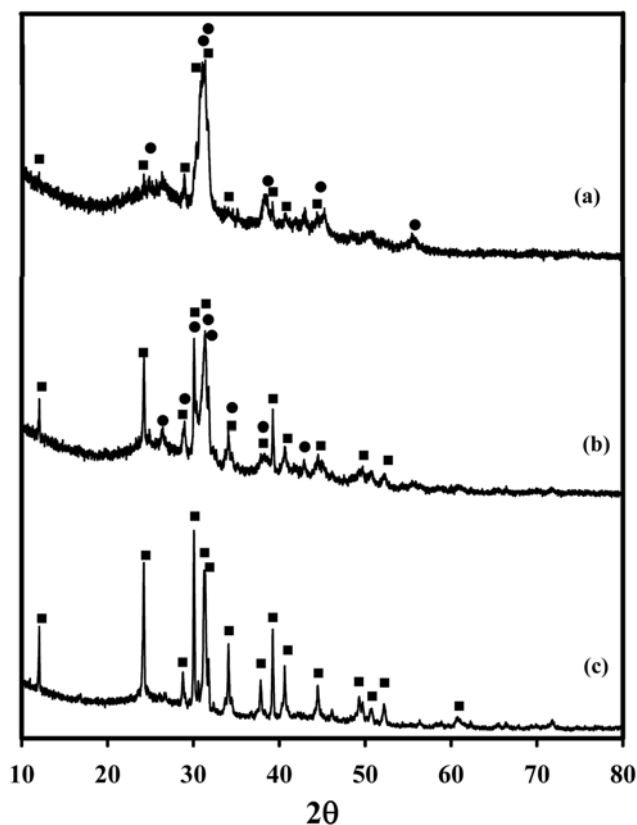


Fig. 2. The XRD patterns of the KACI30 sorbent after CO_2 absorption in the presence of various water vapors such as 1.3 vol% H_2O (a), 5.0 vol% H_2O (b), and 9.0 vol% H_2O (c) and 1 vol% CO_2 ; (●) K_2CO_3 ; (■) KHCO_3 .

In the case of 1.3 vol% H_2O , its total CO_2 capture capacity was about 40 mg CO_2 /g sorbent. This value was equivalent to about 42 percent of the theoretical value of the sorbent, when one mole of K_2CO_3 absorbed a stoichiometric amount of one mole of CO_2 and one mole of H_2O ($\text{K}_2\text{CO}_3 + \text{H}_2\text{O} + \text{CO}_2 \rightleftharpoons 2\text{KHCO}_3$) to form potassium hydrogencarbonate (KHCO_3) [8-14,17,18]. This low capture capacity in the presence of excess water vapor could not be explained by the above reaction mechanism. As the inlet water concentration increased, the total CO_2 capture capacity increased to 86.3 mg CO_2 /g sorbent, which was about 90.4 percent of the theoretical value. Although high CO_2 capture capacity was obtained, the point to note here is that the slope of the breakthrough curve is very low as shown in Fig. 1(a), indicating slow absorption of CO_2 .

Fig. 2 shows the XRD patterns of the KACI30 sorbent after CO_2 absorption in the presence of various water vapors such as 1.3 vol% H_2O (a), 5.0 vol% H_2O (b), and 9.0 vol% H_2O (c) and 1 vol% CO_2 . In the case of 1.3 vol% H_2O and 5.0 vol% H_2O , the peaks of K_2CO_3 were observed as shown in Fig. 2(a) and (b), while in the case of 9.0 vol% H_2O , it disappeared in the XRD patterns of Fig. 2(c). This indicates that K_2CO_3 was not completely converted into KHCO_3 under the concentration of 1.3 vol% H_2O and 5.0 vol% H_2O in spite of the excess water vapor as compared to 1.0 vol% CO_2 .

2. The Water Absorption of the Sorbent without CO_2

To identify this fact, the water absorption of the sorbent itself was carried out in the presence of various concentrations of water vapor.

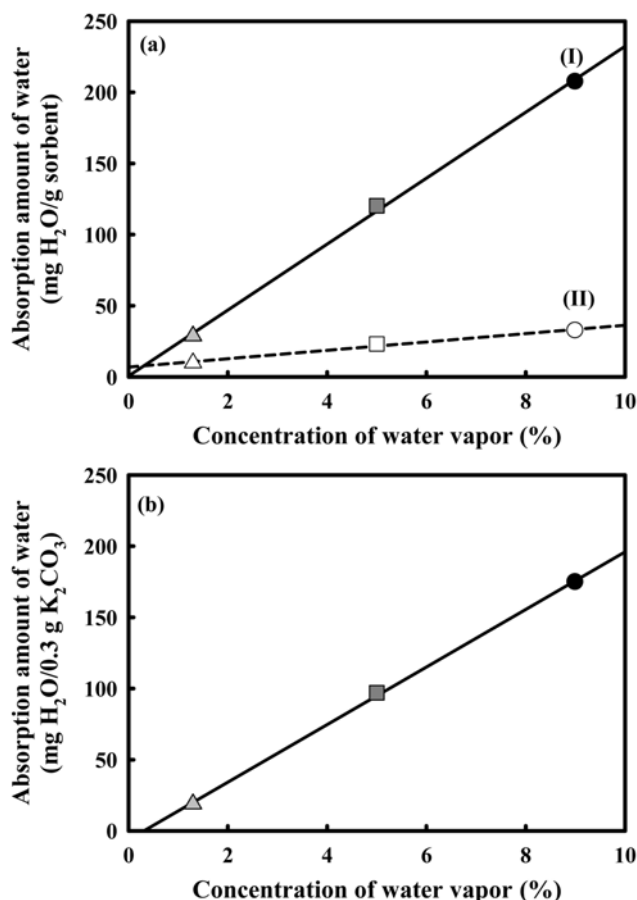


Fig. 3. The amount of water (a) absorbed on KACI30 (I) and 0.7 g of AC (II) at various concentrations of water vapor at 60 °C and the difference (b) between the absorption amounts of water for KACI30 (I) and that for AC (II) support.

Fig. 3(a) shows the amounts of water absorbed on the KACI30 sorbent (I) and 0.7 g AC (II) support, which are calculated from their breakthrough curves at various concentrations of water vapor. As shown in Fig. 3(a-I), the amount of water absorbed on the sorbent depends on the concentration of water vapor. When concentration of water vapor increased from 1.3 to 9.0 vol% H_2O , the absorption amount of water per 1 gram of the KACI30 sorbent increased linearly from 28.8 mg H_2O /g sorbent to 207.4 mg H_2O /g sorbent. Considering the following reaction to form the active species ($\text{K}_2\text{CO}_3 + 3/2 \text{H}_2\text{O} \rightarrow \text{K}_2\text{CO}_3 \cdot 1.5\text{H}_2\text{O}$) [8,10,11] the theoretical value for water absorption per 1 g KACI30 was 58.6 mg H_2O /g sorbent (58.6 mg H_2O /0.3 g K_2CO_3). However, the amount of water absorbed in the case of 9 vol% H_2O is more than 3.5 times of the theoretical value of the sorbent. This result suggests that activated carbon (AC), used as a support of KACI30, should adsorb a large amount of water. To check this fact, the absorption amounts of water on the activated carbon at various concentrations of water vapor are measured as shown in Fig. 3(a-II). With an increase in concentration of water vapor from 1.3 vol% H_2O to 9.0 vol% H_2O , the amount of the water absorption increased linearly from 9.9 to 32.6 mg H_2O . The difference between the water absorption amounts for KACI30 (Fig. 3(a-I)) and those for AC (Fig. 3(a-II)) should represent the amount of water used to convert the sorbent from K_2CO_3 into the active species, $\text{K}_2\text{CO}_3 \cdot$

1.5H₂O. The results are shown in Fig. 3(b). When the absorption of water of the sorbent was performed with 5 vol% and 9 vol% H₂O at 60 °C (25% and 46% relative humidity, respectively), the absorption amounts of water were 97.9 and 174.8 mg H₂O/0.3 g K₂CO₃, respectively. It must be noted that these values are higher than the theoretical value for water absorption per 1 g of KACI30 (0.3 g K₂CO₃). It was thought that a new structure or new water consumption reaction, with the exception of the formation of K₂CO₃·1.5H₂O, might be needed to explain the large absorption amounts of water.

3. Structure Identification

Fig. 4 shows the XRD patterns of the sorbent before (a) and after the absorption of water with various concentrations of water vapor such as 1.3 (b), 5.0 (c), and 9.0 (d) vol% H₂O. The important point

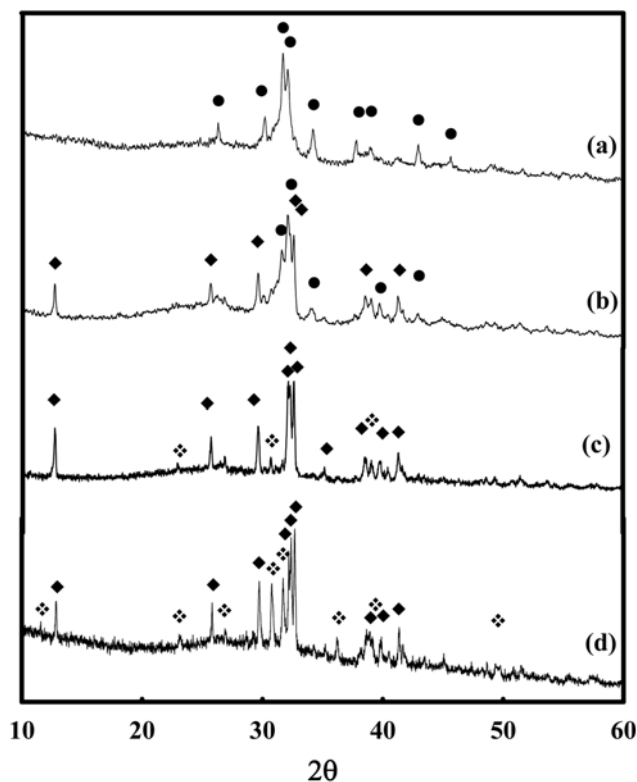


Fig. 4. XRD patterns of KACI30 before (a)/after pre-treatment with various concentrations of water vapor such as 1.3 (b), 5.0 (c), and 9.0 (d) vol% H₂O at 60 °C; (●) K₂CO₃; (◆) K₂CO₃·1.5H₂O; (◆) K₄H₂(CO₃)₃·1.5H₂O.

to note is that in the case of 1.3 vol% H₂O (b), K₂CO₃ did not completely convert into K₂CO₃·1.5H₂O. This fact agreed with the previous result that the absorption amount of water did not reach 58.6 mg H₂O/g sorbent. It was thought that this result was caused by the equilibrium of absorption and desorption of water at low relative humidity. When the absorption of water of the sorbent was performed under 5.0 (c) and 9.0 (d) vol% H₂O, K₂CO₃ could be completely converted into K₂CO₃·1.5H₂O. It was also found that a new structure assigned to a K₄H₂(CO₃)₃·1.5H₂O phase (JCPDS No. 20-0886), with the exception of K₂CO₃·1.5H₂O, was formed by absorption of water vapor. These facts indicate that K₄H₂(CO₃)₃·1.5H₂O, as well as K₂CO₃·1.5H₂O, could be formed by the absorption of water under the concentration range from 5.0 to 9.0 vol% H₂O.

4. The Effects of Water on Pre-treatment and CO₂ Absorption

It has been reported that the formation of K₂CO₃·1.5H₂O plays an important role in CO₂ capture capacity [8], and that the K₂CO₃·1.5H₂O phase formed from the K₄H₂(CO₃)₃·1.5H₂O phase during pre-treatment with excess water vapor before CO₂ absorption, was directly related to CO₂ capture capacity [11]. Pre-treatment was defined as the absorption process of water vapor before CO₂ absorption. In this section, to investigate the effects of water vapor on pre-treatment and CO₂ absorption, CO₂ breakthrough curves at 60 °C after pre-treatment in the presence of various concentrations of water vapor were compared and their net and total CO₂ capture capacities are shown in Table 2. When CO₂ was absorbed in the presence of 9 vol% H₂O without pre-treatment, the net and total CO₂ capture capacities of the sorbent were 37.7 and 82.0 mg CO₂/g sorbent, respectively. After pre-treatment with 9 vol% H₂O, the net CO₂ capture capacity increased to 62.8 mg CO₂/g sorbent, while the total CO₂ capture capacity was slightly increased to the value of 86.4 mg CO₂/g sorbent (equivalent to about 91 percent of the theoretical value). This result indicates that the pre-treatment process plays an important role in the net CO₂ capture capacity, because the active species, K₂CO₃·1.5H₂O, is already formed by the pre-treatment with water vapor, as mentioned in the XRD results of a previous section. When CO₂ absorption of the sorbent was performed in the absence of water, even after pre-treatment with 9 vol% H₂O, the net and total CO₂ capture capacities were only 50.2 and 67.4 mg CO₂/g sorbent, respectively. As the water concentration increased from 1.3 vol% H₂O to 9 vol% H₂O, the net and total CO₂ capture capacities increased from 50.2 mg CO₂/g sorbent to 62.8 mg CO₂/g sorbent, and from 67.4 mg CO₂/g sorbent to 86.4 mg CO₂/g sorbent, respectively. It is clear that the concentration of water vapor in the CO₂ absorp-

Table 2. The net CO₂ capture capacity and total CO₂ capture capacity of KACI30 in the presence of various concentrations of water vapor and 1 vol% CO₂ at 60 °C

Concentration of water vapor (vol%)		CO ₂ capture capacity (mg CO ₂ /g sorbent)		Total CO ₂ capture capacity/ theoretical value × 100 (%)
Pretreatment	CO ₂ absorption	Net CO ₂ capture capacity	Total CO ₂ capture capacity	
0	9.0	37.7	82.0	85.9
1.3	9.0	37.7	83.0	86.9
5.0	9.0	44.0	84.0	87.9
9.0	9.0	62.8	86.4	90.5
9.0	5.0	56.5	75.4	78.9
9.0	1.3	50.2	74.8	78.3
9.0	0	50.2	67.4	70.6

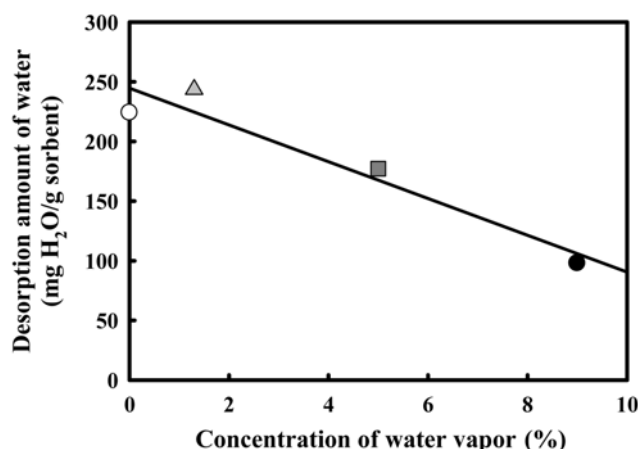


Fig. 5. The desorption amounts of water during CO₂ absorption in the presence of various concentrations of water vapor at 60 °C after pretreatment with 9 vol% H₂O.

tion process, as well as in pre-treatment process, is an important factor in obtaining the enhanced CO₂ capture capacity of the sorbent. In particular, the total CO₂ capture capacity of the sorbent depends on the concentration of water vapor, despite the complete formation of K₂CO₃·1.5H₂O through its pre-treatment with water vapor.

5. The Behavior of Water Vapor in CO₂ Absorption Process

Fig. 5 shows the desorption amount of water during the CO₂ absorption under the respective water concentration after pre-treatment with 9 vol% H₂O for 2 h. The amounts of water decreased from 224 mg H₂O/g sorbent to 108.7 mg H₂O/g sorbent, with an increase in the inlet water concentration. It was impossible to explain the desorption amount of water by the following reaction only: K₂CO₃·1.5H₂O + CO₂ + H₂O → 2KHCO₃ + 1.5H₂O, in which the desorption amount of water was constant regardless of the concentration of water vapor due to excess water vapor during the CO₂ absorption. It could be deduced that the high desorption amount of water was due to the physisorbed water and the conversion process such as

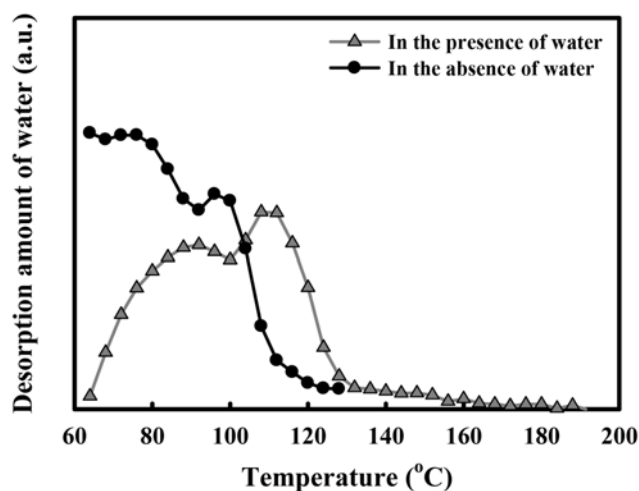


Fig. 6. TPD experimental results of sorbent activated through pre-treatment with 9 vol% H₂O at 60 °C under conditions with/without 9 vol% H₂O.

the conversion of K₄H₂(CO₃)₃·1.5H₂O to K₂CO₃·1.5H₂O and the transformation of K₂CO₃·1.5H₂O to K₂CO₃, as well as the reaction. Fig. 6 shows the TPD results of the sorbent under conditions with/without 9 vol% H₂O after pre-treatment with 9 vol% H₂O, when the temperature ramping rate was 1 °C/min. In both cases, the TPD result of the sorbent showed two types of water peaks. In the presence of 9 vol% H₂O, however, the desorption temperature of water increased as compared with that in the absence of water. This result indicates that there are two kinds of species within the sorbent after pre-treatment with 9 vol% H₂O, and that two peaks are shifted to the higher temperatures in the presence of 9 vol% H₂O. The two kinds of species agreed well with the XRD result shown in Fig. 4(d) (K₄H₂(CO₃)₃·1.5H₂O and K₂CO₃·1.5H₂O). It has been reported that a K₂CO₃ phase of the KACI30 sorbent transformed into a K₄H₂(CO₃)₃·1.5H₂O phase by pre-treatment under the excess water vapor (52 vol% H₂O) at 90 °C, and the K₄H₂(CO₃)₃·1.5H₂O phase could be converted into the K₂CO₃·1.5H₂O phase by drying under N₂ at 60 °C [11]. As shown in Fig. 7, this suggested that K₄H₂(CO₃)₃·1.5H₂O could be converted into K₂CO₃·1.5H₂O during CO₂ absorption at 60 °C and that the first peak was due to conversion of K₄H₂(CO₃)₃·1.5H₂O into K₂CO₃·1.5H₂O.

6. The Effect of Relative Humidity on Water Desorption and CO₂ Capture Capacity

The amount of water vapor desorbed was measured at various CO₂ absorption temperatures under 9 vol% H₂O, after pre-treatment with 9 vol% H₂O. As shown in Fig. 8(a), in spite of the same

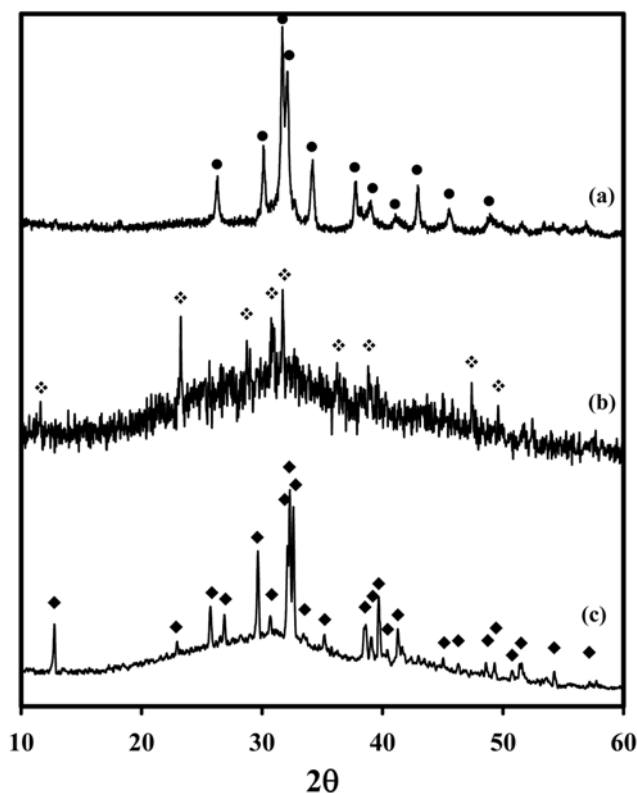


Fig. 7. XRD patterns of the KACI30 sorbent before (a) and after (b) pretreatment with the excess water vapor (52 vol% H₂O) and after (c) drying for 40 min with N₂ at 60 °C; (●) K₂CO₃; (◆) K₄H₂(CO₃)₃·1.5H₂O; (◆) K₂CO₃·1.5H₂O.

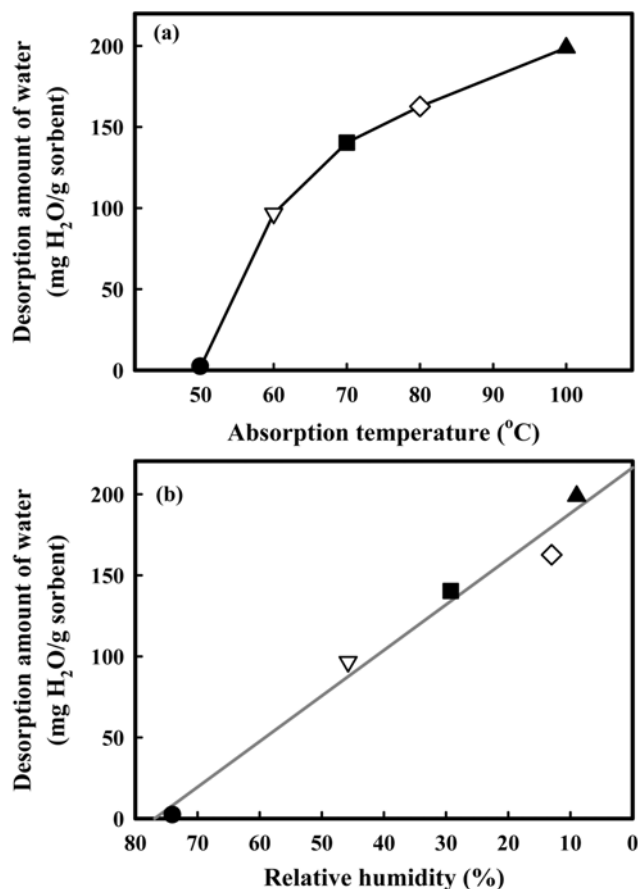


Fig. 8. The amount of water desorbed at various CO₂ absorption temperatures (a) and relative humidity (b).

concentration of water vapor (9 vol% H₂O), it was observed that the desorption amount of water increased with an increase in temperature. Fig. 8(b) shows the desorption amount of water as a function of relative humidity calculated at each temperature. The amount of water desorbed was linearly increased with a decrease in relative humidity. It was found that the desorption amount of water vapor directly depended on relative humidity, indicating that relative humidity was more important than the concentration of water vapor in the reactor.

Fig. 9 shows the CO₂ capture capacity of the sorbent as a function of absorption temperature and relative humidity in the presence of 9 vol% H₂O after pre-treatment with 9 vol% H₂O at 60 °C. As shown in Fig. 9(a), the sorbent showed CO₂ capture capacity of about 87 mg CO₂/g sorbent at 50 °C and 60 °C, while the value gradually decreased with an increase in temperature. To identify the effect of relative humidity on total CO₂ capture capacity, the CO₂ capture capacity was represented as a function of relative humidity. It was found that the total CO₂ capture capacity of the sorbent almost linearly decreased with a decrease in relative humidity. In a separate experiment, it was known that KHCO₃ desorbed most CO₂ in the temperature range between 80 °C and 160 °C [10]. This means that the KHCO₃ phase was starting to transform to K₂CO₃ by thermal decomposition from 80 °C. These results indicated that the CO₂ capture capacity of the sorbent could be affected by the thermal decomposition of KHCO₃ between 80 °C and 100 °C. As a result, to obtain

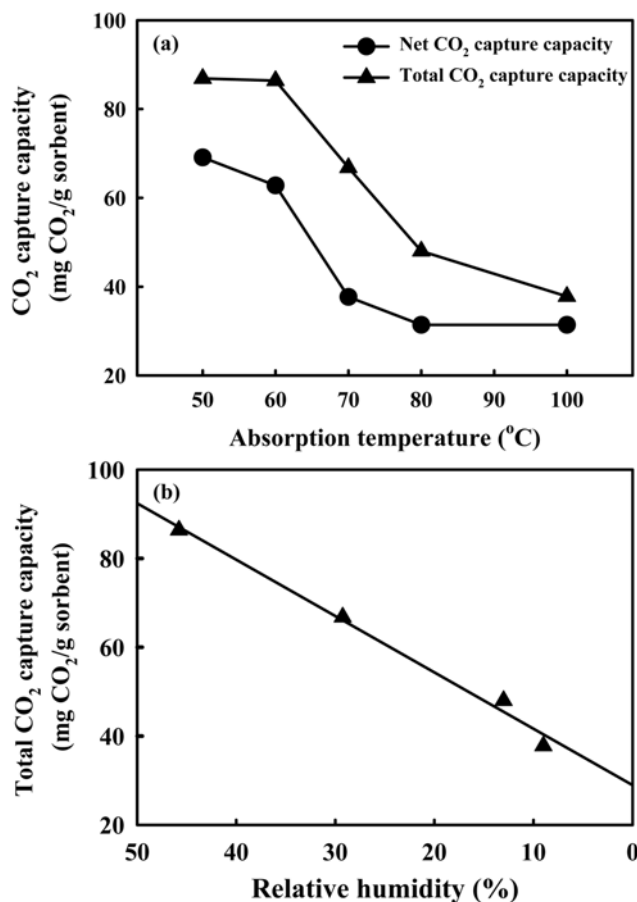


Fig. 9. CO₂ capture capacity of the sorbent in the presence of 9 vol% H₂O and 1 vol% CO₂ as a function of absorption temperature and relative humidity.

excellent CO₂ capture capacity, the sorbent always needs sufficient relative humidity during CO₂ absorption between 50 °C and 70 °C. It was concluded that the relative humidity in the reactor plays a very important role in preventing the transformation from K₂CO₃·1.5H₂O to the original phase, K₂CO₃, as well as in producing the K₂CO₃·1.5H₂O from K₂CO₃, during CO₂ absorption.

CONCLUSIONS

KACI30 showed not only a high CO₂ capture capacity of 86.4 mg CO₂/g sorbent, which was 91% of the theoretical value of 30 wt% K₂CO₃, but also excellent CO₂ absorption rate after pre-treatment with 9 vol% H₂O. The role of water vapor in pre-treatment was to enhance the net CO₂ capture capacity by converting the K₂CO₃ phase to active species, K₂CO₃·1.5H₂O, through pre-treatment with sufficient water vapor. It, however, was found that K₂CO₃·1.5H₂O could be easily transformed again to K₂CO₃ by thermal desorption even at low temperatures under very low concentration of water vapor due to the low relative humidity during CO₂ absorption. To enhance CO₂ capture capacity, it is very important to maintain the active species as well as to convert the entire K₂CO₃ to the K₂CO₃·1.5H₂O phase during CO₂ absorption between 50 °C and 70 °C. The sufficient relative humidity in the reactor plays a very important role in preventing the active species from transforming to the original phase, K₂CO₃,

in CO₂ absorption process. In conclusion, in the CO₂ absorption process, the CO₂ capture capacity of the potassium-based sorbent depends on the relative humidity in the reactor and CO₂ absorption temperature.

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