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CO_2 absorption and regeneration of alkali metal-based solid sorbents

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Abstract

Potassium-based sorbents were prepared by impregnation with potassium carbonate on supports such as activated carbon (AC), TiO_2, Al_2O_3, MgO, SiO_2 and various zeolites. The CO_2 capture capacity and regeneration property were measured in the presence of H_2O in a fixed-bed reactor, during multiple cycles at various temperature conditions (CO_2 capture at 60 °C and regeneration at 130–400 °C). Sorbents such as K_2CO_3/AC, K_2CO_3/TiO_2, K_2CO_3/MgO, and K_2CO_3/Al_2O_3, which showed excellent CO_2 capture capacity, could be completely regenerated above 130, 130, 350, and 400 °C, respectively. The decrease in the CO_2 capture capacity of K_2CO_3/Al_2O_3 and K_2CO_3/MgO, after regeneration at temperatures of less than 200 °C, could be explained through the formation of $KAl(CO_3)_2(OH)_2$, $K_2Mg(CO_3)_2$, and $K_2Mg(CO_3)_2\cdot 4(H_2O)$, which did not completely converted to the original K_2CO_3 phase. In the case of K_2CO_3/AC and K_2CO_3/TiO_2, a $KHCO_3$ crystal structure was formed during CO_2 absorption, unlike K_2CO_3/Al_2O_3 and K_2CO_3/MgO. This phase could be easily converted into the original phase during regeneration, even at a low temperature (130 °C). Therefore, the formation of the $KHCO_3$ crystal structure after CO_2 absorption is an important factor for regeneration, even at the low temperature. The nature of support plays an important role for CO_2 absorption and regeneration capacities. In particular, the K_2CO_3/TiO_2 sorbent showed excellent characteristics in CO_2 absorption and regeneration in that it satisfies the requirements of a large amount of CO_2 absorption (mg CO_2/g sorbent) and fast and complete regeneration at a low temperature condition (1 atm, 150 °C).

Keywords: Carbon dioxide; Absorption; Potassium carbonate; Potassium hydrogen carbonate; Regeneration

1. Introduction

Carbon dioxide (CO_2) is a greenhouse gas that is released into the environment due to the use of fossil fuels and causes global climate warming, which may be disastrous to the environment. CO_2 can be removed from flue gas and waste gas streams by various methods such as membrane separation [1], absorption with a solvent [2,3], and adsorption using molecular sieves [4–6]. These methods, however, are costly and consume a lot of energy.

One of the improved techniques for the removal of CO_2 is CO_2 chemical absorption, with regenerable solid sorbents. The use of solid sorbents containing alkali and alkali earth metals for CO_2 absorption has been reported in many literatures [7–17]. In these papers, alkali metal carbonate could be applied to CO_2 absorption at low absorption temperatures (50–60 °C), with thermal regeneration easily occurring at a low temperature. On the other hand, calcium oxide was applicable at much higher absorption temperatures (less than 860 °C). The sorption of CO_2 on the K_2CO_3–Al_2O_3 composite sorbent, in the presence of water vapor, was studied by in situ IR spectroscopy and X-ray diffraction analysis [13]. Several studies regarding the efficient chemical absorption over K_2CO_3, supported on carbon [8,9] or other different porous matrics such as silica gel, Al_2O_3 and vermiculite [12], were also reported by using cyclic fixed-bed operations under moist conditions, for the recovery of carbon dioxide from flue gases. Thus, several additives or supports such as activated carbon, SiO_2, Al_2O_3, and others have

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been used in the alkali metal-based sorbents to absorb CO_2. Nonetheless, these sorbents have a disadvantage in that there is a decrease of reactivity during multiple absorption/regeneration at low temperatures (40–200 °C). The reasons for the decrease of the CO_2 capture capacity and regeneration property of these sorbents, however, have not been clearly defined.

One of the objectives of this work was to develop a potassium-based regenerable solid sorbent for use in the CO_2 absorption/regeneration process, at low temperatures below 150 °C. The CO_2 capture capacity and regeneration property of several potassium-based sorbents were studied in a fixed-bed reactor using multiple tests. In addition, the changes in the physical properties of the sorbents before/after CO_2 absorption and mechanism were investigated with the aid of Power X-ray diffraction (XRD; Philips, X'PERT).

2. Materials and methods

2.1. Preparation of sorbents

Potassium-based sorbents used in this study were prepared by the impregnation of K_2CO_3 on porous supports such as activated carbon (AC, Aldrich), TiO_2, MgO (Aldrich), SiO_2 (Aldrich), Al_2O_3 (Aldrich), NaX (Aldrich) and CsNaX. CsNaX was prepared by the ion-exchanged method with NaX and cesium acetate [18]. TiO_2 was prepared by the sol–gel method from alcoholic metal alkoxide (Titanium *n*-butoxide, Aldrich) under acidic conditions [19]. Five (5.0) grams of support were added to an aqueous solution containing 2.5 g of anhydrous potassium carbonate (K_2CO_3, Aldrich) in 15 ml of de-ionized water. Then, it was mixed with a magnetic stirrer at room temperature for 24 h [9]. After stirring, the mixture was dried in a rotary evaporator at 60 °C. The dried samples were calcined in a furnace under N_2 flow (100 ml/min) for 4 h at 300 °C. The ramping rate of the temperature was maintained at 3 °C/min. The amount of alkali metal impregnated was determined by using a Varian spectra AA 800 atomic absorption spectrophotometer.

2.2. Apparatus and procedure

CO_2 absorption and regeneration processes were performed in a fixed-bed quartz reactor with a diameter of 1 cm, and placed in an electric furnace under atmospheric pressure. One-half (0.5) gram of sorbent was packed into the reactor, and space velocity (SV) was maintained at 3000 h^{-1} in order to minimize severe pressure drops and channeling phenomena. All volumetric gas flows were measured under standard temperature and pressure (STP) conditions. The temperature of the inlet and outlet lines of the reactor was maintained above 100 °C, in order to prevent the condensation of water vapor injected to the reactor. The column used in the analysis was a 1/8 in. stainless tube packed with Porapak Q. The conditions of CO_2 absorption and regeneration and the composition of mixed gases are shown in Table 1. Before CO_2 absorption, moist nitrogen with 9 vol.% H_2O at 60 °C was supplied to sorbents for 120 min by using a saturator. When the CO_2 concentration of the outlet gases reached the same level as the inlet gas (1 vol.%) in the CO_2 absorption process, moisture nitrogen containing 9 vol.% H_2O only was introduced in the multiple tests, in order to regenerate the sorbents until the CO_2 concentration reached 200 ppm. The outlet gases from the reactor were automatically analyzed every 4 min by a thermal conductivity detector (TCD; Donam Systems Inc.), equipped with an auto sampler (Valco Instruments CO. Inc.).

Table 1
Experimental conditions of potassium-based sorbents

	Pretreatment	CO_2 absorption	Regeneration
Temperature (°C)	60	60	130–400
Pressure (atm)	1	1	1
Flow rate (ml/min)	40	40	40
Gas composition (vol.%)	H_2O: 9; N_2: balance	CO_2: 1; H_2O: 9; N_2: balance	H_2O: 0, 9; N_2: balance

3. Results and discussion

3.1. Comparison of CO_2 capture capacities of various sorbents

Table 2 shows the total CO_2 capture capacities and the net CO_2 capture capacities of various sorbents after pre-treatment in the presence of 9 vol.% H_2O for 2 h at 60 °C. The phrase ''total CO_2 capture capacity'' describes the amount of CO_2 absorbed until the output concentration of CO_2 reached 1 vol.%, which is the identical value of concentration with that of the inlet. The net CO_2 capture capacity is defined as the amount of CO_2 absorbed per 1 g sorbent until the CO_2 concentration emitted from the reactor remained less than 200 ppm. The total CO_2 capture capacities of the potassium carbonate (30 wt.%), impregnated on the supports such as activated carbon (AC), Al_2O_3, MgO, and TiO_2 (K_2CO_3/AC, K_2CO_3/Al_2O_3, K_2CO_3/MgO, and K_2CO_3/TiO_2), were 86.0, 85.0, 119.0, and 83.0 mg CO_2/g sorbent, respectively. The total CO_2 capture capacities of K_2CO_3/AC, K_2CO_3/TiO_2, and K_2CO_3/Al_2O_3 were about 90% of the theoretical value of the sorbent when 1 mol of K_2CO_3 absorbed a stoichiometric amount of 1 mol of CO_2. The K_2CO_3/MgO sorbent observed a higher total CO_2 capture capacity than that of the theoretical

Table 2
The net CO_2 capture capacities and the total CO_2 capture capacities of various sorbents prepared by impregnation of potassium carbonate (30 wt.%) in the presence of 9 vol.% H_2O at 60 °C

Sorbent	Net CO_2 capture capacity	Total CO_2 capture capacity
K_2CO_3/AC	56.5	86.0
K_2CO_3/Al_2O_3	56.5	85.0
K_2CO_3/USY	6.3	18.9
K_2CO_3/CsNaX	0	59.4
K_2CO_3/SiO_2	0	10.3
K_2CO_3/MgO	75.4	119.0
K_2CO_3/CaO	37.7	49.0
K_2CO_3/TiO_2	44.0	83.0

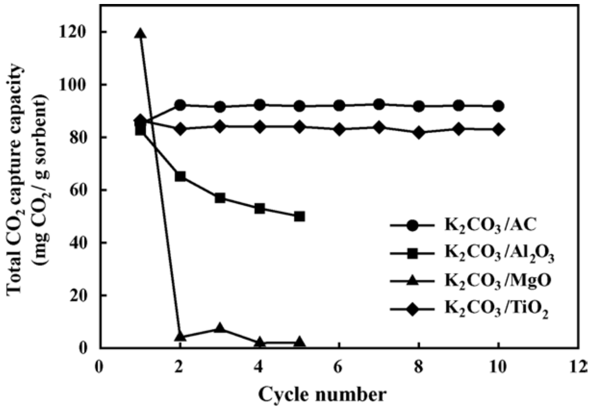

Fig. 1. The total CO_2 capture capacities of various sorbents as a function of cycle number (absorption at 60 °C and regeneration at 200 °C).

value. In addition, the net CO_2 capture capacity of this sorbent was higher than that of other sorbents.

Fig. 1 shows the total CO_2 capture capacities of sorbents such as K_2CO_3/AC, K_2CO_3/Al_2O_3 K_2CO_3/MgO, and K_2CO_3/TiO_2, as a function of cycle number. When both CO_2 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_2 absorbed per 1 g sorbent. The total CO_2 capture capacities of all sorbents observed were about 83–119 mg CO_2/g sorbent at the first cyclic number as shown in Fig. 1. The total CO_2 capture capacity of the K_2CO_3/AC sorbent after one cycle was significantly increased from 86 to 92 mg CO_2/g sorbent. The total CO_2 capture capacity of the K_2CO_3/TiO_2 sorbent was maintained during the multiple CO_2 absorption/regeneration cycles. In the case of K_2CO_3/Al_2O_3 and K_2CO_3/MgO, the total CO_2 capture capacities, however, dramatically reduced after two cycles. In particular, the total CO_2 capture capacity of K_2CO_3/MgO rapidly decreased from 119 to 4 mg CO_2/g sorbent. These results indicated that the K_2CO_3/Al_2O_3 and K_2CO_3/MgO sorbents were not completely regenerated at low temperatures below 200 °C. In addition, it was thought that the changes of the sorbent structure during CO_2 absorption depended upon the nature of support.

3.2. Structure identification of the sorbents before/after CO_2 absorption by XRD

As was mentioned previously, K_2CO_3/AC and K_2CO_3/TiO_2, unlike K_2CO_3/Al_2O_3 and K_2CO_3/MgO, showed very high CO_2 capture capacities and no deactivation of sorbents during multiple cycles, even after 10 cycles. In order to investigate these properties, the structural change of sorbents before/after the absorption was examined by XRD. Fig. 2 shows the XRD patterns of K_2CO_3/Al_2O_3 and K_2CO_3/MgO before/after CO_2 absorption. In the case of the K_2CO_3/Al_2O_3 sorbent, the XRD results of the fresh sorbents, which were calcined at 300 °C under nitrogen, showed two phases such as K_2CO_3 and $KAl(CO_3)_2(OH)_2$ phase as shown in Fig. 2(a: I). The new diffraction lines appeared with 2θ of 15.9, 21.6, 26.7, 28.4, 31.7, 35.8, 41.9, and 45.7, which were assigned to the $KAl(CO_3)_2(OH)_2$ phase (JCPDS no. 21-0979). It was obvious that $KAl(CO_3)_2(OH)_2$ was formed by the reaction of the alumina support with K_2CO_3 during calcinations at 300 °C. After CO_2 absorption at 60 °C, the XRD pattern of this sorbent showed two phases including $KHCO_3$ (JCPDS no. 70-0095) and $KAl(CO_3)_2(OH)_2$. The increase of the intensity of the $KAl(CO_3)_2(OH)_2$ peak after CO_2 absorption was shown in Fig. 2(a: II), as compared with Fig. 2(a: I). Fig. 2(b: I) showed the XRD patterns of the K_2CO_3/MgO fresh sorbent, which was calcined at 300 °C before reaction. It showed that two phases were assigned K_2CO_3 and MgO. After CO_2 absorption at 60 °C, the XRD of the K_2CO_3/MgO sorbent showed three phases, which were assigned to $K_2Mg(CO_3)_2$ (JCPDS no. 75-1725), $K_2Mg(CO_3)_2 \cdot 4(H_2O)$ (JCPDS no. 83-1955) and $Mg(OH)_2$ (JCPDS no. 83-0114), as shown in Fig. 2(b: II). It has been noticed that the $KHCO_3$ phase was not observed in

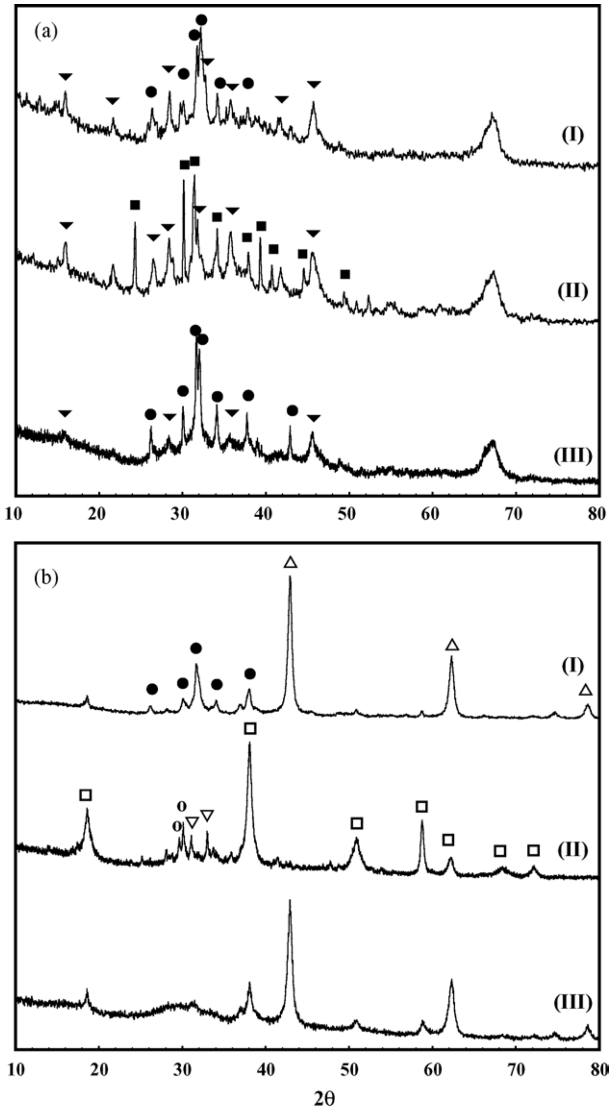

Fig. 2. The XRD patterns of K_2CO_3/Al_2O_3 and K_2CO_3/MgO before/after CO_2 absorption: (a) K_2CO_3/Al_2O_3; (b) K_2CO_3/MgO; (I) fresh; (II) after reaction with 9 vol.% H_2O and 1 vol.% CO_2 at 60 °C; (III) after regeneration under N_2 at 200 °C; (●) K_2CO_3; (■) $KHCO_3$; (▼) $KAl(CO_3)_2(OH)_2$; (△) MgO; (□) $Mg(OH)_2$; (▽) $K_2Mg(CO_3)_2$; (○) $K_2Mg(CO_3)_2 \cdot 4(H_2O)$.

the XRD of the K_2CO_3/MgO sorbent after CO_2 absorption at 60 °C. It has been reported that potassium hydrogencarbonate ($KHCO_3$) was formed through the reaction, $K_2CO_3 + H_2O + CO_2 \rightleftharpoons 2KHCO_3$, in the presence of moisture over potassium-based sorbents [8,9,11,20]. From the results obtained by XRD, it was concluded that new structures such as $KAl(CO_3)_2(OH)_2$, $K_2Mg(CO_3)_2$, and $K_2Mg(CO_3)_2{\cdot}4(H_2O)$ were formed during CO_2 absorption. Also, it could be suggested that the CO_2 capture capacity of K_2CO_3/MgO, which is higher than that of the theoretical value, was due to the formation of $K_2Mg(CO_3)_2$ and $K_2Mg(CO_3)_2{\cdot}4(H_2O)$ during absorption. The spent K_2CO_3/Al_2O_3 and K_2CO_3/MgO sorbents were not completely converted into the original phase (K_2CO_3) after regeneration at 200 °C, as shown in Fig. 2(a: III) and (b: III). These results indicated that the decrease in the capacities of the K_2CO_3/Al_2O_3 and K_2CO_3/MgO sorbents after regeneration, at temperatures lower than 200 °C, was due to the formation of new structures such as $KAl(CO_3)_2(OH)_2$, $K_2Mg(CO_3)_2$ and $K_2Mg(CO_3)_2{\cdot}4(H_2O)$, which were not completely converted into the original K_2CO_3 phase.

The XRD patterns of K_2CO_3/AC and K_2CO_3/TiO_2, before/after reaction, were shown in Fig. 3. In the case of the K_2CO_3/AC sorbent, the phase of this sorbent before reaction showed the K_2CO_3 phase, as shown in Fig. 3(a: I). This sorbent after CO_2 absorption showed one phase, $KHCO_3$. This result indicates that the K_2CO_3 phase of fresh sorbent was completely converted into $KHCO_3$. In the XRD pattern of the K_2CO_3/TiO_2 sorbent after CO_2 absorption, only the $KHCO_3$ phase was observed except for the TiO_2 phase. This result, also, indicated that the K_2CO_3 phase was completely converted into $KHCO_3$ during CO_2 absorption, in the presence of 9 vol.% H_2O as the K_2CO_3/AC sorbent. Moreover, in the case of the K_2CO_3/TiO_2 sorbent, the new structure was not observed during CO_2 absorption. These sorbents were easily regenerated by completely converting the entire $KHCO_3$ phase into the K_2CO_3 phase at 200 °C. In other words, this result indicates that the formation of the $KHCO_3$ phase, after CO_2 absorption, is an important factor regarding regeneration at low temperatures below 200 °C. It is concluded that the K_2CO_3/TiO_2 sorbent, also, has potential as a new regenerable solid sorbent for CO_2 absorption.

3.3. Regeneration property by TPD

In order to understand the regeneration properties of various sorbents in detail, a TPD test was performed after CO_2 absorption. The test was carried out by measuring the concentration of CO_2 desorbed when the temperature ramping rate was 1 °C/min. The TPD experimental results are shown in Fig. 4(a). In the case of the K_2CO_3/AC sorbent, most CO_2 was desorbed in the temperature range between 100 and 150 °C. The TPD result of the K_2CO_3/AC sorbent was similar to that of the K_2CO_3/TiO_2 sorbent. In the case of the K_2CO_3/MgO sorbent, most CO_2 was desorbed at temperatures of more than about 300 °C. On the other hand, the TPD result of the K_2CO_3/Al_2O_3 sorbent showed two types of CO_2 peaks. One was desorbed at the initial experiment temperature of 106 °C and the other was desorbed at temperature above 290 °C. This indicates that there are two kinds of structures within the K_2CO_3/Al_2O_3 sorbent after CO_2 absorption. To explain these results, the TPD tests for pure $KHCO_3$ and K_2CO_3 were performed and the results were shown in Fig. 4(b). In the case of $KHCO_3$, the CO_2 desorption peak, observed at around 130 °C, was almost consistent with those of K_2CO_3/AC and K_2CO_3/TiO_2. For pure K_2CO_3, however, CO_2 was not desorbed during the TPD test under nitrogen (N_2) at a temperature below 600 °C. From these results, the desorbed CO_2 of various sorbents (K_2CO_3/AC, K_2CO_3/TiO_2, and K_2CO_3/Al_2O_3) at around 130 °C could be explained by the decomposition of $KHCO_3$. These results were, also, completely consistent with the XRD results mentioned previously. It was thought that the CO_2 peak for K_2CO_3/Al_2O_3, at a high temperature range of 260–320 °C, was due to the $KAl(CO_3)_2(OH)_2$ structure. In

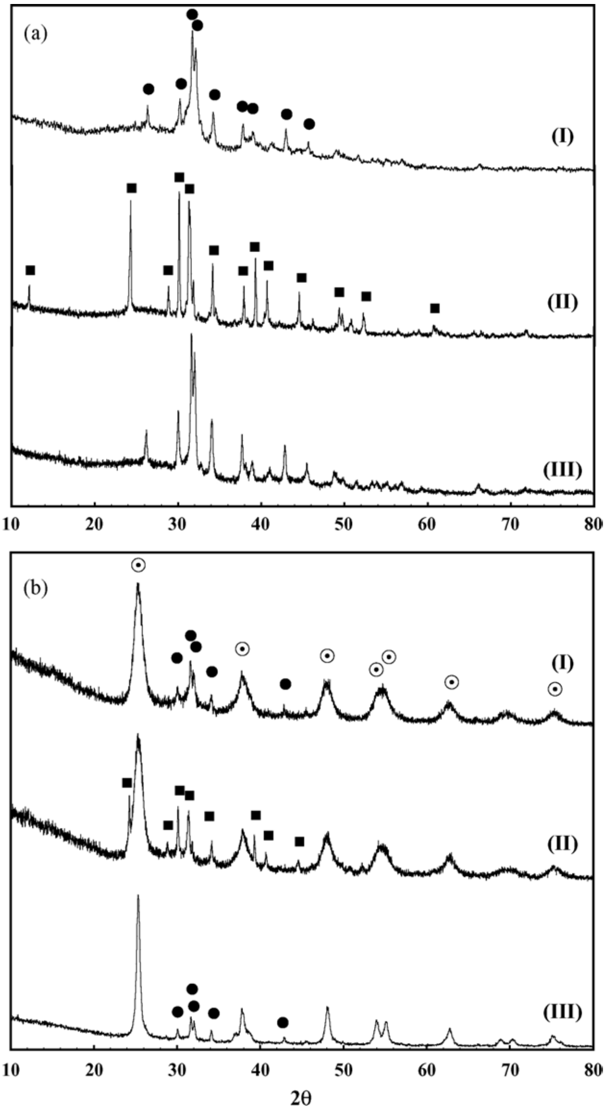

Fig. 3. The XRD patterns of K_2CO_3/AC and K_2CO_3/TiO_2 before/after CO_2 absorption: (a) K_2CO_3/AC; (b) K_2CO_3/TiO_2; (I) fresh; (II) after reaction with 9 vol.% H_2O and 1 vol.% CO_2 at 60 °C; (III) after regeneration under N_2 at 200 °C; (●) K_2CO_3; (■) $KHCO_3$; (⊙) TiO_2.

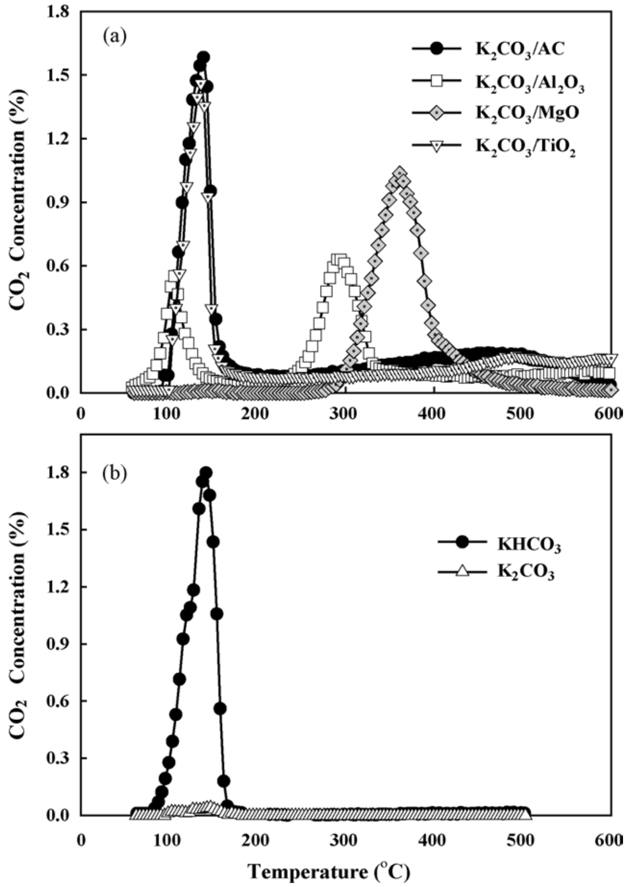

Fig. 4. TPD results after CO_2 absorption at 60 °C: (a) sorbents; (b) $KHCO_3$ and K_2CO_3.

addition, these results, also, indicated that the regeneration temperature of K_2CO_3/AC and K_2CO_3/TiO_2 was lower than that of K_2CO_3/Al_2O_3 and K_2CO_3/MgO due to the formation of the only $KHCO_3$ crystal structure after CO_2 absorption.

3.4. Regeneration property by XRD

From the TPD results and separated experiment results, it was suggested that sorbents such as K_2CO_3/AC and K_2CO_3/TiO_2, K_2CO_3/Al_2O_3 and K_2CO_3/MgO were completely regenerated at temperatures above 130, 130, 300, and 350 °C, respectively. The XRD patterns of these sorbents, after regeneration under nitrogen at each temperature (130, 130, 350, and 400 °C, respectively), were observed and were shown in Fig. 5. The separated K_2CO_3 crystal structure was observed in the XRD patterns of all the sorbents. In particular, in the case of K_2CO_3/AC and K_2CO_3/TiO_2, the $KHCO_3$ phase was completely converted into the K_2CO_3 phase even at 130 °C. In addition, the new structures such as $KAl(CO_3)_2(OH)_2$, $K_2Mg(CO_3)_2$, and $K_2Mg(CO_3)_2 \cdot 4(H_2O)$, which were observed after CO_2 absorption, were completely converted into the K_2CO_3 crystal structure at high regeneration temperatures. These results indicated that a sufficiently high temperature was needed to convert the new structures into the K_2CO_3 crystal structure. It is concluded that the formation of the only $KHCO_3$ crystal structure, through the CO_2 absorption in the presence of water, plays an important role in enhancing regeneration capacity.

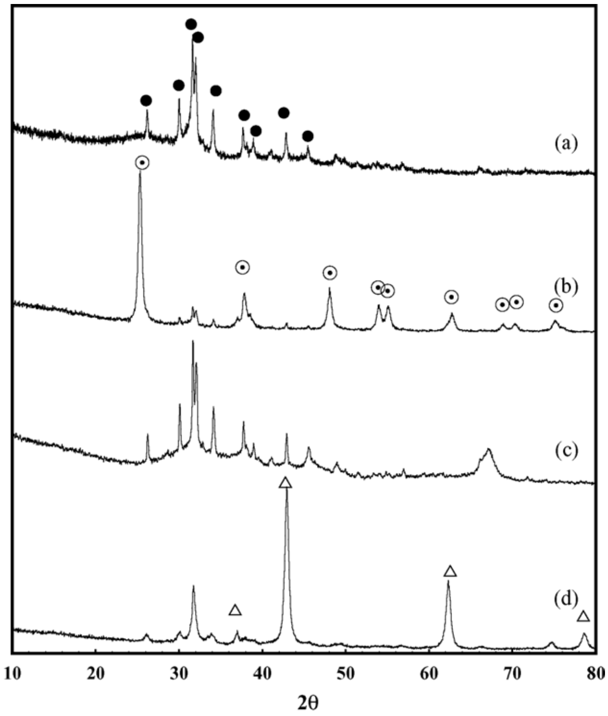

Fig. 5. XRD patterns of sorbents after regeneration under N_2 at each temperature: (a) K_2CO_3/AC, 130 °C; (b) K_2CO_3/TiO_2, 130 °C; (c) K_2CO_3/Al_2O_3, 350 °C; (d) K_2CO_3/MgO, 400 °C; (●) K_2CO_3; (⊙) TiO_2; (△) MgO.

3.5. Comparison of regeneration capacity of K_2CO_3/TiO_2 and K_2CO_3/AC

The CO_2 capture capacities of K_2CO_3/TiO_2 and K_2CO_3/AC were tested during the multiple absorption and low temperature regeneration cycles and were shown in Fig. 6. The regeneration process of K_2CO_3/TiO_2 and K_2CO_3/AC, after absorption at 60 °C, was performed at 150 °C. The total CO_2 capture capacities of K_2CO_3/AC and K_2CO_3/TiO_2 were maintained

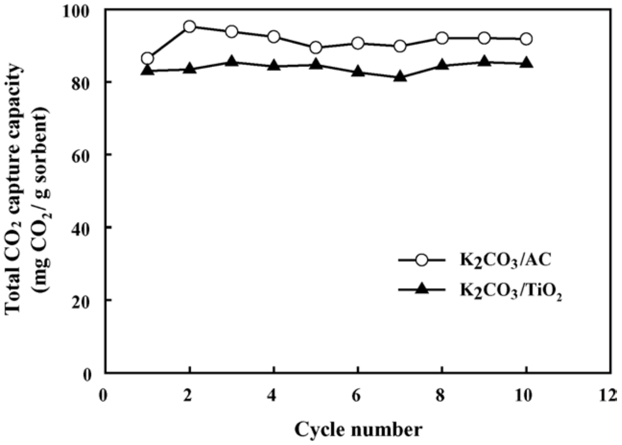

Fig. 6. The CO_2 capture capacities of K_2CO_3/TiO_2 and K_2CO_3/AC during the multiple absorption/regeneration cycles (absorption at 60 °C and regeneration at 150 °C).

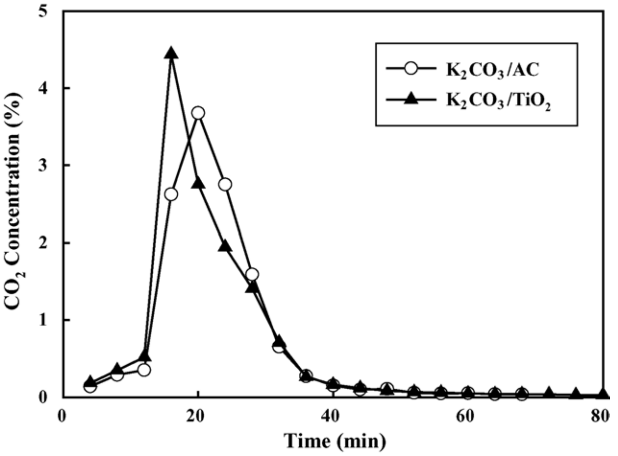

Fig. 7. CO_2 breakthrough curves of K_2CO_3/AC and K_2CO_3/TiO_2 during regeneration at 150 °C.

during multiple cycles. The total CO_2 capture capacities of these sorbents were between 83 and 92 mg CO_2/g sorbent. This value is about 85–95% of the theoretical value of sorbent loaded 30 wt.% K_2CO_3. It was clear that these spent sorbents could be completely regenerated at 150 °C.

In order to identify the regeneration ability of sorbents, the CO_2 concentration of the K_2CO_3/AC and K_2CO_3/TiO_2 sorbents were recorded during regeneration at a low temperature (150 °C) after purging under N_2 for 12 min, as shown in Fig. 7. The temperature ramping rate between 60 and 150 °C was 10 °C/min. It was observed that the maximum concentration of CO_2 was observed at approximately 16 min in the case of the K_2CO_3/TiO_2 sorbent and, then, the outlet concentration of CO_2 rapidly decreased. As shown in Fig. 7, the rate of regeneration of the new sorbent (K_2CO_3/TiO_2) was a little more than that of the K_2CO_3/AC at a low temperature (150 °C). The amount of CO_2 desorbed, which was calculated from the CO_2 concentration curves shown in Fig. 7, was 83–86 mg CO_2/g sorbent in both cases. These results showed that most of the CO_2 absorbed during CO_2 absorption was desorbed during the regeneration. Considering that the regeneration capacity was an important factor in addition to the CO_2 capture capacity, the K_2CO_3/TiO_2 sorbent, of which the regeneration temperature was lower than 150 °C, could be used as the sorbent that had the potential for CO_2 absorption.

4. Conclusions

The CO_2 capture capacity of K_2CO_3/AC, K_2CO_3/TiO_2, K_2CO_3/MgO, and K_2CO_3/Al_2O_3 was 86, 83, 119, and 85 mg CO_2/g sorbent, respectively. Also, it was possible to suggest that spent sorbents such as K_2CO_3/AC, K_2CO_3/TiO_2, K_2CO_3/Al_2O_3, and K_2CO_3/MgO could be completely regenerated at 150, 150, 350, and 400 °C, respectively. The nature of support plays an important role for CO_2 absorption and regeneration capacities. The CO_2 capture capacities of K_2CO_3/Al_2O_3 and K_2CO_3/MgO, after regeneration at low temperatures less than 200 °C, decreased due to the formation of $KAl(CO_3)_2(OH)_2$, $K_2Mg(CO_3)_2$, and $K_2Mg(CO_3)_2{\cdot}4(H_2O)$, which were not completely converted to the original K_2CO_3 phase. In the case of K_2CO_3/AC and K_2CO_3/TiO_2, it was possible to regenerate even within a low temperature range (130–150 °C), due to the formation of a $KHCO_3$ crystal structure during CO_2 absorption, unlike K_2CO_3/Al_2O_3 and K_2CO_3/MgO. In particular, the rate of regeneration of the new sorbent (K_2CO_3/TiO_2) was a little more than that of K_2CO_3/AC even at a low temperature (150 °C). Considering that the regeneration capacity was an important factor in addition to the CO_2 capture capacity, K_2CO_3/TiO_2 could be used as a sorbent that had the potential for CO_2 absorption.

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