

Article

Limitation of K_2CO_3 as a Chemical Agent for Upgrading Activated Carbon

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Abstract: The chemical activation of a carbon precursor with KOH generally results in an activated carbon (AC) with a high specific surface area. However, this process generates a large volume of wastewater that includes dissolved alkali metals, existing mainly as K_2CO_3 . Thus, wastewaters with a high concentration of dissolved K_2CO_3 can potentially be used in place of KOH as a chemical agent. In the present study, to reduce the thermal stability of K_2CO_3 , which decomposes at temperatures greater than 891 °C, K_2CO_3 was chemically impregnated into carbon precursors prior to activation of the precursors. The thermochemical properties and activation efficiency of the carbon precursors treated with K_2CO_3 were compared with those of carbon precursors treated with KOH. Analysis by XPS indicated that C–O–K complexes formed on the surface of the carbon precursors; in addition, their peak intensities were approximately the same irrespective of the chemical agent used. However, the specific surface area of the K_2CO_3 -impregnated AC was 2162 m²/g, which was ~70% of that of the KOH-impregnated AC (3047 m²/g) prepared using the same K/C molar ratio of 0.5. XRD results confirmed that both K_2CO_3 and KOH transformed into $KHCO_3$ and $K_4H_2(CO_3)_3 \cdot 1.5H_2O$ during the impregnation. The peak intensities of these compounds in the XRD pattern of the K_2CO_3 -impregnated carbon precursors were two times greater than those in the pattern of the KOH-impregnated carbon precursors. These compounds eventually transformed into K_2CO_3 , which hardly participated as a chemical agent at the temperature used in the present study (850 °C). Therefore, recrystallisation of K_2CO_3 , even during the impregnation, appeared to adversely affect the degree of activation. Nevertheless, the specific surface area of the K_2CO_3 -activated AC was still ~1.6 times greater than that of the untreated carbon precursor (1378 m²/g), suggesting that the use of wastewater as a chemical agent is feasible for resource recycling.

Keywords: activated carbon; chemical activation; KOH; K_2CO_3 ; impregnation; recycling



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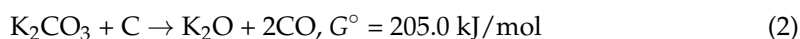
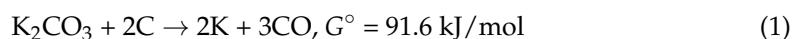
1. Introduction

Activated carbon (AC) has many reactive sites on its surface; it is therefore widely used as an adsorbent in the medical, automotive, water purification, and air-quality-control industries [1,2]. The AC market in Korea is divided into price-leading and technology-leading markets [3]. In particular, the demand for AC with a specific surface area greater than 2000 m²/g has been increasing rapidly because of strengthened air-quality regulations. According to the Korea International Trade Statistics [4], the amount of high-specific-surface-area AC imported in 2018 was ~5000 tonnes (~9.1% of the total imported AC) and originated from two specific countries (i.e., the United States and Japan).

Both physical and chemical activation processes are used to prepare AC. However, harsher conditions (e.g., a longer residence time and a higher loading of reagents) are necessary to prepare AC with a high specific surface area. This phenomenon is more pronounced with physical activation, where pores are developed by oxidative gases (i.e., steam and CO₂). Chemical activation has advantages over physical activation with respect

to being conducted at a lower activation temperature and providing a greater production yield [5–7]. Potassium salts such as KOH and K_2CO_3 have been widely used in the manufacture of AC, most commonly using KOH. It has been found that AC prepared by KOH is highly microporous with higher specific surface area. However, the high costs associated with chemical activation have been pointed out as a drawback and limitation, stemming from the excessive use of chemicals and the generation of byproducts such as wastewater by the cleaning processes [8,9]. To address this aspect, some researchers suggest the possibility of recycling the chemical, but KOH is transformed as carbonate by reacting with CO_2 during the activation [8–10]. Due to this reason, washing water recovered alkali metals mainly in the form of K_2CO_3 , which is normally a less effective activation agent [11].

The melting points of KOH and K_2CO_3 , which are commonly used as chemical agents for treating AC, differ greatly. KOH melts at $380\text{ }^\circ\text{C}$ and decomposes at $769\text{ }^\circ\text{C}$, whereas K_2CO_3 melts at $891\text{ }^\circ\text{C}$ and decomposes at $>1200\text{ }^\circ\text{C}$. As such, K_2CO_3 is less reactive than KOH as an agent during the activation process because of its high thermal stability [8,12,13], as documented by Lu et al. [14]. Even when the activation temperature is less than $830\text{ }^\circ\text{C}$, the Gibbs free energy (G°) of K_2CO_3 would not thermodynamically favor its reaction with carbonous materials:



However, Hayashi et al. [12,13] have confirmed that wet impregnation of K_2CO_3 lowers its decomposition temperature to $890\text{ }^\circ\text{C}$. This effect has been verified on various biomasses (i.e., almond, coconut, oil palm, pistachio, and walnut shell) with specific surface areas as high as $2000\text{ m}^2/\text{g}$ after impregnation with K_2CO_3 at $800\text{ }^\circ\text{C}$ [15].

Even if chemical activation has advantages over physical activation with respect to production yield and reaction temperature, it still requires a large amount of chemicals and generates large volumes of wastewater. Thus, using wastewater with a high concentration of K-containing compounds as a chemical agent would be an attractive method to reduce the manufacturing costs of AC. However, most previous studies on the surface modification of AC have focused on optimizing conditions by controlling the ratio of agents; consequently, only the effects of appropriate agents have been identified. Even though carbon precursors have been impregnated with K_2CO_3 to upgrade the adsorption performance of the resultant AC, the literature contains little research on why K_2CO_3 is less effective than KOH [11].

In the present study, activation reaction characteristics with an existing alkali metal were compared for reuse of a K-containing compound in the form of K_2CO_3 in alkaline wastewater. To overcome the unfavorable thermochemical properties of solid-state K_2CO_3 , we induced easier surface bonding of alkali-metal cations on the precursor surfaces via impregnation. The effects of introducing an alkali metal onto the surface of the carbon precursors, especially the activation properties of the alkali metal, were characterized. The results of this study are expected to suggest an approach for economically feasible chemical activation through effective utilization of high-concentration alkaline solutions generated in the AC washing process.

2. Materials and Methods

2.1. Preparation of Carbon Precursors and Chemical Activation

The carbon precursor in this study was commercial AC (JIG-SC-2040 BT, JAYEON SCI., Korea), which was produced from wood and exhibited a specific surface area of approximately $1378\text{ m}^2/\text{g}$. It has relatively high moisture (13.0%) and volatile (18.2%) contents while fixed carbon is about 63.4%, which is different from that of coconut-based AC [16]. For chemical activation, two types of K compounds (KOH, K_2CO_3) were used. Prior to the chemical activation, $\sim 30\text{ g}$ of AC was mixed with 200 mL of K compound solution and then impregnated in a rotary evaporator (N-1300, Eyela, Japan); the K/C molar

ratio was varied. After evaporation of the solution, the samples were dried at 105 °C for 24 h in preparation for activation. Five grams of impregnated carbon precursor was placed in a tubular furnace under flowing N_2 , and the temperature of the furnace was increased at 5 °C/min until the activation temperature (850 °C) was reached; the activation temperature was then maintained for 2 h. After the activation was terminated, the sample was washed with distilled water several times until the leachates were neutral to remove impurities.

2.2. Analytical Methods

To identify thermal properties of activators and carbon precursors impregnated, thermogravimetric analysis (TGA) was performed. All of the samples were heated to 1000 °C at a rate of 1 °C/min under a N_2 atmosphere. To quantify the amount of K impregnated onto the carbon surface, we used X-ray fluorescence (XRF) spectroscopy (Shimadzu, Japan). The surface properties of the carbon were characterised by XPS (K-Alpha+, Thermofisher, Waltham, MA, USA) and X-ray diffraction (XRD, SmartLab, Rigaku). The specific surface area was measured by N_2 adsorption (ASAP-2010, Micromeritics, Norcross, GA, USA) at −196 °C. We confirmed the pore distribution by varying the relative pressure (P/P_0) of the specimen after pretreatment and calculated the sub-segment distribution using the Dubin–Astakhov formula and the Barrett–Joyner–Halenda method for intermediate and large-scale machining. The porous size distribution (PSD) was calculated using the density functional theory.

3. Results and Discussion

3.1. Changes in Thermal Stability of Chemical Agents

Figure 1 shows the TGA results for the commercial AC used as a carbon precursor, the chemical activation agents (KOH and K_2CO_3), and the AC impregnated with chemical agents. The weight losses of the specimens activated using KOH and K_2CO_3 reflect the different thermal properties of the activating agents. K_2CO_3 did not function as an activating agent at 850 °C because of its high thermal stability relative to that of KOH. However, the initial point of weight loss, which indicates the decomposition temperature, for AC impregnated with K_2CO_3 shifted ~200 °C lower than that of K_2CO_3 . In fact, the TGA curves for the AC samples impregnated with agents varied depending on the carbon-to-agent ratio in the mixture. Thus, the TGA curves for this type of mixture are generally plotted along with the thermal properties of the two materials. However, at temperatures greater than 750 °C, the AC impregnated with K_2CO_3 exhibited a greater weight loss than the sample containing untreated AC. Given that K_2CO_3 exhibits negligible weight loss until 891 °C because of its thermal stability, the greater weight loss of the K_2CO_3 -impregnated compared with that of the untreated AC indicates that the thermal properties of K_2CO_3 changed as a result of the impregnation. These results indicate that K_2CO_3 dissolved in wastewater from the chemical activation with KOH can be reused through the impregnation process.

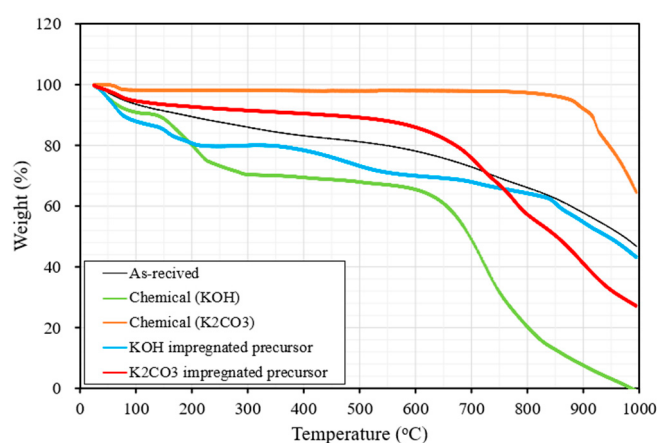


Figure 1. Thermogravimetric analysis results of chemical agents and carbon precursors impregnated with each chemical.

3.2. Changes in Surface Properties of Carbon Precursors Impregnated with KOH and K₂CO₃

3.2.1. XPS Analysis

Table 1 shows the XPS results used to verify the surface characteristics of the impregnated AC precursors. The XPS results confirm that the O and K contents of the samples increased with increasing amount of impregnated alkali metal. Because the surface properties of the carbon precursors are associated with an oxidation–reduction reaction during the activation process [17,18], an increase in O, which is an electron donor, on the carbon surface will strongly affect the pore development of the carbon matrix. Kopyscinski et al. [19] and Quyn et al. [20] have both proposed that C–O–K bound during the alkali-metal (i.e., K₂CO₃) impregnation is reduced to C–K at the surface of the carbon body, accompanied by the generation of CO. Lee et al. [21] and Punsuwan et al. [22], who qualitatively and quantitatively measured the gases emitted during the KOH activation of AC, also confirmed that CO is the main syngas ($K_2O + C \rightarrow 2K + CO$ at 800–850 °C [17,18]).

Table 1. XPS survey analysis of the KOH/K₂CO₃-impregnated carbon precursors.

Preparation of Carbon Precursors	K/C Ratio	C 1s (285.15 eV)	K 2p (294.25 eV)	N 1s (401.11 eV)	O 1s (532.73 eV)
KOH impregnation	0.5	27.1	9.17	N.D	63.7
K ₂ CO ₃ impregnation	0.2	77.5	0.99	0.89	20.6
	0.4	34.0	17.2	1.09	47.7
	0.5	23.9	8.19	N.D	67.9

To more closely investigate these binding properties of the carbon precursors, we deconvoluted the K and O peaks in the XPS spectra (Table 2). The K peak was deconvoluted into two peaks with binding energies of 293.0 eV (K 2p_{3/2}) and 295.6 eV (K 2p_{1/2}). The peaks for each of these main peaks were further deconvoluted into two peaks, which were identified as the K component of K–O bound to the surface (K_o) and K surrounded by carbonate (K_{CB}) [19]. These deconvolution characteristics are similar for the K 2p_{3/2} and K 2p_{1/2} peaks of carbon precursors impregnated with KOH and K₂CO₃. Deconvolution of the C 1s spectra for both KOH-impregnated AC and K₂CO₃-impregnated AC reveals peaks attributable to C=C (284.4 eV), C–C (285.9 eV), C–O (287.1 eV), C=O (288.5 eV) and COOH (289.7 eV). The intensity of the deconvoluted C 1s peaks varies depending on the K/C ratio but is independent of the agent at the same K/C ratio of 0.5. Thus, the increase in the specific surface area after activation of carbon precursors impregnated with KOH and K₂CO₃ at the same K/C ratio would be similar irrespective of the agent.

Table 2. Deconvolution of K 2p and C 1s XPS spectra of the KOH/K₂CO₃-impregnated carbon precursors.

Agent	K/C Ratio	K 2p _{3/2}		K 2p _{1/2}		C 1s				
		K _o (293.0)	K _{CB} (295.7)	K _o (294.5)	K _{CB} (296.6)	C=C (284.4)	C–C (285.9)	C–O (287.1)	C=O (288.5)	COOH (289.7)
KOH	0.5	49.6	15.9	25.7	8.88	51.7	22.5	7.59	10.2	7.95
	0.2	53.9	11.5	25.9	8.68	71.7	16.8	5.65	3.62	2.15
K ₂ CO ₃	0.4	54.2	10.3	30.9	4.54	56.3	14.5	6.79	8.36	14.1
	0.5	50.0	15.2	26.3	8.59	50.8	23.7	7.62	10.4	7.40

K_o: K–O groups, K_{CB}: potassium surrounded by carbonate, Numbers in parentheses represent binding energy in eV.

3.2.2. XRD Results

Figure 2 shows the results of the XRD analysis of the carbon precursors impregnated with KOH and K₂CO₃ at a K/C molar ratio of 0.5. Similar XRD peak patterns are observed for the carbon precursors impregnated with the different activating agents. The main peak is attributed to KHCO₃; however, weak peaks of K₄H₂(CO₃)₃ were also observed. Although K₄H₂(CO₃)₃ is known as an intermediate composition of KHCO₃ crystals exposed to a CO₂

atmosphere [23,24], it eventually transforms to KHCO_3 : $\text{K}_4\text{H}_2(\text{CO}_3)_3 \cdot 1.5\text{H}_2\text{O}_{(\text{s})} + \text{CO}_{2(\text{g})} \rightleftharpoons 4\text{KHCO}_3 + 0.5\text{H}_2\text{O}_{(\text{g})}$. Nevertheless, the large differences in the specific surface areas of the upgraded ACs impregnated with KOH and K_2CO_3 , as shown in Figure 3, are difficult to explain. Because KHCO_3 also transforms into K_2CO_3 at temperatures greater than 140°C ($2\text{KHCO}_{3(\text{s})} \rightleftharpoons \text{K}_2\text{CO}_{3(\text{s})} + \text{H}_2\text{O}_{(\text{g})} + \text{CO}_{2(\text{g})}$) [25], the amounts of K_2CO_3 formed on the carbon precursors would adversely affect the degree of activation. Given that the peaks of $\text{K}_4\text{H}_2(\text{CO}_3)_3$ and KHCO_3 are more intense for the carbon precursor impregnated with K_2CO_3 than for that impregnated with KOH, the K_2CO_3 -impregnated sample would favor the formation of K_2CO_3 during activation. Because K_2CO_3 is thermally stable to 891°C , it hardly participated as a chemical agent at the highest temperature used in the present study (850°C), which is why the specific surface area of the carbon precursor impregnated with K_2CO_3 was lower than that of the precursors impregnated with KOH.

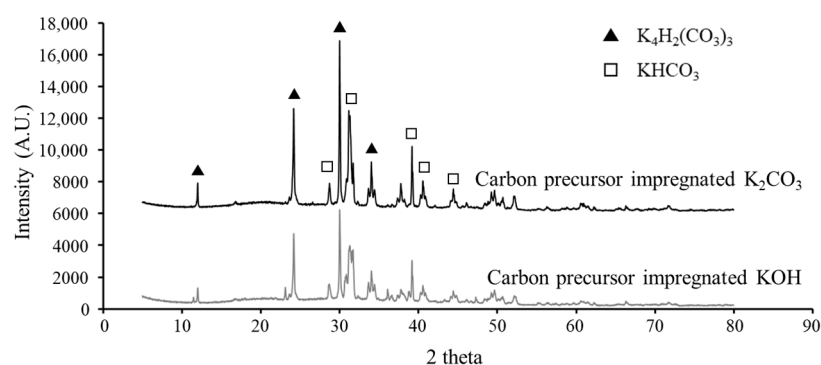


Figure 2. XRD patterns for KOH/ K_2CO_3 -impregnated carbon precursors.

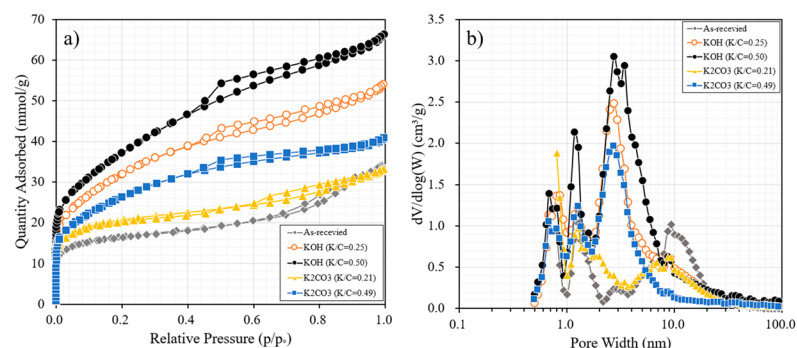


Figure 3. (a) N_2 isotherms and (b) pore size distributions of activated carbons prepared by impregnation with KOH and K_2CO_3 at different K/C molar ratios.

3.3. Chemical Activation with KOH and K_2CO_3

For chemical activation, carbon precursors were prepared by being physically and chemically mixed with KOH and K_2CO_3 . The specific surface area of the AC after chemical activation with KOH was superior to that of the AC after chemical activation with K_2CO_3 , irrespective of the mixing method (Figure 3). The specific surface area of the AC activated with KOH at a K/C ratio of 0.5 increased from $1400\text{ m}^2/\text{g}$ to $3047\text{ m}^2/\text{g}$, which is $\sim 30\%$ greater than that of the AC activated with K_2CO_3 ($2162\text{ m}^2/\text{g}$) under otherwise identical conditions. These results clearly show differences in the thermal properties of the ACs treated using KOH and K_2CO_3 . However, the specific surface area of the AC impregnated with K_2CO_3 at the same K/C ratio was $\sim 24\%$ greater than that of the AC mixed with K_2CO_3 physically. Thus, the physical properties of K_2CO_3 were changed during the impregnation (Figure 1), enabling the K doped into the carbon precursors to more easily intercalate into the carbon matrixes.

Figure 3 shows the N_2 isotherms and PSD for the ACs treated using KOH and K_2CO_3 at various K/C ratios. The shape of the N_2 isotherms and PSDs varies with the K/C ratio;

however, no substantial differences are observed between the ACs treated using these two agents. Nevertheless, AC impregnated with K_2CO_3 at a twofold K loading during the activation process was similar to that of AC treated with KOH at a K/C ratio of 0.25. These results reflect the relatively low reactivity of K as a chemical agent when impregnated as K_2CO_3 compared with that of K impregnated as KOH at the same K/C ratio.

4. Conclusions

In the present study, the reactivity of K_2CO_3 as a chemical agent was compared with that of KOH to evaluate the feasibility of recycling alkaline wastewaters generated as a byproduct during chemical activation. Wet impregnation with K_2CO_3 on carbon precursors effectively lowered the thermal stability of K_2CO_3 , as indicated by a decrease in its melting point. However, the specific surface area ($2162\text{ m}^2/\text{g}$ at K/C = 0.5) of the K_2CO_3 -impregnated AC was still only ~70% of that of the KOH-impregnated AC ($3047\text{ m}^2/\text{g}$ at K/C = 0.5). The XPS and XRD results showed similar surface binding properties (C–O–K) and potassium compounds ($KHCO_3$, $K_4H_2(CO_3)_3$) in the precursors impregnated with these two agents (K_2CO_3 and KOH). However, the intensity of $KHCO_3$ peaks in the XRD patterns differed dramatically dependent on the agent used. This result indicated that the degree of formation of C–O–K complexes, which participate in the activation reaction, strongly affected the carbon surface even if the binding energies and peak intensities were similar. Consequently, the specific surface area of the K_2CO_3 -impregnated AC was lower than that of the KOH-impregnated AC even though the high thermal stability of K_2CO_3 had been overcome. Nevertheless, the specific surface area of the K_2CO_3 -activated AC was still ~1.6 times greater than that of the carbon precursor ($1378\text{ m}^2/\text{g}$), suggesting the possibility of resource recycling by reusing wastewaters as a chemical agent.

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