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Removal of Volatile Toluene Using $\text{K}_2\text{CO}_3$-Activated Carbon Adsorbents Prepared from Buckwheat Hull

Tomoya Takada *©, Ryo Tanaka and Ryoto Ono

Department of Applied Chemistry and Bioscience, Chitose Institute of Science and Technology, Bibi, Chitose 066-8655, Japan; m2200080@photon.chitose.ac.jp (R.T.); m2210060@photon.chitose.ac.jp (R.O.)
* Correspondence: t-takada@photon.chitose.ac.jp

Abstract: Carbon adsorbents for use in the removal of gaseous toluene from the air were prepared from buckwheat (Fagopyrum esculentum Moench) hull. A chemically-activated adsorbent was prepared via the impregnation of raw hull powder with potassium carbonate, followed by thermal decomposition. The chemically-activated adsorbent exhibited improved adsorption capacity for toluene compared to the adsorbent prepared without chemical activation. Toluene concentration in the air decreased from 220 ppm to 160 ppm during 24 h of adsorption using unactivated adsorbent. Only a trace amount of toluene remained after the adsorption under the same conditions using $\text{K}_2\text{CO}_3$-activated adsorbent. This improvement was explained based on experimental results, specifically, iodine adsorption tests, methylene blue adsorption tests, and microscopic observations. Chemical activation dramatically increased the specific surface area of the adsorbent and created mesopores capable of adsorbing toluene. This study revealed that a mesoporous adsorbent for use in volatile toluene removal can be prepared from waste biomass (buckwheat hull) by chemical activation using potassium carbonate.

Keywords: adsorbent; buckwheat hull; toluene; $\text{K}_2\text{CO}_3$; chemical activation

1. Introduction

The removal of volatile organic compounds (VOCs) from the air is quite important in industry. Typical VOCs emitted by processes used in the chemical and petroleum industries contain aromatics, ethers, aldehydes, halogenated compounds, and so on [1]. Toluene is among the most important VOCs emitted by various industrial processes. It has been widely used as a representative solvent in the manufacture of paints, inks, rubbers, adhesives, and various other chemical substances. Toluene is emitted to the atmosphere via vaporization, leakage, and in exhausts. The emission of gaseous toluene is a public health concern because toluene is neurotoxic, and exposure can cause headaches, dizziness, mental depression, cognitive dysfunction, and many other symptoms [2,3].

Many methods have been proposed for use in the removal of VOCs from the air, including adsorption, catalytic decomposition, absorption, photocatalytic oxidation, thermal oxidation, and bioremediation. Among them, adsorption is widely applied. It is considered to be a superior VOC removal method because of its low energy consumption, cost effectiveness, and high capacity for VOC removal, even at low VOC concentrations [4,5]. Various adsorbents have been used for this purpose; in particular, activated carbon is commonly used. Activated carbon is one of the most important forms of industrially used carbon. It has a very high specific surface area and a well-developed surface pore structure, which make it suitable for use as an adsorbent for the removal of gaseous pollutants [6].

Activated carbon can be prepared from various plant-derived biomasses. Coconut shell and sawdust are the main raw materials used in commercial production. Other natural substances have also been used, for example, sugarcane bagasse [7], straws [8], rice husks [9], corncob [10], spent coffee grounds [11], nutshells [12], and many other...
agricultural wastes [13]. In Japan, buckwheat (Fagopyrum esculentum Moench) is frequently consumed as an ingredient in foods such as noodles, crackers, and beverages. Buckwheat hull is generated during the production of buckwheat flour. It was once used to stuff pillows, but this practice is now less common because of concerns regarding allergic effects and vermin. Buckwheat hull has thus become an agricultural waste. Therefore, applications that effectively utilize buckwheat hull are strongly desired. Although buckwheat hull is expected to be a good raw material for activated carbon production, this possibility has not undergone much progress recently. Kuwamura and Takahashi reported that a carbonaceous product was obtained by the thermal decomposition of buckwheat hull at 300–550 °C [14]. In this example, however, the only activation method used was heating. Other activation methods, such as steam and chemical activation, were not employed. The activation of the prepared activated carbon using chemicals is expected to significantly increase its adsorption capacity.

In the present study, activated carbons were prepared from buckwheat hull to produce adsorbents for the removal of gaseous toluene. To introduce the pores needed for toluene adsorption, chemical activation with potassium carbonate (K₂CO₃) was applied. The main aim of this work was to evaluate the effect of the K₂CO₃ chemical activation of adsorbents derived from buckwheat hull on their gaseous toluene removal capacity. The toluene removal capacities of the adsorbents prepared with and without K₂CO₃ activation were compared. The effect of K₂CO₃ activation on toluene adsorption was investigated through I₂ adsorption, methylene blue adsorption, scanning electron microscope (SEM) observation, and toluene adsorption.

K₂CO₃ chemical activation is known to introduce pores and increase the specific surface area of adsorbents [15]. Typical chemical activating agents for the preparation of activated carbon include K₂CO₃, ZnCl₂, H₃PO₄, NaOH, and KOH. Among them, K₂CO₃ is the least harmful and the least corrosive; these are advantageous characteristics for its industrial applications in activated carbon production. Despite these advantages, there are a few examples of the preparation of the activated carbon from buckwheat hull by chemical activation using K₂CO₃. In the present case, the K₂CO₃-activated adsorbent adsorbed much more toluene than the unactivated adsorbent. The enhanced toluene removal capacity was explained by the differences in specific surface areas and mesopore structures between the unactivated and K₂CO₃-activated adsorbents. These results showed that chemical activation via K₂CO₃ was effective in improving the toluene adsorption capacity of the buckwheat hull-derived carbon adsorbent.

2. Materials and Methods

Adsorbents were prepared from buckwheat hull obtained in Abira Town, Hokkaido, Japan. The buckwheat hull was ground and sifted to obtain a powder with particle sizes of 1 mm to 180 µm. The hull powder (10 g) was added to an aqueous potassium carbonate (K₂CO₃, Junsei Chemicals, Tokyo, Japan) solution consisting of 10 g of K₂CO₃ and 15 mL of water. The mixture was then stored in a desiccator for 24 h under reduced pressure to impregnate the hull powder with K₂CO₃ and vaporize the water. After impregnation, a pasty mixture was obtained. The mixture was further dried at 110 °C for 24 h. The dried mixture was ground with a mortar and pestle and heated in a tubular furnace (FT-01 VAC-WM, Full-tech, Yao, Japan) in nitrogen flow under reduced pressure. The heating temperature and time were 800 °C and 2 h, respectively. The obtained carbonaceous product was boiled in water for 1 h to remove potassium-containing products and unreacted K₂CO₃. Next, the carbonaceous product was filtered and dried at 110 °C until it was completely dry. An adsorbent was also prepared without K₂CO₃ impregnation via the simple thermal decomposition of buckwheat hull powder under the same conditions. The prepared adsorbents were characterized by iodine and methylene blue adsorption tests, X-ray diffractometry measurement, scanning electron microscopy (SEM), and Fourier transform infrared absorption (FT-IR) spectroscopy.
The buckwheat hull powders that were prepared with and without K$_2$CO$_3$ impregnation were both converted to black carbonaceous solids via heating. However, the product yields differed somewhat. The calculated yields (amount of product/amount of buckwheat hull powder) were 26 wt% and 23 wt% for the unactivated and K$_2$CO$_3$-impregnated hull powders, respectively. The low yield of activated carbon is common. For example, the yield of activated carbons obtained through gas activation has been reported to be about 10% [16]. Muroyama et al. reported that the yield of activated carbons obtained from bean curd via K$_2$CO$_3$ activation was about 5–20% depending on the heating temperature used [17]. These low yields are due to the gasification of carbon with various reactants, such as H$_2$O, O$_2$, CO$_2$, K$_2$O, K$_2$CO$_3$, and so on. When the hull powder was carbonized in the presence of K$_2$CO$_3$, the reduction of K$_2$CO$_3$ via carbon occurred, and K$_2$CO$_3$ and carbon reacted to generate metallic potassium, CO, and CO$_2$ [18]. Consequently, some of the carbonaceous product was lost. Burning was observed when the K$_2$CO$_3$-impregnated product and the inner wall of the quartz tube used for carbonization were in contact with water. This behavior indicates the formation of metallic potassium through the reduction of K$_2$CO$_3$ via carbon.

Figure 1 shows photographs of 1 g samples of the unactivated and K$_2$CO$_3$-activated adsorbents. Their bulk densities are clearly different. This difference suggests that K$_2$CO$_3$ activation effectively introduces pores into adsorbents. This significant lowering of the bulk density via K$_2$CO$_3$ activation is caused by the thermal decomposition of K$_2$CO$_3$ itself and/or the reduction of K$_2$CO$_3$ via carbon. Both of these reactions generate CO and CO$_2$, and they effectively create pores, as shown later in the paper. This pore formation results in the decrease in apparent bulk density. The apparent bulk densities of the adsorbents were roughly estimated from the weight (1 g) to volume ratios, which were calculated from the height and diameter of the particle layers in the test tubes. The densities were estimated to be 0.5 g/cm$^3$ and 0.1 g/cm$^3$ for the unactivated and K$_2$CO$_3$-activated adsorbents, respectively.

![Figure 1. Photographs of unactivated adsorbent (A) and K$_2$CO$_3$-activated adsorbent (B).](image)

The I$_2$ adsorption test was performed as follows. First, a 0.05 mol/L iodine (I$_2$) aqueous solution containing potassium iodide (KI) was prepared. KI was used to make I$_2$ soluble. The molar ratio of I$_2$ and KI in the solution was 1:3. Each prepared adsorbent (0.1 g) was weighed and added separately to 50 mL of the aqueous I$_2$ solution. The mixtures were stirred at 25 °C for 5 h in the dark. They were then centrifuged to separate the adsorbent from the solution, and the I$_2$ concentration of the supernatant was determined via redox titration using a 0.1 mol/L aqueous sodium thiosulfate (Na$_2$S$_2$O$_3$) solution. The amount of adsorbed I$_2$ was determined from the difference in the measured I$_2$ concentrations before and after adsorption. The detection limit of I$_2$ amount was expected to be 1.27 mg under the experimental condition; 0.01 mL (the minimum scale) of the Na$_2$S$_2$O$_3$ solution corresponded to 1.27 mg of I$_2$.

The methylene blue adsorption test was performed using a 0.40 mmol/L aqueous methylene blue solution. This solution (20 mL) was mixed with an appropriate amount of
the unactivated or K$_2$CO$_3$-activated adsorbent. The mixture was stirred for 2 h at 25 °C and then centrifuged to separate the adsorbent from the solution. The visible light absorbance of the supernatant at 650 nm was measured using a UV–visible spectrophotometer (V-630, JASCO, Tokyo, Japan). The amount of adsorbed methylene blue was determined using a calibration curve.

Powder X-ray diffraction (XRD) patterns of the prepared adsorbents were recorded with an X-ray diffractometer (RINT-2000, Rigaku, Tokyo, Japan) using CuK$_\alpha$ radiation and a scan rate of 2°/min. SEM images (VE-8800, Keyence, Osaka, Japan) and FT-IR spectra (FT/IR-6600, JASCO, Tokyo, Japan) were recorded. The adsorbents were tableted using potassium bromide before the FT-IR measurements.

Toluene adsorption tests were conducted using a Tedlar® bag (AAK-5, GL Science, Tokyo, Japan). The tests were conducted at ambient temperature (15–16 °C). An adsorbent (0.2 g) was placed in a corner of the bag, and the corner was partitioned with a clip. Then, toluene and air were mixed in the rest of the bag to make a gaseous air–toluene mixture with a toluene concentration of 200–240 ppm. After the toluene was vaporized, the initial toluene concentration was measured using a gas detector tube (No. 122, GASTEC, Kanagawa, Japan). The clip used for partitioning was then removed to enable adsorption. The change in toluene concentration was observed in the same manner for 120 min or 24 h. Figure 2 shows the experimental procedure for the toluene adsorption test. The gas detector tube indicates toluene concentration by coloration with I$_2$ generated through the reduction of I$_2$O$_5$ via toluene. The stated detection limit was 1 ppm, and the stated coefficients of variation of measured concentration were 5% (10–100 ppm) and 10% (100–300 ppm).

![Figure 2. Experimental procedure for toluene adsorption test.](image)

3. Results and Discussion

3.1. Characterization of Adsorbents

Figure 3 shows the XRD patterns of the unactivated and K$_2$CO$_3$-activated adsorbents. These patterns indicate that the products had turbostratic graphite structures; the peaks corresponding to C(002), C(100), and C(101) were quite broad, and the peak found at 43.0° was not split into C(100) and C(101) peaks. The diffraction angles of the C(002) peaks were 25.9° and 28.0° for the unactivated and K$_2$CO$_3$-activated adsorbents, respectively. This indicates that the spacing between the carbon layers in the two adsorbents was different. In addition, buckwheat is known to contain various minerals such as K, Mg, Ca, Fe, Mn, and Na [19], for which the corresponding diffraction peaks were not obvious in the XRD patterns, indicating that the products containing these minerals were removed during the carbonization process and/or subsequent treatment.
Figure 3. Powder XRD patterns of unactivated adsorbent (red) and K$_2$CO$_3$-activated adsorbent (purple).

Figure 4 shows the FT-IR spectra of the unactivated and K$_2$CO$_3$-activated adsorbents. Due to the strong infrared absorption of carbon, the background absorption is very significant; thus, it is quite difficult to clearly identify the intrinsic peaks of surface functional groups. The absorption peaks around 1000–1700 and 3400–4000 cm$^{-1}$ were distinguished by careful background subtraction and indicate the presence of O–H and C=O bonds in surface hydroxyl and/or carboxyl groups. The band around 1500–1700 cm$^{-1}$ also corresponds to the aromatic C=C stretching mode. The band around 1000–1300 cm$^{-1}$ corresponds to a C–O stretching mode of carboxyl groups and/or surface hydroxyl groups. These structures are commonly found in activated carbon. Other unusual features were not confirmed.

Figure 5 shows SEM images of the unactivated and K$_2$CO$_3$-activated adsorbents. The surface morphology of these two adsorbents is quite different. Individual particles of the unactivated adsorbent have relatively smooth surfaces (Figure 5A), whereas those of the K$_2$CO$_3$-activated adsorbent are highly porous (Figure 5B). This result indicates that K$_2$CO$_3$ activation effectively created pores on the surface of the buckwheat hull-derived adsorbent. As can be seen in Figure 5B, the pores created are mainly classified as mesopores and/or macropores.
The amount of adsorbed iodine is listed in Table 1. This measurement method is based on a modified Japanese industrial standard [20]. The approximate specific surface area of a sample can be calculated from the amount of adsorbed I$_2$; the surface area occupied by an I$_2$ molecule is 0.40 nm$^2$. Assuming that I$_2$ covers the adsorbent surface to form a molecular monolayer, the approximate specific surface area, $SSA$ (m$^2$/g), is given by Equation (1). Here, $n$ is the amount (number) of the adsorbed I$_2$ molecules and $w$ (g) is the weight of the sample adsorbent.

$$SSA = \frac{n \times (0.40 \times 10^{-18})}{w}$$

(1)

| Sample                  | $m_{I_2ads}$/mg/g | $m_{MBads}$/mg/g |
|-------------------------|-------------------|------------------|
| Unactivated             | 250               | 4                |
| K$_2$CO$_3$-activated    | 1300              | 130              |

Note that I$_2$ also exists in the form of I$_3^-$ under the presence of I$^-$ (I$_2$ + I$^-$ $\rightleftharpoons$ I$_3^-$), so the calculated $SSA$ are estimates. The surface area occupied by an I$_3^-$ ion is different from that of an I$_2$ molecule, but the $SSA$ obtained here have been calculated using the surface area occupied by an I$_2$ molecule (0.40 nm$^2$). The $SSA$ was found to be significantly larger following activation with K$_2$CO$_3$. The $SSA$ of unactivated and K$_2$CO$_3$-activated adsorbents were calculated to be 240 m$^2$/g and 1200 m$^2$/g, respectively. This increase was caused by the infiltration of K$_2$CO$_3$ into the hull powder surface, followed by the removal of products formed from K$_2$CO$_3$ (K, K$_2$O) after carbonization [21], which generated pores on the adsorbent surface. These results are consistent with the SEM observations presented above. The amount of adsorbed methylene blue is also listed in Table 1. This measurement method is also based on a modified Japanese industrial standard [20]. The adsorption capacity for methylene blue was also found to increase significantly after activation, indicating that the mesopores capable of accepting the methylene blue molecule grew markedly during activation. By contrast, the unactivated adsorbent exhibited a very low methylene blue adsorption capacity. This result indicates that carbonization without activation does not result in effective mesopore formation. The kinetic diameters of I$_2$, methylene blue, and toluene were 0.4 nm, 1.4 nm, and 0.6 nm, respectively. Therefore, mesopores capable of accepting methylene blue are also capable of accepting toluene. Moreover, the kinetic diameters of I$_2$ and toluene are quite similar, suggesting that micropores capable of accepting I$_2$ also contribute to the toluene adsorption. In this respect, the $SSA$ shown above correlates to the toluene uptake. The difference in toluene concentration decrement observed using the two adsorbents (see Figure 6) qualitatively correlates to their $SSA$.
Figure 6. Toluene concentration as a function of time during (A) 120 min and (B) 24 h of observation for unactivated adsorbent (red) and K$_2$CO$_3$-activated adsorbent (purple).

3.2. Removal of Gaseous Toluene from Air

Figure 6a shows the toluene concentration versus time for the unactivated and K$_2$CO$_3$-activated adsorbents. The initial concentration was approximately 230 ppm. When the adsorbents were in contact with air containing toluene, the toluene concentration decreased. A comparison of the observed concentration changes reveals a clear difference between the unactivated and activated adsorbents. For the unactivated adsorbent, the concentration decreased with time but it became constant at approximately 180–170 ppm. This result indicates that the amount of adsorbed toluene became constant after approximately 60 min of exposure. In contrast, for the K$_2$CO$_3$-activated adsorbent, the decrease in toluene concentration was more significant, and the concentration continued to decrease during the entire observation (0–120 min). This result indicates that the toluene adsorption did not reach equilibrium during the observation; the amount of toluene adsorbed on the activated adsorbent at equilibrium may have been much larger than that on the unactivated adsorbent. Therefore, the toluene adsorption capacity of the adsorbents derived from buckwheat hull was improved by activation with K$_2$CO$_3$. This increase in the toluene adsorption capacity is consistent with the increase in SSA and the introduction of mesopores via K$_2$CO$_3$ activation that were mentioned above.

To investigate whether the K$_2$CO$_3$-activated adsorbent could completely remove toluene under the conditions used here, the toluene concentration was observed for 24 h. Figure 6b shows the toluene concentration versus time during 24 h of observation. The K$_2$CO$_3$-activated adsorbent is clearly capable of adsorbing a large amount of gaseous toluene from the air. The final toluene concentration after 24 h of adsorption was very low and only a trace amount of toluene was detected. In contrast, when the unactivated adsorbent was used, a considerable amount of toluene remained (160 ppm), even after adsorption for 24 h.

The initial toluene concentration used (200–240 ppm) was much higher than the occupational threshold limit value set by the American Conference of Governmental Industrial Hygienists (20 ppm) [22]. The K$_2$CO$_3$-activated adsorbent was able to lower the toluene concentration below the threshold limit value under the experimental conditions used here. These results indicate that K$_2$CO$_3$ activation is effective for the preparation of adsorbents to be used in the thorough removal of volatile toluene. In the present study, 0.2 g of the adsorbent was used to track the time course of toluene concentration. Maximum toluene adsorption capacity was not immediately determined on the basis of the present observation because the final toluene concentration achieved under the present condition was almost zero. The determination of the maximum adsorption capacity will be a subject for future work.
4. Conclusions

The toluene adsorption capabilities of unactivated and K$_2$CO$_3$-activated carbonaceous adsorbents derived from buckwheat hull were compared. The K$_2$CO$_3$-activated adsorbent adsorbed much more toluene than the unactivated adsorbent. The enhanced toluene removal capacity was explained by the differences in specific surface areas and mesopore structures between the unactivated and K$_2$CO$_3$-activated adsorbents. These results showed that chemical activation via K$_2$CO$_3$ was effective in improving the toluene adsorption capacity of the buckwheat hull-derived carbon adsorbent. In the present case, the K$_2$CO$_3$-activated adsorbent was able to reduce the relatively high initial toluene concentration (220 ppm) to almost zero after 24 h. Although the maximum adsorption capacity was not precisely determined in these experiments, an impressive change in the adsorption capacity via K$_2$CO$_3$ activation was confirmed. This is an important finding with regard to the utilization of buckwheat hull as a raw material for activated carbon.

In the present case, the main adsorption mechanism of toluene is the simple physisorption of toluene molecules on the carbon surface; no chemical reactions between toluene and the adsorbent surface occurred in this case. Therefore, adsorbates other than toluene, such as benzene, xylene, and water vapor, may also be competitively adsorbed on the adsorbent prepared in this work. In addition, temperature also affects the adsorption behavior of toluene. The selectivity of toluene adsorption, the effect of humidity, and the effect of temperature on the adsorption capacity of toluene are subjects that remain in need of further study.

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