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Benzene Adsorption from Dry and Humid Air on Activated Carbons from Japanese Cypress Wood Prepared by CO₂ and K₂CO₃ Activation

Andrei Veksha, Md. Azhar Uddin, Eiji Sasaoka, and Yoshiei Kato

Abstract—Activated carbons (ACs) were prepared by physical (CO₂) and chemical (K₂CO₃) activation of wood (Japanese cypress). Porous properties of the ACs, such as BET surface area and micropore volumes $V_{mic}(N_2)$ and $V_{mic}(CO_2)$, were characterized by N₂ and CO₂ adsorption isotherms. Short activation times caused formation of micropores with sizes smaller than 0.7 nm, which had a restricted access for N₂ molecules. As a result, the ACs prepared through short activation time exhibited $V_{mic}(N_2)$ smaller than $V_{mic}(CO_2)$. The ACs were used for benzene adsorption at 5 ppmv concentration and relative humidities (RHs) of 0, 50 and 70 %. The largest benzene adsorption capacities at these RHs were exhibited by the AC prepared with K₂CO₃ activation at 800 °C. At RH 0 %, the benzene adsorption on the ACs was governed by $V_{mic}(N_2)$, when it was smaller than $V_{mic}(CO_2)$, and by $V_{mic}(CO_2)$, when it was smaller than $V_{mic}(N_2)$. The lower determination coefficients for the relationship between benzene uptake by the ACs and the volume of micropores at RHs 50 and 70 % compared to RH 0 % were attributed to the increased influence of surface chemistry of the ACs in the presence of moisture. In particular, the benzene adsorption at RHs 50 and 70 % was detrimentally affected by larger amounts of ash and surface oxygen groups.

Index Terms—Activated carbon, Benzene adsorption, CO₂ activation, K₂CO₃ activation, Relative humidity.

I. INTRODUCTION

Activated carbons (ACs) are widely used adsorbents for the removal of volatile organic compounds (VOCs) from air. Adsorption on ACs depends on their porous and chemical characteristics, such as micropore volume, pore size distribution, surface groups, oxygen and nitrogen contents [1]–[3]. Furthermore, the removal efficiency of ACs is influenced by the type of adsorbate, concentration and the presence of moisture [4].

ACs can be produced from any carbonaceous material either by physical activation with steam, CO₂ and air or by chemical activation with various inorganic agents (H₃PO₄, ZnCl₂, KOH etc.). The properties of the resulting carbons are affected by both the initial precursor and the activation conditions [5].

In the current study, Japanese cypress wood was used as a precursor for the preparation of ACs by physical (CO₂) and chemical (K₂CO₃) activation. The ACs were tested for

benzene adsorption from air at low concentrations (5 ppmv) in dry and humid conditions. Benzene is a common VOC used in organic synthesis and is a trace component emitted from paints, exhaust gases of vehicles and cigarette smoke. Recently, the development of adsorbents for benzene removal has been gaining attention due to benzene's toxicity and carcinogenic properties. The purposes of this study were to a) investigate the effects of temperature and time during K₂CO₃ activation on porosity formation and compare porous properties of the ACs prepared by chemical and physical activation and b) determine the influence of porous and surface chemical properties of the ACs on benzene adsorption in the absence and presence of moisture.

II. EXPERIMENTAL

A. Preparation of the ACs

ACs were prepared from Japanese cypress wood. Wood chips were ground using a mixer, pelletized by pressing at room temperature and dried in oven at 110 °C for 24 h. In case of physical activation, the pellets were heated with a temperature increment of 10 °C/min to 900 °C, carbonized for 1 h in nitrogen (300 ml/min) and then activated with CO₂ (300 ml/min) for 0.25, 0.5 and 1 h. The prepared ACs were denoted as HP0.25, HP0.5 and HP1 (numbers in the name correspond to the activation time).

For chemical activation, dried pellets were carbonized at 550 °C in nitrogen (300 ml/min) for 1 h. The char was ground and sieved in order to select particles with sizes between 1.2 and 1.7 mm. The char particles were mixed with K₂CO₃ in a ratio of 1:3 by weight. Deionized water was added to the mixture for dissolution of K₂CO₃ (15 ml of water per 1 g of char). The char was soaked in K₂CO₃ solution for 1 h at room temperature, heated in oven at 55 °C for 12 h and then at 110 °C for 24 h. The dried particles were placed into a reactor, heated with a temperature increment of 10 °C/min to 700, 800 and 900 °C and activated for 0.75 h in nitrogen (300 ml/min). The activation at 900 °C was also carried out for 1.5 and 4 h. After activation, the samples were boiled with 3 N HCl for 0.5h, washed with cold deionized water until a neutral pH and the absences of chloride-anion in the filtrate were achieved followed by drying at 110 °C. The dried ACs were treated with nitrogen (300 ml/min) at 900 °C for 1 h in order to reduce the amount of surface functional groups. The prepared ACs were denoted as I0.75, J0.75, K0.75, K1.5 and K4 (the letters I, J and K corresponded to the activation temperatures 700, 800 and 900 °C, respectively, and the numbers in the name to the activation time).

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TABLE 1: PROPERTIES OF THE ACS PREPARED BY ACTIVATION WITH CO₂ AND K₂CO₃

Sample	Activation conditions			Burn-off [%]	S _g [m ² /g]	V _{mic} (N ₂) [ml/g]	V _{mes} [ml/g]	V _{mic} (CO ₂) [ml/g]	Ash [%]
	Activator	Temperature [°C]	Time [h]						
HP0.25	CO ₂	900	0.25	21	521	0.27	0.02	0.33	0.8
HP0.5	CO ₂	900	0.5	37	609	0.31	0.03	0.34	1.4
HP1	CO ₂	900	1	70	852	0.43	0.05	0.35	4.5
I0.75	K ₂ CO ₃	700	0.75	25	551	0.29	<0.01	0.41	<0.1
J0.75	K ₂ CO ₃	800	0.75	27	816	0.43	<0.01	0.50	<0.1
K0.75	K ₂ CO ₃	900	0.75	35	865	0.45	0.02	0.41	0.9
K1.5	K ₂ CO ₃	900	1.5	39	917	0.47	0.05	0.40	1.9
K4	K ₂ CO ₃	900	4	69	1317	0.64	0.16	0.30	1.5

A. Characterization of the ACs

The ACs were characterized by burn-off, ash content, N₂ (-196 °C), CO₂ (25 °C) and H₂O (25 °C) adsorption isotherms. The burn-off was determined as the ratio of weight loss of carbon during activation to the initial weight of char. BET surface areas S_g were measured with N₂ adsorption isotherms in the relative pressure range of 0.04-0.31. The micropore volumes V_{mic}(N₂) and V_{mic}(CO₂) were calculated using Dubinin-Radushkevich equation and liquefied gas densities of 0.81 and 0.85 g/ml, respectively [6]. Micropore volume V_{mic}(CO₂) characterizes the adsorption in micropores smaller than 0.7 nm whereas V_{mic}(N₂) corresponds to the adsorption in micropores with sizes up to 2 nm. Mesopore volume V_{mes} was found from nitrogen adsorption as the difference of total pore volume calculated at the relative pressure P/P_s = 0.96 and micropore volume V_{mic}(N₂).

Prior to ash content determination, the dried ACs were placed in crucibles of known weight and heated at 110 °C for 2 h in order to remove the residual moisture. After measuring the weight, the crucibles with the ACs were placed in a muffle furnace and heated in air at 650 °C for 3 h. The crucibles were cooled to room temperature in a desiccator and then weighed. The complete burning of the samples was confirmed by their constant weight after the additional heating in the furnace at 650 °C for 1 h.

Temperature programmed desorption (TPD) method was used to characterize surface oxygen groups of the ACs. The surface oxides decompose upon heating releasing CO₂ and CO. The amounts of evolved CO₂ and CO from the ACs were monitored by quadrupole mass spectrometer as described previously [7].

B. Benzene Adsorption Experiments

The experimental setup for benzene adsorption has been described elsewhere [7]. Benzene adsorption was carried out from air at 25 °C and relative humidities (RHs) of 0, 50 or 70 %. The total gas flow was 250 ml/min and contained 5 ppmv of benzene. The tube reactor with inner diameter of 4 mm was loaded with 0.05 ml of dried AC (particle size of 0.35 mm, carbon bed height of 4 mm). The concentration of benzene was monitored by gas chromatograph equipped with flame ionization detector (without chromatographic column). Benzene adsorption capacities of the ACs were calculated when the concentration of benzene in outlet gas reached 100 % of the concentration in inlet gas using breakthrough curves.

III. RESULTS AND DISCUSSION

A. Properties of the ACs

Table 1 illustrates the characteristics of the ACs. The burn-offs of chemically activated samples increased with activation temperature from 25 % for I0.75 to 35 % for K0.75 and longer activation time from 35 % for K0.75 to 69 % for K4. The enhancement of K₂CO₃ activation with higher temperature and longer time was reported previously, although the preparation procedures were not exactly the same [8], [9]. Activation of wood with CO₂ was more intensive than with K₂CO₃, which could be seen from the shorter activation time required for HP0.25, HP0.5 and HP1 to reach the burn-offs that are similar to those of K0.75, K1.5 and K4. However, the chemically activated samples K0.75, K1.5 and K4 exhibited larger surface areas than ACs prepared with CO₂ activation. The increase in temperature from 700 °C to 800 °C resulted in significant enhancement of surface area in J0.75 compared to I0.75 whereas raising the temperature to 900 °C produced only slight development of surface area in K0.75. The micropore volumes V_{mic}(N₂) of the ACs prepared with K₂CO₃ activation at 800 °C and 900 °C were larger than at 700 °C. Similarly to surface areas, micropore volumes V_{mic}(N₂) of the ACs prepared at 900 °C increased with longer activation time. Additionally, the longer K₂CO₃ activation at 900 °C resulted in the formation of larger amount of mesopores (K4).

The development of micropore volume V_{mic}(CO₂) was different from V_{mic}(N₂). The highest V_{mic}(CO₂) was exhibited in J0.75 prepared at 800 °C (Table 1). Since V_{mic}(N₂) and V_{mes} of K0.75 increased compared to J0.75, the decrease in V_{mic}(CO₂) of K0.75 could be partially attributed to the widening of micropores with sizes smaller than 0.7 nm. Apparently, the micropore widening could also be a reason of the decrease in V_{mic}(CO₂) of K0.75, K1.5 and K4 with longer activation time. The micropore volumes V_{mic}(N₂) of HP0.25, HP0.5, I0.75 and J0.75 were smaller than V_{mic}(CO₂), indicating that some of the micropores with sizes smaller than 0.7 nm were not accessible to N₂ molecules.

B. Benzene Adsorption at RH 0 %

Fig. 1 depicts the breakthrough curves of the ACs. The breakthrough curves obtained at RH 0 % were used for calculation of adsorption capacities of the ACs (Fig. 2). At RH 0 %, longer CO₂ activation improved benzene adsorption on the ACs in the increasing order of HP0.25, HP0.5 and HP1. However, K₂CO₃ activation affected adsorption of benzene adversely and adsorption capacities of K0.75, K1.5 and K4

decreased with longer activation time. Adsorption on I0.75 was similar to HP0.5, suggesting no benefits of chemical activation at 700 °C compared to physical activation. On the contrary, J0.75 and K0.75 prepared at 800 °C and 900 °C, respectively, exhibited the largest adsorption capacities at RH 0 % among the tested samples. The adsorption capacities of the ACs at RH 0 % plotted against their micropore volumes $V_{mic}(CO_2)$ and $V_{mic}(N_2)$ are shown in Fig. 3a and 3b, respectively. Low determination coefficients (R^2) indicated weak correlations between benzene uptake by the ACs and their $V_{mic}(CO_2)$ and $V_{mic}(N_2)$. These results differed from a previous report, which showed good correlation between benzene adsorption capacities of the ACs and the amount of micropores [10]. However, this report was on the adsorption of benzene at concentration 200 ppmv, which is higher than in our study.

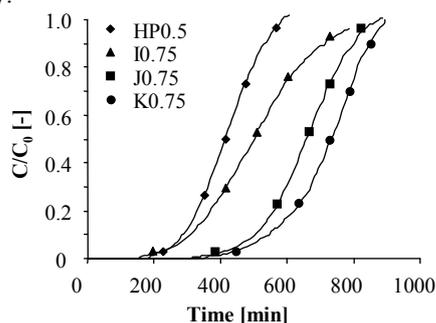


Fig. 1. Breakthrough curves of the ACs (25 °C, RH 0 %).

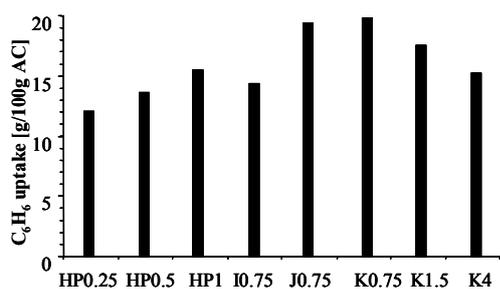


Fig. 2. Benzene adsorption capacities of the ACs at RH 0 %.

The decrease in benzene uptake by K1.5 and K4 compared to K0.75 could be associated with the decrease in $V_{mic}(CO_2)$, i. e. with the reduced amount of micropores smaller than 0.7 nm (Table 1). The enhancement of $V_{mic}(N_2)$ of these ACs did not improve their benzene adsorption. In contrast, the benzene adsorption on HP0.25, HP0.5 and HP1 increased from 12.1 g/100 g of AC on HP0.25 to 15.5 g/100 g of AC on HP1, while their $V_{mic}(CO_2)$ were changed only from 0.33 to 0.35 ml/g, respectively (Table 1). On the other hand, these ACs exhibited an increase in $V_{mic}(N_2)$ from 0.27 ml/g in HP0.25 to 0.43 ml/g in HP1, suggesting that the enhancement of benzene adsorption was contributed by the development of $V_{mic}(N_2)$ rather than $V_{mic}(CO_2)$. One possible explanation. For this phenomenon is that benzene could not access some of the micropores smaller than 0.7 nm in HP0.25 and HP0.5. The limited access into micropores could also be the reason for lower benzene adsorption on I0.75 compared to K0.75. According to Table 1, both ACs had same $V_{mic}(CO_2)$, but I0.75 exhibited smaller $V_{mic}(N_2)$ than K0.75. Finally, the benzene uptake at RH 0 % by J0.75 and K0.75 suggested that these ACs had similar volumes of micropores suitable for benzene adsorption, which would be possible if adsorption of benzene occurred only in micropores accessible for both CO_2

and N_2 gases. Based on the benzene uptake by the ACs and their porous properties, it was concluded that the benzene adsorption on the ACs was governed by $V_{mic}(N_2)$ when $V_{mic}(N_2)$ was smaller than $V_{mic}(CO_2)$ and by $V_{mic}(CO_2)$ when $V_{mic}(CO_2)$ was smaller than $V_{mic}(N_2)$. In other words, the amount of “effective” micropores responsible for benzene adsorption was limited by the lowest value of two micropore volumes deduced from CO_2 and N_2 adsorption (for instance, 0.27 ml/g in HP0.25 and 0.41 ml/g in K0.75). Fig. 3c illustrates a good correlation between benzene uptake at RH 0 % and the “effective” volumes of micropores V_{mic} (“effective”) of the ACs ($R^2 = 0.89$).

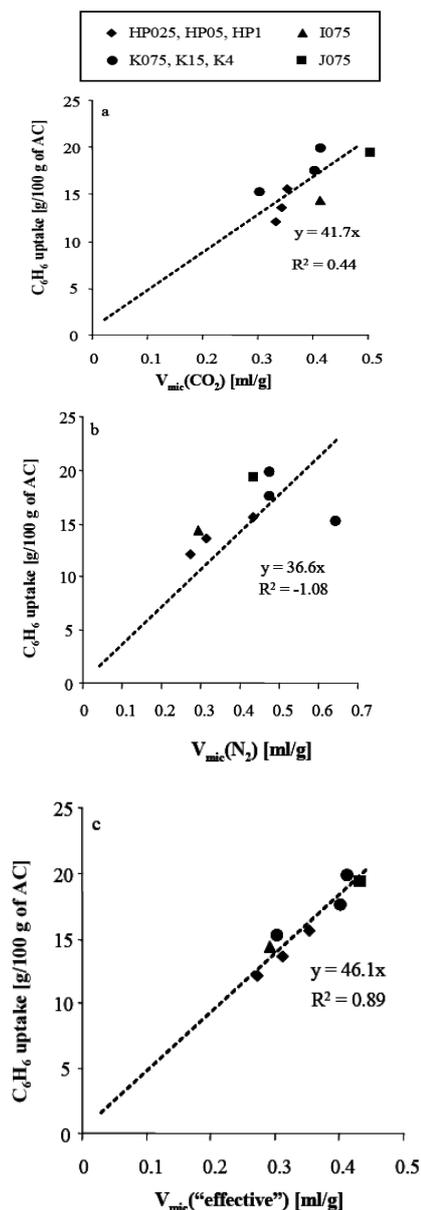


Fig. 3. Correlation between benzene uptake at RH 0 % and a) $V_{mic}(CO_2)$; b) $V_{mic}(N_2)$ and c) V_{mic} (“effective”)

C. Effect of Moisture on Benzene Adsorption

Benzene uptake by the ACs in the presence of moisture is shown in Fig. 4. At RH 70 %, J0.75 prepared with K_2CO_3 at 800 °C exhibited the largest benzene uptake among the tested ACs (Fig. 4a). The benzene uptake at RHs of 50 % and 70 % suggested that longer activation with CO_2 and K_2CO_3 did not favor benzene adsorption in the presence of moisture. The

adsorption of benzene on HP0.25, HP0.5 and HP1 remained almost the same whereas the adsorption on K0.75, K1.5 and K4 dropped with longer activation time. At RH 50 %, the adsorption of benzene on the ACs, except K4, exhibited the similar trend as at RH 70 % (Fig. 4b). The higher benzene uptake by the ACs at RH 50 % compared to RH 70 % could be attributed to the weaker competition by water for the adsorption sites.

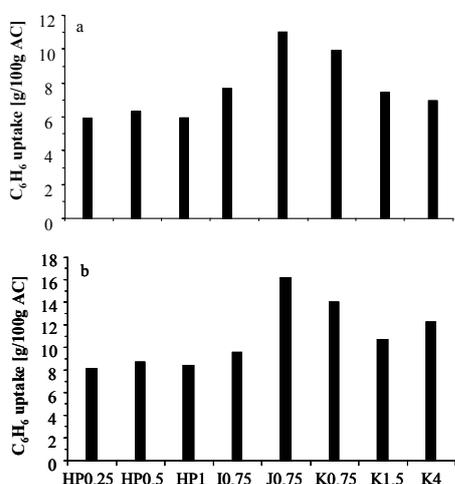


Fig. 4 Benzene adsorption on the ACs at RH a) 70 % and b) 50 %.

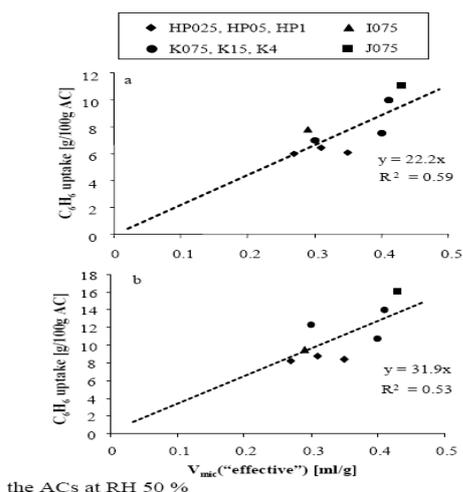


Fig. 5. Relationship between benzene uptake at a) RH 70 % and b) 50 % and "effective" micropore volumes of the ACs.

The relationship between benzene adsorption capacities of the ACs at RHs 50 and 70 % and "effective" micropore volumes is shown in Fig. 5. The values of R^2 for the correlations of benzene adsorption obtained at RHs 50 and 70 % and "effective" micropore volumes were lower than at RH 0 %. These results suggested that influence of other factors on benzene adsorption increased in the presence of moisture. Fig. 6 depicts the initial parts of water adsorption isotherms of the ACs prepared by K_2CO_3 activation ($P/P_s < 0.2$). At low relative pressures, water uptake is mainly attributed to interactions of water molecules with polar sites on the surface of ACs [11]-[13]. K1.5 exhibited the larger water uptake than the other ACs, which was partially contributed by the higher ash content (Table 1). Previously, it was reported that inorganic impurities on the surface of ACs may alter water adsorption [14].

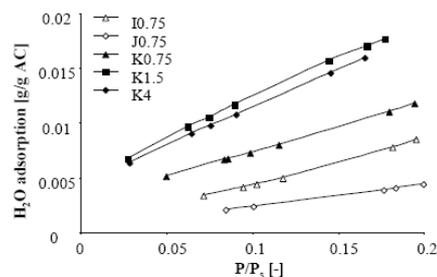


Fig. 6. Water adsorption on the ACs prepared by K_2CO_3 activation.

TABLE 2: RATIOS OF BENZENE UPTAKE BY THE ACs AT RHs 50 AND 70 % TO THEIR UPTAKE AT RH 0 %

Sample	$V(C_6H_6)_{70\%} / V(C_6H_6)_{0\%}$	$V(C_6H_6)_{50\%} / V(C_6H_6)_{0\%}$
HP0.25	0.50	0.68
HP0.5	0.45	0.64
HP1	0.33	0.54
I0.75	0.54	0.66
J0.75	0.57	0.83
K0.75	0.50	0.71
K1.5	0.43	0.61
K4	0.46	0.81

The amount of surface oxides in the ACs was determined by CO_2 and CO TPD. The TPD profiles of the ACs are shown in Fig. 7. The amounts of CO_2 and CO evolved from K1.5 were larger than from K0.75 and K4. The surface oxides favor water adsorption, due to the formation of hydrogen bonds with water molecules [15]. Therefore, larger water uptake by K1.5 at $P/P_s < 0.2$ could also be caused by greater amounts of surface oxygen groups. Table 2 illustrates the ratio of benzene adsorption on the ACs from humid air to the adsorption in dry conditions. Among the ACs prepared by K_2CO_3 activation at $900^\circ C$, K1.5 showed the lowest ratios of benzene adsorption at RHs 50 and 70 % to the adsorption at RH 0 %, which was consistent with the pronounced hydrophilic character of this AC suggested by water adsorption isotherm (Fig. 6).

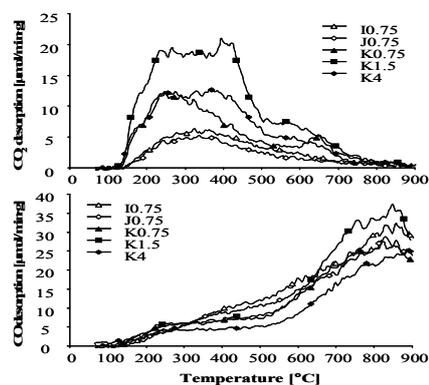


Fig. 7. CO_2 and CO TPD profiles of the ACs prepared by K_2CO_3 activation.

Water adsorption increased on K0.75 compared to J0.75 (Fig. 6), which was in agreement with the increase in the amounts of ash and surface oxygen groups evolved as CO_2 (Table 1 and Fig. 7). Nevertheless, the amount of CO evolved from K0.75 at $300-600^\circ C$ was slightly lower than from J0.75. Water adsorption on I0.75 at $P/P_s < 0.2$ was higher than on J0.75 (Fig. 6), despite the low ash contents and similar TPD

profiles of both ACs (Table 1 and Fig. 7). The decomposition of surface oxides evolved as CO was incomplete at 900 °C (Fig. 7). It was reported that the complete removal of ethers and carbonyls may require heating up to 1100 °C [16]. It is possible that the amount of surface oxides that remained intact after heating up to 900 °C during TPD was larger in I0.75. The higher ratios of benzene adsorption on J0.75 at RHs 50 and 70 % to the adsorption at RH 0 % compared to those of I0.75 and K0.75 were consistent with the lower water uptake at $P/P_s < 0.2$ (Table 2 and Fig. 6). The ratio of benzene adsorption on K4 at RH 70 % to the adsorption at RH 0 % was considerably lower than that of J0.75. However, the ratio for K4 at RH 50 % was almost as high as for J0.75 (0.81 and 0.83, respectively). This phenomenon could not be attributed solely to the hydrophilic character of the ACs and requires further investigation.

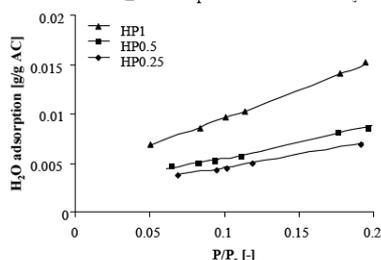


Fig. 8. Water adsorption on the ACs prepared by CO₂ activation.

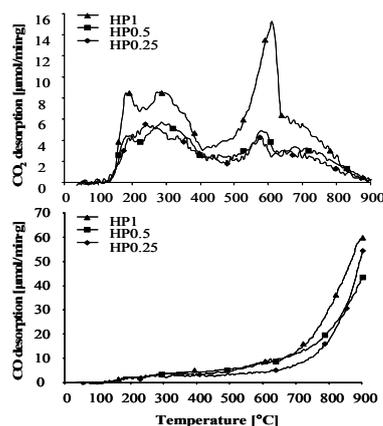


Fig. 9. CO₂ and CO TPD profiles of the ACs prepared by CO₂ activation

According to Table 2, the tolerance towards moisture during benzene adsorption decreased in the ACs prepared by CO₂ activation in order of HP0.25 > HP0.5 > HP1. The water adsorption at $P/P_s < 0.2$ suggested that it could be caused by the enhancement of hydrophilic character of their surfaces (Fig. 8). The longer activation resulted in the accumulation of ash components in the ACs (Table 1). Additionally, HP1 had larger amounts of evolved CO₂ and CO during TPD compared to HP0.25 and HP0.5 (Fig. 9).

IV. CONCLUSION

Adsorption of benzene from air was carried out on the ACs prepared from Japanese cypress wood by CO₂ and K₂CO₃ activation. Development of porosity during activation depended on the activation method, temperature and time. The chemical activation produced the ACs with larger

micropore volumes than physical activation. At RH 0 %, mainly “effective micropores” smaller than 0.7 nm, in which both CO₂ and N₂ were adsorbed, were suitable for benzene adsorption. At RHs 50 and 70 %, besides the micropore volumes, adsorption of benzene was also affected by the surface chemistry of the ACs. The increase in the amounts of ash and surface oxygen groups decreased benzene uptake by the ACs in the presence of moisture.

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