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Adsorption Characteristics of Acetaldehyde on Activated Carbons Prepared from Corn-Based Biomass Precursor

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The ACs (R-1/2 and R-1/4) having two different textual and chemical properties are prepared from corn-based biomass precursor and evaluated together with a wood-based activated carbon (WAC) at room temperature using a gas chromatograph. The results obtained from the correlation studies indicate that the pore size distribution (below 8 Å) and the relatively lower energetic heterogeneity of ACs on acetaldehyde adsorption are considerable factors rather than that of a specific surface area and surface chemistry. The adsorption equilibrium of ACs is well correlated with the Sips equation. The pseudo second-order equation was better in describing the ACs' adsorption kinetic of acetaldehyde.

Keywords acetaldehyde; activated carbon; adsorption; corn grain; water vapor

INTRODUCTION

With growing concerns of an indoor decoration, the concentration of aldehyde compounds accumulated in indoor air has been one of the major issues and needs to be removed. Especially, one of the main aldehyde compounds namely "acetaldehyde" is recognized as a toxic material. Acetaldehyde is usually emitted by decorated materials, cigarette smoke, paint, polymerized plate, binder, etc. (1). The excessive exposure of the high acetaldehyde concentration to the human body causes serious symptoms such as headache, nausea, DNA damage, and abnormal muscle development (2).

There are several methods to remove acetaldehyde compounds in air such as oxidation, absorption/adsorption, condensation, and biofiltration. Among them, the adsorption process has been known as the most effective method to remove acetaldehyde because of its high selectivity, process simplicity, and safety (3).

Activated carbon (AC) has been widely used in the adsorption process as one of the most potential adsorbents due to the proper properties such as large surface area, broad pore size distribution, microporocity, and thermal stability. In general, the ACs can be prepared from various precursors such as biomasses, coals, resins, synthetic polymers, petroleum coke, phenolic resins, peat, etc.

Biomass precursors have been used for the preparation of highly porous ACs owing to the properties of high carbon and low inorganic contents, relatively cheaper, and an abundant amount (4–6). There are numerous biomass sources such as wheat, misconstrues, pinecone, coconut shell, cotton residues, almond shells, cotton, olive stones, cherry stones, corn straw, corn hulls, corn stove, corn cob, rice husk, and rice hull. These sources have been utilized in various fields (6).

In this work, the main objectives were to prepare the novel ACs having different physical properties and surface chemistry from corn grains and to assess their adsorption capacities of acetaldehyde compared to a commercial AC.

EXPERIMENTAL

AC Characterization

Three ACs having the different physical and chemical properties were selected in this study. The corn grain was chosen for the preparation of AC. The procedures are as follows: The corn grain was grinded and dried at 60°C for 24 h. The powdered sample was carbonized in a horizontal furnace up to 600°C and kept at 600°C for 1 h under nitrogen atmosphere. Then, the samples (R-1/2, R-1/4) physically mixed with varying the mass ratio between the carbonized char and chemical activation agent (potassium hydroxide, KOH) were activated up to 800°C and maintained at 800°C for 1 h under nitrogen atmosphere. The prepared samples were washed by using the distilled hot water and

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were dried at 120°C for 24 h in order to remove the remaining moistures (3–5). The commercial wood-based activated carbon (WAC) was chosen for comparison of the adsorption capacity of acetaldehyde and water vapor.

The physical properties of all samples were determined by nitrogen adsorption/desorption isotherms at 77 K (Micrometrics ASAP 2020). The specific surface area was calculated by the basis of BET theory in the relative pressure range of 0.01–0.05. The total pore volume (V_T) was calculated from the nitrogen adsorption data at a relative pressure range of 0.99 and the total pore width was measured using the following equation ($4V_T/S_{BET}$). The Dubinin-Radushkevich (DR) equation was used for micropore volume (V_{DR}). The pore size distribution (PSD) and the pore volume of samples were evaluated by the density functional theory (DFT) (3–5).

The surface functional groups of all samples were examined using the Fourier transform infrared spectroscopy (FT-IR, BIO-RAD, FTS-175C) and the X-ray photoelectric spectroscopy (XPS, Multi lab 2000/Thermo electron corporation, England). In the case of FT-IR analysis, the activated carbons were mixed with KBr at a mass ratio of AC/KBr (1:30) to form into a pellet (7,8).

Water Vapor Adsorption

Adsorption experiments of water vapor on all samples were carried out in an isolated chamber which kept at 50% relative humidity at 30°C. About 50 mg of activated carbons was put onto a weight machine (AR2140, OHAUS/US) in the isolated chamber, respectively. Then, the weight change was recorded as a function of time. When the weight change of a sample was maintained within the range of about ±0.1 mg, the adsorption equilibrium state was assumed.

Acetaldehyde Adsorption Using Gas Chromatograph

Adsorption of acetaldehyde (Stem Supply, SA, Australia) was studied using a cuboids stainless (Top-face glass) airtight reactor with a total volume of 3.8 L. The reactor had three rubber openings for the injection of acetaldehyde, connecting to an air pump to ensure mixing of air inside the reactor and sampling aperture connected to a gas chromatograph (GC)/flame ionization detector (FID) (HP 5890 series II, Wilmington, USA) to measure acetaldehyde concentration variations. The reaction took place at room temperature of 25°C around 60 min.

About 0.5 g of activated carbon was sprinkled in a glass Petri-dish (9 cm of diameter) in the center of the reactor. After closing the reactor door, the adsorption capacities on all samples were measured. With varying the initial acetaldehyde concentration acetaldehyde was injected through the injection cavity using an airtight syringe. The concentration of acetaldehyde was recorded at fixed 3.8 min intervals.

RESULTS AND DISCUSSION

Physical and Chemical Properties of ACs

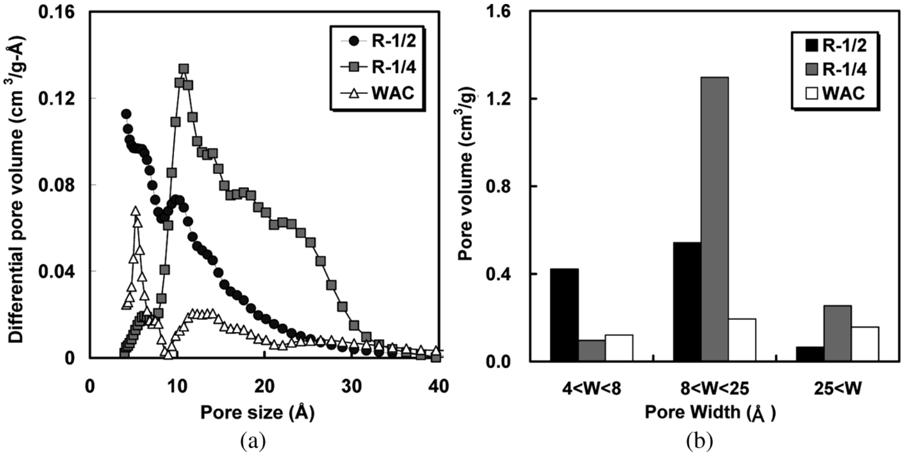
The textual properties and surface functional groups of AC have highly influenced on organic compound adsorption. The textural properties of the ACs were determined by N_2 adsorption/desorption isotherms at 77 K (Table 1). The DFT equation was used to investigate the effect of PSDs of ACs on acetaldehyde adsorption. R-1/4, which is one of the ACs prepared from corn grain, possesses the values of the largest specific surface area and total pore volume. In the contrast of the R-1/4, R-1/2 shows the relatively narrow pore size distribution, mostly in the range of 4–10 Å, as shown in Fig. 1.

The surface functional groups of ACs, especially the surface oxygen functional groups, are measured by FT-IR and XPS. The FT-IR spectra have been used to identify the functionality of AC qualitatively. The XPS has been also used to assess the atomic compositions quantitatively. The FT-IR spectra of ACs were analyzed in the range of 4000–1000 cm^{-1} (Fig. 2). All the ACs showed a couple of absorption peaks at 1375 and 1575 cm^{-1}. The absorption peak at 1375 cm^{-1} was considered as the methyl group (7) and the other at 1575 cm^{-1} was ascribed to the C−C stretching vibrations of polyaromatic C=C (7,8). However, the FT-IR characteristic peaks of all the ACs show the weak intensities due to natural black body of AC. Therefore, the XPS was used for examining the surface binding state and elemental speciation in detail. The XPS spectra have two distinct peaks considered as carbon and oxygen. The elemental surface compositions of graphitic carbon (285 eV), phenolic (286.7 eV), carbonyl (288.4 eV), carboxylic (289.7 eV), and carbonate group (290.7 eV) are listed in Table 2. In the case of ACs prepared from corn grain, the percentage occupied by the oxygen containing groups on the AC surface increases with increasing the mass ratio between the corn grain char and KOH. It is also observed that the percentage of the oxygen containing groups of the R-1/4 is similar with that of the WAC.

TABLE 1
Physical properties of ACs (3–5)

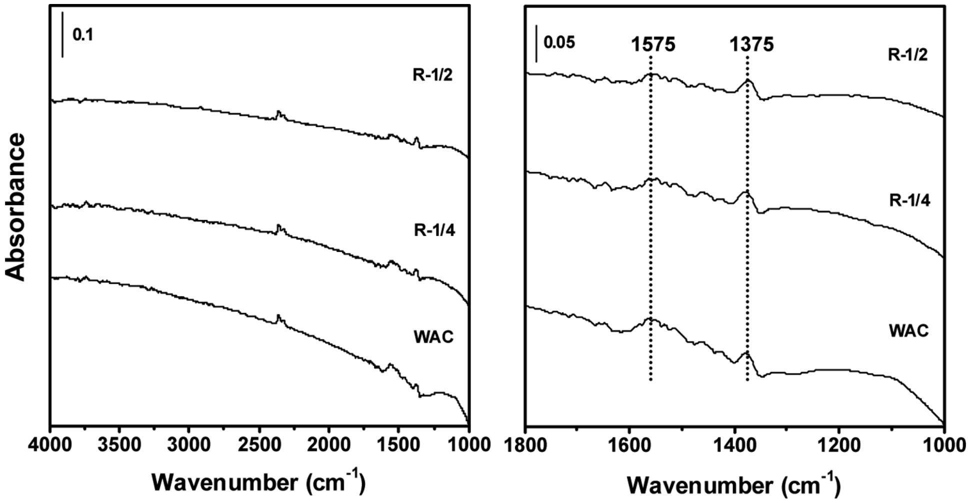
Sample	[a]S_{BET} (m^2/g)	Pore volume (cm^3/g) V_{DR}	V_{DFT}	[b]A.P.W (nm)
R-1/2	2269	0.94	1.03	1.07
R-1/4	3199	1.69	1.65	1.41
WAC	882	0.58	0.47	2.62

[a]BET: Specific surface area (relative pressure range: 0.01–0.05).
[b]Average pore width.



(a) (b)

FIG. 1. The DFT pore size distributions of ACs (3–5).



(a) (b)

FIG. 2. FT-IR spectra of all the ACs in the range of 4000–1000 cm^{-1} (3).

TABLE 2
Distribution of oxygen containing groups on ACs determined by XPS (3–5)

	C1s				
Sample	[a]G.C (%)	C–O– (%)	C=O (%)	O–C=O (%)	Carbonate (%)
R-1/2	80.55	12.85	–	6.59	–
R-1/4	68.15	8.02	10.28	7.22	6.33
WAC	65.09	13.13	3.8	3.38	3.43

[a] Graphitic carbon.

Acetaldehyde Adsorption

The adsorption capacities of acetaldehyde on ACs were assessed using gas chromatograph at room temperature. The adsorption equilibrium isotherms of acetaldehyde are shown in Fig. 3. The R-1/2 shows the highest adsorption capacity compared to the R-1/4 and the WAC. According to the previous work, the adsorption capacity of acetaldehyde ($\sigma = 3.8$ Å) on AC strongly depends on various factors such as the specific surface area, pore structure, pore size distribution, energetic heterogeneity between the adsorbent and the adsorbate, and the oxygen-containing groups on AC (9–13). Thus, the adsorption property correlations among the textual property and the surface chemistry as well as the energetic heterogeneity on acetaldehyde

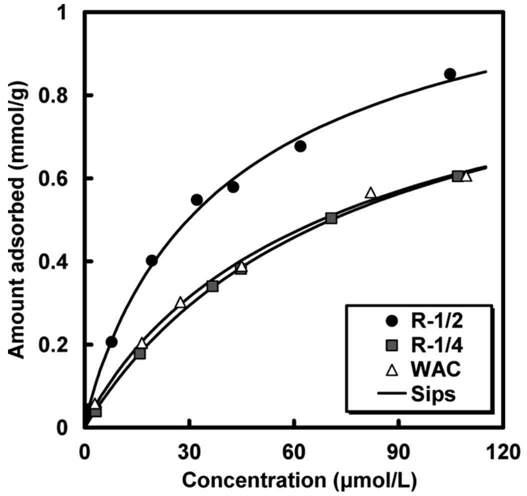


FIG. 3. Adsorption equilibrium isotherms of acetaldehyde on ACs at room temperature.

adsorption were examined to find out the optimum adsorption condition.

In general, it has been known that the acetaldehyde adsorption capacity increases with increasing the specific surface area and pore volume of AC (12,13). In this study, a linear correlation between the specific surface area and the capability of AC on acetaldehyde adsorption is not observed (Table 3). On the other hand, the acetaldehyde adsorption capacity of AC is enhanced with the increment of amount of micropore pore volume (below 8 Å). These results indicate that the adsorption capacity of acetaldehyde on the ACs is subjected to a correlation between the molecule size of acetaldehyde and the pore size of AC. Therefore, it is proven that the pore size distribution (below 8 Å) of the used ACs is strongly predominant on acetaldehyde adsorption.

The oxygen functional groups on the surface of AC usually determine the charge of the surface, hydrophobicity, and the electronic density of the graphene layers (9,12,13). In general, these oxygen containing groups on the surface of AC interact with acetaldehyde (14). Thus, the adsorption capacity of acetaldehyde is influenced by the existing amounts of oxygen groups on the surface of ACs (12,13). However, the R-1/2, which shows the highest adsorption capacity, mostly consists of graphitic carbon (about 80%). Interestingly, the R-1/4 having the similar percentage of oxygen containing group with the WAC shows the similar adsorption capacity with the WAC. It is again confirmed from the correlation studies that the acetaldehyde adsorption on the ACs used is mainly influenced by the pore size distributions below 8 Å.

TABLE 3
Adsorption equilibrium parameters of acetaldehyde on ACs

	Sips			
Sample	q_m	b	n	DN
R-1/2	1.229	0.033	0.893	0.014
R-1/4	1.042	0.013	0.998	0.003
WAC	1.065	0.018	0.920	0.022

The correlation study between the acetaldehyde adsorption capacity and the surface energetic heterogeneity of ACs was investigated. In general, the energetic heterogeneity of a solid surface can be described by the adsorption energy distribution (AED). The AED is calculated by following the integral equation (15–19).

$$\theta(p) = \int_{\Delta} \theta_1(p, U) \cdot F(U) dU \qquad \text{(i)}$$

where $\theta(p)$ is the experimental adsorption isotherm data, Δ is the integration region, U is the adsorption energy, $F(U)$ is the adsorption energy distribution, and $F(U)dU$ denotes the fraction of the surface with adsorption energies between U and $U + dU$. $\theta_1(p, U)$ is the local adsorption isotherm.

According to the previous works, the Fowler-Guggenheim (F-G) local adsorption equation has been used to assess the localized monolayer adsorption on single component in gas phase (18,19). The F-G equation can be described as a following expression.

$$\theta_1(p, U) = \frac{bp \exp\left(\frac{zw\theta}{kT}\right)}{1 + bp \exp\left(\frac{zw\theta}{kT}\right)} \qquad b = b_0(T) \exp\left(\frac{U}{kT}\right) \qquad \text{(ii)}$$

where T is the absolute temperature, p is the equilibrium pressure, z is the number of closest adjacent molecules in the monolayer, w is the interaction energy between the two nearest neighboring molecules, k is the Boltzmann constant, b is the Langmuir constant, and the pre-exponential factor $b_0(T)$ can be calculated from the partition function for an isolated molecule. In this work, the generalized nonlinear regularization method, which can be generally adopted for solving the difficulties associated with numerically ill-posed problems, was employed to evaluate the surface energetic heterogeneity of ACs. The energy distributions were calculated with using the regularization parameter $\alpha = 0.001$ (16,20–22).

The calculated AEDs of acetaldehyde on all the ACs are shown in Fig. 4. The relative energetic heterogeneity can be assessed by the AEDs of each AC (17). Furthermore, the peak of AED shifted towards the higher adsorption energy is attributed to the development of the microporocity of AC (23). In this work, when the AED moves to higher values, the order of their peak results are R-1/2 > WAC > R-1/4.

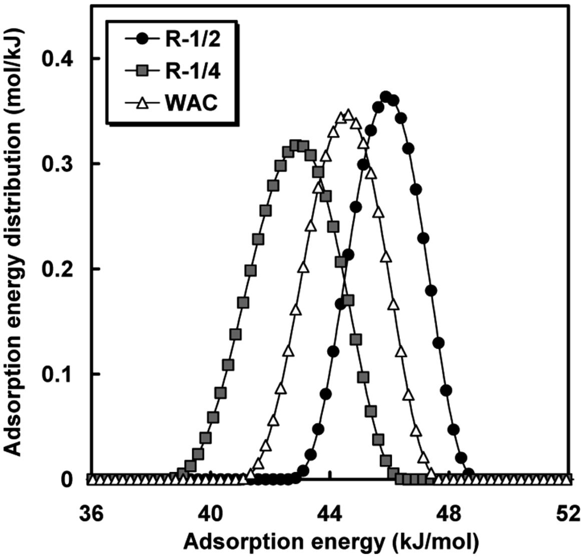


FIG. 4. Adsorption energy distributions of acetaldehyde on ACs at room temperature.

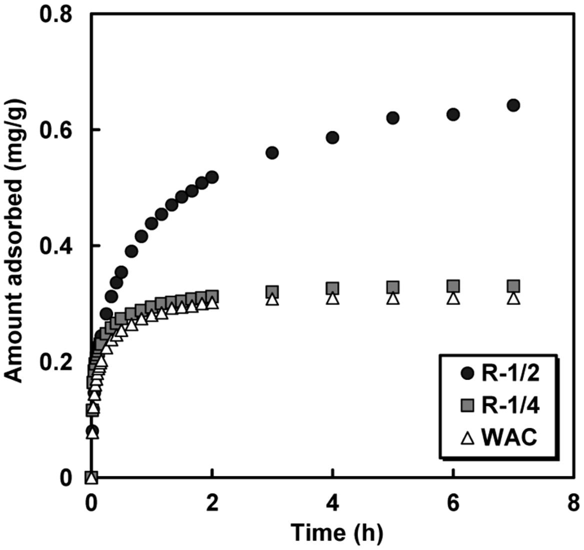


FIG. 5. The adsorption isotherms of water vapor on ACs.

These results are in agreement with the order of the adsorption capacities of acetaldehyde on ACs. Hence, the AED results indicate that the relatively higher adsorption energy on acetaldehyde adsorption is a considerable factor to attain the higher adsorption capacity as similar as the effect of the adsorption capacity influenced by textural property.

Water Vapor Adsorption

As mentioned in the previous section, the R-1/4 with the largest specific surface area and highest pore volume has the similar adsorption capacity companions with the WAC. In general, the water vapor adsorption on the surface of AC is correlated with hydrophilic functional groups (24). Thus, considering the effect of oxygen containing groups on the surface of the ACs, the water vapor adsorption property was assessed as a function of time. The adsorption isotherms of water vapor are shown in Fig. 5. It is found that the order of adsorption capacity of water vapor on ACs is the same as with the order of acetaldehyde adsorption capacity. According to the previously reported work, the high adsorption amount of water vapor on the carbon sample of the lower surface area can be explained by the higher concentration of surface oxygen groups on the surface of these carbon samples compared to the activated carbon samples of the higher surface area (25). As can be seen in Table 2, the percentage of oxygen groups on the surface of R-1/4 and WAC is comparable. Therefore, these results could well explain the correlation for the adsorption property between the R-1/4 and WAC.

In this study, the reasonable effects of the oxygen groups existed on the surface of ACs are not found on acetaldehyde and water vapor adsorption. On the other hand, it is confirmed that the adsorption capacity of acetaldehyde and water vapor on the ACs predominantly depend on the pore size distribution (below 8 Å) of AC, especially small molecular adsorption.

Adsorption Isotherm

The adsorption isotherm models such as Sips, Toth, Langmuir, and Freundlich isotherm models have been widely used to describe the reasonable correlations between adsorption characteristics and experimental data. In this work, the Sips model was used as follows.

$$q_e = \frac{q_m \cdot b \cdot C_e^{1/n}}{1 + b \cdot C_e^{1/n}} \qquad \text{(iii)}$$

where q_m, b, and n are Sips isotherm parameters, q_e is the amount adsorbed at equilibrium, C_e is the concentration of adsorbate at equilibrium condition. Besides, considering the fitness of the Sips isotherm model the average of absolute value of Δn (DN) was employed as follows

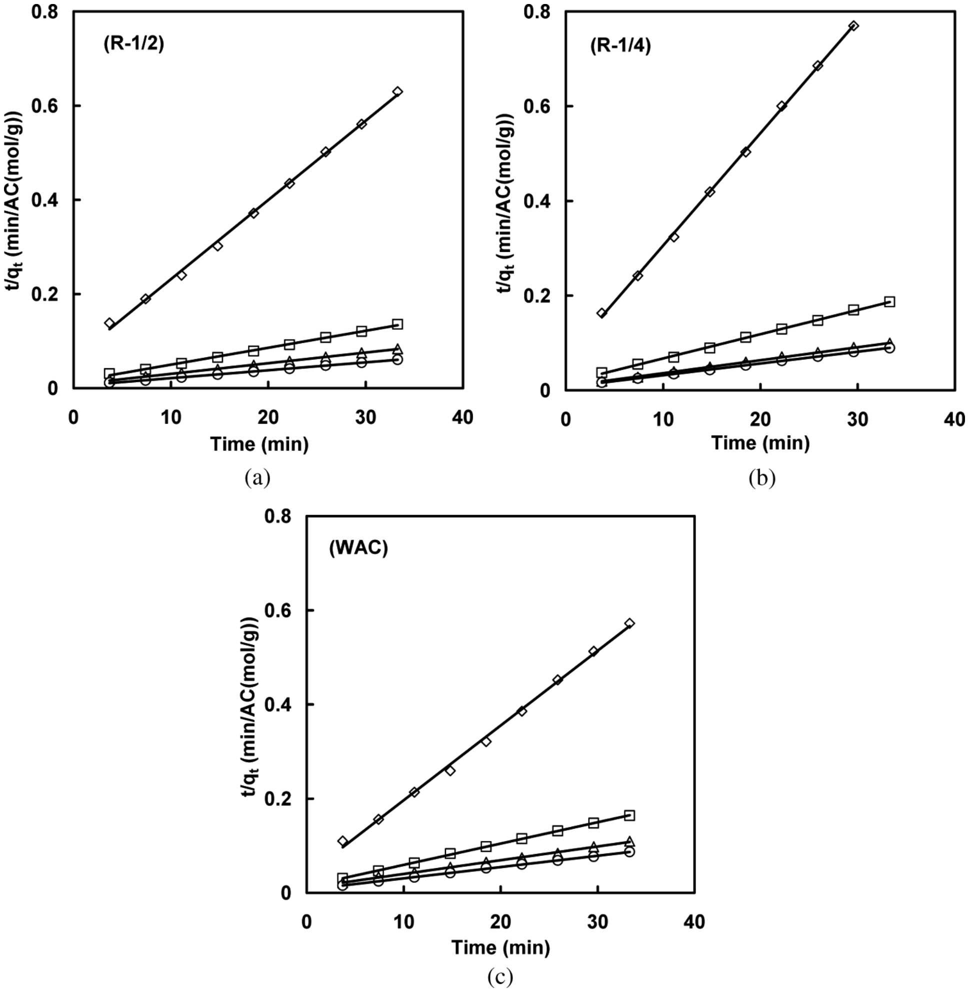
$$Average(DN) = \Delta n = \frac{1}{j}\sum_{i=1}^{j}\left|N_{cal} - N_{\exp}\right| \qquad \text{(iv)}$$

where N_{exp} is the experimental data and N_{cal} is the adsorbed amount calculated from Sips equation. j is the number of experimental points.

The isotherm parameters calculated from Sips and the DN values are listed in Table 3. The parameters (q_m and b) obtained from the Sips equation are in agreement with the order of adsorption capacity of acetaldehyde (R-1/2 > WAC ≥ R-1/4). Also, the parameters (n) generally

TABLE 4
Parameters of adsorption kinetics of acetaldehyde on ACs at room temperature

		Pseudo first-order model			Pseudo second-order model		
Sample	C_0 (μmol/L)	q_e (mmol/g)	k_f (mmol/g · min)	R^2	q_e (mmol/g)	k_s (g/mmol · min)	R^2
R-1/2	10.7	0.28	5.73	0.92	0.06	4.51	0.99
	42.8	0.64	4.56	0.85	0.28	0.92	0.99
	85.6	0.61	6.57	0.99	0.45	0.62	0.99
	107	0.76	5.25	0.93	0.59	0.64	0.99
R-1/4	10.7	0.20	8.93	0.95	0.04	8.27	0.99
	42.8	0.47	6.06	0.88	0.19	1.66	0.99
	85.6	0.53	9.08	0.96	0.37	0.83	0.99
	107	0.53	9.44	0.95	0.41	0.82	0.99
WAC	10.7	0.23	5.99	0.87	0.06	8.30	0.99
	42.8	0.47	6.94	0.94	0.22	1.50	0.99
	85.6	0.51	9.32	0.90	0.34	0.88	0.99
	107	0.57	7.99	0.93	0.42	0.84	0.99



(a) (b) (c)

FIG. 6. Pseudo second-order kinetics on ACs by varying the initial acetaldehyde concentrations at room temperature (○ 107 μmol/L, △ 85.6 μmol/L, □ 42.8 μmol/L, ◇ 10.7 μmol/L).

regarded as the parameter characterizing the system heterogeneity are values around 0.89–0.99. These results are related with energetic heterogeneity of ACs surface as mentioned in the previous section. Besides, it is observed from the *DN* values that the Sips equation well represents the good correlations with experimental data.

Adsorption Kinetics

The adsorption kinetics of acetaldehyde on ACs were assessed with varying the initial concentrations of acetaldehyde at room temperature. The empirical equations (pseudo first-order and second-order equation) were used for evaluating the correlation between the adsorbed amount and the required time. The pseudo first-order (v) and second-order (vi) equations are described as the following mathematical expressions:

$$\frac{dq_t}{dt} = k_f \cdot (q_e - q_t) \tag{v}$$

$$\frac{dq_t}{dt} = k_s \cdot (q_e - q_t)^2 \tag{vi}$$

where q_e is the quantity of acetaldehyde adsorbed at equilibrium and q_t is the adsorbed quantity of acetaldehyde. k_f and k_s are the kinetic constant of the pseudo first-order and second-order, respectively. The suitability studies of the equations used were checked through the corresponding correlation coefficient (R^2).

The parameters determined by the pseudo first-order and the pseudo second-order equation are given in Table 4. The adsorption kinetics of acetaldehyde on ACs are well illustrated by the pseudo second-order equation compared to the pseudo first-order equation. Also, the values of q_e calculated from the pseudo second-order equation are consistent with experimental data and the values of R^2 are in the range of 0.99 to 1.0. Accordingly, the pseudo second-order equation is suitable to explain the correlation between experiment data and the adsorption kinetics of acetaldehyde on all the ACs, as shown in Fig. 6.

CONCLUSION

Activated carbons (ACs) having different physical and chemical properties were prepared from biomass precursor using corn grain. Adsorption characteristics of acetaldehyde on corn-based ACs (R-1/2 and R-1/4) and a commercially available wood-based activated carbon (WAC) were assessed at room temperature using a gas chromatograph. The effects of acetaldehyde adsorption on ACs were investigated in terms of textural properties, energetic heterogeneity, and surface chemistries. In addition, the adsorption properties of water vapor were evaluated in terms of the effect of the oxygen containing groups on the surface of ACs on acetaldehyde adsorption. The results indicate that the influences of pore size distribution (below 8 Å) and energetic heterogeneity of ACs on acetaldehyde adsorption were highly predominant than that of specific surface area and surface chemistry. The Sips equation well represents the experimental results of acetaldehyde adsorption. Moreover, the pseudo second-order equation well illustrates kinetic data of acetaldehyde adsorption rather than the pseudo first-order equation.

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