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Original Article

Reduction of Adsorption Capacity of Coconut Shell Activated Carbon for Organic Vapors Due to Moisture Contents

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Abstract: In occupational hygiene, activated carbon produced from coconut shell is a common adsorbent material for harmful substances including organic vapors due to its outstanding adsorption capacity and cost advantage. However, moisture adsorption of the carbon generally decreases the adsorption capacity for organic vapors. In a previous report, we prepared several coconut shell activated carbons which had been preconditioned by equilibration with moisture at different relative humidities and measured the breakthrough times for 6 kinds of organic vapor, in order to clarify the effect of preliminary moisture content in activated carbon on the adsorption capacity in detail. We found that the relative percent weight increase due to moisture adsorption of the carbon specimen had a quantitative effect, reducing the breakthrough time. In this report, we carried out further measurements of the effect of moisture content on the adsorption of 13 kinds of organic vapor, and investigated the relationship between moisture adsorption and the reduction of the breakthrough time of activated carbon specimens. We also applied the data to the Wood's breakthrough time estimation model which is an extension of the Wheeler-Jonas equation.

Key words: Activated carbon, Adsorption capacity, Breakthrough time, Moisture adsorption, Organic vapors, Wheeler-Jonas equation

Introduction

In industrial hygiene, activated carbon is used as an adsorbent for harmful substances typified by various kinds of organic vapor. Currently, there are many kinds activated carbon products, which are produced from various raw materials and they have different configurations and specific surface areas. In Japan, activated carbon produced from coconut shell is a traditional and popular material for gas adsorption, because it has good adsorption capacity for various chemical substances, and a marked cost advantage over other adsorbent materials[1–5]. Coconut shell activated carbon is also used in the gas filters of respirators for organic vapors.

The adsorption capacities of gas filters of respirators containing activated carbons are specified by the breakthrough time, and it is indicated that the adsorption capacities are affected by temperature, flow rate, concentration of representative organic vapors and moisture content in the test gas flow. Among these influencing factors, moisture content has been the focus of many quantitative researches, and there are many reports[6–19] regarding its effect on gas filters or experimental packed beds of activated carbon at room temperature (approximately at 293–298 K). Further, Wood *et al*. have reported the effective estimation of the breakthrough time of activated carbon beds for organic vapors[20, 21] which covers the effect of all levels of relative humidities by extension of the Wheeler-Jonas breakthrough time equation[22–24] modified by Yoon *et al*[25, 26]. These researches revealed that the adsorption capacity of acti-

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vated carbon for organic vapors is reduced by the moisture content of the carbon.

In a previous report[27], we measured the adsorption capacity as the breakthrough time of coconut shell activated carbons which were preconditioned with moisture adsorption and evaluated the reduction of the capacity with reference to the relative weight increase due to the preliminary water adsorption of the carbon specimens. First, we prepared several activated carbon specimens which had adsorbed moisture at different degrees by the use of airflow humidified at a steady state. Then, the breakthrough time of each specimen for 6 kinds of organic vapor was measured by a test gas flow which had the same relative humidity as the preconditioning airflow. We used test gas flows with a single organic vapor at 300 ppm and relative humidities from 30 to 70% for the measurements. Adsorption capacities of the specimens decreased for water adsorption of 30% or higher, sharply in many cases. In addition, we found that the data plots of the breakthrough time against water adsorption showed good agreement with quadratic function approximations for a wide range of water adsorption. It was notable that the relative percent weight increase due to moisture adsorption of the carbon specimens had a clear quantitative effect, reducing the breakthrough time for organic vapors.

In this report, we carried out further measurements on 13 kinds of organic vapor using a method similar to that of previous reports[27, 28], and investigated the relationships between water adsorption and the reduction of the breakthrough time of the activated carbon specimens. We used the relative percent weight increase of the carbon specimens due to water adsorption as a simple indicator of moisture content of the specimens, as a substitute for the relative humidity of the test gas flow. We also applied the results to the Wood's breakthrough time estimation model, an extension of the Wheeler-Jonas equation.

Materials and Methods

Coconut shell activated carbon specimens in this research were granular ones taken from several major kinds of gas filter products for organic vapors which are commercially available in Japan. The characterization of the carbon specimens, the apparatus and the conditions for the preconditioning and the measurements of the breakthrough times of the specimens for organic vapors were same as those described in our previous report[27]. Among the carbon specimens examined in the report, we used S1[27], which has the largest N_2-BET specific surface area, for the more detailed measurements of the adsorption of organic vapors.

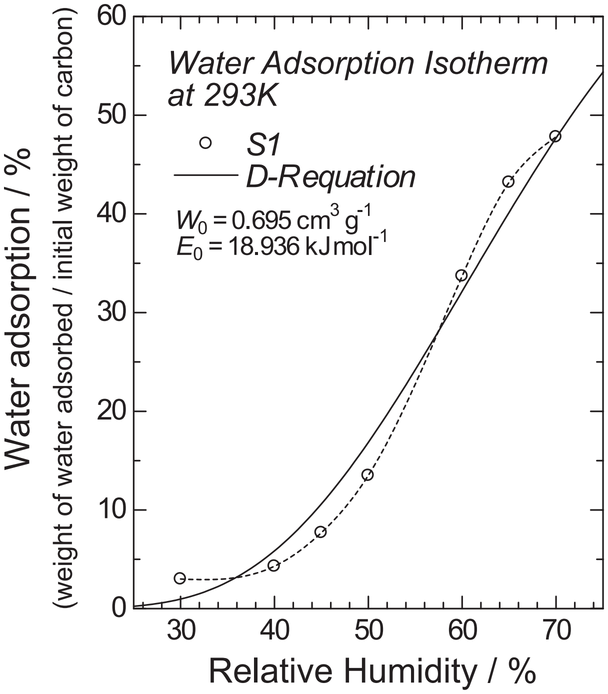


Fig. 1. Water adsorption isotherm of the activated carbon specimen S1 and its approximation by the D-R equation ($\beta_{H_2O} = 0.075$) at 293 K.

Results and Discussion

Calculation of the breakthrough time by extension of the Wheeler-Jonas equation

The data measured on the basis of the relative weight increase of the carbon specimen due to water adsorption is easily converted to the relative humidity of the preconditioning gas flow by referring to the water adsorption isotherm (Fig. 1). Following this conversion, we applied the estimation model of the breakthrough time by Wood *et al*[21]. In this model, the service lives of cartridges or activated carbon beds for organic vapors are estimated by extension[21, 29–43] of the Wheeler-Jonas breakthrough time equation (Equation 1). The basic equation is as follows.

$$t_b = \frac{W_e W}{C_0 Q} - \frac{W_e \rho_B}{k_v C_0} \ln\left(\frac{C_0 - C}{C}\right) \qquad (1)$$

where:
t_b = breakthrough time (min),
W_e = adsorption capacity of carbon (g/g-carbon),
W = carbon bed weight (g),
C_0 = challenge vapor concentration (g/cm^3),
C = breakthrough concentration (g/cm^3),
Q = airflow rate (cm^3/min),
ρ_B = bulk density of carbon (g/cm^3),
k_v = adsorption rate coefficient (min^{-1}).

In the estimation, a single vapor is assumed to be the adsorbate, and the Dubinin-Radushkevich (D-R) equation (Equation 2)[44, 45] is used to calculate the value of W_e for individual organic vapors:

$$W_e = W_0 d_L \exp\left[-\left(\frac{RT}{\beta_{OV} E_0}\right)^2 \left\{\ln\left(\frac{p_{sat}}{p}\right)\right\}^2\right] \quad (2)$$

where:
W_0 = micropore volume or adsorption space (cm^3/g-carbon),
d_L = liquid density of adsorbate (g/cm^3),
R = ideal gas constant (8.315 kJ $\cdot$ K^{-1}/mol),
T = temperature (K),
β_{OV} = affinity coefficient of organic vapor (dimensionless parameter),
E_0 = adsorption energy of a reference adsorbate (kJ/mol),
p_{sat} = bulk liquid saturation vapor pressure at the temperature (kPa),
p = pressure of vapor (kPa).

In Equation 2, the value of p_{sat} was determined by the Antoine equation[46]. The value of β_{OV} was calculated from a correlation equation (Equation 3) with molar polarizability.

$$\beta_{OV} = 0.0862 P_e^{0.75} \quad (3)$$

$$P_e = \left(\frac{n_D^2 - 1}{n_D^2 + 2}\right)\left(\frac{M_W}{d_L}\right) \quad (4)$$

where:
P_e = molar polarizability (cm^3/mol),
n_D = refractive index (dimensionless parameter),
M_W = molecular weight of adsorbate (g/mol).

Among the above parameters, d_L, n_D and M_W are available from several references[47, 48]. Further, Wood *et al.* used a simultaneous equation (Equation 5) for calculation of equilibrium volumes of water and organic vapors as liquids competing for available micropore volume or adsorption space W_0.

$$\begin{cases} V_{adsOV} = \left(W_0 - V_{adsH_2O}\right)\exp\left[-\left(\frac{RT}{\beta_{OV} E_0}\right)^2 \left\{\ln\left(\frac{p_{satOV}}{p_{OV}}\right)\right\}^2\right] \\ V_{adsH_2O} = \left(W_0 - V_{adsOV}\right)\exp\left[-\left(\frac{RT}{\beta_{H_2O} E_0}\right)^2 \left\{\ln\left(\frac{p_{satH_2O}}{p_{H_2O}}\right)\right\}^2\right] \end{cases} \quad (5)$$

$$\frac{p_{H_2O}}{p_{satH_2O}} = \mathrm{RH} \quad (6)$$

where:
V_{adsOV} = equilibrium volume of adsorbed organic vapor (cm^3/g),
V_{adsH_2O} = equilibrium volume of adsorbed water vapor (cm^3/g),
β_{H_2O} = affinity coefficient of water vapor (dimensionless parameter),
RH = relative humidity.

The estimation model is composed of the above equations and several other kinetics and adsorption equilibrium equations. In the report[21], Wood *et al.* utilized data from many previous measurements by other researchers for model testing, and their calculations are consistent with the data at high challenge concentrations, higher than or equal to 1,000 ppm, of organic vapors. In contrast, the results of their calculations markedly disagree with some experimental data at low challenge concentrations, below 100 ppm.

The experimental conditions used in this report are described in Table 1. Our challenge concentration was 300 ppm. In the calculation of the breakthrough time, the unit of vapor concentration was converted from ppm to g/cm^3 by the ideal gas law. We used the skew parameter (dimensionless parameter), S[33] = 1.22, reported in the reference data of Wood *et al*[33]. E_0 was obtained from a plot of the linearized form of the D-R water adsorption isotherm equation[21]:

$$\ln(W_e) = \ln(W_0 d_L) - \left[\left(\frac{RT}{\beta_{H_2O} E_0}\right)^2 \left\{\ln\left(\frac{p_{satH_2O}}{p_{H_2O}}\right)\right\}^2\right] \quad (7)$$

In the plot, W_0 is also obtained from the intercept of

Table 1. Conditions of measurements and the calculation of breakthrough time in this research

C_0 / ppm (challenge concentration)	C / ppm (breakthrough concentration)	Q / cm^3 min^{-1} (airflow rate)	T / K (temperature)	ρ_B / g cm^{-3} (bulk density of carbon)
300	6	30,000	293	0.364
W / g (carbon bed weight)	W_0 / cm^3 g^{-1} (micropore volume)	E_0 / kJ mol^{-1} (adsorption energy)	β_{H_2O} (affinity coefficient of H_2O)	$S = k_{v1\%} / k_{v10\%}$ (dimensionless parameter)
35	0.695	18.936	0.075	1.22

$k_{v1\%}$ = adsorption rate coefficient for $C/C_0 = 0.01$ breakthrough fraction,
$k_{v10\%}$ = adsorption rate coefficient for $C/C_0 = 0.1$ breakthrough fraction.

ln (W_e), approximated as the straight line of ln ($W_0 d_L$). W_e obtained W_0 = 0.646 (cm^3/g-carbon) from the plot of the water adsorption isotherm, and this is almost the same as the experimental micropore volume of W_0 = 0.695 (cm^3/g-carbon) obtained by N_2 gas adsorption in a previous report[27]. In this simulation, we adopted the base value of the water affinity coefficient β_{H_2O} = 0.075 from the shape of the water adsorption isotherm (Fig. 2 in the reference[21] by Wood *et al.*). The D-R equation with the above parameters provides a good approximation of the observed curve of the water adsorption isotherm (Fig. 1).

Figure 2 (a) shows the result of the calculation of the breakthrough times of organic vapors by the estimation model without modification. The calculated breakthrough times scatter in the graph, and no particular correlation of the breakthrough time with kind of adsorbate was found. In the model, it is necessary to adjust the water affinity coefficient β_{H_2O} to be 1.67 times larger than the base value when RH is larger than 50%[21]. However, this adjustment enlarged the scattering of calculated the results, and the adjustment of the water affinity coefficient was not performed for the plots of Fig. 2 (a). We attributed this discrepancy to the affinity coefficients β_{OV} calculated with Equation 3 and shown in Table 2, because the equation is a correlation[32] from experimental data found in the foregoing reports. That is, the actual values of the affinity coefficients of S1 for many organic vapors under experimental conditions are different from the values calculated by the equation. Therefore, we adjusted the affinity coefficients with reference to the breakthrough time data at RH = 40% (Table 2). The recalculation of the results with the adjusted affinity coefficients is plotted in Fig. 2 (b). In the Figure, the above mentioned adjustment of the water affinity coefficient was also carried out, and we used β_{H_2O} = 0.125 for the data at RH > 50% when required. In this result, calculated breakthrough times show good agreement with the experimental values. However, narrow range scattering remains at RH ≥ 60%, and the estimation needs further improvement.

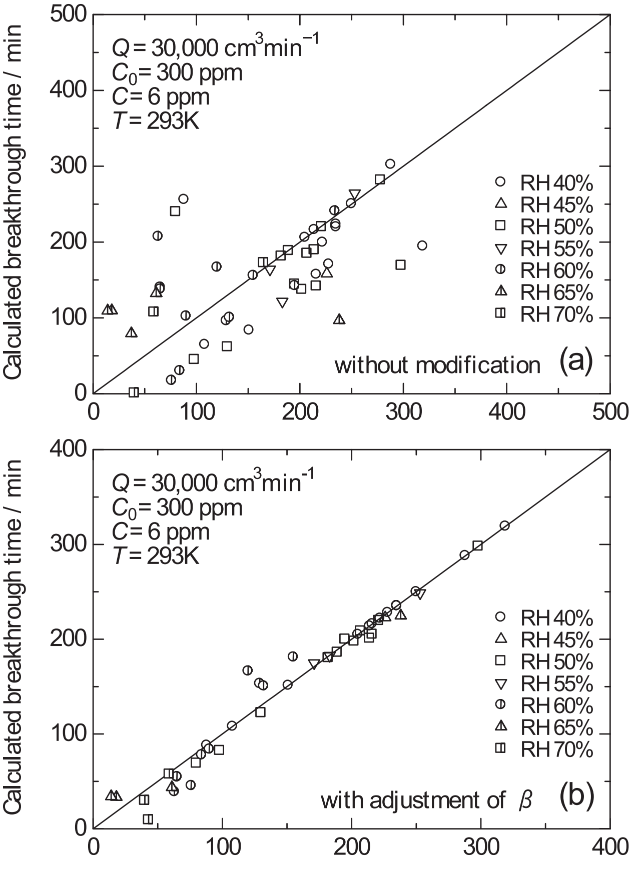


Fig. 2. Comparisons of breakthrough times of organic vapors calculated by the Wood's estimation model with experimental ones for the activated carbon specimen S1.

(a) Calculation by the Wood's model without modification of affinity coefficients, (b) Recalculation of breakthrough times with adjustment of affinity coefficients.

Reduction of the breakthrough time of the coconut shell activated carbon specimen due to moisture

The estimation model with the Wheeler-Jonas breakthrough time equation by Wood *et al.* is effective and comprehensible, and its further development is fascinating. However, the calculations are composed of many equations, which contain many parameters related to specific properties of adsorbates and activated carbon beds. In addition, the model also requires adjustments

Table 2. Affinity coefficients β_{OV} for calculation by the estimation model with the D-R equation

Organic Vapor	Calculated β_{OV}	Adjusted β_{OV}
n-Hexane	1.108	1.380
n-Heptane	1.234	1.364
Acetone	0.700	0.770
2-Butanone	0.837	0.969
Methyl acetate	0.741	0.856
Ethyl acetate	0.883	1.003
Benzene	0.998	0.997
Toluene	1.136	1.080
4-Methyl-2-pentanone	1.106	0.601
1,2-Dimethoxyethane	0.940	1.376
Cyclohexane	1.040	1.037
Carbon tetrachloride	1.006	1.000
2-Propanol	0.749	0.766

The values were calculated by Equation 3, and then adjusted with reference to the experimenal breakthrough times at RH = 40%.

to the parameters in the calculation, particularly at high relative humidity. As described above in the calculation procedure, the relative humidity of the test gas flow is usually taken as an indicator of the breakthrough time in representations of adsorption capacity. In contrast, we used relative percent weight increase due to water adsorption of the activated carbon specimen, as a substitute for relative humidity, since the relative percent weight increase is a simpler indicator of the humidification state of activated carbon in working.

Figure 3 shows the breakthrough times as adsorption capacities of S1 for 13 kinds of organic vapor. The activated carbon specimens were preconditioned with different moisture adsorptions at equilibria. In the figure, the breakthrough time of each measurement is plotted against the preliminary water adsorption, in percentage, of the specimen. Under these conditions, the amount of water adsorption in the 0–50% range in the saturated state corresponds to the equilibrated relative humidity of the airflow in the 30–70% range. The organic vapors used as adsorbates in this research included several types of organic substances (alcohols, aromatics, chain and chlorinated hydrocarbons, cyclic compounds, esters, ethers and ketone compounds); however, they showed a similar pattern in the reduction of the breakthrough time. The breakthrough time of S1 shows a gentle decrease for organic vapors in the 0–30% range of water adsorption, and the declining curves of breakthrough times are roughly approximated by the straight lines, which are shown by the broken lines in Fig. 3. However, the breakthrough time of the specimen shows a steeper decrease at water adsorption of 30% or higher in many cases of measurement. This critical point of effect of water adsorption seems to be significant for the utilization of activated carbon in respirators, and it also approximately corresponds to an equilibrated atmosphere with relative humidity of 58% at 293 K.

We found that the plots (except for 2-propanol) show good agreement with quadratic function approximations, which are shown by solid lines for a wide range of water adsorptions in Fig. 3. Note, however, that the approximation curve and the dotted line almost overlap in the case of ethyl acetate. Absolute values of coefficients of correlations for 2 types of approximations, linear ($|R_1|$) and quadratic ($|R_2|$) functions, for the organic vapors are listed in Table 3. In general, the quadratic function approximations show closer agreement with the breakthrough time plots than linear function approximations, and it is remarkable that the relative percent weight increase due to moisture adsorption of the carbon specimen had a clear quantitative effect on the breakthrough times for organic vapors.

Regarding the reduction of the breakthrough time of the carbon specimens, the specific reduction for each kind of adsorbate (organic vapor) is also an important subject. Figure 4 (a) shows the relationship between molar volumes of adsorbed organic vapors and the breakthrough times of the carbon specimens, which were preconditioned to contain water at approximately 4.6%.

$$V_m = \frac{M_W}{d_L} \quad (8)$$

where:

V_m = molar volume of adsorbed organic vapor (cm^3/mol).

Figure 4 (b) shows the transition of breakthrough times with percentage of water adsorption of the carbon specimens. The results seem to demonstrate no significant molar volume dependence of the breakthrough time. We also investigated other correlations with several parameters (e.g., boiling point of organic vapors and molar polarizability calculated by Equation 4); however, we found no significant specific reduction of the breakthrough time for any of the organic vapors used in this research.

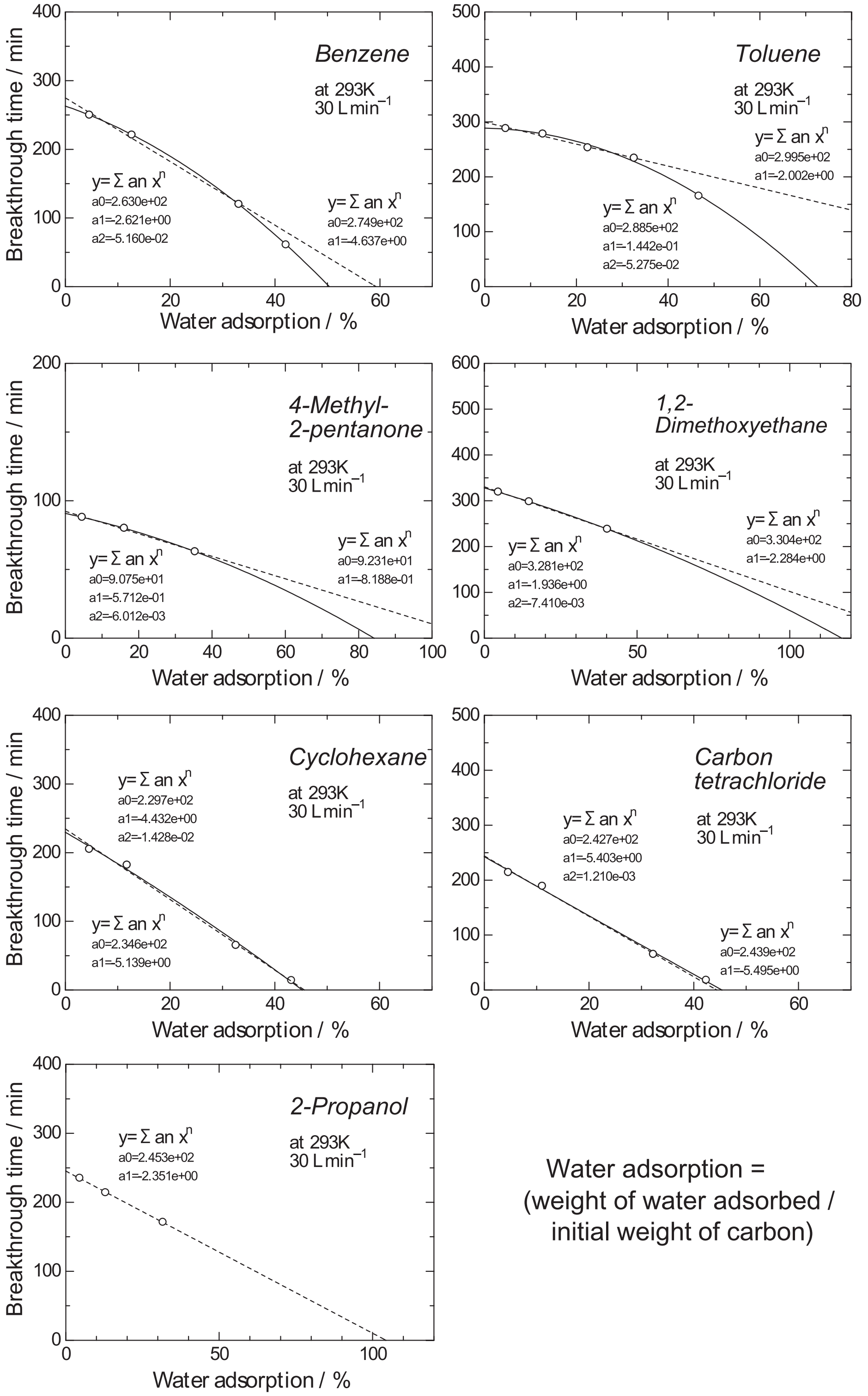
Reduction of the breakthrough time of activated carbon due to moisture adsorption: data from foregoing research

The above results were obtained for a single activated carbon product; however, we also investigated the reduction of the breakthrough time due to moisture with other data sets for different products of activated carbon, which were taken from several foregoing reports by other researchers.

There are many foregoing reports regarding the effect of moisture on gas filters or experimental packed carbon beds. In these reports, the relative humidity of the test gas flow was taken as the critical factor for the evaluation of the adsorption capacity of the activated carbon, and most of the reports mention little about the quality of the activated carbon. However, some reports[9, 13, 18)] do show the water adsorption isotherms of the activated carbon specimens, from which breakthrough time reduction can be related to the relative percent weight increase due to water adsorption of the activated carbon.

In Fig. 5, we converted the data plots of the preceding papers to show the relation between the breakthrough time and the relative percent weight increase due to water adsorption of the activated carbon specimens. Figure 5 also shows the approximated curves for the plots.

We need to take note that the original data plots have



Water adsorption =
(weight of water adsorbed /
initial weight of carbon)

Fig. 3. Reduction of the breakthrough times due to preliminary water adsorption by S1 for organic vapors (300 ppm), and linear and quadratic function approximations.

The range of approximation with linear function (broken lines) is approximately from 0 to 30%, and that with quadratic function (solid lines) is for the whole range of water adsorption. Note that the approximate curve and the straight line almost overlap in the case of ethyl acetate.

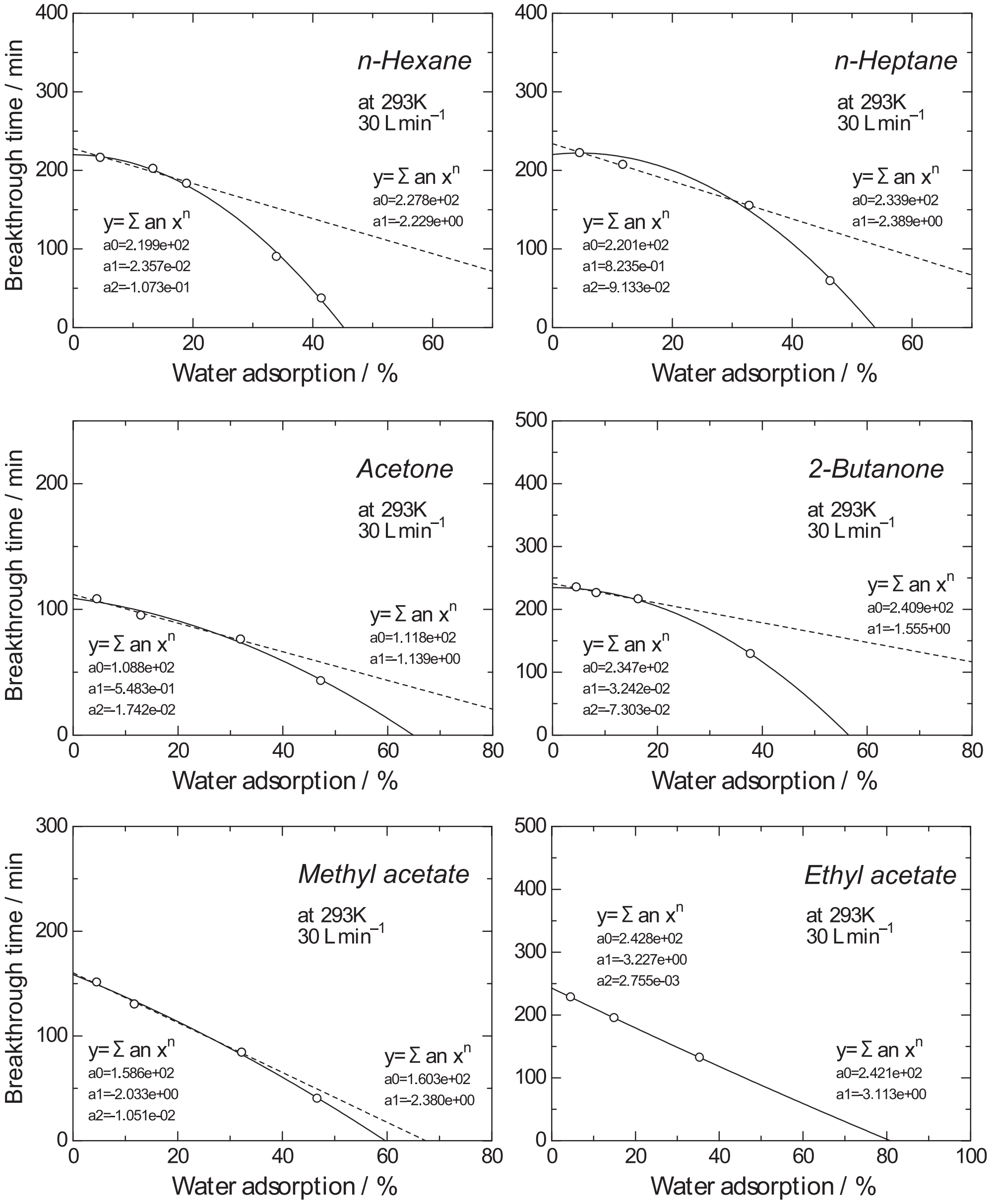


Fig. 3. (*continued.*)

different measurement conditions of airflow rate, challenge concentration of organic vapors, breakthrough concentration and other components as described in Table 4. In addition, the activated carbon specimens in the preceding papers are not necessarily coconut shell activated carbon, and the raw materials of several specimens are not even specified. However, it is worthy of attention that these plots have similar quantitative results for the effect of water adsorption as found in our measurements, and the breakthrough times of the specimens decreased for water adsorptions of around 30% or higher, sharply in the cases of (a), (b) and (d) in Fig. 5. The converted data plots do not necessarily conform to quadratic function approximation. However, the results support the potential of relative percent weight increase due to water adsorption of activated carbon as a simple indicator of the breakthrough time reduction.

Conclusion

We measured the breakthrough times of 13 kinds of organic vapor as the adsorption capacity of coconut shell activated carbons, which were preconditioned with different moisture adsorptions. Then, we evaluated the reduction of the adsorption capacity with reference to the relative weight increase due to the preliminary water adsorption of the carbon specimens. We used test gas flows with a single organic vapor at 300 ppm and rela-

Table 3. Absolute values of coefficients of correlation for the approximated lines or curves in Fig. 3.

Organic Vapor	$\lvert R_1 \rvert$ (linear approximation)	$\lvert R_2 \rvert$ (quadratic approximation)
n-Hexane	0.9770	0.9991
n-Heptane	0.9995	0.9955
Acetone	0.9937	0.9946
2-Butanone	0.9852	0.9988
Methyl acetate	0.9983	0.9987
Ethyl acetate	0.9998	1.0000
Benzene	0.9979	1.0000
Toluene	0.9936	0.9961
4-Methyl-2-pentanone	0.9982	1.0000
1,2-Dimethoxyethane	0.9997	1.0000
Cyclohexane	0.9954	0.9982
Carbon tetrachloride	0.9976	0.9986
2-Propanol	0.9998	—

$\lvert R_2 \rvert$ for 2−Propanol is omitted because of the poor fit of the quadratic approximation curve.

tive humidities ranging from 30 to 70% for measurements of 2% breakthrough time. In each measurement, the test gas flow had the same relative humidity as the preconditioning airflow. The breakthrough times of the carbon specimens decreased at water adsorptions of 30% and higher, sharply in many cases. The data plots of the breakthrough times showed good agreement with quadratic function approximations for a wide range of water adsorptions. We also found similar results for data published in the previous reports by other researchers, indicating the relative percent weight increase due to moisture adsorption of activated carbon is a simple indicator of the reduction of its adsorption capacity measured as the breakthrough time. We found no significant specific reduction of breakthrough time related to the kind of organic vapor in this research. The breakthrough time estimation model by extension of the Wheeler-Jonas equation by Wood *et al.* shows good agreement with our data particularly at RH of 40–55% after adjustment of the affinity coefficients.

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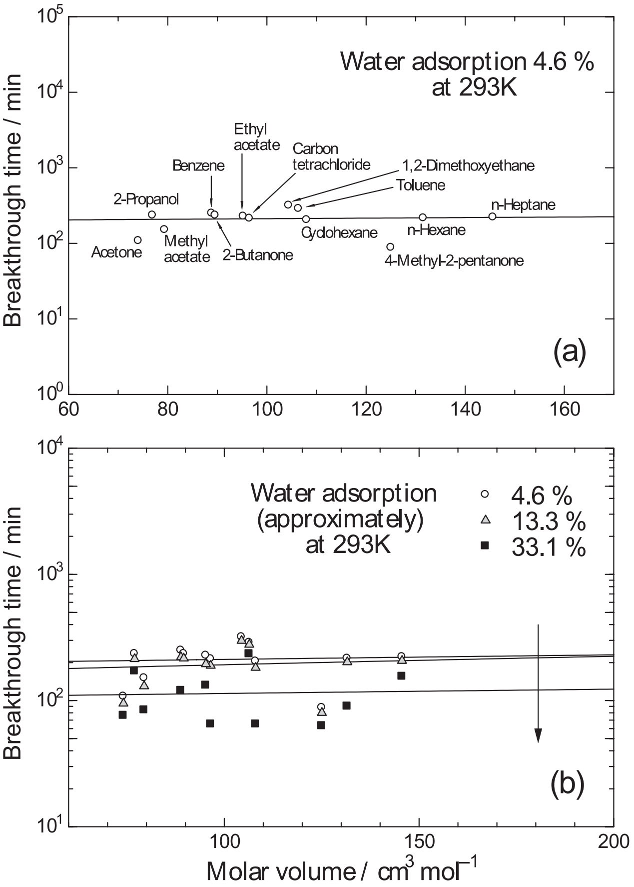
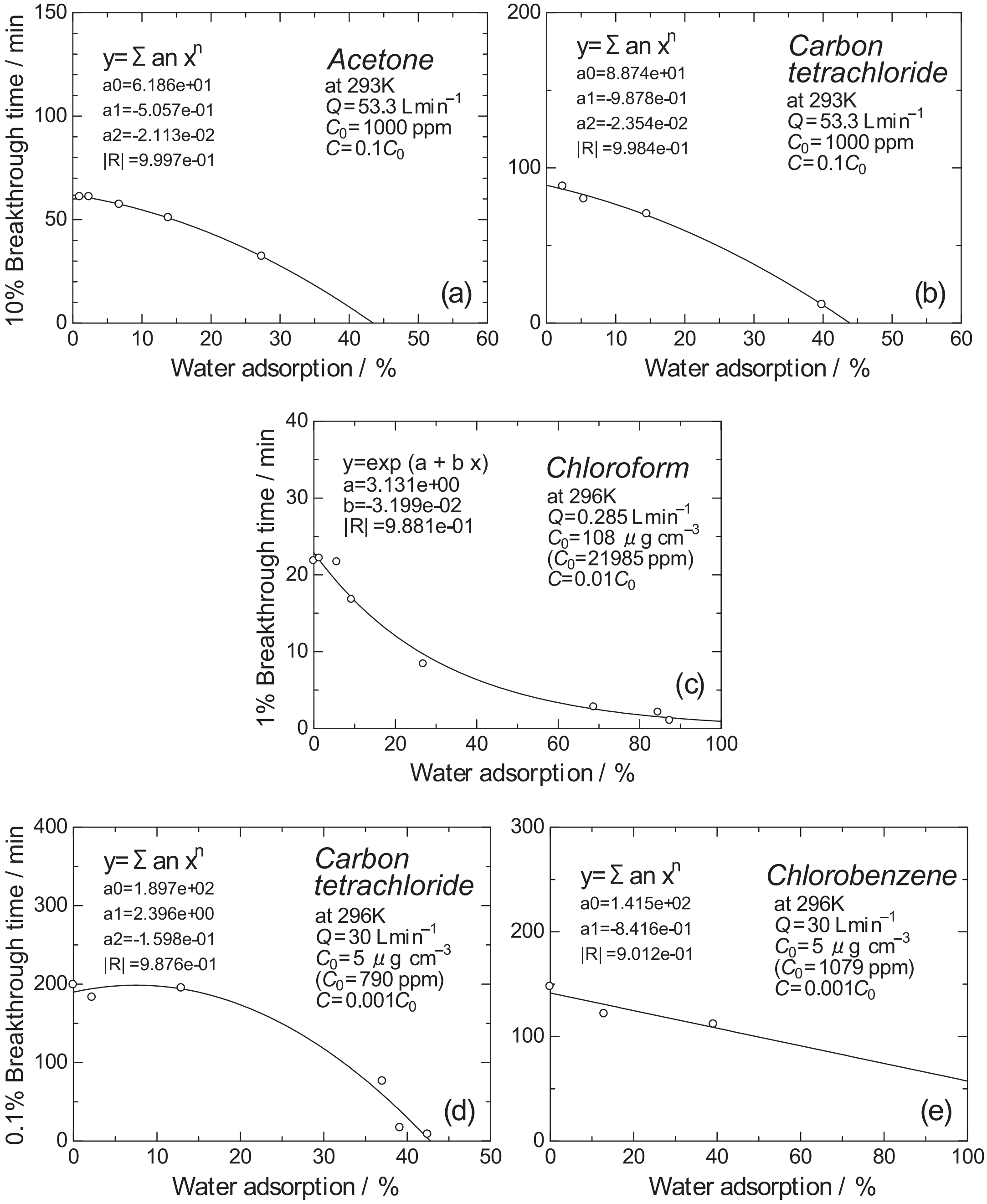


Fig. 4. Relationships between the breakthrough time of the humidified activated carbon and the molar volume of adsorbed organic vapors.

(a) Breakthrough time of each organic vapor for the carbon specimen which contains water approximately 4.6%.

(a) Transition of breakthrough time for the carbon specimens with different water contents.



Water adsorption = (weight of water adsorbed / initial weight of carbon)

Fig. 5. Reduction of the breakthrough times due to preliminary water adsorption of activated carbon specimens in the data retrieved from the preceding reports.

(a) petroleum base (b) coconut shell base: Nelson *et al.*[9]),

(c) North American Carbon Co., Columbus, OH, NACAR G-352, Lot 2905: Jonas *et al.*[13]),

(d) (e) Calgon Carbon Co., BPL-HA carbon: Lodewyckx *et al.*[18]).

Table 4. The measurement conditions in the preceding reports by other researchers

Fig.	Ref. No.	Cartridge or Activated carbon	W / g	ρ_B / g cm^{-3}	Organic Vapor	C_0 / ppm	C / C_0	Q / L min^{-1}	T / K
5 (a)	9	American Optical Co., Model R-51 (petroleum base)	74	0.43	Acetone	1,000	0.1	53.3	293
5 (b)	9	Mine Safety Appliance Co., Model 44135 (coconut base)	52	0.38	Carbon tetrachloride	1,000	0.1	53.3	293
5 (c)	13	North American Carbon Co., Columbus, OH, NACAR G-352, Lot 2905	2.25	0.351	Chloroform	21,985	0.01	0.285	296
5 (d)	18	Calgon Carbon Co., BPL-HA carbon	78	0.49	Carbon tetrachloride	790	0.001	30	296
5 (e)	18	Calgon Carbon Co., BPL-HA carbon	78	0.49	Chlorobenzene	1,079	0.001	30	296

(a) petroleum base (b) coconut shell base: Nelson *et al.*[9)],

(c) North American Carbon Co., Columbus, OH, NACAR G-352, Lot 2905: Jonas *et al.*[13)],

(d) (e) Calgon Carbon Co., BPL-HA carbon: Lodewyckx *et al.*[18)].

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