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Original Article

Quantitative Evaluation of the Effect of Moisture Contents of Coconut Shell Activated Carbon Used for Respirators on Adsorption Capacity for Organic Vapors

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Abstract: Activated carbon is an elemental material used for hygienic applications, particularly as an adsorbent for harmful gases and vapors. In Japanese industrial and occupational hygiene, activated carbon produced from coconut shell is a traditional and popular adsorbent material due to its excellent adsorption ability and cost advantage. In this research, in order to clarify the effect of the preliminary content of moisture on the adsorption capacity in detail, we prepared several coconut shell activated carbons which were preconditioned by equilibration with moisture at different relative humidities. We measured their adsorption capacities as breakthrough times for 6 kinds of organic vapor, and attempted to determine the relationships between the relative weight increase of water adsorption and the decrease of adsorption capacities of the activated carbon specimens for the organic vapors. The procedure of the quantitative evaluation of the effect of moisture and the results are useful for practical applications of activated carbon, particularly those used as adsorbents in workplaces.

Key words: Activated carbon, Adsorption capacity, Breakthrough time, Gas filter, Moisture adsorption, Organic vapors, Respirator

Introduction

In industrial and occupational hygiene, activated carbon is generally used for adsorbents of harmful substances typified by various kinds of organic solvent vapors. Currently, there are many kinds of products of activated carbon, which are produced from various raw materials, having various configurations and specific surface areas. Among them, popular products of activated carbon used for gas filters of respirators on the Japanese market are those made from coconut shell. They have good adsorption ability for organic vapors and a marked cost advantage compared with other porous adsorbent materials. Consequently, the products are used as effective materials for air purification in workplaces, and appropriate handling and storage of them are important subjects for practical and effective use.

Adsorption capacities of cartridges or canisters of respirators containing activated carbon as their contents are specified by breakthrough time, and it is indicated that their adsorption capacities are affected by temperature, flow rate of the sampled air, the concentrations of representative organic vapors and the moisture content in the airflow. Among these influencing factors, moisture content has been the focus of many quantitative researches, and there are many reports[1–14)] regarding its effect on the gas filters of respirators or experimental packed beds of activated carbon at room temperature (approximately at 293–298 K).

Around the time of World War II, Burrage *et al.* reported[1)] that the adsorption capacity of several kinds of charcoal for carbon tetrachloride (CCl_4) vapor is reduced by moisture in the vapor and also by pre-adsorption of

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moisture by the carbons, and they tried to estimate the effect quantitatively. Later, in the 1970s, Nelson *et al.* reported[3–5] the effects of the above mentioned influencing factors on adsorption capacity of activated carbon for many kinds of organic vapor in detail. The measurements in their reports were carried out using both petroleum and coconut shell activated carbons for comparatively high concentrations of organic vapors (1,000 ppm). They also investigated the effect of preconditioning of carbon specimens, similar to that reported by Burrage *et al.*[1], and performed multiple measurements with combinations of relative humidity preconditioning (50–80%) and test gas flow (20–90%). Preconditioning with moisture was continued until equilibrium was established. However, the relative humidity of each test gas flow was not necessarily the same as that of the preconditioning. In this regard, Nelson *et al.* concluded that relative humidity of the test gas flow had a more marked influence on the adsorption capacity of the activated carbons than that of the preconditioning. Furthermore, they also reported that there was little effect of relative humidity on the breakthrough time of the carbon beds when it was below 50%, and that temperature was not a dominant factor at room temperature. In their conclusion, they noted that the factors influencing adsorption capacity are many and complicated, and the kind of organic vapor can not decide the breakthrough time of the activated carbon bed simply.

Subsequently, the effect of moisture on adsorption of several kinds of specific organic vapor was investigated[7–9]. Werner *et al.* reported the effect of moisture on the adsorption of trichloroethylene vapor[7]. They used vapors at concentrations of 300–1,000 ppm and at relative humidities of 5 and 85%. Their results are slightly different from those of Nelson *et al.*[3–5], in that particularly in adsorption of trichloroethylene vapor at low concentrations, the moisture effect was not negligible even at relative humidities below 50%. That is, all levels of relative humidity could affect the lifetimes of the carbon beds of their specimens. Jonas *et al.* reported the effect of moisture on the adsorption of chloroform vapor[8]. They used a wide range of relative humidities (13–95%), for both preconditioning of activated carbon specimens and in the test gas flows. They indicated that preconditioning had little effect on adsorption when relative humidity was below 40%, and that preliminary water adsorption by the carbon bed had greater impact on adsorption capacity than the moisture content of the chloroform vapor flow.

In recent years, several reports have re-examined the adsorption capacity of gas filters of respirators[10–14], and they have mostly investigated substitutes for carbon tetrachloride as a standard substance for evaluating the adsorption capacity of gas filters for organic vapors. In these researches, it is notable that measurements were carried out by gas filters principally “as received” or “as purchased” without preconditioning, unlike previous papers. Among these reports, Moyer *et al.* reported[11, 12] the measurements of lifetimes of respirator cartridges as received using several kinds of organic vapor with moisture contents 50 and 80%, and they also indicated a reduction of lifetime under high humidity conditions.

Since the 1990s, Wood *et al.* have reported kinetics studies of the adsorption rate and the capacity of the activated carbon bed for organic vapors[15–19]. They have also reported estimations of the adsorption capacities of organic vapors of activated carbon beds[19] considering the effect of all levels of relative humidity by extension of the Wheeler-Jonas breakthrough time equation[18, 20]. In their report[19], they used many previous measurement data by other researchers for model testing, and their calculations are consistent with the data at high concentrations of organic vapors, greater than or equal to 1,000 ppm. However, the results of their calculations are inconsistent with the experimental data at low concentrations below 100 ppm, and long breakthrough time range in each figure. In this regard, they concluded that the estimation model needs further improvement.

In Japan, gas filters of respirators for organic vapors mostly contain activated carbon, which is usually made from coconut shell. In contrast, the reports by European and American researchers investigated various carbon specimens including charcoal, coconut shell activated carbon and petroleum activated carbon. Furthermore, there are many reports which have little mention of the quality of material of the contents. Nelson *et al.* stated that petroleum activated carbon was increasingly being used for economic reasons[5]. At present, coconut shell activated carbon is assumed to be affected by moisture content more than petroleum activated carbon[3–5], and further analysis of this point in detail is desirable. Moreover, relative humidity varies significantly from season to season in indoor atmospheres without air-conditioning in Japan. In order to estimate the effect of moisture uptake by coconut shell activated carbon on its adsorption capacity for organic vapors, we measured the adsorption capacity as the breakthrough time of carbon specimens which had been preconditioned with moisture. In this research, we used the relative percent weight increase of the carbon specimens due to water adsorption as the benchmark of moisture content of the specimens, in place of the relative humidity of the test gas flow used in the cited above reports. We tested the adsorption of organic vapors at 300 ppm following test conditions stipulated by Japanese official standards for gas masks[13], and at relative humidities from 30 to 70%. In this paper, we report the details of the measurements and the results of quantitative analysis.

Materials and Methods

The coconut shell activated carbon specimens used in this research were granular ones taken from several major kinds of gas filters for organic vapors, and the filters were commercially easily-available in Japan. The isotherms of nitrogen at 77 K, N_2-BET specific surface area, pore size distributions and pore volumes of the activated carbon specimens were measured by BELSORP 36 (BEL JAPAN Inc.) with N_2 gas ($\geq$ 99.9995%). Pore size distributions and pore volumes of the specimens were calculated by the MP method (microporous region radius of pores, r<1.0 nm) and the Dollimore-Heal (D-H) method (mesoporous region radius of pores, 1.0 nm <R< 25 nm). In addition, we performed X-ray fluorescence (XRF) analysis excluding light elements (atomic number Z=1–8,10) by RIGAKU RIX 2000, and elemental analysis for CHN by PerkinElmer 2400 II CHNS/O analyzer. We measured the true density of the specimens by Micromeritics multivolume pycnometer 1305 with He gas ($\geq$ 99.99995%). Ash measurements were carrried out with a combustion condition of 1173 K for 0.5 h in air in Muffle Furnace FP 100 (Yamato Scientific Co., Ltd.) using 500 mg of each specimen. We also performed analysis of energy dispersive X-ray spectrometry (EDX) on the ash of the carbon specimens by HORIBA EMAX-7000 with HITACHI S-4700 scanning electron microscope (SEM).

The apparatus for preconditioning of the activated carbon specimens with humidified airflow and the measurements of adsorption capacity of the specimens for organic vapors were similar to those described in the previous report by Furuse *et al*[13]. The apparatus consisted of two prominent parts, one for generating an airflow containing an organic solvent vapor at a constant concentration with constant relative humidity, and the other for testing the breakthrough time of the test column, described below, filtering the airflow containing the organic vapor. They were set in a thermostatic air box at 293 K and the exit vapor concentration from the test column was monitored by a gas chromatograph system equipped with a hydrogen flame ionization detector (FID) manufactured by Ohkura Riken Co., Ltd.

After drying at 348 K, we packed the carbon specimens in an acrylic resin tube with an internal diameter of 70 mm, and the preconditioning and measurements of adsorption capacity were carried out. The packed carbon bed, 35 g, was sandwiched between 2 metal meshes in the test column. The length of the packed bed was approximately 25 mm, and the bulk density of the carbon specimens for each measurement was approximately 0.364 g/cm^3. The flow rate of the humidified air for preconditioning and the test gas containing the organic vapor was adjusted to 30 l/min, and the temperature was kept at 293 K. The moisture adsorption of the activated carbon specimens was determined by the weight increase of the specimen, and the adsorption capacity was determined by the breakthrough times for each organic vapor at a concentration of 300 ppm.

First, we prepared several activated carbon specimens which had adsorbed moisture at different degrees by the use of airflow humidified at a steady state. The preconditioning was carried out by passing the humidified airflow for long enough to attain an equilibrium state. Then, the breakthrough times for 6 kinds of organic vapor were measured by a test gas flow which had the same relative humidity as the preconditioning airflow, and the relationships between the degrees of moisture adsorption and the reduction in the adsorption capacities for the organic vapors were analyzed. In this research, we determined the breakthrough time as the time when the concentration of each organic component in the exit gas flow reached 6 ppm. Organic solvents used in this research were products of Wako Pure Chemical Industries Ltd.

Results and Discussion

We examined 3 types of coconut shell activated carbons taken from contents of gas filters of respirators for a comparative examination in advance of measurements of adsorption capacity.

Figure 1 shows N_2 adsorption and desorption isotherms (at 77 K) of the activated carbon specimens in this research. They have characteristic curves with a flat plateau shape, and the curves are classified as type I isotherms (defined by IUPAC) showing that all of the specimens were microporous carbon materials; however, there were apparent differences among their specific surface areas and chemical characteristics as follows.

The porous properties and true densities of the specimens are described in Table 1, and the results of elemental analysis for CHN of the specimens are described in Table 2. In the specimens, the micropore structure mainly contributes to the specific surface area, and the orders of value of micropore volume and the specific surface area were in agreement with each other among the specimens. Further, the results of XRF analysis for the principal compositions of the specimens calculated by the FP method assuming oxide compositions are described in Table 3. In the analysis and calculation, light elements (atomic number Z=1–8,10), including carbon, were excluded from objects for clarity; the specimens consisted mainly of carbon. As a result of the XRF analysis, we found that the specimens had several elements in common, and these elements were typical components of coconut shell. These results were confirmed by qualitative measurements of the EDX analysis for the ash of

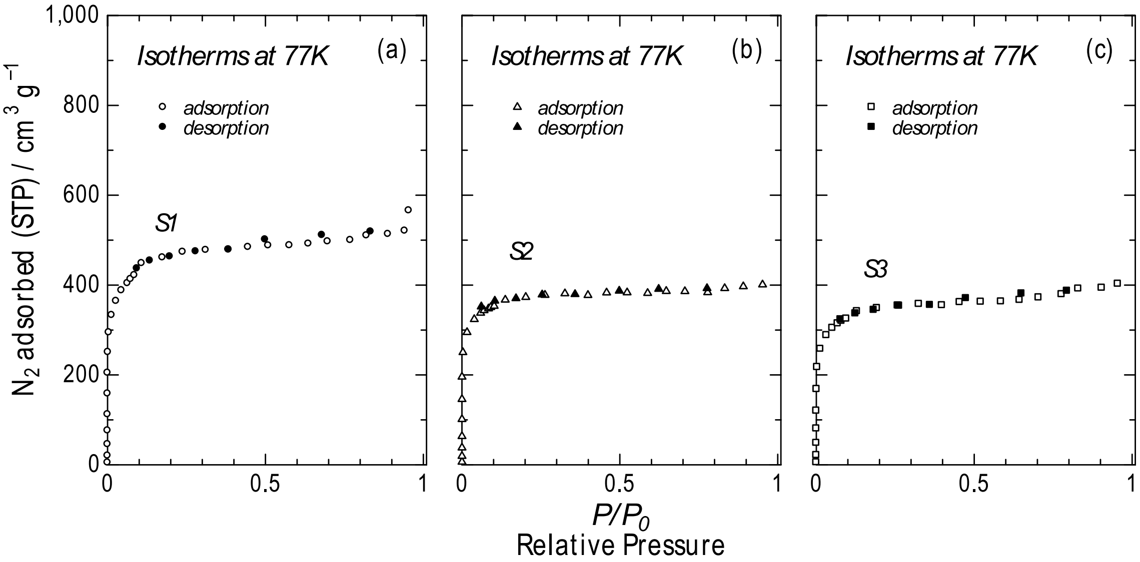


Fig. 1. N_2 adsorption and desorption isotherms at 77K of activated carbon specimens in this research.
STP means Standard Temperature and Pressure.

Table 1. Porous properties of the activated carbon specimens in this research

SAMPLE	S1	S2	S3
Cartridge	SHIGEMATSU OV	KOKEN KGC	SUMITOMO 3M No.3001
N_2-BET specific surface area/m^2 g^{-1}	1,768	1,435	1,333
V_{micro}/cm^3 g^{-1}	0.695	0.577	0.486
V_{meso}/cm^3 g^{-1}	0.1374	0.0344	0.0828
True density/g cm^{-3}	2.310(10) N=5	2.209(7) N=6	2.033(6) N=5

V_{micro} is micropore volume calculated by the MP method, and V_{meso} is mesopore volume calculated by the D-H method. Regarding true density, 2.310(10) means 2.310 ± 0.010, and N=measurement number of times.

Table 2. Results of elemental analysis of the activated carbon specimens

SAMPLE	Content/mass %		
	C	H	N
S1	67.3	0.5	0.2
S2	92.0	0.5	0.2
S3	77.9	0.5	0.2

Error of each measurement is ≤ 0.3 mass %.

Table 3. Results of XRF analysis for the principal elements of the activated carbon specimens by the FP method assuming oxide compositions

SAMPLE	Content/mass %				
	K	Ca	Fe	P	Si
S1	14.0	51.6	4.3	7.6	4.7
S2	74.6	9.7	3.0	2.7	2.7
S3	71.2	13.9	4.1	1.6	3.0

In the analysis and calculation, light elements (atomic number Z=1–8, 10) were excluded from objects.

each carbon specimen which gave similar results. The order of the true densities of the specimens corresponded to that of the residues in ash measurements (Table 4). However, the residues were small compared to the entire amount of the specimens, and the contribution of ash content to true density was not profound. Among the specimens, S1 had the highest true density. As seen in Tables 2–4, S1 also seems to have an oxygen content greater than that of S2 or S3, and we consider that the higher oxygen content was the main factor for the high true density of S1. S2 had a higher true density and a higher carbon con-

Table 4. Ratio of weight of residue on ignition in ash measurements of the activated carbon specimens

SAMPLE	S1	S2	S3
Ash residue /mass %	0.5	0.3	0.1

tent than S3. However, in this case, S2 had remarkably poor pore development in the mesoporous region compared to the other specimens, and the porous structure influences the density. Among the specimens, S1 had the largest N_2-BET specific surface area, and we used S1 for detailed measurements of the adsorption capacities for the organic vapors.

Figure 2 (a) shows the variation of the weight increase of the activated carbon specimens (S1–S3) with time during exposure to humidified airflow with relative humidities of 50 and 60%, and a flow rate of 30 l/min. We have defined moisture adsorption by activated carbon specimens as the weight increase of the carbon packed bed, and "water adsorption" in the figure is the weight increase relative to the initial weight of the dry packed bed of the carbon specimen without the acrylic tube of test column. The figure shows that none of the specimens reached the equilibrium state, even 6 h, and their temporal variations are almost similar at each relative humidity level. Further, Fig. 2 (b) shows the variation of the weight increase of S1 for 3 levels of humidified airflows relative humidity 50–70% and the flow rate 30 l/min. From these results, we have assumed that water adsorption by the specimen S1 reach equilibrium state within 18 h under the experimental conditions in this research. We carried out preconditioning of each specimen for 18 h, and prepared activated carbon specimens with different preliminary moisture contents.

Figure 3 (a) shows water vapor adsorption isotherms for the activated carbon specimens (S1–S3) at 293 K. Each specimen was preconditioned as described above at one of several relative humidity levels. The shapes of their isotherms are similar; however, the curve of S1 shows greater water adsorption than those of S2 and S3 at all relative humidity levels. The water adsorption of the specimens is expected to depend on their micropore structures and the degree of their hydrophobic properties, which are mainly dependent on their surface chemical components. S1 had the largest specific surface area and the smallest carbon content among the specimens, and both of these properties would affect the water adsorption isotherms. The curves for S2 and S3 show a crossing of the isotherms. S2 seems to have the highest hydrophobicity among the specimens from the high carbon content in the results of CHN analysis (Table 2). As a consequence, water adsorption of S2 fell below that of S3 in the range from 40 to 60% of relative humidity. However, at higher relative humidities, S2 showed higher water adsorption than S3, and this result is consistent with their specific surface areas. Figure 3 (b) compares the isotherm of S1 with isotherms from the previous reports[4, 8, 21]. In the figure, a steep increase in the weight of S1 occurs at relatively low humidity, and this feature indicates that S1 is more susceptible to moisture than the other activated carbons. In the previous reports of the moisture effect on the adsorption ability or capacity of cartridges of respirators[1–14], there is little description of the porous structures and the specific surface areas of activated carbon contents of the cartridges. However, from the comparison of the curves in Fig. 3 (b), the specific surface area of S1 is

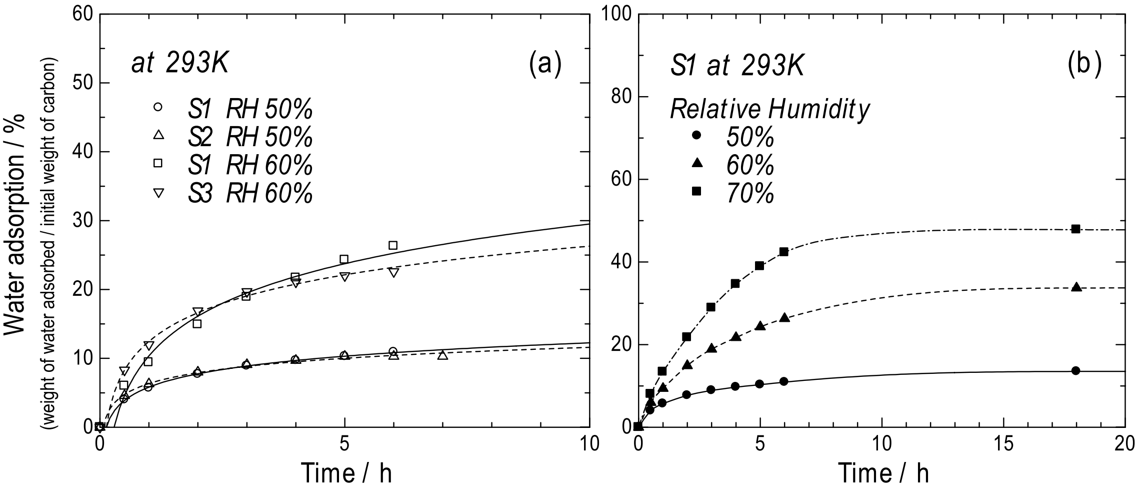


Fig. 2. Variation of the weight increase of the activated carbon specimens (S1–S3) with time by the exposure to humidified airflow (30 l/min).

RH in the figure (a) means Relative Humidity.

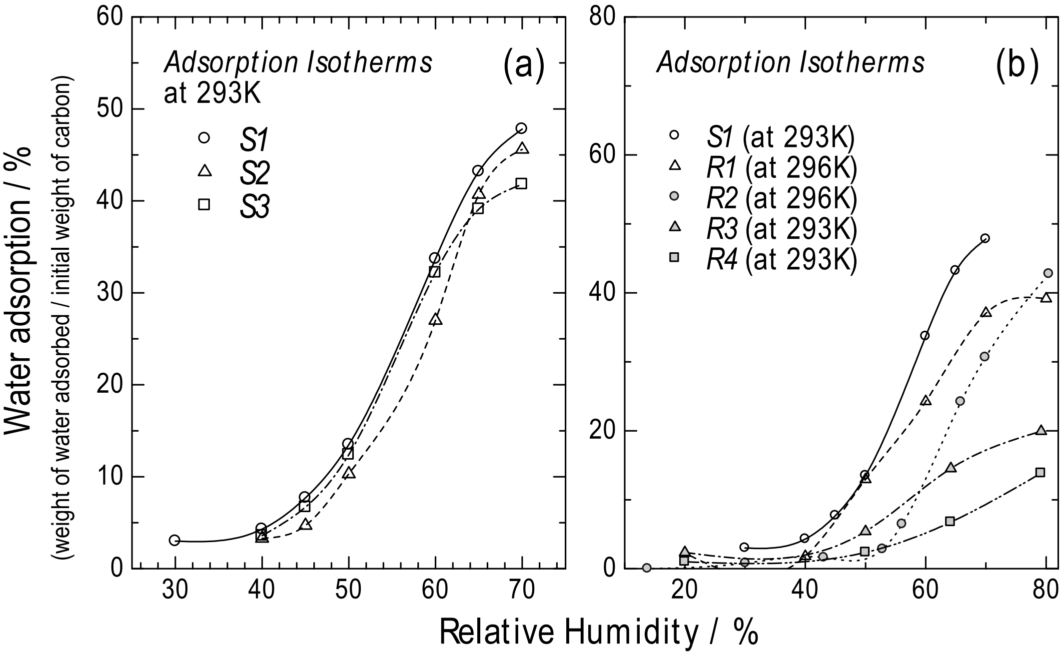


Fig. 3. (a) Water adsorption isotherms of the specimens (S1–S3) in this research. (b) Comparison of water adsorption isotherms by S1 and the data of the previous reports.

R1 (Calgon Carbon Co., BPL-HA carbon, V_{micro}=0.42–0.49 cm^3/g): Lodewyckx *et al.*[21)],

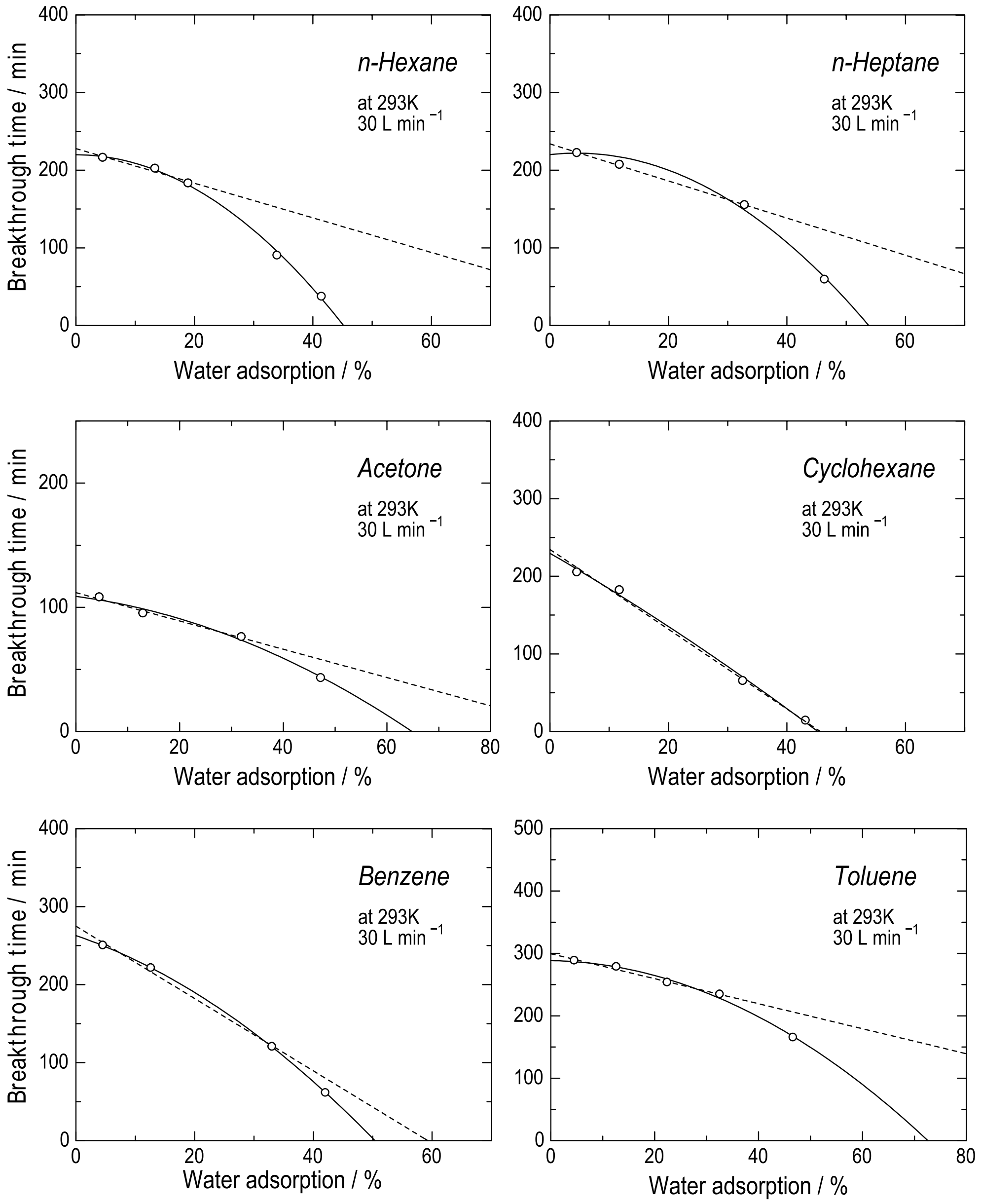
R2 (North American Carbon Co., Columbus, OH, NACAR G-352, Lot 2905): Jonas *et al.*[8)],

R3 (coconut shell base: Mine Safety Appliance Co., Model 44135) and R4 (petroleum base: American Optical Co., Model R-51): Nelson *et al.*[4)]

expected to surpass those of R1–R4. In addition, in the 1980s, Urano *et al.*[22)] and Matsumura *et al.*[23, 24)] reported surface structures of typical coconut shell activated carbons for adsorption of organic vapors available at the time in Japan. The specific surface areas of the carbons measured by N_2 gas in their reports were approximately 900–1,300 m^2/g, whereas those of the activated carbon specimens in this research were higher (Table 1). Technical advances have increased the porosity and specific surface area of activated carbons and also this seems to have increased the moisture adsorption effect on the carbon. This is an important issue which should be investigated in the days ahead.

Figure 4 shows the adsorption capacity of S1, which was preconditioned with gradual different moisture adsorption until equilibrium, for 6 kinds of organic vapor. In the figure, the breakthrough time of each measurement is plotted as a function of the preliminary water adsorption in percentage at the saturated state by the specimen. In the saturated state, the amount of water adsorption in the range 0–50% corresponds to an equilibrated relative humidity of airflow in the range of 30–70% (Fig. 3 (a)). In the previous reports[1–14)], the relative humidity of the test gas flow was taken as the index of adsorption capacity in relation to the breakthrough time. However, the weight increase by preliminary moisture adsorption relative to the weight before preconditioning is another simple indicator of the adsorption capacity of the carbon bed, and we have used this indicator for the breakthrough time in the figures. The adsorption capacity of the specimen S1 shows a gentle decrease for the organic vapors in the range of 0–30% water adsorption, and the declining adsorption capacities are roughly approximated by straight lines, which are shown by broken lines in Fig. 4. The declining tendency of adsorption capacity at low relative humidity is consistent with the results of the previous reports by Nelson *et al*[3–5)]. However, the adsorption capacity of the specimens for the organic vapors showed a sharp decrease at water adsorption of 30% or higher in many cases of measurement. This critical point of water adsorption seems to be significant for the practical use of respirators, and it also approximately corresponds to an equilibrated atmosphere with relative humidity of 58% (Fig. 3 (a)). It is important to mention here that relative humidity of 58% is not uncommon in Japanese climate. Furthermore, we found that the plots agreed well with quadratic functions shown by solid lines in Fig. 4, approximating a wide range of water adsorption in the graphs.

Absolute values of coefficients of correlations of linear ($|R_1|$) and quadratic ($|R_2|$) function approximations for the organic vapors are listed in Table 5. In Fig. 4, the data plots show comparatively good agreement with linear



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

Fig. 4. Effect of preliminary water adsorption on breakthrough time for organic vapors (300 ppm) of the specimen S1 and linear and quadratic function approximations for the data.

The interval of linear function approximation (broken lines) is approximately from 0 to 30%, and that of quadratic function approximation (solid lines) is all range of water adsorption. The breakthrough time was measured by a test gas flow which had the same relative humidity as the preconditioning airflow.

approximations in the range below 30% of water adsorption. However, on the whole, the quadratic approximation shows better agreement with the breakthrough time plots than the linear approximation, and it is remarkable that the relative weight increase due to moisture adsorption of the carbon specimen has a clear quantitative effect on the adsorption capacity for the organic vapors. We consider that these results will also be useful for the practical handling and storing of coconut shell activated carbons, because several types of organic substances (chain hydrocarbons, ketone compounds, aromatics and cyclic compounds) showed a common pattern in the results. We will further investigate the approximations of the breakthrough time plots and the effect of moisture content with additional measurements using other organic vapors in a following paper.

Table 5. Absolute values of coefficients of correlations of approximated curves or lines in Fig. 4.

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
Cyclohexane	0.9954	0.9982
Benzene	0.9979	1.0000
Toluene	0.9936	0.9961

Conclusion

We have measured the adsorption capacity as the breakthrough time of coconut shell activated carbons, which had been 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. We first prepared several carbon specimens with moisture at different levels by equilibration with humidified airflow at certain steady state moisture content. Then, the breakthrough time of each specimen for 6 kinds of organic vapor was measured using a test gas flow which had the same relative humidity as the preconditioning airflow. The test gas flow contained organic vapor concentration of 300 ppm and had relative humidity ranging from 30 to 70%.

The adsorption capacities of the specimens decreased sharply at water adsorption of 30% and higher state in many cases of our measurements. In addition, we found that the data plots of the breakthrough times versus water adsorption showed good agreement with quadratic functions approximating a wide range of water adsorption in the graphs. We note that the relative weight increase due to moisture adsorption of the carbon specimens had a clear quantitative effect on the breakthrough time for the organic vapors.

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