

# ALDEHYDES ADSORBENT PRODUCED BY CARBONIZATION OR ACTIVATION OF RICE HUSK

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## Abstract

Rice husk (RH) was converted into an adsorbent intended for use in the removal of two types of aldehyde vapors. Japanese RH was carbonized at 250-800°C in N<sub>2</sub> or was activated at 875°C in CO<sub>2</sub>. The carbonized and the activated RHs were exposed to formaldehyde vapor at 1.0 vol. ppm or acetaldehyde vapor at 100 vol. ppm (both N<sub>2</sub> carrier) in enclosed 5 L-gas sampling bags. The RH activated at 875°C reduced the concentrations of formaldehyde and acetaldehyde in the bags the fastest. The time-rate of the aldehydes adsorption of the activated RH was higher than that of the commercial granular coconut-shell activated carbon (GCSAC), whereas the specific surface area and the total pore volume of the activated RH (466.9 m<sup>2</sup>/g and 0.35 cm<sup>3</sup>/g) was much lower than that of the GCSAC (1119.7 m<sup>2</sup>/g and 0.57 cm<sup>3</sup>/g). The surface basic property of the activated RH and the RH carbonized at high temperature attributed to the intrinsic inorganic matters of K and Ca was useful to enhance an uptake of the aldehydes on their surfaces.

## Introduction

Aldehydes are common indoor air pollutants which can be given off from construction materials, mainstream smoke of cigarettes and so on (WHO Europe, 2001). In particular, formaldehyde (HCHO) and acetaldehyde (CH<sub>3</sub>CHO) have been well recognized as toxic volatile organic compounds which can cause human's allergy and serious diseases. The Japan Society for Occupational Health (2006) determines the occupational exposure limits of formaldehyde and acetaldehyde, which are recommended as reference values for preventing adverse health on workers, to be 0.5 and 50 vol. ppm (hereafter simply ppm), respectively. Activated carbons (ACs) have been studied for the sake of adsorption of formaldehyde (Doming-Gracia et al., 1999) and acetaldehyde (El-Sayed and Bandosz, 2002a). The adsorption of aldehydes on oxide such as silica gel (Ghosh and Hines, 1990) and silica (Natal-Santiago et al., 1999) has been also studied.

Japan produced 10.9 million tons of rice grain in 2004 (Food and Agricultural Organization of UN, 2006). A large quantity of rice husk, one-fifth by mass of rice grain production, is also produced every year. The most common disposal method of rice husk (RH) was incineration on farms. However, incineration of RH on farms is now prohibited in Japan because it produces ash, fumes, and toxic organic gases, leading to serious air pollution. It is socially beneficial to convert RH considered to be agricultural waste in Japan into an adsorbent intended for use in the removal of formaldehyde and acetaldehyde from the atmosphere.

The characteristic nature of RH is high-content inorganic matters. The content of the RH ash, which is most composed of SiO<sub>2</sub>, is about 20 mass% (Kumagai et al., 2007). Carbonization or activation process can convert RH into a composite made of carbon and silicate. The present study investigated the aldehydes adsorption performance of carbonized or activated RHs with an emphasis on realistic service conditions. The aldehydes adsorption of the carbonized RH or the activated RH in an enclosed bag in which ambient pressure was maintained was observed in relation to its pore structure and its chemical composition.

## Experimental Methods

### Material

A RH sample was obtained by rice threshing at Akita Komachi rice grown in Nishiki-mura (Akita Prefecture, Japan) in autumn of 2005. The raw RH was carbonized or activated in a stainless steel cylinder (SUS 304) whose respective external and bore diameters were 50 and 44 mm using an electrical furnace (KT-1053R; Advantec Toyo Kaisha, Tokyo, Japan).

The raw RH was carbonated at 250, 400, 600, and 800°C in N<sub>2</sub> flow at 500-700 mL/min. The raw RH was heated from room temperature to the desired carbonizing temperature in 1 h at the constant heating rate; it was then maintained at the desired temperature for 1 h. It was then cooled naturally to room temperature. In order to produce the activated RH, the raw RH was heated to 875°C in N<sub>2</sub> flow at 500-700 mL in 1h at the constant heating rate; it was maintained at 875°C in CO<sub>2</sub> flow at 500-700 mL for 1 h and cooled naturally to room temperature. The reason why 875°C was chosen for the activation temperature was that it provided the largest specific surface area in the tested activation temperatures. The RH ash was also produced by heating the raw RH at 800°C in atmospheric air. The heating and cooling manners were similar to those for producing the carbonized RH

Commercial granular coconut-shell activated carbon (GCSAC) was also prepared for a comparison purpose. Granule size of the GCSAC (GW10/32, Kuraray Chemical Co., Ltd., Osaka, Japan) was adjusted to be 1-3 mm using sieves. This was also subjected to the tests and analyses which were implemented for the heat-treated RHs.

### **Material Characterization**

The ash content of the raw RH, the carbonized RH, and the activated RH (all weighed 0.1 g) was precisely determined from the residual-ash ratio after incineration at 800°C for 1 h in air flow (100 mL/min) using a thermogravimetric analyzer (TGA-51; Shimadzu Corp., Japan). A CHN/S analyzer (2400 I; PerkinElmer Inc., USA) was used to determine the hydrogen, carbon, nitrogen and sulfur contents of the samples. The composition of the RH ash was determined using a fluorescent X-ray analyzer (XRF-1700; Shimadzu Corp., Japan). The content of oxygen excluding that in ash was determined to be a difference of hydrogen, carbon, nitrogen, sulfur, and ash. All the samples were dried at 105°C for 3 h prior to analyses. It was verified beforehand that the above periods were sufficient to saturate the sample weights in drying.

A gas-adsorption analyzer (Autosorb-3B; Quantachrome Instruments, FL, USA) was used to evaluate the sample porosity, correlating it with the aldehydes adsorption property. N<sub>2</sub> adsorption isotherms, providing the surface area based on the BET theory (Bansal and Goyal, 2005), and the total pore volume at a relative pressure of 0.995 were measured. The samples (ca. 0.02 g) were degassed at 200°C for > 4 h prior to isotherm measurement. The BET surface area ( $S_{\text{BET}}$ ) was calculated using the N<sub>2</sub> adsorbed volume at relative pressures ( $P/P_0$ ) of 0.05–0.30. The liquid N<sub>2</sub> volume, referring to N<sub>2</sub> adsorbed volume at  $P/P_0$  0.995, was determined to be the total pore volume ( $V_t$ ). The volume of micropores of which pore diameter is <2 nm was obtained following the t-method (Do, 1998), giving the volume of external pores including mesopores and macropores ( $V_e = V_t - V_m$ ).

### **Formaldehyde and Acetaldehyde Adsorption Tests**

After the drying process, the sample was enclosed quickly in a gas-sampling bag made of polyvinyl fluoride film (Tedlar®; DuPont, USA) and residual air in the bag was degassed using a small pump for 3 min. The formaldehyde vapor adjusted at the concentration of 1.0 ppm or acetaldehyde vapor at 100 ppm (dry nitrogen gas based, <1% RH at 22–24°C) provided by Taiyo Nippon Sanso Corp., Tokyo, Japan, was injected 5 L to the degassed bag. These concentrations are both twice higher than the occupational exposure limits (OEL) determined by the Japan Society for Occupational Health, simulating likely service conditions such in an air purification process for industrial occupations. The contact time started when the gas injection started. Kitagawa-type detecting tubes targeting formaldehyde (Komyo Rikakagaku Kogyo K.K., Kawasaki, Japan) and Gastec-type detecting tubes targeting acetaldehyde (Gastec Corp., Ayase, Japan) were used to measure each aldehyde concentration in the bag. The gas of 200 mL was evacuated from the bag for every measurement. The gas in the bag was maintained at ambient pressure during the adsorption test, which was confirmed using a manometer (PG-100, Copal Electronics Co., Ltd, Tokyo, Japan). The concentration in the bag was evaluated at 22–24°C as a function of the contact time.

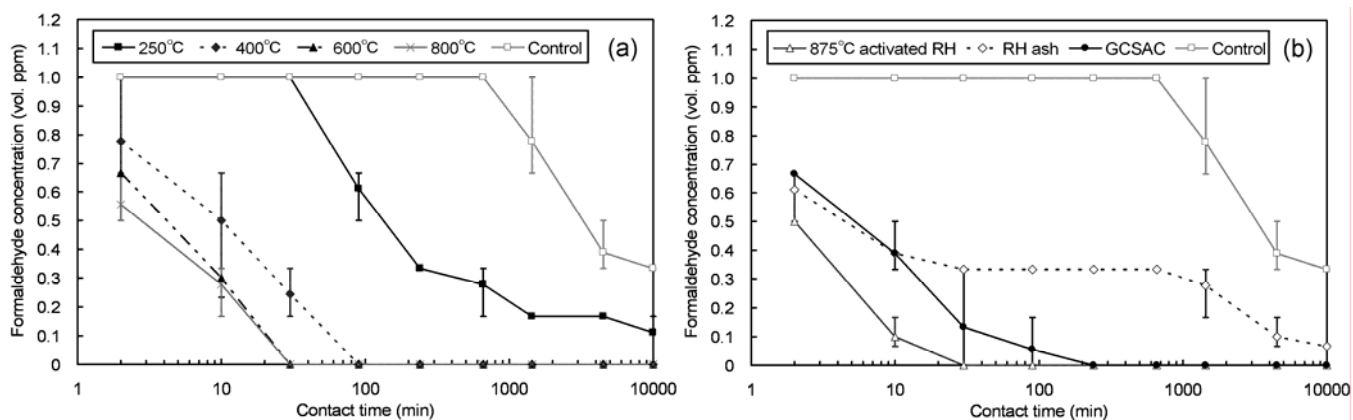
## **Results and Discussion**

### **Formaldehyde and Acetaldehyde Gas Adsorption Properties of Heat-treated RHs**

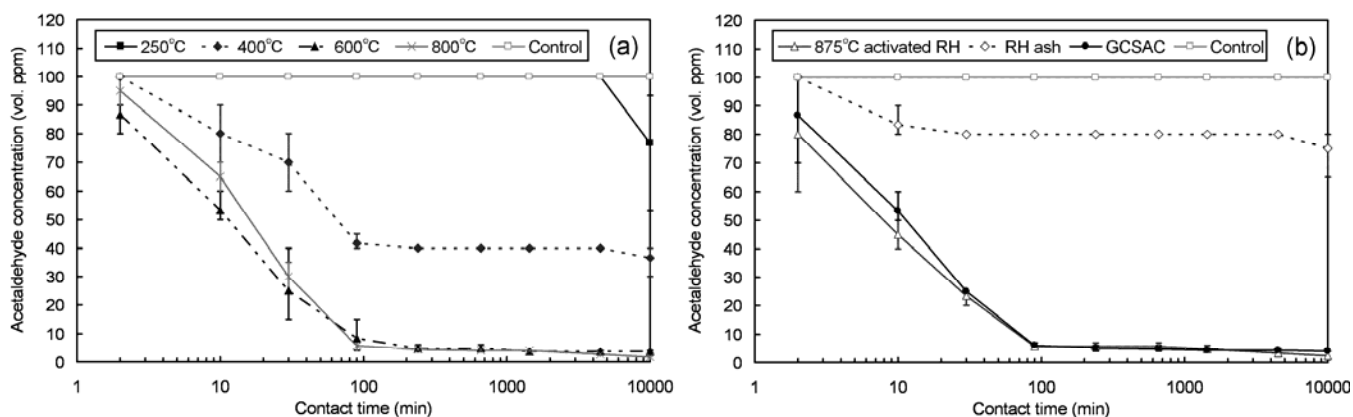
The formaldehyde and acetaldehyde concentrations in the gas-sampling bags of 5 L in which the heat-treated RH was enclosed were measured in order to observe their aldehydes adsorption around the respective OEL levels.

**Figure 1** shows the formaldehyde adsorption property of the RHs carbonized at 250–800°C, the RH activated at 875°C, the RH ash, and the GCSAC, of which weights were all adjusted to be 1.00 g. The control indicates the formaldehyde concentration in the gas in which no sample was enclosed. It is found that the carbonizing temperature had a large influence on the time-rate of the formaldehyde adsorption of the carbonized RHs. The RH carbonized at the lowest temperature (250°C) required the longest time (>100 min) to reduce the formaldehyde concentration to the OEL level (0.5 ppm). The time-rate of the formaldehyde adsorption of the carbonized RHs increased with increasing the carbonizing temperature. The fastest formaldehyde adsorption appeared on the activated RH, requiring 2 min to reduce the concentration to the OEL level, and 10 min to 0.1 ppm. Although the RH ash reduced the formaldehyde concentration to 0.4 ppm at the early period (<10 min), further formaldehyde adsorption was not observed. This result suggests that the adsorption capacity of the RH ash was not large compared to the other heat-treated RHs. The time-rate of the GCSAC, employed as a reference, was lower than that of the activated RH; it is approximately similar to that of the RH carbonized at 600°C.

**Figure 2** shows the acetaldehyde adsorption behavior of the heat-treated RHs and the GCSAC. The weights were also all adjusted to be 1.00 g. With increasing the carbonizing temperature, the time-rate of the acetaldehyde adsorption increased at 250–600°C, whereas it slightly decreased at 800°C. The RH carbonized at 600°C displayed the fastest acetaldehyde adsorption in the carbonized RHs. The acetaldehyde adsorption of the RH carbonized at the lowest temperature (250°C) was minor. The fastest acetaldehyde adsorption appeared on the activated RH, which was identically observed on the formaldehyde adsorption test. The acetaldehyde concentration was reduced to the OEL level (50 ppm) in 8 min, and to 10 ppm in 70 min. The slight acetaldehyde adsorption was observed on the RH ash, in which the acetaldehyde concentration was not reduced to <80 ppm during the adsorption test. The acetaldehyde adsorption performance of the GCSAC was inferior to that of the activated RH. It was on a level with that of the RH carbonized at 600°C.



**Figure 1.** Formaldehyde adsorption of heat-treated rice husks (RHs) and commercial activated carbon in enclosed bags. (a) RHs carbonized at different temperatures for 1 h, (b) RH activated at 875°C for 1 h, ash of RH and granular coconut-shell activated carbon (GCSAC). Control is a result with no sample in the enclosed bag. Data are the averages of three separate measurements. The error bars indicate the maximum and minimum values of the three measurements.



**Figure 2.** Acetaldehyde adsorption of heat-treated rice husks (RHs) and commercial activated carbon in enclosed bags. (a) RHs carbonized at different temperatures for 1 h, (b) RH activated at 875°C for 1 h, ash of RH and granular coconut-shell activated carbon (GCSAC). Control is a result with no sample in the enclosed bag. Data are the averages of three separate measurements. The error bars indicate the maximum and minimum values of the three measurements.

### Chemical and Pore Characteristics of Heat-treated RHs

The formaldehyde and acetaldehyde adsorption of the heat-treated RHs is inferred to be correlated with their chemical compositions and pore structures. Changes in the composition and yield of the RH due to the carbonization and activation processes are shown in **Table 1**. It is found that the ash content of the raw RH was 17.1 mass%, and higher carbonizing temperature led to higher ash content of the samples. The highest ash content was observed on the activated RH, which was more than half of the mass. The yield of the RHs carbonized at 600 and 800°C was <40 mass%. The changes in composition of the carbonized RHs indicate that the hydrogen and oxygen contents attributable to the ligneous composition decrease with the carbonizing temperature. The activation process resulted in the lowest yield (26.5 mass%) and further lowering of hydrogen, carbon, and oxygen contents. The primary component of the activated RH was not carbon, but inorganic matters related to the ash formation. The GCSAC was found to be most composed of carbon. The ash content of the GCSAC was much lower than those of the heat-treated RHs. **Table 2** shows the composition of the RH ash determined using a fluorescent X-ray analyzer. It is found that most inorganic matters contained in the raw RH were Si, K and Ca. The components of K and Ca were probably provided from soil fertilizers.

Pore structures of the heat-treated RHs and the GCSAC were also analyzed. The specific surface area ( $S_{\text{BET}}$ ) and the volume of several types of pores ( $V_t$ ,  $V_m$  and  $V_e$ ) were obtained and tabulated in **Table 3**.  $S_{\text{BET}}$  and  $V_t$  of the carbonized RHs increased with the carbonizing temperature at 250–600°C and decreased at 800°C. The carbonization at 600°C resulted in very porous structure, in which micropores were most generated in the carbonized RHs. The declined  $S_{\text{BET}}$  and  $V_t$  appearing on the RH carbonized at 800°C were attributed to the decrease in  $V_m$ , which was related to a degeneration of micropores. The largest  $S_{\text{BET}}$ ,  $V_t$  and  $V_m$  in the heat-treated RHs, which were about 2.5-fold greater than those of the RH activated at 600°C, were observed on the activated RH. It is interesting to note that the RH ash had a large  $V_t$  attributed to a large volume of

external pores. The GCSAC had the largest  $S_{BET}$ ,  $V_t$  and  $V_m$  in all the samples. A large volume of micropores was responsible for the largest  $S_{BET}$  and  $V_t$ . The highest time-rate of the aldehydes adsorption was not observed on the GCSAC, but on the activated RH. The time-rate of aldehydes adsorption of the GCSAC was comparable to that of the RH carbonized at the high temperature (600 or 800°C). It is therefore revealed that the aldehydes adsorption performance of the tested samples was not simply determined by their porosity.

**Table 1.** Compositional changes and yields of the rice husks (RHs) carbonized at different temperatures for 1 h, the RH activated at 875°C for 1 h, and the commercial granular coconut-shell activated carbon (GCSAC). Unit: mass%. The content of oxygen except for that in ash is calculated:  $O = 100 - (\text{Ash} + H + C + N + S)$ . Data are the averages of three separate measurements.

Sample		Ash	H	C	N	O	S	Yield
Raw RH		17.1	5.2	40.5	0.4	36.1	0.7	100
Carbonizing temperature of RHs (°C)	250	17.6	5.1	42.2	0.5	33.9	0.7	93.0
	400	32.2	2.7	47.5	0.5	16.6	0.4	46.1
	600	39.8	1.4	51.3	0.5	6.9	0.2	38.4
	800	42.6	0.4	51.0	0.4	5.6	0.1	35.9
875 °C activated RH		58.8	0.1	38.3	0.4	2.3	0	26.5
GCSAC		0.5	0.4	92.8	0.3	5.9	0.1	-

**Table 2.** Composition of the ash produced by incineration of raw RH. Unit: mass%. Data are the averages of three separate measurements.

SiO <sub>2</sub>	K <sub>2</sub> O	CaO	P <sub>2</sub> O <sub>5</sub>	Detectable others
91.1	5.3	1.6	0.6	1.4

**Table 3.** Pore characteristics of the rice husks (RHs) carbonized at different temperatures for 1 h, the RH activated at 875°C for 1 h, the RH ash, and the commercial granular coconut-shell activated carbon (GCSAC). Data are the averages of three separate measurements.

Sample		$V_t$ (cm <sup>3</sup> /g)	$V_m$ (cm <sup>3</sup> /g)	$V_e$ (cm <sup>3</sup> /g)	$S_{BET}$ (m <sup>2</sup> /g)
Carbonizing temperature of RHs (°C)	250	0.04	0	0.04	10.9
	400	0.06	0	0.06	18.3
	600	0.14	0.05	0.09	166.0
	800	0.11	0.02	0.09	120.3
875 °C activated RH		0.35	0.12	0.23	466.9
RH ash		0.28	0	0.28	134.6
GCSAC		0.57	0.43	0.14	1119.7

### *Mechanisms of Adsorption of Formaldehyde and Acetaldehyde Vapors on Heat-treated RHs*

The formaldehyde and acetaldehyde adsorption tests under the concentrations around the respective OEL levels (0.5 and 50 ppm) were implemented for the heat-treated RHs. Higher carbonizing temperature was useful to promote both the aldehydes adsorption. The highest time-rate appeared at the carbonizing temperature of 800°C for the formaldehyde adsorption and 600°C for the acetaldehyde adsorption. The activated RH showed the highest time-rate for both the aldehydes adsorption, which was superior to the commercial GCSAC. The RH ash had decent adsorption abilities for both the aldehydes adsorption, with a susceptibility to formaldehyde adsorption.

Rong et al. (2002) and Boonamnuyvitaya et al. (2005) reported that surface hydrophilic and polarized oxygen-containing functional groups as well as large specific surface area and pore volume were useful to enhance the formaldehyde adsorption on ACs, which were verified using coffee residue-based ACs and oxidized Rayon-based AC fibers, respectively. The dipole interaction and hydrogen bonding between the formaldehyde and the oxygen containing functional groups were responsible for the increase in the formaldehyde uptake. EI-Sayed and Bandosz (2001) showed that surface oxygen-containing functional groups had a strong interaction with acetaldehyde, resulting in the increased acetaldehyde uptake. An interaction between acetaldehyde molecules and hydrocarbon moiety with the AC pore walls was also a noticeable factor to determine the acetaldehyde adsorption. They also mentioned that micropores in ACs provided predominantly active centers for the acetaldehyde adsorption process. Therefore, an increase in the micropore volume led to an increase in the acetaldehyde uptake (EI-Sayed and Bandosz, 2002b). The molecular size of formaldehyde is smaller than that of acetaldehyde, meaning that predominant adsorption sites for formaldehyde should be also micropores.

Yang et al. (2005) studied the adsorption of formaldehyde and acetaldehyde vapors using silica gel. Silica gel can adsorb aldehydes due to hydrogen bonding between silanol groups on the surface of silica gel and aldehyde groups. They further described that silica gel adsorbed acetaldehyde more effectively than formaldehyde, which were related to the pore size of the used silica gel. Busca et al. (1987) studied the role of basic sites on the adsorption and the transformation of formaldehyde on inorganic oxides using FTIR spectroscopy. It was conformed that basic sites such as MgO could polymerize formaldehyde into  $(-CH_2O-)_n$ , finally leading to transformations into methoxy groups and formate ions. Transformations of acetaldehyde at the basic sites on oxides should be also likely (Kiss, 2005).

The high ash content was observed on the RH carbonized at high temperatures (600 and 800 °C) and the activated RH, showing the high time-rate of both formaldehyde and acetaldehyde adsorption. Si, K, and Ca were identified in the RH ash, indicating that K and Ca might provide basic character on the carbonized and the activated RH. Therefore, surface pH of the samples was evaluated by adding it into distilled water. The dried sample of 0.05 g was added to distilled water of 100 mL. Distilled water containing the sample was stirred for 1 h and then conserved for 48 h, during which the temperature of the distilled water was hold at 25°C. The sample was filtered and the pH of solution was measured, providing information about the acidity and basicity of the sample surface. **Table 4** shows surface pH of the heat-treated RHs and the GCSAC. The pH of the distilled water added with the samples was higher than that of distilled water with no sample (control), indicating that all the sample surfaces had a basic property. The surface pH of the samples is found to be closely consistent with the ash content shown in **Table 1**. **Table 2** suggested that principal compositions identified in the RH ash were SiO<sub>2</sub>, K<sub>2</sub>O, and CaO. The surface basicity of the heat-treated RHs was attributed to intrinsic inorganic matters of K and Ca.

**Table 4.** Surface pH of the rice husks (RHs) carbonized at different temperatures for 1 h, the RH activated at 875°C for 1 h, the RH ash, and the commercial granular coconut-shell activated carbon (GCSAC). Control is a result with no sample in distilled water. Data are the results of one measurement.

Sample		pH
Carbonizing temperature of RHs (°C)	250	5.3
	400	5.7
	600	6.1
	800	6.3
875 °C activated RH		7.5
RH ash		9.0
GCSAC		5.2
Control		5.1

The compositional analysis also suggested that the carbonized and the activated RHs contained the remaining hydrogen and oxygen excluding that in ash, indicating that oxygen-containing groups existed in carbon and/or silicate matrices in the carbonized and the activated RHs. The contents of hydrogen and oxygen excluding that in ash were found to decrease with the carbonizing temperature. The activation process resulted in further lowering of those contents.

The RH ash most composed of the intrinsic inorganic matters showed a small capacity of the aldehydes adsorption, in which smaller capacity was observed on the acetaldehyde adsorption. Although the RH ash had a relatively large total pore volume, it had no micropores. Hence, it was reasonable to understand that the aldehydes uptake of the RH ash was due to basic character. Formaldehyde showed a stronger sensitivity to adsorption on the RH ash than acetaldehyde, which was inferred to be due to higher hydrophilicity of formaldehyde. Silanol groups on SiO<sub>2</sub> in the RH ash might contribute to the aldehydes adsorption, which cannot be verified in detail in the present study.

The porosity evaluation suggested that the RH carbonized at 400°C had very small total pore volume and no micropores. Therefore, the aldehydes adsorption on the RH carbonized at 400°C can be explained by a formation of oxygen-containing groups. The effect of basic property resulting from K and Ca seemed to be minor. The aldehydes adsorption on the RH carbonized at 600 and 800°C, and on the activated RH was a combination adsorption resulting from micropores, oxygen-containing functional groups both in carbon and silicate matrices, and surface basic character. A contribution of oxygen-containing groups to their aldehydes adsorption was minor because their contents of remaining hydrogen and oxygen were reduced as shown in **Table 1**. The micropores volume of the RH carbonized at 600°C was larger than that at 800°C, while their external pore volumes were comparable. This indicates that micropore-filling was effective for the acetaldehyde adsorption, which was probably due to its lower hydrophilicity than that of formaldehyde. The activated RH showed the largest micropore volume and the highest surface basicity in the heat-treated RHs, leading to the highest time-rate of both the aldehydes adsorption. The largest micropore volume in all the samples was observed on the GCSAC. However, the surface of the GCSAC was neutral. The combination of micropore and surface basic property of the activated RH was shown to be useful to the aldehydes adsorption, with a higher time-rate than that of the GCSAC. It is finally concluded that such the useful properties to remove formaldehyde and acetaldehyde vapors from ambient air were obtainable from agricultural waste of RH.

## Conclusions

Japanese RH was carbonized at 250, 400, 600, and 800°C in N<sub>2</sub> gas or was activated at 875°C in CO<sub>2</sub> gas. The carbonized RH, the activated RH, and the RH ash were exposed to formaldehyde gas at 1.0 vol. ppm or acetaldehyde gas at 100 vol. ppm (both N<sub>2</sub> gas carrier) in enclosed 5 L-bags in which ambient pressure was maintained. The RH activated at 875°C reduced the concentrations of formaldehyde and acetaldehyde vapors in the bags the fastest. The time-rate of the aldehydes adsorption of the activated RH was higher than that of commercial GCSAC, whereas the specific surface area and the total pore volume of the activated RH was much lower than that of the GCSAC. The surface basic property of the activated RH and the RH carbonized at high temperature provided from intrinsic inorganic matters of K and Ca was useful to increase the time-rate of the aldehydes adsorption. A composite of basic surface property resulting from the inorganic matters and microporous structure resulting from carbon in RH ligneous composition was beneficial to the aldehydes adsorption. Agricultural waste of RH was shown to be capable of providing such beneficial properties to remove formaldehyde and acetaldehyde vapors.

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