Efect of Potassium Hydroxide Concentration and Activation Time on Rice Husk-Activated Carbon for Water Vapor Adsorption

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Abstract: Rice husk carbon by-product from the industrial combustion is a promising source to produce a vast amount of activated carbon adsorbent. This research prepared rice husk-activated carbon adsorbent by varying the concentration of potassium hydroxide solution (5, 10, 15, 20 % w/v) and activation time (2, 4, 6, 8 hours). Fourier-transform infrared spectral characterization (FTIR) indicated a significant effect before and after activation, especially the presence of hydroxyl groups. Based on the iodine adsorption, the specific surface area of the produced-activated carbon was approximately 615 m^2/g. Experimental results showed that increasing potassium hydroxide concentration and activation time increases the water vapor adsorption capacity of the activated carbon. Compared with the rice husk carbon, the KOH-activated carbon enhanced the water vapor adsorption capacity to 931%. In the adsorption observation, changing the temperature from 15 to 27 ℃ caused a higher water vapor uptake onto the activated carbon. Two adsorption kinetics (pseudo-first- and pseudo-second-order models) were used to evaluate the adsorption mechanism. This research found that rice husk-activated carbon performed a higher water vapor adsorption capacity than other adsorbents (silica gel, zeolite, and commercially activated carbon).

Keywords: *activated carbon, adsorption kinetics, desiccant, rice husk, water vapor adsorption*

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INTRODUCTION

Water vapor adsorption is an essential process in several industrial practices, such as water harvesting [1], dehumidification [2], and desalination. As an example, in the dehumidification drying process, reducing water vapor from air enhances the driving force, which can shorten the drying time and retain heat-sensitive ingredients in the food product [3]. This is due to the significant difference between the concentration of water vapor on the food's surface and the air as the drying medium. Then, the mass transfer of water vapor from the product to the air can be fastened.

The need for water vapor adsorption led to the production of efficient materials for moisture control. Some studies presented different types of water adsorbents, including Covalent Organic Frameworks [4], Metal-Organic Frameworks (MOFs) [5], zeolite [6], silica gel [7], mesoporous silica [8], and activated carbon [9]. Covalent Organic Frameworks (COFs) and Metal-Organic Frameworks (MOFs) are mostly used in the water harvesting process because of their remarkable adsorption properties, which are highly tunable and high porosity [10]. However, those materials require careful handling due to potential structural damage and complex synthesis processes, making them relatively higher cost compared to other adsorbents [11]. Meanwhile, widely known water vapor adsorbents or desiccants (zeolite, silica gel, and activated carbon) are easily regenerated and more stable in structure.

There are several requirements in the adsorbent selection, including toxicity, adsorption capacity, cycles, and easiness in regeneration using low heat energy. Activated carbon is a non-toxic and thermally stable adsorbent with a high water adsorption capacity due to its large surface area [12]. Also, activated carbon can be produced from natural sources such as agricultural by-products. Thus, activated carbon is a promising adsorbent due to its abundance, low cost, and potential for sustainable water vapor removal.

Rice husk is a natural adsorbent that has been applied for various applications, including heavy metal and dye removal in wastewater [13], [14], CO_2 adsorption [2], and urine purification [15]. In its application as a water vapor adsorbent, rice husk was exploited by Warsiki et al. [16] that produce rice husk-$CaCl_2$ composite desiccant. They reported that environmental humidity (water activity) and temperature influenced the adsorption capacity of the adsorbent. However, the study did not compare rice husk-$CaCl_2$

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adsorption with other adsorbents. Moreover, untreated rice husk still contains contaminants in its pores and thus is still low in adsorption capacity. Another research showed that rice husk silica had the potential as a silica gel substitute in water vapor adsorption [17]. The adsorption mechanism of rice husk silica and silica gel was similar and affected by silanol groups on their surface. Nevertheless, commercial silica adsorbed twice as much water vapor than silica produced from rice husk. Therefore, this research focuses on synthesizing natural-based adsorbent with high water vapor adsorption.

This work aims to produce activated carbon from rice husk as a water vapor adsorbent and its comparison with other commercial adsorbents (commercial activated carbon, silica gel, and zeolite). In the process, several studies used potassium hydroxide, zinc chloride, hydrochloric acid, and sodium hydroxide as activation agents to activate rice husk carbon [18], [19], [20]. In this study, potassium hydroxide (KOH) was selected as the activation agent to increase the pore volume and adsorption capacity of the adsorbent water vapor [21]. The activation agent concentration and the activation time were studied to assess the water vapor adsorption property.

EXPERIMENTAL PROCEDURE

Materials

Rice husk char was bought from a local shop in Pedurungan, Semarang, Central Java. The char was sun-dried before being activated. The experiment was conducted using potassium hydroxide (90% purity, technical grade), hydrochloric acid (concentration of 32%, technical grade), distilled water, iodine solution (concentration of 1%, Merck & Co., Inc.), sodium thiosulfate (98% purity, Merck & Co., Inc.), and amylum (99% purity, Merck & Co., Inc.). Commercial adsorbents that were used as a comparison were coal-based commercial activated carbon (size of 4 – 8 mesh), white silica gel (size of 2 – 4 mm), and natural zeolite bought from a local shop (CV. Indrasari, Semarang, Central Java).

Carbon Activation

Before activation, rice husk char was prepared by pulverizing and sieving to 20 and 25 mesh. Char activation was conducted by adding 25 grams of rice husk char to 150 mL of 5% w/v KOH solution and

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letting it stand for 8 hours. The activated char (activated carbon) was filtered and washed using HCl and distilled water until the washing solution achieved a neutral pH. The wet activated carbon was dried in an oven at 110°C to the achievement of a constant mass and was placed in a desiccator. The activation procedure was repeated using KOH solution at different concentrations (10, 15, and 20% w/v) and activation times (2, 4, and 6 hours).

Iodine Number Adsorption

The iodine adsorption experiment was begun by mixing the activated carbon and 0.1 N iodine solution for 10 minutes. After the filtration, 10 mL of filtrate was titrated with 0.1 N sodium thiosulfate ($Na_2S_2O_3$) until became light yellow. The solution was then titrated by 1% amylum until clear from the blue color. The iodine adsorption capacity was calculated by the equation of (Suliestyah et al., 2020)

$$\text{Iodine number} = \frac{(V_1N_1 - V_2N_2)126.9 \times 5}{W} \tag{1}$$

with V_1 and V_2 are volume of iodine and sodium thiosulfate (mL), respectively, N_1 is the normality of iodine (N), N_2 normality of sodium thiosulfate (N), and W is the mass of the sample (g).

Adsorption capacity

Fig. 1 describes the experimental setup of the adsorption test. The adsorption capacity of the produced activated carbon was evaluated by placing 10 grams of activated carbon in an isolated mason jar. Previously, the jar was filled with a specific amount of water. The adsorption capacity was the total mass of water adsorbed into the sample that was measured by the gravimetric method. This test was conducted under temperatures of 15 °C, room temperature (27 °C), and 40 °C for several days until equilibrium. In comparison, several commercial adsorbents were also examined with iodine and water vapor adsorption. The adsorption kinetics was evaluated using several models presented in Table 1. The adsorption capacity of prepared activated carbon was then compared to other commercial adsorbents (commercial activated carbon, natural zeolite, and silica gel).

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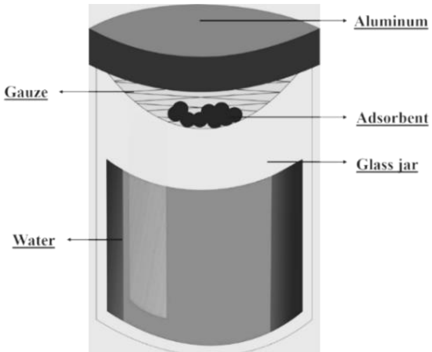

Fig. 1. Experimental set-up of the adsorption capacity evaluation

Table 1. Adsorption kinetics equation (Gurses et al. 2006; Qiu et al. 2009)

Model	Equation
Pseudo-first-order	$q_t = q_e(1 - e^{-k_1 t})$
Pseudo-second-order	$q_t = \dfrac{k_2 {q_e}^2 t}{1 + k_2 q_e t}$

Notes: q_t = moisture adsorption at a certain time; q_e = moisture adsorption at equilibrium; t = adsorption time; k_1 = constant parameter of pseudo-first-order; k_2 = constant parameter of pseudo-second-order

Materials Characterization

The functional groups in the activated carbon samples were examined using Fourier Transform Infrared Spectroscopy (FTIR) employing PerkinElmer Frontier Infrared Spectrometer version 10.6.1, with a resolution of 1 cm^{-1} in a region of 400 to 4000 cm^{-1}. X-ray diffractions (XRD PANalytical X'Pert PRO, Malvern Panalytical Ltd.) with CuKα radiation of 1.54060Å were used to investigate the crystallinity of natural zeolite. This instrument operated at 40 kV, 30 mA and the diffractograms were observed from 10.03° to 82.19° on a 2θ scale with a 0.7° step size. The chemical compositions of natural zeolite were observed using X-ray Fluorescence (Panalytical Minipal 4, Malvern Panalytical Ltd.). Three random samples were taken from the same source as the zeolite samples, named Zeolite A, B, and C. The crystal structure and the chemical composition of natural zeolite were shown in XRD patterns and XRF analysis in Fig. 2. According to XRD patterns evaluated by Highscore plus 3.0e (PANalytical B.V., The Netherlands), the peaks of zeolite samples indicate similar crystal structure (mordenite). This result was compatible with XRF analysis that showed a common compound of mordenite mineral (Na_2, Ca, K_2) $Al_2Si_{10}O_{24}\cdot 7H_2O$ [22].

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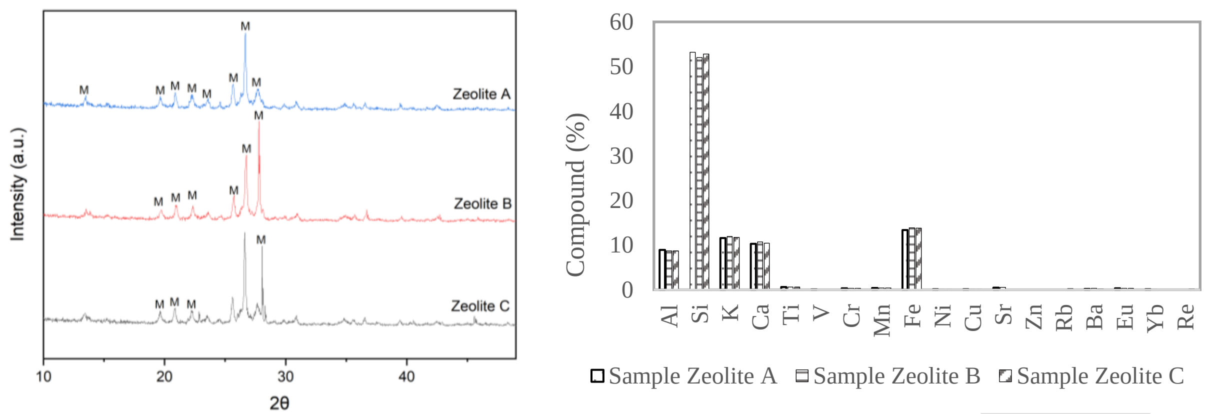

(a) (b)

Fig. 2. (a) XRD patterns of natural zeolite used in this study (b) compounds found in natural zeolite used in this study

Results and Discussion

Fourier transform infrared (FTIR) characterization

Fig. 3 shows the FTIR spectra of rice husk char and activated carbon, which highlight the chemical functional groups. Several functional groups with water affinity were discovered by this characterization, as well as some water that had bound to the activated carbon. A clear difference between the two samples was a higher peak of aromatic ring C=C at 1600 – 1500 cm^{-1} in the activated carbon and a C-O band of rice husk char at 1300 – 1050 cm^{-1} [23]. A C-H functional group at a wavenumber of 1374 cm^{-1} corresponds to bending vibrations in methyl groups [24]. It also found a higher broad peak in the range of wavenumber of 3600 – 3200 cm^{-1}, representing the O-H functional group and indicating the moisture-enriched surface of the activated carbon [13]. The oxygen-containing functional groups had the ability to fasten the water vapor adsorption performance of the activated carbon [24].

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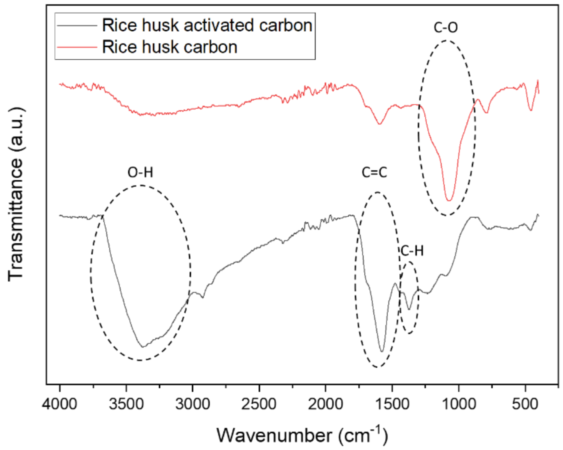

Fig. 3. FTIR spectra of rice husk char and activated carbon

Iodine Adsorption

According to Mianowski et al. [25], iodine adsorption can represent the surface area of activated carbon for a range of 200 – 850 mg/g iodine number. Therefore, the surface area of rice husk activated carbon in this study was around 615 m^2/g and higher than other tested commercial adsorbents (Fig. 4). According to this result, the product can be potentially developed as a high-capacity water vapor adsorbent. However, this result was still lower than nano-porous carbon with iodine adsorption of more than 700 mg/g [20]. This result indicates that the activated carbon product from this experiment had a higher lack of well-developed porosity than that of nano-porous carbon from rice husk.

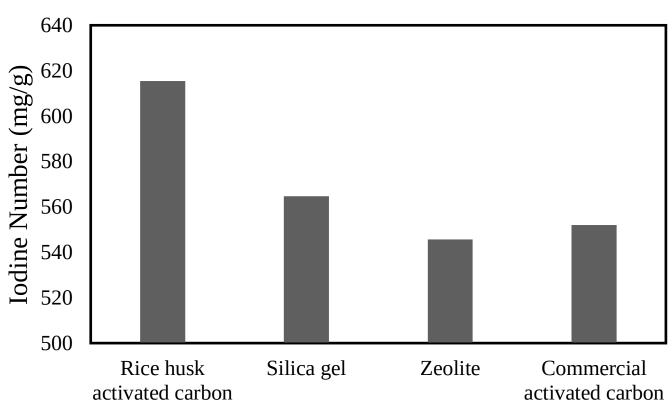

Fig. 4. Iodine adsorption of several adsorbents

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Effect of potassium hydroxide concentration

In the activation stage, the chemical reaction that occurred was [26]

$$6KOH + 2C \rightarrow 2K + 3H_2 + 2K_2CO_3 \quad (2)$$

Based on the reactions, KOH and carbon decomposed into potassium compounds (K and K_2CO_3) and hydrogen. The activated carbon was then washed with acid and water to remove them, clearing carbon pores [27]. Therefore, the water vapor adsorption of carbon was increased after the activation (Fig. 5). Untreated rice husks possessed the lowest adsorption capacity (0.04 g water/g adsorbent). Fig. 5 also presents water vapor adsorption of the activated carbon that was treated using different KOH concentrations for 8 hours. The data shows that manufacturing activated carbon using higher KOH concentration impacted a higher adsorption capacity. The highest adsorption capacity (0.42 g vapor/g adsorbent) belonged to activated carbon obtained from activation using 20% w/v KOH solution, and the lowest was found at 5% w/v KOH solution.

This finding confirmed the FTIR characterization that showed a hydroxyl functional group on the activated carbon. Also, the theory stated that a higher activation agent concentration facilitates higher carbon degradation to produce more pores [28]. However, the produced activated carbon was still lower than activated carbon derived from coffee shells [24] and tobacco stems [29]. The significance of different concentrations and adsorption times were evaluated with ANOVA summarized in Table 2, showing a significant impact of both factors (p-value < 0.05).

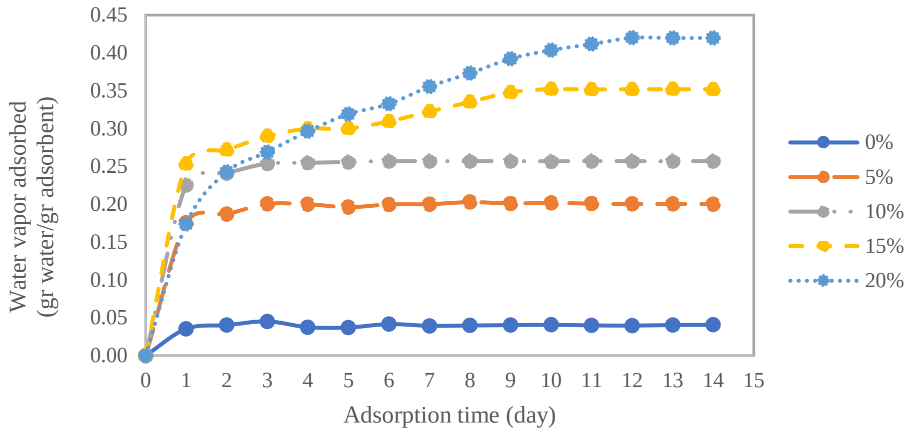

Fig. 5. Water vapor adsorption of activated carbon at different concentrations

Table 2. Two-way ANOVA of water vapor adsorption of the activated carbon at different adsorption times and KOH concentrations

Source of Variation	**SS**	**df**	**MS**	**F**	***P-value***	**F crit**
Adsorption time	28.9739	14	2.0696	10.7222	< 0.001	1.8726
Concentration	77.3276	4	19.3319	100.1570	< 0.001	2.5366
Error	10.8089	56	0.1930			
Total	117.1103	74				

Effect of Activation Time

The analysis of the activation time was conducted for activated carbon that was activated using 20% w/v KOH solution for 2 – 8 hours. Fig. 6 depicts the adsorption capacity at different activation times. The highest adsorption capacity was achieved at 8 hours of carbon activation (0.420 g vapor adsorbed/g adsorbent). Compared to the untreated rice husk char, activating the carbon for 8 hours increased the adsorption capacity up to 9 times. Analysis of the variance of this result also demonstrated that varying the activation period has a significant effect on the adsorption capacity (Table 3).

Previous studies reported a different relationship between the activation time and adsorption capacity [24], [28]. Sun et al. [24] stated that changes in activation time did not have a significant effect on the maximum adsorption capacity. In contrast, Yang et al. [28] found a fluctuation in the adsorption capacity of activated carbon at different activation times. From a range of 0.5 to 3.0 hours, the maximum methylene blue adsorption was at 2 hours. That result occurred because of an excessive increase in reaction rate between KOH and carbon causing a higher growth of porous structure. Extending the activation duration can increase the contaminants degradation and maximize the microporous formation [30].

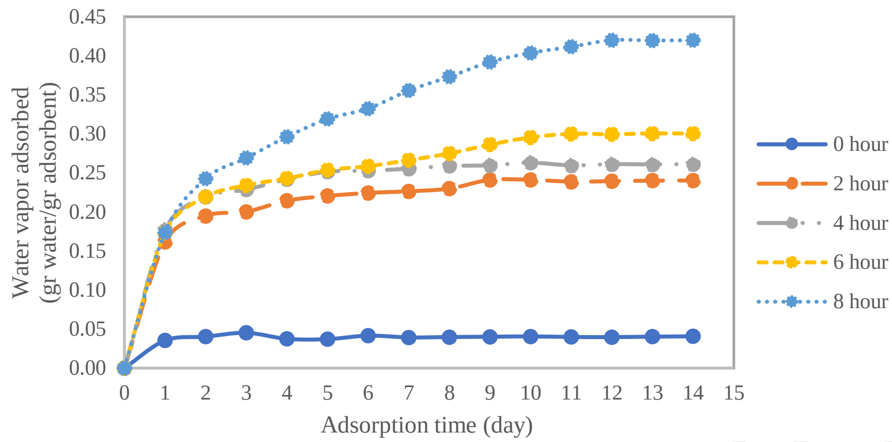

Fig. 6. Water vapor adsorption capacities at different activation times

Table 3. Two-way ANOVA of water vapor adsorption of the activated carbon at different activation times

Source of Variation	**SS**	**df**	**MS**	**F**	***P-value***	**F crit**
Adsorption time	30.0561	14	2.1469	13.3901	< 0.001	1.8726
Activation time	66.2628	4	16.5657	103.3209	< 0.001	2.5366
Error	8.9786	56	0.1603			
Total	105.2976	74				

Effect of Adsorption Temperature

The effect of adsorption temperature on the water vapor adsorption capacity of activated carbon was presented in Fig. 7. The test observed that a change of temperature of 15 °C to 27 °C affected the adsorption capacity. At 40 °C, there was no significant difference in the maximum water vapor adsorption capacity. More details, ANOVA indicated that the adsorption temperature and time had a significant effect on the water vapor adsorption (Table 4). Before, Chairunnisa et al. [12] and Cardenas et al. [31] explored the water vapor adsorption of activated carbon at 20 - 40°C. They found that increasing the adsorption temperature caused a reduction in the adsorption capacity. Combined with our results, the phenomenon that happened was a rapid formation of water clusters at temperatures lower than 30°C, and at higher temperatures, this water cluster became less stable [12]. Higher temperatures sped up the movement of water molecules and reduced the attraction of adsorbent and water [32].

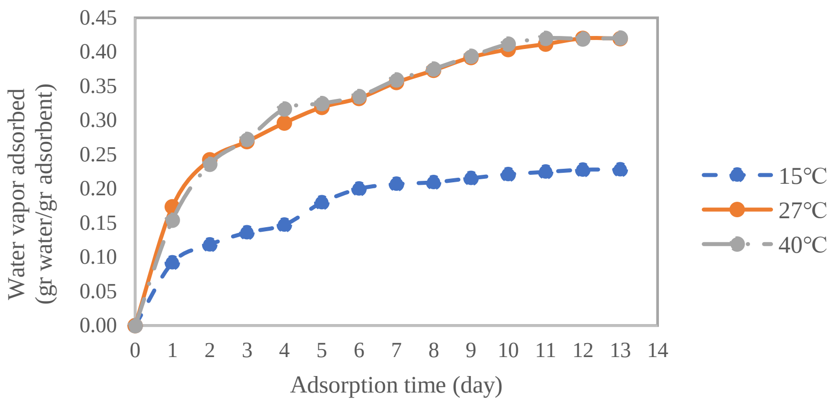

Fig. 7. Water vapor adsorption capacity at different temperatures

Table 4. Two-way ANOVA of water vapor adsorption capacity of the activated carbon at different temperatures

Source of Variation	**SS**	**df**	**MS**	**F**	***P-value***	**F crit**
Time	0.3975	13	0.0306	31.5170	< 0.001	2.1192
Temperature	0.1939	2	0.0970	99.9622	< 0.001	3.3690
Error	0.0252	26	0.0010			
Total	0.6166	41				

Fig. 8 displays the effect of the adsorption temperature of several adsorbents. Interestingly, different adsorbents exhibited a different relationship. For example, when the temperature increased from 15 °C to 40 °C, the maximum adsorption capacity of commercial activated carbon decreased by 33%. Furthermore, the adsorption capacity of other adsorbents increased when the temperature increased from 15°C to 40°C. Also, this comparison indicated that the produced activated carbon from rice husk adsorbed more water vapor than other tested commercial adsorbents. This result was in agreement with the iodine adsorption test that showed a higher surface area of rice husk-activated carbon than other adsorbents.

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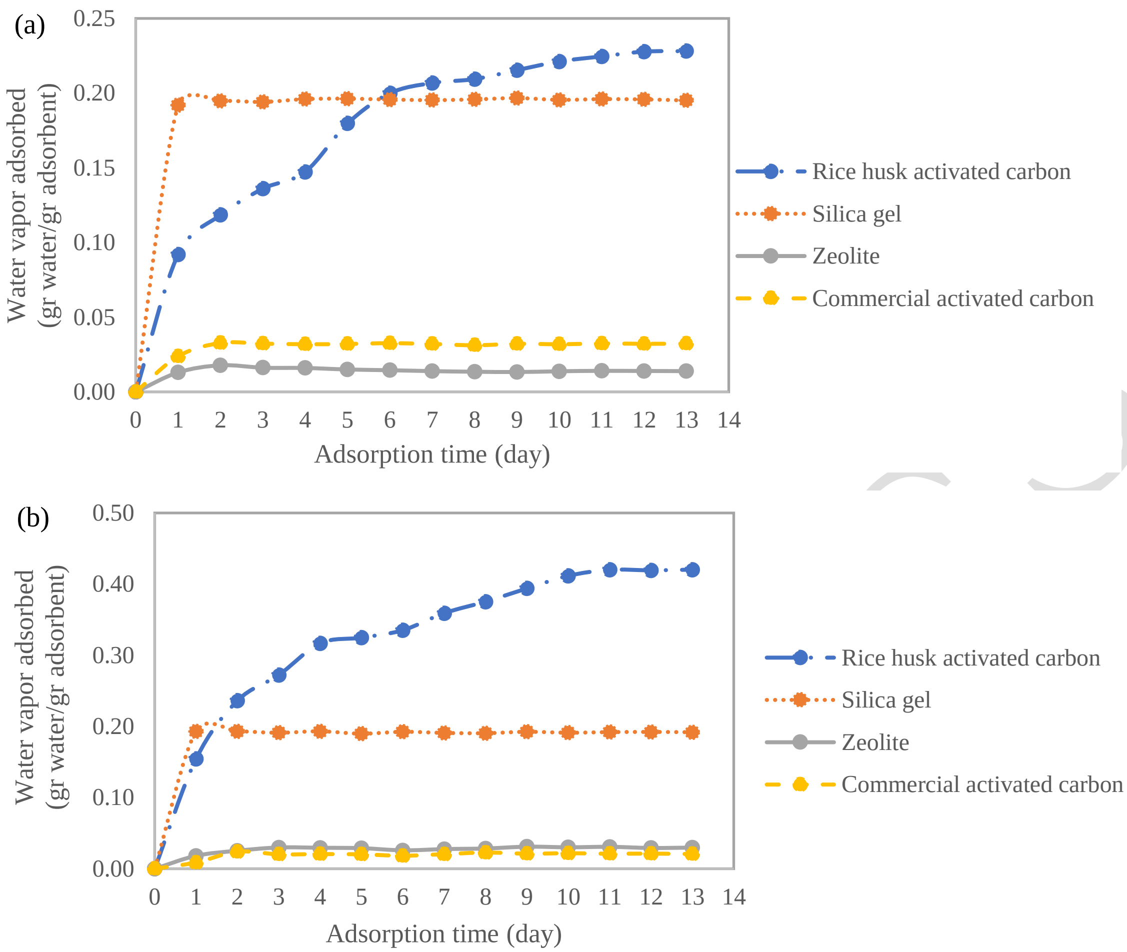

Fig. 8. Water vapor adsorption of several adsorbents at temperatures of (a) 15°C, (b) 40°C

Adsorption Kinetics

The adsorption kinetics of prepared activated carbon (20% w/v KOH solution and 8 hours activation) was evaluated by pseudo-first- and pseudo-second-order models. The kinetic study was analyzed by varying the adsorption temperature at 15 °C, 27 °C, and 40 °C. The parameters of the two models are provided in Table 5. The most suitable model was determined by coefficient of determination (R^2) and SSE. Based on the calculation, two models show good fits with R^2 close to 1.0 and SSE close to 0. Furthermore, the pseudo-first-order model described the adsorption mechanism of activated carbon prepared at the highest concentration and longest activation time was better than the second-order (R^2 values closer to 1 and SSE closer to 0). It implied that the main mechanism for the adsorption of water vapor on that activated carbon was physisorption. Meanwhile, the pseudo-second-order model described some samples better than the

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first-order, agreeing to the chemisorption process [33].

Table 5. Parameters of non-linear pseudo-first- and pseudo-second-order model

Concentration (% w/v)	Pseudo-First-Order				Pseudo-Second-Order			
	k_1	q_e	R^2	SSE	k_2	q_e	R^2	SSE
5	0.0013	0.2001	0.8383	9.6×10^{-6}	0.0240	0.2001	0.8921	1.9×10^{-5}
10	0.0009	0.2572	0.9396	6.3×10^{-5}	0.0714	0.2569	0.9551	4.5×10^{-5}
15	0.0005	0.3506	0.6702	0.0008	0.0034	0.3506	0.8340	0.0002
20	0.0002	0.4184	0.9618	0.0006	0.0012	0.4184	0.9492	0.0007
Activation time (hour)								
2	0.0003	0.2405	0.9614	0.0006	0.0078	0.2405	0.9561	6.0×10^{-5}
4	0.0003	0.2656	0.9864	0.0008	0.0062	0.2656	0.9905	3.0×10^{-5}
6	0.0003	0.3013	0.8805	0.0005	0.0031	0.3013	0.9309	0.0001
8	0.0002	0.4184	0.9613	0.0006	0.0010	0.4184	0.9532	0.0008
Temperature (°C)								
15	0.0002	0.2326	0.9686	0.0001	0.0019	0.2326	0.9398	0.0003
27	0.0002	0.4184	0.9613	0.0006	0.0010	0.4184	0.9532	0.0008
40	0.0003	0.4224	0.9425	0.0007	0.0009	0.4224	0.9772	0.0011

CONCLUSION

Activated carbon from rice husks was activated by varying the concentration of KOH as the activation agent and the activation time. The Fourier transform infrared (FTIR) spectral characterization represents that there was a significant effect on the activation of rice husk activated carbon as indicated by differences in functional groups before and after activation, including the addition of the hydroxyl group which made the activated carbon more hydrophilic, and the presence of the C=C group indicated an increase in carbon content. Based on the iodine adsorption test, the surface area of the activated carbon produced was around 615 m^2/g. The adsorption test showed that an increase in KOH concentration of up to 20% (w/v) and an activation time of 8 hours could increase the adsorption capacity of the resulting activated carbon (up to 0.420 g/g). Adsorption was also examined at 15°C, 27 °C, and 40 °C and showed an increase in adsorption capacity with increasing temperature. The produced activated carbon that was activated using 20% w/v KOH for 8 hours showed a good fit with the pseudo-first-order adsorption kinetics model. According to the comparison, activated carbon from rice husk showed a higher adsorption

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capacity than silica gel, zeolite, and commercial activated carbon. This research found that activated carbon from rice husks is a promising material to be applied to the dehumidification system of a drying process.

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