

1 **Effect of Potassium Hydroxide Concentration and Activation Time on Rice Husk-Activated Carbon** 2 **for Water Vapor Adsorption**

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10
11 **Abstract:** Rice husk carbon by-product from the industrial combustion is a promising source to produce a
12 vast amount of activated carbon adsorbent. This research prepared rice husk-activated carbon adsorbent by
13 varying the concentration of potassium hydroxide solution (5, 10, 15, 20 % w/v) and activation time (2, 4,
14 6, 8 hours). Fourier-transform infrared spectral characterization (FTIR) indicated a significant effect before
15 and after activation, especially the presence of hydroxyl groups. Based on the iodine adsorption, the specific
16 surface area of the produced-activated carbon was approximately 615 m²/g. Experimental results showed
17 that increasing potassium hydroxide concentration and activation time increases the water vapor adsorption
18 capacity of the activated carbon. Compared with the rice husk carbon, the KOH-activated carbon enhanced
19 the water vapor adsorption capacity to 931%. In the adsorption observation, changing the temperature from
20 15 to 27 °C caused a higher water vapor uptake onto the activated carbon. Two adsorption kinetics (pseudo-
21 first- and pseudo-second-order models) were used to evaluate the adsorption mechanism. This research
22 found that rice husk-activated carbon performed a higher water vapor adsorption capacity than other
23 adsorbents (silica gel, zeolite, and commercially activated carbon).

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

25 INTRODUCTION

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

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

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

46 Rice husk is a natural adsorbent that has been applied for various applications, including heavy metal and
47 dye removal in wastewater [13], [14], CO₂ adsorption [2], and urine purification [15]. In its application as
48 a water vapor adsorbent, rice husk was exploited by Warsiki et al. [16] that produce rice husk-CaCl₂
49 composite desiccant. They reported that environmental humidity (water activity) and temperature
50 influenced the adsorption capacity of the adsorbent. However, the study did not compare rice husk-CaCl₂

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

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

64 **EXPERIMENTAL PROCEDURE**

65 **Materials**

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

73 **Carbon Activation**

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

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

81 **Iodine Number Adsorption**

82 The iodine adsorption experiment was begun by mixing the activated carbon and 0.1 N iodine solution for
83 10 minutes. After the filtration, 10 mL of filtrate was titrated with 0.1 N sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$)
84 until became light yellow. The solution was then titrated by 1% amylum until clear from the blue color.
85 The iodine adsorption capacity was calculated by the equation of (Sulistyah et al., 2020)

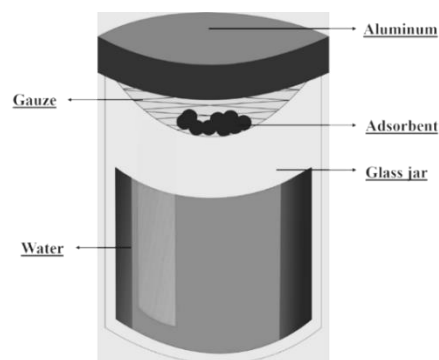
$$\text{Iodine number} = \frac{(V_1N_1 - V_2N_2)126.9 \times 5}{W} \quad (1)$$

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

88 **Adsorption capacity**

89 Fig. 1 describes the experimental setup of the adsorption test. The adsorption capacity of the produced
90 activated carbon was evaluated by placing 10 grams of activated carbon in an isolated mason jar.

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



98

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

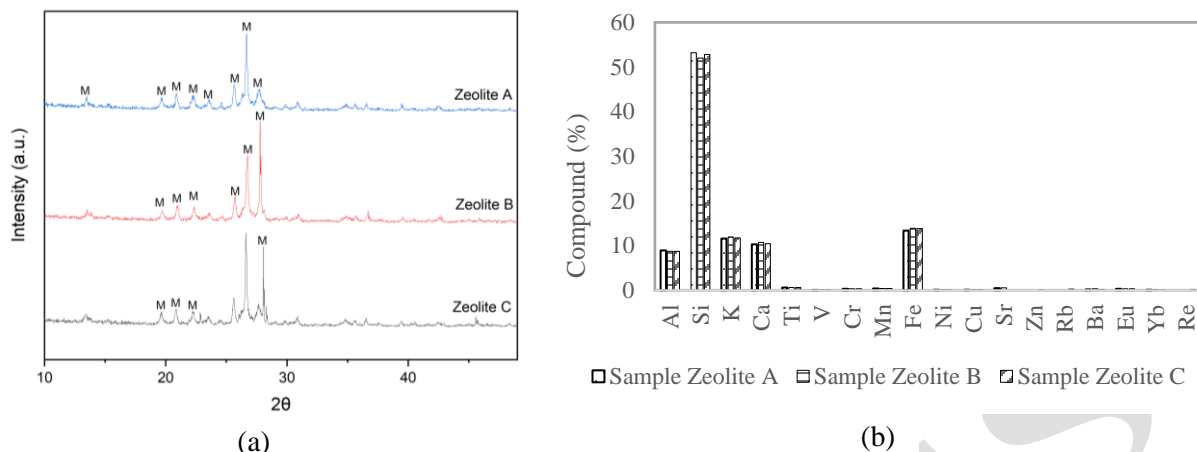
100 Table 1. Adsorption kinetics equation (Gurses et al. 2006; Qiu et al. 2009)

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

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

101 **Materials Characterization**

102 The functional groups in the activated carbon samples were examined using Fourier Transform Infrared
 103 Spectroscopy (FTIR) employing PerkinElmer Frontier Infrared Spectrometer version 10.6.1, with a
 104 resolution of 1 cm^{-1} in a region of 400 to 4000 cm^{-1} . X-ray diffractions (XRD PANalytical X'Pert PRO,
 105 Malvern Panalytical Ltd.) with $\text{CuK}\alpha$ radiation of 1.54060 \AA were used to investigate the crystallinity of
 106 natural zeolite. This instrument operated at 40 kV , 30 mA and the diffractograms were observed from
 107 10.03° to 82.19° on a 2θ scale with a 0.7° step size. The chemical compositions of natural zeolite were
 108 observed using X-ray Fluorescence (Panalytical Minipal 4, Malvern Panalytical Ltd.). Three random
 109 samples were taken from the same source as the zeolite samples, named Zeolite A, B, and C. The crystal
 110 structure and the chemical composition of natural zeolite were shown in XRD patterns and XRF analysis
 111 in Fig. 2. According to XRD patterns evaluated by Highscore plus 3.0e (PANalytical B.V., The
 112 Netherlands), the peaks of zeolite samples indicate similar crystal structure (mordenite). This result was
 113 compatible with XRF analysis that showed a common compound of mordenite mineral (Na_2 , Ca , K_2)
 114 $\text{Al}_2\text{Si}_{10}\text{O}_{24} \cdot 7\text{H}_2\text{O}$ [22].

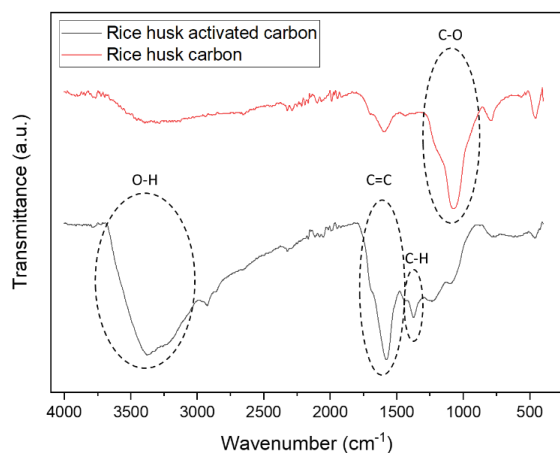


115 Fig. 2. (a) XRD patterns of natural zeolite used in this study (b) compounds found in natural zeolite used
 116 in this study

117 Results and Discussion

118 Fourier transform infrared (FTIR) characterization

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



128

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

130 **Iodine Adsorption**

131 According to Mianowski et al. [25], iodine adsorption can represent the surface area of activated carbon

132 for a range of 200 – 850 mg/g iodine number. Therefore, the surface area of rice husk activated carbon in

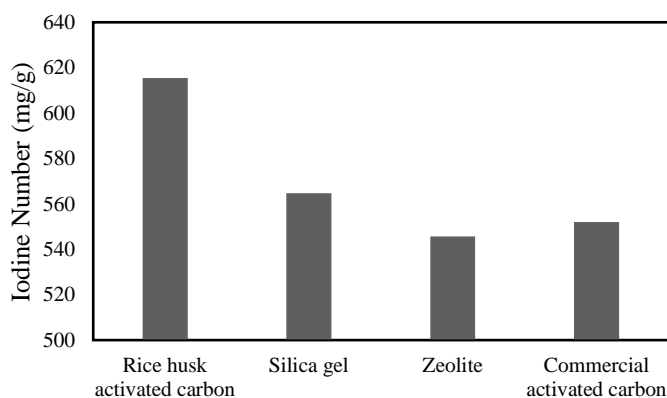
133 this study was around 615 m²/g and higher than other tested commercial adsorbents (Fig. 4). According to

134 this result, the product can be potentially developed as a high-capacity water vapor adsorbent. However,

135 this result was still lower than nano-porous carbon with iodine adsorption of more than 700 mg/g [20].

136 This result indicates that the activated carbon product from this experiment had a higher lack of well-

137 developed porosity than that of nano-porous carbon from rice husk.

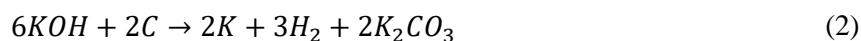


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139 Fig. 4. Iodine adsorption of several adsorbents

140 **Effect of potassium hydroxide concentration**

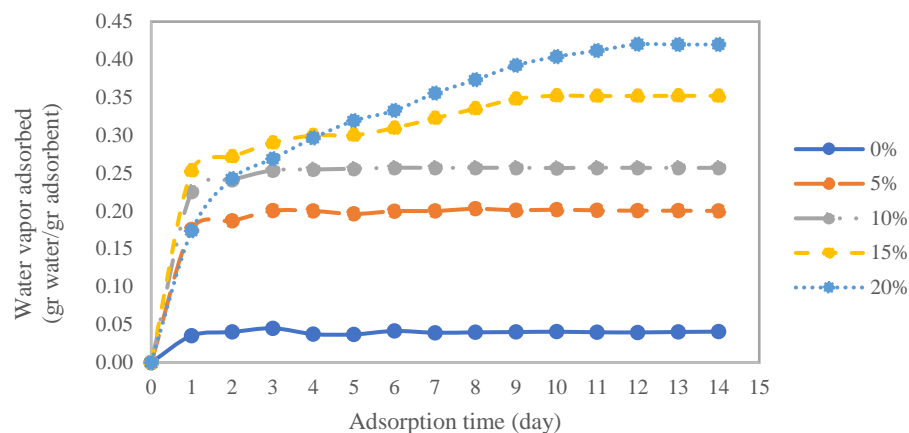
141 In the activation stage, the chemical reaction that occurred was [26]



142 Based on the reactions, KOH and carbon decomposed into potassium compounds (K and K_2CO_3) and
143 hydrogen. The activated carbon was then washed with acid and water to remove them, clearing carbon
144 pores [27]. Therefore, the water vapor adsorption of carbon was increased after the activation (Fig. 5).

145 Untreated rice husks possessed the lowest adsorption capacity (0.04 g water/g adsorbent). Fig. 5 also
146 presents water vapor adsorption of the activated carbon that was treated using different KOH
147 concentrations for 8 hours. The data shows that manufacturing activated carbon using higher KOH
148 concentration impacted a higher adsorption capacity. The highest adsorption capacity (0.42 g vapor/g
149 adsorbent) belonged to activated carbon obtained from activation using 20% w/v KOH solution, and the
150 lowest was found at 5% w/v KOH solution.

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



157
 158 Fig. 5. Water vapor adsorption of activated carbon at different concentrations
 159 Table 2. Two-way ANOVA of water vapor adsorption of the activated carbon at different adsorption times
 160 and KOH concentrations

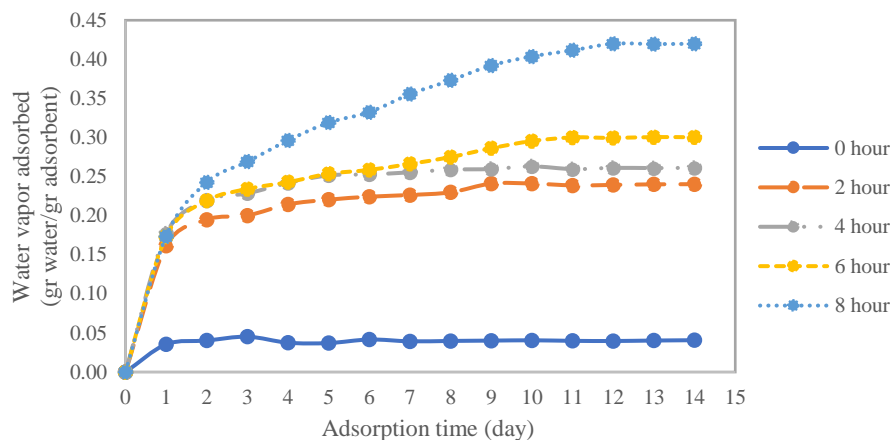
Source of Variation	SS	df	MS	F	P-value	F crit
Adsorption time	28.9739	14	2.0696	10.7222	< 0.001	1.8726
Concentration	77.3276	4	19.3319	100.1570	< 0.001	2.5366
Error	10.8089	56	0.1930			
Total	117.1103	74				

161 Effect of Activation Time

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

168 Previous studies reported a different relationship between the activation time and adsorption capacity [24],
 169 [28]. Sun et al. [24] stated that changes in activation time did not have a significant effect on the
 170 maximum adsorption capacity. In contrast, Yang et al. [28] found a fluctuation in the adsorption capacity
 171 of activated carbon at different activation times. From a range of 0.5 to 3.0 hours, the maximum
 172 methylene blue adsorption was at 2 hours. That result occurred because of an excessive increase in

173 reaction rate between KOH and carbon causing a higher growth of porous structure. Extending the
 174 activation duration can increase the contaminants degradation and maximize the microporous formation
 175 [30].



176

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

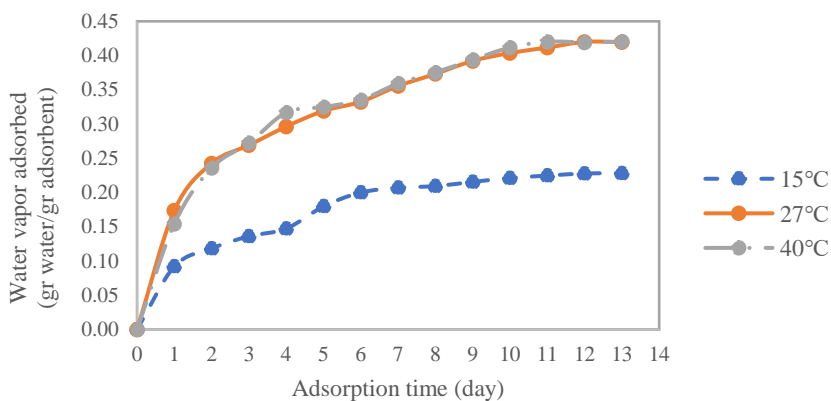
178 Table 3. Two-way ANOVA of water vapor adsorption of the activated carbon at different activation times

Source of Variation	SS	df	MS	F	<i>P-value</i>	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				

179 **Effect of Adsorption Temperature**

180 The effect of adsorption temperature on the water vapor adsorption capacity of activated carbon was
 181 presented in Fig. 7. The test observed that a change of temperature of 15 °C to 27 °C affected the
 182 adsorption capacity. At 40 °C, there was no significant difference in the maximum water vapor adsorption
 183 capacity. More details, ANOVA indicated that the adsorption temperature and time had a significant effect
 184 on the water vapor adsorption (Table 4). Before, Chairunnisa et al. [12] and Cardenas et al. [31] explored
 185 the water vapor adsorption of activated carbon at 20 - 40°C. They found that increasing the adsorption
 186 temperature caused a reduction in the adsorption capacity. Combined with our results, the phenomenon
 187 that happened was a rapid formation of water clusters at temperatures lower than 30°C, and at higher

188 temperatures, this water cluster became less stable [12]. Higher temperatures sped up the movement of
 189 water molecules and reduced the attraction of adsorbent and water [32].



190

191 Fig. 7. Water vapor adsorption capacity at different temperatures

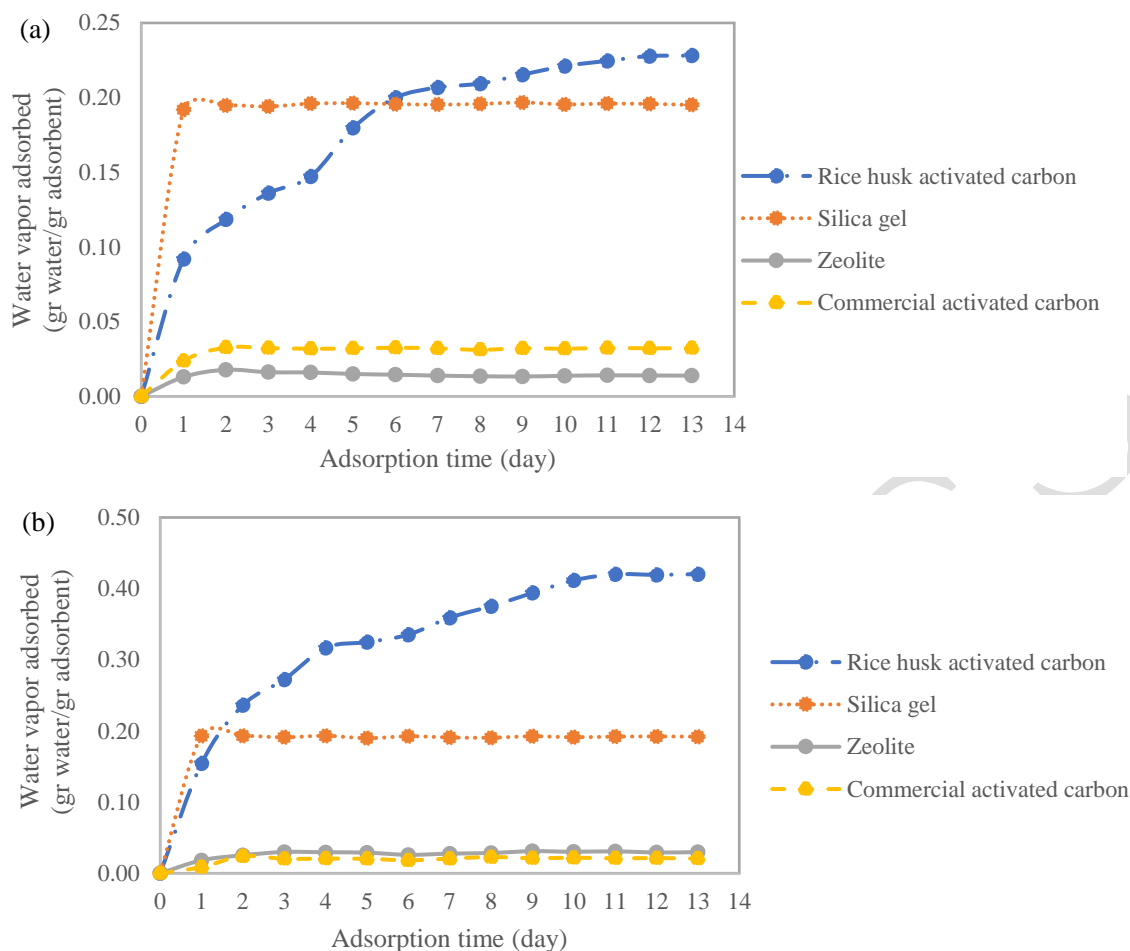
192 Table 4. Two-way ANOVA of water vapor adsorption capacity of the activated carbon at different

193 temperatures

Source of Variation	SS	df	MS	F	<i>P-value</i>	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				

194

195 Fig. 8 displays the effect of the adsorption temperature of several adsorbents. Interestingly, different
 196 adsorbents exhibited a different relationship. For example, when the temperature increased from 15 °C to
 197 40 °C, the maximum adsorption capacity of commercial activated carbon decreased by 33%. Furthermore,
 198 the adsorption capacity of other adsorbents increased when the temperature increased from 15°C to 40°C.
 199 Also, this comparison indicated that the produced activated carbon from rice husk adsorbed more water
 200 vapor than other tested commercial adsorbents. This result was in agreement with the iodine adsorption
 201 test that showed a higher surface area of rice husk-activated carbon than other adsorbents.



202

203

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

205 **Adsorption Kinetics**

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

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

216 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

217 CONCLUSION

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

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

233 ACKNOWLEDGEMENT

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