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Synthesis and Characterization of Activated Carbon from Waste Compedak Fruit (Artocarpus Champeden) Activated H_3PO_4 as Adsorbent of Methylene Blue

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Abstract

Synthesis of activated carbon from cempedak peel waste is carried out to utilize biomass waste. This study aimed to synthesize and characterize activated carbon from cempedak peel waste. The synthesis was carried out by chemical activation using phosphoric acid with a dry impregnation ratio of 1:4 (g sample:g H_3PO_4). Samples impregnated for 24 hours were then heated at 250 ℃—and then calcined at 350 ℃ and 450 ℃. The XRD and FTIR characterization results indicated that the activated carbon obtained had an amorphous structure and the activated carbon obtained had hydroxyl, carbonyl, and carboxylic groups. Activated carbon with the highest yield was obtained at a temperature of 350 ℃, namely 43%. The results of determining the water content of activated carbon obtained are 8.36% at 350 ℃ and 7.1% at 450 ℃. The value of water content and ash content of activated carbon from the skin of this cempedak fruit has met the Indonesian National Standard (SNI 06-3730-1995). The best-activated carbon yield was at a calcination temperature of 450, with the percentage of adsorption efficiency on methylene blue of 98.88%.

Keywords: Activated carbon, cempedak peel, phosphoric acid, methylene blue

Introduction

Some of the biomass waste can be utilized in the manufacture of activated carbon, such as coconut shells (Azevedo et al., 2007; Sawalha et al., 2020 et al., 2020), corn cob waste (Aisiyah, 2016), fruit peels (Prahas et al., 2008; Musa, 2013), fruit seeds (Olivares-Marin et al., 2006), grain skins (Juwita et al., 2017). Activated carbon is usually produced from carbon-rich materials such as organic materials (lignin, wood, and coal). Coal is a natural resource that is difficult to renew. The price is relatively high, so agricultural residues can be an alternative material to produce activated carbon because they are sourced from renewable and cheaper materials (Prahas et al., 2008; Lim et al., 2020).

Cempedak plant (Artocarpus champeden) is a tropical plant that grows in Indonesia. Cempedak is a type of fruit whose yields are pretty abundant, and people only eat the flesh and seeds, while the skin of the cempedak fruit is a problem for the environment (Rahmawati et al., 2021). One way to increase the economic value of cempedak skin waste is to co. Convert it into activated carbon to be used as an adsorbent (Kurniasari et al., 2012; Putri et al., 2020). Activated charcoal can be activated through a physical activation process and a chemical process. The physical activation process can be carried out by giving water vapor or CO_2 gas, while chemically, it is done by adding certain chemical substances (Jamilatun et al., 2015; Sudrajat, 2011; Aryani et al., 2019). Sahara et al. (2017) reported that activated charcoal produced from the stems of the gumitir plant (Tagetes erecta) through chemical activation using phosphoric acid (H_3PO_4) resulted in activated charcoal with a better pore structure and a carbon content of 81,41%.

Not all pigments from dyestuffs will stick; there are still residual dyes wasted along with solvents, which can pollute the aquatic environment and be carcinogenic to living things (Muhtar, 2013; Teddy et al., 2018). Methylene blue dye is difficult to decompose by micro-organisms due to a benzene nucleus in its structure (Sunarsih & Dahani, 2018; Dini & Wardhani, 2014; Darajat et al., 2008).

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Based on the above background, research was carried out on the synthesis and characterization of activated carbon from phosphoric acid activated cempedak peel waste as an adsorbent for methylene blue dye. This study aims to obtain an adsorbent material using cempedak peel waste. The urgency of the research is to increase the economic value of cempedak skin waste and to overcome the pollution of methylene blue dye.

Methods

Equipment and material

The tools in this research are Fourier Transform Infra-Red (FTIR), Difraktogram X-ray (XRD), UV-Vis, furnace, and pH meter. The materials used in this study were Cempedak fruit skin waste, distilled water, phosphoric acid/H_3PO_4 (p.a *Merck*), methylene blue dye (p.a *Merck*), nitrogen gas, and Whatman filter paper no.42

Sample preparation

The peel of the cempedak fruit that has been taken washed with distilled water several times until clean, then separated from the carpel fibers and cut into small pieces. Then it was dried for 24 hours in an oven at 105 °C. The sample was then cooled in a desiccator. The dried samples were crushed and sieved using a 100 mesh sieve and stored in a desiccator (Prahas et al., 2008).

Impregnation of samples using H_3PO_4

A dehydrated sample of 20 g was impregnated into 85% H_3PO_4 with a ratio of 1:4 for 24 hours. The suspension was dried in an oven at 60 °C and carbonized at 250 °C for 30 minutes; calcination was continued at 350 and 450 °C for 45 minutes. The sample is cooled in a desiccator. Then the sample was filtered and rinsed with distilled water until the pH was neutral and then dried at 105 °C. Characterization was carried out using XRD and FTIR (Prahas et al., 2008).

Making carbon from cempedak fruit peel without activator

A 500 g of the prepared sample was put into a furnace. Carbonized at 250 °C for 30 minutes, and calcination was continued at 450 °C for 45 minutes. The resulting carbonization is cooled in a desiccator. Then grinded and sieved using a 100 mesh sieve.

Characterization by XRD and FTIR

Identification of activated carbon characteristics

Identifying the characteristics of activated carbon refers to the Indonesian National Standard (SNI 06-3730-1995) on general requirements and testing of activated carbon. Identification of factors carried out is an analysis of yield and determination of water and ash content.

Methylene blue calibration curve creation

0.1 grams of methylene blue dye were carefully weighed into a 100 mL beaker to make 100 mL of a standard 1000 ppm solution. The methylene blue dye was then dissolved with 20 mL of distilled water. The solution was put into a 100 mL volumetric flask and diluted with distilled water to the limit mark. Dilutions were carried out to obtain 0.1, 0.5, 1.0, 3.0, 5.0, and 7.0 ppm. Then the absorbance was measured at max = 665 nm using a UV-Vis spectrophotometer, and standard curves were made.

Methylene blue dye adsorption with carbon without activator

As much as 25 mL of methylene blue dye solution with a concentration of 50 ppm was put into 3 Erlenmeyer pieces, adding 0.1 grams of carbon. Shaker for 24 hours to obtain the filtrate and measure the absorbance at max = 665 nm. The absorbance value obtained is entered into the methylene blue calibration curve to determine the remaining methylene blue concentration.

Methylene blue dyestuff adsorption with temperature activated carbon H_3PO_4 85%

25 mL of methylene blue dye solution with a concentration of 50 ppm was put into 3 Erlenmeyer flasks followed by the addition of 0.1, 0.3, and 0.5 gram of activated carbon to every flask and then shaken for 24 hours to obtain the filtrate and measure the absorbance at max = 665 nm. The absorbance value obtained was entered into the methylene blue calibration curve to determine the remaining methylene blue concentration.

Results and Discussion

Chemical activation with H_3PO_4

The prepared cempedak powder was activated by impregnating the sample into H_3PO_4 85%. Activation with H_3PO_4 was carried out to reduce the formation of tar compounds from lignin in pieces that could cover the carbon pores, thereby reducing the absorption of activated carbon (Prahas et al., 2008). The impregnated carbon with H_3PO_4 was calcined at 350 °C and 450 °C. The temperature variation was carried out to determine its effect on the thermal stability of the resulting impregnated carbon structure and on the ability to adsorb methylene blue dye. Activated carbon was obtained after filtering, rinsing, and drying processes.

Manufacture of cempedak leather carbon without activator

The carbon from the cempedak peel powder without adding an activator was made as a visual comparison with activated carbon with a calcination temperature of 450 °C. The prepared sample was put into a furnace and carbonized at 250 °C for 30 minutes, and calcination was continued at 450 °C for 45 minutes. Visually seen in **Figure 1a**, cempedak peel powder turns black due to the carbonization process, which hydrates the mineral components in the sample and leaves carbon.

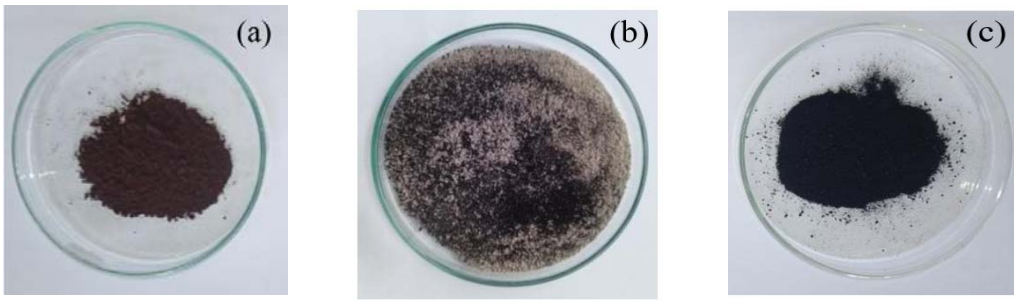

Figure 1. a). Carbonization sample 250 °C, b) Carbon without H_3PO_4, calcination 450 °C; and c) activated charcoal with H_3PO_4, calcination 450 °C

According to Pari (2011) & Kumar & Tyagi (2018), carbonization (**Figure 1**) occurs in the arrangement of carbon elements to form aromatic carbon and becomes more intensive with increasing temperature. Hemicellulose is the first compound that undergoes decomposition changes in chemical components because the hemicellulose structure is branched and amorphous. The calcined sample at 450 (**Figure 1b**) shows that some of the carbon has already formed ash. It shows that the ash content of the carbon produced from the cempedak shell without adding an activator before calcination increases with increasing temperature. Activated carbon (**Figure 1c**) visually does not look like ash because, as an activating agent, H_3PO_4 can absorb mineral content in the material used as activated carbon to prevent the formation of ash on activated carbon (Sangi et al., 2012).

Characterization using X-ray diffractogram (XRD)

X-ray diffraction characterization on prepared carbon with a scan angle range (2θ = 20°–80°) using a Cu radiation source with a wavelength (λ) of 1.54 Å. This analysis aims to determine the changes in the structure of the cempedak fruit peel powder after being impregnated and calcined at high temperatures. To determine the crystalline phase of carbon formed from carbon without an activator with a calcination temperature of 25 and activated carbon with a calcination temperature variation of 350 °C. Characterization results using XRD are shown in **Figure 2**.

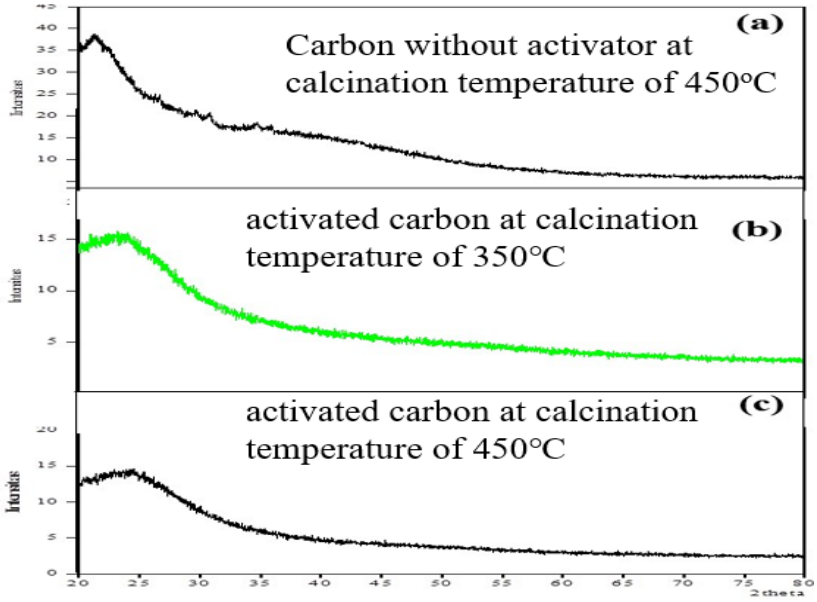

Figure 2. XRD diffractogram of carbon without activator calcining temperature 250 °C, activated carbon calcination temperature 350 °C, and (c) activated carbon calcining temperature 450 °C.

The X-ray diffraction results showed carbon with an amorphous structure pattern, so it was impossible to determine the crystalline phase of the carbon formed. According to Prabu & Raghu (2017), carbon with an amorphous compound structure has a turbostratic structure originating from a graphite layer with a microcrystalline network. The calcination process with temperature

variations without impregnation and with H_3PO_4 impregnation showed a change in the diffraction pattern of a shift in the value of 2θ that appeared at the peak of the carbon diffractogram after the calcination process. Carbon with a calcination temperature of 250 °C has a characteristic peak for the (110) plane, which corresponds to the graphite structure where the diffraction peaks are seen at an angle of 2θ approx. 21°–22°. These results are the same as those Aisiyah (2016) obtained in the study of activated carbon from corn cobs waste (2θ=21.36°. At calcination temperatures between 350 and 450 °C, the peaks become wider, indicating a characteristic for the (002) plane, which corresponds to the graphite structure seen at an angle of 2 around 25°. The same result was reported by Prabu & Raghu (2017) is 2θ=25°. According to Darmawan et al. (2015) & Perdani et al. (2021), in the study of the structure of activated carbon from wood, diffraction results were obtained which characterize the characteristics of graphite peaks with a combination of diffractogram patterns formed at peaks with angles of about 10°–30° and 25°–26° which are indicated as carbon markers with turbostratic properties.

Characterization using the fourier transform infrared (FTIR)

The utilization of activated carbon in the industrial sector in terms of application as an adsorbent is more focused on its adsorption properties. In this case, it is related to the functional groups produced by activated carbon, such as hydroxyl, carbonyl, and carboxylic groups, which can give amphoteric properties to carbon, so that activated carbon can act as an acid or a base (Saputro, 2010; Eletta et al., 2020). In this study, sample characterization by FTIR was conducted to determine the functional groups formed on the carbon of the prepared cempedak peel. Carbon IR spectra can be seen in **Figure 3.**

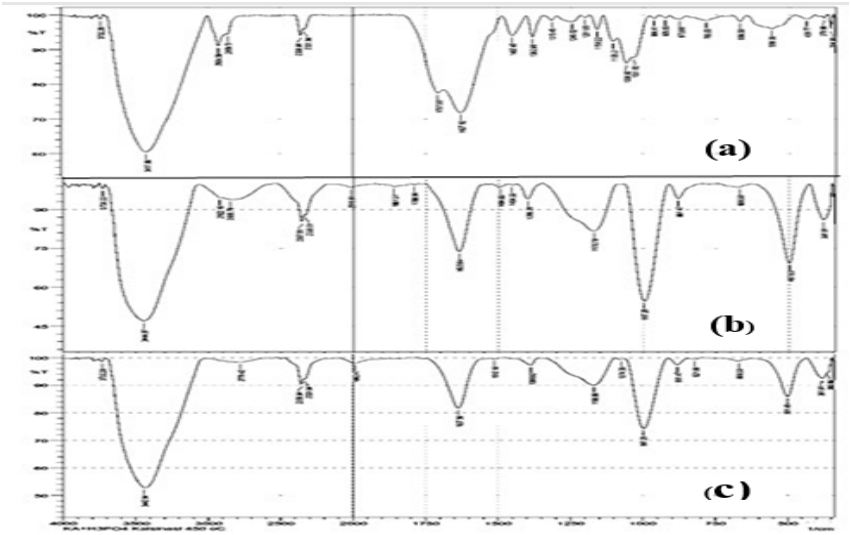

Figure 3. FTIR spectrum, a) carbon without activator calcination temperature 250 °C, b) activated carbon calcination temperature 350 °C, and c). 450 °C

The analysis results using an IR spectrophotometer showed a shift in the wavenumber and a change in the intensity of the resulting carbon. However, the transition is not too significant, so there are still characteristic vibrations from the absorption pattern with the type of bond -OH, -CH, -C=C, and -C-O on the carbon functional group, which is also found in the primary material (cempedak peel powder). The same result was also stated by Prahas et al. (2008) & Nagalakshmi et al. (2019) that there are still the same functional groups between the jackfruit peel samples and the activated carbon functional groups produced. The functional groups on the synthesized carbon can be seen in **Table 1**.

Table 1. Synthesized carbon functional groups

Functional Groups	Activated Carbon theorists	Activated carbon (°C)		
-OH	3700–3000	250	350	450
-C=C	1700–1600	3417.86	3444.87	3442.94
-C-O	1300–1000	1627.92	1635.64	1637.56
-C-H	3000–2700	1058.92	1170.79	1168.86
		2924.09	2922.16	2779.42

The results of this FTIR spectrum indicate that the carbon from the cempedak shell has functional group suitability with the standard structure of the theoretically activated carbon (-OH, C=C, -CH, and -CO), where each wavenumber of the resulting peak is in the range activated carbon wave (Lewoyehu, 2021). The resulting activated carbon has an absorption pattern with the type of bond –OH, C=C, C-H, and C-O and can then be used as an adsorbent for solutions and gases.

The activation process with the H_3PO_4 activator on cempedak peel powder causes a shift in IR absorption and the formation of new absorption in a particular wavenumber region. In research, Nurhasni et al. (2018) reported that activated carbon experienced a decrease in intensity and a shift in the wavenumber of the –OH functional group after activation with acid, followed by a change in the wavenumber of other functional groups. The –OH active group undergoes protonation after being activated with acid, resulting in a decrease in vibration, which causes a shift in wavenumber and a reduction in the intensity of –OH.

Physical characteristics of activated carbon from cempedak fruit peel

The cempedak peel carbon tested physical properties included yield, moisture content, and ash content. The impregnation treatment with H_3PO_4 and temperature variations in the sample resulted in different carbon characteristics, as listed in **Table 2.**

Table 2. Physical properties of activated carbon from cempedak leather

Sample	Rendement (%)	Water Content (100)	Ash Content (%)
Activated Carbon (SNI)	-	Max 15	Max 10
Carbon without activator	37.38	10.15	8.3
Carbon Actevated 350 ^{o}C	43.22	8.36	7.2
Carbon Actevated 450 ^{o}C	41.72	7.1	4.8

According to Prahas et al. (2008), jackfruit skin is a complex composite material made from natural polymers (cellulose, lignin, and hemicellulose). In the activation process at high temperature, the polymer structure decomposes and releases non-carbon elements, mainly hydrogen, oxygen, and nitrogen, in the form of liquid (tar) and gas, then forms a carbon skeleton in aromatic sheets.

The calcination process encourages depolymerization, dehydration, and aliphatic conversion with aromatic compounds, thereby increasing the yield of activated carbon (Prahas et al., 2008). As for the result of activated carbon at temperatures of 350 and 450 °C, it seems to be decreasing (from 43.22% to 41.72%). It indicates that activated carbon's activation temperature is inversely proportional to the yield. The increase in the calcination temperature at the time of activation causes the reduction of volatile substances to be more significant, affecting the weight of the obtained results. These results follow the research results on activated carbon from jackfruit skin with an H_3PO_4 activator that has been done previously by Prahas et al. (2008), namely, the yield of raw materials is 36%, while the result of carbon with H_3PO_4 is around 42.15%.

The determination of the water content of carbon is carried out to determine the hygroscopic properties of carbon. The excellent water content of carbon must have a small value because a significant moisture content can reduce the adsorption capacity of carbon. Based on the results of determining the water content in **Table 3**, it can be seen that the value of the water content of activated carbon H_3PO_4 (8.36% and 7.1%) is smaller than carbon without activator (10.15%). It is because H_3PO_4 is a potent hydrating agent so that it can bind water molecules contained in carbon. Furthermore, the higher temperature in the manufacture of activated carbon also further increases the dehydration process. The water in activated carbon will evaporate more, and the water content will be lower. These results are by the determination of the carbon moisture content of coconut shells for treatment at temperatures of 500, 600, 700, and 800 °C by Jamilatun et al. (2015), with the result that the water content decreases with each increase in the temperature treatment of each carbon, namely 5.7, 5.6%, 4.2, and 4.0%.

Activated carbon made from natural ingredients contains carbon compounds and mineral content such as metals. Determination of ash content aims to determine the scope of metal oxides in carbon because excessive ash can cause blockage of the pores on the carbon so that the carbon surface area is reduced (Pari, 2011; Ramayana et al., 2017). Results of the characterization of the ash content of carbon without an activator at a temperature of 250 °C (8.3%) according to **Table 3** show that the increasing carbonization temperature causes the amount of ash content to increase, while for activated carbon H_3PO_4 at temperatures of 350 °C and 450 °C in this study. It has a lower total ash

content than carbon without activator, 7.2%, and 4.8%, respectively. The effect of temperature on carbon ash content was proposed by Lestari et al. (2017) in the manufacture of carbon from coconut shells at 225, 250, 275, 300, and 325 °C. Then obtained less ash content on carbon at a temperature of 325 °C with the result of 0 – 2.04%, that the higher the temperature for the manufacture of activated carbon, the greater the number of volatile substances that are oxidized so that it will affect the surface area of the activated carbon and cause a decrease in ash content. Meanwhile, the effect of phosphoric acid on carbon ash content was stated by Sahara et al. (2017) that phosphoric acid can cause corrosion of metals so that based on this, metals contained in carbon will corrode when activation using phosphoric acid causes the ash content of activated carbon to be lower than that of unactivated carbon

Methylene blue dye adsorption with activated carbon

Before determining the percentage of adsorption of methylene blue dye, the absorbance measurement of the standard solution of methylene blue dye was first measured. Measurements were carried out using a UV-Vis spectrophotometer at a wavelength of 665 nm. The adsorption of methylene blue in this study was carried out with various treatments to know the effect of treatment variations and determine the percentage of adsorption from each treatment. Variations of treatment given include adsorption with carbon without the addition of an activator with a calcination temperature of 250 °C as much as (A) 0.1 grams, (B) 0.3 grams, (C) 0.5 grams, activated carbon with a calcination temperature of 350 °C as much as (D) 0.1 grams, (E) 0.3 grams; (F), 0.5 grams, and activated carbon with a calcination temperature of 450 °C as much as (G) 0.1 grams, (H) 0.3 grams, (I) 0.5 grams.

The adsorption process is carried out by stirring the suspension using a shaker for 24 hours. Stirring is carried out to achieve adsorption equilibrium between methylene blue dye and the carbon surface. The results of the adsorption of methylene blue with each treatment can be seen in **Table 3**.

Table 3. Results of measurement of absorption of methylene blue dyes on a variation of temperature and absorbent mass treatment

Treatment Type	Temperature (^{0}C)	M (g)	abs	Co (ppm)	Ce (ppm)	Co-Ce (ppm)	q (mg/g)	E (%)
A	250	0.1	0.105	50	1.2234	48.7766	12.1819	97.55
B	250	0.3	0.357	50	3.9835	46.0165	3.8347	92.03
C	250	0.5	0.521	50	5.7798	44.2202	2.2110	88.44
D	350	0.1	0.069	50	0.8921	49.1709	12.2804	98.34
E	350	0.3	0.056	50	0.6867	49.3133	4.1094	98.62
F	350	0.5	0.077	50	0.9167	49.0840	2.4542	98.16
G	450	0.1	0.049	50	0.6100	49.3900	12.3351	98.78
H	450	0.3	0.044	50	0.5553	49.4447	4.1203	98.88
I	450	0.5	0.066	50	0.7962	49.2038	2.4601	98.62

Information:
m = mass of adsorbent
Co = concentration of methylene blue before the adsorption process
Ce = concentration of methylene blue before the adsorption process
q = adsorption capacity
E = percentage of adsorption efficiency

The adsorption efficiency in treatment C showed the lowest results because, in this treatment, the adsorption process of methylene blue dye only depended on the ability of carbon, without adding an activator with a calcination temperature of 250 °C. The addition of the H_3PO_4 activator and increased calcination temperature in the activated carbon production process showed a significant increase in the adsorption efficiency results. It can be seen from the percentage of adsorption efficiency for treatments D to I, which is in the range of 98%, so it can be seen that the addition of an H_3PO_4 activator and treatment of increasing the calcination temperature in the process of making activated carbon are factors that can affect the absorption of activated carbon in adsorption of dyes—methylene blue.

An optimum amount of adsorbent at 350 and 450 °C on the absorption of methylene blue dye with an adsorbent amount of 0.3 g resulted in a higher adsorption efficiency than the amount of adsorbent 0.1 and 0.5 g, each of which was 98.62% and 98.88%. An adsorbent with chemical activation treatment using H_3PO_4 has a higher adsorption efficiency than an adsorbent without an activator. Chemical activation using H_3PO_4 was carried out to increase the negative charge on the adsorbent's surface to increase the adsorbent's ability to adsorb methylene blue, a cationic dye (positively charged). Based type of treatment, increasing the activation temperature adsorbent to the kind of treatment at a temperature of 450 °C had a better adsorption efficiency than the adsorbent with the type of treatment at a temperature of 350 °C. It shows that increasing the activation temperature can increase the ability of the adsorbent to adsorb dye with the

assumption that there is an increase in the surface area of the adsorbent so that the adsorption efficiency is also better. These results are under the results obtained in the study of carbon from jackfruit skin by (Prahas et al., 2008).

Carbon adsorption capacity is also affected by active groups on the carbon surface. The existence of each functional group has different adsorption capabilities, thus enabling the adsorption process to occur with several interaction mechanisms. Two possible interaction mechanisms can occur between the active site on the surface of the adsorbent and the adsorbate, namely chemical adsorption (covalent bonds) and physical adsorption (van der Waals electrostatic forces, hydrogen bonds, and -π interactions. The covalent bond is more vital than physical adsorption. The relatively weak physical interaction will form a mayer layer determination of the adsorption mechanism on activated carbon from cempedak fruit peel. Refers to research on activated carbon from jackfruit peel by Prahas et al. (2008) and activated carbon from salak peel by Angela et al. (2015), which shows activated carbon adsorption on methylene blue dye obtained occurs physically.

Conclusions

Cempedak fruit peel can be synthesized into activated carbon through chemical activation using an 85% H_3PO_4 activator at 350 and 450 °C with a yield of 41–43%. The determination of the water content and ash content has met the Indonesian National Standard (SNI 06-3730-1995). Characterization using XRD and FTIR showed that the synthetic activated carbon obtained was an amorphous compound with hydroxyl, carbonyl, and carboxylic groups. The best-activated carbon adsorption results were at a calcination temperature of 450 °C with an adsorption efficiency percentage of 98.88% for methylene blue dye.

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