# Effect of Activator Type on Activated Carbon Characters from Teak Wood and The Bleaching Test for Waste Cooking Oil

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## Effect of Activator Type on Activated Carbon Characters from Teak Wood and Bleaching Test for Waste Cooking Oil

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#### **Abstract**

The starting material for activated carbon was biomass from teak woodcutting, which consists of 47.5% cellulose, 14.4% hemicellulose, and 29.9% lignin. The surface area and iodine number of activated carbons are the factors determining the adsorption ability. This study aims to determine the effect of the activator type on activated carbon characters and test the absorption ability for waste cooking oil. The synthesis stages include carbonization, chemical activation, and then physics activation. The activation process consists of two steps. Firstly. the chemical activation via adding  $H_2SO_4$ , and  $H_3PO_4$  at room tengerature for 24 hours, the second, physical activation by heating at various temperatures of 300, 400, and 500 °C for two hours. The characterizations of activated carbon include water content, ash content, iodine number, functional groups, and surface area. Furthermore, the activated carbon was used as an adsorbent for waste cooking oil for 60 minutes at 100 °C with a stirring of 500 rpm. The results were analyzed using UV-Vis spectrophotometry at a maximum wavelength of 403 nm. The iodine numbers of activated carbon ranged 481.1-1211.4 mg/g and 498.8-1204 mg/g for H<sub>3</sub>PO<sub>4</sub> and H<sub>2</sub>SO<sub>4</sub>, respectively. Activated carbon with H<sub>3</sub>PO<sub>4</sub> of 15% and an activation temperature of 400 °C has the highest surface area of 445.30 m<sup>2</sup>/g. The H<sub>2</sub>SO<sub>4</sub> dan H<sub>3</sub>PO<sub>4</sub> activators can be used to improve the quality of activated carbon in absorbing dyes in waste cooking oil, where the optimum concentration is 10-15% (v/v). The H<sub>3</sub>PO<sub>4</sub> activator tends to produce a higher bleaching percentage than H<sub>2</sub>SO<sub>4</sub>.

Keywords: activator type, activated carbon, waste cooking oil, bleaching, teak wood

### 1. Introduction

Activated carbon is a term used for carbon-rich materials, containing internal pore structures, high surface area, and chemical group on the surface. Activated carbon is the oldest and most popular adsorbent in the water and wastewater treatment industry (Bhatnagar et al., 2013). Wood is a crucial component for building and handicraft. The sawmill industry produces 10.6% sawdust, 25.9% thin wood, and 14.3% cuts with a total waste of 50.8% of the amount of raw material. Indonesia's total sawn wood production reaches 10 million m3 per year. The abundance of sawmills, especially teak wood, it can be utilized as a variety of more useful items, one of which is activated carbon. Activated carbon can be synthesized from biomass on a porous and contains cellulose, hemicellulose, and lignin (Arami-Niya et al., 2010).

Lignocellulosic chemicals are the primary constituents of the wood (Danish & Ahmad, 2018). Lignocellulose content in teak wood is composed of carbohydrates, namely cellulose (47.5%) and hemicellulose (14.4%), as well as

non-carbohydrate compounds, namely lignin (29.9%) (Sumarna, 2004).

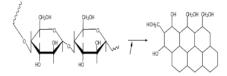


Figure 1. Carbonization reaction (Johannes, 2011)

Figure 1 shows the carbonization reaction in which in that process, the water molecules and volatile compounds such as CO, CH<sub>4</sub>, and H<sub>2</sub> evaporate at 120 °C. At 200 °C, an exothermic reaction occurs due to the breakdown of lignocellulose to acetate, methanol, aldehydes, and other gases such as CO<sub>2</sub> and H<sub>2</sub>O. Finally, the remaining gas comes out at temperatures between 400-500 °C and the remaining dry carbon and tar residue compounds. The use of woody biomass as raw material for activated carbon has been reported by Danish & Ahmad (2018), from coconut husk/shell (Sesuk et al., 2019), from sugarcane (Rawal et al., 2018), and bagasse (Guo et al., 2020), from black wattle bark waste (Lütke et al., 2019), from

Dates (Hussein et al., 2015) even from coal (Li et al., 2019). The type of raw material affects the properties of activated carbon. In addition, other factors such as the activation method, activator agent, and conditions of the activation process (Kumar & Jena, 2016) including temperature and activation contact time (Polii, 2017) affect the character of activated carbon.

The activation process aims to remove impurities from the carbon after the carbonization of biomass; therefore, the surface area increases and contains chemical functional groups (Berhe Gebreegziabher et al., 2019). Activation could be carried out in the form of chemical activation employing of raw materials soaked into chemicals used such as alkali, alkaline earth metal salts, and some inorganic acids (Ozdemir et al., 2014). Chemical activation also aims to obtain active sites on activated carbon, such as functionalization using triethylenetetramine (Tran et al., 2017).

The activation of physics is carried out by heating at high temperatures. Different activators agents will have different effects on the activated carbon character. The activator that has utilized in the activation of carbon was H₃PO₄ (Oginni et al., 2019). In the study, the precursor biomass was impregnated with H<sub>3</sub>PO<sub>4</sub>. Then activated at 900 °C. The surface area of activated carbon was obtained from biomass 1373 and 999 m<sup>2</sup>/g. Activated carbon from date press cake, an agro-industrial waste was carried out by Hasanzadeh et al. (2020) using thermochemical activation with NaOH (NAC) and KOH (KAC). Both activated carbon NAC and KAC produced micropore structures with a surface area of 2623.2 m<sup>2</sup>/g and 2760.0 m<sup>2</sup>/g. Meanwhile activated carbon fibers were prepared from liquefied wood using a ZnCl<sub>2</sub> activator by Liu et al. (2016). Besides Lin et al. (2020) prepared activated carbon from pamelo peel used ZnCl<sub>2</sub> as an activator. From the studies of Liu and Lin, the surface area of activated carbon was 1423 m<sup>2</sup>/g and 1361 m<sup>2</sup>/g respectively. According to Yorgun & Yildiz (2015) among the various activator agents, phosphoric acid is preferred because of its smaller environmental and economic impact. Phosphoric acid also allows the development of micro and mesoporous pores from the resulting activated carbon.

Waste cooking oil is residual frying oil derived from types of cooking oil such as corn oil, vegetable oil, refined oil, and others. Cooking oil is slightly brownish. It has a rancid odor due to the oxidation of the oil during the heating process at high temperatures and repeated use so that the use of used cooking oil in the long

term can damage human health and cause cancer and other diseases. For this reason, proper handling is needed to make waste cooking oil can be beneficial and does not cause harm to human health and the environment. Several studies that have been carried out to handle used cooking oil include the one converted into biodiesel (Sriatun et al., 2015), (Sriatun et al., 2018), adsorption using activated carbon in the manufacture of solid soap (Riyanta & Nurniswati, 2016), adsorption of used cooking oil by zeolites (Alamsyah et al., 2017), (Sobrinho et al., 2019). The adsorption process is carried out by adsorbing the oil on particular contact time and temperature variation; therefore, the level of oil clarity has increased.

This research studied the effect of sulfuric and phosphoric acid as activators. This is based on the nature of corrosive acids decomposes the hemicellulose and lignin components, due to the elemental cellulose is more resistant to acid. The acid will hydrolyze the mlignocellulose glycosidic chain and cut the bonds aryl ether in lignin (Megiyo et al., 2017). The acidity of sulfuric acid is stronger than phosphoric acid, which is expected to give different effects.

In previous studies, no one has published the purification of used cooking oil using activated carbon which is activated by two methods, namely acid activation and heating at a temperature of 300 °C - 500 °C. Therefore, this study examined the effect of the two acids on the characters of activated carbon from teak wood and tested the ability to bleach the waste cooking oil.

### 2. Method

### 2.1. Materials

The remaining pieces of teak wood from sawn wood from the Central Java region.  $H_2SO_4$  (97%),  $Na_2CO_3$  (99%),  $ZnCl_2$  (98%), KI (99%), Iodine (99,8%), and  $Na_2S_2O_3$  (99%). All chemicals are from Merck. Aquadest, waste cooking oil, packaging oil from the supermarket.

### 2.2. Preparation of activated carbon

Carbonization is a burning process of biomass containing cellulose, lignin, and hemicellulose into a whole carbon element. Firstly, pieces of teak wood were cleaned and dried. Then they burned without oxygen with a closed drum until becomes charcoal. This study uses traditional combustion, so it requires observation to ensure carbonization has been completed by

seeing the smoke that comes out while burning. If the smoke produced is black and thick, it shows the formation of charcoal, otherwise. Meanwhile, if the smoke comes out a little, it indicates the formation of charcoal as a whole and needs to be monitored to avoid being ashes completely.

Furthermore, the carbon is crushed using porcelain crucible. After that, the carbon is sieved of 100 mesh, so that the size of carbon was homogeneous. This sample is called activated carbon without activator or activated carbon before activation and coded AC NA.

### 2.3. Chemical and physical activations

In chemical activation, carbon reacted with activator agents, i.e  $\rm H_2SO_4$  and  $\rm H_3PO_4$  in which the concentration varies 1%, 5%, 10%, 15%, and 20% (v/v). The activation process was carried out at a carbon/activator ratio of 1:1, contact time of 24 hours. Afterward, the product is washed with distilled water until a pH of 6-7. The carbonization process produces impurities such as tar compounds that can close the pores of activated carbon. In addition, chemical activation produces residual acid which must be removed. Therefore, it is necessary to wash until a pH of 6-7.

Table1. Code of samples

	300 °C		400 °C		500 °C	
%	H <sub>2</sub> SO <sub>4</sub>	H <sub>3</sub> PO <sub>4</sub>	H <sub>2</sub> SO <sub>4</sub>	H₃PO₄	H <sub>2</sub> SO <sub>4</sub>	H <sub>3</sub> PO <sub>4</sub>
1	S 1-	P 1-	S 1-	P 1-	S 1-	P 1-
	300	300	400	400	500	500
5	S 5-	P 5 -	S 5-	P 5-	S 5-	P 5-
	300	30 0	400	400	500	500
10	S 10-	P 10-	S 10-	P 10-	S 10-	P 10-
	300	300	400	400	500	500
15	S 15-	P 15-	S 15-	P 15-	S 15-	P 15-
	300	300	400	400	500	500
20	S 20-	P 20-	S 20-	P 20-	S 20-	P 20-
	300	300	400	400	500	500

In the final step, the product was calcined at a temperature of 300  $^{\circ}$ C, 400  $^{\circ}$ C, and 500  $^{\circ}$ C for two hours. The activated carbon product is weighed, and the yield is determined. Each sample is coded as in Table 1.

#### 2.4. Determination of yield (SNI 01-1682-1996)

The yield of activated carbon was determined by calculating the ratio of the weight of activated carbon to the weight of activated carbon before being activated using Eq. 1.

$$Yield\ (\%) = \frac{W_a}{W_b} \ x \ 100\%$$
 (1)

 $W_a$  = weight of carbon after activation  $W_b$  = weight of carbon before activation

### 2.5. Determination of water content (SNI 01-1682-1996)

The sample was weighed as much as 1 g and put into a porcelain crucible which has known the weight. Then the sample in the crucible is dried using an oven at 105 °C for 24 hours. The dried sample is cooled in a desiccator and weighed until the weight is constant. The water content is calculated using Eq. 2.

Water content (%) = 
$$\frac{W_i - W_f}{W_i} \times 100\%$$
 (2)

 $W_i$  = initial weight of sample  $W_f$  = final weight of sample

#### 2.6. Determination of ash content (SNI 01-1682-1996)

The sample was weighed as much as 1 g and put in a porcelain crucible that has known the weight. The sample was heated in a furnace at a temperature of  $600~^\circ\text{C}$  for four hours. The sample is cooled in a desiccator and then weighed until the weight is constant. The ash content is calculated using Eq. 3.

$$Ash content (\%) = \frac{w_f}{w_i} \times 100\%$$
 (3)

 $W_i$  = initial weight of sample  $W_f$  = final weight of sample

### 2.7. Determination of Iodine Numbers (IN) (SNI 01-1682-1996)

The activated carbon is weighed as much as 0.50 g and mixed with 50 mL of 0.1 N Iodine solution. After that, the mixture is shaken with a shaker for one minute, then transferred into the centrifugal tube until the activated carbon drops. Next, 10 mL of the filtrate is titrated by 0.1 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution. If the yellow color of the solution starts to faint, so a 1% starch solution is added to the solution as an indicator. Then the solution is titrated until the dark blue becomes a clear color. The iodine number is calculated using Eq. 4.

IN 
$$\binom{mg}{g} = \frac{\left(10 - \frac{VxN}{0.1N}\right)x \ 12.69x5}{W}$$
 (4)

IN: Iodine number (mg/g)

V = Volume of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (mL)

N = Normality of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (N)

12.69 = The amount of iodine corresponds to one mL of 0.1 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution

5 = dilution factor W = weight of activated carbon (g)

### 2.8. Determination of the surface area by Gas Sorption Analyzer (GSA)

Determination of activated carbon structural properties was undertaken by a Gas Sorption Analyzer (GSA) (Quantachrome NovaWin version 11.0) with an outgas time of 3.0 hours, analysis gas of nitrogen, outgas temperature of 300 °C, bath temperature of 77.3 °K. The surface area of activated carbon is determined by a multi-point Brunauer–Emmett–Teller (BET), the total pore volume, and pore size was conducted by the desorption isotherms of the Barret–Joyner–Halenda (BJH) method.

### 2.9. Determination of functional groups by FTIR Spectrometer.

The functional groups of activated carbon were determined using an FTIR Spectrometer (PerkinElmer Spectrum Version 10.03.06) using the KBr method. The absorption of functional groups is observed at the wavenumber of  $400 - 4000 \ cm^{-1}$ .

### 2.10. Adsorption or bleaching of waste cooking oil

Bleaching of used cooking oil is carried out through the interaction of activated carbon (at various concentrations and activator agents) of 0.1 gram with 10 mL of waste cooking oil, at  $100\,^{\circ}\text{C}$  in a stirring at 500 rpm, the contact time of 60 minutes. Heating on the adsorption process aims to auto-oxidize the oil. Activated carbon adsorbents and oils are separated using centrifuges. The filtrate in the form of waste cooking oil (WCO) is characterized by UV-Vis spectrophotometry at  $\lambda$  420–550 nm. Packaged oil is used as a blank (AO). The bleaching ability of activated carbon is obtained using Eq.5.

Bleaching (%) = 
$$100 - \frac{A_{WCO}}{A_{AO}} x$$
 100% (5)  $A_{WCO}$  = absorbance of WCO  $A_{AO}$  = absorbance of AO

### 3. Results and discussion

### 3.1. The yield of activated carbon

The yield is one of the factors that are considered due to commercial production. The yield is an important factor in production effectiveness. Figure 2 shows the yield of activated carbon using  $\rm H_2SO_4$  not influenced by

concentration but influenced by temperature. The higher the temperature, the less the yield. An increase in the activation temperature causes the reaction rate in pore formation to accelerate so that more carbon is converted to CO2 gas so that less carbon remains. The smallest yield was obtained in S 1-500 activated carbon since the greatest mass loss. It is possible that during chemical activation, not much of the impurity was lost due to the low activator concentration so that when activated carbon was still a lot of impurities were physically activated, many impurities are lost and the resulting low yield. While the highest yield was found in S 5-300 activated carbon of 69.72%.

Figure 2 also shows that the yield of activated carbon from teak wood with an  $\rm H_3PO_4$  activator ranges from 14.77 to 63.16%. This result has a similarity to the yield of activated carbon from passiflora ligularis, Inga feuilleei, and native plants of South America (Montalvo Andia et al., 2020).

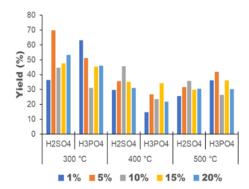


Figure 2. The yield of activated carbon

The data provides information at a temperature of 300 °C tends to have a higher yield. This is caused by the temperature is quite low which only aims to open pores and only undergoes a little oxidation process. In addition, at a temperature of 300 °C cellulose and hemicellulose were gone but there was still lignin. While the temperature of 400 °C tends to have a little yield.

### 3.2. Water content

Determination of the water content aimed to know the hygroscopic properties. The smaller the water content, the better the quality of activated carbon. The high water content indicates the activated carbon has a high affinity to adsorb water molecules which could close the pores. The water content in carbon without activation is 8.94%.

Figure 3 shows the water content of activated carbon fluctuated in range 2.1%-11.84% independent of the activator type and concentration and the activation temperature. Theoretically, the water content decreased when increasing activation temperature and activator concentration. Due to the treatments will encourage impurities resulting from carbonization and decomposition of other elements from the activated carbon pore. Therefore, a larger pore volume of activated carbon formed and smaller water content.

Referring to SNI 01-1682-1996, the maximum of water content in activated carbon is 15%, so all activated carbon from activation and carbon without activation are eligible.



Figure 3. The water content of activated carbon after activation

#### 3.3. Ash content

Ash content is used to determine inorganic components or metal oxides contained in activated carbon. The SNI 01-1682-1996 standard declares that ash content <10%. Excessive ash can clogged pores and reduced the surface area of activated carbon. Ash content of activated carbon before activation was 5.776%.

Figure 4 shows that the ash content of activated carbon by H<sub>2</sub>SO<sub>4</sub> relatively lower than H<sub>3</sub>PO<sub>4</sub>. The lowest ash content is found in activated carbon S 20-300, i.e. 0.87%. In contrast, the highest one is found in the P 20-400 sample. The high ash content is caused by the oxidation process of activated carbon during physical activation. This has an impact on the less optimal absorption of activated carbon. It is possible for inorganic components in the ash such as metal oxides to spread into the pores of the activated carbon, so that the performance of the activated carbon decreases. Meanwhile, the decrease of ash content is thought to be due to the remaining metal oxides dissolving in H<sub>2</sub>SO<sub>4</sub> resulting in ions and forming sulfate salts that dissolve in washing.

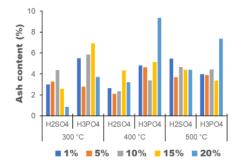


Figure 4. The ash content of activated carbon after activation

#### 3.4. Iodine Number (IN)

The adsorption ability of activated carbon to iodine solution is one of the important parameters that indicate the quality of activated carbon. The higher the iodine absorption, the better of activated carbon quality. Therefore, the absorption of iodine is an important indicator in assessing activated carbon. In this study iodine number (IN) of activated carbon without activator is 260,11 mg/g. Meanwhile, the IN for other samples varies depending on the activator concentration and the activation temperature.

Referring to Figure 5, the iodine number of carbon samples varies and does not have a good trend, but there appears to be a decrease in IN when at high concentrations of 20% and temperatures of 400 °C and 500 °C. Activated carbon produced by activation of H<sub>3</sub>PO<sub>4</sub> 15% and heating 400 °C (P 15-400 sample) has the largest IN, namely 1211.4 mg/g followed by H<sub>2</sub>SO<sub>4</sub> 15% at 400 °C (S 15-400 sample) which is 1204.1 mg/g. Meanwhile, IN <500 mg/g is owned by activated carbon as a result of activation H<sub>3</sub>PO<sub>4</sub> 15% and heating 300 °C (P 15-300 sample) and  $H_2SO_4$  5% at 500 °C (S 5-500 sample). However, the IN of all activated carbon results from all treatments higher than IN of activated carbon without activation which was only 260.11 mg/g. Sulfuric acid and phosphoric acid at various concentrations followed by heating can increase the degradation of activated carbon, resulting in an increased dehydration effect.

Activation of carbon occurs through interactions with activator agents penetrating and spreading into gaps or pores between hexagonal structural carbon; consequently, the carbon surface eroded. When we used  $H_2 {\rm SO}_4$  as an activator,  ${\rm SO}_4^{2^-}$  ions enter the hexagonal cracks of carbon, and then they could bind the impurities that cover the pores. Sulfuric acid

also acts as an oxidizing agent for molecules on the surface of activated carbon. Activated carbon can decompose inorganic and organic compounds such as functional groups attached to impurities (Al-Qodah & Shawabkeh, 2009); therefore, when it becomes activated carbon on the surface, it contains more oxygen as C=O is more active. It can observe changes in functional groups in carbon to activated carbon in Figure 6.

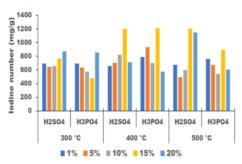
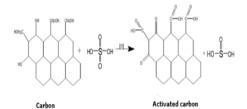


Figure 5. The iodine number of activated carbon



**Figure 6.** Mechanisms of functional group change in activated carbon upon acid activation

Furthermore, evidence of changes related to the presence of functional groups on activated carbon can be seen in the FTIR spectra in Figure 7 and Figure 8. The presence of heating energy as a physical activation aims to remove residual impurities. The higher activation temperature, the more energy is given, therefore the more pores that open, so that the surface area increases.

The SNI quality standard requires the minimum IN of activated carbon is 750 mg/g, so not all samples in this study fulfill it. However, it can be said that acid activator, both sulfuric and phosphoric acid at a concentration of 15% followed by heating at 100 °C, is proven to be effective in increasing the adsorption capacity of activated carbon. The IN results of activated carbon obtained in this study are according to (Andas & Wazil, 2019), where the IN of activated carbon resulted from activation with H<sub>3</sub>PO<sub>4</sub> is 1299.3 mg/g.

### 3.5. The Functional group of activated carbon by FTIR.

The adsorption ability of activated carbon is not only determined by the characters such as water content, ash content, and iodine number. The adsorption ability is also influenced by the chemical composition of activated carbon in the form of a functional group which is an active group of activated carbon. Of all samples with various concentration and activation temperatures, only a few samples with low (1% or 5%) and high (15% or 20%) concentrations were taken for functional group analysis by FTIR. The sample selection is considered to represent the structure of the sample to obtain the desired information.

Figure 7 and Figure 8 present patterns of peaks absorption at the wavenumber 3450 cm $^{-1}$ , 2350 cm $^{-1}$ , 1628 cm $^{-1}$ , 1378 cm $^{-1}$  and 650 cm $^{-1}$ . But there is very weak adsorption at a wavenumber of 2350 cm $^{-1}$  in sample activated carbon without activator, and P 20-300, according to Montalvo Andia et al. (2020) the band observed at 2353 cm $^{-1}$  corresponds to vibrations present in alkyne groups C  $\equiv$  C. The low intensity of the spectra due to the release of volatile substances during the carbonization.

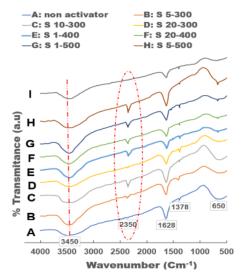


Figure 7. FTIR spectra of activated carbon by H<sub>2</sub>SO<sub>4</sub> activator in various activation temperature

The peak at the wavenumber 3450 cm<sup>-1</sup> is assumed as the O-H group. The intensity of this O-H group arises due to the degradation of materials from lignin and cellulose. The stretching vibrations of C=O and C-H groups are shown by the peaks at wavenumbers 1628

cm<sup>-1</sup> and 1378 cm<sup>-1</sup> respectively. Furthermore, the C=C bending vibration of the aromatic ring is found in the wavenumber 650 cm<sup>-1</sup>.

The spectra in Figure 7 and 8 appear a very wide absorption band dan a very weak intensity at the wave number 1008 -  $1200~cm^{-1}$ . This shows that the activated carbon contains alcohol, phenol, ester, and ether groups (Andas & Wazil, 2019) which are related to peaks at wavenumbers  $1628~cm^{-1}$ . The activated carbon after being activated by sulfuric acid or phosphoric acid at a concentration of 20% and temperatures of  $300~^{\circ}$ C,  $400~^{\circ}$ C, and  $500~^{\circ}$ C inclined to have a lower adsorption intensity. Reduction in the intensity of the peaks and narrowing of the adsorption band indicates the loss of volatile compounds after the activation using  $H_2SO_4$  and  $H_3PO_4$ .

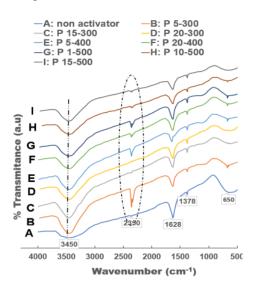


Figure 8. FTIR spectra of activated carbon by H<sub>3</sub>PO<sub>4</sub> activator in various activation temperature

The sulfuric and phosphoric acid act as oxidation agents, they can oxidize organic compounds on the carbon surface. There is a change in the primary alcohol group into carboxylic acids and secondary alcohols to ketones. This underlies the formation of the activated carbon structure. The difference in the chemical structure of carbon and activated carbon is characterized by changes in functional groups and shifts in absorption bands. The absorption band at wave number 3450 cm<sup>-1</sup>, namely the without activation treatment (non activator) OH group of carbon is wider than that of activated carbon. This indicates that the carbon without activation still contains a lot of OH groups compared to the activated carbon.

The FTIR absorption patterns of activated carbon in this study are in accordance with Om Prakash et al. (2020) for activated carbon prepared from arhar stalks using KOH activator in 1:2 wt ratio and heating at 800 °C for two hours. In addition, the FTIR spectra patterns of activated carbon in this study also have similarities to the standard activated carbon spectra pattern used in the research of (Rahmadani & Kurniawati, 2017) and (Mistar et al., 2020). From the FTIR data, it can be concluded that the decrease in peak intensity and the narrowing of the adsorption band occurs along with the increase in the activator concentration and the activation temperature.

### 3.6. Surface area, pore-volume, and pore size of activated carbon

The surface area of activated carbon as adsorbent is an important parameter. Activated carbons act as a good adsorbent if they have a high surface area. The determination of surface area and pore size, only a few samples were selected to be analyzed namely S 5-400, P 5-400, S 15-500, and P 15-500, considering that they have low water content, ash content, and high iodine numbers.

Figure 9 shows nitrogen adsorption isotherm of activated carbon at 77 °K. The nitrogen adsorption-desorption pattern between activated carbon without the activator (before the activation treatment) and after the addition of H<sub>2</sub>SO<sub>4</sub> and H<sub>3</sub>PO<sub>4</sub> activators that followed by heating was different. Based on the IUPAC classification, Figure 9 shows the isotherm is a mixture of types I and IV. Activated carbon without activator (AC NA) has a type I isotherm which is associated with microporous structures. The type I isotherm shows low adsorption of nitrogen gas at a relative pressure of P/Po ~ 1. Meanwhile, S 5-400, P 5-400, S 15-500, and P 15-500 samples have a type IV isotherm which is a mixture of microporous and mesoporous structures. At intermediate and high relative pressures, the isotherms are of type IV with a hysteresis loop of type H4 associated with monolayermultilayer adsorption followed by capillary condensation in narrow slit-like pores (Kumar & Jena, 2016). During activation using H<sub>2</sub>SO<sub>4</sub> and H<sub>3</sub>PO<sub>4</sub> and heating at 400°C and 500 °C volatile matter, residual tar, and unorganized carbon are driven out of the material, resulting in the formation of pores on the surface (Muniandy et al., 2014).

The volume of nitrogen adsorption slightly increases with increasing  $H_2SO_4$  concentration and activation temperature from S 5-400 to S 15-500 indicating the gradual development of

the porous structure. However, it is the opposite when the concentration of phosphoric acid and the activation temperature increases, the volume of adsorbed nitrogen decreases which is connected with the deterioration of porous structure. The deterioration of the the porous structure is most probably due to the destruction of the wall between adjacent micropores by reaction with phosphoric acid (Sych et al., 2012).

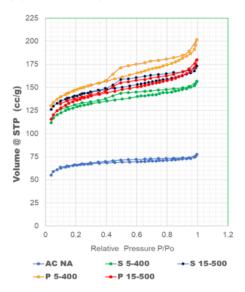


Figure 9. The Pattern of adsorption isotherms of activated carbon from teak wood

**Table 2.** Surface area, pore volume and pore size of carbon before and after activation

carbo	carbon before and after deavadon				
Sample Code	Pore Volume (cc/g)	Average pore diameter (Å)	Surface area (m²/g)		
AC NA	0.018	30.889	192.634		
S 5-400	0.043	34.112	384.882		
P 5-400	0,089	34,168	445,3		
S 15-500	0.055	31.052	416.536		
P 15-500	0,071	34.107	412.58		

Table 2 presents the surface area, porevolume, and pore size of activated carbon. From Table 2, it is known that activated carbon before the activation treatment has a surface area of 192.694  $\text{m}^2/\text{g}$ . Based on Table 2, the surface area of activated carbon after adding the activator with both  $\text{H}_2\text{SO}_4$  and  $\text{H}_3\text{PO}_4$  and heating increased two times become 384,882 to 445.3  $\text{m}^2/\text{g}$ . The surface area of activated

carbon is almost the same as the surface area of activated carbon from teak sawdust reported by (Ismadji et al., 2005), which is 341.78 m²/g from the vacuum pyrolysis method and 412.73 m²/g from atmospheric pyrolysis. The increase of the surface area value is proportional to the increase in the iodine number discussed previously.

The activated carbon of S 15-500, P 5-400, and P 15-500 have a high surface area and pore volume even though the pore size does not increase significantly. It is assumed that activated carbon is more pores. Furthermore, in the previous discussion about FTIR spectra in Figure 6 and 7, these samples do not have many functional groups, so there is an assumption that the role in the adsorption is the pores. Another information from Table 2 is that all activated carbons both before (AC NA) and after activation using H2SO4 and H3PO4 activators have an average pore diameter of 30-34 Å which is included in the mesoporous category according to the IUPAC classification. Although the activated carbon before activation (AC NA) has a pore size of 30.889 Å. Based on the IUPAC classification, it is mesoporous in structure, however, the surface area is only 192.694 m<sup>2</sup>/g, and the ability to absorb nitrogen very small, much smaller than the other samples. Therefore, the AC NA structure tends to be like a micropore. It is possible that AC NA only has a few pores and is a closed pore type (Schubert & Hüsing, 2012). Another possibility is that the mouth of the pore is large but inside is narrow. It is clear that both activations using acid and heating are required.

### 3.7. Bleaching of waste cooking oil

Dyes of cooking oil include a-carotene,  $\beta$ -carotene, xanthophyll, chlorophyll, sometimes anthocyanin, and others (Ketaren, 1986). When the components of cooking oil are oxidized or degraded it can produce other colors. Used cooking oil contains impurities that cause the oil to turn brown. The addition of activated carbon can adsorb the dye therefore the cooking oil is paler (yellowish).

Bleaching is a stage of the refining process to remove dyes from oil. Generally, bleaching of oil is carried out by heating; therefore, the components such as water, aldehydes, ketones resulting from oil degradation, fatty acids with a low weight of molecule can be evaporated (Ketaren, 1986). The presence of these components reduces the quality of cooking oil and causes health problems. In this study, bleaching of the waste cooking oil used activated carbon. The bleaching ability of activated carbon in various activators

concentration and activation temperature is shown in Figure 10.

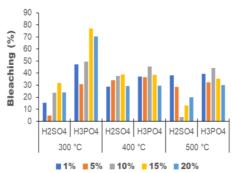


Figure 10. The bleaching ability of activated carbon from teak wood

The level of the paleness of used cooking oil is determined by the absorbance amount of 2.28, whereas as a comparison, packaged cooking oil (pure oil) has an absorbance value of 0.023. It is assumed that components of cooking oil such as carotenoids and vitamin E are oxidized by peroxides. Besides, it is also possible to have dissolved components in oil when cooking. During the cooking process, the oil undergoes degradation, oxidation, hydrolysis, and polymerization reactions due to interactions with heat, air, and water. Oxidation reactions can also occur during the storage period.

Figure 10 shows that the percentage of used cooking oil bleaching by activated carbon with H<sub>2</sub>SO<sub>4</sub> activator at almost all concentrations has increased to a temperature of 400 °C and decreased when the temperature was raised to 500 °C. The energy of heating causes more functional group formation on the surface and pore opening. However, when the temperature is higher, it is possible for the decomposition of the active group, or the ash formation that can cover the pore. Therefore, it reduces the interaction ability with the used cooking oil components. Meanwhile, in the H<sub>3</sub>PO<sub>4</sub> activator, the percentage of bleaching was relatively the same at various activation temperatures except for samples P 15-300 and P 20-300. Figure 9 also shows that the percentage of bleaching tends to decrease at the activator concentration of 20%, where it is possible that a high concentration of H<sub>2</sub>SO<sub>4</sub> and H<sub>3</sub>PO<sub>4</sub> damage the activated carbon walls. The bleaching ability of activated carbon correlated to surface area and iodine number characters.

### 4. Conclusions

From the results and discussion, it can be concluded that teak wood can be used as raw

material for the production of activated carbon. All activated carbon after activation using H<sub>2</sub>SO<sub>4</sub> or H<sub>3</sub>PO<sub>4</sub> followed heating have water content and ash content in accordance with the SNI requirements (01-1682-1996). The water content of all activated carbons with H<sub>3</sub>PO<sub>4</sub> activator ranged 2.10-9.57%, with H<sub>2</sub>SO<sub>4</sub> ranged 3.36-11.74%. The ash content of activated carbons with  $H_3PO_4$  was 2.78-9.36%and H<sub>2</sub>SO<sub>4</sub> was 0.87-5.46%. Meanwhile, the iodine numbers ranged 481.1-1211.4 mg/g and 494.8-1204 mg/g for  $H_3PO_4$  and  $H_2SO_4$ , respectively. Activated carbon with H<sub>3</sub>PO<sub>4</sub> of 15% and an activation temperature of 400 °C has a surface area of 445.30 m<sup>2</sup>/g. The H<sub>2</sub>SO<sub>4</sub> dan H<sub>3</sub>PO<sub>4</sub> activators can be used to improve the quality of activated carbon in absorbing dyes in used cooking oil, where the optimum concentration is 10-15% (v/v). The H<sub>3</sub>PO<sub>4</sub> activator tends to produce a higher bleaching percentage than H<sub>2</sub>SO<sub>4</sub>.

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