

Synthesis Activated Charcoal of Malapari (*Pongamia pinnata*) Fruit peel

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ABSTRACT

Objective: Utilizing *malapari* peel as a raw material of activated charcoal and determine its best quality.

Method: The samples were carbonized at a temperature of 300°C, 400°C, and 450°C. The resulting charcoal was activated chemically and physically. Activated charcoal was characterized based on SNI 06-37301995. The best quality activated charcoal was determined by its specific surface area. The best quality *malapari* peel was washed with 10% HCl. Furthermore, the best quality activated charcoal before and after being washed with 10% HCl was analyzed micrograph using Scanning Electron Microscopy-Energy Dispersive Analysis X-Ray (SEM-EDAX).

Results: Carbonization of samples at 450°C, continued activation by 2% phosphoric acid, and physical activation at 750°C by vapor streaming for 60 minutes produced the best quality activated charcoal by a methylene blue adsorption capacity of 119.50 mg / g. The active charcoal was washed by 10% HCl was able to increase the adsorption capacity of the methylene blue solution to 193 mg/g fulfilling the requirements of SNI 06-3730-1995 by specific surface area and pore surface of 715 m²/g and 545.04 μm².

Conclusion: The best quality activated charcoal was produced by 450°C temperature carbonization process, activated using 2% phosphoric acid followed by physics activation at 750°C by vapor flow for 60 minutes.

Keywords: activated charcoal, activation, carbonization, *malapari* peel

I. INTRODUCTION

Currently, the utilization of *malapari* as a biofuel was found in India [2]. But the process left an untapped peel. Meanwhile, the prospect of bio-fuel processing based on raw fruit very promising because of its easy breeding. One hectare *malapari* population can produce 9 tons of dried *malapari* seeds, although the plant age

is more than 50 years old but still produce seeds [16]. So it needs to be studied early on the utilization of the *malapari* peel in order to increase added value and not pollute the environment.

Malapari fruit peel texture is quite hard, likely contains lignin and selolusa that can be used as a source of carbon in the manufacture of activated charcoal. Several researchers have

also reported waste utilization as a carbon source in the production of activated charcoal such as solid waste agar [4], tapioca solid waste [13], coconut and straw fibers [18] and wood puppa logging waste [10].

The process of making activated charcoal can be through two stages, namely carbonization, and activation. The process of carbonization is by using pyrolysis method, namely the thermochemical decomposition process with high temperature to the organic material without air. There are two activation processes, namely physical activation, and chemical activation. The principle of physical activation is the provision of water vapor or CO₂ to the heated charcoal, whereas the chemical activation is the immersion of the charcoal in chemical solutions such as CaCl₂, ZnCl₂, H₃PO₄, NaOH, KOH, and Na₂SO₄ [20].

In this research, malapari fruit peel becomes activated charcoal by carbonization at 300°C, 400°C and 450°C temperature. Meanwhile, the activation process was carried out by immersion in 1% and 2% H₃PO₄ solution, so that the activated charcoal was produced chemically, then the activated charcoal was heated at the activation furnace with a temperature of 750°C while steam for 60 and 90 minutes, to obtain activated charcoal Chemically-physically. H₃PO₄ not only functions as an activator but also as a protective material from heat [19]. So the presence of H₃PO₄ can also be interpreted as a substance that slows down the rate of carbon oxidation reaction so that the yield of activated charcoal is increased.

II. METHOD

Time and Location of Research

This research was conducted from February to June 2014 at Laboratory Center for Forest Products Research and Development Indonesia.

Sample

The Sample was used in the research was malapari fruit peel from *Batu Karas*, West Java.

Carbonization

Calipari fruit peel was weighed and then carbonized with three treatments at 300°C, 400°C, and 450 °C pyrolyzes. Pyrolysis was performed in a stainless steel furnace for 4 hours. Then the carbonization was turned off and allowed to cool (\pm 20 hours). The charcoal was removed and the rendement was determined, followed by the activation process.

Activation

The charcoal of malapari fruit peel was chemically activated by soaking in 1% and 2% phosphoric acid solution for 24 hours. Then it was drained, washed and dried by solar heat, to obtain chemically activated charcoal [22]. Thereafter, chemically activated charcoal went into the physical activation furnace reactor. The furnace reactor was prepared by increasing the temperature to 750 °C gradually until reaching the constant temperature and the pressure was made constant at 35 mbar. Water vapor is also carried in the reactor with time 60 and 90 minutes [14]. After the activation process was completed, the furnace reactor was allowed to cool (\pm 24 hours) and in this process would be produced charcoal activated chemically-physically. The activated charcoal of malapari fruit peel yielded was weighed and determined its rendement and smoothed with a size of 200 mesh using Milling Herzog for 1 minute. This process produces 12 treatments such as Table 1.

Table 1 The condition of activated charcoal making of malapari fruit peel

Treatment	Carbonization (K)			Activation Chemically (H)		Activation Physically (750 °C) (S)	
	300 °C (3)	400 °C (4)	450 (45) C	H ₃ PO ₄ 1% (1)	H ₃ PO ₄ 2% (2)	Steam 60 second (6)	Steam 90 second (9)
K3H1S6	√			√		√	
K3H1S9	√			√			√
K3H2S6	√				√	√	
K3H2S9	√				√		√
K4H1S6		√		√		√	
K4H1S9		√		√			√
K4H2S6		√			√	√	
K4H2S9		√			√		√
K45H1S6			√	√		√	
K45H1S9			√	√			√
K45H2S6			√		√	√	
K45H2S9			√		√		√

Activated charcoal from malapari fruit peel obtained in characterization based on SNI 06-3730-1995. These characteristics include determination of moisture content, ash content, flying ash content, bound carbon content, an adsorption capacity of benzene, iodine and methylene blue. The specific surface area of activated charcoal of malapari fruit peel was determined by the adsorption capacity of methylene blue. The activated charcoal of malapari fruit peel with the highest specific surface area was the best quality activated charcoal. The activated charcoal was washed with HCl 10% to remove the impurities. Activated charcoal of best quality malapari peel before and after washing with HCl 10% analyzed micrograph by Scanning Electron Microscope-Energy Dispersive Analysis X-Ray (SEM-EDAX) to see different the pore area and the impurity surface area.

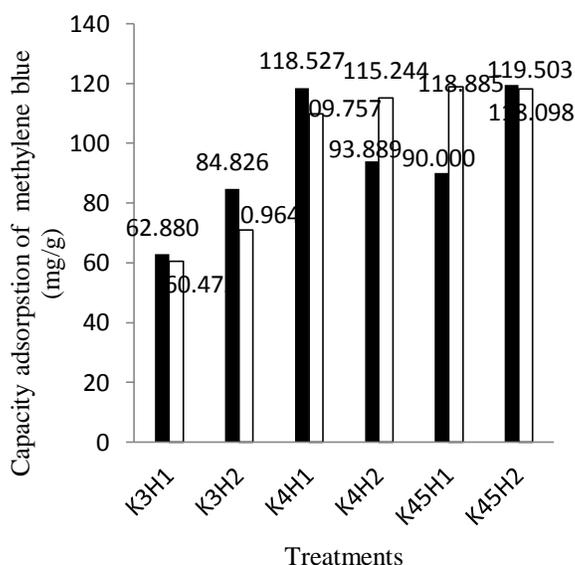


Figure 2 The effect of chemical-physical activation with water vapor flowing for 60 minutes (■) and 90 minutes (□) on methylene blue adsorption capacity.

III. RESULT

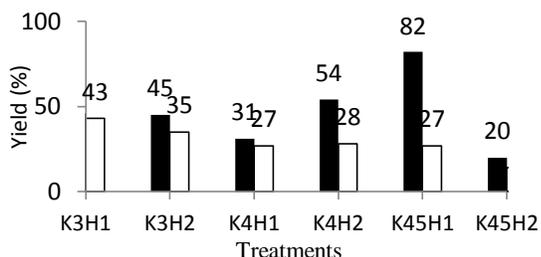


Figure 1 The influence of chemical-physical activation with the duration of water vapor 60 minutes (■) and 90 minute (□) on the activated charcoal rendement.

IV. DISCUSSION

The activated charcoal of malapari peel generated from different carbonization temperatures and means of activation will produce different characteristics. It was due to the rate of decomposition of different feedstocks with an increase in carbonization temperature. The more components that break down the more potential pore formation. At 300°C there has been decomposition of selolusa and at temperatures above 300°C not only

decomposed of selolusa, but also decomposed of lignin. The decomposed component forms charcoal, CO, CO₂, CH₄, phenol, ash, and tar [11]. It had evidenced by the decrease of charcoal yield along with the increase of carbonization temperature due to the formation of volatile components such as CO, CO₂, and CH₄. During the carbonization process, the carbonaceous material undergoes a fragmentation process to form a thermostable aromatic structure that initiates polyaromatic formation. The process occurs at a carbonization temperature of ± 400°C [15]. The size of the formed charcoal pore was still small because it was covered by ash, tar, and resin formed during the carbonization process. Components formed from the decomposition of lignin during the carbonization process were shown in Figure. 3. The ash, tar, and resin components covering the pores can be removed through the activation process. The use of H₃PO₄ as activator material can expand activated charcoal pore by dissolving the ash

and tar covering its pore [10]. H₃PO₄ also plays an important role in the formation of mesoporous and microporous structures in the inner structure of activated charcoal [21]. Thus, an increase in H₃PO₄ concentration may provide an increased potential for the expansion and formation of activated charcoal pores. Further physical activation also plays an important role in the formation of pores through the process of decomposition of hydrocarbons to form volatile compounds because of the heat provided. The surface area of activated charcoal will increase with the loss of such volatile compounds [9]. This physical activation is accompanied by the flow of water vapor into the furnace reactor causing the reaction of carbon oxidation to form CO₂ and H₂ gases. The oxidation reaction increases with the increase of water vapor flow, so that the activated charcoal yield decreases (Figure 1). The yield of activated charcoal decreases with the increase of water vapor streaming time [13].

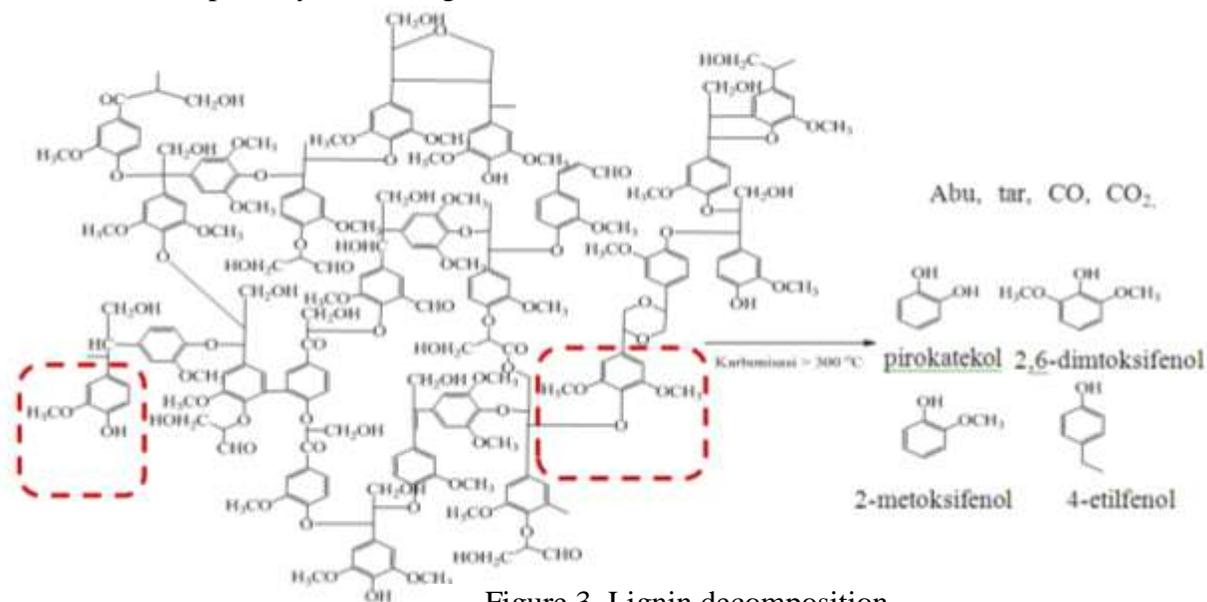


Figure 3 Lignin decomposition

The yield of activated charcoal also decreases with increasing H₃PO₄ concentration due to the increasing of metal oxide, tar and resin together (Figure 1). The results characteristics of activated charcoal of malapari fruit peel indicate that only water content and flying substances meets the requirements of SNI 06-3730-1995. Low levels of carbon bound, iodine

adsorption capacity, benzene, and methylene blue are influenced by high ash content. High ash content can cover the pores of activated charcoal of malapari peel, so its function as an adsorbent to the solution and gas decreases. The best quality activated charcoal of malapari peel derived from carbonized raw materials at 450 °C, chemically activated using H₃PO₄ 2%

followed by physical activation at 750 °C with vapor flow for 60 min. The adsorption capacity of methylene blue solution of 119.50 mg/g (Figur. 2) with LPS of 443.08 m²/g. It's adsorption capacity to methylene blue is greater than the activated charcoal from coconut shell activated with ZnCl₂ [3] and activated charcoal from kapok seeds activated with 85% phosphoric acid [23], but smaller than the activated charcoal from tea seed shells activated with ZnCl₂ [8] and activated charcoal from the bark of Chinese trees [6]. The higher the adsorption capacity of the methylene blue solution, the greater the specific surface area. Large the specific surface area causes more adsorbate molecules that can interact with adsorbate molecules.

Activated charcoal of malapari peel's ability to adsorb methylene blue increases with increasing carbonization temperature and phosphoric acid concentration (Figure 2). Increasing carbonization temperature can improve the decomposition of raw material components such as selolusa and lignin. Decomposition of the component causes the formation of pores, but is still covered by the impurities of ash and tar. The use of phosphoric acid as an activator serves to dissolve tar and minerals contained in the ash so that the pores open. Increasing the concentration of phosphoric acid may increase the dissolution of the impurities. Increased concentrations of phosphoric acid may increase pore volume [9]. The Activated charcoal of malapari peel adsorption capacity of the methylene blue solution decreased slightly with the increase of water vaporization time. It was probably caused by increased carbon oxidation, which could damage some of the pores that have been formed.

The best quality activated charcoal of malapari peel was washed with HCl 10% to remove the impurities. This washing aims to reduce the content of metal oxides and other impurities that potentially cover the pores of Activated charcoal of malapari peel. Analysis result using SEM-EDAX known surface Activated charcoal of malapari peel before washed with 10% HCl looks dirty (Figure 4). The impurity

components are oxides of sodium, magnesium, potassium, calcium, aluminum, silicon, sulfur, and chlorine (Figure 5). After washing, all impurities are lost except chlorine and potassium by 0.18% and 0.36% (Figure 6).

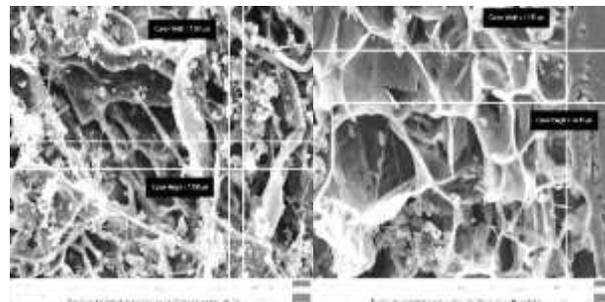


Figure 4 Micrograph activated charcoal of malapari peel and its composition before (left) and after (right) was washed with HCl 10%

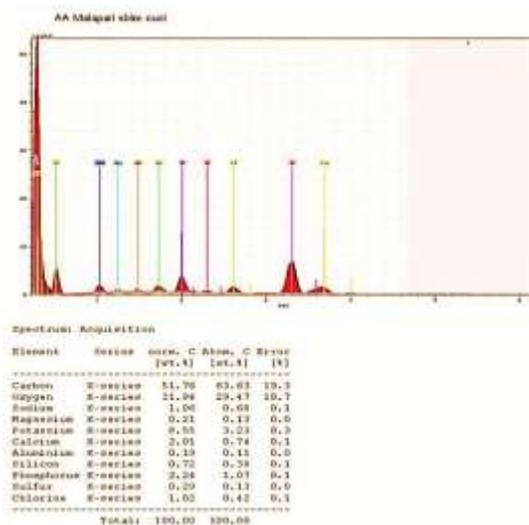


Figure 5 The content of metal on activated charcoal malapari before washed with HCl 10%

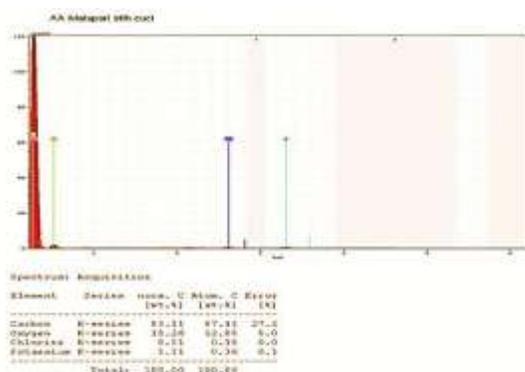
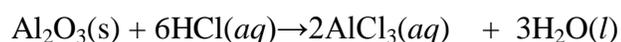
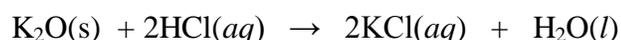
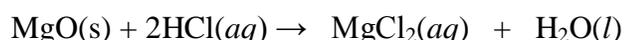
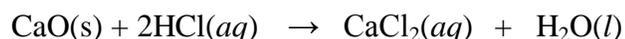
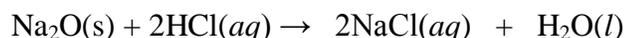


Figure 6 The content of metal on activated charcoal malapari after washing with HCl 10%

The metal oxides formed during the carbonization process and the physics activation disappears from the charcoal activated surface as they change from the solid phase to the dissolved phase. The reaction involved was the salting reaction. This reaction produced salt and water. All of the salts formed from the oxide are soluble in water and could form an electrolyte solution [21]. Most of the chloride-containing compounds could be water-soluble at 25°C, except those containing Ag⁺, Hg²⁺, and Pb²⁺. The metal oxide dissolution reaction was shown in the following reaction [7].



The conversion of metal oxides to dissolved salts caused activated charcoal malapari peel surfaces to become cleaner (Figure. 5), resulting in increased pore area and specific surface area of 35 μm² and 443 m²/g to 138 μm² and 715 m²/g respectively. The adsorption capacity of methylene blue also increased from 119.50 mg/g to 193 mg/g which has fulfilled the requirements of SNI 06-3730-1995 [5]. The resulting surface area is close to the commercial surface area of 200-325 mesh, ie 750 m²/g [1].

The surface area of activated charcoal malapari peel production is greater than the activated charcoal from kapok seed [23].

V. CONCLUSION

The activated charcoal of malapari fruit had been successfully created. Its characteristic was only water content and flying substance which fulfill the requirements of SNI 06-3730-1995. The best quality Activated charcoal of malapari peel was produced from a carbonized sample at 450°C, activated with 2% H₃PO₄ and continued activation at 750°C along by water vapor flow for 60 min. The adsorption capacity of methylene blue is 119.50 mg/g. Advanced washing by 10% HCl increases the pore area of Activated charcoal of malapari peel and its adsorption capacity to methylene blue to 193 mg/g (fulfilling the requirements of SNI 06-3730-1995). Specific surface area and Activated charcoal of malapari peel pores of 715 m²/g and 138 μm².

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