SURFACE STRUCTURE AND CHEMICAL COMPOSITION OF COCONUT SHELL CHARCOAL USING NIRA AREN (*Arenga pinnata*) ACID ACTIVATION

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ABSTRACT

Coconut shell is a waste of copra manufactured in North Sulawesi, Indonesia. Pyrolysis coconut shell charcoal is potentially a raw material for bio-adsorbent for health. Bio-adsorbent activated carbon is produced from coconut shells using an acetic acid activator from palm sap (Arenga pinnata). Coconut shell pyrolysis was carried out at a temperature of 400 - 600°C and an acetic acid activator was obtained by fermenting the palm for six months. The results of the FT-IR analysis of coconut shell charcoal activated with CH_3COOH 3 M and CH_3COOH fermented palm sap showed a decrease or removal of impurity functional groups and an increase in coconut shell charcoal functional groups. The results of SEM-EDS analysis of coconut shell charcoal with CH_3COOH activator fermented palm sap has a larger average pore diameter of 7.82 μm. The elemental content of carbon C is 87.88 % by mass. Thus, coconut shell charcoal activated using CH_3COOH resulting from palm sap fermentation has potential as an adsorbent for industrial liquid waste.

Keywords: *coconut shell charcoal, nira aren, surface structure, chemical composition.*

INTRODUCTION

The part of the coconut plant that has not been used optimally is the coconut shell. So far, coconut shells are only considered waste from the coconut processing industry, especially from the coconut oil industry and are only disposed of or burned directly and indirectly [1]. If processed further, it will produce higher economic benefits and value [2]. One way to process coconut shells is through the pyrolysis process [3]. The pyrolysis process will obtain coconut shell charcoal, liquid smoke, tar, and non-condensable gases. Coconut shell charcoal can be further processed into briquettes, activated carbon [4, 5], carbon electrodes [6 - 8], and bio-coke [9, 10].

The main composition of coconut shells consists of cellulose, lignin, and hemicellulose with C, O, H, and N atoms. These organic materials contain functional groups such as hydroxyl R-OH, alkane R-(CH_2)n- R', carboxyl R-COOH, carbonyl R-CO-R', ester R-CO-O-R', linear and cyclic ether groups R-O-R' with varying amounts. The most common chemical reaction is combustion, which combines fuel with oxygen to form a product compound. This chemical transformation is potential energy on a molecular scale. In this case, it is related to the position of atoms and molecular structures [1].

Nano-carbons from coconut shells can be made in two stages: carbonation and activation. Carbonation is an indoor combustion process without oxygen and other chemicals [11]. At the same time, activation is required to convert the carbonization product into an adsorbent with a large surface area and a high-purity carbon C element composition [12 - 14]. Activation is the treatment of charcoal which aims to enlarge the pores, namely by breaking hydrocarbon bonds or oxidizing surface molecules so that the charcoal undergoes changes in properties, both physical and chemical, namely the surface area increases and affects the adsorption power [15].

Chemical activation in the study using acetic acid fermented palm sap. This is the novelty of this research using CH_3COOH fermented palm sap. Where palm sap is a local resource in North Sulawesi, Indonesia. Improving the purity of acetic acid fermented from palm sap is done by re-distilling the acetic acid. Activation of coconut shell charcoal using palm sap acetic acid will obtain information on surface structure, pore size, and chemical composition of carbon C elements. As a comparison, this research used 3M CH_3COOH. Choosing CH_3COOH with a concentration of 3M is expected to produce coconut shell charcoal with a high level of purity. The characteristics of coconut shell charcoal with activation of acetic acid produced by palm sap fermentation were characterized using FT-IR and SEM-EDS.

EXPERIMENTAL

Tools and materials

The tools include some glassware commonly used in laboratories, agar mortar, 100 mesh sieve (USA standard Testing Sieve), gravity convection oven, carbolite electric furnace model 2132 (Max Temperature 1200^{o}C), Mettler balance model AE 200, thermometer, and clamp. The materials used include coconut shell from North Sulawesi Province as a source of carbon raw materials, starch from p.a Merck, acetic acid from p.a. Merck, universal indicator, Whatmann paper no. 42, palm sap, and aquadest.

Research procedure

Coconut shells from the old deep-coconut species that have not fallen from the tree are the primary raw materials for the research sample. The coconut shell is cleaned of attached fibres and shaped into a smaller size. Coconut shell chips are put in a pyrolysis reactor with a process temperature of 400 - 600^{o}C. The coconut shell charcoal obtained was ground and crushed to uniform particle size [16]. The coconut shell charcoal powder obtained from the pyrolysis results was then sieved with a 100 mesh sieve to obtain carbon powder that passed a 100 mesh sieve. Carbon powder was activated using two activators, 3M CH_3COOH and CH_3COOH fermented palm sap, each for 4 h at boiling. Then the carbon was washed with distilled water until the washing water showed a pH of 7. Then it was dried in an oven at 383 K overnight.

FT-IR measurement using the Shimadzu FT-IR Spectrophotometer IRprestige-21 type works in a scan range of 4000 - 340 cm^{-1}, resolution 4 cm^{-1}, and scans 2 - 3 s. FT-IR analysis using IR solution software for spectrum measurement and peak detection. The JCM-6000 PLUS SEM tool works at a voltage of 15.00 kV and an energy range of 0 - 20 keV. The SEM tool us the JED-230 Analysis Station Plus for EDS analysis. The JED-2300 Analysis Station Plus is equipped with JEOL's DrySD™ (Dry Silicon Drift Detector), a high-speed analyser, and analytical software specially designed for JEOL electron microscopes. Particle analysis software is used to determine the pore diameter of charcoal [16]. Measurement of pore diameter to obtain macropore size by comparing the average pore diameter of coconut shell charcoal activated using 3M CH_3COOH and CH_3COOH fermented palm sap.

RESULTS AND DISCUSSION

FT-IR analysis was used to determine the functional groups contained in the charcoal based on the absorption band pattern at the wave number. The absorption band pattern of coconut shell charcoal activated by 3M CH_3COOH and CH_3COOH fermented palm sap is shown in Fig. 1. The FT-IR spectrum of coconut shell charcoal activated by 3M CH_3COOH and CH_3COOH fermented palm sap has almost the same absorption band pattern. The comparison shows that absorption occurs but undergoes a shift in wavenumber. Absorption with CH_3COOH activator occurred at wave numbers 3446.79 cm^{-1}, 2914.44 cm^{-1}, 2875.86 cm^{-1}, 1612.49 cm^{-1},

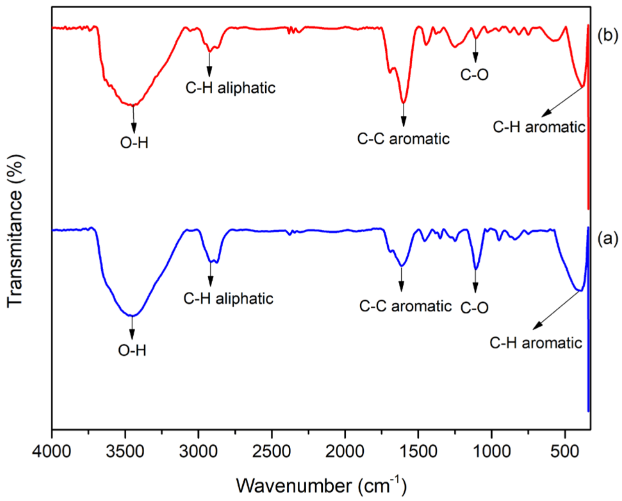

Fig. 1. FT-IR spectrum of coconut shell charcoal activated by (a) 3M CH_3COOH and (b) CH_3COOH fermented palm sap.

Table 1. The absorption band of the FT-IR spectrum of coconut shell charcoal activated by (a) CH_3COOH 3 M and (b) CH_3COOH fermented palm sap.

Wavenumber, cm^{-1}		Functional groups
CH_3COOH 3 M	CH_3COOH fermented palm sap	
3446.79	3446.87	O-H
2875.86	2922.16	C-H aliphatic
1612.49	1600.92	C=C aromatic
1107.14	1107.14	C-O
387.69	383.83	C-H aromatic

1107.14 cm^{-1}, and 387.69 cm^{-1}. Meanwhile, absorption with CH_3COOH activator from palm sap fermentation occurred at wave numbers 3446.87 cm^{-1}, 2922.16 cm^{-1}, 2872.01 cm^{-1}, 1600.92 cm^{-1}, 1107.14 cm^{-1}, and 383.83 cm^{-1}. Loss of absorption and a decrease in absorption intensity indicate reduced impurities in the charcoal. This indicates the formation of aromatic compounds, constituents of the hexagonal charcoal crystal structure.

The functional groups can be seen in Table 1. The absorption appears at a wavenumber of about 3400 cm^{-1} indicating the O-H group. Absorption at wavenumber 2900 to 2800 cm^{-1} indicates the presence of aliphatic C-H groups, and at wavenumber 1500 to 1400 cm^{-1} indicates the presence of aromatic C=C, which is a hexagonal shape. The presence of the C-O group indicates absorption at wavenumber from 1300 to 800 cm^{-1}. Furthermore, the absorption in wavenumber 700 to 300 cm^{-1} indicates the presence of an aromatic C-H group from confusion. This shows that the appearance of the C=C group is a feature of the carbon structure [17, 18].

The distribution of coconut shell charcoal pores is not uniform [16, 19]. The sample appears in the form of granules caused by grinding. As a result of the milling technique on the model, the sample cracked (Fig. 2). This result follows the theory that coconut shell charcoal is a porous material. The non-uniformity of the pore structure indicates the presence of filled tar in some pores [20]. In addition, the outer surface of coconut shell charcoal looks rough due to the breakdown of the structure due to the release of more volatile elements due to heating treatment at high temperatures [21, 22].

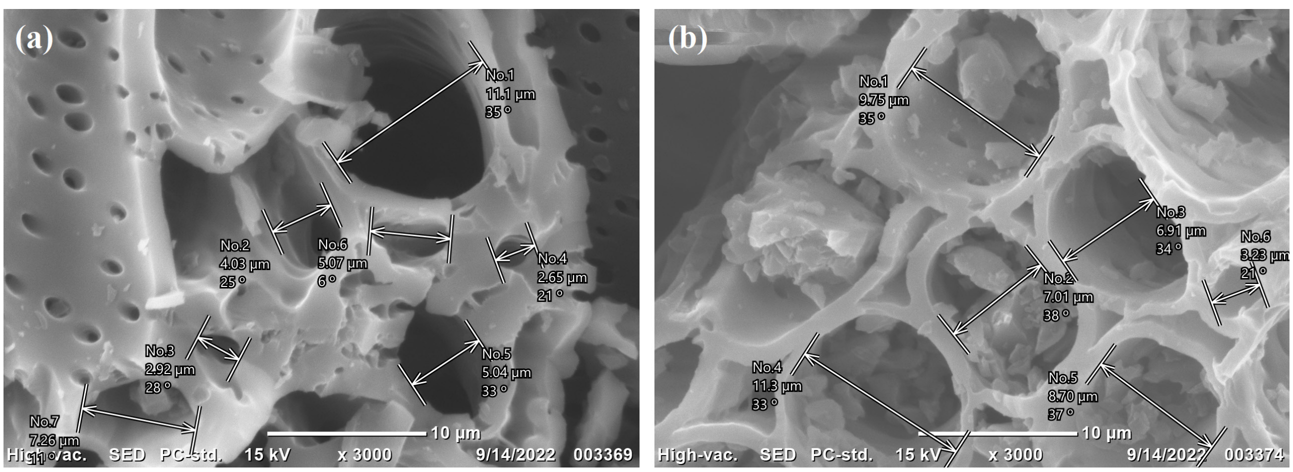

Fig. 2. SEM image of coconut shell charcoal with x3000 magnification activated by (a) 3M CH_3COOH and (b) CH_3COOH fermented palm sap.

Table 2. Pore length of coconut shell charcoal activated by 3M CH_3COOH and CH_3COOH fermented palm sap.

CH_3COOH	CH_3COOH fermented palm sap
11.1 µm	11.3 µm
7.26 µm	9.75 µm
5.07 µm	8.70 µm
5.04 µm	7.01 µm
4.03 µm	6.91 µm
2.92 µm	3.23 µm
2.65 µm	

Table 3. Results of EDS analysis of coconut shell charcoal activated by 3M CH_3COOH and CH_3COOH fermented palm sap.

CH_3COOH		CH_3COOH fermented palm sap	
Element	Mass, %	Element	Mass, %
C	85.61	C	87.88
O	10.15	O	10.53
Al	3.74	Al	1.09
Ca	0.50	Ca	0.50

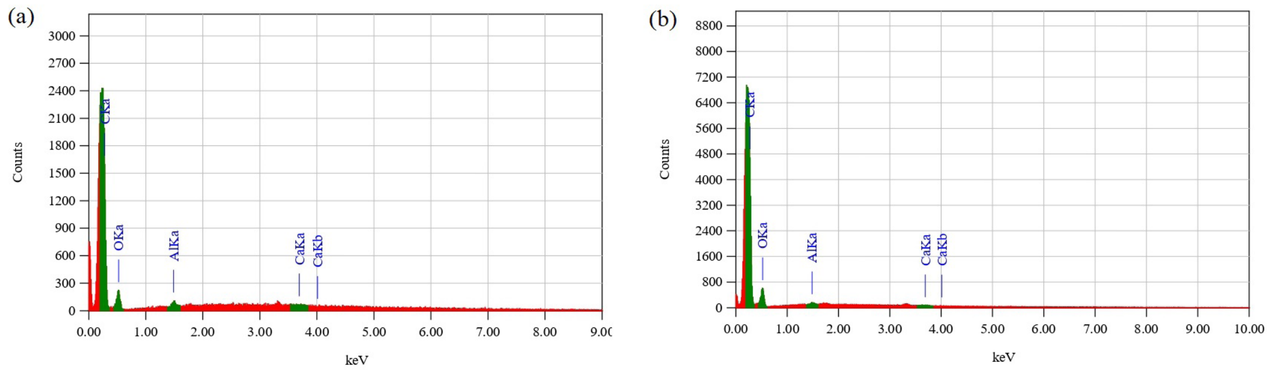

Fig. 3. EDS analysis result using the activator (a) CH_3COOH 3 M and (b) CH_3COOH fermented palm sap.

It appears that the pore diameter of coconut shell charcoal produced by pyrolysis and activation varies. The pore diameter of coconut shell charcoal activated by (a) 3M CH_3COOH and (b) CH_3COOH fermented palm sap can be seen in Table 2. The pore diameter of coconut shell charcoal activated by CH_3COOH ranged from 2.65 to 11.1 µm (mean pore size is 5.81 µm). While the pore diameter of coconut shell charcoal activated by CH_3COOH fermented palm sap is 3.23 to 11.3 µm (mean pore size is 7.82 µm). The pore diameter of coconut shell charcoal, which CH_3COOH from fermented palm sap activated, was more significant than that activated by 3M CH_3COOH. However, the charcoal pore diameter is still not uniform.

The results showed that these pores were macropores, with a pore diameter of more than 50 nm [23 - 25]. These results indicate that the pore diameter of coconut shell charcoal activated using CH_3COOH resulting from palm sap fermentation has potential as an adsorbent for industrial liquid waste. Adsorbents with large pore sizes have a higher absorption capacity compared to adsorbents with small pores. The non-uniformity of pore diameter was influenced using chemicals CH_3COOH and CH_3COOH fermented palm sap. In addition, it is influenced by an imperfect carbonization process caused by the presence of leftover raw materials. This raw material contains other chemically bonded elements, such as oxygen and hydrogen [26, 27].

The chemical composition of coconut shell charcoal because of EDS analysis using the activator 3M CH_3COOH and CH_3COOH from fermented palm sap is given in Fig. 3 and Table 3. The results of the EDS analysis of coconut shell charcoal activator 3M CH_3COOH showed that the elemental composition of carbon C 85.61 %, aluminium Al 7.58 %, and calcium Ca 0.75 %. The results of the EDS analysis of coconut shell charcoal activator CH_3COOH from palm sap fermentation showed the chemical composition of the carbon element C 87.88 %, aluminium Al 1.09 %, and calcium Ca 0.50 %. The elemental composition of carbon C using the CH_3COOH activator from fermented palm sap is more significant than using 3M CH_3COOH. The presence of element O, Al, and Ca indicate that the carbonization process is not perfect. This is following the SEM results. An increase in carbon content is associated with an increase in the number of pores [28 - 30].

CONCLUSIONS

The FT-IR analysis of coconut shell charcoal activated with CH_3COOH 3M and CH_3COOH fermented palm sap showed a decrease or removal of impurity functional groups and an increase in coconut shell charcoal functional groups. The results of SEM-EDS analysis of coconut shell charcoal with CH_3COOH activator fermented palm sap has a larger average pore diameter of 7.82 μm. Thus, coconut shell charcoal activated using CH_3COOH resulting from palm sap fermentation has potential as an adsorbent for industrial liquid waste. The elemental content of carbon C is 87.88 % by mass.

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