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Utilization of Tropical Forest Cacao Dried Leaves for Environment Improvement

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ABSTRACT

Cacao (Theobroma cacao L.) is a plant that grows in a tropical forest environment that requires shade to avoid whole light. This plant can be broadly divided into two parts: the vegetative part, which includes roots, stems, and leaves, and the generative function, which includes flowers, fruit, and seeds. The productive part of cacao is a product with high economic value, while the vegetative part, such as dry leaves, has not been widely used. This study aims to determine the characteristics of activated carbon from cacao leaves taken from plantation locations near tropical forests in Central Sulawesi and its use to improve water quality by reducing TSS and improving pH values. The parameters observed were yield, moisture, ash, and fixed carbon. The quality of activated carbon meets the technical quality requirements for activated carbon (SNI 06-3730-1995) for water content and ash content, namely 5.1% (maximum 15%) and 1.12% (maximum 10%), but has a bound carbon content of 37.97% (minimum 65%). Generally, the performance of tofu wastewater treated with activated charcoal changes with increasing pH value and decreasing TSS value.

1. INTRODUCTION

The natural environment for cacao plants is a tropical forest; thus, rainfall (1.100 mm – 3.000 mm per year), temperature 18℃-32℃, humidity, light intensity, and wind are limiting factors for the spread of cacao plants [1]. Climatic factors relevant to cacao growth are annual rainfall and its yearly distribution. If it is too low, insufficient water is available to the plant, leading to stress and death, depending on the degree of drought. On the other hand, too high annual rainfall can cause negative impacts in the form of erosion [2]. Cacao plants are planted between 10 North Latitude (NL) and 10 South Latitude (SL). However, in general, the spread of cacao plantations is located in the area of 7 North Latitude (NL) to 18 South Latitude (SL) and quite tolerant in the 20 South Latitude (SL) to 20 South Latitude (SL) Region. Cocoa growing areas in Indonesia are located from 5 North Latitudes (NL) to 10 South Latitudes (SL), and this area is considered ideal if accompanied by an altitude of not more than 800 m above sea level [3]. Cacao grows well in soils with a pH of 6 – 7.5 [4]. The cacao plant has a stem, branches, roots, flowers, and fruit. Cacao leaves are part of the rarely used stem but are a large part of the cacao plant besides the fruit. Old leaves will fall to the ground and pollute the plant environment. These dried leaves can be used as raw materials to manufacture activated carbon.

Activated carbon is a material or material that has very many wide pores [5-9]. These pores serve to absorb any contaminants that pass through them. If the water is filtered with activated carbon, the impurities can enter the pores and get trapped [10]. If numbers are made, 450 grams of activated carbon can contain approximately 40 hectares of the surface area [11]. Unlike charcoal burned at a temperature of 100℃, started carbon undergoes a combustion process with a 6-8 times hotter temperature in an airtight chamber. The combustion process that produces this carbon finally makes activated charcoal have more pores than ordinary charcoal. And the presence of these pores makes the charcoal more active and absorbs high. Activated carbon works by absorption or absorption. That is, when there is material through the activated carbon, the material contained in it will be absorbed. So, do not be surprised if this material can take some of the content that is not good from polluted water. It can even clear up cloudy water while removing odors from the water [12]. The use of activated carbon in water filters can be felt by people in big cities, especially those who already use water filters, both simply and technologically. Some benefits of activated carbon for water filters and purifiers are absorbing odors, purifying, taking chlorine, creating a fresh taste for water, and non-absorbable materials [13].

In this study, the sample to be tested is a tofu wastewater sample [14]. Industrial waste in tofu processing contains large amounts of protein, fat, carbohydrates, minerals, and chemical residues used during cleaning and processing [15]. The presence of high levels of organic matter in wastewater and materials carried in water in the processing of the tofu industry will cause disturbances to the environmental ecosystem. The waste can be sustainable if there is no good handling and countermeasures. The most apparent impact of this organic waste is the emergence of a pungent odor and cloudy water [16]. With activated carbon from dry cacao leaves, it is hoped that the pollution of tofu wastewater in the environment can be minimized by lowering the TSS level and increasing the pH value before it is disposed of in the background.

2. MATERIALS AND METHODS

The research design is an experiment in the laboratory using a Muffle Furnace STM-6-12 Thermocouple: N/K Type, Max Temperature: 1200℃ for the pyrolysis process.

2.1 Activated charcoal sample making

The cacao leaves used as starting material is the yellow leaf (not brown) and are still on the cocoa tree because it contains biomass such as lignin and cellulose. The samples were then dried in the sun to remove the water content. Dry pieces will be easily mashed. The wind-dried samples were weighed and mashed using a blender, put in an electric furnace, and heated at 250℃ for 1 hour. The black product was cooled for 24 hours, and the charcoal was weighed. The powder was filtered with a pass size of 80 mesh. Graphical steps of sample preparation are shown in Figure 1 below.

Figure 1. Preparation of activated carbon from Cacao leaves

2.2 Charcoal activation

The filtered powder was activated physically by soaking the purified charcoal powder with 3M $ZnCl_2$ for 24 hours, then washed with cold water and filtered/drained. SEM-EDS performed pore analysis. The water and ash content were then calculated.

2.3 Activated charcoal application to improve water quality

Activated charcoal from dry cacao leaves is mixed into a water sample, namely tofu wastewater, with a mixed composition of 1 gram of activated carbon in 100 ml. The solution is stirred until homogeneous, left for 1 hour, 4 hours, and 8 hours, and then filtered with filter paper. The filtered water (treated with activated charcoal) was compared to the quality of the water with water (not given activated charcoal). The water parameters measured were turbidity (TSS) and pH.

3. RESULTS AND DISCUSSION

Preparing activated carbon from cacao leaves goes through 3 stages: carbonization, activation, and calcination. Pyrolysis carbonization is the process of writing by incomplete combustion of materials containing carbon at high temperatures [17]. The cacao leaves used are old yellow cacao leaves that have been dried. Pyrolysis is carried out using a steel furnace. After the carbonization stage, the results obtained from the sample are black carbon in the form of coarse powder. The following is the decomposition reaction of chemical compounds in the carbonization process [18].

1. Cellulose pyrolysis reaction

$$(C_6H_{10}O_5)_n \rightarrow C_6H_8O + CH_3COOH + 3CO_2 + 2H_2O + CH_3OH + 5H_2 + 3CO$$

2. Lignin decomposition reaction

$$\text{Lignin} \rightarrow C_{18}H_{11}CH_3\,(\text{tar}) + C_6H_5OH + C_7H_8O_2 + CO + CO_2 + CH_4 + H_2$$

3. Reaction for the formation of carbon

$$(C_x H_y O_z)_n + O_2 \rightarrow C(s) + CO(g) + H_2O(g)$$

The carbon obtained from the pyrolysis carbonization is activated. The activation process using the $ZnCL_2$ solution was carried out for 24 hours. $ZnCl_2$ inhibits tar formation in the activation process and encourages aromatization to produce a porous activated carbon product. The amount of $ZnCl_2$ and the carbonization temperature affect the surface area of activated carbon obtained, while carbonization time tends to have no effect [19]. Soaking in an activation agent aims to degrade or hydrate organic molecules during the carbonization process, limit tar formation, and assist in decomposing organic compounds. The process also helps remove hydrocarbon deposits produced in the carbonization process and protect the carbon surface, reducing the possibility of oxidation [20]. After activation, the precipitate is filtered, and the residue is washed using hot water to purify the carbon and neutralize the pH. The precipitate was filtered, and the residue was dried in the oven for 1 hour and cooled.

The activated carbon produced was 33 grams of coarse powder and black. The color change after pyrolysis carbonization occurs because the cacao leaves, which contain hydrocarbon and mineral compounds, undergo incomplete combustion with little air. During pyrolysis, heat energy encourages oxidation, so complex carbon compounds are mostly decomposed into carbon or charcoal [21]. Carbonization occurs when the organic materials in the sample are broken down, namely cellulose, hemicellulose, and lignin, into carbon.

3.1 Water content

The value of water content in activated carbon is related to the hygroscopic nature of activated carbon in water. The moisture content of activated carbon from dry cacao leaves was 5.1% (Table 1). The water content obtained has met the quality requirements of technical activated charcoal SNI 06-3730-1995 and meets the conditions with a water content of less than 15%. The moisture content of charcoal is closely related to the specific gravity of the material used because it is hygroscopic. Materials with low specific gravity have a bond structure between carbon particles that are less compact and have cavities so that they are filled with water vapor from the air, which results in more excellent water content when compared to heavy materials such as wood [22]. However, the density level of the material has no significant effect on the material's moisture content, ash content, volatile matter content, carbon content, and calorific value. However, the material's density level significantly affects the compressive strength and fuel power of the material [23].

Table 1. Data analysis of Biochar from Cacao leaf

Sample	Pyrolysis Temperature	Yield (%)	Water content (%)	Ash content (%)
Cacao leaf	250	33	5.1	1.12

3.2 Ash content

The value of ash content in activated charcoal is related to the metal oxide content. The ash content of activated charcoal from dry cacao leaves was 1.12% (Table 1). The ash content has met the quality requirements of technical activated charcoal SNI 06-3730-1995. Excessive ash content causes the clogging of activated charcoal pores, reducing the surface area of activated charcoal [24]. Ash content cannot be burned or can no longer produce heat, so the less ash content, the better the calorific value of the fuel [25]. The ash content determines the amount of metal oxidation in activated carbon. Activated carbon made from natural ingredients contains carbon compounds and several minerals. The ash content will indicate the mineral content contained in activated carbon [26].

Activated carbon in this study has an ash content of 1.12%. The results of carbon ash content obtained in this study met SNI 06-3730-1995 because it was less than 10%. Theoretically, the higher the pyrolysis temperature, the higher the ash content. However, in this study, the ash content was not high, meaning there was no increase in the pyrolysis temperature, which triggered the oxidation of volatile substances, including carbon [17].

Besides the moisture and ash content, another property of the material is that the synthesized activated carbon is Powdered Activated Carbon (PAC). Activated carbon can be classified based on its form, namely: Powdered Activated Carbon (PAC), Granular Activated Carbon (GAC), and Extruded Activated Carbon (EAC). PAC is used in the liquid phase. GAC is used in the liquid and gas phases, while EAC is used in the gas phase. Because activated carbon from dried cacao leaves will be used to improve the quality of liquid waste, activated carbon is made in the form of PAC.

3.3 Bonded carbon content

The value of activated carbon-bound carbon content relates to the pure carbon content wrapped in activated charcoal after activation (Figure 2). The dried cacao leaves' activated carbon secured carbon content was 27.15% (Table 2). The bound carbon content that meets the technical quality requirements of SNI 06-3730-1995 is activated charcoal with a bonded carbon content of 65%. The low level of bound carbon illustrates that the purity of the activated charcoal is still relatively small, and the surface of the activated charcoal still contains non-carbon compounds. The condition is probably due to the pyrolysis temperature, which is too low, so the carbonization process does not take place ideally [20].

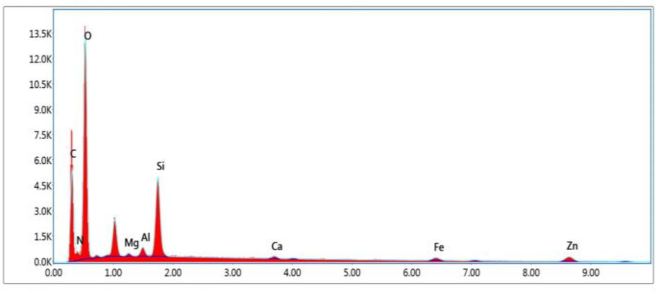
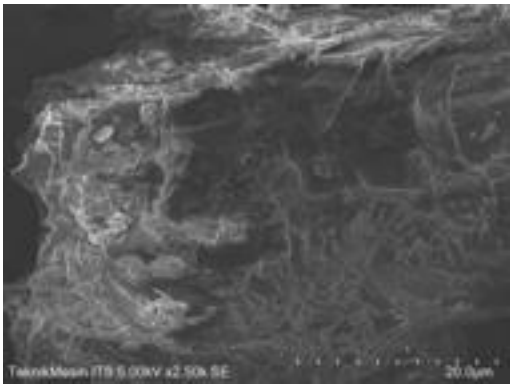


Figure 2. EDS analysis shows the distribution of carbon and other elements in activated charcoal of dry cacao leaves

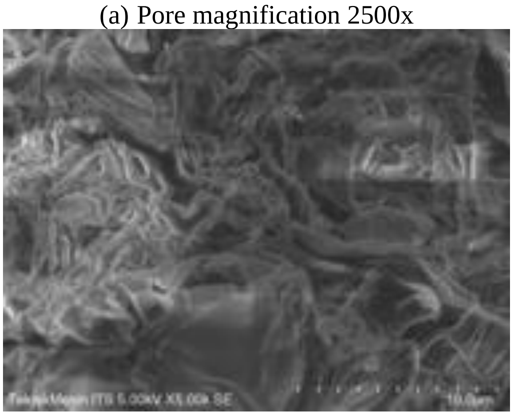
Table 2. Elemental content biochar

Element	Weight %	Atomic %
C	27.15	37.97
N	4.82	5.78
O	44.48	46.69
Mg	0.40	0.28
Al	1.03	0.64
Si	8.07	4.82
Ca	0.70	0.29
Fe	2.25	0.67
Zn	11.11	2.85

The variation influences the size of the carbon content bound to the activated carbon in the ash and volatile matter content [24]. If the pyrolysis temperature is not maximal, it will affect the amount of yield; thus, the amount of bound carbon produced is less [20].



(a) Pore magnification 2500x



(b) Pore magnification 5000x

Figure 3. Pore surface structure analysis was performed using a scanning electron microscope (SEM) with magnification 2500x (a), and 5000x (b)

This analysis aims to determine the surface topography of a material due to changes in carbonization temperature and activation. SEM is one type of microscope used to determine the microstructure of solids, such as a sample's morphology, composition, and surface crystallographic information [27]. Pore morphology analysis of dried cocoa leaf activated carbon using the pyrolysis method was carried out using Scanning Electron Microscopy (SEM) with magnifications of 2500 and 5000 times. SEM is to determine the difference in the shape of the pore surface of calcined pyrolysis-activated carbon at 250°C for 1 hour.

In Figure 2, the activated carbon test results show that the particle size from the SEM is 20 m and 10 m, indicating that activated carbon is classified as microporous. The smaller the pore diameter of the carbon and the larger the pore volume, the greater the surface area and absorption of the carbon [28]. In this study, testing at a temperature of 250°C for 1 hour showed that the particle size was obtained through spherical and needle-like plates. The surface appeared to form pores compared to activated carbon from banana peels and banana stems, young coconut peels, and corn stalks, which was calcined at a temperature of 400°C-600°C with the results of SEM-activated carbon shaped like irregular lumps [5, 29, 30]. The SEM analysis showed that the dried leaves of cocoa that had not been carbonized did not show any pores (Figure 3).

Activation of charcoal into activated charcoal tends to cause an increase in the number and diameter of the pores. In addition, $ZnCl_2$ at a concentration of 10% has opened larger and smaller pores. The pores' performance shows that $ZnCl_2$ can reduce hydrocarbon compounds still attached to the surface of the charcoal.

The formation of pores is due to the evaporation of volatile substances from the raw material due to the carbonization process [17, 20]. Carbonization has caused the components of the material to be degraded to produce gaseous products (CO, CO_2, hydrogen, and methane), liquid products (tar, hydrocarbons, wood vinegar, water), and solid products, namely charcoal [20].

3.4 Activated carbon application to improve environment quality

The tofu industry production process produces several types of waste, such as tofu dregs and wastewater [31]. In the tofu production process, large amounts of water are used in each production process, such as soaking, washing soybeans, clumping, and printing, where this wastewater is directly discharged into the environment without any prior treatment. This wastewater is used as a sample in this study [32]. TSS testing on wastewater in the field uses the Gravimetric method [33] according to the reference, namely the reference to SNI 06-6989.3-2004.

The water performance changed with the treatment of mixing activated carbon from dry cacao leaves into the three tested water samples. The quality of the tofu wastewater studied experienced a decrease in the TSS value even though it did not meet the standards (Table 3) because there was still eight mg/L TSS in the water even though the time used had reached 8 hours with 1 gram of activated carbon. However, using activated carbon from dry cacao leaves showed a 50% decrease in TSS levels within 8 hours. It will likely take a long time, and a higher amount of activated carbon from dry cacao leaves so that the TSS value in tofu wastewater can decrease or even reach zero. Because the amount of bound carbon present in the activated carbon sample from dry cacao leaves is also not too high (< 65%), the best alternative for further research is to use more activated carbon (>1 g) and a longer contact time (>8 hours).

Table 3. Measurement of TSS level and pH value in tofu waste with variations in contact time

Biochar weight (grams)	Time (hours)	pH	TSS (mg/L)
0	0	4.43	16
1	1	4.82	16
1	4	4.85	12
1	8	4.91	8

Figure 4 shows changes in TSS levels and pH values based on variations in the contact time of activated carbon with tofu wastewater. TSS levels and pH values decreased with increasing contact time, meaning the longer the contact time of activated carbon with tofu wastewater, the more the pH value will increase, but the TSS level will decrease.

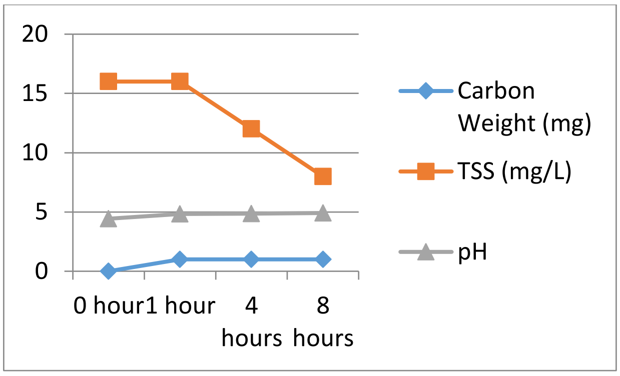


Figure 4. Decreasing pH value and TSS levels based on variations in contact time of activated carbon

Suppose the TSS value is expected to decrease with the addition of the amount of activated carbon and contact time. In that case, the pH value is likely to increase so that the trend of increasing the pH value toward normal water can be seen. This study shows that a variation of the contact time of 8 hours and the amount of activated carbon 1 g can increase the pH of tofu wastewater from 4.43 to 4.91, meaning an increase of 0.48 points (Table 4). This condition indicates hope that the pH of tofu wastewater can reach the standard pH value of water. i.e., 7 with the addition of the amount of activated carbon within 8 hours.

Table 4. Measurement of TSS and pH levels in tofu waste with variations in biochar weight

Biochar weight (grams)	Time (hours)	pH	TSS (mg/L)
0	0	4.43	16
0.5	8	4.79	12
1	8	4.91	8
1.5	8	5.15	2

Figure 5 shows changes in TSS levels and pH values based on variations in activated carbon use. The more carbon, the pH value increases while the TSS value decreases.

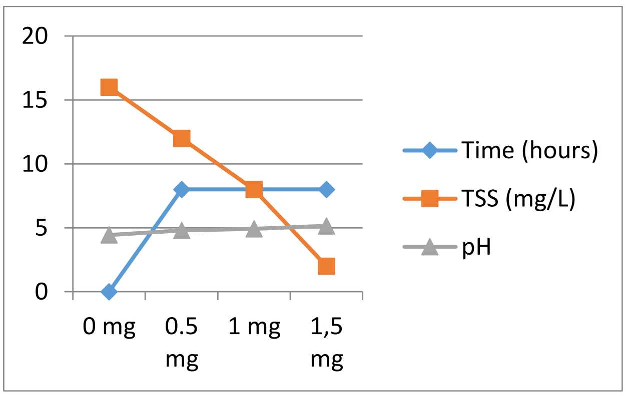


Figure 5. Decreasing pH value and TSS levels based on variations weight of activated carbon

One of the characteristics of tofu liquid waste is tofu liquid waste which is generally in high-temperature conditions [34]. A high temperature is needed because it is always hot in making tofu, either at the time of clumping or at the time of filtering, namely at a temperature of 60–80℃ [35]. Washing using cold water during the process cannot reduce the temperature of the liquid waste. Tofu liquid waste is light yellow and is accompanied by a white suspension [15]. The temperature of liquid tofu waste is generally higher than the of raw water, which is 400℃-460℃. Increased temperature in the aquatic environment will affect biological life, the solubility of oxygen and other gases, water density, viscosity, and surface tension [36]. Adding activated carbon into tofu waste before the waste is discharged into the environment can reduce the negative impact on the environment by increasing the pH value and decreasing the amount of TSS [37].

High levels of TSS in water can cause several environmental problems, such as increasing water turbidity and reducing penetration and transmission of sunlight, inhibiting aquatic plants' photosynthesis and resulting in disrupted oxygen production [38]. If the dissolved oxygen content in water is low, it causes organisms in the water to die due to lack of oxygen. High TSS in water can cause several environmental problems, such as increasing water turbidity, and reducing penetration and transmission of sunlight, thereby inhibiting the photosynthesis process of aquatic plants and resulting in disrupted oxygen production [39]. If the dissolved oxygen content in water is low, it causes organisms in the water to die due to a lack of oxygen [40].

The results of the measurement of the pH value show that the pH value tends to increase and has the potential to meet the established quality standards. The pH concentration is between 4-5 due to the addition of whey (liquid containing acid), causing the quality of the liquid waste to have acidic properties. The pH value at the final disposal (outlet) must meet the quality standards because it is a liquid waste. Water conditions that are very acidic or alkaline will endanger the survival of organisms because they will cause metabolic and respiratory disorders [41]. A pH value above neutral will increase the concentration of ammonia which is very toxic to organisms [42].

4. CONCLUSION

Most cacao waste, namely leaves, can be processed into activated carbon which can be used as an adsorbent to improve the condition of tofu wastewater so as not to harm the environment. Cacao leaves are taken randomly from tropical forests that residents use to grow cacao. The temperature of carbonation or pyrolysis still needs to be increased to produce a larger yield. The activation process with $ZnCl_2$ to open the pores of the activated carbon surface gives the appearance of spherical pores and needle-like plates, and the size shows macrospores. It is just that the number of pores is not too much due to the low carbonation temperature, which is only 250℃. For further research, if necessary, it is needed to increase the carbonation rate to make activated carbon from dry cacao leaves with several variations of pyrolysis temperature so that the results can be compared, starting from the yield to the pore morphology. Although the application for the improvement of tofu wastewater shows the ability of activated carbon from cacao leaves to increase the pH value and reduce the TSS value, research on the parameters of tofu wastewater must be done. It can also be classified according to the type of dry leaf of cacao based on where it grows to determine the effect of the growing location on its adsorption ability.

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