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REUSE OF AGRICULTURAL WASTE TO ADSORB IRON CONTENT IN ACID MINE WATER

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Abstract

The adsorption process is the most desirable process in the treatment of waste containing heavy metals of acid mine drainage because it is economical, efficient, effective, and inexpensive. The adsorbent that is generally used is an activated carbon made from materials containing carbon, such as agricultural waste, namely lemongrass waste (*Cymbopogon S.P*), langsung fruit peel (*Lansium domesticum cortex*) and coconut shell (*Cocos nucifera L.*). The activated carbon using H_3PO_4 displayed more pore formation, larger, and cleaner than activated carbon with NH_4OH . The best activated carbon in absorbing Fe^{3+} metal in acid mine water was at a mass of 4 grams with a percent removal. The equation used in the adsorption process was the Langmuir and Freundlich isotherm adsorption equation. The Langmuir isotherm equation obtained from maximum adsorption capacity of activated carbon langsung fruit peel was 38.89 mg/g with $K_L=76.69$ mol/L and $R^2=0.9750$ followed by 16.51 mg/g; $K_L=43.31$ mol/L and $R^2=0.9069$ for activated carbon lemongrass waste and 53.11 mg/g with $K_L=73.20$ mol/L and $R^2=0.9494$ for activated carbon from coconut shell. Meanwhile, the Freundlich equation, K_F value was 83.23 mol/L; $R^2=0.9512$ on langsung peel activated carbon; $K_F=16.49$ mol/L; $R^2=0.9042$ on activated carbon of lemongrass waste, and $K_F=20.09$ mol/L; $R^2=0.9162$ on coconut shell activated carbon. Based on the data, the adsorption isotherm curve was more closely follows the Langmuir isotherm model with a linear regression coefficient that was relatively closer to 1 for the three types of adsorbents. This also indicates that the adsorption only takes place in one layer (monolayer).

INTRODUCTION

Mining business activities, besides being able to improve the economy and meeting community needs, can also have an impact on environmental damage such as ecological disturbances, damage to natural flora and fauna, air, water, soil pollution, instability of soil and rock masses, land-scape degradation, and global warming [1]. Mining activities will produce changes and impacts on the environment. One of the impacts of mining activities that is not managed properly is acid mine drainage. Acid mine water is a serious problem for the environment because acidic water has a characteristic of low pH value and contains high levels of heavy metals and

sulfates [2]. The accumulated heavy metals found are as iron, manganese, copper, cadmium, lead, copper and nickel [3]. If the metal is present in excess, it can cause adverse effects on human health, depending on which part of the heavy metal is bound in the body and the amount of exposure dose. The toxic effects of heavy metals are able to block the work of enzymes so that they interfere with the body's metabolism, cause allergies, and become mutagens, teratogens, or carcinogens for humans or animals [4]. Iron (Fe) doses exceed 20 mg/kg body weight in humans cause toxicity with an LD50 Fe of 60 mg/kg [4]. Consumption of Fe supplements exceeding 45 mg/day can cause gastric irritation while children can die if exposed orally to 200 mg

to 5.85 g of Fe. One of the shortcomings of the human body is the absence of a control mechanism for the disposal of Fe in the body. The Ministry of Health Republic Indonesia No. 907/MENKES/SK/VII/2002 also said about the requirements and supervision of drinking water quality, which is maximum iron in drinking water is 0.3 mg/L. That is why iron is one of the heavy metals.

Several techniques have been developed in the treatment of waste containing heavy metals but the adsorption process is the most desirable because it is economical, efficient, effective and inexpensive [5]. The adsorption is a process in which one or more constituents of a fluid solution will be more concentrated on the surface of a particular solid (adsorbent). In this way, the components of a solution, whether from a gas or liquid solution can be separated from each other [6]. The adsorbent which is generally used for the treatment of metal ion waste is activated carbon. The activated carbon can adsorb organic compounds well [7], and the production costs are relatively cheap because the raw material for making activated carbon can come from biomass waste [8]. The activated carbon can be made from materials that contain carbon, either from plants, animals, or minerals.

In this study, the use of activated carbon was focused on the mining industry, especially on waste from the mining industry, namely the extraction of Fe metals in acid mine drainage through an adsorption process with adsorbent raw materials from agricultural waste such as activated carbon produced from lemongrass plant waste (*Cymbopogon SP*), langsat fruit peel (*Lansium domesticum cortex*) and coconut shell (*Cocos nucifera L.*).

The average production of Indonesian coconuts is 15.5 billion grains/year, equivalent to 3.02 million tons of copra, 3.75 million tons of water, 0.75 million tons of shell charcoal, 1.8 million tons of coir fiber, and 3.3 million tons of coir dust [9]. Meanwhile, the production of lemongrass is around 2900 tons/year, if we convert that amount, about 50% is waste, then the amount of lemongrass waste is around 1450 tons/year [10]. Indonesia also produces 71,887 tons/year of langsat fruit. If we convert that amount, about 50% is waste, then the amount of waste such as langsat peel, then we get the amount of langsat peel waste around 35,945.5 tons/year [10]. Therefore, in this study we aimed to reuse of plant waste for reducing the waste in the environment and for treating acid mine drainage derived from mining industry. Aside from that we aimed to determine the type of activators and the optimum mass for the efficiency of reducing the concentration of Fe metal as well as the adsorption of isothermal model.

MATERIALS AND METHODS

Materials

Agricultural wastes used as raw materials for making activated carbon include langsat peel, lemongrass, and coconut shell. Furthermore, the chemicals used as activators

are H_3PO_4 and NH_4OH solutions with 20% concentration, while the equipment includes Crusher, oven dryer (Memmeth DIN 12880-KI), furnace (SX-2.8-12 Boc Huanghua Faithful Instrument Co.Ltd), analytical balance (Shimadzu AW-220), Screening (filter size 850 micron), and other necessary tools according to the implementation stage. The stages of the tools used are as follows:

1. Equipment for making carbon from agricultural waste: A stove with the brand SX-2.8-12 Boc Huanghua Faithful Instrument Co.Ltd.
2. Equipment for carbon Characterization: Brand SEM-EDX JEOL JSM-6360LA, analytical balances, ovens, porcelain dishes, desiccators, furnaces, and glassware commonly available in laboratories.

This study was conducted in two stages. The first is an activity that includes the process of writing, making activated carbon, and analyzed the quality of the resulting product. Meanwhile, the second stage is the application of the activated carbon from agricultural waste to adsorb Fe in acid mine water.

Carbon Manufacturing Process

Agricultural waste, such as langsat peel, lemongrass, and coconut shells were cleaned to remove existing impurities. Subsequently, the lemongrass waste and langsat fruit skins were cut into pieces while the coconut shell which was marched with a crusher of approximately size 1-2 cm was dried in an oven at 105°C for 24 hours. The waste was transferred into a furnace and burnt at a temperature of 300°C for 2 hours. The results of the composing were characterized by analysis of water content, ash content, volatile matter, fixed carbon, and iodine number. The results of the characterization of carbon obtained were compared with SNI 01-1682-1996 regarding the quality requirements of coconut shell carbon.

Chemical Activation

Activation with H_3PO_4 Solution

The 70 grams of each carbon was weighed and immersed in a 20% H_3PO_4 solution for 24 hours. Subsequently, it was dried in an oven for 60 minutes at 105°C, and the experiment was repeated twice for each.

Activation with NH_4OH Solution

The 70 grams of each carbon was weighed and immersed in a 20% NH_4OH solution for 24 hours. Subsequently, it was dried in an oven for 60 minutes at 105°C, and the experiment was repeated twice for each.

Sample Preparation and Carbon Proximate Analysis

Agricultural waste such as langsat fruit peel, lemongrass waste, and coconut shell was ground using a mortar while the powder was sieved with an 850-micron sieve. Furthermore, it was characterized based on water content, volatile matter (volatile matter), ash content, bound carbon (fixed carbon), and iodine number.

Application of Activated Carbon for Acid Mine Water Treatment and Determination of Adsorption Isotherms

The activated carbon that fulfil of the standards from the results of improving the quality was then applied as an adsorbent in the treatment of acid mine waste water (artificial Fe metal) by mixing the each of activated carbon from agricultural waste in the liquid waste.

The treatment of acid mine waste water was carried out by adding 2, 3, 4, 5, 6, and 7 g of activated carbon from agricultural waste into the wastewater sample (the mother liquor sample contains FeSO_4 as much as 127.20 ppm) with a volume of 100 mL in each Erlenmeyer flask. Then, the mixture was stirred using a rotary shaker at 50 rpm for 60 minutes and further filtered with whatman 42 paper. The liquid resulting from the separation was analyzed for its metal ion content by atomic adsorption spectrophotometry. The initial metal ion solution was also analyzed in the same way. Based on the calculation results obtained, the amount of metal ions adsorbed was expressed in mg/g. These data can be used to determine the adsorption isotherm model. Spectrophotometric test using SNI 6989.4:2009 was used for the total determination of Iron.

Data analysis was carried out by determining the final concentration of FeCl_3 after went through the adsorption process from the adsorbate concentration to the adsorption capacity. The results of this analysis were then entered into the Langmuir and Freundlich isotherm equations to determine the adsorption isotherm model on the adsorbent.

The amount of Fe ions adsorbed by agricultural waste adsorbent (Q_e) was calculated for each run by the following equation:

$$Q_e = \frac{(C_0 - C_e)V}{w} \quad (1)$$

where Q_e (mg/g) is the adsorption capacity; C_0 (mg/L) is the initial concentration of Fe; C_e (mg/L) is the equilibrium concentration of Fe; V (L) is the volume acid mine water, and W (g) is the weight of adsorbent added [11-12]. The Langmuir isotherm model equation is:

$$q = q_{\max} \left(K_L \frac{C_e}{1 + K_L C_e} \right) \quad (2)$$

where q (mg/g) is the amount of heavy metal ions adsorbed onto the unit mass of the polymeric beads; K_L is the

Langmuir equilibrium constant, which is related to the affinity of binding sites; C_{eq} is the equilibrium Ni^{2+} ion concentration, and q_{\max} is the maximum adsorption capacity (theoretical monolayer saturation capacity) [13]. The main characteristics of the Langmuir equation, constants K_L and q_{\max} , are determined from a linearized form of the Langmuir equation as follows:

$$\frac{C_e}{q_e} = \frac{1}{q_{\max} K_L} + \frac{C_e}{q_{\max}} \quad (3)$$

Therefore, a plot of C_e/q versus C_e gave a straight line with slope of $1/q_{\max}$ and intercept of $1/(K_L q_{\max})$.

The Freundlich isotherm model was based on adsorption on a heterogeneous surface, developed an empirical equation [14]:

$$q_e = K C_e^{1/n} \quad (4)$$

where q_e is the adsorption (mg/g); C_e is the concentration of adsorbate in the solution (mg/L); K_F and n are empirical constants that are characteristics of the system, indicating the adsorption capacity and the adsorption intensity, respectively. The above equation can be linearized as the following form and also can be used to confirm the applicability of the model [14]:

$$\ln q_e = \ln K + \frac{1}{n} \ln C_e \quad (5)$$

where K_F is the measure of sorption capacity, and $1/n$ is the sorption intensity.

RESULTS AND DISCUSSION

In the process of making agricultural waste carbon (langsat fruit peel, lemongrass and coconut shell waste), the cooking temperature used was 300°C. The basis of choosing this temperature variation is that too high temperature contributes risk of further oxidation of the carbon, so that it can damage the C-C carbon bond [15]. In addition, the selection of temperature is also based on the research of [16], which states that the composing process consists of four sequential steps, namely evaporation of water at a temperature of 100-120°C, decomposition of cellulose at a temperature of 270-310°C, decomposition of lignin at a temperature of 310-510°C, and finally carbon purification at a temperature of 500-1000°C. For this reason, the 300°C cooking temperature range was chosen. As for the quality of carbon/carbon from agricultural waste (langsat fruit peel, lemongrass and coconut shell waste), it is compared with SNI 01-1682-1996 regarding the quality requirements of coconut shell carbon. The quality of carbon/carbon from agricultural waste (langsat fruit peel, lemongrass and coconut shell waste) is compared to coconut shell carbon because there is no carbon/carbon standard for agricultural

waste (langsar fruit peel, lemongrass waste and coconut shell).

Table 1. Carbon Proximate Test Results (Composition At 300°C Temperature) from Agricultural Waste (Langsat Fruit Peel, Lemongrass Waste and Coconut Shell)

Parameter	Unit	SNI 01-1682-1996	Agricultural Waste		
			Lemongrass Waste	Langsat Fruit Peel	Coconut Shell
Water content	%	Max 6	5.28	5.88	4.23
Ash Level	%	Max 5	4.39	4.13	4.11
Volatile Matter	%	Max 20	9.86	5.37	6.93
Fixed Carbon	%	Min 70	91.47	96.62	90.73
Iodine Number	g Iod/100 g	-	29.00	26.91	27.15

Based on Table 2 and Table 3, it can be seen that those that meet the requirements according to SNI 06-3730-1995 (technical activated carbon quality requirements) are carbon activated with H_3PO_4 solution. According to [17], the high moisture content of activated carbon is influenced by the hygroscopic nature of activated carbon, the amount of water vapor in the air, the length of the cooling, milling and sieving processes. The high ash content of activated carbon is due to the lack of cleanliness in the washing process after the carbon is activated with NH_4OH . The high content of volatile matter in activated carbon with NH_4OH is due to the low nitrogen and sulphur burned [18]. The banded carbon content that does not meet the SNI standard (min. 65%) is activated carbon with NH_4OH . The low content of bound carbon is due to the high ash content of the activated carbon.

According to [19], good banded carbon content as raw material for activated carbon ranges from 70-80%. The adsorption of activated carbon to iodine solution indicates the ability of activated carbon to adsorb components with low molecular weight. This happens because the ash content of activated carbon activated with H_3PO_4 is lower than that of activated carbon activated with NH_4OH so that it affects the amount of iodine adsorption. The high ash content resulted in the number of activated carbon pores being covered with ash, thus affecting the adsorption of iodine. Based on the results of the proximate analysis, it is known that the activated carbon that has the best quality is the carbonized carbon at a temperature of 300°C which is activated with H_3PO_4 .

Table 2. Proximate Test Results of Carbon from Agricultural Waste (Langsat Fruit Peel, Lemongrass Waste and Coconut Shell) Soaked with H_3PO_4

Parameter	Unit	SNI 06-3730-1995	Agricultural Waste		
			Lemongrass Waste	Langsat Fruit Peel	Coconut Shell
Water content	%	Max 15	12.48	14.38	14.13
Ash Level	%	Max 10	4.41	4.33	4.09
Volatile Matter	%	Max 25	10.36	4.31	4.93
Fixed Carbon	%	Min 65	65.47	78.62	67.03
Iodine Number	g Iod/100 g	Min 750	876.46	813.70	913.70

Table 3. Proximate Test Results of Carbon from Agricultural Waste Soaked with NH_4OH

Parameter	Unit	SNI 06-3730-1995	Agricultural Waste		
			Lemongrass Waste	Langsat Fruit Peel	Coconut Shell
Water content	%	Max 15	16.33	15.47	14.50
Ash Level	%	Max 10	12.68	14.62	11.55
Volatile Matter	%	Max 25	43.97	52.09	38.08
Fixed Carbon	%	Min 65	37.02	29.82	50.87
Iodine Number	g Iod/100 g	Min 750	509.00	600.91	675.15

The topography of carbon was identified to determine the surface structure of the carbon, especially the cavity or porosity of the carbon with magnifications of 3000x and 5000x. Based on the results of the SEM-EDX analysis, the three types of carbon have different topography and porosity (Table 4 and Table 5). The skin of langsat fruit and coconut shell that have not been treated with any treatment looks more porous when compared to lemongrass waste (Table 4). The carbon cavity of agricultural waste (langsat fruit peel, lemongrass waste, coconut shell) which was charred at 300°C looked more open and elongated (Table 5). This happens because at high heating temperatures, a complete decomposition reaction of cellulose and lignin occurs as

stated by [16]. The observation of the topography of carbon using SEM-EDX shown in Table 5 shows that in activated carbon using both acid and base, there are more pores, bigger and cleaner pores than carbon that has not been activated. However, the pore formation was more large and clean in carbon activated with H_3PO_4 than in carbon activated with NH_4OH . The formation of many pores is also caused by the evaporation of volatile substances from the raw material due to the pyrolysis process. This causes the raw material components to be degraded into gas products (CO , CO_2 , H_2 , CH_4), liquid products (liquid smoke, tar, hydrocarbons and water) and solid products (carbon) [20].

Table 4. SEM-EDX Test Results Carbon (Production At 300°C)

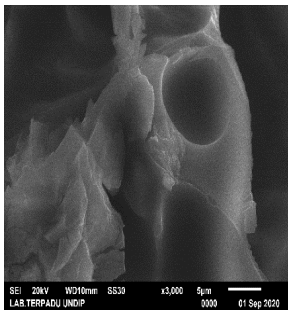
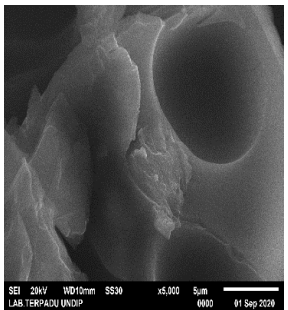
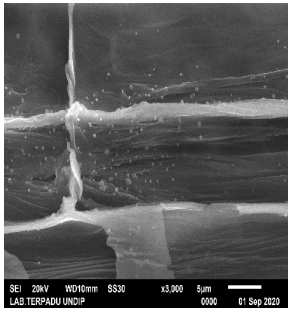
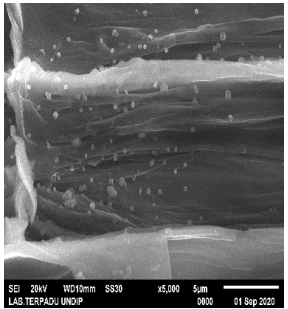
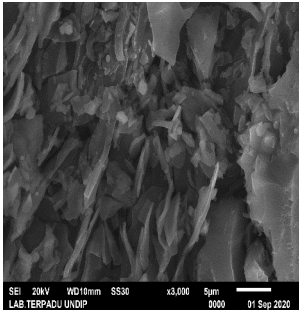
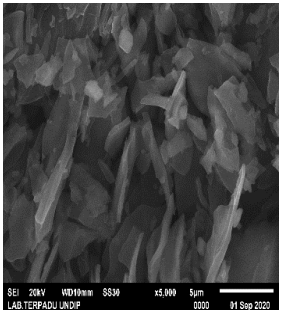
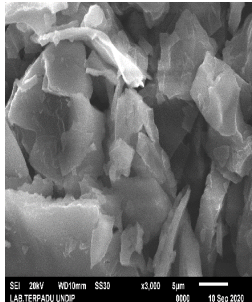
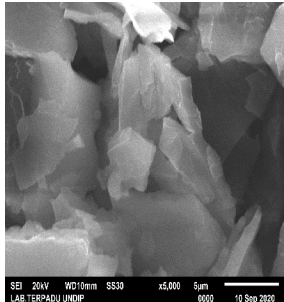
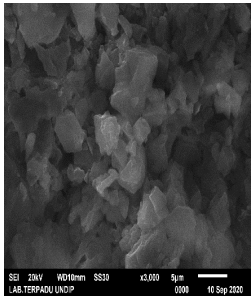
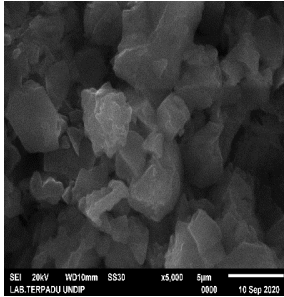
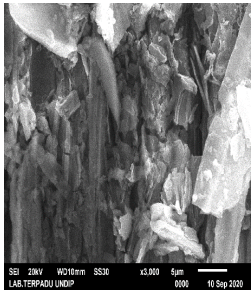
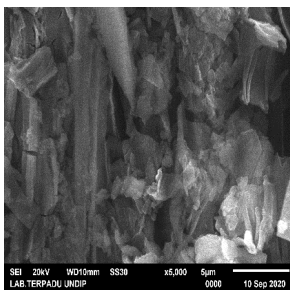
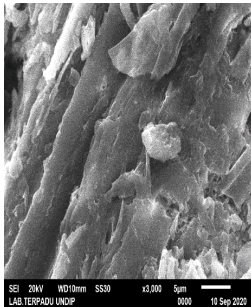
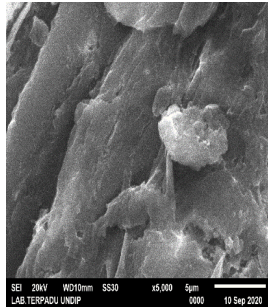
Agricultural Waste	Scale		Result
	3000X	5000X	
Langsat Fruit Peel			Fitting coeff.: 0.0186 Element Mass% C 75.56 O 22.88 Mg 0.09 K 0.84 Ca 0.16 Zr 0.47 Total 100.00
Lemongrass Waste			Fitting coeff.: 0.0205 Element Mass% C 67.45 O 28.46 Mg 0.19 Si 0.11 P 0.37 S 0.09 Cl 0.30 K 2.83 Cu 0.19 Total 100.00
Coconut Shell			Fitting coeff.: 0.0202 Element Mass% C 72.45 O 22.70 Na 0.94 Si 0.93 P 0.09 S 0.08 Cl 0.67 K 1.83 Zr 0.31 Total 100.00

Table 5. SEM-EDX Test Results Carbon from Agricultural Waste (Langsat Fruit Peel, Lemongrass Waste and Coconut Shell) soaked with H_3PO_4 and NH_4OH

Agricultural Waste	Solution	Scale		Result
		3000X	5000X	
Langsat Fruit Peel	H_3PO_4			Fitting coeff.: 0.0218 Element Mass% C 78.62 O 18.74 Mg 0.26 P 0.21 K 1.29 Ca 0.31 Cu 0.33 Zn 0.23 Total 100.00
	NH_4OH			Fitting coeff.: 0.0298 Element Mass% C 73.73 O 23.93 Mg 0.12 P 1.42 K 0.36 Ca 0.12 Cu 0.30 Total 100.00
Lemongrass Waste	H_3PO_4			Fitting coeff.: 0.0283 Element Mass% C 65.66 O 32.45 Mg 0.14 Si 0.69 P 0.76 K 0.17 Ca 0.13 Total 100.00
	NH_4OH			FITTING COEFF.: 0.0269 ELEMENT MASS% C 65.58 O 32.57 Mg 0.18 Si 0.66 K 0.52 Ca 0.27 Zn 0.22 Total 100.00

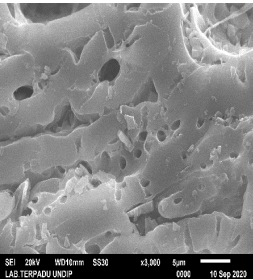
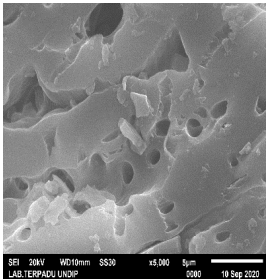
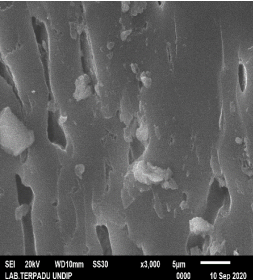
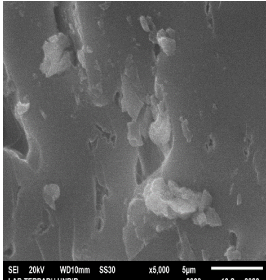
Coconut Shell	H ₃ PO ₄			Fitting coeff.: 0.0302 Element Mass% C 66.90 O 32.56 P 0.54 Total 100.00
	NH ₄ OH			Fitting coeff.: 0.0250 Element Mass% C 72.87 O 25.50 Na 0.12 K 0.48 Cu 0.40 Zn 0.25 Zr 0.37 Total 100.00

Table 6. AAS Test Results of Activated Carbon Agricultural Waste (Langsat Fruit Peel, Lemongrass Waste and Coconut Shell) in Acid Mine Water (Fe Metal Parameter)

Parameter	Agricultural Waste	Activated Carbon Mass (gram)	Results (mg/L)	Removal (%)	Method Specification
Fe	Langsat Fruit Peel	Preliminary Solution	127.20	0	Sni 6989.4:2009
		2	49.45	61.12	
		3	43.51	65.79	
		4	38.03	70.10	
		5	37.99	70.13	
		6	37.15	70.80	
		7	37.03	70.89	
	Lemongrass Waste	2	94.19	25.95	
		3	68.24	46.35	
		4	55.98	55.99	
		5	55.52	56.35	
		6	55.03	56.74	
		7	55.00	56.76	
	Coconut Shell	2	21.06	83.45	
		3	15.06	88.16	
		4	12.47	90.20	
		5	12.25	90.37	
		6	12.09	90.50	
		7	11.99	90.58	

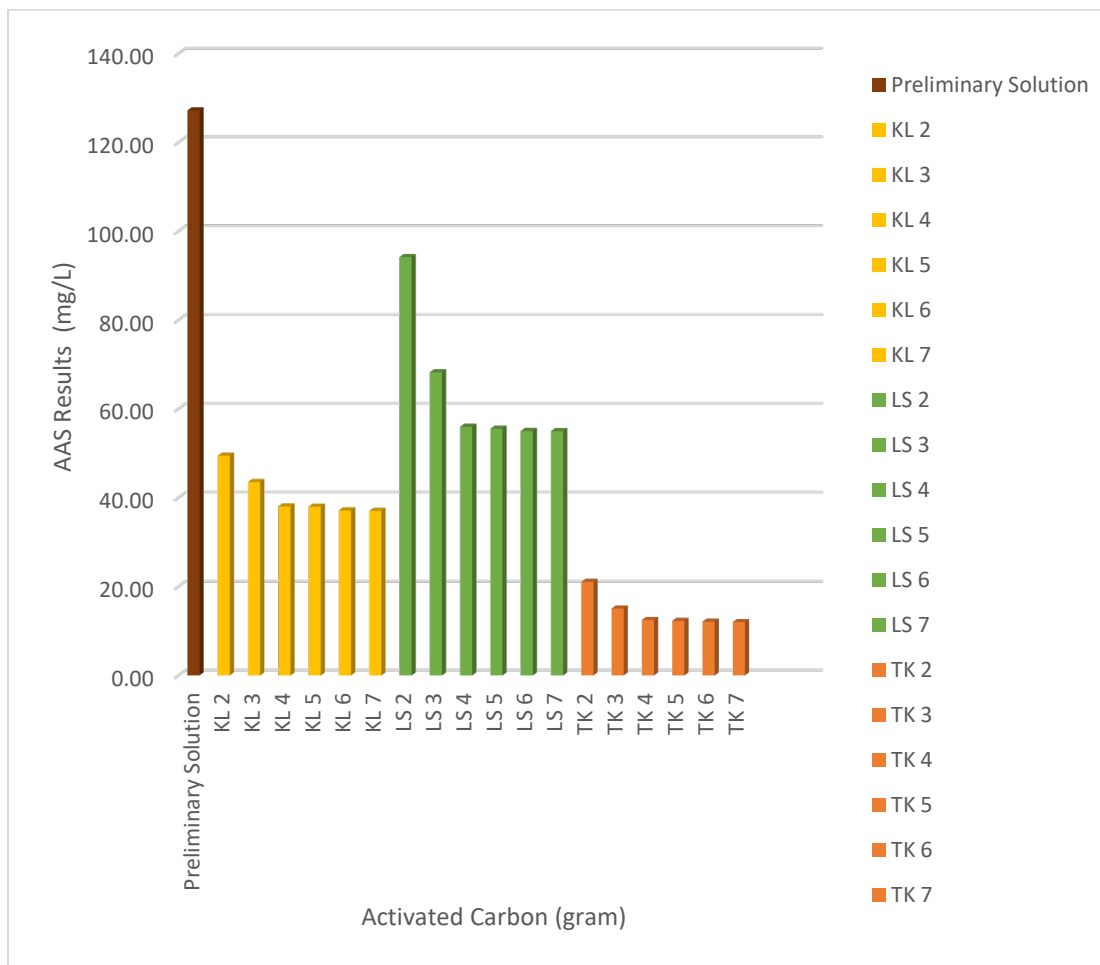


Figure 1. Graph of AAS Test Results for Activated Carbon Agricultural Wastes (Langsat Fruit Peel, Lemongrass Waste and Coconut Shell) in Acid Mine Water (Fe Metal Adsorption)

Based on the AAS test results for Fe metal, a graph can be made as shown in Figure 1. The calculation results of the Langmuir and Freundlich isothermal adsorption equations for Mn are shown in Table 7.

According to the Langmuir adsorption model, the value of q_{max} can be seen that the activated carbon of langsat peel waste, lemongrass waste and coconut shell on the Fe metal, each reaches a maximum adsorption capacity of 38.91 mg/g; 16.50 mg/g and 53.13 mg/g, respectively. The value of adsorption capacity can be caused by the outer layer of activated carbon that has been saturated with the adsorbed so that the adsorbent can no longer adsorb other metal molecules. The higher the concentration of the waste solution containing metals, the more molecules that collide and interact with the adsorbent, so that the adsorption ability increases [21].

If $0 < KL < 1$ it can be stated that the adsorption is favorable, $KL > 1$ is not favorable, $KL = 1$ linear adsorption and $KL = 0$ means irreversible and also explains the poor

adsorption but cannot be changed [22-24]. While in the Freundlich isotherm model, if the value is $0 < 1/n < 1$ then it can be stated that the adsorption is favorable and indicates cooperative adsorption. If the value of n (Freundlich isotherm) between 2 and 10 is a good adsorption [25]. Meanwhile, based on the calculation of the value of $1/n$, the results of the value of $1/n$ are all below or less than 0. This indicates that the adsorption of activated carbon from agricultural waste (langsat peel waste, lemongrass and coconut shell waste) with chemical processes follows the isotherm model. The Langmuir adsorption, in addition to the value of the relation coefficient (R^2) which is closer to 1 [26], so it can be assumed that the adsorption of acid mine drainage by the removal of Fe metal that occurs on the surface of the adsorbent from agricultural waste (langsat peel waste, lemongrass waste) and coconut shell) are homogeneous and the adsorbate is adsorbed in a single form (monolayer).

Table 7. Langmuir and Freundlich equations for Fe metal in the adsorption process

Metal	Agricultural Waste	Langmuir Isotherm			Freundlich Isotherm		
		q_{\max} (mg/g)	K_L (mol/L)	R^2	K_F (mol/L)	1/n	R^2
Fe	Langsat Fruit Peel	38.89	76.69	0.9750	83.23	0.000001	0.9512
	Lemongrass Waste	16.51	43.31	0.9069	16.49	0.000001	0.9042
	Coconut Shell	53.11	73.20	0.9494	20.09	0.000001	0.9162

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CONFLICT OF INTEREST

Authors declare that there is no conflict of interest regarding the publication of this manuscript.

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