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Utilization of Langsat Fruit Peel Waste to Adsorb Metal Contents from Acid Mine Drainage

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Abstract: The feasibility of using agricultural wastes, specifically the langsat fruit peel (*Lansium domesticum cortex*) as a low-cost sorbent (activated carbon) to remove Cu and Mn from acid mine drainage was investigated to determine the adsorption of the isothermal model in this study. Activated carbon could be made from the langsat fruit peel with 20% H_3PO_4 and 20% NH_4OH chemical activation. Before being activated, the langsat fruit peel was charred at 300°C for 2 hours to produce carbon. The SEM-EDX results showed that a high carbon value of 78.62% was obtained from the activation process carried out with H_3PO_4. The pore formation was also larger and cleaner in carbon activated with H_3PO_4 than NH_4OH. A BET surface area of 1.4456 m^2/g was obtained for the activated carbon produced from the langsat peel waste. In addition, a BJH adsorption cumulative volume of 0.000701cm^3/g was recorded for pores. The BJH adsorption average pore diameter (4V/A) for the activated carbon was 31.31399 nm, indicating a mesoporous scale size. The best activated carbon in terms of metals adsorption in acid mine drainage was in a mass of 4 grams, with each percent removal of 91.42% for Mn metal and 15.74% for Cu metal. The Langmuir and Freundlich isotherm adsorption equations were used in the adsorption process. Based on the data obtained, the isotherm curve corresponded more to the Langmuir model, indicating absorption occurred in a monolayer, with a linear regression coefficient, which was relatively closer to 1.

Keywords: acid mine drainage, activated carbon, adsorption, langsat fruit peel, metal contents.

利用 朗萨果皮废料吸附酸性矿山排水中的金属含量

摘要：研究了使用农业废弃物，特别是 朗萨 果皮（家蚕皮层）作为低成本吸附剂（活性炭）从酸性矿山排水中去除铜和锰的可行性，以确定本研究中等温模型的吸附。活性炭可以用 20% H3PO4 和 20% NH4OH 化学活化的 朗萨 果皮制成。在被激活之前，朗萨果皮在 300°C 下烧焦 2 小时以产生碳。扫描电镜-EDX 结果表明，用 H3PO4 进行的活化过程获得了 78.62%的高碳值。与 NH4OH 相比，用 H3PO4 活化的碳中的孔形成也更大且更清洁。从 朗萨 果皮废料生产的活性炭的赌注 表面积为 1.4456 平方米/克。此外，孔隙的京华吸附累积体积为 0.000701 立方厘米/克。活性炭的京华 吸附平均孔径(4 五/一个)为 31.31399 纳米，表明介孔尺度尺寸。就酸性矿山排水中的金属吸附而言，最好的活性炭质量为 4 克，每百分比去除 91.42%的锰金属和 15.74%的铜金属。朗缪尔 和 弗伦德利希等温吸附方程用于吸附过程。根据所得数据，等温曲线更符合朗缪尔模型，表明吸收发生在单层中，线性回归系数较接近 1。

关键词：酸性矿山排水, 活性炭, 吸附, 朗萨果皮, 金属含量。

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1. Introduction

Acidic water is one of the main problems in the mining industry because it contains dissolved heavy metals [1], which need to be reduced [2]. Furthermore, it is often obtained from the geochemical reaction of sulfide minerals exposed to air. Its drainage poses a serious threat to human health, animals, and ecological systems because it contains non-biodegradable heavy metal contaminants, such as Cu^{2+}, Mn^{2+}, Fe^{3+}, Zn^{2+}, Cd^{2+}, and Pb^{2+}, which accumulate in living organisms where they cause various diseases [3]. In addition, the high metal and acid content of the drainage can contaminate surface water, groundwater, and soil [4], [5]. It also damages plants, thereby adversely affecting animals, aquatic species, and humans [6], [7].

Consequently, several techniques have been developed to treat waste containing heavy metals, and the most desirable method is the adsorption process because it is economical, efficient, effective, and cheap. The technique has been proven to be an excellent method for wastewater due to its numerous advantages compared to conventional processes. Low-cost adsorbents are an effective and economical method for removing heavy metals. However, materials with high adsorption capacity have been developed to reduce the dose and minimize disposal problems. Bioadsorbents are a better option because they have no adverse effect on human health or the environment [8], [9]. Adsorption is a process where one or more constituents of a liquid solution, which can either be in the form of a gas or liquid, are more concentrated on the surface of a solid (adsorbent), thereby causing separation [10]. Furthermore, activated carbon is one of the adsorbents often used to treat metal ion waste. It can be obtained from organic materials, including agricultural waste, such as langsat fruit peel waste (*Lansium domesticum cortex*).

The adsorption isotherm model has been used in wastewater treatment to predict the ability of adsorbents to remove pollutants up to a certain discharge value. A balance occurs between the amount of adsorbed pollutant and the amount remaining in the solution when the adsorbent mass and the effluent stream are in contact for a long time. Langmuir and Freundlich's models only apply to the batch adsorption systems where sufficient time is provided for balance. Furthermore, during the flow through the adsorbent, many pollutants are expected to come in contact with the active sites. Hence, they are retained on the medium's surface [11]. The Langmuir and Freundlich equations are the most widely used during waste removal. Langmuir's model assumes that metal adsorption is a process that occurs on a homogeneous surface without any interaction between the adsorbed ions until a complete monolayer is formed, while Freundlich's model is based on adsorption on heterogeneous surfaces [12]. Therefore, this study aims to predict the ability of adsorbents to remove pollutants up to a certain discharge level using the Langmuir and Freundlich isotherm adsorption model.

2. Experiment

2.1. Size of Dataset

The materials used include langsat peel and 20% H_3PO_4 and 20% NH_4OH solution, which served as the activators. Furthermore, the equipment consists of a 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 tools needed for the implementation stage, namely:

1. Equipment for making carbon from agricultural waste: A stove with 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 laboratory glassware.

This study was conducted in two stages, and the first was a series of activities that involved writing, making activated carbon, and analyzing the quality of the product. Meanwhile, in the second stage, the product obtained from the agricultural waste was used to adsorb Cu and Mn in the acid mine drainage.

2.2. Carbon Manufacturing Process

Langsat peel was cleaned to remove its impurities, cut into pieces, and dried in an oven at 105˚C for 24 hours. The waste was then transferred into a furnace and burnt at 300˚C for 2 hours.

2.3. Chemical Activation

2.3.1. H_3PO_4 Activation

Seventy grams of carbon were weighed in two places, and they were immersed in a 20% H_3PO_4 solution for 24 hours. Subsequently, they were dried in an oven for 60 minutes at 105˚C, and the experiment was repeated twice.

2.3.2. NH_4OH Activation

70 g of carbon were weighed in two places, and they were immersed in a 20% NH_4OH solution for 24 hours. Subsequently, they were dried in an oven for 60 minutes at 105˚C, and the experiment was repeated twice.

2.4. Application of Activated Carbon for Acid Mine Drainage Treatment and Determination of Adsorption Isotherms

Activated carbon, which met the standard requirements, was applied as an adsorbent when treating acid mine wastewater (artificial Cu and Mn) to form liquid waste. This treatment was carried out by mixing 2g, 3g, 4g, 5g, 6g, and 7g of the absorbent with a 100mL sample containing 58.52 ppm MnO_2 and 64.30 ppm $CuSO_4$ in 2 Erlenmeyer. Furthermore, the mixture was stirred using a rotary shaker at 50 rpm for 60 minutes and then filtered with Whatman 42 paper. The liquid obtained from the separation was analyzed for its metal ion content before and after the treatment using atomic absorption spectrophotometry, and the results were expressed in mg/g. These data were then used to determine the adsorption isotherm model. The spectrophotometric test used SNI 6989.5:2009 and SNI 6989.6:2009 for the determination of total Mn and Cu content, respectively. Subsequently, data analysis was carried out by determining the final concentrations of MnO_2 and $CuSO_4$ after treatment using the adsorbent concentration and adsorption capacity. The results were then inputted into the Langmuir and Freundlich equations to determine the adsorption isotherm model of the adsorbent.

The amount of Cu and Mn ions adsorbed by agricultural waste (Q_e) was calculated for each experiment using the equation below:

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

where Q_e (mg/g) is the adsorption capacity, C_0 (mg/L) is the initial concentration of Cu and Mn, C_e (mg/L) is the equilibrium concentration of Cu and Mn, V (L) is the volume of acid mine water, and W (g) is the weight of adsorbent added [13], [14]. The Langmuir isotherm model equation is illustrated below:

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

where q (mg/g) is the number of heavy metal ions adsorbed into the unit mass of the polymeric beads, K_L is the Langmuir equilibrium constant 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) [15].

The Freundlich isotherm model assumes that adsorption occurred on a heterogeneous surface, and it was developed into an empirical equation [15]:

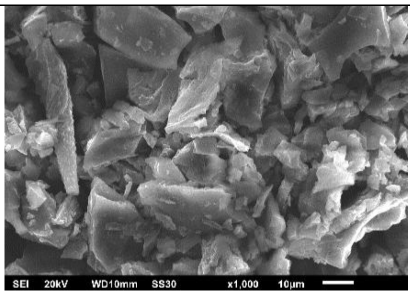
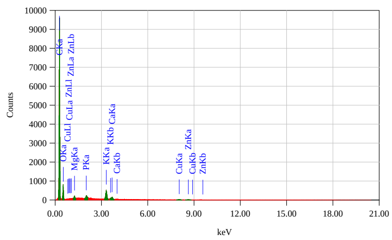
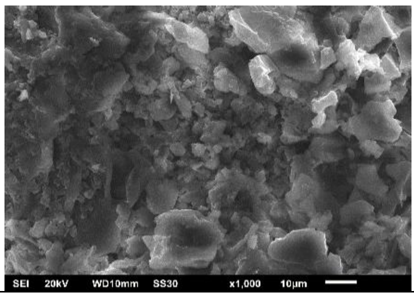
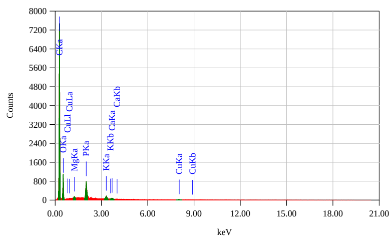
$$q_e = KC_e^{1/n} \quad (3)$$

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 the empirical constants, which indicate the adsorption capacity and intensity, respectively.

3. Results and Discussion

Table 1 shows the observation results of product topography using SEM-EDX. Furthermore, a carbon content value of 78.62% was obtained from the H_3PO_4 activation, which was higher than NH_4OH. The pores on the product activated with acid were more abundant and cleaner than others, activated with an alkaline solution. As stated in [16], the formation of many pores was caused by the evaporation of volatile matter from the raw material due to pyrolysis. This porosity causes the degradation of raw material components into gases (CO, CO_2, H_2, CH_4), liquids (liquid smoke, tar, hydrocarbons, and water), and solid (carbon). As also reported in [17], [18], [19], the porosity of adsorbents can affect their adsorption power. Therefore, many pores and a relatively large surface area increase their ability to adsorb organic and inorganic compounds in wastewater.

Table 1 Results of SEM-EDX test of carbon soaked with H_3PO_4 and NH_4OH

Scale	Solution	SEM	EDX	Content Composition
1000X	H_3PO_4			Fitting Coefficient: 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 Coefficient: 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

The BET (Brunaeur Emmet Teller) and BJH (Barret Joyner Halenda) test results revealed that the activated charcoal obtained from the peel waste had a surface area of 1.4456 m^2/g. In addition, a cumulative adsorption volume of 0.000701cm³/g was recorded for pores ranging within 1.7000 nm-300.0000 nm diameter. Furthermore, the BJH adsorption average pore diameter (4V/A) was 31,31399 nm. These results indicate that activated carbon from langsat peel waste has a mesoporous scale pore with a diameter ranging from 2-50 nm [20].

Table 2 Results of AAS test of langsat fruit-peel activated carbon (Mn metal parameter)

Parameter	Activated Carbon Mass (gram)	Results (mg/L)	Removal (%)	Method Specification
Mn	Preliminary Solution	37.00	0	SNI 6989.4:2009
	2	6.14	83.41	
	3	3.19	91.37	
	4	3.17	91.42	
	5	3.05	91.77	
	6	2.98	91.94	
	7	2.92	92.10	

A graph was plotted based on the AAS test results, as shown in Figure 1.

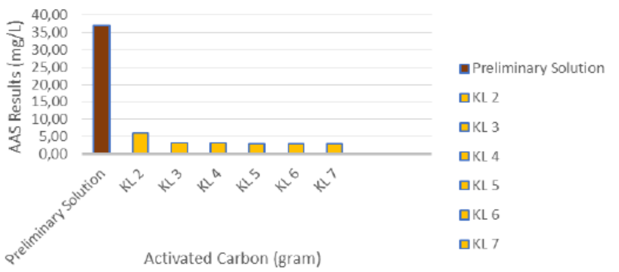


Fig. 1 Graph of AAS test results for langsat fruit-peel activated carbon in acid mine drainage (Mn metal adsorption)

Table 3 AAS test results for langsat fruit-peel activated carbon (Cu metal parameter)

Metal	Langmuir Isotherm			Freundlich Isotherm		
	q_{max} (mg/g)	K_L (mol/L)	R^2	K_F (mol/L)	1/n	R^2
Cu	1.7170	76.6426	0.9483	1.7159	0.000001	0.9255

Based on the AAS test results for Cu metal, a graph can be made as shown in Figure 2 below:

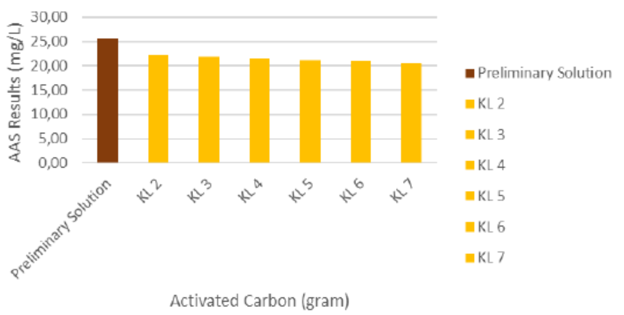


Fig. 2 Graph of AAS test results for langsat fruit-peel activated carbon in acid mine drainage (Cu metal adsorption)

Tables 2 and 3 show that a maximum metal removal of 91.42% was obtained for Mn treated with activated charcoal, while a value of 15.74% was recorded for Cu. The maximum values occurred at an optimal dose of 4 grams, after which they remained constant due to the adsorbent nature [15].

Furthermore, the mass positively correlates with the adsorption of heavy metal ions, as shown in Figures 1 and 2. This was determined by the size or surface area, which increased along with the weight of the adsorbent, leading to a higher percentage of metal removal at a higher dose [21], [22], [23]. Conversely, the percentage of metal adsorption can decrease as the dose increases, which is caused by the formation of agglomerates, leading to a decrease in the overall surface area [24], [25].

Table 4 shows the calculation results of the Langmuir and Freundlich isothermal adsorption equations for Mn.

Table 4 The Langmuir and Freundlich equations for Mn in the adsorption process

Metal	Langmuir Isotherm			Freundlich Isotherm		
	q_{max} (mg/g)	K_L (mol/L)	R^2	K_F (mol/L)	1/n	R^2
Mn	15.4632	76.5878	0.9521	15.4304	0.000001	0.9059

Table 5 shows the calculation results of the Langmuir and Freundlich isothermal adsorption equations for Cu:

Table 5 The Langmuir and Freundlich equations for Cu in the adsorption process

Parameter	Activated Carbon Mass (gram)	Results (mg/L)	Removal (%)	Method Specification
Cu	Preliminary Solution	25.60	0	SNI 6989.4:2009
	2	22.17	13.41	
	3	21.95	14.28	
	4	21.57	15.74	
	5	21.13	17.48	
	6	21.00	17.97	
	7	20.59	19.58	

Based on the Langmuir adsorption model and the q_{max} value obtained in Tables 4 and 5, a maximum capacity of 15.4632 mg/g was recorded after using activated carbon for Mn adsorption. Meanwhile, a value of 1.7170 mg/g was obtained from the adsorption of Cu, which is in line with the order that Mn > Cu. The adsorption capacity can be reduced by the outer layer of the activated carbon when it is saturated with the adsorbed materials. The higher the concentration of the waste solution containing metals, the higher the number of molecules that collide and interact with the adsorbent to increase its capacity. Based on the Freundlich parameters, all activated carbon obtained from the agricultural waste for Mn and Cu adsorption had a value of 1/n, lower than 1. This result indicated that the process was favorable under these conditions. This finding is consistent with an assumption that the adsorption is favorable and cooperative when the value is $0 < 1/n < 1$. Therefore, it can be concluded that the process occurred on a heterogeneous surface, and it was single-layered for all activated carbon obtained from the agricultural waste. Tables 4 and 5 show that the adsorption of Mn and Cu by the absorbent produced was closely related to the Langmuir isotherm model. This was indicated by the relation coefficient (R^2) value, which was closer to 1 compared to the Freundlich model.

4. Conclusion

The present study aimed to assess the capability of agricultural wastes, specifically the langsat fruit peel (*Lansium domesticum cortex*) as a low-cost sorbent (activated carbon) to remove Cu and Mn from acid mine drainage and determine the adsorption of the isothermal model of the heavy metals. The SEM-EDX results showed that carbon produced from langsat fruit peel, which was then activated using H_3PO_4, had a larger, cleaner, more pore formation and greater C value of 78.62% compared to others activated with NH_4OH. The pore formation was also larger and cleaner in carbon activated with H_3PO_4 than NH_4OH. A BET surface area of 1.4456 m^2/g was obtained for the activated charcoal produced from langsat peel waste. Furthermore, a BJH adsorption cumulative volume of 0.000701cm^3/g was recorded for pores. The BJH adsorption average pore diameter (4V/A) for the charcoal was 31.31399 nm, which indicates that it has a mesoporous scale size. The results indicated that the langsat fruit peel activated carbon is an effective adsorbent for removing Cu and Mn metals. The best activated carbon in terms of metals adsorption in acid mine drainage was in a mass of 4 grams, with each percent removal of 91.42% for Mn metal and 15.74% for Cu metal. Based on the data, the adsorption isotherm curve closely corresponded with the Langmuir isotherm model (adsorption occurred on the monolayer). This was indicated by the R^2 value obtained from the Langmuir equation, closer to 1 than that of Freundlich.

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