The Application of Carbon from Discarded Langsat Fruit Peel to Adsorb Copper and Manganese from Acid Mine Drainage

Lailan Ni`mah[1], Sri Rachmania Juliastuti[1] and Mahfud Mahfud[1,*]

[1] Department of Chemical Engineering, Institut Teknologi Sepuluh Nopember,
Surabaya 60111, Indonesia

`mahfud@chem-eng.its.ac.id`

Abstract. The study aimed to explore the practicality of utilizing agricultural waste, specifically langsat fruit peel (*Lansium domesticum* cortex), as an economical sorbent, namely activated carbon, for the removal of copper (Cu) and manganese (Mn) from acid mine drainage. Additionally, it sought to assess the adsorption behavior through isothermal modeling. Prior to activation, the langsat fruit peel underwent charring at 300°C for 2 hours. Activated carbon was produced from langsat fruit peel through chemical activation using 20% H_3PO_4 and 20% NH4OH. The SEM-EDX analysis revealed a significant carbon content of 78.62% when H_3PO_4 was employed for activation, and this process resulted in larger and cleaner pore structures compared to NH_4OH activation. The activated carbon derived from langsat peel waste exhibited a BET surface area of 1.4456 m2/g, with a BJH adsorption cumulative volume of 0.000701 cm^3/g for its pores. The BJH adsorption average pore diameter (4V/A) indicated a mesoporous scale size, measuring at 31.31399 nm. In terms of its efficacy in adsorbing metals from acid mine drainage, the most favorable results were observed when using 4 grams of activated carbon, achieving a removal rate of 91.42% for Mn and 15.74% for Cu. To model the adsorption process, both Langmuir and Freundlich isotherm equations were applied. The data analysis revealed that the Langmuir model closely matched the isotherm curve, suggesting monolayer adsorption. Furthermore, the linear regression coefficient for the Langmuir model approached 1, indicating a strong fit to the data.

Keywords: Acid Mine Drainage, Activated Carbon, Process of Adsorption, Use Of Langsat Fruit Peel, Presence of Metal Substances.

1 Introduction

One of the main concerns in the mining sector is the presence of acidic water, which carries heavy metals such as besi, mangan, tembaga, cadmium, timbal, dan nikel[1] that undergo dissolution[2], this should be reduced[3] because The high content of metals and acids in acid mine water can contaminate surface water, groundwater, and soil[4],

ICESDM 2023, November 02, Banjarmasin, Indonesia

DOI 10.4108/eai.2-11-2023.2348004

thus posing a threat to plants, having adverse effects on wildlife, aquatic species, and humans[5]. Furthermore, it often originates from the geochemical interaction of sulfide minerals ejected at significant altitudes, and their discharge poses a serious threat to the well-being of humans, wildlife, and the natural environment. This is due to the presence of non-degradable heavy metal contaminants such as Cu2+, Mn^{2+}, Fe^{3+}, Zn^{2+}, Cd^{2+}, and Pb^{2+}, which accumulate in organisms and lead to various illnesses[6]. The elevated metal content and acidic nature of the waste can contaminate surface water, groundwater, and soil [7], [8]. It damages plants, thereby negatively impacting animals, aquatic species, and humans[9], [10]. As a result, several methods precipitation, ion exchange, membrane technology [11], electrochemistry [4], electrodialysis [12], biological processes [13], and adsorption [14]; [15]; [16] have been devised to manage waste containing heavy metals, with the most effective approach being the adsorption process due to its affordability, efficiency, effectiveness, and cost-effectiveness. Adsorption is an excellent choice for wastewater treatment, offering numerous advantages compared to conventional processes. Employing low-cost adsorbents is an efficient and cost-effective means of heavy metal removal. Nonetheless, efforts are ongoing to develop materials with higher adsorption capacities to reduce dosage and disposal concerns. Bio-adsorbents are a popular choice as they have a minimal impact on human health and the environment [17], [18]. Adsorption is a process in which at least one component of a fluid, whether it be a gas or another liquid, is concentrated on the surface of a solid in such a way that it leads to separation[19]. Hence, activated carbon is a commonly employed substance in the recycling of metal ion waste. This common substance has the capacity to include different forms of refuse, such as domestic waste and the discarded peels of langsat fruit.

During the carbonization process, despite the increased surface area, absorption remains relatively low due to the presence of tar residues covering the pores. Therefore, an activation process is required to further enhance the carbon surface. One method is through chemical activation since, in chemical activation, tar can dissolve during immersion[20].

The operation of the adsorption isotherm is frequently employed in wastewater recycling, especially when there is a concern about reaching a critical turbidity threshold caused by substantial pollutant release. This situation arises from an imbalance between the contaminants absorbed in the water and the introduction of additional waste into the river flow. The Freundlich-Langmuir model is applicable to this adsorption system when an adequate amount of water is provided, leading to alterations. Throughout the adsorption process, various impurities present in the conduits are separated by the medium[21]. The Langmuir and Freundlich models are frequently applied in the treatment of model waste that can adsorb metal contaminants, involving a process that takes place on the water's surface without any adsorbing particles until a complete monolayer is established. In contrast, the proposed model is based on adsorption occurring on a heterogeneous surface[22]. Therefore, this experimental study aims to determine the observation limit for removing impurities up to a specific water flow rate using the adsorption isotherm models presented by Langmuir and Freundlich.

2 Method

2.1 Dataset size

The materials used in this process include langsat peels from South Kalimantan and a 20% H_3PO_4 0,1 N and 20% NH_4OH 0,1 N solution, which serve as activators. The equipment comprises a crusher, an oven dryer (Memmeth DIN 12880-KI), a furnace (SX-2.8-12 Boc Huanghua Faithful Instrument Co.Ltd), a precision balance (Shimadzu AW-220), a screening apparatus (with a mesh size of 850 microns), and various other tools required for the experimental phase. Specifically: for the production of carbon from agricultural waste, a furnace from SX-2.8-12 Boc Huanghua Faithful Instrument Co.Ltd is used. For carbon characterization, equipment includes the SEM-EDX JEOL JSM-6360LA, precision balances, grills, porcelain dishes, desiccators, heaters, and laboratory glassware.

This study was conducted in two distinct phases. The first phase involved a series of activities focused on the synthesis of structured carbon, its production, and the evaluation of its potential. Subsequently, in the second phase, the carbon derived from agricultural waste was employed to adsorb copper (Cu) and manganese (Mn) from contaminated mine discharge.

2.2 Process of Carbon Production

Langsat peels were cleaned to remove contaminants, cut into segments, and subjected to drying on a grill at 105°C for 24 hours. Subsequently, the waste material was transferred to a furnace and heated to 300°C for a duration of 2 hours.

2.3 Activation of the Carbon

Activation using H_3PO_4: A total of 70 grams of carbon was divided into two portions and immersed in a 20% H_3PO_4 solution for 24 hours. Subsequently, they underwent drying in an oven at 105°C for an hour, and this initial process was repeated twice.

Activation using NH_4OH: Seventy grams of carbon were divided into two portions and soaked in a 20% NH_4OH solution for 24 hours. Afterwards, they were dried on a grill for one hour at 105˚C, and this procedure was repeated twice.

2.4 Utilizing Activated Carbon in Acid Mine Drainage Treatment and Establishing Adsorption Isotherms

Activated carbon that met the required standards (SNI 06-3730-1995 for the technical specifications of activated carbon quality) was employed as an adsorbent for the treatment of acidic mine wastewater containing simulated copper (Cu) and manganese (Mn) ions, with the goal of converting it into liquid waste. This involved combining 2g, 3g, 4g, 5g, 6g, and 7g of the activated carbon with a 100mL sample solution containing 37.00 ppm MnO_2 and 25.61 ppm $CuSO_4$ in two separate Erlenmeyer flasks.

Subsequently, the mixture was agitated using a rotary shaker at 50 rpm for one hour, followed by filtration through Whatman 42 paper. The liquid obtained after filtration was analyzed for its metal ion content using atomic absorption spectrophotometry, and the results were expressed in mg/g. This data was then utilized to determine the adsorption isotherm model. The spectrophotometric analysis adhered to SNI 6989.5:2009 and SNI 6989.6:2009 standards for the quantification of total Mn and Cu content. Subsequently, data analysis was carried out by establishing relationships between MnO_2 and $CuSO_4$ concentrations after treatment, using 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 particles adsorbed by the ordinary waste, which was not stabilized for each experiment, was determined using the following formula:

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

In this equation, Qe (mg/g) represents the adsorption capacity, C_0 (mg/L) denotes the initial concentration of Cu and Mn in the solution, Ce (mg/L) represents the equilibrium concentration of Cu and Mn, V (L) stands for the volume of the acidic mine water, and W (g) indicates the mass of the adsorbent added[23], [24]. The Langmuir isotherm model equation is defined as follows:

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

In this context, q (mg/g) represents the quantity of significant metal ions adsorbed per unit mass of the polymeric sites, K_L signifies the Langmuir equilibrium constant indicating the affinity of binding sites, C_e stands for the equilibrium Ni^{2+} ion concentration, and max denotes the maximum adsorption capacity (theoretical monolayer saturation capacity)[25].

The Freundlich isotherm model assumes that adsorption occurs on a heterogeneous surface, and it has been formulated into a precise equation[25]:

$$q_e = K_F C_e^{1/n} \tag{3}$$

In this equation, q_e represents the adsorption capacity (mg/g), C_e signifies the concentration of the adsorbate in the solution (mg/L), and K_F and n are the empirical constants that respectively represent the adsorption capacity and the adsorption intensity.

3 Result and Discussion

3.1 Morphological and surface analysis of activated carbon through SEM-EDX testing.

The characteristics of activated carbon can be examined through sample tests, one of which is SEM-EDX. This can be seen in Figure 1 for activation using H_3PO_4 and Figure 2 for activation using NH_4OH.

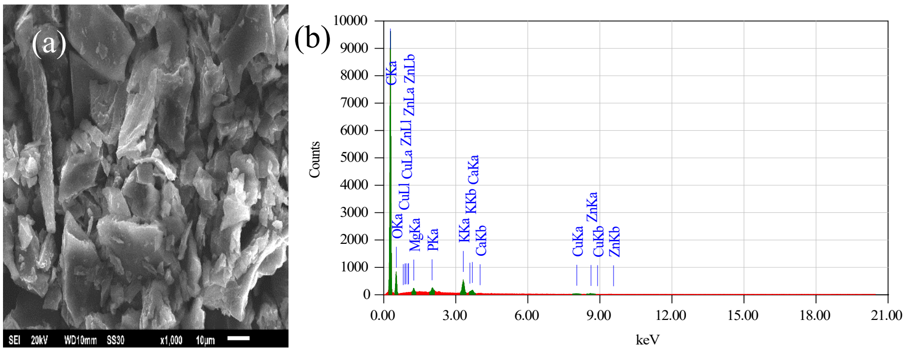

Fig. 1. (a) SEM micrographs of activated carbon with H_3PO_4 x1000 and (b) EDX contents of activated carbon treated with H_3PO_4

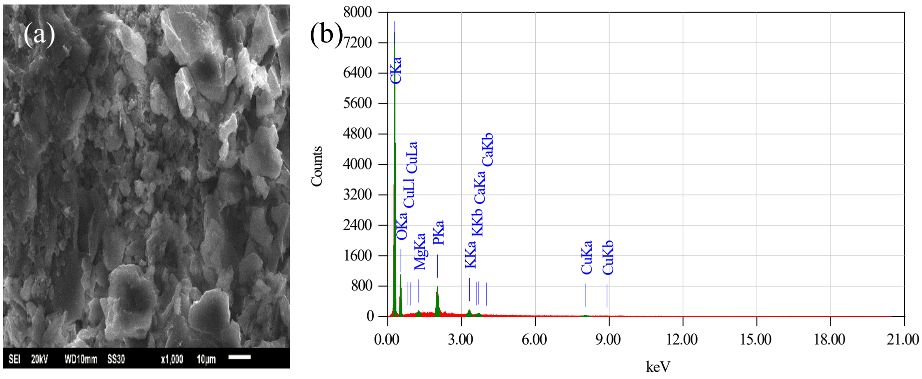

Fig. 2. (a) SEM micrographs of activated carbon with NH_4OH x1000 and (b) EDX contents of activated carbon treated with NH_4OH

The table presented above provides insights into the findings from the SEM-EDX analysis of production materials. It reveals that carbon content amounted to 78.63%, which is higher in cases where H_3PO_4 activation was employed, as compared to NH_4OH activation. Additionally, the products activated with acid exhibited more pronounced and cleaner pores when compared to those activated with an alkaline solution. This distinction arises due to hydrolysis, a process involving the conversion of ingredients into gases such as CO, CO_2, H_2, CH_4, as well as liquids like liquid smoke, hydrocarbons, water, and carbon density[26]–[28] through our investigations, we have found that the absorption capacity can be influenced by the porosity of the material.

An EDX analysis was performed to determine the elements or atomic makeup of the activated carbon. The results of the EDX Analysis are displayed in Table 1.

Table 1. results of the adsorbent analysis using EDX

Element	Mass (%) (NH_4OH activation treating)	Mass (%) (H_3PO_4 activation treating)
Carbon	78.62	73.73
Oxygen	18.74	23.93
Magnesium	0.26	0.21
Phosporus	0.21	1.42
Kalium	1.29	0.36
Calcium	0.31	0.12
Cuprum	0.33	0.30
Zinc	0.23	-

Table 1 presents the results of the EDX analysis performed on activated carbon derived from langsat fruit peel activation with chemical. The table shows that the components of the activated carbon obtained from langsat fruit peel include the elements C, O, Mg, P, K, Ca, Cu, and Zn. The highest proportion is observed in carbon and oxygen. The data acquired by producing activated carbon from agricultural waste, specifically langsat fruit peel, using H_3PO_4 for activation beside using NH_4OH. The product of the carbonization stage is a solid with high carbon content, typically in the range of 25-50% by mass, depending on the raw material and process parameters used[29]. In this activation process, there is a removal of hydrogen, gases, and water from the carbon surface, leading to physical changes on the surface as well as an increase in carbon content and other constituents.

As a result, we believe that the presence of significant and extensive pores with a larger surface area demonstrates an enhanced capability to absorb various organic and inorganic compounds in both domestic and industrial wastewater treatment plants. Our experimental findings, using the Brunauer Emmet Teller (BET) and Beret Joyner Halenda (BJH) methods, indicated that the charcoal material derived from langsat fruit peel waste exhibited a remarkable surface area of 1,445.7 square meters per gram. Simultaneously, we recorded a cumulative volume of observations amounting to 0.00000701 cubic centimeters per gram for pores within the size range of 1,7000 MN to 300,000 MN. These results clearly indicate that the activated carbon derived from langsat peel waste possesses mesoporous structures with diameters ranging from 2 to 50 nanometers[30].

In the application of activated carbon for adsorbing metals in acid mine water waste, a reduction in metal content is obtained as detailed in Table 2 and Table 3.

Table 2. Analytical results from the AAS test of activated carbon derived from langsat fruit peel, specifically focusing on the parameter related to Mn metal

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	

Table 3. Analytical results from the AAS test of activated carbon derived from langsat fruit peel, specifically focusing on the parameter related to Cu metal

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	

Tables 2 and 3 illustrate an impressive removal rate of 91.42% for Mn when treated with activated carbon, while a value of 15.74% was recorded for Cu. These optimal values were observed at a dosage of 4 grams, and they remained consistent due to the adsorbent's characteristics[25].

Additionally, there is a strong correlation between the mass and the adsorption of heavy metal ions. This relationship is attributed to the increased surface area and sites available on the adsorbent, leading to a higher degree of metal removal at higher dosages[31]–[33]. The extent of metal adsorption may decrease as the dosage increases, a phenomenon attributed to the formation of agglomerates that results in a reduction of the overall surface area[34], [35].

Table 4. The Langmuir and Freundlich models applied to the adsorption process of Cu:

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

Table 5. The Langmuir and Freundlich models applied to the adsorption process of Mn

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

Considering the Langmuir adsorption model and the qmax values obtained from Tables 4 and 5, a maximum capacity of 15.4632 mg/g was achieved after employing activated carbon for Mn adsorption. Meanwhile, for Cu adsorption, a value of 1.7170 mg/g was obtained, confirming the preference for Mn over Cu adsorption. It should be noted that the outer layer of activated carbon can be saturated as it absorbs the materials. The greater the concentration of the waste solution containing metals, the more particles impact and aid the adsorbent in reaching its maximum capacity. In terms of capacity, all activated carbon derived from agricultural waste for Mn and Cu adsorption exhibited a value of 1/n, which was less than 1. This suggests that the adsorption process was effective under these conditions. This observation aligns with the theory that when the value is between 0 and 1, the adsorption is efficient and robust. Therefore, it can be inferred that the process occurred on a heterogeneous surface and was single-layered for all activated carbon derived from agricultural waste. Tables 4 and 5 indicate that the adsorption of Mn and Cu by the produced material strongly adhered to the Langmuir isotherm model, as evidenced by the higher coefficient of determination (R^2) values approaching unity, contrasting with the Freundlich model.

4. Conclusion

Carbon that has been activated using H3PO4 complies with the standards outlined in SNI 06-3730-1995 (technical criteria for activated carbon quality) and features pores within the mesoporous range (with pore diameters ranging from 2 to 50 nm). The adsorption isotherm pattern on this activated carbon aligns more closely with the Langmuir isotherm model, suggesting monolayer adsorption.

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