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Preparation of magnetic activated carbon based on breadfruit (*Artocarpus altilis*) peel waste by impregnation method for lead absorption in wastewater

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ABSTRACT

Magnetic activated carbon has been made from breadfruit skin waste using the impregnation method. The research began with the drying of breadfruit skin followed by carbonization. Furthermore, breadfruit skin carbon was activated using H_3PO_4. The final stage of making magnetic activated carbon is impregnation of Fe_3O_4 into activated carbon. The resulting magnetic activated carbon is then applied to treat Pb-containing wastewater. Characterization results show that the surface area of magnetic activated carbon is 1110.804 m^2/g with a pore diameter of 18.59 °A. The surface morphology of magnetic activated carbon shows the presence of Fe in the pore indicated by the presence of white color. Based on SEM EDX analysis, it is known that the Fe content on magnetic activated carbon is 16.6%. The ability of Pb absorption in waste based on the calculation of adsorption capacity is 6.635 mg/g.

Keywords: breadfruit, magnetic, adsorption, impregnation, surface area.

INTRODUCTION

Magnetic activated carbon is activated carbon that is composited with metal precursors so that it has magnetic properties. The metal precursor that is often used in the manufacture of magnetic activated carbon is iron oxide. Magnetic activated carbon can attract metals that are in liquid waste. The use of magnetic activated carbon is often found in water treatment applications, pollutant removal, and chemical separation. The advantage is the ease of the separation process, as it can be quickly pulled out of the mixture using a magnet, thus facilitating the cleaning and treatment process.

The manufacture of magnetic activated carbon can be done by 2 (two) methods, namely the precipitation method and the impregnation method. In the precipitation method, iron precursors are dissolved in water and precipitation agents (such as sodium hydroxide) are added to form iron oxide precipitates. This process is usually carried out at a certain temperature with good stirring. While the impregnation method is done by making a precursor solution usually in the form of iron oxide salts in a suitable solvent. Furthermore, impregnation of activated carbon with magnetic materials is carried out. This process allows magnetic particles to be evenly distributed on the surface of activated carbon (Bo et al., 2024).

Magnetic activated carbon is made by combining carbon raw materials and magnetic particles. Materials in the manufacture of activated carbon include wood, coconut shells, or agricultural waste, salak seeds, bamboo sticks, fly ash, coconut shells, and industrial waste. While magnetic precursors that can be used are Fe_3O_4 (magnetite), Fe_2O_3 (hematite) and nickel. The manufacture of magnetic activated carbon begins with the manufacture of carbon followed by an activation process to produce activated carbon. Activation of activated carbon can be done by two methods, namely physical activation and chemical activation, both of which are done to increase the capacity of the adsorbent. The most widely used chemical activating agents are H_3PO_4, $ZnCl_2$, KOH and NaOH, while for physical activation is usually done by heating at high temperatures and flowing N_2 gas (Zhang et al., 2023), (Al-qodah et al., Z2023) (Mirzaee and Sartaj, 2022).

Several studies have been conducted to make magnetic activated carbon. Magnetic activated carbon made from coconut shell using the precipitation method produces a surface area of 324.7 m^2/g (Mirzaee & Ehsan, 2022). Making magnetic activated carbon from industrial waste has a surface area of 822 m^2/g (Sanchez et.al, 2021). The precipitation process is used in the manufacture of magnetic activated carbon for the absorption of Cr (IV) in liquid waste (Zhenyu et al.,2023). Food industry waste is used as a material for making magnetic activated carbon using the impregnation process for the absorption of mercury in wastewater (Sánchez et al., 2022). Magnetic activated carbon made from bamboo stems using the precipitation method has a surface area of 1388.83 m^2/g (Yang et al., 2023). *Pittosporum pterocarpus* flowers can be processed into magnetic activated carbon with a precipitation process capable of absorbing methyl orange in water 93.3% (Thanh et.al, 2024). Making magnetic activated carbon from commercial activated carbon powder using the precipitation method has an adsorption capacity of 75.76 mg/g (Sirirat et al., 2024). Magnetic activated carbon made from tree seeds of copper pods using the precipitation method produces low adsorption capacity sorbents and the adsorption process is exothermic (Adithya et al., 2024).

Testing the quality of magnetic activated carbon produced is done through characterization tests and performance tests. Characterization tests carried out include surface morphology using SEM, metal content test in magnetic activated carbon using SEM EDX, surface area test using BET method and functional group test using FTIR. Performance tests were conducted to determine the adsorption capacity and maximum ability of metal absorption. The performance test of magnetic activated carbon was carried out through the use of magnetic activated carbon on the absorption of metals in wastewater.

In this study, magnetic activated carbon made from breadfruit peel waste using the impregnation method was made. The activator used in the stage of making activated carbon is H_3PO_4 while as a precursor in the impregnation stage Fe_3O_4 is used. Magnetic activated carbon was characterized by surface morphology test with SEM EDX, surface area with surface area analyzer using BET method. Performance tests were carried out calculating the adsorption capacity on the absorption of Pb in wastewater.

MATERIAL AND METHODS

Materials

Breadfruit peel waste was obtained from breadfruit processing industry around Surabaya. Fe_3O_4 was used pro analysis purchased from a chemical store in Surabaya. H_3PO_4 pro analysis was purchased from a chemical store in Surabaya. Pb waste used was liquid waste.

Preparation of activated carbon made from breadfruit skin

The breadfruit skin that has been cleaned from impurities is dried in an oven at 110 °C until a moisture content of 10% is obtained. Next, the carbonation process was carried out on the dried breadfruit skin using a furnace. The resulting breadfruit skin carbon was reduced in size until a size of 200 mesh was obtained. The activation process was carried out using chemical activation and physical activation. Chemical activation was carried out by soaking the carbon in 1 M H_3PO_4 solution for 8 hours followed by filtering and drying using an oven. Physical activation was carried out after chemical activation using a microwave at 450 watts. During physical activation, the microwave was supplied with N_2 gas to keep the microwave environment free of air. The activated carbon produced was then analyzed by SEM EDX and surface area.

Preparation of magnetic activated carbon by impregnation

Activated carbon resulting from activation was weighed according to the variables, then added Fe_3O_4 and distilled water until submerged, then stirred. The mixture was filtered and dried. There are five variables of activated carbon (AC) mass ratio with Fe_3O_4 or iron oxide (IO), namely, (AC:IO = 2:1); (AC:IO = 2:2); (AC:IO = 2:3); (AC:IO = 2:4); (AC:IO = 2:5). Next, the mixture was heated in a furnace for 3 hours at 450 °C. The magnetic activated carbon was then cooled in a desiccator to room temperature. Characterization of magnetic activated carbon was done through SEM-EDX analysis and surface area.

Surface morphology analysis and compositions

The surface morphology of magnetic activated carbon was analyzed using SEM EDX. This analysis is intended to determine the shape of the surface morphology so that it will be seen the presence of Fe on the internal surface of magnetic activated carbon and components that exist on the surface of magnetic activated carbon. Analysis is carried out in seven stages, namely sample preparation, sample coating, SEM processing, scanning and data recording, morphological analysis, composition analysis, result interpretation. The sample preparation stage is aimed at ensuring that the sample is dry and free of contaminants. Next, the sample was coated with gold or carbon to prevent charge collection. The next stage is SEM setup by adjusting the voltage, electron beam current, and scanning mode. The next step is scanning and recording data by moving the electron beam over the surface. The SEM will produce high-resolution images of the sample surface. Record these images for further analysis at the morphological analysis stage. The analysis is carried out by observing the resulting images to assess the morphology and surface structure of the magnetically activated carbon and pay attention to pore size, distribution, and particle shape. Compositional analysis was conducted to determine the elements contained in the magnetic activated carbon. Interpretation of the results is done to gain insight into the physical and chemical characteristics of the magnetic activated carbon. This information can be used for specific applications, such as adsorption or catalysis.

Surface area analysis

The active surface area of magnetic activated carbon was analyzed using a surface analyzer with the Brunner-Emmet-Teller (BET) method. This tool serves to determine the diameter and volume of pores, as well as the specific surface area of the material. Based on the principle of adsorption-desorption of adsorbate gas. The gas adsorption mechanism is the absorption of gas (nitrogen, argon and helium) on the surface of a solid material to be characterized at a fixed temperature. If the volume of gas (nitrogen, argon, or helium) that can be absorbed by a solid surface at a certain temperature and pressure is known and the theoretical surface area of one molecule of gas absorbed is known, the total surface area of the solid can be calculated. Surface area is the number of pores on each unit area of the sample. While the specific surface area is the surface area per unit gram.

Determination of adsorption capacity

The adsorption capacity of magnetically activated carbon was carried out on Pb adsorbs in wastewater. At this stage, adsorption of Pb-containing wastewater was carried out with a batch system. The wastewater was first analyzed for Pb heavy metal content using an AAS instrument. Magnetite activated carbon was applied to 50 mL of wastewater with predetermined variables with a rotating speed of 150 rpm and an adsorption time of 3 hours. This process was carried out with a batch system. The mass variations of magnetic activated carbon used were 1, 2, 3, 4 and 5 grams. After the adsorption process, the magnetite activated carbon was separated from the wastewater by filtering. The liquid waste is then analyzed for Pb content using AAS to calculate the adsorption capacity. Calculation of the adsorption capacity of magnetic activated carbon is calculated using the Langmuir isotherm equation, Equation 1.

$$\frac{Ce}{Qe} = \frac{Ce}{Qm} + \frac{1}{k\,Qm} \tag{1}$$

where: *Ce* is the equilibrium concentration (mg/L), *Qe* is the equilibrium adsorption (mg/g), *k* is the adsorption constant (L/mg), while *Qm* is the adsorption capacity (mg/g). It is calculated using Equation 2

$$Q_e = \frac{(Co-Ce)V}{m} \tag{2}$$

where: *Co* is initial concentration (mg/L), *Ce* is equilibrium concentration (mg/L), *V* is volume of sample (L), while m is mass of magnetic activated carbon.

RESULT AND DISCUSSION

Characterization of magnetic activated carbon

Surface area

The surface area of magnetic activated carbon was analyzed using a surface analyzer with the BET method. This analysis is done by using gas adsorption, usually nitrogen, to measure the surface area. The data obtained helps in determining the specific surface area of activated carbon calculated using the BET equation. The surface area of magnetic activated carbon is very large ranging from 500 to 1200 m² per gram, depending on the synthesis and modification methods used. To determine the effect of making magnetic activated carbon on surface area, this study measured the surface area of activated carbon before impregnation and after impregnation. The results of the surface area analysis of breadfruit skin carbon, breadfruit skin activated carbon and breadfruit skin magnetic activated carbon are presented in Table 1.

Based on Table 1, it can be seen that there is an increase in the surface area of breadfruit skin carbon due to the activation and impregnation process. The surface area obtained is magnetic activated carbon > activated carbon > carbon. Breadfruit skin carbon from the carbonation process has the smallest surface area because the pores on the carbon are still covered with dirt and covered. The activation process using a combination of chemical and physical activation using microwave has the ability to remove impurities on the carbon surface and open the pores on the carbon surface so that it has a larger active surface compared to carbon before activation. The iron oxide impregnation method on the surface of activated carbon makes the surface of activated carbon more porous. This is evidenced by the results of the analysis with BET which shows that the surface area of magnetic activated carbon is greater than the activated carbon before impregnation.

Activation of breadfruit peel carbon using H_3PO_4 produces activated carbon with a surface area of 987.5830 m²/g. This value is greater than the activated carbon of orange peel with HCl activator which has a surface area of 630.34 m²/g (Elangovan et al., 2024). This indicates that H_3PO_4 can be recommended as an activator for the manufacture of activated carbon. Iron oxide impregnation process can be used to increase the surface area of activated carbon. The surface area of magnetic activated carbon made from breadfruit skin has higher surface area characteristics compared to magnetic activated carbon made from oak tree trunks which has a surface area of 264.97 m²/g (Elangovan et al., 2024). Magnetic activated carbon made from biowaste has a surface area of 481.3 m²/g (Sarra et.al, 2024).

Table 1. Surface area of carbon, activated carbon and magnetic activated carbon

Carbon type	Surface area (m²/g)
Carbon	262.004
Activated carbon	987.5830
Magnetic activated carbon	1110.804

Pore diameters

The pore diameter of magnetic activated carbon is the size of the small pore holes in the structure of magnetic activated carbon, which allows the material to adsorb and trap molecules of other substances. These pores are very important in determining the adsorption ability of activated carbon. The characteristics and size distribution of these pores affect the effectiveness of activated carbon in various applications. Smaller pores are suitable for adsorbing small molecules, while larger pores can capture larger molecules. Its magnetic properties are generated through processes that usually involve doping with ferromagnetic materials or the addition of magnetic particles during synthesis. The size of the pore diameter is influenced by the manufacturing method.

The magnetic activated carbon produced in this study was determined using the Barrett-Joyner-Halenda (BJH) method. The results of the pore diameter analysis of carbon, activated carbon and magnetic activated carbon are presented in Table 2.

Table 2 shows that the pore diameter becomes larger when the breadfruit skin carbon undergoes an activation process. This proves that the activation process can open pores on the carbon surface. However, when the activated carbon undergoes impregnation into magnetic activated carbon, the pore diameter size becomes smaller. This is due to the impregnation process of iron oxide into the pores on the surface of activated carbon so that iron oxide sticks to the surface of activated carbon pores and causes the diameter to be smaller than the diameter of the activated carbon pores. The decrease in pore diameter is 34.63%.

Table 2. Comparison of pore diameters

Carbon type	Pore diameter (Å)
Carbon	23.07
Activated carbon	28.44
Magnetic activated carbon	18.59

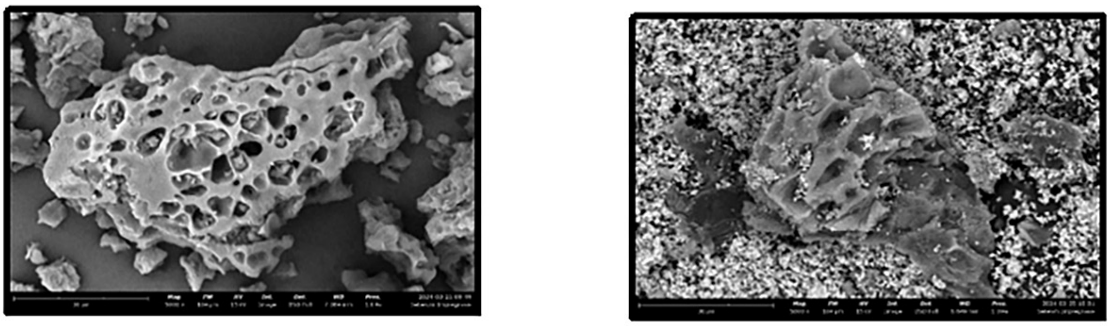
Surface morphology

Surface morphology is a physical characteristic that describes the surface structure of the material including the shape, size, and distribution of the pores present. The surface morphology of activated carbon is usually characterized by a porous and irregular structure, which can be produced through physical or chemical activation processes. In this study, the surface morphology of activated carbon and magnetic activated carbon was analyzed using SEM. SEM test is a qualitative test that aims to analyze differences in surface morphology, while EDX test is a quantitative test to determine the constituent elements of activated carbon (without magnetite) and magnetite activated carbon. The magnetite activated carbon tested is the carbon that has the best %removal results, namely the 2:3 ratio. Surface morphology analysis can prove the presence of Fe particles in carbon which indicates the success of the impregnation process in producing magnetic activated carbon. SEM analysis conducted in this study using 5000× magnification. The surface morphology of breadfruit peel activated carbon is presented in Figure 1a while the surface morphology of magnetically activated carbon is presented in Figure 1b. Figure 1a shows that the surface of the activated carbon is porous which is caused by the activation process on the breadfruit skin carbon. In the figure it also appears that the pore size on the surface of the breadfruit skin activated carbon is large. Meanwhile, Figure 1b shows the presence of iron oxide on the surface of breadfruit skin magnetic activated carbon due to the impregnation process. This phenomenon is indicated by the white colour that covers the pores of activated carbon due to iron oxide impregnation. The same phenomenon is also shown in research making magnetic activated carbon made from coconut shell, water melon and chestnut shell (Mirzaee and Sartaj, 2022; (Rashda et al., 2024; Sánchez et al., 2021).

Surface composition

The elements contained in the carbon were analyzed by EDX. The elemental contents observed in activated carbon (without magnetite) and magnetite activated carbon were C, O, Na, Si, K, Ca, Fe, and Zn which are presented in Table 3.

Based on Table 3, it can be seen that activated carbon (without magnetite) contains the three most elements, namely carbon (C), oxygen (O) and zinc (Zn) with percentages of 82.482%, 11.812% and 1.802% respectively. Meanwhile, magnetite activated carbon contains the three most elements, namely carbon (C), oxygen (O), and iron (Fe) with percentages of 51.400%, 22.200% and 16.600% respectively. The presence of carbon and oxygen elements proves that the sample is activated carbon. The carbon element contained in activated carbon is higher than magnetite activated carbon because the hydrothermal composite process reduces the carbon element. The element of iron (Fe) is also present in activated carbon (without magnetite), but with a small percentage of 1.401%, because the raw material for making activated carbon, namely breadfruit peel, contains little Fe. In magnetite activated carbon, the Fe element has increased to 16.600%,



(a) (b)

Figure 1. (a) Surface morphology of activated carbon and (b) magnetic activated carbon

Table 3. EDX analysis results of activated carbon and magnetic activated carbon

Element symbol	Element name	Weight concentration (%)	
		Activated carbon	Magnetic activated carbon
C	Carbon	82.482	51.400
O	Oxygen	11.812	22.200
Na	Sodium	0.200	0.500
Si	Silicon	0.801	1.700
K	Potassium	0.501	0.900
Ca	Calcium	1.001	5.400
Fe	Iron	1.401	16.600
Zn	Zinc	1.802	1.00

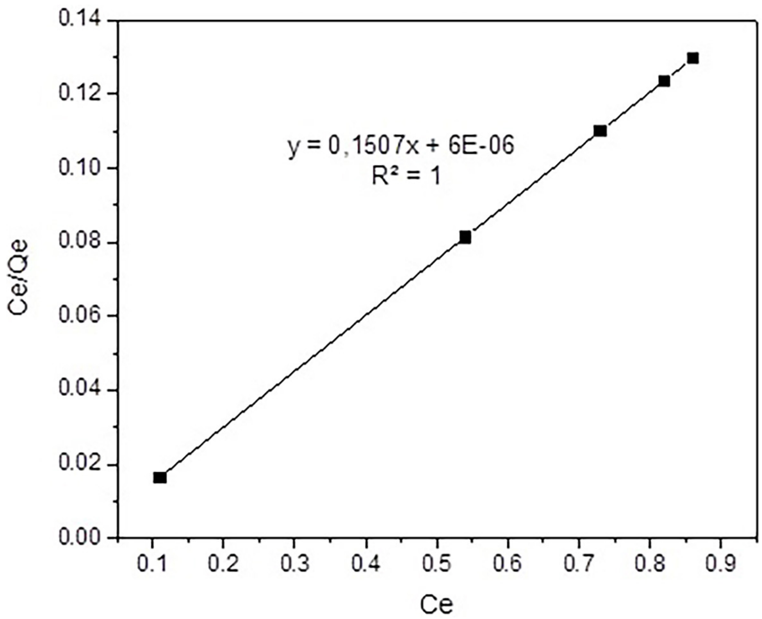


Figure 2. Langmuir isotherm model for Pb adsorption using magnetic activated carbon made from breadfruit peel

which means that with the impregnation method, Fe_3O_4 is successfully grafted and fills the pores of activated carbon. In the manufacture of magnetic activated carbon made from rice husk using the precipitation method produced Fe on the surface of 4.35% by weight (Jayalakshmi et al., 2024).

Determination of maximum adsorption capacity of magnetic activated carbon

The combination of high surface area and magnetic properties allows magnetic activated carbon to have advantages in adsorption applications. The maximum capacity value of adsorption describes the interaction between the adsorbed substance and the sorbent. The adsorption capacity of magnetic activated carbon can vary depending on several factors, including the type of activated carbon used, experimental conditions, and the nature of the substance being adsorbed. The maximum adsorption capacity can be determined through the Langmuir Isotherm equation model (Rao and Ramanaiah, 2024).

The adsorption capacity of magnetic activated carbon on Pb absorption in wastewater is calculated from the linear equation using the formula in Equation 1. By plotting Ce/Qe with Ce, the values of Qm and KL can be determined. The maximum adsorption capacity obtained based on the study is 6.6357 mg/g with a Langmuir constant (KL) of 25117 L/mg. Thus, each gram of magnetite activated carbon can adsorb a maximum of 6.6357 mg/g of Pb metal (Figure 2). Bamboo stem-based magnetic activated carbon has a maximum adsorption capacity of 22.17 mg/g on the adsorption of Bisphenol A in water (Yao et al., 2023). In the sorption of Fe(II) in water, banana peel-based magnetic activated carbon has a maximum adsorption capacity of 10.02 mg/g (Ika et al., 2023).

CONCLUSIONS

Magnetic activated carbon made from breadfruit skin has been made with iron oxide precursor using impregnation method. Based on the research that has been done, it can be concluded that the impregnation process can increase the surface area from 987.5830 m^2/g for activated carbon and 1110.804 m^2/g for magnetic activated carbon made from breadfruit skin. The surface morphology of magnetic activated carbon shows a white color due to the presence of Fe on the surface of activated carbon. The presence of Fe is also indicated by analysis using SEM EDX, which is 16.6% by weight of Fe for magnetic activated carbon while breadfruit skin activated carbon without Fe composite does not show the presence of Fe. The maximum adsorption capacity of magnetic activated carbon on Pb absorption in wastewater is 6.6357 mg/g.

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