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ADSORPTION KINETICS OF BANANA STEM ACTIVATED CARBON IN REDUCING PHOSPHATE LEVELS

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Abstract

Introduction: *High levels of phosphate in water are caused by wastewater pollution such as laundry waste water causing eutrophication. Adsorption is a method that can be used to reduce phosphate level. Banana stem that contains high levels of cellulose can be used as the main ingredient for making activated carbon. The aims of this study were to analyze the adsorption capacity and adsorption kinetics of banana stem activateld carbon in relducing phosphatel lelvells.* ***Methods:*** *The design of this research was true experiment with a pretest-posttest controlled group. Adsorption process was carried out with batch method that used three variations of adsorbate concentration and four variations of mixing time. Adsorption took place at pH 3 and 30 rpm of mixing time. Adsorption capacity was analyzed using the Langmuir isotherm and Freundlich isotherm models. Adsorption kinetic was analyzed using pseudo-first-order and pseudo-second-order kinetic models.* ***Results and Discussion:*** *The X-Ray Diffraction (XRD) difactogram and Scanning Electron Microscopy (SEM) results prove that the activateld carbon was successfully made. The iodine number of banana stelm activateld carbon was 698.12 mg/g. The results showed that activated carbon from banana stem successfully reduced phosphate levels in water with adsorption capacity 0.10708 mg/g and following the Langmuir isotherm model. The kinetics adsorption of banana stem activated carbon was validated by the pseludo-selcond-ordelr kinetics model with a kinetics constant of 0.17137 g/mg.min.* ***Conclusion:*** *The Langmuir models indicated that adsorption of phosphate occurred in monolayer. Modifications of activated carbon were needed based on characterization results.*

INTRODUCTION

One of the important nutrients for the growth of organisms is phosphate. However, phosphate can be a problem to the environment in high levels. The fertilizer manufacturing industry is a contributor that increases phosphate level in water bodies. Phosphate is produced from ammonia and phosphoric acid production activities (1). The laundry industry contains liquid waste in the form of detergent water. The composition of the constituent materials in detergent causes the laundry wastewater to have a fairly high phosphate level. Laundry wastewater in the Keputih area of Surabaya contains 14,148 mg/L of phosphate level (2). These results mean that detergent waste contains high levels of phosphate and exceeds the quality standard based on Indonesian Minister of Environment Regulation No. 5 of 2014 (2). Phosphates can be harmful to kidney (3).

The untreated domestic and industrial waste water will contribute to the increase of phosphate levels until it exceeds the maximum limit. This condition can cause water pollution such as eutrophication. Eutrophic conditions allow algae and aquatic plants to reproduce quickly so the dissolved oxygen levels in the water will decrease (4). Adsorption is one of the methods that can be used to overcome liquid waste pollution such as phosphate (5). Adsorption is a separation process in which certain components in the fluid phase move to the surface of a solid substance that can adsorb (6).

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A molecule can be adsorbed if the adhesion force between adsorbate molecules with adsorbent molecules is larger than those with cohesive forces on each molecule.

Adsorption is divided into two types, chemical adsorption and physical adsorption. Chemisorption occurs in the form of a chemical reaction that requires activation energy. The reaction that forms the chemical reaction increases the heat of adsorption. Compared to physical adsorption, adsorption time is longer and regeneration is difficult. In one way reaction, the reaction products cannot react again to form reactants. The reaction proceeds from left to right. The reactants produced cannot be recycled back to the original substance. The reaction stops only when one or all reactants are used up. Physical adsorption is reversible, occurs rapidly with little heat adsorption, interactions are thought to generate only Van der Wall force, occur in all adsorption processes, and occur at low temperatures. Dynamic equilibrium reactions can occur when the reaction taking place is a reversible reaction. Reactions are indicated by two opposite arrows. The reaction occurs in two directions, left to right and right to left, and the reaction products can be recycled to the starting materials. The reaction does not stop because the components of the substance never run out (7).

There are factors that influence the adsorption capacity of a material. The first is physical and chemical characteristics of adsorbents among surface area, pore size, and chemical adsorption. A large adsorbent surface will increase the amount of adsorbate that is adsorbed. The other factors are chemical characteristics of adsorbate including molecular size, molecular polarity, and chemical composition. Adsorbate concentration, stirring time, solution characteristics (pH and temperature), and adsorption time also influence the process of adsorption (8). The adsorption process affects the contact time because it shows the process of diffusion and the process of attachment of the adsorbate to the surface of the adsorbent. The stirring speed affects the adsorption process. A slow stirring speed will slow down the adsorption process, while a stirring speed that is too high will cause damage to the adsorbent structure so that adsorption is less than optimal.

There are several factors that influence a substance sticking to the adsorbent, which creates a pattern of certain isotherms and kinetics that become process model adsorption. Isotherm study is necessary to determine the effectiveness of adsorbent adsorption capacity (9). The equilibrium and adsorption capacity can be determined through isotherm models such as the Freundlich, Langmuir, Radlich Peterson, and Temkin (10). In the batch adsorption process, kinetic studies provide information regarding optimal condition, adsorption mechanisms, and rate control measures can be performed. Adsorption kinetics is important in determining adsorbate uptake by adsorbent and time required for completion of the adsorption process. Pseudo-first-order describes a process with a single mechanism. Pseudo-second-order describes the existence of two mechanisms that happened (9).

The adsorption process can be carried out in a batch with a stirring system, where the adsorbent which is usually in powder form is added, mixed, and stirred. The adsorbent that is usually used is activated carbon. Activated carbon is a multifunctional material where almost various types of industry use it. Activated carbon is an adsorbent with broad surface layer with a granular or powder shape. Activated carbon contains 85-95% carbon, produced from materials containing carbon which is treated in a special way to get a larger surface area. Wide activated carbon surface ranges from 300-3500 m^2/gram; this is related to the internal pore structure which causes activated carbon to have properties as an adsorbent.

Banana is one type of fruit that can live well in Indonesia. Banana production in Indonesia in 2019 was 7,280,658 tons (11). The large population and the increase of awareness of nutrition caused the increasing of fruit consumption. The stems of bananas in Peunaron District, East Aceh Regency have not been utilized optimally so they can harm humans and the ecosystem (12). Banana stem rots quickly because it contains 96% of water. Banana stems have a chemical composition in the form of cellulose amounting to 50% with potential to be used as an absorbent. Research using activated carbon from banana stems has been carried out to reduce several types of compounds in water (13). This experiment aims to analyze the capacity of adsorption and adsorption kinetics of banana stem activated carbon in decreasing the concentration of phosphate

METHODS

Production of Banana Stem Activated Carbon

The scraps of banana stem were collected, cleaned, dried, and carbonized. The drying process was carried out under the sun until it dried. The drying process was continued using an oven at 105^{0}C for an hour. Carbonization was obtained by burning the raw material at 400^{0}C for an hour. The material formed into a carbon was crushed until its particles had a size of 100 mesh. The carbon was activated by NaOH with a concentration of 2.5% for 24 hours and then cleaned using distilled water to neutralize the pH.

Characterization Test of Banana Activated Carbon

Characterization tests were carried out on banana stem activated carbon by calculating iodine number, content of water, content of ash, morphology, and structure. The water content test was performed by measuring 1 gram of banana stem activated carbon and placing it in a porcelain cup with known weight. The samples were placed in an oven at 115^{0}C for 3 hours. The content of water can be measured by the following formula:

$$\text{Water content (\%)} : \frac{W_1}{W_2} \times 100\%$$

Description:

W_1 = final sample weight
W_2 = initial sample weight

The method for analyzing the content of ash was by weighing 2 gram of banana stem activated charcoal and placing them in a porcelain cup with known weight. The samples were burned using a furnace at 800^{0}C for 2 hours. The resulting ash was cooled and then weighed. The content of ash can be calculated using the following formula:

$$\text{Ash content (\%)} : \frac{W_1}{W_2} \times 100\%$$

Description:

W_1 = residual sample weight
W_2 = initial sample weight

To test the iodine number value, 0.5 g of banana active charcoal was weighed and placed in in a beaker. Up to 25 mL of 0.125 N iodine solution was poured into the beaker glass. The solution was mixed for 15 minutes, then stored at a room temperature for 2 hours, and then filtered. A total of 10 mL of filtrate was removed and poured into an Erlenmeyer flask, and titrated with sodium thiosulfate until the color became light. Starch with 1% concentration was added to the solution until the dark blue color turned colorless. The iodine number can be calculated by using the following formula

Iodine number (mg/g) :

$$\frac{mL - V\ x\ \frac{N\ Na_2SO_4}{N\ Iodine}}{W} \times 12{,}693 \times 5$$

Description:

mL = sample volume (mL)
V = volume of titration (mL)
W = the mass of activated carbon (g)

Banana stem activated carbon is also characterized by X-Ray Diffraction (XRD) to determine the structure of active carbon and reveal the main minerals, and by Scanning Electron Microscopy (SEM) to display the morphology of activated carbon surface. The XRD test was performed with X'pert PRO PANalytical at long angle analysis and the SEM test was performed with FEI Inspect S50 at 1000x, 2500x, and 5000x magnification.

Experimental Design

The design of this study was purely experimental with a control group before and after testing. The subjects were divided into treatment groups and untreated control groups. The samples in this experiment were artificial water so it can control the phosphate concentration (adsorbate concentration) easily.

Adsorption was using batch method. The samples were made by dissolving the orthophosphate compound (KH_2PO_4) with distilled water according to the specified concentration. Samples were made in three various concentrations: 2 ppm, 4 ppm, and 6 ppm. As much as 250 mL sample on each concentration was added 4 grams of activated carbon. The sample was stirred using a shaker at 30 rpm for 3, 6, 9, and 12 hours. All research samples were conditioned on 25 ± 3^{0}C of temperature and pH 3. The control group had the same treatment with the samples group but no adsorbent added.

The results were analyzed using the Langmuir isotherm and Freundlich isotherm to determine the optimum adsorption capacity. Pseudo-first-order and pseudo-second-order kinetic models were also used to determine the adsorption kinetics

RESULTS

Characterization Test of Banana Stem Activated Carbon

Characterization tests for banana stem activated carbon include XRD, SEM, iodine number value, content of water, and content of ash. The characterization test results were analyzed in accordance with Indonesia National Standard 06-3730-1995 concerning technical activated carbon. The characterization results are shown in Table 1. Banana stem activated carbon has a water content value that is in accordance with the requirements, while its ash content and iodine number do not accord to the requirements based on Indonesia National Standard 06-3730-1995.

Table 1. Analysis of Banana Stem Activated Carbon Characterization

Parameter	Results	Standard
Content of water (%)	2.6	Max. 15
Content of ash (%)	18.9	Max. 10
Iodine number (mg/g)	698.12	Max. 750

The XRD test result at Figure 1 shows a diffraction pattern with several irregular peaks and dominant

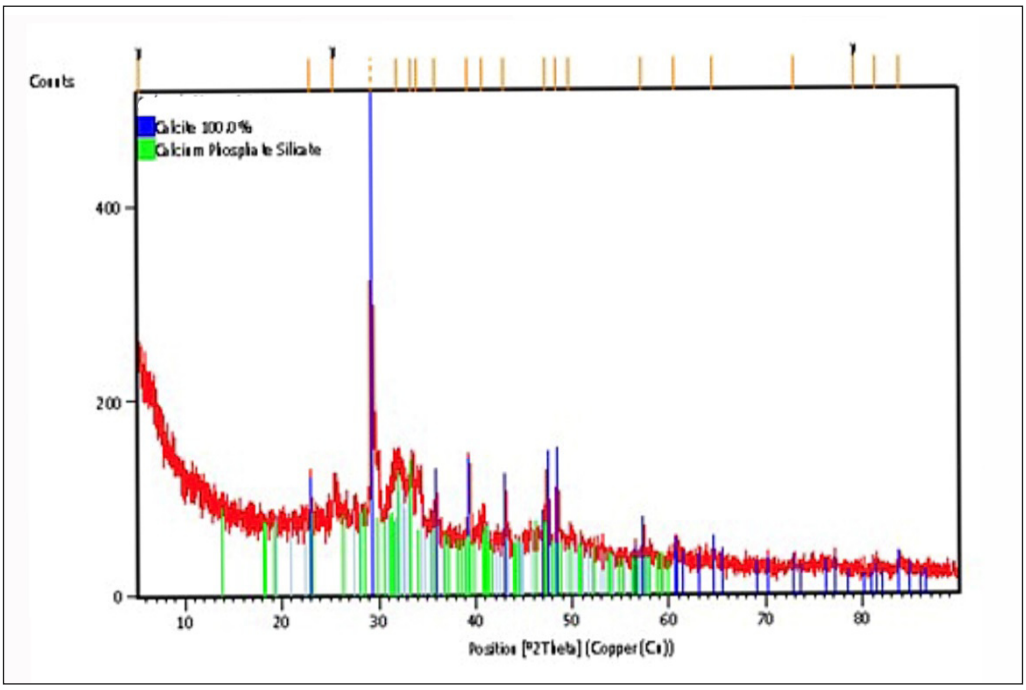

Figure 1. The Diffractogram XRD Result of Banana Stem Activated Carbon

minerals. There were two peaks that are the highest among the other peaks. The dominant minerals were Calcium Carbonate and Calcium Phosphate Silicate. The surface structure of the banana stem activated carbon was analyzed to see the pores formed. Examination of the surface structure of activated carbon was carried out using magnifications of 1000x, 2500x and 5000x. The SEM test results of banana stem activated carbon in Figure 2 show that banana stem activated carbon surface does not have many pores. Activated carbon has pores of various shape and size on its surface and contains many impurities.

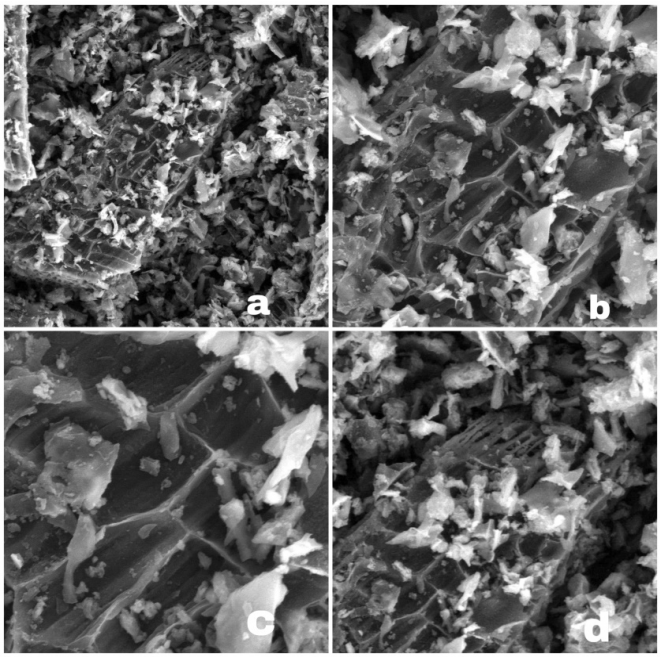

Figure 2. The SEM Analysis Results with 1000x Magnification (a), 2500 x Magnification (b), 5000x Magnification (c), and the Form of the Pores (d)

Adsorption of Phosphate

The phosphate adsorption result is shown in Table 2. The highest decrease of the concentration of phosphate using activated carbon from banana stems occurred in samples with 6 ppm of an initial phosphate concentration. As much as 2.41 ppm of phosphate concentration was adsorbed by the adsorbent in an optimum mixing time of 9 hours. The average result of phosphate reduction using activated carbon from banana stems was 1.7275 ppm. Adsorbate concentration and mixing time give different results in reducing phosphate content.

Table 2. The Result of Phosphate Adsorption

Activated Carbon	Co (ppm)	t (hours)	Ce (ppm)	The Average of Phosphate Removal (ppm)
Banana stem	2	3	1.33	0.67
		6	0.99	1.01
		9	0.97	1.03
		12	1.32	0.68
	4	3	2.10	1.90
		6	1.95	2.05
		9	1.80	2.20
		12	1.90	2.10
	6	3	3.67	2.33
		6	3.67	2.33
		9	3.59	2.41
		12	3.98	2.01

Adsorption Capacity

The adsorption capacity showed that the adsorbates accumulated on the surface of banana activated charcoal and acted as an adsorbent at equilibrium. Activated carbon from banana stems is used for the phosphate adsorption process, which reaches

equilibrium after 9 hours of mixing. The adsorption capacity data are shown in Table 3. The adsorption of banana stem activated carbon to reduce phosphate levels was determined using the Langmuir isotherm and Freundlich isotherm models. An illustration of the Langmuir and Freundlich isotherm models for banana stem activated carbon to reduce phosphate levels is shown in Figure 3. The results showed that the phosphate adsorption process using banana stem activated carbon adsorbent followed the Langmuir isotherm pattern. The adsorption capacity value can be determined based on the relationship between Ce/(x/m) and Ce in the Langmuir isotherm diagram. The optimum phosphate adsorption capacity using banana stem activated carbon was 0.1071 mg/g.

Table 3. Adsorption Capacity Data

Activated Carbon	Co (ppm)	t (hours)	Ce (ppm)	x/m (mg/g)	Ce/ (x/m)	Log Ce	Log (x/m)
Banana stem	2	9	0.97	0.0644	15.0679	-0.0132	-1.9128
	4	9	1.80	0.1375	13.0909	0.2553	-0.8617
	6	9	3.59	0.1506	23.8340	0.5551	-0.8222

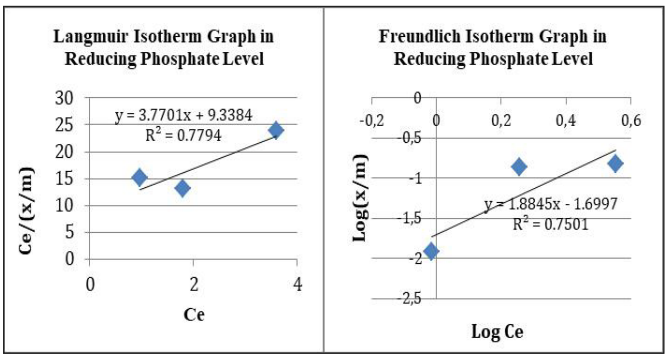

Figure 3. The Langmuir and Freundlich Isotherm in Reducing Phosphate Level

Adsorption Kinetics

Adsorption kinetics were determined by looking for the adsorption speed of activated carbon in reducing phosphate levels. The optimum concentration is 6 ppm and the variations in mixing time used are 180 minutes, 360 minutes, 540 minutes and 720 minutes. The data needed to find the adsorption kinetics value can be seen in Table 4. The adsorption kinetics model of banana stem activated carbon in reducing phosphate levels was determined using pseudo-first-order and pseudo-second-order kinetics models. The value of the rate constant can be determined based on the kinetic model equation with the regression value (R2) closest to linearity. The graph of the adsorption kinetics of activated charcoal from banana stems can be seen in Figure 4. The adsorption kinetic model of banana activated carbon has followed the pseudo-second-order kinetics models with the result of regression value R2 = 0.9761

Table 4. Adsorption Kinetic Data

Co (ppm)	t (minutes)	Ce (ppm)	Co-Ce (ppm)	Log (Qe-Qt)	t/qt
6	180	3.67	2.33	-2.3006	1236.0520
	360	3.67	2.33	-2.3006	2472.1030
	540	3.59	2.41	-5.3010	3585.0620
	720	3.98	2.02	-1.6130	5702.9700

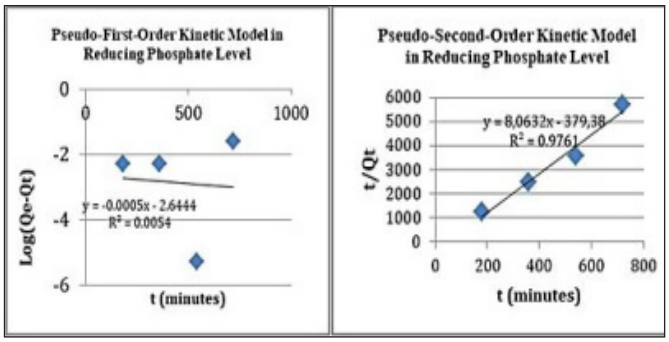

Figure 4. Adsorption Kinetic Models of Banana Stem Activated Carbon in Reducing Phosphate Level

DISCUSSION

Characterization Test of Banana Stem Activated Carbon

The water content of banana stem activated carbon is in accordance to the requirements based on Indonesia National Standard 06-3730-1995 concerning technical activated carbon. The low content of water of activated carbon is caused by the evaporation of the water content in both materials during the carbonization process. The ash content of banana stem activated carbon is not in accordance to the requirements. The high ash content in banana stem activated carbon is caused by the formation of fine mineral due to imperfect carbonization process (14).

The iodine number of banana stem activated carbon was 698.12 mg/g. According to Indonesia National Standard 06-3730-1995, the iodine number of banana stem carbon was not in accordance to the requirements. The imperfect carbonization process caused the low iodine number. The results of the iodine number were in accordance with research that activated carbon from banana stems has an iodine number value below the specified minimum limit (15).

The XRD diffractogram results of banana stem activated carbon showed the highest difference of peaks. Banana stem activated carbon has amorphous and crystalline peaks. Carbonization with temperature under 800^{0}C produced amorphous activated carbon, while at temperature of 800^{0}C-1000^{0}C it produced crystalline activated carbon. The amorphous form was characterized by curves with blunt and sloping peaks. The carbonization process affects the structural form of activated charcoal (16). SEM results showed that pores

have formed on the surface of the banana stem activated carbon. Impurities on the surface of the activated carbon were caused by the raw material not being dry enough at the time of carbonization.

Adsorption of Phosphate

Banana stem activated carbon can adsorb phosphate because of the pores on the surface. The activation process increased the pore surface area of activated carbon. The pH in low value of the solution helps the bonding process between the negative adsorbate molecules and the positive molecules of active sites on the active carbon (17).

Banana stem activated carbon was able to adsorb phosphate effectively in the 6 ppm adsorbate concentration sample and a mixing time of 9 hours, that left 3.59 ppm of phosphate concentration. This research is in line with previous research that banana stem activated carbon can reduce phosphate concentration in laundry wastewater with a reduction percentage of 80% (14).The adsorption is supported by the results of characterization tests for iodine number value, content of water, and content of ash that has quality under the requirement in accordance to Indonesia National Standard 06-3730-1995. Differences of adsorbate concentration and mixing time also provided the different phosphate adsorption results. The increase of adsorbate concentration caused the increase of adsorption capacity (18). Long mixing times also affected the adsorption result because of a greater chance of collision between the adsorbate and activated carbon (19). This research is supported by previous research which states that increasing contact time has a positive impact on reducing phosphate concentrations using adsorbent from banana stem activated carbon (14). The collision caused the adsorbate molecules to bond with the surface of the activated carbon so that the phosphate concentration can be reduced until it is in equilibrium phase. This condition is caused by the adsorbent surface being saturated due to all active sites having bound to the adsorbate molecules (20). Phosphate adsorption using activated carbon continued until the equilibrium time and after that no significant changes occurred (21)

Adsorption Capacity

The adsorption isotherm is described as the relationship between adsorbed molecules and their concentration at equilibrium at a fixed temperature. The types of adsorption isotherms that are often used to analyze adsorption isotherms are those of Langmuir and Freundlich (22). This type of adsorption can be used to study the adsorption mechanism. Phosphate adsorption by using activated carbon from banana stem followed the Langmuir isotherm which defines that the maximum adsorption capacity occured in monolayer. Subsequent adsorption only occured on the adsorbent surface which had not been bonded by adsorbate. The isotherm of the Langmuir equation is carried out using the following formula:

$$\frac{Ce}{x/m} = \frac{1}{a.b} + \frac{1}{a} Ce$$

Description:

Ce = adsorbate concentration at the equilibrum (ppm).

x/m = the mass of adsorbate that adsorbed by each gram of activated carbon (mg/g).

a = the optimum capacity of adsorption (mg/g)

Adsorption capacity is determined by calculating the amount of phosphate absorbed in each gram of activated carbon. High adsorbate concentrations caused the increase of phosphate level that was adsorbed. The adsorbate concentration can be calculated from the data in Table 3 by using the formula. The data showed that the highest capacity of adsorption occurred at the 6 ppm concentration of adsorbate with 0.15063 mg/g an adsorption capacity value. The value of phosphate adsorption also can be calculated from the linear equation in Figure 3. The linear equation was y = 3.7701x + 9.3384. From this linear equation, the optimum adsorption capacity of banana stem activated carbon in reducing the concentration of phosphate was 0.10708 mg/g.

The result was in accordance to another experiment which explained that phosphate adsorption follows the Langmuir isotherm (23). The Langmuir isotherm model defines that the optimum capacity of adsorption occurs in a monolayer (24). The Langmuir isotherm model assumes that the adsorbent surface is homogeneous and the amount of adsorption energy is equivalent for each adsorption site. The active centers on the surface of the adsorbent have the same adsorption capacity for phosphate. Adsorption continued to take place on the adsorbent surface which had not been covered by adsorbate molecules until the equilibrium was reached. The carbonization process and method determine the type of activated carbon isotherm in reducing phosphate concentration. The low iodine value also supports the results that activated carbon from banana stems followed the Langmuir isotherm in phosphate adsorption.

Adsorption Kinetics

Adsorption kinetics can be used to show the

value of adsorption that occurred on adsorbent against adsorbate and affected by time. The adsorption of phosphate by banana stem activated charcoal followed a pseudo-second-order adsorption kinetics model. The pseudo-second-order adsorption kinetics model was carried out using the following formula:

$$\frac{t}{qt} = \frac{t}{qe} + \frac{1}{k.qe^2}$$

Description:

qe = adsorption capacity at the equilibrium
qt = adsorption capacity at the time t
t = contact time
k = kinetic constant

The decision is based on the regression value (R2) that is closest to linearity. Pseudo-second-order kinetic model defines that the adsorption rate depends on the adsorption ability of each solid phase by the adsorbent. This also follows a pseudo-second-order kinetics model (25). The adsorption capacity is believed to be directly proportional to the number of active sites on the adsorbent.

The pseudo-second-order linear equation for banana stem active carbon in adsorbing phosphate was y = 8.0632x – 379.38. The kinetic constant value of banana stem activated carbon in adsorbing phosphate was 0.17137 g/mg.min. The results of this research are also related with research which explained that phosphate reduction using active alkaline and zeolite follows a pseudo-second-order kinetic model (26).

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CONCLUSION

Banana stems can be applied as an adsorbent for phosphate adsorption in water. Several parameters of characterization test were not in accordance with the requirements based on Indonesia National Standard 06-3730-1995.There were differences in the results of phosphate reduction using various adsorbate concentrations and mixing times. The phosphate concentration was successfully reduced optimally in the sample which had an adsorbate concentration of 6 ppm and a mixing time for 9 hours. Phosphate absorbed on the adsorbent will increase along the increasing adsorbate concentration and mixing time until the equilibrium. The adsorption using banana stem activated carbon followed the isotherm model of Langmuir with an optimum adsorption capacity of 0.10708 mg/g. The Langmuir isotherm defines that the adsorption process only occurs in a monolayer. The phosphate adsorption kinetics followed a pseudo-second-order kinetic model with a kinetic constant value of 0.17137 g/mg.min.

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