KINETIC ANALYSIS OF PHOSPHATE ADSORPTION ON ZEOLITE AND ACTIVATED CARBON IN GREYWATER

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Abstrak

Phosphate is one of the pollutants that exist in greywater. Phosphate pollutant comes from surfactants used in detergent products which are commonly used in household activities. Water phosphate will increase when detergent waste is not managed properly. The presence of phosphate in waters can result in eutrophication which disrupts aquatic life. Some natural adsorbents such as zeolite and activated carbon can adsorb phosphate in greywater. The effectiveness of an adsorbent in removing phosphate in greywater can be analyzed based on the adsorption capacity and adsorption rate. In this research, it is known that zeolite is more effective as an adsorbent in removing phosphate content in greywater. Zeolite and activated carbon follow the Freundilch adsorption mechanism in the adsorption of phosphate in water. Zeolite can adsorb 41.154% with a contact time of 40 minutes, with a Freundilch constant (Kf) = 8.10 L g^{-1} and an adsorption rate of K = 0.0038 m^{-1}. For activated carbon it can adsorb 23.330 % with a contact time of 40 minutes, with a Freundilch constant (Kf) = 1.99 L g^{-1} and an adsorption rate of K = 0.0013 m^{-1}. Based on these data, activated carbon has a faster phosphate adsorption rate than zeolite, but has a smaller adsorption capacity value than zeolite. So that the zeolite can absorb more phosphate in greywater, with an adsorption rate of 0.0038 per minute.

Kata kunci*: adsorption, zeolite, carbon, phosphate, greywater*

1. INTRODUCTION

The natural greywater treatment technique, through filtration with adsorbent media, is the most environmentally friendly and cost-effective technology for the treatment and reuse of greywater. Natural greywater treatment systems use natural media for filtration and biological degradation (Elmagd, 2019). Reuse of greywater is possible, as greywater is far less polluted than domestic wastewater. In wastewater, greywater contains about 30% of the total organic load and 10-20% of the nutrients present in domestic wastewater. Therefore, it is important to characterize the contaminants present in the greywater to determine the total removal required and to select the appropriate treatment course.

Treated greywater is generally used for non-potable purposes such as flushing toilets, irrigation, and washing cars. The reuse of treated greywater not only conserves scarce water resources but also reduces water supply costs and reduces the burden on centralized wastewater treatment systems. This can help minimize the negative impacts and costs of water extraction and wastewater treatment, thereby helping to achieve water sustainability (Shaikh & Ahammed, 2020).

To reduce pollutants in greywater is to use adsorbents. Commonly used adsorbents are zeolite and activated carbon. With the addition of 6 grams of zeolite adsorbent and activated carbon, it can reduce the value of pH, COD, and TSS in the water treatment process (Kharismawat et al., 2018). Zeolite is capable increase the quality of greywater with its decline mark of 21.8% COD, 11.5% pH, 9.2% BOD, and 10.8% TSS (Suhartana & Pardoyo, 2020). One of the pollutants in greywater is phosphate levels. Phosphate in greywater generally comes from Sodium Tripoly Phosphate (STPP), which is one of the ingredients in detergents. In addition, various types of surfactants are used as active ingredients to remove stains or dirt. Surfactants can be anionic (Alpha Olein Sulfonate /AOS, Linear Alkyl Benzene Sulfonate /LAS, or Alkyl Benzene /ABS) a major contributor to increasing water phosphate levels. The presence of phosphate levels in waters can cause eutrophication which disrupts aquatic life (Prihatin & Sugiharto, 2021).

Removal of phosphate levels in water can be done by the adsorption process. Activated carbon adsorbents are known to reduce phosphate levels using 4 grams of adsorbent mass and 100 rpm stirring. Activated carbon activated using an acid solution can adsorb 0.26 mg/g of phosphate compounds. while activated carbon which was activated using an alkaline solution had an adsorption capacity of 0.49 mg/g. Activated carbon activated with an acid solution is known to have a hollow surface dominated by C (55.20%), O (28.86%), and N (8.00%) elements (Illah, S. et al., 2020; Perdani et al., 2021). In addition to activated carbon, zeolite adsorbents are also able to reduce phosphate in greywater with an adsorption rate of 0.1154 g/mg min (Syafaat et al., 2013). So it is necessary to study the adsorption mechanism and adsorption kinetics that occur in phosphate removal using zeolite and activated carbon adsorbents.

Adsorption kinetics is a model used to determine the process speed and stages in an adsorption medium. Adsorption kinetics data can be in the form of adsorption capacity values which show the relationship between adsorbate concentration and time in equilibrium conditions (Syafaat et al., 2013). Using this method, the effectiveness of the adsorbent in removing phosphate in greywater can be determined based on the calculated adsorption rate. So that in this study will be analyzed the kinetics of the adsorption rate in the removal of phosphate content in greywater.

Adsorption capacity is the ability of the adsorbent to absorb a certain amount of adsorbate in the liquid phase. Adsorption capacity is used to determine the adsorptive characteristics of an adsorbent. A solution concentration data resulting from analysis can be calculated as the adsorption capacity (q), using equation 1. The maximum adsorption capacity value will be obtained by using the Langmuir adsorption isotherm equation (equation 2) and the Freundlich adsorption isotherm equation (equation 3). Through this equation, it will be known the adsorptive properties of an adsorbent on the adsorbate. The Freundlich isotherm model describes adsorption interactions that occur due to physical adsorption. The Langmuir isotherm model can explain the presence of a homogeneous adsorbent surface (Huda & Yulitaningtyas, 2018).

$$q = \frac{(C_0 - C_1)\ X\ (\frac{V}{1000})}{m} \qquad (1)$$

Where Co, Ce are the initial total solids and final total solids in the greywater sample (ppm); V is the volume of the greywater sample used (mL); m is the mass of the adsorbent used in the experiment.

$$q = \frac{(C_0 - C_1)\ X\ (\frac{V}{1000})}{m} \qquad (2)$$

$$q = \frac{(C_0 - C_1)\ X\ (\frac{V}{1000})}{m} \qquad (3)$$

Where q is the adsorption capacity balance; qmax is the maximum adsorption capacity; KL is the Langmuir constant; K f is Freundlich's constant; n is the heterogeneity factor (Ungureanu et al., 2020).

Adsorption kinetics states that there is a process of absorption of a substance by the adsorbent as a function of time. The characteristics of the adsorption ability of the adsorbent on the adsorbate can be seen from the adsorption rate. The adsorption rate can be known from the adsorption rate constant (k) and the order of the reaction resulting from an adsorption kinetics model. The first-order kinetic model and the second-order kinetic model are expressed by the equation:

$$q = \frac{(C_0 - C_1)\ X\ (\frac{V}{1000})}{m} \qquad (4)$$

$$q = \frac{(C_0 - C_1)\ X\ (\frac{V}{1000})}{m} \qquad (5)$$

The reaction kinetics model order 1 and order 2 for each parameter is calculated using the graph ln Ce to t and (1/ Ce) to t. The model that fits the research results is the kinetic model with the highest R^2 value (Meila Anggriani et al., 2021).

Kinetic analysis is based on first-order kinetics and second-order kinetics. First-order kinetics is a reaction whose speed depends on one of the reacting substances or is proportional to one of the powers of the reactants. A reaction kinetics model is also needed to predict the rate of transfer of the adsorbate from the solution to the designed adsorbent. The first-order reaction kinetics model is an adsorption reaction kinetics model that explains the entrapment of molecules from the liquid phase to the solid phase. The reaction process of the 1st order is a reversible reaction in which the equilibrium is established from the solution phase (liquid) and the solid phase. The mechanism of adsorption of metal ions on the adsorbent is a non-dissociation molecular entrapment which is described as diffusion control. Meanwhile, the kinetics of the 2nd order adsorption reaction assumes that the adsorption capacity is adjusted to the number of active sites on the surface of the adsorbent. So the kinetics of Order 2 depends on the ability of each adsorbent (Wahidatun et al., 2015).

2. METHODS

This study uses a set of adsorption columns with a continuous system (Figure 1). Each adsorption column was filled with zeolite adsorbent and activated carbon adsorbent with a height of 15 cm. The greywater sample used is a laundry waste sample located in the Rajabasa area, Bandar Lampung. The greywater sample is then flowed into the adsorption column which already contains the adsorbent (zeolite or activated carbon). Greywater samples flowed into the adsorption column with a flow rate of 200 ml/s. Greywater samples flowed for 40 minutes and test samples were taken every 10 minutes for phosphate analysis. The contact time of the sample and the adsorbent for 40 minutes was used to determine the adsorption kinetics of each adsorbent. Furthermore, the test sample obtained was analyzed for its phosphate content.

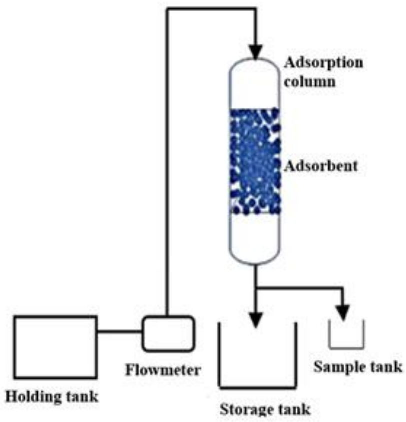


Figure 1. Adsorption Column Design of Continue System

Analysis of phosphate levels in test samples using a UV-Vis spectrophotometer using ascorbic acid in water and wastewater samples according to SNI 06-6989.31-2005. Making a standard curve, make a working phosphate solution by taking 0 mL; 5mL; 10mL; 20 mL and 25 mL of anhydrous dihydrogen phosphate standard solution (KH2PO4) which has a concentration of 10 ppm in 250 mL then add distilled water until it is right at the tera mark. To obtain a phosphate level of 0.0 mg P/L; 0.2mg P/L; 0.4 mg P/L; 0.8 mg P/L and 1.0 mg P/L.

Then prepare a mixed solution consisting of 50 mL of H2SO4 5 N, 5 mL of potassium antimonyl tartrate solution (1.3715 g in 500 ml of distilled water), 15 mL of ammonium molybdate solution (20 g in 500 mL of distilled water and 30 mL of ascorbate solution 0.1 M). To 50 mL of the working solution, 1 drop of phenolphthalein indicator is added. If a pink color is formed, add drop by drop H2SO4 5N until the color disappears. Then add 8 mL of the mixed solution and react for 20 minutes. The solution that has finished reacting is marked with a blue solution change, then it is measured using a spectrophotometer with a wavelength of 898 nm. The same thing is done for the other working solutions and the greywater test sample. The phosphate level in the test sample is obtained from the equation of the calibration curve of the working solution obtained.

The results of the analysis of phosphate levels in the test samples were used to determine the kinetics of phosphate adsorption from zeolite adsorbents and activated carbon adsorbents. By using Equation 2 and Equation 3, the adsorption mechanism that occurs in each adsorbent will be obtained. While the adsorption rate of each adsorbent on phosphate will be obtained using Equation 4 and Equation 5. So that it can be determined which adsorbent is more effective in carrying out phosphate removal in greywater.

3. RESULT AND DISCUSSION

Adsorption is one form of an absorption process. There are two important components in the adsorption process, namely the mobile phase (adsorbate) and adsorbent (adsorbent). The adsorption process begins with a diffusion process through the macropores and a mass transfer process that occurs on the active surface of the adsorbent. Furthermore, adsorbate molecules are adsorbed through small pores (micropores) and are retained in them (Bimantio & Ferhat, 2022). This research identified the ability of zeolite and activated carbon adsorbents in reducing phosphate content in greywater. The adsorbent is arranged in an adsorption column with a height of 30 cm (Figure 1). Each adsorbent was then analyzed for its adsorption capacity on the phosphate present in the greywater. The results of the phosphate adsorption process using zeolite and activated carbon used the Uv-Vis spectrophotometer test method as shown in Table 1.

Tabel 1. Results of the phosphate adsorption process in *greywater*

Type adsorbent	Time (minute)	Concentration Phosphate (mg/L)	efficiency Absorption (%)
Zeolite	0	4,229	0
	10	3,567	15,646
	20	2,971	29,735
	30	2,879	31,910
	40	2,489	41.154
Activated carbon	0	3,920	0
	10	3,455	11,865
	20	3,377	13,865
	30	3,330	15,065
	40	3,006	23,330

Based on Table 1, the zeolite adsorbent is more effective in absorbing the phosphate content present in greywater waste. With the same contact time, zeolite can adsorb more phosphate than activated carbon, which is equal to 41.154%. Zeolite can absorb more dissolved adsorbate than activated carbon because it has smaller pores so it can absorb polar molecules such as phosphate in greywater. Activated carbon has many micropores and high reactivity.

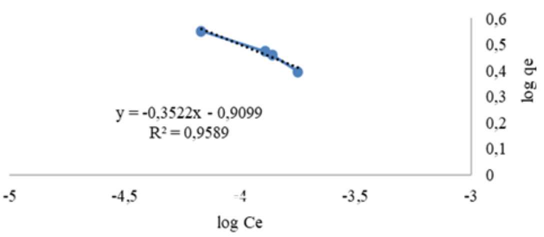
Whereas zeolite has a surface full of carbon groups so that the hydrophilic nature of zeolite is reduced and it is effective for adsorbing other polar molecules (Apriyani & Novrianti, 2020; Asyipa et al., 2021). Zeolites have been known as adsorbents in water treatment, especially for removing cations. Zeolite can achieve a higher removal effect because it is a porous material with a large specific surface area. Salt-modified zeolite is known to be able to remove phosphorus in wastewater with an adsorption capacity of 0.3 mg/g (Shi et al., 2017).

Zeolite and activated carbon are carbon-based microporous adsorbents. Zeolite is an aluminosilicate crystal that has a complex three-dimensional framework. The structure of the zeolite consists of tetrahedrally coordinated Si, Al, or P. The framework has a highly ordered network of micropores resulting in a large surface area and capacity for adsorption uniformly throughout the crystal structure. This microporosity is a characteristic of the adsorption capacity of each zeolite in the addition of phosphate in wastewater (Lin et al., 2019; Merilaita et al., 2021). Meanwhile, activated carbon will form a multilayer layer, where the active side of the carbon has different adsorption energy. This causes the active side of the carbon which has the greatest adsorption energy to be filled first with other pollutants (besides phosphate) contained in the greywater. (Aprian et al., 2021). So activated carbon is less effective in removing phosphate in greywater than zeolite.

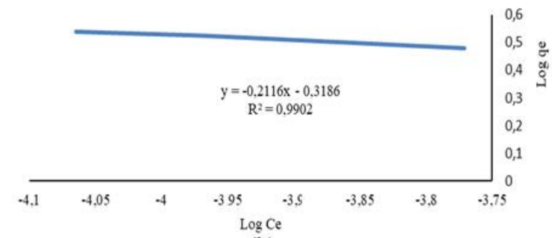
3.1. Phosphate Adsorption Isotherms In Greywater

Adsorption isotherms can be used to determine the adsorption mechanism that occurs on the adsorbate molecule and the adsorbent surface. By using the Langmuir and Freundlich adsorption isotherm equations, it can be seen that the mechanism that occurs between phosphate and adsorbent, is both physisorption and chemisorption. Physical adsorption is physical adsorption, where there is a van der Waals attraction on the surface, resulting in a back-and-forth, multilayer reaction that does not involve electron transfer. Chemisorption is adsorption that occurs due to the binding force on the surface which involves the transfer of electrons between the adsorbate and the adsorbent. In chemisorption, the reaction cannot go back and forth and is monolayer (Zakaria & Djasmasari, 2017).

Based on the data in Table 1, the Langmuir and Freundlich adsorption equation model is obtained through calculation. Adsorption equation models Freundlich for reducing phosphate using zeolite and activated carbon is shown in Figure 1. The Langmuir adsorption equation model for phosphate reduction using zeolite and activated carbon is shown in Figure 2.

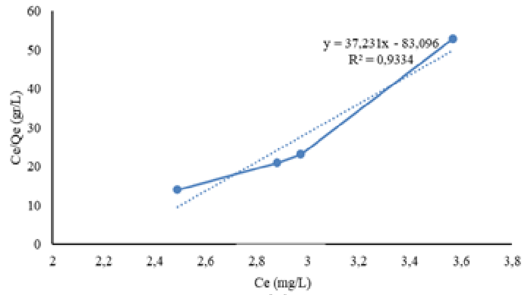


(a)



(b)

Figure 1. Freundlich isotherm of phosphate adsorption using a) zeolite; b) activated carbon



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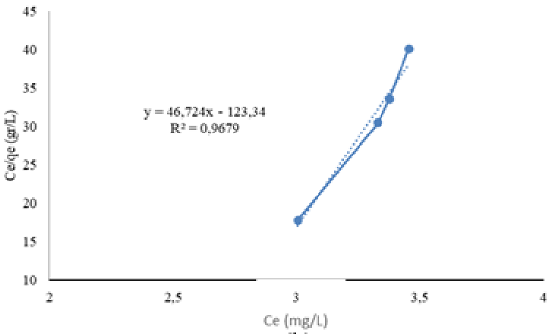

(b)

Figure 2. Langmuir isotherm of phosphate adsorption using a) zeolite; b) activated carbon

Table 2. Parameter analysis of phosphate adsorption isotherms in greywater

***Isotherm* models**	**Parameter**	**Zeolite**	**Activated Carbon**
Langmuir model	R^2	0.9334	0.9679
	q_{max} ,mg g^{-1}	1.2 x 10^{-2}	0.8 x 10^{-2}
	K_L , L g^{-1}	2,232	2,639
Freundlich model	R^2	0.9589	0.9902
	n	2,8	4,7
	K_f , L g^{-1}	8,10	1.99

Based on the results of the linearization of experimental data, phosphate adsorption on zeolite and activated carbon follows the Freundlich adsorption isotherm mechanism. The correlation coefficient (R^2) in the Freundlich isotherm model is higher than in the Langmuir isotherm model. This shows that the Freundlich isotherm model with an R^2 value that is closer to 1 is the best description of the experimental data related to the adsorption mechanism that occurs in the phosphate adsorption process. The mechanism of phosphate adsorption on zeolite and activated carbon takes place in multilayer physisorption. This mechanism indicates the occurrence of bonds between metal ions and adsorbents which are only bound by van der Waals forces (Yenti et al., 2018). This causes the adsorption process to take place on several heterogeneous layers of the adsorbent surface.

In the adsorption mechanism using the Freundlich isotherm model, the surface of the adsorbent is heterogeneous because there is an active site that is only able to adsorb dissolved molecules with different energy levels (Wijayanti & Kurniawati, 2019). In Table 2, the phosphate adsorption process on zeolite has a higher Kf value than the phosphate adsorption process on activated carbon. The value of Kf in the adsorption process of phosphate on zeolite is 8.10 L g-1. This shows that more phosphate can be adsorbed in the zeolite pores than in the activated carbon pores, because the higher the value of Kf, the greater the adsorption capacity that occurs on the active site of the adsorbent.

Although the Langmuir isotherm model does not adequately describe the experimental data obtained on phosphate adsorption, it can explain that there is more than one type of adsorption site available on the adsorbent surface. The existence of a cooperative adsorption system in the Langmuir adsorption model, explains the existence of binding on identical surfaces but can accommodate many molecules. So more adsorption energy is needed which is affected by the presence of various adsorbates bound to the active site of the adsorbent (Al-Ghouti & Da'ana, 2020). Based on the parameter analysis of phosphate adsorption isotherm in greywater, the adsorbent has a higher adsorption capacity value, namely q max = 1.2 x 10 -2 mg g-1. This is a factor causing zeolite to have a greater efficiency value for phosphate absorption than activated carbon.

3.2. Kinetics of Phosphate Adsorption in Greywater

The kinetics of phosphate adsorption in greywater describes the rate of phosphate binding to changes in contact time on the adsorbent. The value of adsorption kinetics can be used to define the efficiency of the adsorption process. Where in this process the kinetics of phosphate adsorption will be influenced by the adsorption factor and the mass transfer stages that occur in the transfer of phosphate ions in solution towards the active side found on the surface of the adsorbent. The kinetic value is also needed in predicting the rate of transfer of the adsorbent from the solution to the adsorbent (Wahidatun et al., 2015).

The kinetics of phosphate adsorption using zeolite and activated carbon can be identified by graphing the experimental data of phosphate adsorption on zeolite and activated carbon. The kinetics of order 1 and the order kinetics of phosphate adsorption can be seen in Figure 3 and Figure 4.

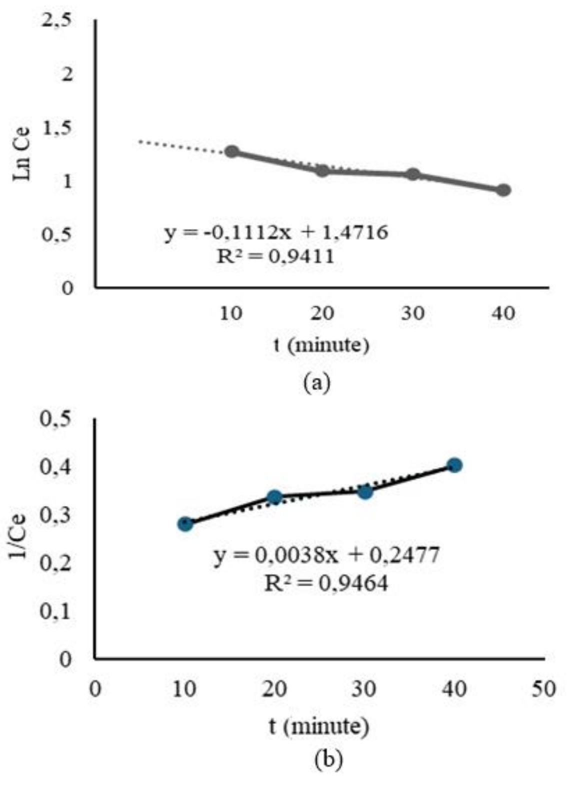

Figure 3. a) first order b) second order kinetics adsorption phosphate using zeolite

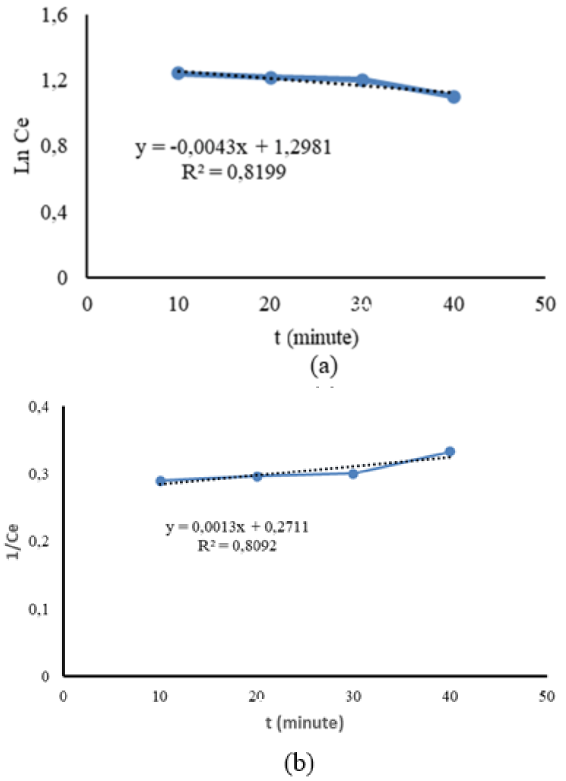

Figure 4. a) first order b) second order kinetics adsorption phosphate using carbon active

Table 3. Parameters of Phosphate Adsorption Kinetics

adsorbent	Reaction order	K (minutes) $^{-1}$	R^2
Zeolite	1	0.1112	0.9411
	2	0.0038	0.9464
Activated Carbon	1	0.0043	0.8199
	2	0.0013	0.8092

Based on the adsorption parameter data in Table 3, phosphate adsorption using zeolite follows a second-order adsorption kinetics model, with a correlation coefficient value (R^2) that is closest to 1. For phosphate adsorption using activated carbon, it tends to follow a 1st order adsorption kinetics model, but the R-value = 0.8199. So it is not enough to describe the kinetic processes that occur in phosphate adsorption with activated carbon adsorbents. In contrast to the R^2 value in the zeolite which follows the second order adsorption kinetics model, namely R^2 = 0.9464 with a K value = 0.0038 m^{-1}. The K value defines the speed at which the adsorption process takes place. The smaller the K value, the faster the adsorption process takes place.

Based on Table 3, activated carbon has the smallest K value, but the coefficient of determination is only 81.99%, which means that the correlation between time and concentration is still influenced by other factors by 20%. In contrast, zeolite has a coefficient of determination of 94.64%, which means there is a correlation between time and concentration so that it can describe the kinetic processes that occur accurately.

4. CONCLUSION

Based on research on phosphate adsorption in greywater, it is known that zeolite is more effective in the adsorption process of phosphate contained in greywater than activated carbon. Zeolite can adsorb 41.154% phosphate in contact time for 40 minutes. The phosphate adsorption process using zeolite follows the Freundlich adsorption isotherm mechanism model, where there is a heterogeneous active site on the surface of the adsorbent.

The Kf value in the phosphate adsorption process on the zeolite was 8.10 L g^{-1} with the adsorption rate on phosphate being K = 0.0038 m^{-1}. Activated carbon can adsorb 23.33% of phosphate with a contact time of 40 minutes. The phosphate adsorption process using activated carbon follows the Freundlich adsorption isotherm mechanism model, with a value of Kf = 1.99 and the adsorption rate of phosphate is K = 0.0013 m^{-1}. The value of Kf on activated carbon is lower than the Kf value of zeolite, but activated carbon has a higher rate of adsorption on phosphate than zeolite.

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