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Kinetics and Isotherm Study of Ion Phosphate Adsorption by Lontor Natural Clay

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Abstract

The clay used taken from Lontor Village, Central Maluku Regency, Banda Besar Island. The clay was activated at 350 °C for 3 hours using a furnace. This study aims to determine the effectiveness of physically activated Lontor natural clay in reducing levels of phosphate ions using the adsorption method. The adsorption capacity of clay is determined based on two parameters. The parameters are the optimum contact time used are 2, 3, 4, 5, and 6 hours and variations of the concentration are 10, 20, 30, 40, and 50 mg/L of phosphate. The concentration of phosphate adsorbed on the clay was measured by a UV-Vis spectrophotometer at a wavelength of 665 nm. The results showed that the optimum conditions for the adsorption of phosphate ions by Lontor natural clay were obtained at the contact time of 5 hours and the concentration of 40 mg/L. The adsorption of phosphate ions by Lontor activated natural clay followed the pseudo-second-order Lagergren adsorption kinetics model with a rate constant value (k_2) of 3.39 x 10^{-1} g mg^{-1} min^{-1}, and following the Freundlich adsorption isotherm models with a correlation coefficient (R^2), Freundlich constant (K_f), and n values, respectively are 0.9513, 0.57 $mg^{1-1/n}$ $L^{1/n}$/g, and 0.40 g/L.

Keywords: Adsorption, phosphate ion, isotherm, kinetics, Lontor natural clay

INTRODUCTION

Indonesia has many potential natural resources that have not been utilized optimally, one of which is clay. Clay is a material consisting of excellent particles measuring < 2 m (Wijaya et al., 2004). Maluku has several places with natural clay-producing areas, namely Latuhalat (Latupeirissa & Fransina, 2014), Ouw (Bijang et al., 2021), and Lontor**.** Lontor Village located in Banda Besar Island, Central Maluku Regency. Lontor natural clay is generally only used as raw material for ceramics and brick making.

Besides being used as a raw material for making bricks and clay ceramics, it can also be used in industry as an adsorbent, catalyst, and resin for ion exchange (Wijaya et al., 2002). Clay is widely used as an adsorbent because it has a high specific surface area, high porosity, abundance in nature, and a relatively lower price than other adsorbents (Waghmare et al., 2015).

Phosphate compounds are one of the parameters used to determine the presence of water pollution. Excessive phosphate in the environment can cause eutrophication (Auliah, 2009). Eutrophication is an environmental problem caused by excessive phosphate waste (Sinta et al., 2015). Phosphate compounds in the water impact the extensive use of phosphate compounds caused by human activities or geochemical processes and can also come from industrial waste (Pohan et al., 2016).

The adsorption method is a method that can absorb a substance (ion or molecule) on the surface of the adsorbent. Several factors affect the adsorption process: surface area, concentration, particle size, temperature, pH, and contact time. Adsorption methods are generally based on the interaction between a substance or metal with functional groups in the adsorbent, namely through the formation of complexes and or cation exchange (Bath et al., 2012). This method is effective considering that the process is easy, the time required is relatively short, does not cause side effects to human health and the environment, and is economical because it does not require a lot of money (Lesbani et al., 2015).

Previous research by Darmadinata et al. (2019) reviewed the potential applications of clay minerals. It concluded that clays exhibit enormous potential for phosphate adsorption due to their large specific surface area, chemical and mechanical stability, layered structure, and high cation exchange capacity. In addition, Auliah (2009) showed the adsorption of phosphate ions in water by activated clays based on the Langmuir isotherm with R^2 = 0.861. Furthermore, research by Maslukah et al. (2020) also showed the kinetics of adsorption and desorption of phosphate ion (PO_4^{2-}) in sediments of Semarang and Jepara waters based on the pseudo-second-order kinetics model and followed the Langmuir adsorption isotherm.

METHODOLOGY

Instrumentals

The tools that have been used in this research are set of tools glass (Pyrex), Analytical balance (Denver Instrument XP-3000), centrifuge (Hettich EBA 20), Oven (Shel Lab), Desiccators, Tanur (VULCAN A-550), Shaker (GFL 3005), Stopwatch, Lumpang and pestle, 100sieve *mesh,* Porcelain dish, Petri dish, Funnel, Dropper pipette, Volume pipette, Stirring rod, FTIR Spectrophotometer (Prestige 21Shimazu), and UV-Vis Spectrophotometer (APEL PD-3000UV).

Materials

The materials that have been used in this research are Lontor natural clay, K_2HPO_4 (pa Merck), Ammonium molybdate (pa Merck), $SnCl_2$ (pa Merck), bi-distilled, distilled water, Whatman filter paper No. 42, and Aluminum foil.

Methods

Sample Preparation

Lontor natural clay samples were soaked, and the top clay fraction was removed, then washed with distilled water until clean. The results obtained were filtered with a vacuum pump to obtain clay free from impurities. Furthermore, clay is dried in an oven at a temperature of 80 ^oC for 6 hours. The dry clay was ground and sieved using a 100 sieve *mesh* and stored in a desiccator.

Physical Activation of Lontor Natural Clay

The prepared Lontor natural clay sample was put into a porcelain dish and then heated using a furnace (physical activation) at 350 °C for 3 hours. Furthermore, the physically activated Lontor natural clay was stored in a desiccator.

Preparation of Phosphate Solution

Preparation of 1000 ppm Phosphate main liquor

A total of 1.83 g of K_2HPO_4 was weighed and dissolved using bi-distilled water. After that, it was put into a 1000 mL volumetric flask and bi-distilled to the exact volume, then homogenized.

Preparation of 100 ppm Standard Phosphate Solution

A total of 10 mL of phosphate mother liquor was diluted with distilled water in a 100 mL volumetric flask to the exact volume.

Preparation of standard phosphate solution for determination of optimum contact time

Phosphate standard solutions of 5, 10, 15, 20, 25, and 30 ppm were prepared by taking respectively 5, 10, 15, 20, 25, and 30 mL of 100 ppm standard phosphate solution then diluted with bi-distilled in a 100 mL volumetric flask to mark the limit.

Preparation of standard phosphate solutions for determining the optimum concentration

Phosphate standard solutions of 5, 10, 15, 20, 25, 30, 35, 40, 45, and 50 ppm were prepared by taking respectively 5, 10, 15, 20, 25, 30, 35, 40, 45, and 50 mL of 100 ppm standard phosphate solution then diluted with bi-distilled in a 100 mL volumetric flask to mark the limit.

Determining the Optimum Contact Time

A total of 1 g of clay was put into a 100 mL Erlenmeyer glass containing 50 mL of phosphate solution with a concentration of 30 ppm. The suspension was shaken using a shaker at a speed of 200 rpm with various contact times of 2, 3, 4, 5, and 6 hours at room temperature. The suspension was filtered using Whatman filter paper No. 42, then washed and put into an Erlenmeyer. The filtrate was then centrifuged at 2000 rpm for 15 minutes. After that, 1 mL of ammonium molybdate and 0.4 mL of $SnCl_2$ added to the filtrate were homogenized to form a blue solution. The absorbance of the solution was measured using a UV-Vis spectrophotometer at a wavelength of 665 nm (Ngibad, 2019)

Determining the Optimum Concentration

A total of 50 mL of phosphate solution with 10, 20, 30, 40, and 50 ppm concentrations were placed into the clay sample. Each added 1 g of clay sample. The suspension was shaken using a shaker at 200 rpm for the optimum time at room temperature. The rest was filtered using Whatman filter paper No. 42, then washed and put into an Erlenmeyer. The filtrate was then centrifuged at 2000 rpm for 15 minutes. After that, 1 mL of ammonium molybdate and 0.4 mL of $SnCl_2$ were added to the filtrate then homogenized to form a blue solution. Furthermore, the absorbance of the filtrate was measured using a UV-Vis spectrophotometer at a wavelength of 665 nm (Ngibad, 2019).

Phosphate Ion Analysis with the spectrophotometer UV-Vis

A total of 50 ml of the filtrate of each suspension was put into a 100 mL Erlenmeyer. After that, 1 mL of ammonium molybdate was added to the filtrate and homogenized, then 0.4 mL of $SnCl_2$ was added to form a blue solution. Next, the solution was measured absorbance with a UV-Vis spectrophotometer at a wavelength of 665 nm (Ngibad, 2019). A graph was made between concentration and absorbance. The chart was plotted on the standard solution curve.

Determining the Adsorption Kinetics

A total of 1 g of clay was put into a 100 mL Erlenmeyer glass containing 50 mL of phosphate solution with a concentration of 30 ppm. The suspension was shaken using a shaker at a speed of 200 rpm with various contact times of 120, 180, 240, 300, and 360 minutes at room temperature. The suspension was filtered using Whatman filter paper No. 42, then washed and put into an Erlenmeyer. The filtrate was then centrifuged at 2000 rpm for 15 minutes. After that, 1 mL of ammonium molybdate and 0.4 mL of $SnCl_2$ were added to the filtrate, then homogenized to form a blue solution. Then the absorbance of the solution was measured using a UV-Vis spectrophotometer at a wavelength of 665 nm (Ngibad, 2019).

Determining the Adsorption Isotherms

A total of 50 mL of phosphate solution with 10, 20, 30, 40, and 50 ppm concentrations are added to 1 g of clay sample each. The suspension was shaken using a shaker at 200 rpm for the optimum time at room temperature. The rest was filtered using Whatman filter paper No. 42, then washed and put into an Erlenmeyer. The filtrate was then centrifuged at 2000 rpm for 15 minutes. After that, 1 mL of ammonium molybdate and 0.4 mL of $SnCl_2$ added to the filtrate were homogenized to form a blue solution. Furthermore, the absorbance of the filtrate was measured using a UV-Vis spectrophotometer at a wavelength of 665 nm (Ngibad, 2019)

Determining the Concentration of Phosphate Adsorbed in Clay

The adsorbed phosphate concentration was determined by measuring the remaining phosphate concentration using a UV-Vis spectrophotometer at a wavelength of 665 nm. The amount of phosphate adsorbed is determined by subtracting the initial concentration from the concentration of phosphate remaining in the solution. This action is to determine the phosphate concentration in the sample. Standard solutions of the phosphate series were used with various concentrations of 5, 10, 15, 20, 25, 30, 35, 40, 45, and 50 ppm. The concentration of residual phosphate that is not adsorbed on the clay is calculated from the absorbance data from the sample analysis, with the regression equation obtained from the calibration curve Equation 1.

$$Y = ax + b \tag{1}$$

Description:
Y = Absorbance of phosphate
x = Concentration of residual phosphate (ppm)
a = Slope (slope)
b = Intercept (intersection)

From the above equation, the residual phosphate concentration (ppm) can be calculated using Equation 2.

$$x = \frac{y-b}{a} \, x \text{ Dilution factor} \tag{2}$$

To determine the concentration of adsorbed phosphate used the Equation 3.

$$C = C_o - C_e \tag{3}$$

Description:
C = concentration of adsorbed phosphate (ppm)
C_o = Initial phosphate concentration (ppm)
C_e = residual phosphate concentration (ppm)

Determining the Adsorption Power of Clay to Phosphate

The amount of phosphate adsorbed mg/g of the adsorbent, which is referred to as the adsorption capacity, is determined by using Equation 4.

$$\frac{x}{m} = \frac{(Co-Ce)\ V}{m} \qquad \text{if } V_o = V_e \tag{4}$$

$$\frac{x}{m} = \frac{(Co\ .\ Vo)-(Ce\ .\ Ve)}{m} \qquad \text{if } V_o \neq V_e \tag{5}$$

Description:
$\frac{x}{m}$ = amount of adsorbed substance (mg/g)
C_o = Initial phosphate concentration (mg/L)
C_e = Concentration of residual phosphate (mg/L)
V = Volume of solution (L)
V_o = Initial volume of solution (L)
V_e = The final volume of solution (L)
m = weight of adsorbent (g)

Determining the Phosphate Clay Adsorption Capacity

The adsorption capacity was determined by using the Freundlich and Langmuir adsorption isotherm pattern.

Freundlich's equation :

$$\frac{x}{m} = k\ C_e^{\ 1/n} \tag{6}$$

$$\log \frac{x}{m} = \log k + \frac{1}{n} \log C_e \tag{7}$$

Langmuir's Equation:

$$\frac{Ce}{x/m} = \frac{1}{b.K} + \frac{1}{b}\, Ce \tag{8}$$

RESULTS AND DISCUSSION

Lontor Natural Clay

The clay used in this study came from Lontor Village, Banda Besar Island, Central Maluku Regency. The clay is initially soaked and washed with distilled water to separate the clay from impurities. This process is carried out several times until a relatively clean clay is obtained. The natural clay was soaked in distilled water for 24 hours, and then the uppermost clay fraction was separated from the natural clay for further washing with distilled water. The clay was then dried in an oven at 80 °C for 6 hours to remove water molecules contained in the space between the clay layers. The dry clay was then ground and sieved through a 100 sieve *mesh*. Clay grinding is done to reduce the size of the clay particles so that the surface area will also increase. While sieving aims to uniform the particle size and obtain a clay sample that is completely clean of impurities. Clay before and after preparation is shown in Figure 1.

(a) (b)

Figure 1. Pictures of Lontor Natural Clay before and after preparation (a) Before preparation; (b) After preparation

Physical Activation of Natural Lontor Clay

The purpose to increase the adsorption capacity and effectiveness of the clay adsorption as an adsorbent, clay activation was carried out. Activation is the most common way to increase adsorbent absorption. The prepared clay was activated by heating in a kiln at 350 °C for 3 hours. The effect of physical activation on the clay aims to open the interlayer space of the clay, remove impurities in the internal part of the clay, and improve the performance of the clay by increasing the adsorption capacity (Fransina & Latupeirissa, 2014).

The physical characteristics observed after calcining can be seen in Figure 2, a slight change in the material's color to be darker than the original color before calcining. Lontor natural clay before activation is light brown, while it is dark brown after activation.

(a) (b)

Figure 2. Pictures of Lontor Natural Clay before and after physical activation (a) Before activation; (b) After activation

Determining the Optimum Contact Time

According to the Lambert-Beer equation: A = abc, a solution's concentration (c) is directly proportional to its absorbance (A). Absorbance measurement to determine the optimum contact time for phosphate ion adsorption was carried out using a UV-Vis spectrophotometer at a wavelength of 665 nm. Parameters for determining the optimum contact time of phosphate ions can be seen in Table 2.

The data in Table 1 and Figure 3 show the optimum contact time for phosphate ion absorption obtained at a contact time of 5 hours, with the percentage of phosphate ion adsorbed at 96.36%. While Auliah, (2009) research obtained a contact time of 8 hours, with the percentage of phosphate ion adsorbed at 70.27%. The more the adsorption time increases, the more adsorbate is absorbed. This condition is because the longer the contact time, the more clay particles collide and interact with phosphate ions so that the adsorption ability increases. After reaching a contact time of 5 hours, the absorption decreased. This number is caused by the adsorbate molecules that do not entirely bind to the adsorbent so that saturation occurs on the adsorbent, which causes a solution equilibrium. Some of the adsorbents release the adsorbate, which causes a decrease after optimum conditions (Auliah, 2009).

Determining the Optimum Concentration

Concentration variations were carried out to determine the maximum absorption concentration during the optimum contact time. The absorbance measurement to determine the optimum concentration of phosphate ion adsorption was carried out using a UV-Vis spectrophotometer at a wavelength of 665 nm. Parameters for determining the optimum concentration of phosphate ions can be seen in Table 2.

Table 1. Parameters for determining optimum contact with initial phosphate concentration 30 mg/L

Contact Time (Hours)	Residual Phosphate Concentration (mg/L)	Adsorbed Phosphate Concentration (mg/L)	Adsorbed Phosphate Ions (%)
2	1.4199	28.5801	95.27
3	1.3837	28.6163	95.39
4	1.3097	28.6903	95.64
5	1.0921	28.9079	96.36
6	1.2092	28.7908	95.97

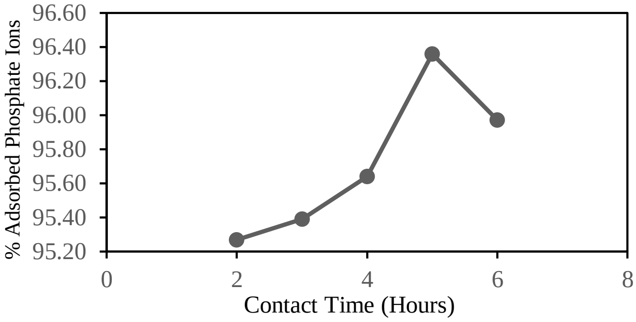

Figure 3. The Curve for determining the optimum contact time

Table 2. Parameters for determining the optimum concentration

Initial concentration of Phosphate Ion (mg/L)	The Concentration of Residual Phosphate (mg/L)	The Concentration of Adsorbed Phosphate (mg/L)	Adsorbed Phosphate Ion (%)
10	0.9446	9.0554	90.55
20	1.2151	18.7849	93.92
30	1.4639	28.5361	95.12
40	1.4813	38.5187	96.30
50	1.8850	48.1150	96.23

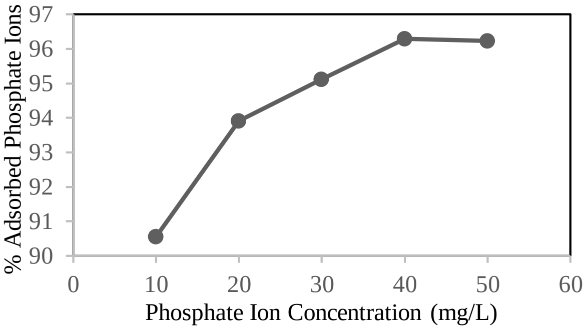

Figure 4. The Curve for determining the optimum phosphate ion concentration

The data in Table 2 and Figure 4 show that the optimum concentration of phosphate ion absorption was obtained at a concentration of 40 ppm, with the percentage of phosphate ion adsorbed at 96.30%. While Auliah (2009) research obtained a concentration of 10 ppm, with the percentage of phosphate ion adsorbed at 87.65%. At concentrations of 10 to 40 mg/L, there was an increase in adsorption. The more significant the phosphate solution concentration, the more clay particles collide and interact with the phosphate. The adsorption ability increases, while the adsorption ability decreases at concentrations above 40 mg/L. This condition is because the clay layer has been saturated with phosphate, which causes a solution equilibrium to occur so that there is phosphate that is not absorbed by the clay (Sinta et al., 2015).

Phosphate Ion Adsorption Kinetic Study

Phosphate ion adsorption kinetics study was carried out with various contact times of 120, 180, 240, 300, and 360 minutes, at a phosphate concentration of 30 mg/L. All adsorption processes take place at room temperature. The value of q_e tested is a value that is close to the value of q_t, which is 1.5 mg/g. The adsorption kinetics parameters can be seen in Table 3. From the first order Lagergren pseudo curve in Figure 9, the value of R^2 was obtained at 65.79%. While the Curve is a second-order Lagergren shown in Figure 10, the value of R^2 reached a value of 99.99%, which is greater than the value of R^2 on the Curve Lagergren pseudo-first-order. While in Maslukah et al. (2020) research, the R^2 value in the pseudo-first-order is 42% while the R^2 value in the pseudo-second-order is 99.99%.

Table 3. Adsorption Kinetics Parameters

t (Minutes)	C_e (mg/L)	C_{ads} (mg/L)	Q (%)	q_t (mg/g)	t/q_t	(q_e-q_t) (mg/g)	Log (q_e-q_t)
120	1.4199	28.5801	95.27	1.4326	83.7638	0.0674	-1.1713
180	1.3837	28.6163	95.39	1.4359	125.3569	0.0641	-1.1931
240	1.3097	28.6903	95.64	1.4365	167.0727	0.0635	-1.1972
300	1.0921	28.9079	96.36	1.4495	206.9679	0.0505	-1.2967
360	1.2092	28.7908	95.97	1.4438	249.3420	0.0562	-1.2503

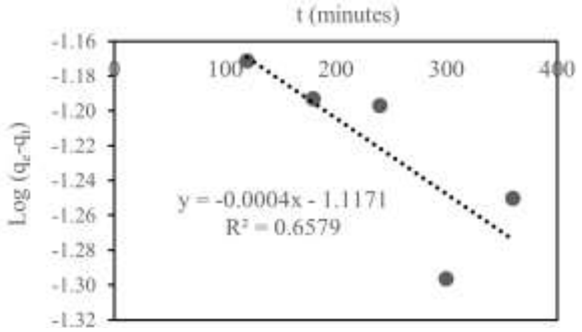

Figure 5. Pseudo-first-order Lagergren plot for phosphate ion adsorption

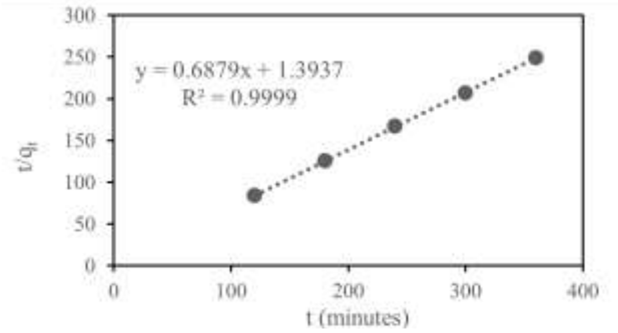

Figure 6. Pseudo-second-order Lagergren plot for phosphate ion adsorption

Phosphate Ion Analysis by spectrophotometer UV-Vis

Analysis of Phosphate Ion by physically activated Lontor natural clay was carried out using a UV-Vis Spectrophotometer at a wavelength of 665 nm. The reagents used in measuring phosphate are ammonium molybdate and $SnCl_2$, which is indicated by a change in the color of the solution to a blue color. In an acidic solution, orthophosphate reacts with ammonium molybdate to form molybdophosphoric acid, which is then reduced by Stannous Chloride ($SnCl_2$) to intense blue molybdenum. The resulting blue color is directly proportional to the phosphate concentration.

Therefore, phosphate ion adsorption on activated natural clay Lontor follows the pseudo-second-order Lagergren adsorption kinetics model. The k value is an adsorption kinetics parameter that shows how fast or slow an adsorption process is. The greater the value of k, the faster the adsorption process is (Sekewael et al., 2013). The value of k_2 obtained was 3.39 x 10^{-1} g mg^{-1} min^{-1}. Two general kinetic equations for the adsorption process in a solution are applied to the data of this study, is shown in Table 3, Figure 5 and Figure 6. From the results of adsorption data analysis, phosphate ion from Lontor natural clay is physically activated. It is known that the adsorption process tends to follow the pseudo-second-order Lagergren kinetic equation.

Determining the Phosphate Ion Adsorption Isotherm Phosphate

The Batch process carried out an ion adsorption experiment by physically activating Lontor natural clay. The use of the batch method serves to allow a thorough adsorption process to the clay. The experimental data can be seen in Table 4. This study used the Langmuir adsorption isotherm and Freundlich adsorption isotherm models to state the occurred adsorption pattern.

Table 4. Adsorption Isotherm Parameters

C_o (mg/L)	C_e (mg/L)	Log C_e	C_{ads}	x	(x/m) (mg/g)	Log (x/m)	C_e/(x/m)
10	0.9446	-0.0248	9.0554	0.4565	0.4564	-0.3407	2.0697
20	1.2151	0.0846	18.7849	0.9429	0.9428	-0.0256	1.2888
30	1.4639	0.1655	28.5361	1.4312	1.4311	0.1557	1.0229
40	1.4813	0.1706	38.5187	1.9319	1.9317	0.2859	0.7668
50	1.8850	0.2753	48.1150	2.4133	2.4131	0.3826	0.7812

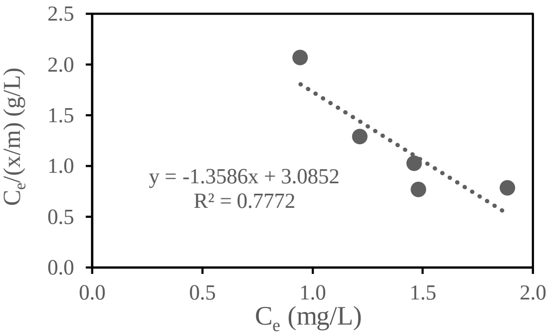

Figure 7. Langmuir adsorption isotherm

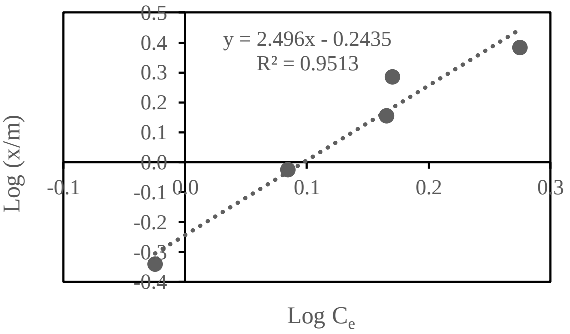

Figure 8. Freundlich adsorption isotherm

By comparing the correlation coefficient (R^2) in Figure 7 and figure 8, the tendency of phosphate ion adsorption in Lontor natural clay can be determined with physical activation based on the existing linearity (Sekewael et al., 2013).

Langmuir Adsorption Isotherm

From the adsorption isotherm parameter data in Table 4, the adsorption isotherm curve in Figure 7 is obtained with a constant value of 0.32 and a constant of -0.43. And are empirical constants related to affinity and absorption capacity parameters. Langmuir isotherm is adsorption that occurs due to a chemical bond between the surface of the adsorbent and the adsorbate. On each active site, only one molecule can be adsorbed so that a monolayer is formed and adsorption occurs in a homogeneous layer.

Freundlich Adsorption Isotherm

The adsorption isotherm parameter data in Table 5 shows that the adsorption isotherm curve in Figure 8 is obtained with a constant k value of 0.57 and an n value of 0.40. While in Auliah (2009) research, the constant k value of 0.32 and an n value of 4.13. The adsorption capacity is related to the adsorption constant or k constant, which is 0.57 $mg^{1-1/n}$ $L^{1/n}$/g. Freundlich adsorption occurs because of the physical bond between the adsorbate (a substance that is adsorbed) and the surface of the adsorbent (a substance that absorbs). The Freundlich isotherm assumes that adsorption occurs in heterogeneous layers and more layers (multilayer).

Based on the graph in Figures 7 and 8, the value of correlation ($R^{2)}$ of 0.7772 Langmuir isotherm, while the value of the correlation ($R^{2)}$ Freundlich isotherm showed a linear relationship with R^2 value of 0.9513. Thus, it can be said that the adsorption of phosphate ions by physically activated Lontor natural clay tends to follow the Freundlich adsorption isotherm, which means that the adsorption occurs in a multilayer layer.

CONCLUSIONS

The optimum contact time for the absorption of phosphate ions by physically activated Lontor natural clay was obtained at a contact time of 5 hours, with the percentage of phosphate ions adsorbed at 96.36%. The optimum concentration of phosphate ion absorption by physically activated Lontor natural clay was obtained at a concentration of 40 mg/L with a percentage of phosphate ion adsorbed at 96.30%. Phosphate ion adsorption by activated Lontor natural clay physically follows the adsorption kinetics model Lagergren pseudo-value second-order rate constant k_2 of 3.39 x

10^{-1} g mg^{-1} $min^{-1.}$ It follows the Freundlich adsorption isotherm models with constant k of 0.57 $mg^{1-1/n}$ $L^{1/n}$/g.

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