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Research Article

Synthesis, Characterization, and Application of Rubber Fruit Shell as an Adsorbent for Phosphate Removal in Real Grey Water

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Abstract

The washing waste from the laundry industry contains phosphate that can pollute the environment. Multiple efforts have been made but have not reached the small to medium-scale laundry industry; this happens because of the high cost of technology. In this paper, the sorption process by rubber fruit shells successfully reduced the pollutants parameter from greywater. This research has succeeded in creating an adsorbent with raw rubber fruit shell waste; studies on manufacturing techniques, adsorbent characteristics, ability to reduce phosphate content, and adsorption isotherm models are well described. This study also promotes the utilization of rubber shell waste that has not been utilized properly. Adsorbents derived from rubber fruit shells reduced phosphate levels by up to 98% by adding 0.5 g of adsorbent to 100 mg/L of phosphate for 60 minutes. The appropriate adsorption isotherm model in this study is the Freundlich isotherm model.

Keywords: Adsorption; greywater; freundlich model; rubber fruit shell

1. Introduction

Environmental pollution is an issue of concern to the world (United Nations Environmental Programme, 2013). Various studies have reported pollution in the environment (soil, water, and air) (Appannagari, 2017). Pollution that occurs damages the environment and can hurt human health, environmental sustainability, animal habitats, and threatens the life of plants. Industrial activity is mainly caused use pollution that occurs; activities that can't be separated from human life are one of the leading causes of the decline in the quality and quantity of the environment. On the one hand, the industry positively impacts economic growth and fulfils human needs. Besides, many industrial activities have not prioritized proper and correct waste management. That is due to the high costs incurred when processing waste, especially in small and handled industries such as laundry (Turkay et al., 2017).

Laundry activities produce used washing waste in detergents containing nitrogen, phosphate, heavy metals, linear alkylbenzene sulphonate, volatile organic acids, alcohols, pH, and oxygen demand (Braga & Varesche, 2014). Various studies have reported the dangers caused by laundry waste activities (Ciabattia et al., 2009; Eriksson et al., 2002; Inyinbor et al., 2019; Samadikun et al., 2021; Šostar-Turk et al., 2005; Watiniasih et al., 2019). In a recent study, greywater can change soil characteristics such as saturated hydraulic conductivity, electrical conductivity, pH, exchangeable sodium percentage, cation exchange capacity, and sodium adsorption on the ratio examined after the irrigation with greywater (Mohamed et al., 2018). A study reported how to solve the problem of greywater, researchers used polyethersulfone ultrafiltration membranes (Mozia et al., 2016), but this treatment needs a high cost and high technology. Another study also reported using Combined chemical coagulation-flocculation/ultraviolet photolysis to solve the greywater properties, and this technology showed can reduce 74% of greywater properties (Terechova et al., 2014). Another method that more expensive and could be applied in rural areas is adsorption. Adsorption is a low-cost potential method and more accessible than other wastewater treatment methods (Budihardjo et al., 2021). Adsorption has successfully reduced the parameter of wastewater using the sorption mechanism (Wibowo et al., 2022). Several resources can be converted into adsorbent, such as coconut shells, bentonite, peat, coal (Naswir et al., 2019; Wibowo & Naswir, 2019).

Although any studies informed about greywater and its essential mitigation, the limitation of the small-scale laundry industry still had problems in treating the greywater. Untreated greywater and its infiltration into the soil have some negative impacts. A recent study reported that greywater was contaminated soil in Toowoomba City (Howard et al., 2005). Many papers published about solving the problem of greywater properties reported several methods, including adsorption, filtration membranes, and chemical coagulation. Still, not all the technology said can be applied in the field. Several studies are limited in the lab scale. Besides, the limitation of resources is one problem. This study prepared a new low-cost and easy-finding resource to solve greywater properties. This paper aims to reduce the phosphate from greywater. This paper also described the material and method, discussion, and comparison with another study. This paper used several variations, including time contact, pH, physicochemical properties of adsorbent, sorption capacity, and isotherm model. Based on this study, agricultural rubber fruit shells are a potential material to solve wastewater from laundry using the adsorption method.

2. Methods

The tools and materials to be used in this research are UV-Vis spectrophotometer, analytical scale, 125 ml Erlenmeyer, 100 ml beaker, 100 ml measuring flask, 250 ml and 1000 ml, 25 ml and 50 ml measuring cups, pH meter, spatula, magnetic stirrer, cuvette, drop burette, 10 ml measuring pipette, 2 ml volumetric pipette; 5 ml; 10 ml; 20 ml and 25 ml; 1000 ml beaker, dropper pipette, rod, funnel, filter paper, watch glass, hot plate. 120 mesh of Activated Carbon, Laundry Liquid Waste Samples, Powder Buffer pH 4 and 6.8, distilled water, Phenolphthalein Indicators, 0.1 M Sodium Hydroxide (NaOH), 0.1 M Hydrochloric Acid (HCl), 5N Sulfuric Acid (H2SO4), Potassium Antimonyl Tartrate (K(SbO)C4H4O6.½H2O), Ammonium molybdate ((NH4) 6Mo7O24.4H2O), Ascorbic acid (C6H8O6) 0.1 M, Potassium dihydrogen phosphate anhydrous (KH2PO4).

2.1. Preparation of Activated Carbon

Activated carbon is made from rubber fruit shells burned at 500° C for 1 hour at 120 mesh. The activated carbon is then activated using a 10% H_3PO_4 solution for 24 hours. The activated carbon is then dried to remove its water content at a temperature of 110° C; then, pH neutralization is carried out using distilled water.

2.2. Wastewater and Artificial Solution

Wastewater from commercial laundry was obtained from commercial laundry in Jambi, Indonesia. In addition, an artificial solution of phosphate was also created to analyze the isotherm models. Artificial solution of phosphate (500mg/L) was made by dissolving 0.715 g of anhydrous potassium dihydrogen phosphate, KH2PO4, with 100 ml of distilled water in a 1000 ml volumetric flask. Then, it was added distilled water right on the mark and homogenize. The sulfuric acid solution

(H_2SO_4) (Concentration: 5N) was made by putting 20.8 mL of concentrated sulfuric acid in a beaker containing 50 mL of distilled water and place it on an ice bath. Then, the solution was diluted with distilled water to 150 mL and homogenized. The antimonyl tartrate potassium solution ($K(SbO)C_4H_4O6.½ H_2O$) was made by dissolving 1.3715 g of potassium antimonyl tartrate with 400 mL of distilled water in a 500 mLvolumetric flask. Then, distilled water was added to the right mark and homogenized. The ammonium molybdate solution ($(NH_4)6M_{07}O_{24}.4H_2O$) was added by dissolving 4 grams of ammonium molybdate in 100 mL of distilled water and homogenized. The ascorbic acid solution ($C_6H_8O_6$) 0.1 M was added by dissolving 1.76 g of ascorbic acid in 100 mL of distilled water. This solution is stable for one week at 4 ^oC. Consequently, 50 mL of 5N H_2SO_4, 5 mL of potassium antimonyl tartrate solution, 15 mL of ammonium molybdate solution, and 30 mL of ascorbic acid solution was mixed. Note: If blue is formed, mixed solutions cannot be used. If turbidity occurs in the hybrid solution, shake and allow a few minutes until the turbidity disappears before use. This hybrid solution is stable for 4 hours.

2.2. Adsorption Test

2.2.1. Calibration Curve

Standard calibration curves for the determination of phosphate compounds were prepared using KH_2PO_4, standard solution concentration of 0.2 mg/L; 0.4 mg/ L; 0.8 mg/L; and 1 mg/L. Measurement of absorbance using a UV-vis Spectrophotometer instrument. The calibration curve is obtained by making a curve between the concentration and the absorption of each concentration.

2.2.2. Time Contact

The contact time of the standard phosphate solution was conducted by varying the contact time. The activated carbon is weighed as much as 0.5 g and then mixed in 100 mL of phosphate solution with a concentration of 100 mg/L, then stirred with variations in contact time of 15, 30, 45, 60, 75, 90, 105, 120 minutes. Separated between adsorbent and adsorbate, then tested with UV-Vis Spectrophotometry to determine the concentration of adsorbed phosphate.

2.2.3. Variation of pH

The determination of the optimum pH in the standard phosphate solution is carried out by varying pH. The activated carbon is weighed as much as 0.5 g, then mixed in 100 mL of phosphate solution with aconcentration of 100 mg/L, then stirred with variations in pH 3, 5, 7, 9, and 11. Making pH variations using 0.1 M HCl and 0 NaOH, 1 M. Separated between the adsorbent and the adsorbate and then tested by UV-Vis Spectrophotometry to determine the concentration of adsorbed phosphate.

2.2.4. Efficiency and Isotherm Model

This determination determines a relationship between the amount of substance adsorbed by the adsorbent with concentration at fixed equilibrium and temperature. Determination of the adsorption capacity of phosphate compounds was carried out at various concentrations, namely 20 mg/L, 40 mg/L, 100 mg/L, 120 mg/L, 140 mg/L, 160 mg/L, 180 mg/L, 200 mg/L, 220 mg/L, 240 mg/L, 300 mg/L, and 400 mg/L in 100 ml phosphate solution with 0.1 g activated carbon. This adsorption is carried out from the results obtained in determining the optimum pH and the optimum time that has been obtained. Then the isotherm model is determined according to the phosphate adsorption.

3. Result and Discussion

3.1. Wastewater from Commercial Laundries

Commercial laundry is one of the small-scale industries that generated a source of pollution to be considered. Several studies reported that commercial greywater has a high surfactant contaminant; this pollutant is a surface-active agent with different ends, namely hydrophilic (like water) and hydrophobic. This active ingredient functions to reduce the surface tension of the water to release dirt that sticks to the material's surface. However, the surfactant is one of the dangerous contaminants for the environment. Colloidal pollutions. Characterized wastewater from the commercial laundry. In the fluid phase, surfactants (detergents) from so-called micellar colloidal suspensions whose dimensions are between nanometer to the micrometre.

Wastewater from commercial laundry has a different characteristic; this condition is caused by the difference in dirt from the clothes, composition of detergent, and technology. Phosphate is one of the pollutants contaminated in greywater. This contaminant can cause some problems to the environment, mostly surface water. When there is an excessive amount of phosphate in the water, it is released. These chemicals will pollute the water and trigger the uncontrolled growth of algae that can cover the water's surface. This alga will interfere with the development of fish and plants in the water because it blocks the incoming sunlight. Algae also seize the oxygen needs of other species in the water, which can cause other organisms to die from lack of oxygen. Although not algae, the presence of detergent foam on the water's surface causes limited contact with air and water, thereby reducing dissolved oxygen.

Wastewater from commercial laundry has a concentration of phosphate 3.1; this concentration is causing the sample to be collected from the small-scale commercial laundry. A recent study reported that wastewater from laundry has characteristics such as nitrogen, phosphate, heavy metals, linear alkylbenzene sulphonate, volatile organic acids, alcohols, pH, and chemical oxygen demand (Braga & Varesche, 2014). These contaminants need to be treated by low-cost and traditional methods. The traditional and low-cost method is an applicable method for removing pollutants. These pollutants are dangerous to the environment, but a recent study also reported the human health impact due to these pollutants.

3.2. Physicochemical of Activated Carbon

3.2.1 Water Content

Water content is one of the specific characteristics of activated carbon. In this study, four samples were measured in different particles size and temperatures (Table 1). Water content can act on sorption ability, the pores of the adsorbent cause it to be filled with water. Thus, less water content could give a better sorption ability. This study informed that higher temperature would provide better water content, and the size of activated carbon will impact water content; the more extensive samples conducted more water content. Water content can affect adsorption ability. The greater the water content of an adsorbent, the smaller the adsorbent's ability to absorb adsorbate; te to the adsorbent pores still filled with water. The result of this study is better than adsorbent derived from cacao, have a water content of 10.49% at 500°C and 5.8% at 600°C for four hours (Masitoh & Sianita, 2013). This value also compares with National Indonesian Standard (SNI), the maximum water content based on the SNI standard is 15%. Thus, all the samples are successfully created based on water content.

Table 1. Water content in adsorbent

No	Temperature (°C)	Size (mesh)	Water content (%)
1.	400	80	9.5
2.	400	120	7.9
3.	500	80	6.5
4.	500	120	4.1

3.2.2. Ash Content

The ash content dramatically affects the adsorption ability of activated carbon. The formation of mineral salts causes the ash content in the adsorbent during the pyrolysis process; the ash content can also be caused by the initial composition of the material used as the adsorbent. The rubber fruit shell is caused by carbon, organic compounds, and minerals. Another factor that causes the high ash level in the manufacture of activated carbon is that the pyrolysis process is not optimal; this can also be caused by air entering the instrument used. The resulting ash content can be seen in the table below in this study.

Table 2. Ash content

No	Temperature (°C)	Size (mesh)	Ash content (%)
1.	400	80	0.5
2.	400	120	1.6
3.	500	80	3.8
4.	500	120	4.8

In this study, it was known that the higher the temperature used, the higher the ash content in the adsorbent. That is due to the higher the temperature. More impurities will be released from the adsorbent surface. Ash content is assumed to be mineral residue left behind from the carbonization process; the materials for the adsorbent (rubber fruit shell) contain carbon and organic compounds and several minerals, where some of these minerals have been lost during carbonization and activation. Moreover, some are thought to be still left in activated carbon. The ash produced in this study can also be caused by non-carbon compounds found in the rubber fruit shells. This study generated a better adsorbent than activated carbon derived from Eucalyptus camaldulensis Dehn. The ash content from the previous survey is 4.88%, with pyrolysis at 5000C (Patnukao & Pavasant, 2008).

3.3. Application of Activated Carbon in Commercial Greywater

The application of activated carbon in laundry liquid waste is carried out by varying the contact time. That is intended so that the adsorbent reaches its equilibrium point; besides contact time, stirring is carried out to accelerate the diffusion process. The test results on the variation of contact time from 15 to 120 minutes can be seen in the table below.

Table 3. The absorbance of the standard curve

No.	Time (minute)	Absorbance (Abs)			Average
		Abs 1	Abs 2	Abs 3	
1	15	0.1157	0.1165	0.1156	0.1159
2	30	0.1004	0.1038	0.1028	0.1023
3	45	0.0836	0.0820	0.0822	0.0826
4	60	0.0553	0.0543	0.0554	0.0550
5	75	0.0971	0.0978	0.0963	0.0970
6	90	0.0674	0.0654	0.0672	0.0666
7	105	0.0800	0.0805	0.0796	0.0800
8	120	0.1030	0.1003	0.1016	0.1016

Based on this study, the average absorbance value is calculated by the equation obtained from the standard phosphate solution calibration curve using a linear regression equation, namely $Y = 0.2169x + 0.0103$. Where Y is the absorbance and X is the concentration with a regression value of $R2 = 0.9448$. so that the final concentration after stirring is known with variations in contact time, the final phosphate concentration after several contacts time in 100 mg/L phosphates is seen in Table 4.

Table 4. Phosphate removal using activated carbon tion

Time (Minute)	Final concentration (mg/L)	Phosphate removal (mg/L)
15	4.87	95.13
30	4.24	95.76
45	3.33	96.67
60	2.06	97.94
75	4.00	95.93
90	2.59	97.41
105	3.21	96.79
120	4.21	95.79

Per cent, phosphate removal from commercial laundry waste can be seen in Fig. 3. The best reduction of phosphate is 60 minutes (97.98%). That is due to the large size of the phosphate molecules, so it takes a longer time (60 minutes) to achieve bond stability on the surface of the activated carbon. The decrease in efficiency after 60 minutes is due to the unstable adsorption process so that the phosphate molecules are released from the adsorbent pores. This result is better than the test results using a combination of activated carbon and zeolite, which can only reduce 90% of the phosphate content by using 40 cm high zeolite in a test column (Agustina et al., 2014).

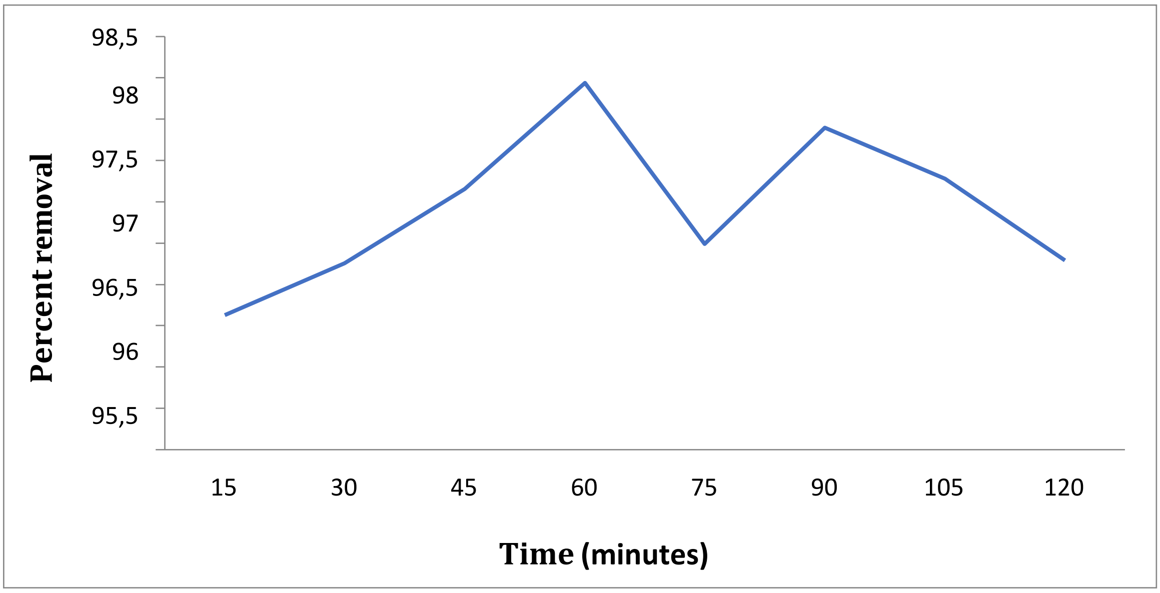


Figure 2. Time impact on phosphate removal using activated carbon

3.4. pH Effect on Phosphate Removal

pH is one of the parameters that affect the adsorption ability. Various studies have informed that the best adsorption is obtained at a pH close to neutral; it is scarce research that states that the best adsorption ability occurs when the pH is acidic or alkaline. The results of this study inform that the best adsorption ability is at pH 5 (Table 5). That is due to a decrease in the mobility of pollutants in polluted water. It is known that various contaminants are mobilized very quickly at acidic pH that the adsorption is not optimal, while at a pH approaching neutral-neutral, pollutant mobility is not as fast as at acidic pH, it is easier to absorb.

Table 5. Removal efficiency in different pH

pH	Final concentration (mg/L)	Phosphate removal (mg/L)
3	3.64	96.36
5	2.71	97.29
7	4.53	95.47
9	5.88	94.12
11	6.81	93.19

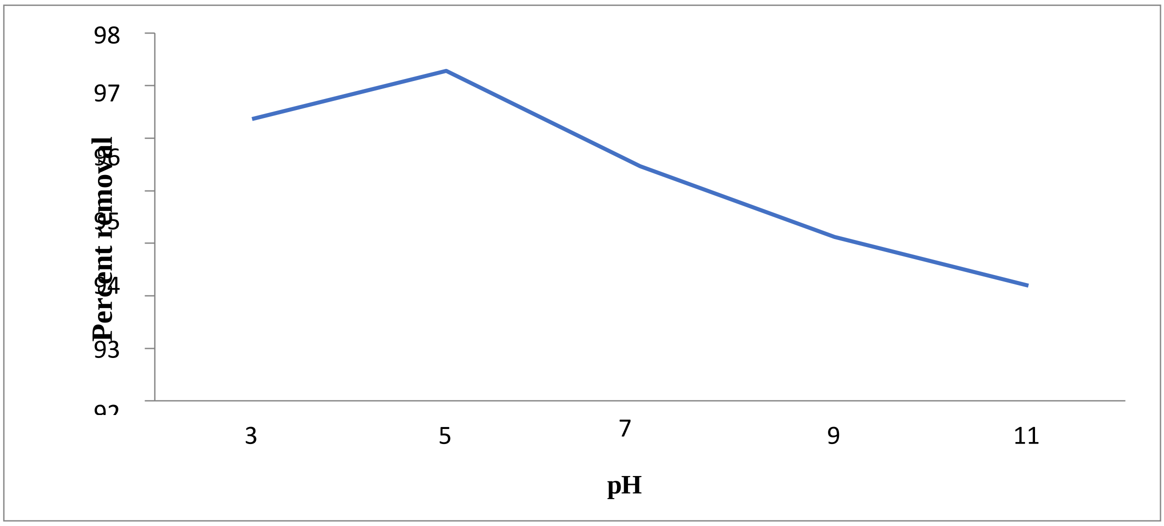


Figure 3. Percent removal of phosphate

3.5. Isotherm Model

Rubber fruit shell-activated carbon has good adsorption ability. Based on this research, it is known that the higher the phosphate concentration, the more phosphate is left in the solution because there is an equilibrium between the phosphate left in the adsorbate and that which is absorbed in the adsorbent, that the higher the phosphate concentration in the solution, the greater the phosphate that the adsorbent can absorb—activated carbon. The adsorption process or adsorption by an adsorbent is influenced by many factors and has certain specific adsorption isotherms. The factors that influence the adsorption process include the type of adsorbent, the kind of substance absorbed, the surface area of the adsorbent, the concentration of the substance being absorbed, and the temperature. Because of these factors, each adsorbent that adsorbs one substance with another will not have the same adsorption isotherm model. It is known that there are two types of adsorption isotherm pattern equations that are often used in the adsorption process in solution, namely the Langmuir and Freundlich adsorption equations. The Freundlich isotherm assumes that the multilayer layer where the bond between the adsorbent and the adsorbate occurs is due to the Van der Waals force so that the bond is not too strong. At the same time, the Langmuir isotherm model defines that the maximum adsorption capacity occurs due to a single layer (monolayer) of adsorbate on the adsorbent surface.

Table 6. Removal efficiency in different concentration

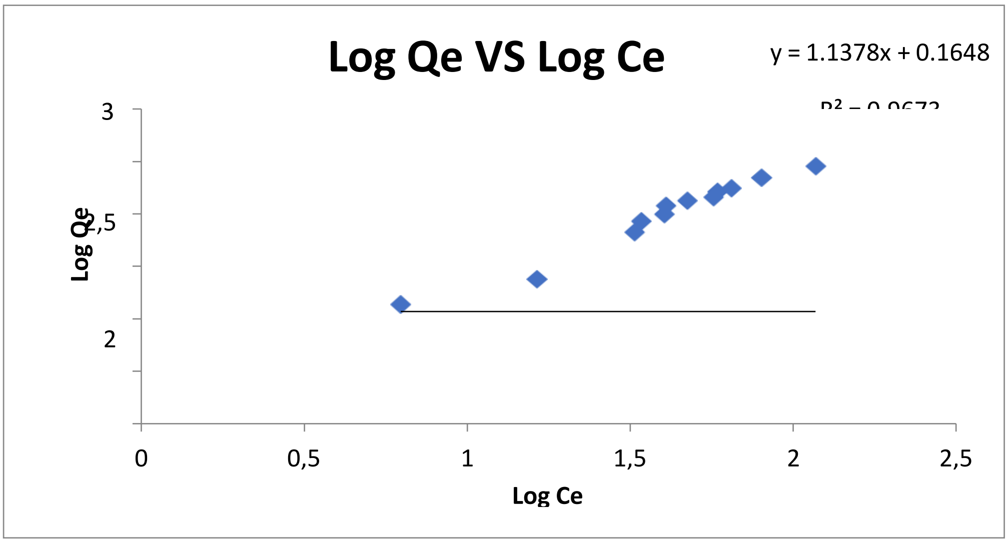
Initial concentration (mg/L)	Phosphate (mg/L)	Phosphate Sorbed (mg/L)
20	6.24	13.76
40	16.31	23.69
100	32.62	67.38
120	34.16	85.84
140	40.20	99.80
160	40.59	119.41
180	47.11	132.89
200	57.03	142.97
220	58.51	161.49
240	64.69	175.31
300	79.85	220.15
400	117.12	282.88

The method used to measure the adsorption process of phosphate levels is the UV-Vis Spectrophotometric method. The measurement results from the UV-Vis Spectrophotometry were then analyzed, and the curve and regression value was determined. The calculation results are shown in the table below.

Table 7. Isotherm model for langmuir and freundlich

No	Co (mg/L)	Sorbed phosphate	Ce (mg/L)	Qe (mg/g)	Ce/Qe	Log Ce	Log Qe
1	20	13.76	6.24	13.753	0.454236784	0.795679743	1.138397443
2	40	23.69	16.31	23.69	0.688507483	1.212473726	1.374565061
3	100	67.38	32.62	67.373	0.484288055	1.513589644	1.828485886
4	120	85.84	34.16	85.833	0.398073227	1.533617261	1.933654292
5	140	99.80	40.20	99.793	0.40290871	1.604306734	1.999100079
6	160	119.41	4.59	119.401	0.340025247	1.608519129	2.077007964
7	180	132.89	47.11	132.882	0.354588942	1.673191344	2.123466156
8	200	142.97	57.03	142.970	0.398901097	1.756110148	2.155244917
9	220	161.49	58.51	161.485	0.362358392	1.767270511	2.208132188
10	240	175.31	64.69	175.307	0.369029229	1.810860024	2.243799258
11	300	220.15	79.85	220.150	0.362721744	1.902288336	2.342714744
12	400	282.88	117.12	282.873	0.414064646	2.068659647	2.451591496

From the table above, the curves of the Freundlich and Langmuir equations are made, with the linear equations of Freundlich and Langmuir isotherms sequentially - also obtained by plotting the Y-axis = Log QE, X-axis = Log Ce, and Y = Ce/Qe axis X = Ce.



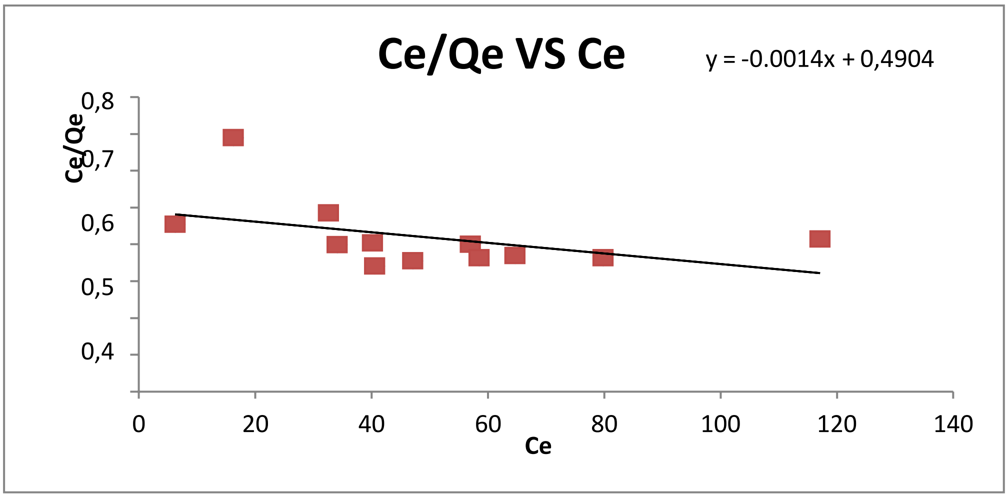


Figure 4. Isotherm langmuir and freundlich models

In this study, testing the Langmuir adsorption equation and the Freundlich adsorption equation is proven by a good linearization graph and has a coefficient of determination R2 ≥ 0.9 (close to number 1), phosphate adsorption using activated carbon follows the Freundlich isotherm equation, it can be seen from the considerable regression value with $R^2 = 0.9673$. This value indicates that this study follows the Freundlich equation, which assumes that the Freundlich isotherm is based on the opinion that the adsorbent has a heterogeneous surface. Each molecule has a different absorption potential. The idea that adsorption occurs multilayer on the adsorbent surface so that the Freundlich isotherm equation is often used in the determination practical because it generally provides a satisfactory correlation. The Freundlich isotherm describes a physical type of adsorption in which adsorption occurs in several layers and the bonds are not strong. Freundlich isotherm, too, assumes that the adsorption sites are heterogeneous. The Freundlich isotherm describes the type of physical adsorption in which the adsorption occurs in several layers and the bond is not strong. The Freundlich constant showed the adsorbate and the adsorbent bond and was obtained experimentally. The Freundlich isotherm has a different model from the Langmuir isotherm model; the Langmuir isotherm defines maximum adsorbent capacity as a single layer (monolayer) of adsorbate on the surface of the adsorbent.

4. Conclusions

Laundry industrial waste has properties that are harmful to the environment. The waste content of laundry detergent is known to pollute soil and water. Various attempts have been made but have encountered difficulties in application in several areas due to the unavailability of raw materials, expensive technology, and demanding access to locations in remote areas. This research has successfully created a simple technology by utilizing the adsorption concept. The adsorbent is made from abundant rubber shell waste and is rarely used. This research has reduced the phosphate content up to 97.94% in an artificial solution. The adsorbent was made at temperature variations of 400 and 500 °C with 100 and 120 mesh particle sizes. Variation in pH is also carried out by making artificial phosphate solutions with acidic to alkaline pH; the optimum pH to reduce phosphate levels is 5. The suitable isotherm model in this study is the Freundlich isotherm model; this shows that the adsorption is physical adsorption; this adsorption indicates that the entrapment process occurs in several layers with weak bonds. Freundlich's adsorption model also informs that the adsorption sites are heterogeneous.

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