Walisongo Journal of Chemistry
Walisongo Journal of Chemistry Vol. 5 Issue 1 (2022), 83-93
ISSN: 2621-5985 (online); 2549-385X (print)
DOI: https://doi.org/10.21580/wjc.v5i1.11755

Adsorption of Cd (II) into Activated Charcoal from Matoa Fruit Peel

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Received: 27 May 2022; Accepted: 30 June 2022; Published: 15 July 2022

Abstract

Cadmium (Cd) is one of the heavy metals with a high level of contaminants that is environmentally harmful and can interfere with human health. This study aims to determine the adsorption capacity and adsorption kinetics of Cd (II) from Matoa fruit peel activated by nitric acid. The adsorption method can be used to treat Cd (II) waste in the water. Activated charcoal is used for adsorption. The charcoal produces a relatively 1.17 % ash content, 3.92 % water content, and a 507.64 mg/g iodine absorption test. Based on the results of FTIR characterization, it is known that the O-H and C=O groups play an important role in adsorption. The SEM-EDX characterization produced a carbon content of 99.21 %. At pH 9, activated charcoal adsorbs Cd (II) metal effectively, with a contact time of 40 minutes and a concentration of 20 ppm. The activated charcoal of Matoa fruit peel activated by nitric acid had an adsorption capacity of 59.75 mg/g. It used a pseudo-second-order reaction for the chemical kinetics equation and the Langmuir adsorption isotherm equation for the adsorption isotherm.

Keywords: *matoa fruit peel; adsorption; Cd (II); activated charcoal*

Introduction

Water is a natural substance found on the earth's surface. Water has many benefits for the human body, such as controlling metabolism, transporting nutrients, and maintaining body temperature balance, however, there are water problems in some areas due to a lack of clean water supplies for human life. The limitations of clean water are caused by excessive human use of clean water, which is contaminated with heavy metals (Fe, Zn, Cd, Hg) above the threshold level (Adetokun et al., 2019; Nejadshafiee & Islami, 2019; Nugroho, 2013). Heavy metal ions in wastewater have relatively high toxicity, which can cause significant environmental problems (Hajjaoui et al., 2022; Kataria et al., 2022; Pratomo et al., 2017).

Cadmium (Cd) is one of the dangerous heavy metals in the water. Cadmium contamination is typically caused by residual substances in the paint, soft drink, smelting, metal plating industries, and others. It is the second most toxic heavy metal after mercury. Because of its toxicity, this metal in water must be present in small quantities (Sembiring et al., 2010). Government regulation no. 82 of 2001 concerning water quality and water quality standard limits for metal Cd content by

Minister of Health of the Republic of Indonesia No. 32 of 2017 is a maximum of 0.01 ppm (Khan et al., 2021; Ministry of Health, 2017; Sasongko et al., 2014).

Heavy metals can cause problems for human health. The toxic impact of Cd (II) metal is that it can cause dangerous diseases in humans, such as lung, kidney, and liver damage, as well as high blood pressure and gastrointestinal problems (Faizal & Fitri, 2014; Rajmohan, 2021). Adsorption is one of the most effective methods for removing hazardous substances from wastewater, and it is commonly used in the treatment of industrial waste liquids. Because the adsorbent used in this process is typically relatively expensive, it is necessary to use an adsorbent that is both inexpensive and environmentally friendly, such as adsorbents made from biomass waste. Adsorbent derived from waste materials not only reduces environmental pollution caused by solid waste, but it can also increase the adsorbent's selling price (Awokoya et al., 2021; Haura et al., 2017; Xu et al., 2022).

The benefits of matoa are only known to be used as medicinal plants in the North Sulawesi region. Matoa plants are commonly recognized for their fruit, which has a distinctive flavor. This plant has previously been studied as a medicinal plant by observing the secondary metabolism contained in Matoa fruit (Kurniawan et al., 2017; Ngajow et al., 2013; Pakaya et al., 2021). The Matoa fruit is one of the fruits that thrives in eastern Indonesia. Whereas this fruit has numerous benefits, it is still used by a small number of people. As a result, this skin degrades and is discarded. According to (Faustina et al., 2014; Kurniawan et al., 2017), the Matoa fruit peel has a high cellulose content of about 50% and has the potential to be used to make paper. The characterization test on the skin of the Matoa fruit produced a cellulose content of 50.6 %, indicating that the Matoa content was higher than that of other materials such as rice straw (27 % -34 %) and bagasse (36%-40%). Because Matoa peel has high antioxidant activity, it has the potential to be an antioxidant source.(Faustina et al., 2014; Pakaya et al., 2021).

Activated charcoal is a carbon compound that has been activated so that it has larger pores and surface area. Therefore, it can increase its adsorption power (Alsohaimi et al., 2020; Eze et al., 2022; Popoola, 2019; Suhendarwati et al., 2014). Activation can be accomplished chemically or physically. Chemical activation is typically accomplished through immersion in water-absorbing alkali hydroxide, carbonate compounds, sulfides, ZnCl2, sulfuric acid, phosphoric acid, and sodium chloride (Setiawati & Suroto, 2010). The sodium hydroxide solution in this activation serves to reduce the lignin compounds present in the Matoa peel, thereby inhibiting the adsorption process. Because lignin can clog the ion transfer process on the active site of the adsorbent, its presence can slow down the adsorption process. According to the study (Safaria et al., 2013), OH-ions in NaOH can break the bonds in the basic structure of lignin. The lignin will then dissolve readily. To decompose the mineral salts in the adsorbent, it must also be activated with nitric acid (Jaouadi et al., 2017; Mentari et al., 2018; Setiaty Pandia & Budi Warman, 2017).

In this study, adsorption of Cd (II) metal was accomplished by using cellulose on activated charcoal from the Matoa fruit peel, which had been activated by nitric acid. This study began with the creation of activated charcoal from the Matoa fruit peel, which was then activated by nitric acid. Following that, the adsorbent will be contacted with a solution of Cd (II) as an adsorbate while taking pH parameters, optimum adsorption contact time, and determining the optimum concentration. It is expected that activated charcoal from the Matoa fruit peel will be able to adsorb Cd (II) metal solution waste during the activation process.

Research Methodology

Materials

A beaker, 50 ml measuring cup, 1 ml measuring pipette, blender, Erlenmeyer, dropper, porcelain cup, desiccator, magnetic

stirrer, filter paper, 80 mesh sieve, analytical balance, oven, furnace, AAS with *Thermo Scientific* brand, FTIR with *Bruker Alpha 2* brand, and SEM with *JEOL JSM-6510LA* brand were the tools used in this study. Whereas the materials used in this study were Matoa fruit peel, 1M NaOH solution, $CdSO_4.8H_2O$, nitric acid, iodine, sodium thiosulfate, and distilled water.

Activation of activated carbon on Matoa peel

The matoa fruit peel was weighed as much as 270 grams and placed in a kiln at 400 °C for 60 minutes. The matoa fruit peel, which had become charcoal, was cooled, and then crushed to 80 mesh, then sieved using an 80-mesh sieve. the matoa fruit peel sample was weighed in an erlenmeyer, then 1m nitric acid was added in a ratio of 1:10, after being soaked for 1 hour, rinsed using distilled water, and then filtered and washed with distilled water until the ph was neutral and dried in an oven for 24 hours at 100°C, then cooled and then characterized using FTIR and SEM-EDX.

Adsorption Test

The activated charcoal adsorbent was weighed at 0.1 grams and put in an Erlenmeyer. Then a sample solution of 10 ppm was added to 50 mL with various variations, namely variations in pH, contact time, and concentration. The sample was stirred by using a magnetic stirrer for 60 minutes, then allowed to stand for 15 minutes, then filtered using filter paper, and the absorbance was measured by AAS.

Results and Discussion

Cellulose Preparation from Matoa Peel

The matoa fruit peel used in this study was Matoa that grows in Pati regency. Matoa fruit peel contains lignin of 28.24% (Faustina et al., 2014; Kurniawan et al., 2017). According to (Li et al., 2016), Materials containing cellulose become hard and inhibited cellulose from binding to metal ions. Therefore, a delignification process was carried out to remove lignin content. The delignification process was carried out by using a 6% NaOH solution because NaOH solution could damage lignin. According to (Rambat et al., 2015) the delignification process using NaOH solution can reduce lignin levels by a percentage of 19.11%. NaOH can separate lignin from cellulose and form bonds within the lignin itself. The separate bonds are hydrogen bonds that link lignin to cellulose. There is a reaction to break the bonds of lignin and cellulose as shown in Figure 1.

Functional Group Analysis by Using FTIR

Analysis with the FTIR instrument aims to determine what functional groups are present in the Matoa fruit peel adsorbent before and after activation. The results of the characterization of charcoal before activation can be seen in Figure 2.

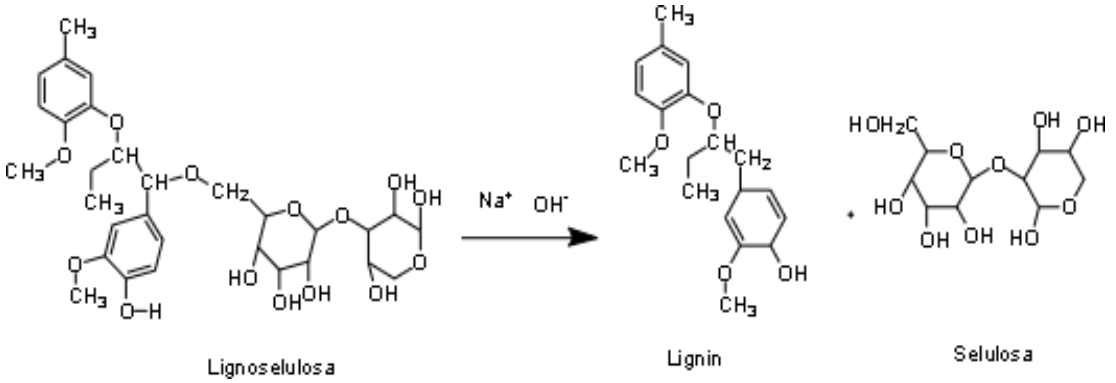


Figure 1. Mechanism of breaking the bonds of lignin and cellulose (Zhu et al., 2016)

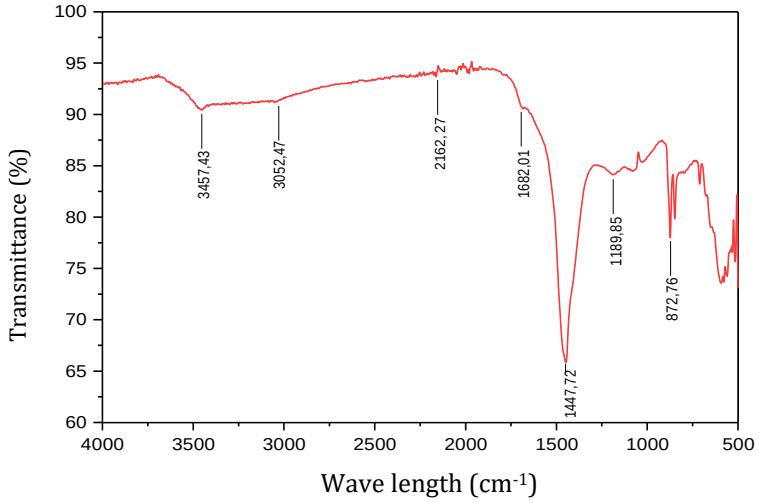


Figure 2 FTIR spectra of atoa fruit peel charcoal before activation

Figure 2 shows that in the presence of an O-H group at a wave number of 3457.43 cm^{-1}, the presence of an O-H bond is usually polar charcoal (Mentari et al., 2018). The presence of C-H groups is indicated by wave numbers 3052.47 cm^{-1}, 2162.27 cm^{-1}, 1447.72 cm^{-1}, and 872.76 cm^{-1}. According to Losev, the C-H functional groups are generally present in materials that contain a lot of cellulose. The C=O groups are at wave number 1682.01 cm^{-1} and the C-N functional groups are at wave number 1189.85 cm^{-1}. The C=O and C-N functional groups have an important role during the adsorption process (Siregar, 2019). While the results of the characterization of charcoal after activation can be seen in Figure 3.

The FTIR spectra in Figure 3 shows that after activation, a new group appeared, namely C=C at a wave number of 1576.27 cm^{-1}. The C=C group indicates an increase in carbon content (Mentari et al., 2018). The difference in functional groups in activated charcoal of Matoa fruit peel before and after activation is shown in Table 1. Functional groups that have been activated by using nitric acid are hydroxyl groups and carboxyl groups which both affect the adsorption process.

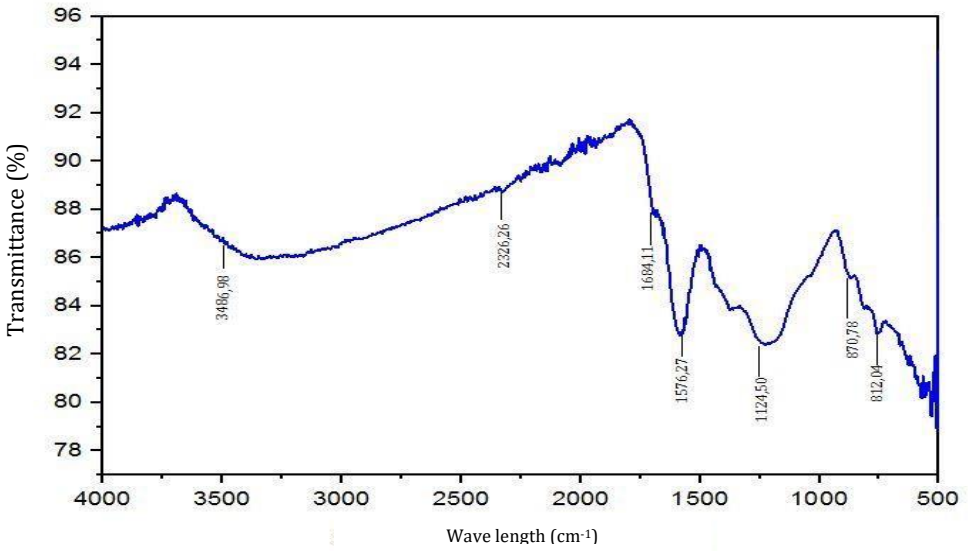


Figure 3. FTIR spectra of Matoa fruit peel charcoal after activation

Table 1. Functional Groups of FTIR Spectra Results on Matoa Fruit Peel Charcoal

Group	Wave Number (cm^{-1})	
	Charcoal Before Activated	**Activated Charcoal**
O-H	3457.43	3486.98 2326.26
C-H	3052.47 2162.27 1447.72 872.76	870.78 812.04
C=O	1682.01	1684.11
C=C	-	1576.27
C-N	1189.85	1124.50

Analysis of Morphology and Element Composition by using SEM-EDX

The surface shape of the matoa fruit peel charcoal was analyzed by using SEM. It was also analyzed by using EDX to find out the elements in the Matoa fruit peel charcoal. The results of SEM characterization on the charcoal surface
before and after activation can be seen in Table 2.

Charcoal after activation has a more regular pore structure. Meanwhile, before activation, the charcoal has irregular pores and there are still impurities. The activation process by using the HNO_3 activator dissolves impurities that still exist in the charcoal so that it will enlarge the pores by breaking the hydrocarbon bonds.

EDX testing was carried out to identify the constituent elements of carbon material. Figure 4 shows that the constituent elements in charcoal before activation are C 79.70%; Na 11.65%; Mg 1.82%; Al 0.15%; K 0.81%; Ca 4.45%; Cu 1.06%. While the constituent elements in activated charcoal consist of 99.21% carbon, Ca 0.29%, and Cu 0.50%.

A material with a high carbon content can adsorb more than a material with a lower one (Chen et al., 2021; Eze et al., 2022; Popoola, 2019). Figure 5 shows that the activated element consists of 99.21% carbon, Ca 0.29%, and Cu 0.50%. There are differences in the elemental composition before and after activation by using nitric acid on the Matoa fruit peel (Table 3). The elemental composition of carbon (C) dominates the Matoa fruit peel charcoal after activation. This is very influential on the adsorption process carried out because it shows the success of this activated charcoal synthesis process.

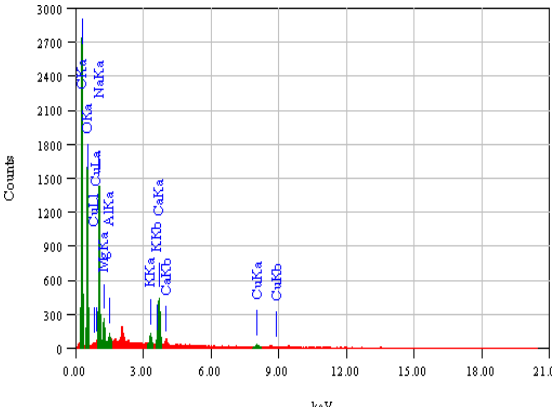


Figure 4. Elemental composition of activated charcoal EDX spectra before activation

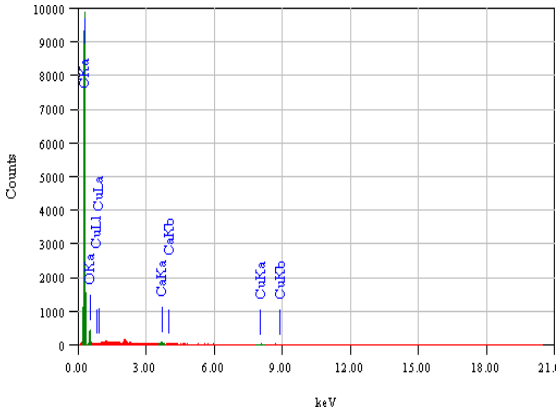


Figure 5. Elemental composition of activated charcoal EDX spectra after activation

Table 2. Comparison of morphological forms of charcoal

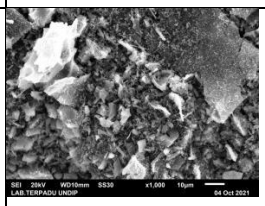
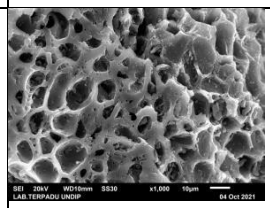
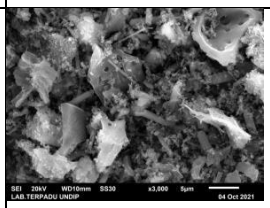
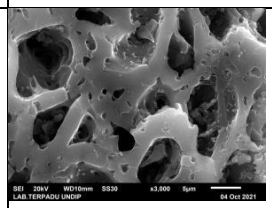
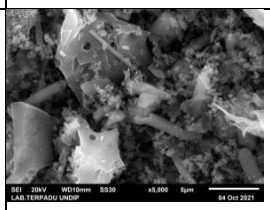
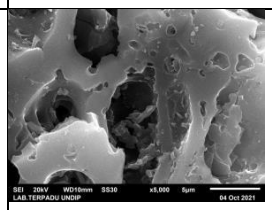
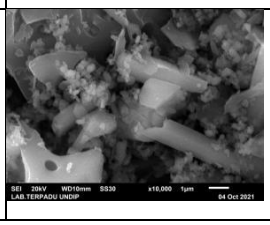
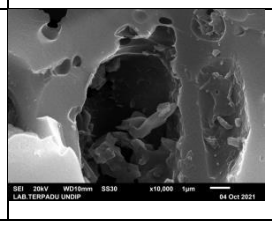
Zoom in	Charcoal before activation	After activation
1000x		
3000x		
5000x		
10000x		

Table 3: Charcoal mass content percentage (%)

Adsorbent Types	Element percentage (% mass)						
	C	Na	Mg	Al	KO	Ca	Cu
before activation	79.70	11.65	1.82	0.15	0.81	4.45	1.06
after activation	99.21	-	-	-	-	0.29	0.50

Determination of Optimum pH

At pH < 6, the Cd (II) ion species formed in the solution is Cd^{2+}, which causes competition between protons and the charge of Cd^{2+} on the carbon surface, which causes small adsorption of Cd (II) ions to occur (Wijaya & Ulfin, 2015).

According to (Wijaya & Ulfin, 2015)), at pH ≥ 8, Cd^{2+} ions will be well adsorbed, but at pH ≥8, there is not only an adsorption process but also a precipitation process in the solution (Table 4).

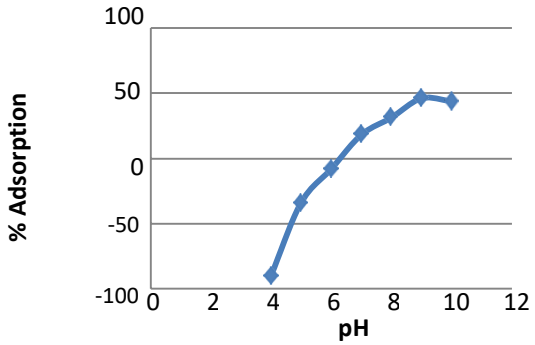


Figure 6. Graph of the pH Variation Effect on Adsorption Percentage

The effect of solution conditions on alkaline pH is the precipitation of hydroxy species such as $Cd(OH)^2$. In this study, the optimum pH was obtained at pH 9, but the decrease in Cd^{2+} ion levels at pH 9 was not only caused by the adsorption process but also the precipitation process.

Table 4 Variation of pH on adsorption capacity

pH	% Adsorpsi
4	-89.963
5	-33.933
6	-8.165
7	18.052
8	31.236
9	46.067
10	43.670

Determination of Optimum Contact Time

The optimum contact time for Cd adsorption determines to determine the ability of charcoal from matoa fruit peel activated by nitric acid to adsorb Cd metal. In this study, the variations used were 20, 40, 60, 80, and 100 minutes. The results of time contact variations are shown in Figure 7.

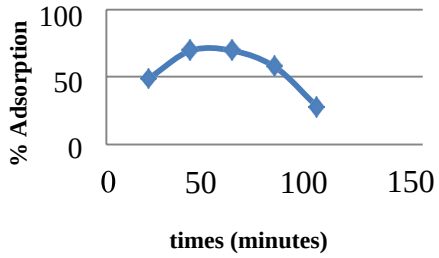


Figure 7. Graph of time variation in adsorption percentage

Based on Table 5, the optimum contact time in the adsorption of Cd (II) metal is 40 minutes, which shows the percentage of adsorption obtained at 69.49437%. At 60 minutes, the adsorption percentage decreased because the optimum time for adsorbent removal had been reached at 40 minutes.

Table 5. The effect of contact time on Adsorption Capacity of Cd(II)

Contact Time (minutes)	% Adsorption
20	48.6678
40	69.4943
60	69.2768
80	57.2594
100	27.7325

The contact time between the adsorbent and the adsorbate that exceeds the optimum contact time was caused by the desorption process and the weak interaction between metal ions and the adsorbent bound to the adsorbent surface (Pratomo et al., 2017). Desorption indicated that a dynamic equilibrium condition has been formed in the adsorption process. The desorption phenomenon was caused by the physical adsorption process. This process was reversible, resulting in the release of ions from the surface of the adsorbent into the wastewater solution.

A determination of adsorption kinetics was carried out to determine the rate of metal absorption that occurred. Determination of adsorption kinetics was carried out by making pseudo-first-order and pseudo-second-order adsorption kinetics curves by using a higher result of R^2 or close to 1. The adsorption kinetics of using nitric acid-activated Matoa peel as an adsorbent can be seen in Table 6. Based on the table, pseudo-second-order is more suitable than pseudo-first-order.

Table 6. Adsorption kinetics of Cd (II) with Matoa fruit peel adsorbent

Kinetics Adsorption	**Pseudo-first-order**			**Pseudo-second-order**		
Unit	Qmax (mg/g)	K_1 (min^{-1})	R^2	Qmax (mg/g)	K_1 (min^{-1})	R^2
Value	89.2857	$2.16x10^{-5}$	0.0262	0.0255	-1.7846	0.9642

This second-order reaction rate model can be used as a reaction rate model for Cd (II) metal adsorption. According to (Huang et al., 2014; Wang et al., 2021) if the kinetic model of adsorption is more suitable than the pseudo-second-order kinetic model, the adsorption that occurs is a chemical adsorption process (chemisorption). It is adsorption involving interaction between the adsorbent and the adsorbate so that the adsorbate cannot move freely to other parts.

Determination of the adsorption isotherm

Determination of the optimum concentration was carried out with several variations of the test: at concentrations of 10, 20, 30, 40, and 50 ppm. This test was carried out at an optimum pH of 9 and a contact time of 40 minutes. The results of this concentration variation can be seen in Figure 8.

Figure 8 shows that the optimum concentration condition is at a concentration of 20 ppm because it shows that the Cd (II) metal that can be adsorbed is 95.9557%. The adsorption isotherm is determined by changing the Langmuir and Freundlich isotherms into an equilibrium curve and determining the equilibrium model according to the regression value (R^2), which is closer to number 1.

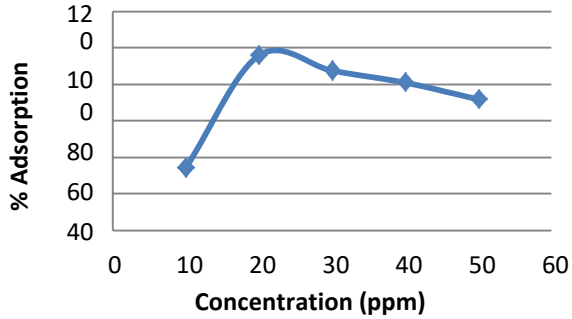


Figure 8: The graph of concentration variation on adsorption

The results of the adsorption isotherm by using activated charcoal Matoa peel can be seen in Table 7 that shows the higher regression value (R^2) is the Langmuir adsorption isotherm. Thus, the Langmuir adsorption isotherm model has the equation y = 2.6982x - 0.0299, and the regression value (R^2) of 0.9608 is more suitable for the adsorption process of Cd(II) metal on activated carbon of Matoa fruit peel.

Table 7. Cd (II) adsorption isotherm with matoa fruit peel as adsorbent

Isotherm Adsorption	**Langmuir**			**Freundlich**		
Unit	Qmax (mg/g)	K_l (min^{-1})	R^2	N	K_f (min^{-1})	R^2
Value	-33.445	-0.01108	0.9608	-22.676	0.4177	0.0088

Conclusions

The water content of activated charcoal from Matoa peel was 3.92 % less than the quality standard of SNI 06-3730-1995; the maximum limit was 15%. Iodine absorption was 507.64 mg/g with a minimum limit of 750 mg/g, and ash content was 1.17 % with a maximum limit of 10%. At a concentration of 20 ppm pH 9 as well as an optimum time of 40 minutes, the adsorption capacity of Cd(II) from activated charcoal of 1M nitric acid Matoa peel was 59.75 mg/g. The adsorption kinetics of activated carbon from Matoa fruit peel activated by nitric acid guided pseudo-second-order, and the adsorption isotherm guided the Langmuir adsorption isotherm with K_L= -0.01108, Q_{max}= -33.4448, and R^2=0.9608.

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