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KINETICS STUDY OF COPPER (Cu) AND CHROMIUM (Cr) ADSORPTION USING GREEN MUSSEL SHELLS (*Perna viridis*) ADSORBENT

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Abstract: Research has been carried out on making green mussel shell adsorbents to absorb Cu and Cr metals. The study of making green mussel shell adsorbents was carried out by thermal activation using a furnace. The results of the activated adsorbent were characterized using FTIR and SEM-EDX. Furthermore, the adsorbent was used to absorb Cu and Cr metal by using several variations, namely variations in the temperature of the adsorbent activation, adsorbent weight, pH, and contact time, and knowing the kinetic model by determining the reaction order. The results obtained from the characterization of green mussel shells contain elements of Ca, about 67.56% at an activation temperature of 500 °C and 67.34% at an activation temperature of 900 °C. The optimum conditions for Cu metal adsorption by green shell adsorbent were obtained at an activation temperature of 500 °C, using an adsorbent of 1 gram under pH 10 and a contact time of 40 minutes. Whereas for Cr metal absorption, the activation temperature was 900 °C, the mass used was 1.5 grams with pH 7 and the contact time was 40 minutes. The adsorption kinetics model of green mussel shells adsorbent on Cu and Cr metals followed pseudo-second-order.

Keywords: *Green Shells, Adsorption, Copper (Cu), Chromium (Cr)*

INTRODUCTION

Waste is a by-product of a production process, both industrial and domestic (household) which causes many problems for the environment. The entry of waste into the water environment might result in physical and chemical changes in water quality and a decrease in water quality caused by the presence of pollutants in the form of organic and inorganic components[1].

One of the uses of inorganic metal components, for example, heavy metals, concentrations of heavy metals whose presence in the environment exceeds the threshold, can certainly be protected because the toxicity of heavy metals is a threat to the health of living things [2]. Heavy metals often used in industry are Cu and Cr metals, especially in the electroplating industry. In a polluted environment, Cu metal is generally in the form of Cu^{2+} ions, and Cr metal is in the form of Cr^{3+} ions and Cr^{6+} ions [3]. Hexavalent chromium Cr (VI) is a class 1 carcinogen classified with multiple complex mechanisms that trigger cancer development [4]. The toxicity possessed by Cu metal will work and affect living organisms if it is in large quantities or exceeds the tolerance value of these organisms, which can cause symptoms of kidney, liver, anemia, and vomiting and can cause sufferers to die [5]. Based on the Regulation of the Minister of Environment of the Republic of Indonesia No. 5 of 2014 concerning Wastewater Quality Standards, it is stipulated that the limit for copper and chromium metals in wastewater should not exceed 0.5 mg/L because waste containing Cu and Cr metals should be treated first before being discharged into the environment.

Adsorption is a method that is widely used because this method is safe, does not provide side effects that endanger health, and does not require complicated and inexpensive equipment. The first step to an effective adsorption process is choosing an adsorbent with high selectivity and capacity[6]. The adsorbent used can be in the form of disposal or utilization of biological waste such as mussel shells, where the shells contain 50-90% calcium carbonate (CaCO3) compounds, 0.69% silica dioxide (SiO2), 0.64% magnesium oxide (MgO), sodium oxide (Na2O) 0.98% and sulfite (SO3) 0.72%. Calcium carbonate is known to be a polar adsorbent to support green mussel shells' function as a promising adsorbent (CaCO3). CaCO3, which undergoes the calcination process, will produce calcite (CaO). The resulting calcite can be used as an adsorbent to adsorb heavy metals[6], [7]. Shellfish production in Indonesia from 2002 to 2006 increased successively, namely by 7 tons, 2,869 tons, 12,991 tons, 16,348 tons, and 18,896 tons [8]. Therefore, using green mussel shells as an adsorbent is quite relevant to increase economic value and reduce environmental burden[9]

RESEARCH METHODS

The research was conducted at the Laboratory of the Department of Chemistry, Faculty of Mathematics and Natural Sciences, Islamic University of Indonesia.

Instruments

This research used: analytical balance, laboratory glassware, magnetic stirrer, scanning

electron microscopy energy dispersive X-Ray (SEM-EDX) testing equipment, atomic absorption spectrophotometer (AAS), Fourier Transform Infra-Red (FTIR), and supporting equipment, namely desiccator, oven, furnace, 150 mesh sieve, and shakers.

Materials

The materials used in this study were distilled water, green mussel shells, pH buffer, $CuSO_4$, and K_2CrO4. (Merck, Germany).

Methods

1. Preparation of Green Mussel Shell Adsorbent

The shells were washed with water several times until they were clean, after which they were allowed to air dry. The shells were reduced in size by pounding them, after which they were calcined using a furnace at temperatures 300, 500, 700, and 900 °C for 5 hours. The calcined mussel shells were put in a desiccator for 1 hour; after that, the mussel shells were mashed and sieved using a 150 mesh sieve.

2. Characterization

The results of the green mussel shell adsorbent at temperatures 300, 500, 700, and 900 °C were then characterized by FTIR to determine the functional group and by SEM-EDX to determine the morphology and elements contained in the green mussel shell.

3. Study Adsorption

a) Determination of Activation Temperature on the Adsorption
Weighed 0.05 grams of green mussel shell adsorbent that had been calcined at temperatures of 300, 500, 500, 700, and 900 °C was put into a beaker containing 10 ml of Cu and Cr metals with a concentration of 25 ppm each. The mixture was shaken at room temperature for 15 minutes. Then the mixture was filtered through filter paper. The solution was analyzed using atomic absorption spectrophotometer (AAS).

b) Determination of the Optimum Mass of Adsorbent
The adsorbents of green mussel shells were weighed at the optimal temperature of 0.01, 0.5, 1, and 1.5 grams. It was put into each glass beaker containing 10 ml of Cu and Cr metals, each with a concentration of 25 ppm. The solution was shaken at room temperature for 15 minutes. Then the mixture was filtered using filter paper. After that, the solution was analyzed using atomic absorption spectrophotometer (AAS).

c) Determination of Optimum pH
The optimum pH is the pH state where the adsorbed concentration is greatest—prepared each Cu and Cr metal solution as much as 10 ml with a concentration of 25 ppm. A buffer solution of each variation was added, namely pH 4, 7, and 10. Then an adsorbent with optimum temperature and mass was added for each variation and stirred for 15 minutes. Furthermore, the mixture was filtered using filter paper, and the filtrate obtained was analyzed by atomic absorption spectrophotometer (AAS).

d) Determination of Optimum Contact Time
Prepared green mussel shell adsorbent at the optimum temperature and mass, put into a glass beaker containing 10 ml of Cu and Cr metals, each with a concentration of 25 ppm and an optimum pH. The mixture was shaken at room temperature with time variations of 5, 10, 20, 30, 40, 45, 60, and 90 minutes. Furthermore, the mixture was filtered using filter paper. The solution was analyzed using an atomic absorption spectrophotometer (AAS).

RESULT AND DISCUSSION

The shells' activation at various temperatures of 300, 500, 700, and 900 °C produced different characteristics, which can be seen in terms of the resulting color and shape.

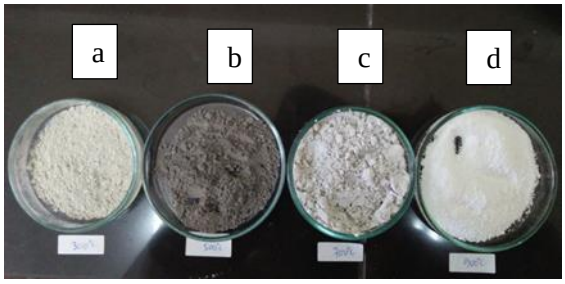

Figure 1. Results of activation of green mussel shells with variations in temperature of (a) 300 °C, (b) 500°C, (c) 700 °C and (d) 900 °C

The results of the shell furnace at an activation temperature of 300 °C have brownish-white characteristics but are almost the same as before heating and have the form of fine granules. Due to the calcination-carbonation process has not occurred and there has not been a change in the shell composition, the color of the carbonized product is still the same as before heating. At activation temperatures of 500, 700, and 900 °C, they have blackish ash characteristics and are different in color from before heating. The physical changes experienced by shell powder

occur because, during the physical activation process, carbon dioxide is released on carbon [10]. The calcination process in mussel shells decomposes $CaCO_3$ compounds into CaO and CO_2 compounds. The reaction from endothermic calcination is as follows [11]:

$$CaCO_3\ (s) \longrightarrow CaO\ (s) + CO_2\ (g)$$

Characterization

1. Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR analysis was carried out to identify the presence of functional groups in the green mussel shell by looking at the absorption peaks in the IR spectra.

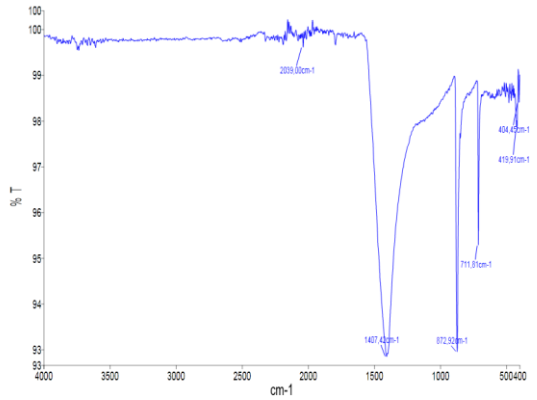

Figure 2. Result of FTIR spectra of green mussel shell in temperature 500 °C.

The results of the spectra contained in the green mussel shell activated at a temperature of 500 °C. The absorption band at wave number 1407.42 cm-1 indicates the presence of a group (C=O), and wave number 872.92 cm-1 indicates an aromatic alkene group (C-C). While the results of the adsorbent after adsorbed Cu metal showed a reduction in the peak and an increase in the absorption peak. It can be seen at 1795.54 cm-1 (C-H group), and a shift in wave number occurred at 700-400 cm-1 Ca-O absorption.

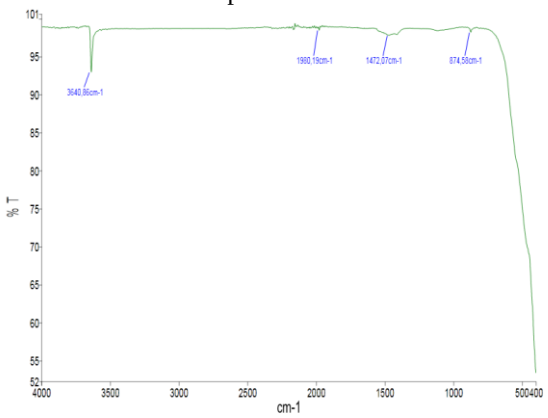

Figure 3. Result of FTIR spectra of green mussel shell in temperature 900 °C.

Meanwhile, the spectra of green mussel shells activated at a temperature of 900 °C (Fig 3.). The spectra obtained from green mussel shells showed the presence of an absorption band at a wave number of 3640.86 cm-1, which is the –OH group that functions to attract positive ions. The negatively charged –OH group will attract positively charged metal ions so that the metal ions can be absorbed [12]. Wave number 1472.07 cm-1 indicates the presence of vibrations from the C-O bending, and wave number 874.58 cm-1 indicates the group (C-C). While the results of the spectra of the green mussel shell adsorbent after adsorption showed there was a shift in the wave number at the absorption of 700-400 cm-1 (Ca-O).

2. Scanning Electron Microscope Energy Dispersive X-Ray (SEM-EDX)

Characterization using SEM-EDX aims to see the morphological structure of the adsorbent. Figure 2 shows an SEM image of a green mussel shell activated at 500 °C. It can be seen that the morphology of the green mussel shell samples showed regular clumps of the same size. The bright color that appears more dominant in the green mussel shell sample is a constituent element with a high atomic number. In contrast, the dark color on the sample's surface is a constituent element with a low atomic number.

Based on the results of the EDX analysis (Figure 4) for shells activated at a temperature of 500 °C, more varied elements are contained therein. More elements are calcium (Ca) 67.56%, and for the rest, there are other elements such as O by 41.27%; C 25,355; N 4.78%; Br 0.55%; Na 0.43% ; P 0.4%; Mg 0.36%; Si 0.33% and Al 0.25%. The remaining elements in the adsorbent are the content of the green mussel shell itself.

The morphology of the green mussel shell activated at a temperature of 900 °C. From Figure 3 (Figure 5.a), it can be seen that the sample's morphology is in the form of bulk or irregular lumps of varying sizes. The bright color more dominant in the green mussel shell sample is the constituent elements with a high atomic number. In contrast, the dark color is the constituent element with a low atomic number.

The EDX results (Figure 5.b) from green mussel shells were activated at a temperature of 900 °C, from the picture shows that the more dominant element is calcium (Ca), 67.34%. The remaining content contained in the shells was 44.39% O, C 20.29%, N 10.8%, and Na 0.96%. Minor elements in activated carbon, namely O, C, N, and Na, are the content derived from green mussel shells.

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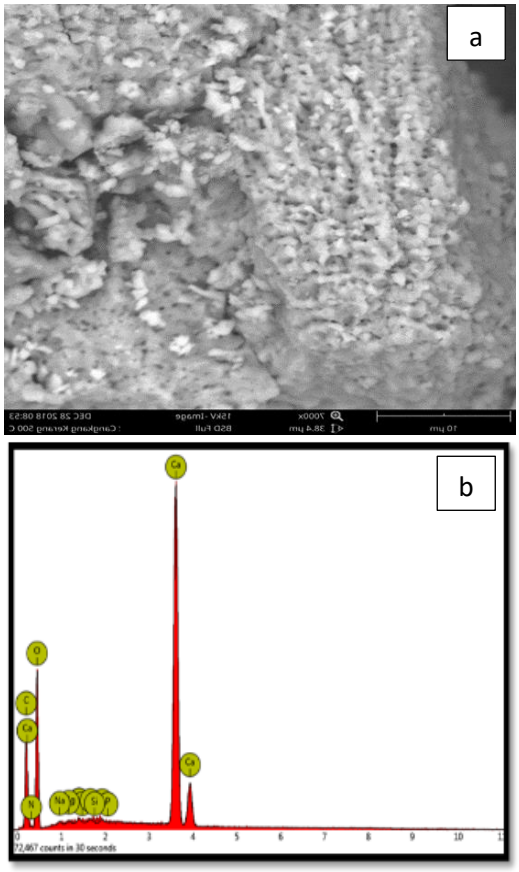
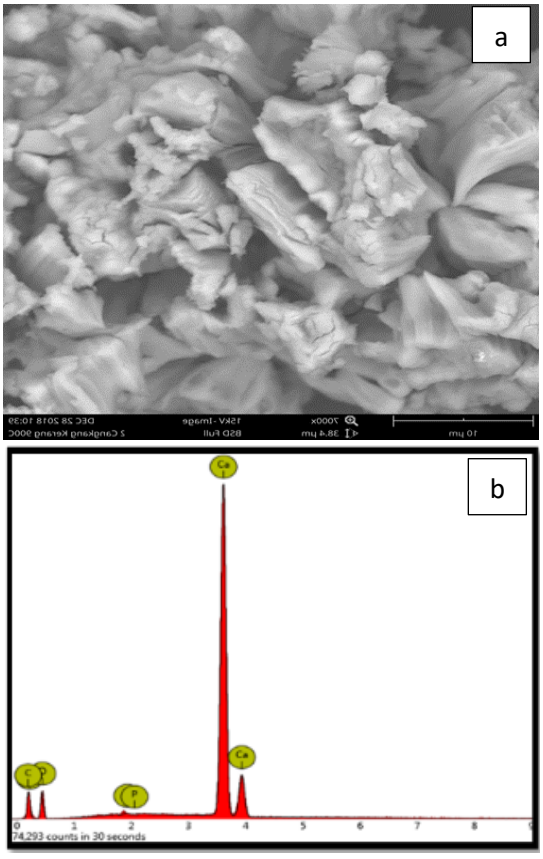


Figure 4. (a) Morphology and (b) EDX analysis result of green mussel shells at an activation temperature of 500 °C



(a) (b)

Figure 5. (a) Morphology and (b) EDX analysis result of green mussel shell at activation temperature of 900 °C

Study Adsorption

1. Determination of Activation Temperature on the Adsorption

Activation of green mussel shells aims to remove non-carbon compounds so that pure carbon elements are obtained, enlarge pores by breaking hydrocarbon bonds or oxidize surface molecules so that carbon changes in properties, namely increasing surface area and increasing adsorption absorption. Based on the analysis that has been carried out, it was found that the effect of the activation temperature of the adsorbent on the adsorption process of Cu and Cr metals was obtained. The amount of Cu adsorbed at each activation temperature variation of the green mussel shell adsorbent is the temperature (300 °C) 0.71815 mg/L; (500 °C) 0.94981 mg/L; (700 °C) 0.93051 mg/L and (900 °C) 0.86229 mg/L. while the amount of Cr adsorbed at the activation temperature variation of the green mussel shell adsorbent was (300 °C) 14.4295 mg/L; (500 °C) 14.5638 mg/L; (700 °C) 14.8322 mg/L and (900°C) 14.9664 mg/L. The percentage of Cu and Cr, metal absorption data, was obtained to clarify the optimum state of activation temperature of the green mussel shell adsorbent on the adsorption of Cu and Cr metals by looking at the curve of the relationship between % Adsorption and activation temperature as shown in Figure 6.

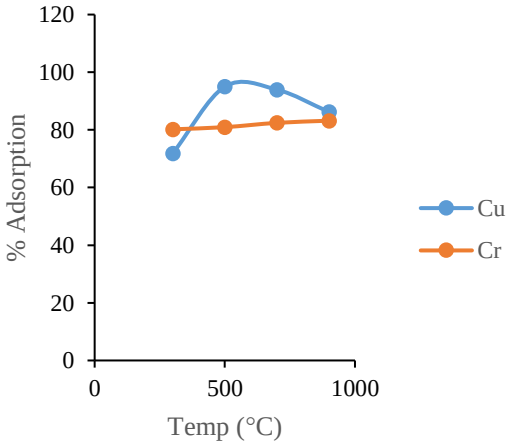


Figure 6. The curve for determining the optimum activation temperature

Green mussel shell adsorbent, which was activated at 300 °C, had a low absorption capacity for metals Cu 71.814% and Cr 80.194% compared to temperatures of 500, 700, and 900 °C. Based on Figure 5.10, the absorption capacity of green mussel shell ash to Cu metal activated at 500 °C has a high absorption capacity of 94.908%. Still, after being activated at 700 °C

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and 900 °C, the absorption decreases, namely 93.951% and 86,229 %. Meanwhile, the absorption of green mussel shell ash to Cr metal at the activation temperature of 500, 700, and 900 °C was 80.939%, 82.431%, and 83.177%, which indicates that the higher the activation temperature, the more Cr metal is adsorbed. It shows that the higher the activation temperature, the more pores open to form a cavity larger in size than Cr metal so that the Cr metal molecule enters the green mussel shell, which is activated at a temperature of 900 °C.

It shows that the higher the activation temperature, the more open pores forming larger cavities than Cr metal, so the metal enters the green mussel shell cavity. In addition, the loss of water compounds in the adsorbent pores causes an area in which the pore surface area increases so that the ability of the adsorbent to adsorb compounds improves [13].

2. Determination of the Optimum Mass of Adsorbent

The optimum mass is where the adsorbent could adsorb most of the adsorbate. Based on the analysis carried out, the results of the determination of the effect of the mass of the green mussel shell adsorbent on the adsorption of Cu and Cr metals are obtained. The amount of Cu metal adsorbed on each mass variation of the green mussel shell adsorbent was 0.05 g, 0.5 g, 1 g, and 1.5 g, respectively 0.73816 mg/L; 0.0688 mg/L; 0.04415 mg/L and 0.02553 mg/L. Meanwhile, the amount of Cr adsorbed on each mass variation of the green mussel shell adsorbent was 0.05 grams, 0.5 grams; 1 gram and 1.5 grams respectively 0.80805 mg/L; 0.09195 mg/L; 0.05768 mg/L and 0.03199 mg/L. These data show that the more ash mass of green mussel shells, the metal concentration of the waste decreases; this is due to the activation process of green mussel shells, which aims to increase the size and distribution of pores and expand the surface of the adsorbent. So the more CaO is directly proportional to the more metal absorbed. To see the relationship curve between the adsorbent's mass and the presentation of the adsorbed Cu and Cr metals.

The results of the mass variation of the adsorbent to adsorb Cu and Cr metals. For Cu metal, the adsorbent mass of green mussel shells is 0.05 grams; 0.5 grams; 1 gram, and 1.5 gram adsorption percentage in each variation is 77.502%; 83.150%; 99.466%, and 86.288%. In comparison, the percentage of adsorbent in each variation for Cr metal is 81.511%; 92.754%; 96.189%, and 96.8144%. The data above shows that the highest percentage of adsorption for Cu metal is 99.466% which indicates the optimum mass used to adsorb Cu metal is 1 gram. It is because, at a higher weight, the number of metal ions in the solution is not proportional to the amount of CaO available. Hence, the optimum mass for Cu metal adsorption is only 1 gram. Meanwhile, the largest percentage for Cr metal is 96.814%, where the optimum mass used is 1.5 grams; according to the theory, the more adsorbents used, the higher the percentage produced. The adsorbent mass used for the adsorption of Cu and Cr metals is different because it is influenced by several factors, namely electronegativity, solubility, cationic size, adsorbent affinity, etc. [8, 14].

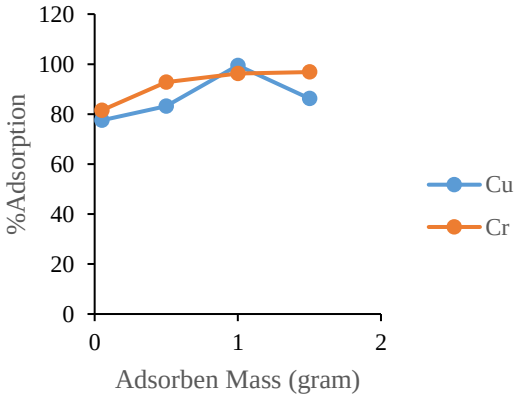

Figure 7. Determining the optimum adsorbent mass

3. Determination of Optimum pH

The acidity condition of the metal ion solution affects the percent absorption, so it is necessary to determine the optimum pH [15].

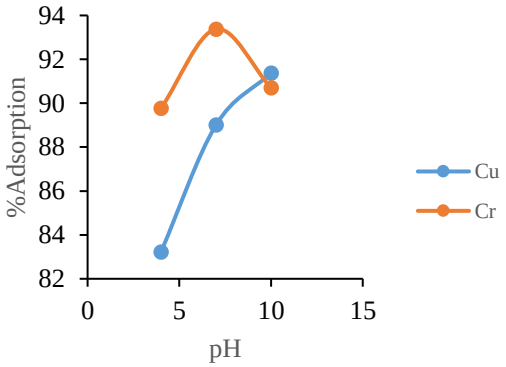

Figure 8. Optimum pH determination curve

Figure 8 shows that at pH 4, the absorption of Cu and Cr metals is relatively low because the surface of the adsorbent is surrounded by H+ ions, so it is suspected that there is competition between H+ ions and metal ions, which can prevent metal interactions with active groups present on the surface. Green mussel shell adsorbent. However, at pH 7 and 10, there was an increase in Cu metal at 89.006% and 91.376%, respectively. It is because metal ions work at pH 7 and 10, and the reaction of Cu metal at alkaline pH formed copper ions (Cu^{2+}). Judging from the HSAB principle, which

explains that Cu (II) ions are classified as intermediate acids, meanwhile, the adsorbent itself has OH- and CO functional groups, which are classified as hard bases, which means Cu (II) ions will interact well with the adsorbent so that it will form a cluster. $Cu(OH)_2$.

Meanwhile, Cr metal increased at pH 7 93.375% and decreased at pH 10 90.702%. At pH 10, there was a decrease. This was because at pH 10, the number of OH- ions in the solution increased, and Cr had precipitated first as the hydroxide $Cr(OH)_2$. This situation probably occurs because the higher the pH, the more alkaline it will be, so the solubility of Cr will decrease, and precipitation will begin. This results in a reduction in the number of Cr ions absorbed by the adsorbent surface [16], [17].

4. Determination of Optimum Contact Time

Optimum contact time is used to determine how long it takes to achieve optimum absorption of Cu and Cr metals. The results obtained from the calculation of the amount of Cu metal in contact time variations of 5, 10, 20, 30, 40, 45, 60, and 90 minutes are 0.03977 mg/L; 0.04231 mg/L; 0.04513 mg/L: 0.0457 mg/L; 0.04584 mg/L; 0.0457 mg/L; 0.04104 mg/L and 0.04203. While the results of the calculation of the amount of Cr absorbed for each variation are 0.03423 mg/L; 0.03604 mg/L; 0.03694 mg/L; 0.03739 mg/L; 0.04009 mg/L; 0.03874 mg/L; 0.03829 mg/L and 0.03739mg/L. % Adsorption data is used to clarify the optimal state of the given contact time variation by looking at the curve of the relationship between contact time and % adsorption of Cu and Cr metals, as shown in Figure 7.

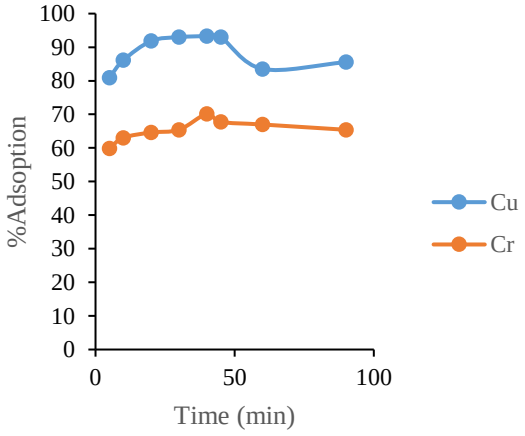

Figure 9. Determining the optimum contact time

The adsorbed Cu and Cr metals increased with increasing contact time. The adsorption presentation of green mussel shell ash in the adsorption of Cu metal at various contact times of 5, 10, 20, 30, 40, 45, 60, and 90 minutes was 80.941%; 86.107%; 91.848%; 92.996%; 93.283%; 92.996%; 83.524% and 85.533%. In comparison, the percentage of the amount of Cr metal that was absorbed in each variation was 59.889%; 63.041%; 64.617%; 65.405%; 70.134%; 67.769%; 66.981%, and 65.405%. At the time of 5, 10, 20, and 30 minutes, there is an increase in absorption every minute. It shows that the empty spaces of the adsorbent are filled with metal, and the active groups on the adsorbent have yet to interact optimally, so an equilibrium state has not been obtained. At the contact time of 40 minutes, an equilibrium occurred between the adsorbent and the adsorbate (metal), so the optimum adsorption occurred with absorption for Cu metal at 93.283% and Cr metal at 70.134%.

Meanwhile, at 45, 60, and 90 minutes, it decreased. The longer contact time between the adsorbent and the adsorbate allows for an increase in metal absorption, but if it is too long, it can reduce the absorption rate. The longer the contact time can cause desorption, namely the release of metal bound by the adsorbent[17]. The adsorption process can experience equilibrium, so further addition of contact time does not significantly affect metal absorption. The physical contact between the metal and the adsorbent causes the metal to be released into the solution over time[18], [19]. Physical contact between the metal and the adsorbent causes the metal to be released into the solution over time. This results in a larger amount of metal being measured, indicating a decreased absorption capacity. Therefore, a period of time is needed to reach the equilibrium state of the metal with the adsorbent [20].

Adsorption Kinetics

Adsorption kinetics is used to determine the rate of absorption that occurs on the adsorbent to the adsorbate and is influenced by time. The contact time required to reach adsorption equilibrium is used to measure the adsorption rate. This study determined the adsorption rate by estimating the reaction order. The reaction order of the rate of a chemical reaction or chemical process is defined as the rate at which a reaction occurs. So from the adsorption kinetics data obtained, it can be understood the dynamics of the adsorption process are based on the reaction order. The adsorption kinetics of Cu and Cr metals by green mussel shell ash was evaluated based on the pseudo-first-order reaction equation and the pseudo-second-order reaction equation. This study used contact time variations of 5, 10, 20, 30, 40, 45, 60, and 90 minutes to determine the order used and look at the values of R_2, k1, and qe_2, which are close to 1. Based on the comparison data, information is obtained that in this study, each metal tends to approach the pseudo-second-order. This is evidenced by the values of R^2, k1, and qe2, which are closer to the value of 1, namely for R^2, k1, and qe_2 for Cu

metal by 1; 22.9885 min-1 and 1 mg/g. Meanwhile, for Cr metal, the values of R^2, k1, and qe2 are 0.9984, 0.00175 min-1, and 0.0379. So the adsorption kinetics model for green mussel shells for the adsorption of Cu and Cr metals follows a pseudo-second-order kinetic model where the results are the same as in previous studies. Adsorption kinetics model for Cu(II) metal adsorption using chitosan from green mussel shells, namely pseudo-second-order pseudo adsorption kinetics with R^2, k1 and qe_2 values of 0.906; 0.472 min-1 and 4.854 mg/g[21-22].

Table 1. Data from the calculation of adsorption kinetics

Metals	Adsorption Kinetics					
	Pseudo first order			Pseudo second order		
	$qe_{1\ (mg/g)}$	$k_{1\ (min-1)}$	R_1	$qe_{2\ (mg/g)}$	$k_{2\ (min-1)}$	R_2
Cu	0.0020	0.0094	0.0088	1	22.9885	1
Cr	0.0076	-0.0069	0.0085	0.0379	0.00175	0.9984

CONCLUSION

Green mussel shells can be used as adsorbents because green mussel shells contain CaCO3, which is orientated with a lot of Ca elements. The Ca content produced 67.56% at an activation temperature of 500 °C and 67.34% at an activation temperature of 900 °C. The results obtained for the adsorption of Cu metal activation temperature 500 °C 94.908%, mass 1 gram 99.466%, pH 10 91.376%, and contact time 40 minutes 93.283%. Meanwhile, for Cr metal, the activation temperature is 900 °C 83.117%, the mass of 1.5 grams is 96.814%, PH 7 is 93.375%, and the contact time is 40 minutes 70.134%. Adsorption of green mussel shells on Cu and Cr metals followed a pseudo-second-order pseudo-kinetic model. Where for Cu metal with $R^2 = 1$, while for Cr metal $R^2 = 0.9984$.

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