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Effect of Weight and Contact Time Adsorption of Activated Carbon from Coal as Adsorbent of Cu(II) and Fe(II) in Liquid Solutions

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Abstract. Experiments have been done for preparation activated carbon with low-rank coal as raw materials taken from PT. Bukit Asam TanjungEnim, Mining unit in South Sumatra, Bangko mining site. This research aims to apply coal activated carbon as an adsorbent for heavy metals Cu (II) and Fe (II) in solution. Chemical activated uses 40% $ZnCl_2$ with 60% coal at 28 mesh, and carbonization was carried out at a temperature of 500°C for 2 hours. Preparation activated carbon from coal aims to see its potential as a metal adsorbent in a liquid solution. The activated carbon was distinguished by iodine number and nitrogen adsorption BET. The iodine number was 1,393 mg/g, the surface area of this activated carbon is 512 m^2/g with a pore volume of 0.297 mL/g and pore diameter of 11.5Å. The low-rank coal activated carbon was used for the adsorption of Cu(II) and Fe(II) ions by using variations in the weight of activated carbon 600, 900, 1,800, 2,400 and 3,000 grams and variations in contact time 3, 6, 9, 12 and 15 hours. The results showed that a maximum removal percentage for Cu(II) 99.88% was obtained for 3,000 grams of activated carbon with a contact time of 15 hours, and maximum removal percentage for Fe(II) 96.24% was obtained for 3,000 grams of activated carbon and 12 hours of contact time. The study produced micropore activated carbon with a large surface area and effectiveness for metals removal. Based on Freundlich isotherm, obtained that adsorption capacity of Cu(II) and Fe(II) were 1.967 and 1.829 mg/g. Based on Langmuir isotherm, obtained that adsorption capacity of Cu(II) and Fe(II) were 0.6277 and 0.955 mg/g.

Keywords: Coal activated carbon, Contact time, adsorber mass, Cu(II) adsorption, Fe(II) Adsorpstion.

INTRODUCTION

Over the last decades, the pollution of water resources has been causing worldwide concern due to the careless disposal of heavy metals. It is well known that some metals are toxic or have harmful effects on living beings. Some of these are copper (Cu) and iron (Fe) which are highly toxic to humans and ecological environment.This problem has received a lot of attention in the last few years.Wastewater from many industries, such as metallurgical, chemical manufacturing, and mining contains these heavy metals. It, therefore, becomes necessary to remove these heavy metals from these wastewaters by appropriate treatment before releasing them into the environment. The wastewater treatment method that will be investigated in this study is adsorption by coal activated carbon

Activated carbon is widely used in drinking water treatment. It is effective in removing chlorinated compounds, complications caused by taste and odor, and many metals. It has the strongest physical adsorption force and the highest volume of adsorption porosity; thus, it has an exceptionally high surface area for adsorption contaminants. Activated carbon is prepared from materials such as diamonds, woods, coconut and walnut shells, and coal[1] [1]. The activated carbon is acknowledged to have low cost, well developed pore structures, and high adsorption capacity. Indonesia has coal reserves of around 37 billion tons in 2018 (Ministry of Energy and Mineral Resources, 2018). The abundance of low-rank coal can be used as a potential low-cost precursor for the activated carbon production.

Making activated carbon with bagasseraw material by CO_2 activation method produces iodine number 730 mg/g [2], [3], [4].Physics activation methods have also been carried out using water vapor with biomass[5] and with peat soil as raw material to produce activated carbon with iodine number 686 mg/g [6]. Physical activation methods using water vapor have also been carried out in the manufacture of activated carbon with Betung

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bamboo as raw material producing iodine number 379 mg/g [7]. While the activation using ozone was carried out by Sugashini [8], and also byAnggarini [9] who used coconut shell raw material to produce activated carbon with iodine number 1,055 mg/g. ZnCl2 activator has been used in making coal-based activated carbon by Monika [10] produced activated carbon with iodine number 1,198 mg/g, and by Suliestyah et al. [11] produced an iodine number of 1,298 mg/g. Activation with the combination of H3PO4-NH4HCO3 with coal raw material has been carried out by Kusdarini et al. [12], producing activated carbon with iodine number 1,238 mg/g. Activation using ZnCl2, K2CO3, NaOH and H3PO4 with coal raw material has been carried out by Kiliç [13], and using NaOH has been carried out by Hardianti et al. [14]

The use of activated carbon as an adsorbent for heavy metals in liquid waste has been tested in various studies, including as an adsorbent for Fe metals [15], Mn and Ag metals [16], and Cu metals [17]. The use of activated carbon as an adsorbent of heavy metals in liquid waste has been tested in various studies, including as an adsorbent [18], which has carried out adsorption studies of Fe, Mn and Al in industrial wastes, as well as [1], [18], [19] has also adsorbed activated carbon against Fe and Mn in solution. The study of the influence of contact time and the weight of the adsorbent in Mn metal adsorption was carried out by Yanou et al. [20]This paper will study the effect of activated carbon weight and contact time on the adsorption of Cu(II) and Fe(II) in liquid solutions. The activated carbon used is made from low-rank coal with a chemical activation method ($ZnCl_2$).

MATERIALS AND METHODS

The method in this study used laboratory experiments. The work scheme starts from the characterization of coal as a raw material for making activated carbon. Followed by the process of synthesis of activated carbon and thecharacterization of activated carbon products, the application of activated carbon as adsorbent of heavy metals Cu (II) and Fe (II) in solution.

Materials Preparation

Low-rank coal was collected from PT Bukit Asam Tanjung Enim South Sumatra. The sampling location was at the Bangko Pit mining pit 1 Layer A2. A constant weight was obtained by timing up the drying process. After drying, a high-speed rotary cutting mill was used to ground the raw material and then calorific value and proximate analysis were carried out. For water content analysis using the ASTM D 3173 method, ASTM D 3174 for ash content, ASTM D 3175 for volatile matter, ASTM D 3172 for fixed carbon, and ASTM D 5865 calorific value. Activated carbon was made with the composition ZnCl2 40% - coal 60% with 28 mesh coal grain size as raw materials for making activated carbon.

Preparation of $ZnCl_2$Treated Coal Carbon

Chemical activation agents was used to directly impregnated the raw material. Ground and sieved *low-rank coal* were treated with $ZnCl_2$ in weight ratios of 40% at room temperature. Magnetic stirrer was used to maintain the continuous mixing of the precursor with chemicals for one hour. After mixing, prepare impregnated samples by drying the solutions at room temperature for 24 h and then drying at 85°C for 72 hours in a temperature controlled oven. After this period, impregnated samples were ready for the carbonization and activation process which were carried out simultaneously. Carbonization was carriedout at 500°C for 120 minuts under the N_2 flow at a heating rate of 5 °C/min.

Characterization of Coal Activated Carbon

By using BET, the surface area of each activated carbon was calculated from N_2 adsorption. The same adsorption data was also used for calculation of the micropore volume. Analysis of Iodine Numbers is carried out based on SNI 06-3730-1995 [21]. Determination of adsorption of iodine was carried out using activated carbon that had been heated in the oven, weighed as much as ± 0.5 g and put into Erlenmeyer. The sample was given an iodine 0.1 N solution of 50 mL, stirred using a shaker for ± 15 minutes, and left for 15 minutes. Next, 10 mL of filtrate is taken and titrated with a solution of 0.1 N. $Na_2S_2O_3$. If the yellow color of the solution is faint, 1 mL of 1% starch solution is added. Titration is done again until the blue color disappears. Repeat three times to determine the iodine adsorption.

$$Iodine\ Number\ (mg/g) = \{(V1N1 - V2N2)X\ 126{,}9\ \times 5\}/W \quad (1)$$

with V1 is the iodine solution analyzed (mL), N1 is iodine normality, V2 is the required thiosulfate solution (mL), N2 is the normality of sodium thiosulfate, and W is the weight of activated carbon.

Adsorption Studies

The experiments used analytical grade iron (II) sulfate ($FeSO_4$) $_2 \cdot 7H_2O$) and copper (II) nitrate ($Cu(NO_3)_2 \cdot 5H_2O$) reagents from J.T. Baker. Deionized water was used to prepare stock solutions f metal ions. Atomic absorption spectrometry (AAS) determined the ion concentrations of metal solution. The adsorption capacity of Cu(II) and Fe(II) on activated carbon was determined by carrying out the batch test. The test was c ducted using dose variation of activated carbon and contact time variation for Cu(II) and Fe(II) respectively with 200 ml of aqueous solution in a beaker and sheet at 150 rpm in room temperature. After reaching the ired contact time, the Atomic Adsorption Spectrometer (AAS) collected and analyzed the aqueous solution for the residual concentration remained.

RESULTS AND DISCUSSIONS

The results of the study included coal rank, the characterization of the activated carbon from the experiment, the effect of the dose of activated carbon and contact time on adsorption of Cu (II) and Fe (II) metals, and Freundlich and Langmuir adsorption isothermal studies.

Coal Rank

The quality of the coal samples tested included proximate analysis and calorific value,as seen in **TABLE 1**. Based on ASTM D388, it can be concluded that coal from Bangko - PT. Bukit Asam Coal Mine TanjungEnim Mining unit used as the raw material in this study is ranked Sub Bituminous A [22], [23].

TABLE 1. Results of Proximate Analysis, calorific value, and iodine number of Coal

Inherent Moisture (%, ADB)	Ash Content (%, ADB)	Volatile Matter (%, ADB)	Fixed Carbon (%, ADB)	Calorific Value(Kal/g)	Iodine Number (mg/g)
11.7	0.78	35.47	52	5,619	347

Characterization Result of Activated Carbon

Iodine number is a parameter commonly used to measure the adsorption of activated carbon expressed in units of mg/g. The activated carbon measurements of iodine number, surface area, pore volume, and pore diameter, can be seen in **TABLE 2.**

TABLE 2. The character of activated carbon.

Coal grain size (mesh)	Iodine Number (mg/g)	Surface Area (m^2/g)	Pore Volume (mL/g)	Pore Diameter (Å)
28	1,393	512	0.297	11.48

TABLE 2shows that the active carbon results of the study have high adsorption with iodine number 1,393 mg/g, which is higher than the coal raw material (437 mg/g at **TABLE 1).** In the carbonization process, the volatile matter in the sample will continue to decrease. This process will increase the number of pore structures and form new porosity, thereby increasing surface area.With the increasing carbonization temperature, the volatiles from the samples continue to evolve. Surface area increases because the devolatilization process further develops the pore structure and creates new porosities. From **TABLE 2** proven that $ZnCl_2$ is a very effective reagent for making high adsorbent activated carbon with a wide surface [24].

This activated carbon has an iodine number higher than the minimum standard of 750 mg / g, also has a rge surface area of 512 m2 / g. According to [25] there are three sizes of active carbons pori, namely micropore (<2 nm), mesopore (2 nm - 50 nm), and macropore (> 50 nm). The activated carbon from this study has a diameter of 11.48 Å, which is equivalent to 1.148 nm, including micropore. This result iodine number can be compared with other research groups who conducted a study on making activated carbon from coal. As

compared to the study done by Monika [10], Kusdarini et al [12] , Hardiyanti,et al [14] , and Suliestyah,et al [11] , which had iodine number 1,198 mg/g, 1,238 mg/g, 562.5 mg/g, and 1,298 mg/g, ours had 1,393 mg/g.

Effect of Adsorbent Dosage and Contact Time to Removal of Cu (II)

To design the optimum treatment systems, a series of batch experiments were conducted with the adsorbent dosage of 600, 1,200, 1,800, 2,400, and 3,000 mg/200 mL of test solution with the contact time of 3, 6, 9, 12, and 15 hours, at initial pH 2. Effect of adsorption dosage contact time on removal percentage of Cu (II) was studied and the results are shown in Fig. 1a and 1b.

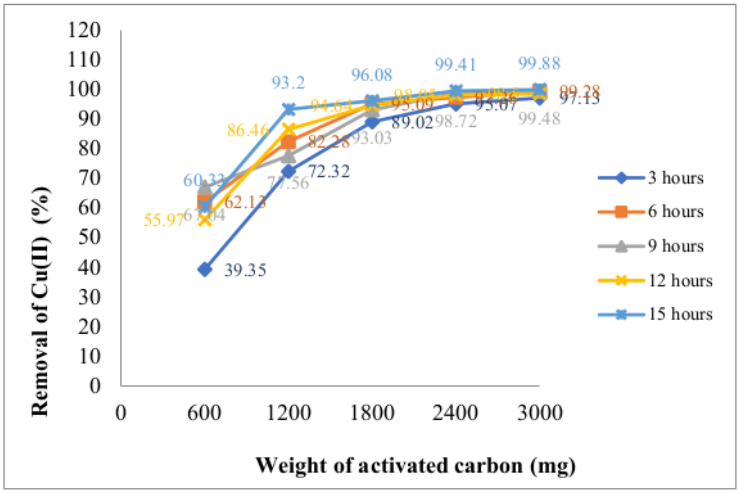

FIGURE.1a. Relationship between the weight of activated carbon against removal of Cu(II)

From **FIGURE1a**, it is observed that the removal percentage of Cu (II) ions increased with adsorbent dosage. It is because the more the amount of activated carbon give rise to the number of pores and the surface area, so the ability to adsorb metals is increasing. The similar trends occurred at all contact time variation (3, 6, 9, 12, and 15 hours). in **FIGURE 1b** shows that the longer the contact time between activated carbon and metal, the higher the percentage of metal adsorption. It is because the longer the contact time will provide a more significant opportunity for the metal to interact with the surface of the activated carbon. The maximum removal percentage (99.88 %) occurred in the weight of activated carbon 3,000 grams at the contact time of 15 hours.

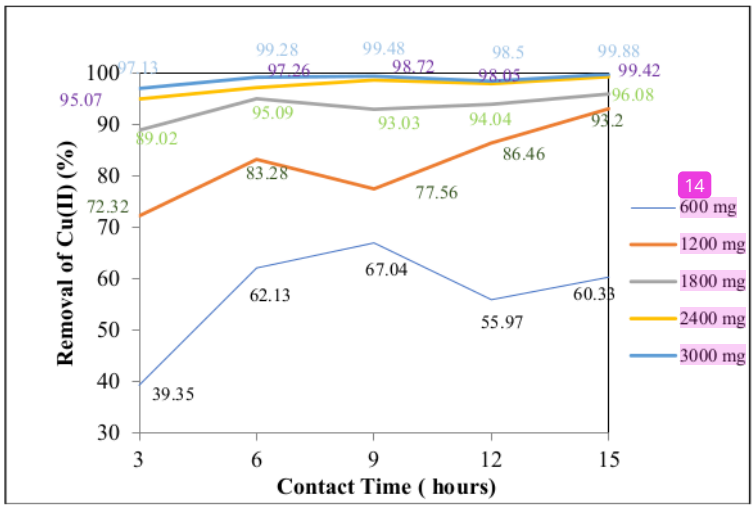

FIGURE.1 b. Relationship between the contact time of activated carbon agains removal of Cu(II)

Effect of adsorbent dosage and contact time to removal of Fe(II)

From **FIGURE 2**a, it is observed that the removal percentage of Fe (II) ions a significant increase between doses of 600 to 300 grams. The similar trends occurred at all contact time variation (3, 6, 9, 12, and 15 hours) are shown in **FIGURE 2b**. The maximum removal percentage (96.24 %) occurred in the weight of activated carbon 3,000 grams at the contact time of 12 hours.

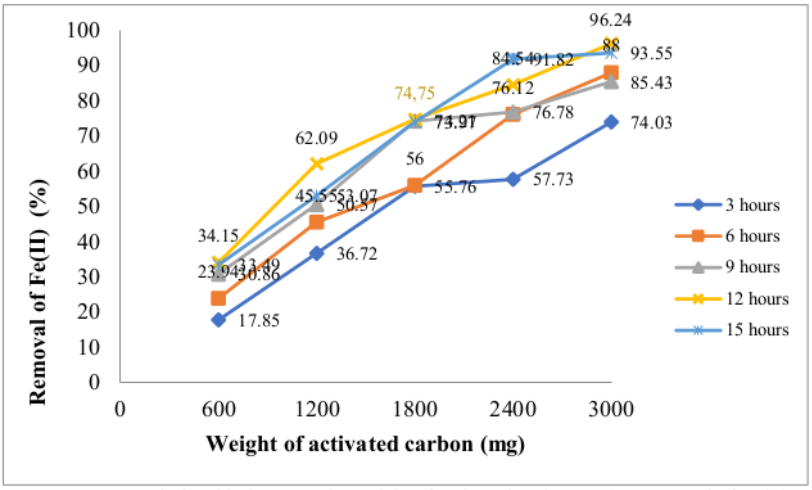

FIGURE2a. Relationship between the weight of activated carbon against removal of Fe(II)

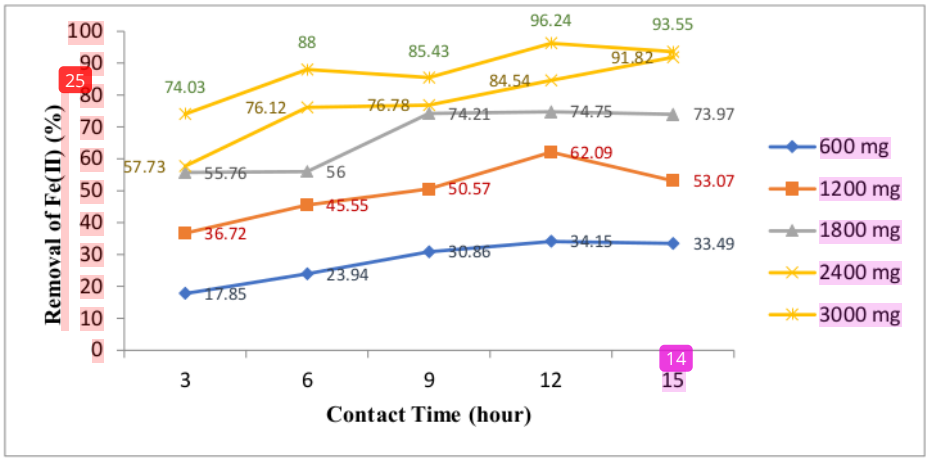

FIGURE 2b. Relationship between the contact time of activated carbon against removal of Fe(II)

TABLE 3. Maximum removal percentage of metal

Metal	Initial concentration (ppm)	Activated carbon weight (mg)	Contact time (hours)	Removal percentage of metal (%)
Cu (II)	16.03	3,000	15	99.88
Fe (II)	16.75	3,000	12	96.24

In **TABLE 3** it appears that metal absorption of Cu (II)> Fe (II) is caused by differences in electronegativity and ionic radii. In this case, the strength of Cu (II) and Fe (II) attached to the negative charge on the surface of activated carbon is measured as the strength of activated carbon adsorption. The higher the electronegativity of metal ions, the higher the strength of adsorption to activated carbon [1]. Another factor that causes differences in the adsorption capacity of activated carbon in metal ions is the ionic radius. The ionic radius of Cu (II) <Fe

(II) because in the electron orbitals, the charge of the Cu (II) ion is attracted more strongly to the nucleus than the Fe (II) ion. Because the activated carbon used is micropore, smaller metal ion sizes are easier to penetrate into the pores [26].

Isothermal Adsorption Studies

Characteristics of activated carbon adsorption in wastewater treatment generally use the Freundlich Isotherm to describe its adsorption capacity. The empirically derived Freundlich isotherm is defined as:

$$\log(x/m) = \log k + 1/n \log Ce \quad (2)$$

By plotting log (x/m) versus log C_e, the constants in the Freundlich isotherm can be determined. From a graph with a slope of 1 / n it can determine the empirical constant and the linear line that intersects the vertical axis is log k. From a graph with a slope of 1 / n it can determine the empirical constant and the linear line that intersects the vertical axis is log k. The Langmuir adsorption isotherm was developed by assuming that the adsorbent activated carbon porosity has the same energy, all of which are available on the adsorbent surface, adsorption on adsorbate occurs in the same surface plane, and adsorption is reversible. The Langmuir adsorption isotherm is then defined from these rational considerations as:

$$Ce/(x/m) = 1/ab + 1/a\ Ce \quad (3)$$

A straight line will be obtained by plotting a graph C_e /(x/m) vs. $1/C_e$. The empirical constant can be obtained from the graph where the slope is $1/ab$ and the linear line will intercept the vertical axis is $1/a$. The notation x is the amount of adsorbed material (mg), m is the weight of adsorbent / activated carbon (g), C_e is equilibrium concentration, k is adsorption capacity for Freundlich adsorption isotherm, n is Freundlich empirical constant, a is adsorption capacity for Langmuir adsorption isotherm, and b is Langmuir constant [27].

The isothermal graph of adsorption of the Freundlich model for Cu (II) and Fe (II) can be seen in **FIGURES3a** and **3b**, mean while the adsorption isothermal graph of the Langmuir model for Cu(II) and Fe(II) can be seen in **FIGURES 4a** and **4b.**

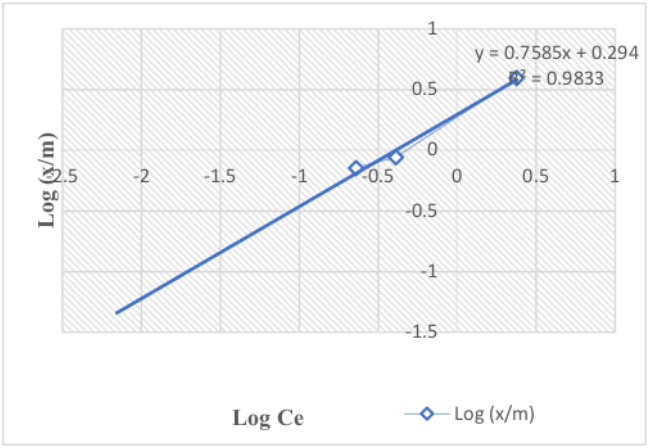

FIGURE 3a. Plot of Isothermal Freundlich equation for Cu(II) (Initial concentration of 16,03 mg/L, the weight ofactivated carbon 600, 1200, 1800, 2400, and 3000 grams, and contact time of 15 hours)

Freundlich and Langmuir adsorption isotherms are obtained through the application of experimental research data. **FIGURES 3a** and **3b** show the application of experimental data obtained for Cu (II) and Fe (II) in the linear Freundlich equation. (1 / n) is the slope of a straight line, and log k is the interception of the line on the y axis. In this study, Cu(II) and Fe(II) adsorption capacity were 1.967 and 1.829 mg/g respectively. From the batch test for Cu(II) and Fe(II), the empirical constant values of n obtained were 1.318 and 1.23 L/mg respectively. It reveals that the multilayer adsorption of Cu (II) and Fe(II) ions on the active carbon is possible.

FIGURES 4a and **4b** show the Ce / (x / m) vs 1 / Ce graph plotted for Cu (II) and Fe (II) to determine the Langmuir constant. The Langmuir isotherm equation applies to adsorption of the monolayer to the surface, where a constant is the adsorption capacity of the adsorbed to activated carbon to form a monolayer. In this study, Cu(II) and Fe(II) adsorption capacity were 0.6277 and 0.955 mg/g respectively. Constants related to the binding affinity of metal ions are empirical constants b in the equation. From the batch test for Cu(II) and Fe(II), the values of b obtained were 10 and 0.86 L/mg respectively.

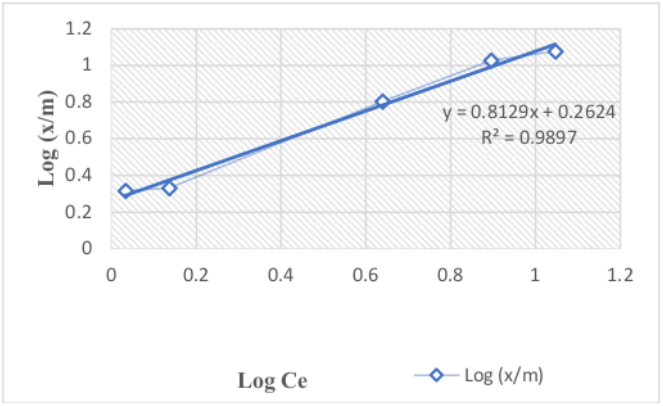

FIGURE 3b. Plot of Isothermal Freundlich equation for Fe(II) (Initial concentration of 16,75 mg/L, the weight of activated carbon 600, 1200, 1800, 2400, and 3000 grams, and contact time of 12 hours)

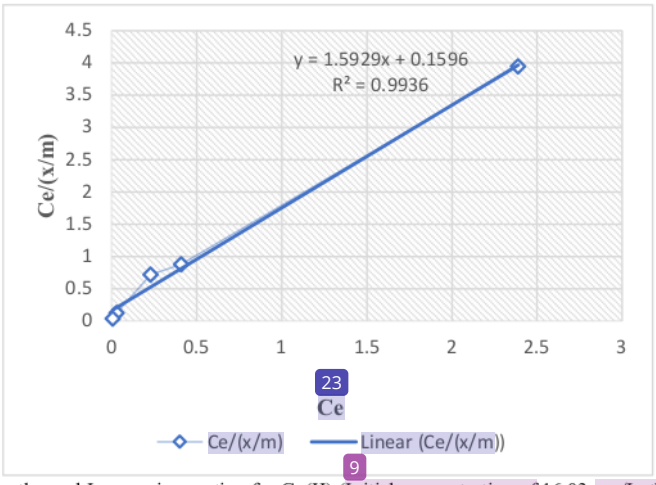

FIGURE 4a. Plot of Isothermal Langmuir equation for Cu(II) (Initial concentration of 16,03 mg/L, the weight of activated carbon 600, 1200, 1800, 2400, and 3000 grams, and contact time of 15 hours)

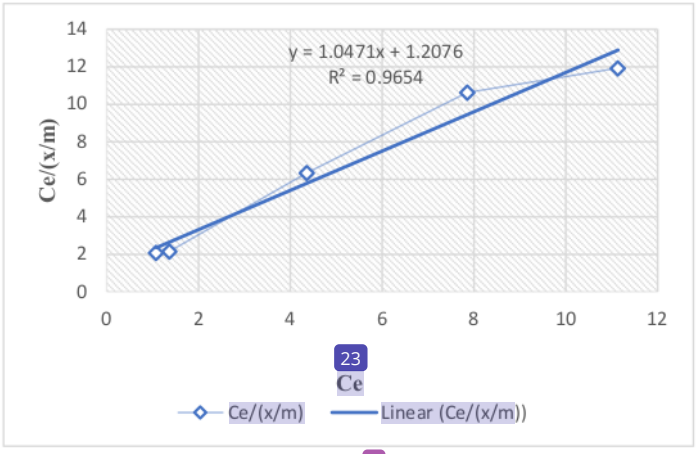

FIGURE 4b. Plot of Isothermal Langmuir equation for Fe(II)(Initial concentration of 16,75 mg/L, the weight of activated carbon 600, 1200, 1800, 2400, and 3000 grams, and contact time of 12 hours)

The experimental results show that based on the Freundlich equation, Cu (II) has a greater activated carbon adsorption capacity compared to Fe (II). This may be related to adsorption characteristics related to electronegativity and ionic radii. Electronegativity is defined as the ability of an element to attract electrons to other elements. Cu(II) has a higher electronegativity than Fe(II), which are 1.9 and 1.8 respectively. This means the strength of Cu(II) to attach negative charge at activated carbon surface is stronger than Fe(II). Based on [26], higher electronegativity results in higher levels of adsorption of metal ions to the surface of activated carbon.

CONCLUSION

Activated carbon is successfully prepared from sub-bituminous coal with iodine number 1,393 mg/g and surface area 512 m^2/g. Application on heavy metal adsorption indicates activated carbon prepared from sub-bituminous can adsorp with removing Cu(II) and Fe(II). The higher the dose of activated carbon and the longer the contact time will result in higher adsorption of Cu and Fe metals. The results showed that a maximum removal percentage for Cu(II) 99.88 % was obtained for 3,000 grams of activated carbon with a contact time of 15 hours, and for Fe(II) 96.24% was obtained for 3,000 grams of activated carbon and 12 hours of contact time.

This study showed that the process performed by activated carbon in the adsorption of Cu(II) and Fe(II) was very interesting. The results of applying Langmuir and Freundlich adsorption isotherms to evaluate experimental data, it is shown that the experimental data are in accordance with the Freundlich and Langmuir isotherms. Based on Freundlich isotherm, obtained that adsorption capacity of Cu(II) and Fe(II) were 1.967 and 1.829 mg/g. Based on Langmuir isotherm, obtained that adsorption capacity of Cu(II) and Fe(II) were 0.6277 and 0.955 mg/g. The results showed that coal activated carbon was effective as an adsorbent for heavy metals Cu (II) and Fe (II) in solution.

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