Study on the Optimization of Mercury Ion (II) Adsorption with Activated Carbon from a Biomass Combination of Palm Bunches and Rice Husk

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Abstract – This research deals with the adsorption of mercury ions [Hg (II)] using a combination of natural biomass between oil palm bunches and rice husks (PB-RH). After drying at 40ºC, PB-RH biomass was carbonized by using a tube furnace for 2 hours at 500 °C and followed chemical activation with citric acid. Activated carbon formed was contacted with an aqueous solution containing Hg (II) ion and stirred at a constant rate of 150 rpm. The concentration of Hg (II) ions in the aqueous phase were analyzed with Atomic Absorption Spectrophotometer (AAS) and the Central Composite Design (CCD) method with Design-Expert software version 6.0.8 was conducted to calculate the optimization of adsorption capacity for mercury ion. The Hg ion adsorption capacity was estimated to observe the performance of the PB-RH biomass combination on the adsorption of Hg (II) ions with some variables, such as contact time, biomass weight, and activator concentration. Results showed that all variable response, contact time, biomass weight, and activator concentration influenced the mercury (II) ion adsorption capacity. The optimum condition of Hg (II) ion adsorption occurred at 30 g of activated PB-RH biomass with 0.6 mole/L concentration of activator during 99.88 minutes with 99.42 mg/L initial concentration of Hg (II). The adsorption capacity occurred at 10.1 mg/g as the optimum condition for the adsorption of Hg (II) ions by PB-RH.

Keywords: adsorption, mercury, palm bunches, and rice husk.

Introduction

Mercury (Hg) is a form of a natural heavy metal element that often pollutes the environment. Most of the mercury comes from nature in the form of other elements combined. In other words, not much mercury is found in a way separate from other elements. In the environment, mercury components are widely distributed in corals, soil, air, water, and living organisms through complex physical, chemical, and biological processes. This element is toxic to one in a billion total parts and is one of the most toxic elements. Nowadays, Mercury poisoning is now one of the most severe health hazards in the world (Sharma, Sharma, & Arya, 2015).

An extensive study has been conducted by many researchers to remove and to minimize the effects of mercury poisoning because it is responsible indirectly for influencing the environment and often causes health hazards. Several researchers continue to develop new alternatives with low-cost mercury removal techniques at a laboratory scale that might be able to be designed for commercial applications, such as the use of biomass as an adsorbent (Salam, Reiad, & ElShafei, 2011). Adsorption is the best conventional method and universal for the processing of organic and inorganic waste (Sumanjit, Seema, Mahajan, & Gupta, 2015) also very easy in reconstruction, operation, and even maintenance (Gupta, Ali, Saleh, Nayak, & Agarwal, 2012)(Yu et al., 2015) with relatively low operating costs. Activated carbon is the most popular material for this adsorption; however, high costs limit their use on a large scale (El-Said, Badawy, & Garamon, 2012). The application of activated carbon in adsorption will increase the absorption capacity but requires a high cost in the process to prepare a

surface area of activated carbon with high uptake and optimal adsorption capacity (Karaoğlu, Doğan, & Alkan, 2010).

Several alternatives using cheap raw materials intended that can lower the total cost required to obtain the optimal results are using natural materials, such as agricultural wastes and useless plants. Natural biomass materials, such as wastes from agriculture, that abundance may have the potential to be cheap absorbents. Because of their low costs, when the lifetime of these materials runs out, they may be removed with low-cost regeneration. The large quantities and availability of agricultural by-products make it a challenge as raw materials for natural adsorbents. Some agricultural wastes can be used as an adsorbent for mercury treatment, including *Bambusa vulgaris var. striata*. It has an adsorption capacity of 218.08-248.05 mg/g (González & Pliego-Cuervo, 2014; Mistar et al., 2019), rice husk (Aprianti, Sheilla, & Machdar, 2018), coconut shell 3.02 mg/g, and rice straw 0.11 mmole/g (Goel, Kadirvelu, & Rajagopal, 2004b). Furthermore, water hyacinth (Zhang et al., 2015) and bamboo (Mistar et al., 2019; Tan, Qiu, Zeng, Liu, & Xiang, 2011) as useless plants, have a potential to be adsorbent to reduce heavy metals. In this study, we modified palm bunches (PB) and rice husk (RH) as an adsorbent to adsorb Hg (II) ion from the water phase.We combined PB-RH biomass as an adsorbent. The purpose of this study was to find the optimal conditions of the Hg (II) adsorption process with activated carbon from a combination of PB-RH biomass using a surface response methodology (RSM).

Materials and Methods
Materials

Palm bunches (PB) was obtained from palm oil plant in Nagan Raya District, Aceh Indonesia. The content of rice husk (RH) was prepared from a rice mill in Aceh Besar District, Aceh Indonesia. The two biomass (PB and RH) were crushed and sieved with a 100-mesh sieve, then were thoroughly washed distilled water and were dried at 90 °C until reaching a constant weight. The dried PB and RH were stored in a desiccator before used. The experimental apparatus used in this study were Dryer (Memmert UF-55), Furnace (Line Thermolyne, FB1410M-33), pH meter (Ohaus 300), Digital Thermometer (Yenaco), Atomic Absorption Spectrophotometer (Shimadzu, AA-6300), Digital Scales (ACIS, BC-500), Shaker (SLM-OS-250), Desiccators (Pyrex®), Dropper (Pyrex®), Erlenmeyer (Pyrex®), Flask (Pyrex®), Buchner Funnel (Pyrex®), and Filter Paper (3M). All chemicals (NaOH, 99.9%; HgCl$_2$; 99.9%; citric acid, 99.5%; etc.) were of analytical reagent grade and were procured from Sigma Aldrich Chemical. Before use, all chemicals were prepared at suitable concentrations.

Adsorbent Preparation

The preparation method was carried out based on our previous work (Suhendrayatna, Zaki, Delima Habdani Harahap, & Verantika, 2018; Zaki, Hadi, & Adha, 2018), consist of adsorbent preparation, activation, and adsorption. PB and RH were carbonized at 500°C in a furnace for 2 hours. The charcoal produced was cooled and follow with the activation process using citric acid with various concentration of 0.2M; 0.4M; 0.6M; and 0.8M, respectively. After this preparation, activated carbon was applied in the adsorption treatment as an adsorbent.

Mercury ion Adsorption and Analysis

A batch experiment was designed in this research. An aqueous solution (100 ml) containing Hg(II) ion was contacted with a suitable weight of activated carbon PB-RH in a shaker at 150 rpm for 100 min at room temperature. Neutral pH was maintained by dripping an appropriate volume of the base (sodium hydroxide) or acid (hydrochloric acid) solution under measuring with a digital pH meter. The adsorbent was removed from the aqueous phase by centrifugation, and Hg ion concentration in the supernatant was analyzed by atomic absorption spectrophotometer. Design-Expert software version 6.0.8 has been conducted to calculate Optimum adsorption of Hg (II) ion based on the Central Composite Design (CCD) method. This method was occurred by applying four factors and three levels to study the effects of contact time, biomass weight, citric acid concentration, and initial concentration on the Hg (II) adsorption capacity.

Results
Statistical analysis

RSM is a combination of statistical and mathematical techniques that are used to model and analyze responses that are influenced by some independent variables or factors x to optimize responses. This methodology is more profitable than traditional single-parameter optimization because it saves time, raw

materials, and space. In this study, there were a total of 30 runs to optimize the four parameters in the current CCD method. Table 1 shows the experimental and prediction results of the Hg (II) ion adsorption capacity based on factorial design. Maximum Hg (II) ion adsorption capacity (7.7 mg/g) occurred at run 16 under the experimental condition of contact time 40 min, biomass weight 25 gram, citric acid concentration 0.2 mole/L, and initial concentration 80 mg/L. Furthermore, the lowest Hg (II) ion adsorption capacity was recorded 1.0 mg/g at the run of 29 under the experimental condition of contact time 60 min, 20-gram biomass weight, 0.4 mole/L citric acid concentration, and 40 mg/L initial concentration.

The statistical model summarizes the general description of the selection of the right model and matches the experimental data. CCD method describes the actual mathematical model applied to predict the Hg (II) ion adsorption capacity based on the suggested model. Based on experimental data being substituted in the quadratic model, the equation of mathematical model that can be applied to the Hg (II) adsorption capacity is as follows.

$$Y = 3{,}59356 - 0{,}14802\ X_1 - 1{,}03973\ X_2 - 0{,}072948\ X_3 + 0{,}014965\ X_4 + 1{,}35666\text{E-}003\ X_1^2 + 2{,}78541\ X_2^2 + 4{,}87860\text{E-}004\ X_3^2 + 7{,}66041\text{E-}004 X_4^2 + 0{,}021333\ X_1X_2 + 9{,}47316\text{E-}005\ X_1X_3 + 1{,}45083\text{E-}003\ X_1X_4 + 0{,}016431\ X_2X_3 - 0{,}047792\ X_2X_4 - 5{,}13171\text{E-}005\ X_3X_4 \dots (1)$$

Equation model (equation 1) consists of each variable that influenced to increase the Hg (II) ion adsorption capacity. These four variables were biomass weight (X_1), citric acid concentration (X_2), contact time (X_3), and initial concentration (X_4). Regarding the most significant coefficient in equation (X1), biomass weight was the most influential variable among these four variables. Base on the equation 1, the actual values for the Hg (II) ion capacity adsorption were tabulated in Table 1 within the prediction column. The prediction value shows the relationship between the predicted values based on the equation model proposed by the response surface. In contrast, the actual amount was provided based on the experimental value. This condition determined the accuracy of the model obtained.

Table 1. Results of Hg (II) ion adsorption based on experimental and predictions

Run	Contact time (minutes)	Biomassa weight (gram)	Citric acid concentration (mole/L)	Initial concentration (mg/L)	Adsorption capacity (mg/g)	
					Experiment	Design expert prediction
1	80	15	0.6	80	6.18	5.6567
2	80	25	0.2	80	7.24	6.3983
3	60	20	0.4	60	3.90	3.3286
4	60	20	0	60	2.39	3.8807
5	60	20	0.4	60	3.56	3.3286
6	40	15	0.2	40	2.59	1.9272
7	60	20	0.8	60	3.99	3.6677
8	60	20	0.4	60	2.52	3.3286
9	80	25	0.6	80	5.72	6.0836
10	60	20	0.4	60	2.58	3.7485
11	80	25	0.6	80	6.78	6.0836
12	60	20	0.4	60	4.32	3.3286
13	60	20	0.4	60	3.62	3.3286
14	80	15	0.2	40	1.63	1.4572
15	80	25	0.6	40	1.52	1.6683
16	40	25	0.2	80	7.77	6.9126
17	40	15	0.6	80	6.93	6.6089
18	40	15	0.6	40	1.36	2.0289
19	60	10	0.4	60	2.09	3.3891
20	60	30	0.4	60	3.67	3.5393
21	60	20	0.4	60	2.80	3.3286
22	80	15	0.6	40	1.96	1.8218
23	60	20	0.4	100	6.94	9.1027
24	100	20	0.4	60	2.58	3.3286
25	80	25	0.2	40	1.23	1.2184
26	80	15	0.2	80	7.69	6.0568
27	40	25	0.6	40	1.20	1.8376
28	40	15	0.2	80	6,93	5.9459
29	60	20	0.4	20	1.00	0.0058
30	40	25	0.2	40	1.30	1.6506

The analysis results of variance and adequacy of the design model were tabulated in Table 2. ANOVA is a CCD factorial that useful for generating the interactions between variables of process and response variables (Zaki et al., 2018). F-ratio, which can determine the effectiveness of a model, was calculated based on components in ANOVA. Table 2 shows that all variables have a P-value higher than 0.05 and only variable D values below 0.05. These results suggested that the variable D (concentration variable) has a significant influence on the adsorption capacity of Hg (II) ions, which has a probability value (probability > F) <0.05, and that value was less than 0.0001.

Table 2. Variant test results (ANOVA) for the surface square of adsorption capacity of Hg (II) ion

Sources		Sum of Squares	DF	Mean Square	F Value	Prob. > F	
Model		127.96	14	9.14	6.36	0.0005	significant
	A	0.064	1	0.064	0.044	0.8363	
	B	0.028	1	0.028	0.019	0.8917	
	C	0.32	1	0.32	0.22	0.6459	
	D	83.70		83.70	58.22	<0.0001	
	A²	0.032	1	0.35	0.022	0.8834	
	B²	0.35	1	0.58	0.24	0.6311	
	C²	0.58	1	2.61	0.40	0.5344	
	D²	2.61	1	1.56	1.82	0.1977	
	AB	6.620E-003	1	6.620E-003	4.605E-003	0.9468	
	AC	1.253E-004	1	1.253E-004	8.717E-004	0.9768	
	AD	0.31		0.31	0.21	0.6511	
	BC	0.060	1	0.060	0.042	0.8405	
	BD	0.53	1	0.53	0.37	0.5522	
	CD	5.884E-003	1	5.884E-003	4.093E-003	0.9498	
Residual		21.56	15	1.44			
	Lack of Fit	18.06	8	2.26			
	Pure Error	3.50	7	0.50	4.51	0.0310	significant
Cor. Total		149.53	29				

Optimization of Hg (II) Adsorption

The quadratic equation was a model obtained from the Design-Expert software version 6.0.8 to predict the adsorption capacity of Hg (II) in this study. The selection of the model was based on various variables, such as the value of R^2. Based on the surface response analysis of this model, two types of graphs can be used to describe the adsorption capacity; they were contour plots and 3D plots.

This plot is applied to facilitate the description of the effects of variables on the response. The contour plot was a 2D plot, which was a cross-section of a 3D plot that was useful to illustrate the impact of interactions between factors on the response. On the other hand, the 3D plot shows the effect of two variables on the response where the variable was created in a fixed condition. The model equation suggested in this study for the analysis of Hg (II) ion adsorption was the quadratic equation model. This model can be analyzed through contour plots, and 3D plots and Figures 1 to 3 show the relationship between three independent variables to the response variable.
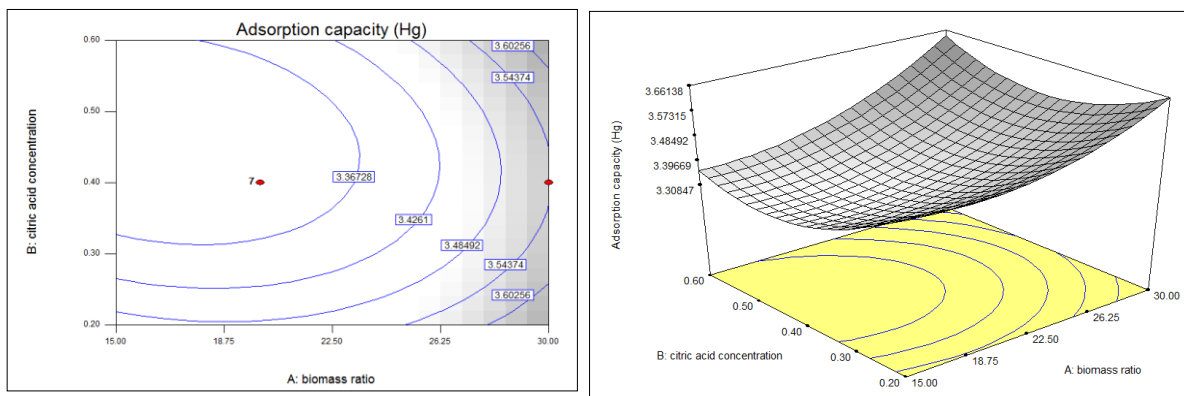
Figure 1. Contour plots and 3D plots for the effect of citric acid concentration and biomass weight on Hg (II) ion adsorption capacity
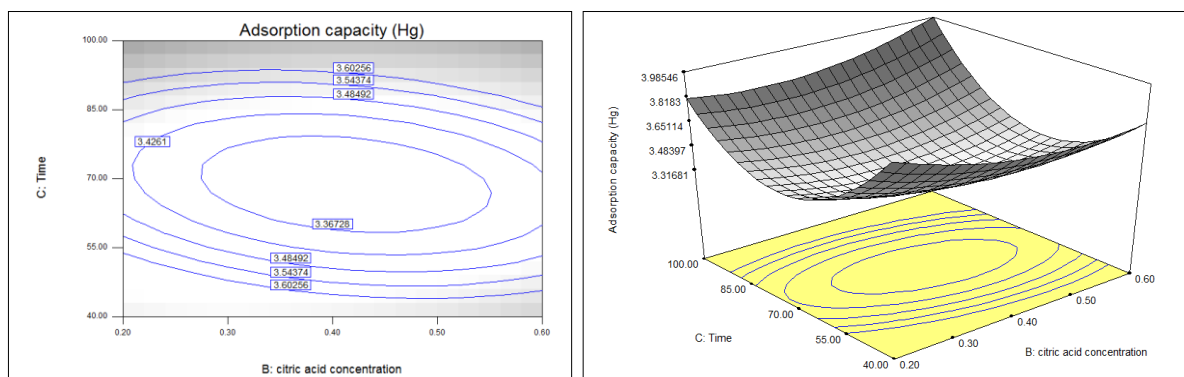

Figure 2. Contour plots and 3D plots for the effect of citric acid concentration and contact time on Hg (II) ion adsorption capacity
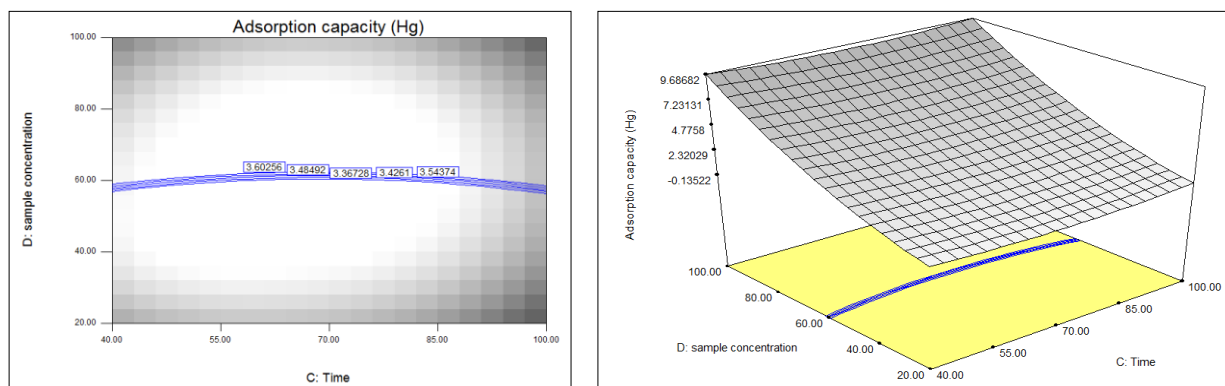

Figure 3. Contour plots and 3D plots for the effect of contact time and biomass weight on Hg(II) ion adsorption capacity

The optimization process in this study was carried out by adding the amount of biomass weight, contact time, and citric acid concentration. Optimization condition for Hg (II) ion adsorption capacity by PB-RH resulting from the calculation of Design-Expert software version 6.0.8 was tabulated in Table 3.

Table 3. Optimization condition for Hg(II) ion adsorption capacity using PB-RH

Contact time (minutes)	Biomassa rasio (g)	Citric acid conc. (mol/L)	Initial con. (mg/L)	Adsorption capacity (mg/g)	Desirability
99.88	30.00	0.60	99.42	10.01	0.95

These results indicate that optimal conditions were achieved at 30 g of activated PB-RH biomass with 0.6 mole/L concentration of activator during 99.88 minutes with 99.42 mg/L initial concentration of Hg (II).

Discussions

Activated carbon has been widely known and used as an absorbent of pollutants in waste treatment processes. Activated carbon consists of porous solids containing 85-95% carbon produced from materials including carbon, which is dehydrated, carbonated, and activated physically and chemically to obtain a large surface and has a high affinity. Activated carbon has a surface area in the range of 600 to 2000 m²/g, which is related to the pore structure that causes has an absorbent property (Bhatnagar & Sillanpää, 2010; Bhatnagar, Vilar, Botelho, & Boaventura, 2010). Carbonation is a process of combining indoors without oxygen and other chemicals. At the same time, activation is a treatment of charcoal to enlarge pores by breaking hydrocarbon bonds or oxidizing surface molecules to changes in properties, both physical and chemical. Activated carbon has been found as a versatile adsorbent, which can reduce various types of pollutants such as metal ions, pesticides, detergents, phenols, and other chemicals (Sud, Mahajan, & Kaur, 2008). However, its application is sometimes limited due to higher costs. Therefore, researchers continue to look for low-cost adsorbents for wastewater treatment, where cost factors play a significant role (Al-Mulla, Jabbar, & AL-Hamadani, 2018). One of the raw materials derived from abundant and inexpensive agricultural materials is rice husk, which has high organic (carbon) content with low inorganic content and can be easily activated. This rice husk has been tested and succeeded in removing heavy metal ions such as arsenic in water (Khalid, Ahmad, Kiani, & Ahmed, 1999), Sb (III), Pb (II) (Khalid et al., 1999; Wang & Lin, 2008), Hg (II) (Khalid et al., 1999), and Cd (II) (Kumar & Bandyopadhyay, 2006).

This study uses natural rice husk combined with palm bunches, followed by citric acid activator to remove Hg (II). The use of activator will affect the ability of activated carbon. Other types of activators that have been studied include KOH, $ZnCl_2$, H_2SO_4, (Yalçın & Sevinc, 2000), depending on the type of pollutant to be sorbed. The citric acid activator used has also been tested inactivation for the absorption of Mn (II) ions (Suhendrayatna et al., 2018). After experimenting with several variables (time, citric acid concentration, and biomass weight), rice husk combined with palm bunches can remove Hg (II) from the water phase with its adsorption capacity of 10.01 mg/g. The optimal conditions were achieved at 30 g of activated PB-RH biomass with 0.6 mole/L concentration of activator during 99.88 minutes with 99.42 mg/L initial concentration of Hg (II). The adsorption capacity was one of the main parameters in the adsorption system. It describes the performance of adsorbents used in the absorption process. A comparison of adsorption capacity values of various types of natural adsorbents is presented in Table 4. Activated Carbon from PB-RH resulted in this study has a relatively low adsorption capacity (10.01 mg/g) in contrast to other Activated Carbon based natural materials reported in the literature. The difference in adsorption capacity is due to each characteristic of natural ingredients having different elemental contents, such as preparation methods and types of functional groups

Table 4. Hg (II) ion adsorption capacity by activated carbon based on natural materials

Activated carbon-based on natural materials	Adsorption Capacity	References
Bambo (*Bambusa vulgaris var. striata*)	218.08 mg/g	(Mistar et al., 2019)
Coconut shell	3.02 mg/g	(Goel, Kadirvelu, & Rajagopal, 2004a)
Bambusa vulgaris var. striata	248.05 mg/g	(González & Pliego-Cuervo, 2014)
Rice straw	0.11 mmole/g	(Goel et al., 2004a)
Rice husks	1.30×10^{-3} mol.dm^{-3}	(Khalid et al., 1999)
$ZnCl_2$-modified walnut shells	151.5 mg/g	(Zabihi, Ahmadpour, & Asl, 2009)
Palm bunches and rice husks	10.01 mg/g	This study

Conclusion

This research leads to the conclusion that the concentration variable has a significant influence on the adsorption capacity of Hg (II) ions from the aqueous phase. By using the quadratic regression model in the optimization analysis, the optimal conditions occurred at 30 g of activated PB-RH biomass with 0.6 mole/L concentration of activator during 99.88 minutes with 99.42 mg/L initial concentration of Hg (II). The adsorption capacity occurred at 10.1 mg/g as the optimum condition for the adsorption of Hg (II) ions by PB-RH.

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