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Adsorption of Mercury(II) using Activated Carbon Produced from *Bambusa vulgaris* var. *striata* in a Fixed-Bed Column

(Penjerapan daripada Merkuri(II) menggunakan Karbon Teraktif yang Dihasilkan daripada *Bambusa vulgaris* var. *striata* dalam Kolum Lapisan Tetap)

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ABSTRACT

Pollution by mercury dissolved in aqueous media causes a crucial problem for health and environment. In this study, activated carbon from Bambusa vulgaris var. striata was produced by chemical activation using NaOH for mercury adsorption. The effects of mercury initial concentrations namely 50 and 100 mg/L on the breakthrough characteristics of the adsorption process were defined. The mechanism of the adsorption process through the fixed-bed column was fitted to the Thomas model. The activated carbon was characterized by scanning electron microscopy and energy-dispersive X-ray spectroscopy. The adsorption study with a continuous system and using the Thomas model showed that the highest adsorption capacity (q_0) of mercury ions is 218.08 mg/g. It can be concluded that activated carbon from Bambusa vulgaris var. striata has a great potential to act as an adsorbent to remove mercury from water.

Keywords: Activated carbon; adsorption; Bambusa vulgaris var. striata; mercury; Thomas model

ABSTRAK

Pencemaran merkuri terlarut dalam air menyebabkan masalah kesihatan kritikal dan mencemarkan alam sekitar. Dalam kajian ini, karbon teraktif daripada Bambusa vulgaris var. striata dihasilkan melalui pengaktifan kimia menggunakan NaOH untuk penjerapan merkuri. Kesan kepekatan merkuri awal iaitu 50 dan 100 mg/L ke atas kejayaan proses penjerapan telah ditakrifkan. Mekanisme proses penjerapan melalui kolum lapisan tetap telah disesuaikan dengan Model Thomas. Karbon teraktif yang dicirikan melalui mikroskopi imbasan elektron dan spektroskopi serakan tenaga sinar-X. Kajian penjerapan dengan sistem yang berterusan dan menggunakan Model Thomas menunjukkan kapasiti penjerapan tertinggi (q_0) ion merkuri adalah 218.08 mg/g. Maka disimpulkan bahawa karbon teraktif daripada Bambusa vulgaris var. striata berpotensi besar untuk bertindak sebagai penjerat untuk mengasingkan merkuri daripada air.

Kata kunci: Bambusa vulgaris var. striata; karbon; merkuri; model Thomas; penjerapan

INTRODUCTION

Heavy metal dumping into the environment has been triggered by the industrial sector and growing human population. Low concentrations of heavy metals, including mercury, cadmium, chromium, and lead, are harmful and toxic to humans, animals, and plants due to their non-biodegradability and toxicity (Ismail et al. 2013). Influence of heavy metal tends to endure immeasurably and will accumulate in the food chain system.

Mercury ions or Hg(II) has caused environmental pollution where it has an unstable, high toxicity, and can easily migrate to all environmental media, such as soil, water, air and plants. Mercury has a liquid property at room temperature. When entering the human body, Hg(II) can join with enzymes causing the loss of enzyme capability as a catalyst in the human body. It can easily enter into the body through the digestive system and skin. The amount of Hg(II) that accumulates is a significant amount and people should be wary as it can cause very adverse health effects (Kabiri et al. 2016). Many cases of Hg (II) poisoning are reported around the world caused by the fish and shellfish

consumption contained of Hg(II) (Hassan et al. 2017). For the environment protection, the process of removing Hg(II), is an essential operation, which is found in many industrial wastes, electrochemical, mining and electroplating (Mondal et al. 2013). Various technologies have been investigated for the heavy metal removal from aqueous solutions, for example, adsorption, membrane filtration, ion exchange, reverse osmosis, solvent extraction, chemical precipitation, and coagulation (Shafiq et al. 2018).

Activated carbon (AC) is a carbon form processed to produce high surface area and optimum adsorption capacity for adsorption or chemical reactions. It can adsorb various heavy metals, gases, and dyestuffs. Because of its adsorption potential, AC is widely used to remove heavy metals, filter, adsorb odours, and detoxify. The method is very popular in the chemical industry, medicine, refineries, water treatment, and mining (Cazetta et al. 2011). The capability of AC as an adsorbent of heavy metals depends on many factors, namely the activation method and the nature of the precursor used. The activation techniques such as impregnation, base, acid, microwave, surfactant, plasma,

and ozone treatment, have been developed to improve the performance of AC for adsorption of most pollutants. Furthermore, precursor materials have also been studied to find appropriate AC for various application (Bhatnagar et al. 2013).

AC has been proven to adsorb Hg(II) contained in the water with a high adsorption capacity. The adsorption studies have been conducted previously using AC made from various sources, such as lignocellulosic materials (Arias et al. 2017), sugarcane bagasse (Sun et al. 2018), scallop shells (Hassan & Hrdina 2018), exhausted coffee waste (Alvarez et al. 2018), flamboyant pods (Vargas et al. 2010), coconut shell (Cazetta et al. 2011) and bamboo (Gonzalez & Pliego-Cuervo 2013). Referring to the results of numerous studies, the AC surface area is a major factor affecting the adsorption process. Gonzalez and Pliego-Cuervo (2014) reported that the AC made from *Bambusa vulgaris* var. *striata* has a potential surface area, pore diameter, and pore volume. Currently, improvement in synthesizing surface-modified AC for various applications is still an emerging research field for further exploitation and development of a more efficient AC (Bhatnagar et al. 2013).

The objective of this present study was to identify the performance of AC produced from *Bambusa vulgaris* var. *striata* with NaOH activation for Hg(II) adsorption using fixed-bed columns. The Thomas model was used in this study for a breakthrough curve study because the model is broadly used for assessing breakthrough curves (Alalwan et al. 2018). Finally, the adsorption capacity (q_0) and the Thomas rate constant (k_T) will be determined. The morphological structure and elemental analysis were also performed to detect the Hg(II) on the surface of AC.

MATERIALS AND METHODS

MATERIALS

Bambusa vulgaris var. *striata* were collected from a local farmer in Banda Aceh, Indonesia. Mercury(II) chloride (HgCl_2), nitric acid (HNO_3), and hydrochloric acid (HCl) used in the experimental work were from Merck (analytical grade). Distilled water with conductivity $<1 \mu\text{S/cm}$ was also used in this study. All chemicals were used as received.

EXPERIMENTAL PROCEDURE

Bambusa vulgaris var. *striata* were washed with water and dried to remove the moisture content. Then, the carbonization process was conducted using a furnace at 500°C of temperature for 2 h at 200 mL/min of nitrogen gas flow rate. The charcoal produced was cooled, triturated by a mechanical milling, and sieved until 125–250 μm in size. The charcoal was then activated with NaOH as an activator. The ratio of NaOH to charcoal used was 3:1 (w/w). Then, pyrolysis was performed at a temperature of 800°C . The AC produced was then washed with HCl and stirred for 30 min at a temperature of 85°C . The residual acid in the product was removed by washing with distilled water several times.

The product was stored in a hermetic container at room temperature. The characteristics of AC used are shown in Table 1 (Mistar et al. 2018). These characteristics illustrate AC from *Bambusa vulgaris* var. *striata* as a good precursor for Hg(II) adsorption from water solutions.

TABLE 1. Characteristics of activated carbon

Parameter	Unit	Value
Surface area	m^2/g	1041.7
Total pore volume	cm^3/g	0.622
Average pore diameter	Å	23.9

The adsorption process was conducted by flowing 20 mL/min of Hg(II) solution and initial concentrations were 50 and 100 mg/L. Stock solutions of Hg(II) were prepared by dissolving amounts of HgCl_2 appropriately in distilled water. Distilled water was used of all solutions preparation. The adsorption process was conducted with 125–250 μm AC filled in a fixed-bed column with a mass of 0.5 g. The column was made of a glass tube. The feed of Hg(II) solution was pumped to the column in an up-flow direction using a peristaltic pump. The analysis of Hg(II) concentration was measured by sampling the samples at a specified time interval. The experiments were performed in the laboratory at ambient condition. Duplicate measurements were conducted for all the variables of operating studied and the values of the average concentration are taken into detail consideration. The data deviation found to be less than 5% of the mean value for the whole experiments. These results showed good reproducibility of data. The schematic experimental set up of the adsorption process can be seen in Figure 1.

THOMAS MODEL FOR FIXED-BED COLUMN STUDIES

The Thomas model application results in a linear regression equation used to determine two important parameters, i.e. the adsorption capacity and rate constant. The Thomas model assumes a Langmuir isotherm with rate expression of a pseudo second-order. Most of the research on continuous adsorption mode using the Thomas model has resulted in a good linear regression equation. Trgo et al. (2011) also reported that the Thomas model can be applied in many flow systems by neglecting the dispersive effects, i.e. axial dispersion and finite resistance to mass transfer. The Thomas model is expressed in (1).

$$\frac{C_t}{C_0} = \frac{1}{1 + \exp\left[(k_T \cdot q_0 \cdot \frac{m}{F}) - (k_T \cdot C_0 \cdot t)\right]} \quad (1)$$

where C_t is the concentration of Hg(II) solution in effluent at a certain time t (mg/L); C_0 is the initial concentration of Hg(II) solution (mg/L); q_0 is the adsorption capacity per gram of adsorbent (mg/g); m is the adsorbent mass in the column (g); F is the feed volumetric flow rate (mL/

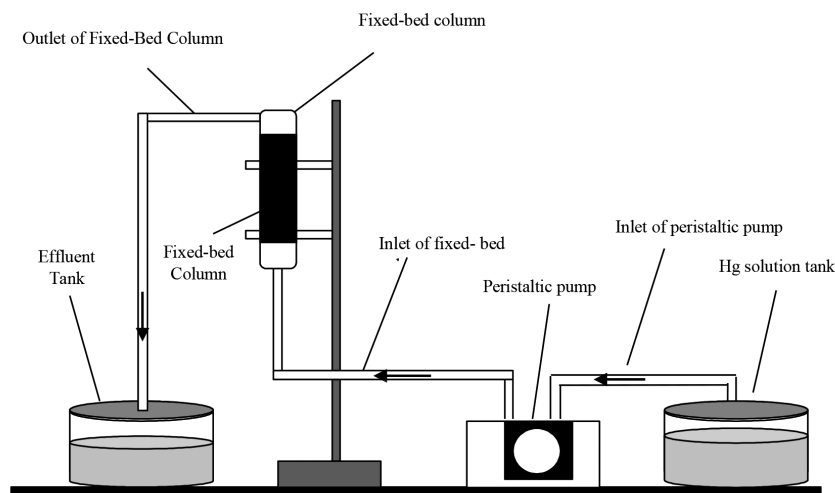


FIGURE 1. Schematic experimental set up of the adsorption process

min); k_T is the rate constant of Thomas model (mL/(min.mg)); and t is the time of the adsorption process (min). The plot between $\frac{C_t}{C_0}$ versus t produces the adsorption pattern curve, commonly referred to as the breakthrough curve. The Thomas model expressed in (1) can be converted to a linear form, as presented in (2).

$$\ln\left(\frac{C_0}{C_t} - 1\right) = (k_T \cdot q_0 \cdot \frac{m}{F}) - (k_T \cdot C_0) \cdot t \quad (2)$$

The linearized form (2) can be used to determine the value of parameters in (1). Based on the plot of $\ln\left(\frac{C_0}{C_t} - 1\right)$ against time t , thus, one can obtain k_T as the slope and q_0 as the intercept.

PRODUCT ANALYSIS

The Hg(II) concentration was measured using a Mercury Analyzer MVU-1A AA-7000 (Shimadzu) where HNO_3 was used to acidify samples prior to analysis. The morphological structure and Hg(II) analysis of the AC from the *Bambusa vulgaris* var. *striata* were provided by a scanning electron microscope (SEM) TM 3000 (Hitachi) equipped with an energy-dispersive X-ray spectroscopy (EDS) unit run at a 25 kV accelerating voltage to ensure the peaks one wishes to record.

RESULTS AND DISCUSSION

BREAKTHROUGH CURVES

Column adsorption studies are essential for large-scale system design. Figure 2 presents the breakthrough curves of the experimental results. As can be seen in Figure 2, during the initial stage of contact of the AC and Hg(II) solution, it is assumed that the plentiful availability of active binding sites on the adsorbent, therefore, Hg(II)

adsorbed increases rapidly with time. Then, the adsorption tempo was slowed down until a nearly constant with the progress of the adsorption process because the decreased availability of active binding sites. The same results were reported by several investigators that studied Hg(II) adsorption by different adsorbents, i.e. sulphur doped zinc oxide (Hassan et al. 2017) and bamboo leaf powder (Mondal et al. 2013). The time of breakthrough as the duration for up to zero concentration of column outlet, increases from around 100 to 200 min, when Hg(II) initial concentration was raised from 50 to 100 mg/L for the same flow rate of 20 mL/min. In addition, the gradient and pattern of the breakthrough curves of the two Hg(II) initial concentrations are different. A higher of Hg(II) initial concentration causes the breakthrough time faster. In general, the gradient and pattern of the breakthrough curve during the time depend on the attainable AC loading with reference to the amount of Hg(II) applied to the fixed bed. The achievable adsorbent loading is the degree of Hg(II) retained by the AC when the fixed bed reaches saturation. A low attainable adsorbent loading will lead to an early breakthrough time (Chu 2010). In this study, it was observed that a higher Hg(II) initial concentration in solution will lead to an early breakthrough time.

LINEAR CURVE AND ADSORPTION CAPACITY

The next step was to determine the adsorption capacity and model of the AC. The Thomas model was used to determine the value of k_T , i.e. the constant of Thomas rate (mL/(min.mg)), which was used to determine q_0 , i.e. the adsorption capacity per gram of adsorbent (mg/g). The results of the mercury adsorption research were made in the breakthrough curves form by approaching the linear regression with t . Figure 2 shows the corresponding predicted value by the Thomas model of (1).

The linearized pattern of the curves in accordance with Hg(II) initial concentrations in solution verifies the Thomas model. Equation (2) shows precise the experimental data

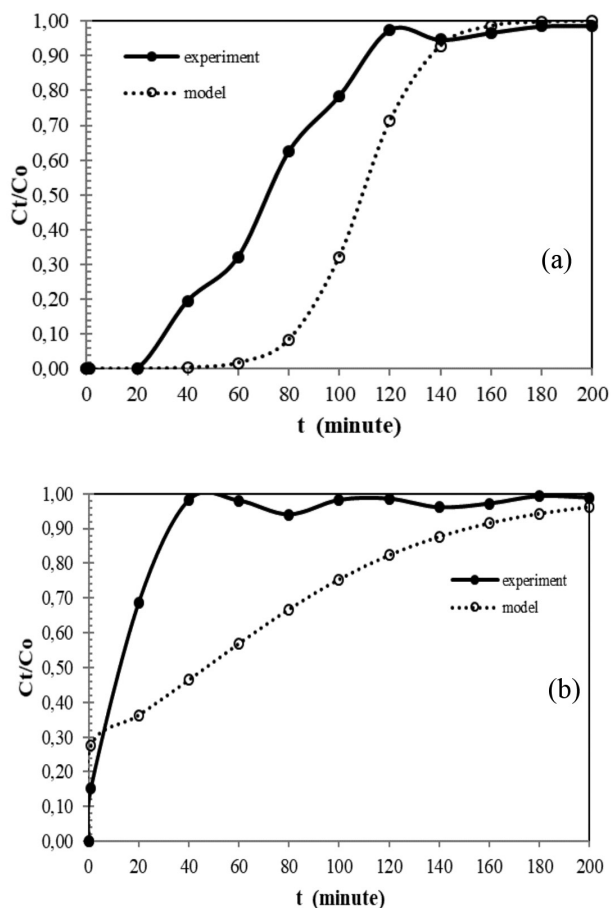


FIGURE 2. Breakthrough curves: (a) 50 mg/L of Hg (II) initial concentration, 20 mL/min of flow rate; and (b) 100 mg/L of Hg (II) initial concentration, 20 mL/min of flow rate

quite worth. The Thomas model parameters, k_T and q_0 , were determined of the experimental data using the method of linear regression by using (2). The calculated generates of the k_T and q_0 values are shown in Table 2.

The decrease in the Thomas rate constant (k_T) values with increased Hg(II) initial concentrations is a common phenomenon observed for the Hg(II) adsorption. There are two contributions of initial concentration to the adsorption rate of Hg(II). Firstly, an increase in the Hg(II) initial concentration causes to decrease the Hg(II) diffusion in the layer of boundary and to gain diffusion in the solid. Secondly, an increase in the initial concentration of Hg(II) in solution causes the forward reduction in covalent interactions depend on interactions of electrostatic of the sites with a weaker affinity for Hg(II) ions (Ismail et al. 2013).

The adsorption capacity is one of the pivotal parameters in the system of adsorption. It describes the performance of adsorbent used in the process. The adsorption capacity value comparison is presented in Table 3. As presented in Table 3, AC from *Bambusa vulgaris* var. *striata* resulted in this study has relatively high adsorption capacity (218.08 mg/g) contrasted with other AC precursors reported in the literature. The differences in adsorption capacities shown by AC from *Bambusa vulgaris* var. *striata* and other adsorbents are due to each characteristics of precursor containing are different, such as preparation method and functional groups types. The cost-effectiveness and easy availability are some additional advantages to make AC from *Bambusa vulgaris* var. *striata* as a prospective adsorbent to remove of Hg(II) from water solutions.

TABLE 2. The results of Thomas model

C_0 (mg/L)	F (mL/min)	k_T (L/min.mg)	q_0 (mg/g)	R^2
50	20	1.652×10^{-3}	218.08	0.8510
100	20	0.210×10^{-3}	187.37	0.7189

TABLE 3. Adsorption capacity of various activated carbon precursor

Activated carbon precursor	q_0 maximum (mg/g)	Reference
Bamboo	2.71	Tan et al. (2011)
Coconut	5.236	Lu et al. (2014)
Bamboo leaf powder	27.1	Mondal et al. (2013)
Coffee waste	31.75	Alvarez et al. (2018)
Coconut shell	38.60	Andal and Sakthi (2010)
Palm shell	83.33	Ismail et al. (2013)
Norit	122.8	Zhu et al. (2009)
Lignocellulosic coconut fiber	144.4	Johari et al. (2014)
Pistachio-nut shells	147.1	Asasian et al. (2012)
<i>Bambusa vulgaris</i> var. <i>striata</i>	218.08	This study
<i>Bambusa vulgaris</i> var. <i>striata</i>	248.05	Gonzalez and Pliego-Cuervo (2014)
Bituminous coal	578	Asasian et al. (2014)
Chitosan-thiomer polymer	2493	Bhatt and Padmaj (2019)

SEM ANALYSIS

SEM analysis was utilized to observe and evaluate the surface morphological of the AC prior and after process of adsorption. Figure 3 shows that the SEM results of the AC from *Bambusa vulgaris* var. *striata* prior and after process of Hg(II) adsorption differ. Before adsorption, the surface of AC from *Bambusa vulgaris* var. *striata* exhibits porous properties with a number of chasms and rucks, indicating a high specific surface area showed on the material (Figure 3(a)). In addition, the AC has a narrow range and quite similar of pore sizes. Meanwhile, a significant disparities in structure of the AC surface after Hg(II) adsorption process were observed. After the adsorption process, assemblies of white needles on the AC surface were observed, which were assumed to be complex formed of Hg(II) ions (Figure 3(b) and 3(c)). Zhu et al. (2009) observed that many extracellular biomass materials of adsorbents can complex or precipitate with ions of heavy metal to generate crystals, oxalate crystal, for instance, which is resulted by oxalic acid and metal ion over the wall of cell and outer matrix of adsorbents. A similar result of AC after Hg(II) adsorption at concentrations 50 mg/L and 100 mg/L was observed.

EDS ANALYSIS

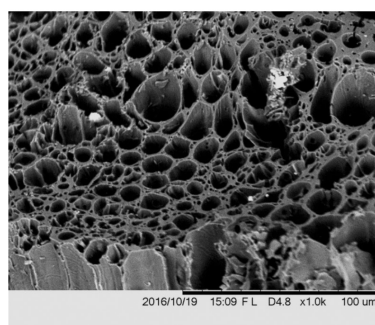
EDS analysis was operated to confirm the appearance of mercury on the AC surface. Figure 4 shows the

corresponding spectrum of EDS and the table of composition. As presented in Figure 4, a significant content of Hg(II) is attendance on the AC surface and is quite regularly distributed, while in Figure 4(b), the content of Hg(II) is less than Figure 4(a). This result shows that carbon and mercury become the main elements of the AC surface made from *Bambusa vulgaris* var. *striata*. It was because the Hg(II) were physisorbed on the AC surface (Ismail et al. 2013).

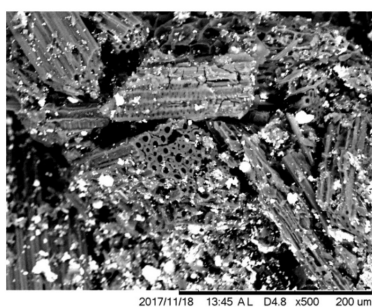
The percentage of Hg(II) adsorbed on the AC surface reduced with enhanced initial concentration of Hg(II). Additionally, at lower of the initial concentration of Hg(II), Hg(II) can easily occupy the lavish existence of active adsorption sites on the AC. However, at higher Hg(II) initial concentration, an adequate quantity of active adsorption sites for Hg(II) to dwell are not obtainable. As consequence, Hg(II) is not totally adsorbed to the AC because the binding sites saturation (Ismail et al. 2013). It can be concluded that EDS analysis allows authentication for the specific Hg(II) adsorption onto the AC from *Bambusa vulgaris* var. *striata*.

CONCLUSION

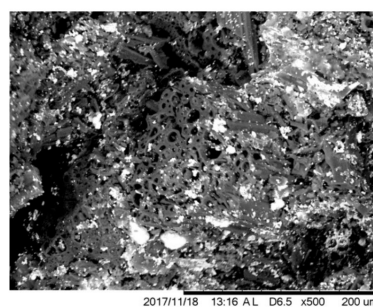
It was observed that AC made from *Bambusa vulgaris* var. *striata* can be used for Hg(II) adsorption in water solution. The Thomas model was used for the description of the



(a)

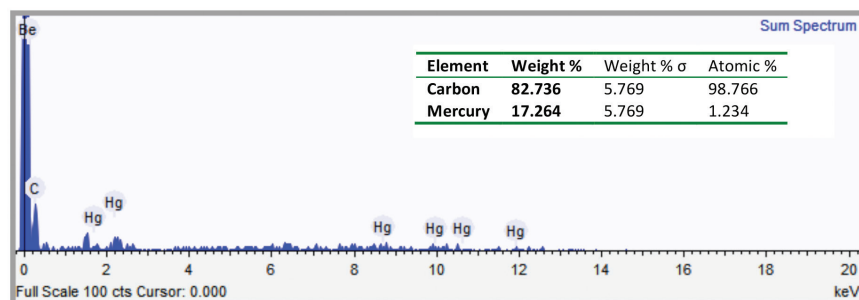


(b)

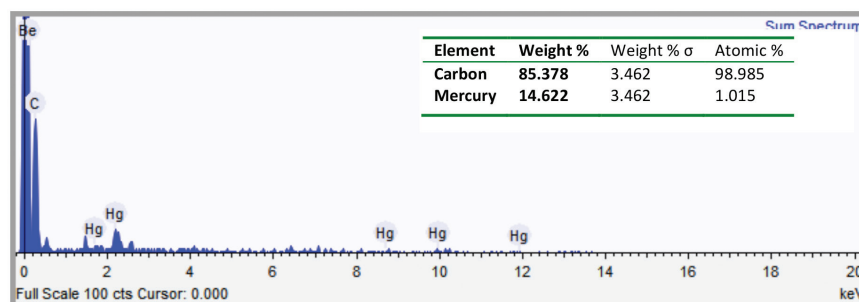


(c)

FIGURE 3. The morphology of activated carbon: (a) before adsorption; (b) after adsorption for 50 mg/L of Hg(II) initial concentration, 20 mL/min of flow rate of; and (c) after adsorption for 100 mg/L of Hg(II) initial concentration, 20 mL/min of flow rate



(a)



(b)

FIGURE 4. Energy-dispersive X-ray spectroscopy analysis of activated carbon after adsorption: (a) 50 mg/L of Hg(II) initial concentration and 20 mL/min of flow rate; and (b) 100 mg/L of Hg(II) initial concentration and 20 mL/min of flow rate.

breakthrough curve. The Hg(II) adsorption was studied using a fixed-bed column by the Thomas model showed that the optimum adsorption capacity at 50 mg/L of Hg(II) initial concentration and 20 mL/min of flow rate is 218.08 mg/g. SEM and EDS analysis detected the attendance of mercury on the AC from *Bambusa vulgaris* var. *striata*. It can be concluded that the AC produced from *Bambusa vulgaris* var. *striata* in this study has the highly potential to adsorb mercury in aqueous solution.

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