

Removal of Cu(II) ions from aqueous solution by activated carbon produced from banana fruit bunch (*Musa paradisiaca*)

Allwar Allwar*, Ade Setiawan, Hendra Agitya Ermawan, Tomy Alviansah

Department of Chemistry, Faculty Mathematics and Natural Sciences, Islamic University of Indonesia, Yogyakarta 55584, Indonesia, Tel. +628112551896; email: allwar@uii.ac.id (A. Allwar)

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ABSTRACT

A low-cost adsorbent of activated carbon prepared from banana fruit bunch (*Musa paradisiaca*) was used for the removal of Cu(II) ion in aqueous solution. Preparation was carried out with different concentrations of sodium hydroxide (NaOH) and under low hydrothermal carbonization method. Experimental results showed that the maximum surface area was obtained at concentration 20% solution of NaOH $33.43 \text{ m}^2 \text{ g}^{-1}$. Surface morphology of activated carbon has the network cavities of mesopores structures with unsmooth surface and irregular size pores. The pores were closed by the fraction of carbon and other impurities such as silicon. Surface chemistry of activated carbon contains highly oxygen-functional groups relating with base properties. The adsorption capacity of Cu(II) was significantly affected by the initial pH of solution, concentration, contact time and mass of adsorbent. Investigation of equilibrium and kinetics adsorption showed the Freundlich isotherm and the pseudo-second-order with $R^2 > 0.9$, respectively. The result of this observation showed that banana fruit bunch is successfully converted to activated carbon and used as adsorbent for the removal of Cu(II) in aqueous solution.

Keywords: Banana fruit bunch; Activated carbon; Cu(II); Sodium hydroxide; Hydrothermal carbonization

1. Introduction

Heavy metal ions such as lead, zinc, copper, cadmium etc. are toxic elements which are easily found in trace amounts in the water effluent. There are increasing chronic diseases in the water environment caused from the interaction between water quality and heavy metal ion [1]. Most of metal ions come from the untreated disposal of industrial and municipal wastes. The presence of metal ion above the maximum tolerable limitation in the water ecosystem might cause a potentially damaging effect on the human psychological and other biological systems [2]. There are many wastewater treatments such as adsorption, ion exchange, precipitation, membrane filtration, electrochemical oxidation, chemical oxidation that have been used to remove metal ions in water [3]. However, these techniques still have

a limitation such as high cost, unavailable raw materials, high energy requirement and poor adsorption capacity. Activated carbon has been well known as a highly porous material with excellent physical and chemical characterization [4]. It was widely used as an adsorbent in the purification and separation methods in wastewater treatment [5]. The significant advantages of activated carbon are large surface area, high pore volume and pore size distribution including variable characteristics of functional group on surface [6,7]. Activated carbon can be prepared from a variety of agricultural wastes such as palm kernel, rich husk, sugarcane bagasse and banana peel that are widely used for the removal of heavy metals. Exploration of banana fruit bunch as raw material might encourage the researchers to find new resources [8–10]. Banana fruit bunches are solid wastes that do not have many applications. It is interesting to introduce

* Corresponding author.

banana fruit bunch as alternative raw materials which have high lignocellulosic compounds [11,12]. Banana fruit bunch could be considered as a candidate of activated carbon because of its low cost, low energy consumption and availability in large quantity, which is a good adsorbent for heavy metal removal [13,14]. This study can reveal the critical raw materials of activated carbon production.

Preparation of activated carbon can be carried out by physical and chemical process. In physical activation process, preparation generally was conducted under steam or carbon dioxide at high temperature in the range of 800°C–1,200°C. In chemical activation process, the raw materials were impregnated using strong acid (H_3PO_4 , H_2SO_4 , etc.), strong base (KOH, NaOH, etc.) or salt ($ZnCl_2$) as activating agents and carbonized in lower temperature ranging from 300°C to 800°C [15,16]. Recently, hydrothermal carbonization process was intensively studied by conversion of biomass to activated carbon. The temperature of carbonization is dependent on the type of raw material ranging from 150°C to 350°C [17].

The main objective of this research is to evaluate the ability of activated carbon for the removal of Cu(II) ions from aqueous solution. Characterization of pore structures was carried out by gas sorption analyzer. Surface morphology was imaged by scanning electron microscopy-energy dispersive X-ray (SEM-EDX). Surface functional groups were determined by Fourier transform spectroscopy infrared (FTIR). Amount of Cu(II) removal was calculated by the adsorption atomic spectroscopy.

2. Materials and methods

2.1. Materials

Banana fruit bunch was collected from local banana seller, cut down into small size (0.5–1.0 mm), washed thoroughly to remove the dirt using distilled water and dried in an oven at 105°C for 24 h. Hydrothermal reactor was designed from stainless steel with 1 cm thickness, 25 cm high and 10 cm diameter. All chemicals such as cupric sulfate ($CuSO_4 \cdot 5H_2O$), sodium hydroxide (NaOH), nitric acid (HNO_3) and hydrochloric acid (HCl) were of analytical grade and used without further purifications.

2.2. Preparation of activated carbon

Accurately weighed samples of dried banana fruit bunch was impregnated at different concentrations: 10%, 20% and 30% of NaOH. The mixtures were refluxed at 80°C for 6 h. It was filtered, washed for few times to reduce the excess of base content while adjusting its pH to 6–7 by using HCl solution and continued to dry in oven at 110°C. Dried activated carbon was transferred into hydrothermal reactor and added 500 mL of distilled water. The temperature of reactor was setup and moderately heated to 250°C for 6 h as contact time. After cooling down, the yield of activated carbon was washed with 1.0 M nitric acid and dried in oven at 110°C for 24 h and then kept in desiccator for further analysis.

2.3. Adsorption capacity of activated carbon

Activated carbon with the highest surface area was used as a model to study the effectiveness of adsorption capacity.

The rate of sorption was studied by batch method at room temperature. The reaction was initiated by adding 50 mg of activated carbon to 50 mL of Cu(II) solution with concentration of 50 ppm. The mixture was stirred for 30 min at pH 7. For next treatment, a similar procedure was carried out with several parameters including pH, adsorbent dose, concentration and contact time. After equilibrium, the reaction was stopped, and the mixer solution was filtered to obtain Cu(II) solution. The residual of Cu(II) was calculated to obtain the amount of adsorbed Cu(II) at equilibrium. The percentage Cu(II) ion removal (%R) and amount of Cu(II) ion per unit mass of adsorbent (q_e , $mg\ g^{-1}$) were calculated using Eqs. (1) and (2), respectively, as follows:

$$\% R = \frac{C_0 - C_e}{C_0} \times 100 \quad (1)$$

$$q_e = (C_0 - C_e) \times \frac{V}{M} \quad (2)$$

where C_0 and C_e are initial and equilibrium concentration of metal ion in solution ($mg\ L^{-1}$), respectively. V and M are volume of metal solution (L) and weight of adsorbent used (g), respectively. The equilibrium adsorptions were evaluated at room temperature using Langmuir and Freundlich isotherm models [18].

2.4. Adsorption isotherms study

Equilibrium adsorptions were evaluated using the Langmuir and Freundlich isotherm models. Langmuir isotherm is considered as monolayer adsorption on the homogeneous micropores, while the Freundlich isotherm occurs on the multilayer on the heterogeneous pores involving mesopores.

The Langmuir isotherm model is shown as Eq. (3).

$$\frac{C_e}{q_e} = \frac{1}{bQ_m} + \frac{C_e}{Q_m} \quad (3)$$

where the value of C_e ($mg\ L^{-1}$) is the concentration of the heavy metal solution at equilibrium and q_e ($mg\ g^{-1}$) is the amount of heavy metal adsorbed at equilibrium. Q_m is the maximum adsorption capacity which indicates the monolayer as fully filled by the adsorbate and b is a Langmuir constant.

The Freundlich isotherm model is calculated from linear parameter as Eq. (4).

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (4)$$

The values of K_f and $1/n$ are the Freundlich constant related to the adsorption capacity and adsorption intensity, respectively. Their values were calculated from the intercept ($\log K_f$) and slope ($1/n$) based on the linear plot of $\log q_e$ vs. $\log C_e$.

3. Result and discussion

3.1. Nitrogen adsorption–desorption isotherm of activated carbon

Determination of nitrogen adsorption–desorption isotherm was carried out at temperature 77 K and the relative

pressure in the range of 0.005–0.99. The nitrogen isotherm was used for studying the pore structure of activated carbon including surface area, pore size distribution and pore volume. Determination of model nitrogen isotherm was carried out according to the IUPAC classification. Nitrogen adsorption–desorption isotherms for activated carbon prepared with 10% and 30% of NaOH exhibited combination of type III and V which are shown in Figs. 1a and c. These isotherm models show a weak interaction between metal ion and adsorbent which displayed the convexities of the isotherm curves toward the relative pressure axis. The isotherm type III related to a nonporous or macroporous structures, and isotherm type V with hysteresis showed clearly mesoporous structures [19,20]. However, activated carbon prepared with 20% solution of NaOH exhibited a combination type II and IV isotherms relating to the mesopores which was shown in Fig. 1b. All nitrogen isotherms show wide hysteresis corresponding to the multilayer with

meso- or macropore characterization. However, the hysteresis loops exhibited uncommon desorption curve or beyond the rule of IUPAC classification. According to the rule of IUPAC classification, the hysteresis curves of adsorption and desorption isotherm should be overlap at relative pressure below 0.4. The reason of this phenomenon is that the pores decrease in size or broken during adsorption–desorption process and closed the open site of porosity.

The effects of concentration of NaOH as activating agent were found to be most important factors in the formation of pore structure. Table 1 summarizes the textural properties of activated carbon including BET surface area (S_{BET}), micropore surface area using Dubinin–Radushkevich (DR) method (S_{micro}), Average pore diameter ($A_{\text{pore diameter}}$) and total pore volume at $P/P_0 = 0.99$ (T_V). The BET surface area increased from 20 to 34 $\text{m}^2 \text{g}^{-1}$ with increasing the concentration, thereafter, decreased to 28 $\text{m}^2 \text{g}^{-1}$ at concentration of 30% solution of NaOH. The effects of concentration of

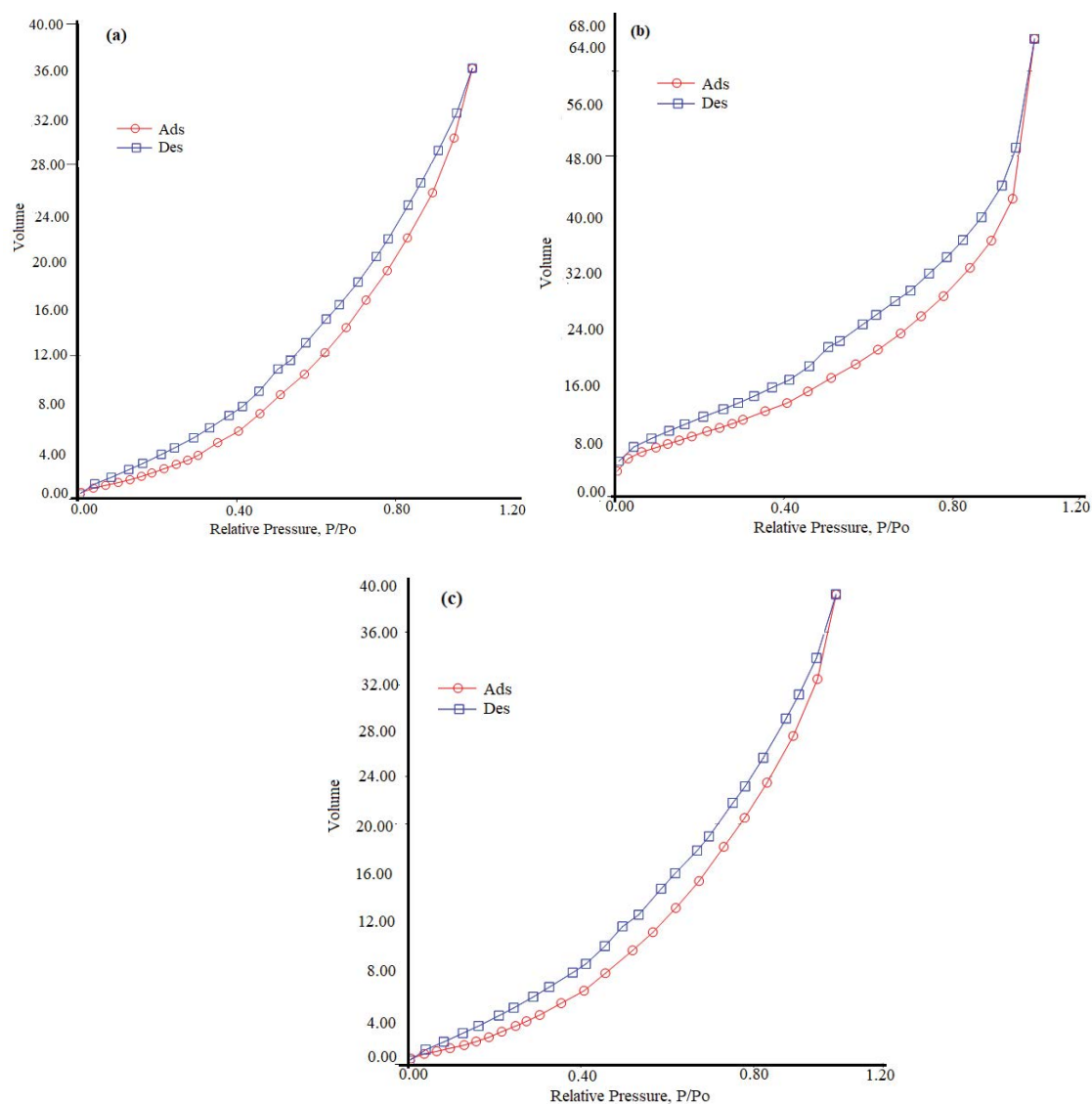


Fig. 1. Nitrogen adsorption–desorption isotherm of activated carbon from banana fruit bunch at (a) 10%, (b) 20% and (c) 30% solution of NaOH.

Table 1
Textural characteristics of activated carbon prepared from banana fruit bunch

Concentration of NaOH (%)	S_{BET} ($\text{m}^2 \text{g}^{-1}$)	DR-method S_{micro} ($\text{m}^2 \text{g}^{-1}$)	$A_{\text{pore diameter}}$ (nm)	T_v ($P/P_0 = 0.99$) ($\text{cm}^3 \text{g}^{-1}$)
10	20	12.07	5.692	0.0557
20	34	41.21	5.974	0.0998
30	28	12.89	4.394	0.0607

NaOH have initiated the reaction between activating agent and cellulose of raw materials. The swelling and dissolution of NaOH into cellulose network caused a broken bonding at the intra- and inter- molecular. The rearrangement of carbon in the cellulose was supported by the hydrothermal carbonization to form porous structures. Increasing the concentration of NaOH may increase the cleavage of hydrogen and oxygen bonds in cellulose. In additional, the excess concentration of NaOH solution may burn off carbon skeleton resulting failure to form pore formation and to collapse the pore wall [21]. In general, the results of BET surface area of the activated carbon were not satisfactory compared with the previous work using the KOH activation [22]. As seen in Table 1, the pore diameters of activated carbon were higher than 2 nm corresponding to the presence of mesopore.

3.2. Functional group of activated carbon

The spectra of FTIR prepared at different concentrations of NaOH are shown in Fig. 2. The major spectra of activated carbon were obtained at the band in the range of $1,582\text{--}1,541 \text{ cm}^{-1}$; $1,390\text{--}1,436 \text{ cm}^{-1}$; $1,030\text{--}1,009 \text{ cm}^{-1}$ and $475\text{--}648 \text{ cm}^{-1}$. A weak band at $3,354 \text{ cm}^{-1}$ is only obtained at activated carbon prepared with 10% of NaOH attributing to the O–H stretching of the hydroxyl group and adsorbed water. The band at $1,582\text{--}1,541 \text{ cm}^{-1}$ indicated the C=C stretching band in aromatic rings. The bands at region $1,390\text{--}1,436 \text{ cm}^{-1}$ are ascribed to C–O–H stretching vibration in carboxylic groups and phenol. The bands observed at $1,030\text{--}1,009 \text{ cm}^{-1}$ are generally attributed to the C–O stretching vibration in phenol and alcohol group or the organic silicone Si–O–Si. The bands at $873\text{--}675 \text{ cm}^{-1}$ are assigned to C–H out-of-plane bending in the aromatic ring. Based on the evaluation of FTIR spectra, it was assumed that the activated carbon derived from banana fruit bunch could contain high oxygen functional groups.

3.3. Morphology structure of activated carbon

Morphology structure is very important to study the pore surface of activated carbons. The effects of different concentration clearly influenced the type of surface structures. Activated carbon images with many tubular pores, long cavity form and irregular types are shown in Figs. 3a and c. However, activated carbon image for 20% of NaOH shows irregular sizes and rough pore developments with high fraction of carbon materials as displayed in Fig. 3b. The EDX analysis proved that there are four major elements found on the surface of activated carbon including carbon, calcium, silicate and oxygen.

3.4. Adsorption study of activated carbon

The low cost of activated carbon from banana fruit bunch was used as adsorbent for removal of Cu(II) from aqueous solution. Adsorption studies were carried out for activated carbon impregnated by 20% of NaOH which has the highest BET surface area. Equilibrium adsorption of Cu(II) was evaluated under various parameters such as pH, concentration, contact time and adsorbent dose. The equilibrium adsorption was evaluated by the Langmuir and Freundlich isotherms and kinetic model.

3.4.1. Effect of pH

Removal of Cu(II) ions from aqueous solution by the activated carbon was carried out by the different pH

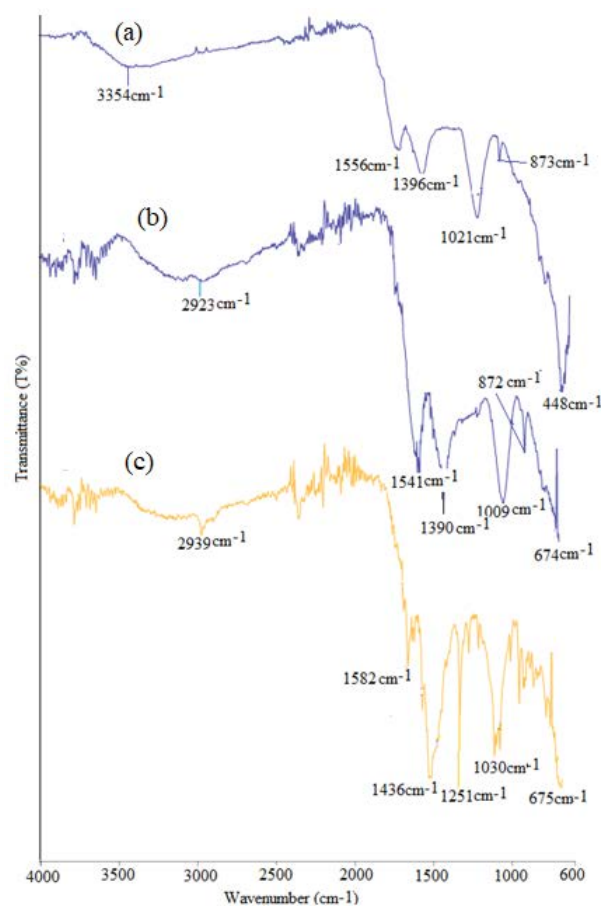


Fig. 2. FTIR spectra of activated carbon from banana fruit bunch at different concentration solution of NaOH: (a) 10%, (b) 20% and (c) 30%.

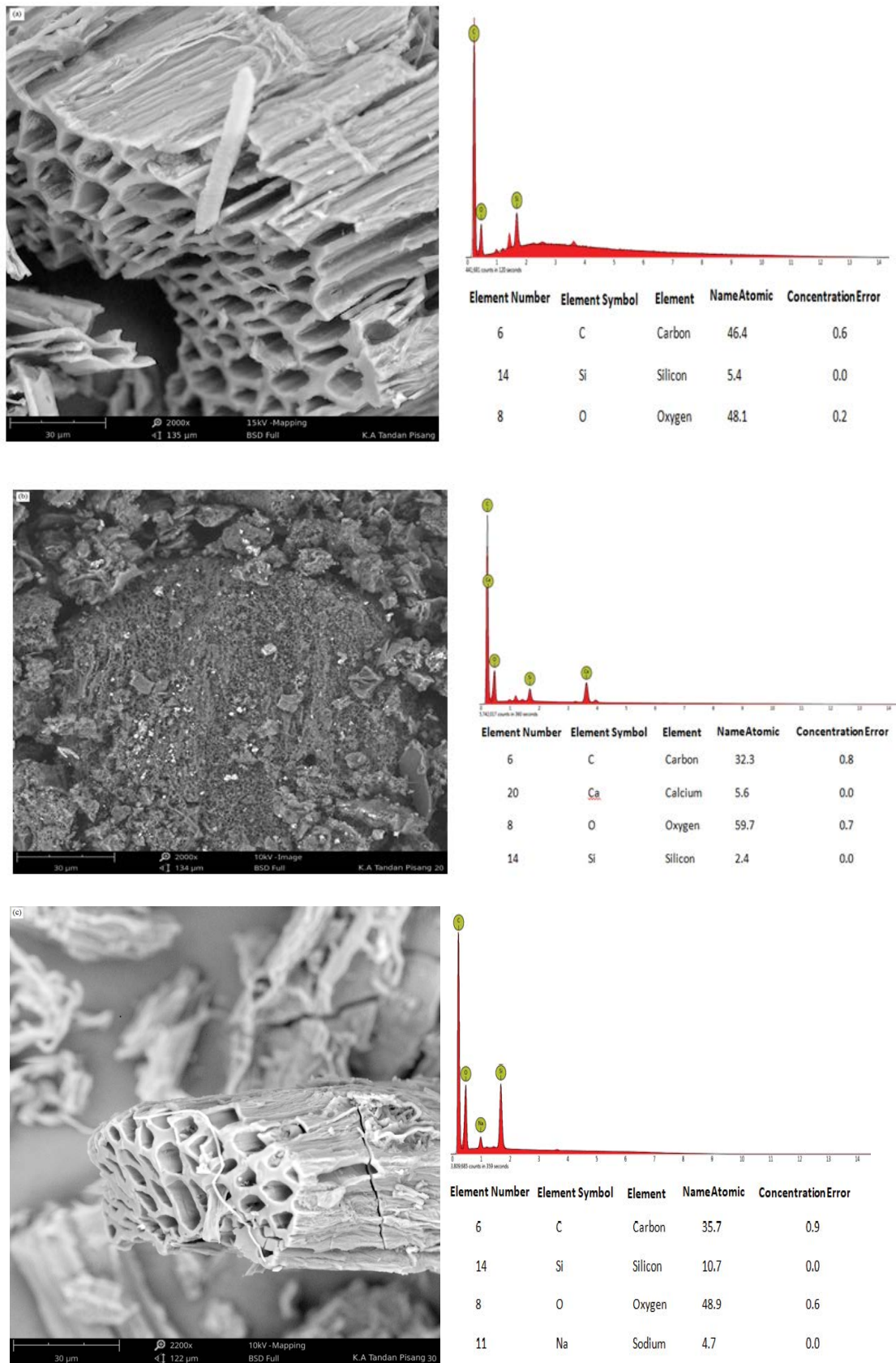
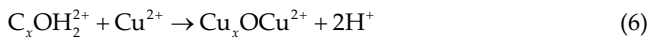
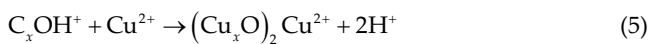


Fig. 3. Morphology structures of activated carbon prepared from banana fruit bunch at different concentrations (a) 10%, (b) 20% and (c) 30% solution of NaOH.

parameters. Percentage of Cu(II) removal vs. different pH is shown in Fig. 4a. The adsorption capacity was clearly influenced by the change of pH solution. The curve sharply increased from pH 3 to pH 5 as an optimum adsorption and started to decrease after pH 5. It was assumed that the adsorption mechanisms on the surface are available in both physical adsorption (Van der Waals, electrostatic or London dispersion) and chemical adsorption (interaction with chemically coordinated bonding). In acidic condition, the negative charges of surface activated carbon rise due to their release of the positive charge (H^+ ion). Attraction that occurred between Cu(II) and surface of activated carbon is more effective at pH 5 through electrostatic force. At a higher pH solution, the surface maintained their positive charges causing repulsion between Cu(II) and surface of activated carbon. As a result, the adsorption of Cu(II) is less effective due to the weak attraction forces between Cu(II) and surface of activated carbon resulting the decrease of adsorption. Interaction of Cu(II) ion in aqueous solution was assumed as shown in Eqs. (5) and (6).



3.4.2. Effect of contact time

Contact time is a very important parameter to observe the effectiveness of adsorption process. The rate of sorption Cu(II) ion at different contact time is shown in Fig. 4b.

The curve sharply increased at initial contact time from 15 to 30 min and tended to plateau after 30 min. The increasing adsorption could be due to high interaction occurring between Cu(II) and adsorbent as a result of available large number of vacant sites. However, after 30 min, the adsorption reached a constant value which was assumed the saturation of porosity with no more Cu(II) adsorbed in solution.

3.4.3. Effect of adsorbent dosages

Adsorption of Cu(II) ions at different adsorbent doses is shown in Fig. 4c. The effects of adsorbent dose showed a significant change of Cu(II) ion removal. It can be seen that removal of Cu(II) ion decreased starting from 0.5 to 1.0 g which was assumed the saturation of adsorbent in the adsorption process of Cu(II). However, the adsorption process sharply increased and tended to plateau up to 2 g. The increasing adsorption could be caused by the increasing active sites such as surface area and pores on the activated carbon resulting in the easier penetration of the metal ion to the adsorption sites. The equilibrium adsorption occurred after the adsorbents dosages reached 1.5 g.

3.4.4. Effect of concentration

Concentration is very important parameter to investigate the equilibrium adsorption. The reaction was carried out at different concentration in the range of 50–200 $mg\ g^{-1}$. Adsorption of Cu(II) ion slightly decreased from 50 to 100 $mg\ L^{-1}$ and sharply dropped to 150 $mg\ L^{-1}$ as shown in Fig. 4d. The reason for this phenomena is unavailable enough vacant sites on activated carbon to adsorb Cu(II) ion.

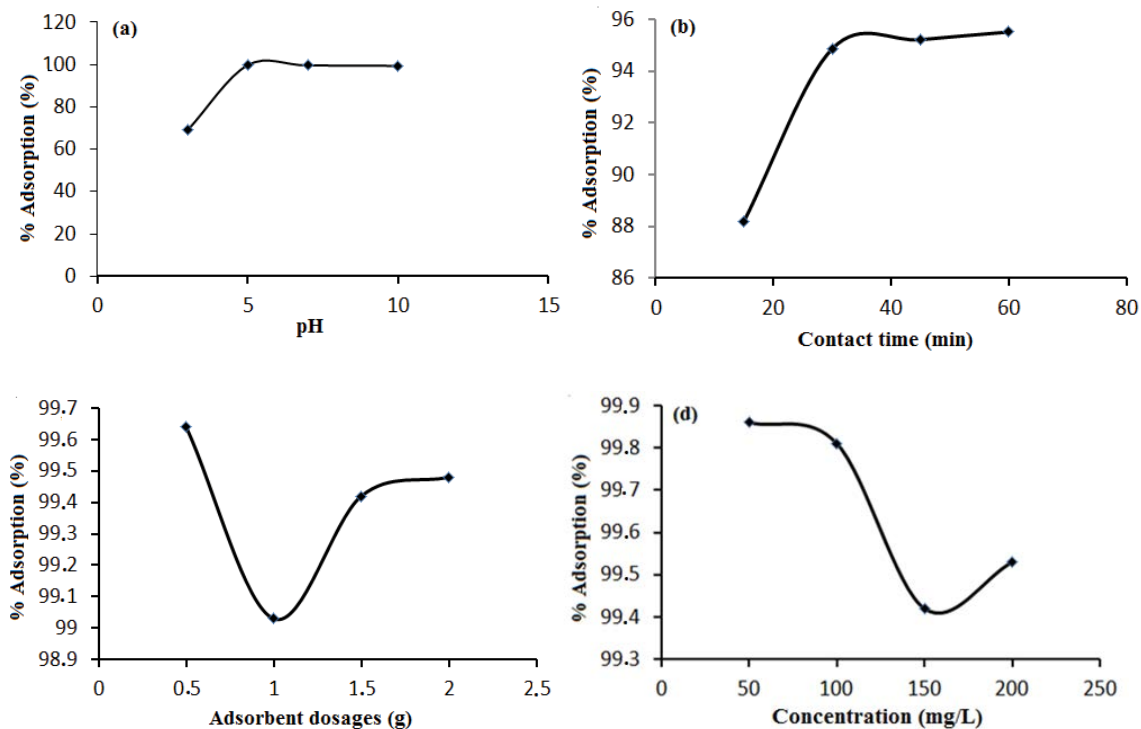


Fig. 4. Adsorption capacity of Cu(II) from aqueous solution (a) pH, (b) contact time, (c) adsorbent dosages and (d) concentration.

The maximum condition of the adsorption of Cu(II) is effective at concentration 50 mg L⁻¹.

3.5. Adsorption isotherm

The equilibrium adsorption of the activated carbon could be evaluated using Langmuir and Freundlich isotherm models. The sorption process is designed and predicted how the metal ion is adsorbed by the adsorbent from the liquid phase at a certain concentrations. In general, the equilibrium adsorption models describe the interaction occurring between adsorbate and adsorbent in the adsorption process. The Langmuir adsorption model was well-known applied to describe the specific homogeneous site on the adsorbent, and successfully used to describe the monolayer adsorption. The curve of Langmuir isotherm model for the adsorption of Cu(II) from aqueous solution at certain concentration is shown in Fig. 5a. The slope and intercept of the linear plot of C_e/q_e against C_e are used to determine the values of maximum adsorption capacity (Q_m) and Langmuir constant (b).

The Freundlich adsorption model was widely used to develop an empirical equation of heterogeneous surface with different classes of porous sites corresponding to the multilayer sorption on the surface. The Freundlich isotherm model for the adsorption of Cu(II) is shown in Fig. 5b. Table 2 displays information about the adsorption of Cu(II) based on the Langmuir and Freundlich isotherms. The maximum adsorption capacity (Q_m) and the correlation coefficient (R^2) were calculated and exhibited fairly good linear fit of the correlation coefficient (R^2) for both Langmuir and Freundlich isotherm models. Comparison of the correlation coefficient (R^2) values for both isotherms showed that the Freundlich isotherm and the Langmuir isotherm are of 0.9401 and 0.8875, respectively. Consequently, the result suggested that the experimental data for the Freundlich isotherm model is better isotherm than the Langmuir isotherm model.

The sorption process of Cu(II) on the activated carbon following the Freundlich isotherm shows heterogeneous pores or multilayer which was supported by the presence of hysteresis loop on the nitrogen adsorption–desorption isotherm. In contrast, the Langmuir isotherm which is presence as the homogeneous pores showed unfavorable properties with correlation coefficient less than 0.9.[23]. This study revealed that activated carbon derived from banana fruit bunch was successfully used as adsorbent for removal of Cu(II) by multilayer process. The activated carbon characteristics were supported by their physical and chemical properties such as predominantly mesoporous structure and rich of oxygen functional groups. The application of activated carbon for removing metal ions has been proved. These result may encourage the research to explore alternative raw material for preparation of activated carbon. Hydrothermally carbonization process was supported to use low energy consumption during process. This work has been enrichment the knowledge of activated carbon from banana fruit bunch, hydrothermally low carbonization process and removal of Cu(II) from aqueous solution.

3.6. Adsorption kinetics

In order to further study the adsorption mechanism of Cu(II) removal from the aqueous solution, the experimental data were calculated based on distinct functions of the pseudo-first-order and pseudo-second-order. The linearized form of kinetic model for the pseudo-first-order was calculated as Eq. (7) [24,25]:

$$\log(q_e - q_t) = \log q_e - \frac{K_1}{2.303} t \quad (7)$$

where q_e is the amount of Cu(II) adsorbed onto the adsorbent at equilibrium (mg g⁻¹), q_t is the amount of Cu(II) adsorbed

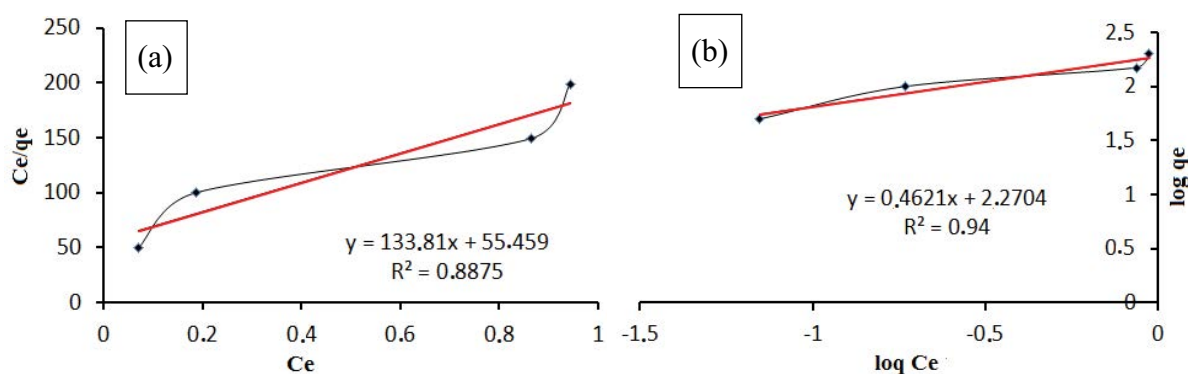


Fig. 5. Isotherm models of (a) Langmuir and (b) Freundlich.

Table 2
Langmuir and Freundlich isotherm model parameters

Metal ion	Langmuir isotherm			Freundlich isotherm		
	Q_m (mg g ⁻¹)	b (L mg ⁻¹)	R^2	K_F	n	R^2
Cu(II)	5.458	4.2115	0.8875	1.9509	2.1645	0.9400

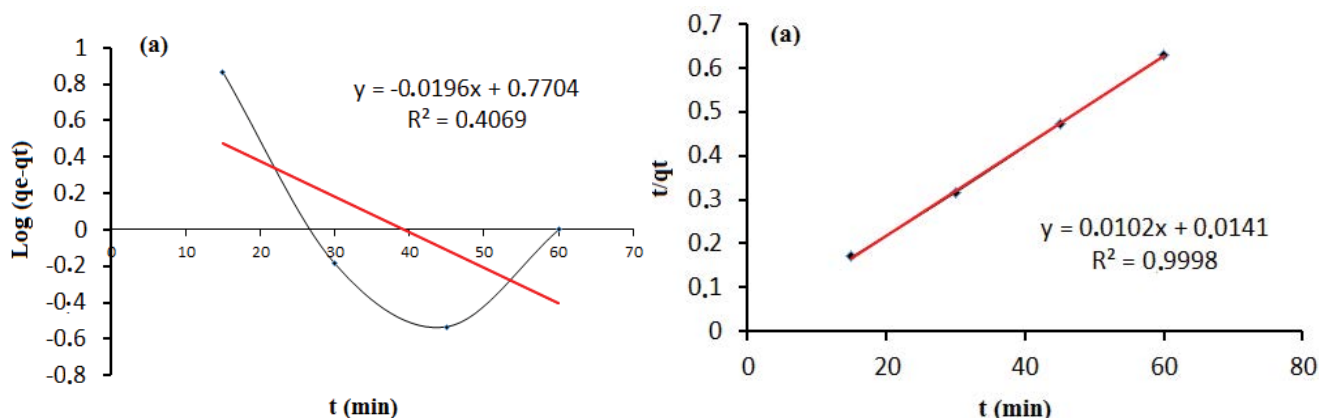


Fig. 6. Adsorption kinetics of (a) the pseudo-first-order and (b) the pseudo-second-order.

Table 3
Pseudo-first and second-order for activated carbon

Adsorbates	Pseudo-first-order			Pseudo-second-order		
	q_e (mg g ⁻¹)	K_1 (min ⁻¹)	R^2	q_e (mg g ⁻¹)	K_2 (g mg ⁻¹ min ⁻¹)	R^2
Cu(II)	5.8939	-0.0451	0.0407	98.0392	0.0074	0.9998

onto the adsorbent at any time t (mg g⁻¹), and K_1 (min⁻¹) is the rate constant of the pseudo-first-order corresponding to the correlation coefficient which is obtained from linear curve of $\log(q_e - q_t)$ vs. t .

The kinetic model for pseudo-second-order is given as Eq. (8):

$$\frac{dq}{dt} = K_2 (q_e - q_t)^2 \quad (8)$$

The integration in linearized form of the pseudo-second-order is shown in Eq. (9) [26].

$$\frac{1}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t \quad (9)$$

where K_2 is the rate constant of pseudo-second-order (g mg⁻¹ min⁻¹), q_e (mg g⁻¹) and q_t (mg g⁻¹) are the amount of Cu(II) adsorbed onto adsorbent at equilibrium and any time t (min⁻¹), respectively. The rate constant of pseudo-second-order (K_2) can be obtained from the slope and intercept of the plot of t/q_t against t .

The linearized forms of plot from $\log(q_e - q_t)$ against t for the pseudo-first-order adsorption of Cu(II) were investigated. The result exhibited that pseudo-first-order adsorption of Cu(II) was not satisfactory because the correlation coefficients (R^2) is far below unity ($R^2 < 0.9$). However, the linearized forms of plot t/q_t against t for the pseudo-second-order showed better presentation with the correlation coefficient (R^2) are closer to unity ($R^2 > 0.9$) as shown in Fig. 6. The result indicates that the adsorption kinetics of Cu(II) removal was favorable following the pseudo-second-order.

Table 3 shows the values of adsorption kinetics consisting of K_1 , K_2 , q_e and corresponding to the correlation coefficients (R^2) of removal of Cu(II) onto activated carbon.

4. Conclusions

In summary, three types of activated carbon were successfully obtained from solid waste of banana fruit bunch as low-cost adsorbent. Activated carbon prepared with 20% of NaOH was employed as a potential adsorbent for the removal of Cu(II) ions from aqueous solution. Activated carbon consists of many tubular pores on the surface which has a good probability to trap Cu(II) ion in the adsorption process. The adsorption equilibrium data described that the Freundlich is better than the Langmuir isotherm model corresponding to the best values of correlation coefficient. The adsorption kinetics model showed a better pseudo-second-order with the correlation coefficient $R^2 = 0.9998$. It was concluded that banana fruit bunch waste can be used as alternative raw material for activated carbon production and employed for the removal of Cu(II) ions from aqueous solution.

Conflict of interests

The authors declare that there is no conflict of interest regarding the publication of this paper

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