

Adsorption of copper (II) ions from aqueous solution using banana stem biochar

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ABSTRACT The pollution of copper (II) ions, (Cu^{2+}) in water has become one of the big concerns since this metal can spread easily through living organisms and the environment. The accumulation of ecotoxicological Cu^{2+} ions is extremely dangerous and causes severe adverse effects on plants, animals, aquatic life and humans. This toxic heavy metal can be removed effectively with an adsorption treatment method by using biochar, a green adsorbent made from biomass feedstock. In this study, the adsorption capacity of biochar derived from banana stems for the removal of Cu^{2+} ions are examined. The dried stem material was carbonized via slow pyrolysis at 300 °C for 1 h. The produced banana stem biochar was characterized by Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray Spectroscopy (EDX). The effect of the adsorption parameters that are initial Cu^{2+} ions concentration (100 – 400 mg/L), biochar dosage (0.1 – 0.6 g), pH solution (2 – 12), and stirring speed (60 – 210 rpm) were investigated in batch adsorption experiments at room temperature. Experimental setup parameters that affect the adsorption have been optimized by the one-factor-at-a-time (OFAT) technique. Adsorption of Cu^{2+} ions on biochar increased until reaching equilibrium with increasing initial Cu^{2+} concentration, increasing pH, increasing stirring speed up to 120 rpm and decreasing dosage. The maximum Cu^{2+} ions amount adsorbed of 300.10 mg/g was achieved with optimum adsorption parameters that are 350 mg/L of initial Cu^{2+} ions concentration, pH 6, 0.1 g of biochar dosage and 120 rpm stirring speed. Due to the high adsorption efficiency, banana stems are potentially to be employed as an alternative adsorbent material for removing Cu^{2+} ions from aqueous solution. As banana stems are abundantly available agricultural wastes, its utilization as adsorbent feedstock will reduce the production and operation cost of removing various toxic metals from wastewater and be beneficial in addressing environmental issues resulting from improper disposal of agricultural wastes.

KEYWORDS: Adsorption; Banana stem; Biochar; Copper (II) ion; Removal

Received 12 February 2024 Revised 25 June 2024 Accepted 26 June 2024 In press 26 June 2024 Online 29 June 2024

© Transactions on Science and Technology

Original Article

INTRODUCTION

Fast urbanization, global industrialization and technological progress result in large quantities of toxic heavy metals being discharged into the water and wastewater. This has sparked interest among researchers due to these metals' poisonous and carcinogenic properties. Heavy metals in water bodies cause environmental pollution by producing a significant toxic impact on aquatic ecosystems and waterborne diseases among the public. Copper (Cu) metal is a popular heavy metal with high concentrations in water resources that become water contaminants. Cu is mostly employed in producing wire, sheet metal, pipes, and other metal products as a metal or alloy. Effluents containing high concentrations of toxic Cu^{2+} ions are mainly discharged from manufacturing industries like electric and electronic components and devices, electric machines, printed circuit boards (PCB), metal plating and printing, batteries, paints and pigments, and chemicals manufacturing processes using copper catalysts and copper salt (Kuwer *et al.*, 2021; Vardhan *et al.*, 2020). Cu compounds are also utilized in agriculture as a treatment for plant diseases. Cu is usually found near industrial settings, waste disposal sites, smelters, landfills, and mines. When Cu and its compounds are released into the water, the dissolved Cu^{2+} ions can be transported in surface waters as free Cu, Cu compounds, or, more frequently, Cu^{2+} ions attached to suspended particles. The presence of Cu^{2+} ions at high concentrations results in eco-toxicity and bioaccumulation in the food chain, making it detrimental to aquatic organisms and the aquatic environment (Katiyar *et al.*, 2021). According to the United States Environmental Protection Agency,

the permissible limit of Cu ions in industrial effluents is 1.3 mg/L while the World Health Organization states the maximum permissible Cu concentration in water used is 2.0 mg/L (USEPA, 1991; WHO, 2011). Humans exposed to these metals, either acutely or chronically, can develop a variety of health issues, such as haemolysis, cirrhosis, anaemia, nephrotoxic and hepatotoxic effects and can even cause death (Zhu *et al.*, 2020). The removal of these rising heavy metals from wastewater represents a research key issue to evaluate the reusability of this wastewater or discharge to the environment.

There are many treatment methods such as chemical precipitation, electrochemical purification, membrane filtration, reverse osmosis and ion exchange that have been used for removing heavy metals from wastewater however in this paper, the adsorption of toxic substances in the waste will be focused on (Bashir *et al.*, 2022; Xiao *et al.*, 2020). Other methods than adsorption have several drawbacks, including costly chemicals, poor removal efficiency, and the formation of secondary wastes during the treatment process (Çelebi, 2020). Adsorption is the most prominent method due to its high reliability, simple design and sludge-free operation. The commonly used adsorbent, activated carbon (AC), is an adsorbent with large porous surface area, controllable pore structure, thermo-stability and low acid/base reactivity. It can eliminate a wide variety of dissolved organic and inorganic contaminants from aqueous medium. The main challenge to its implementation includes high production costs and the complexity of regeneration, notwithstanding its advantages in adsorption processes (Torres-Caban, 2019). Therefore, there is a need to search for another adsorbent potential that would provide low-cost, eco-friendly processes and high efficiency to maintain heavy metal ion pollution even at a relatively low concentration.

According to recent studies, the most effective way to provide adsorbents for waste management and environmental protection is to convert waste materials into biochar. These studies focused on adsorbents derived from those mentioned above or like materials. Compared to activated carbon, biochar requires less energy during manufacture, making it more affordable. Moreover, agricultural biomass is one of the most plentiful renewable resources that have been exploited as the suitable primary source of biochar feedstocks (Ajien *et al.*, 2022; Gajendiran *et al.*, 2023; Li *et al.*, 2023; Thoe *et al.*, 2019).

Bananas are widely grown particularly in tropical nations like Malaysia. The increased demand for banana fruit production results in greater banana stem leftovers. Following harvest, the stems are normally burned as waste without being properly removed or simply discarded, which adds to the already existing environmental issue. Utilizing these banana stems as an adsorbent helps to solve this issue and turn the waste recycling theory into practice. As a lignocellulosic material that comprises cellulose, hemicellulose, and lignin content, banana stem waste has been considered an eco-friendly biochar feedstock for treating and removing various organic, inorganic, heavy metals and dye contaminants in water and wastewater. Previous studies utilized banana stem biochar for the adsorption of methylene blue (Liu *et al.*, 2019), removal of cadmium from industrial phosphoric acid (Bahsaine *et al.*, 2023), adsorption of hexavalent chromium in aqueous solution (Xu *et al.*, 2018) and removal of amoxicillin antibiotic from wastewater (Chakhtouna *et al.*, 2021). Although the resulting banana stem biochar has a smaller surface area and less pore volume, the presence of various functional groups and high carbon content makes the adsorption capacities of biochar on heavy metals and dyes quite comparable to the activated carbon (Bahsaine *et al.*, 2023; Xu *et al.*, 2018; Liu *et al.*, 2019). Besides that, the biochar was produced in a simple and easy procedure and with low energy usage, showing the production costs are more economical. Up to now, no research has been done on the use of banana stem biochar for Cu²⁺ ions adsorption. Therefore, this research attempts to investigate the potential of the banana stem biochar as an alternative adsorbent for the removal of Cu²⁺ ions from an aqueous solution. The adsorption efficiency of the biochar was evaluated by

examining adsorption factors such as initial Cu^{2+} ions concentration, biochar dosage, pH solution and stirring speed. Therefore, this research aims to investigate the potential of banana stem biochar as an alternative adsorbent for the removal of Cu^{2+} ions from an aqueous solution. The biochar capability in adsorbing Cu^{2+} ions was evaluated by examining adsorption factors such as initial Cu^{2+} ions concentration, biochar dosage, pH solution and stirring speed.

METHODOLOGY

Chemicals and Instruments

Copper (II) sulfate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, HCl and NaOH used for analysis were of analytical grade and were purchased from HmbG Chemicals. Copper stock solution (1 g/L) was prepared in a 1 L volumetric flask and then further diluted using distilled water to various Cu^{2+} ions concentration solutions. The instruments include a pH meter (Sartorius PB-10, Germany), an electronic weighing balance (Sartorius TE124S, Germany), an oven (Venticell 55, USA), a muffle furnace (PROTHERM, Turkey), a shaking incubator (Protech SI 1000D, Malaysia) and a UV-VIS Spectrophotometer (Genesys 204001/4 California) for analyzing Cu^{2+} ions concentration after adsorption. The microscopic surface morphology and element components of banana stem biochar were examined using Scanning Electron Microscopy (SEM) which coupled to Energy-Dispersive X-ray Spectroscopy (EDX) (Thermo Fisher, Netherlands).

Preparation of Banana Stem Biochar

The banana stems were collected from Bestari Jaya, Selangor, Malaysia. The stems were cleaned and washed several times with tap water, cut into small pieces and followed by oven-drying at $105\text{ }^\circ\text{C}$ until a steady weight was reached. After that, the samples were placed in a muffle furnace for pyrolysis at $300\text{ }^\circ\text{C}$ for 1 h, then cooled to room temperature. The produced biochar was ground, sieved to obtain a particle size of below $100\text{ }\mu\text{m}$ and later characterized for the properties.

Batch Adsorption Experiments

The Cu^{2+} ions adsorption onto banana stem biochar was investigated in batch mode at room temperature. The experiments were conducted to determine the optimum level of adsorption parameters; initial Cu^{2+} concentration, biochar dosage, pH solution and stirring speed. The parameters have been optimized by the one-factor-at-a-time (OFAT) technique, where only one parameter was investigated at a time, while the other factors remained constant. Batch experiments were carried out with 100 mL of Cu^{2+} ions solution in a 250 mL Erlenmeyer flask. Prior analysis, the Cu^{2+} ions solution was adjusted to the required pH using 0.1 M NaOH or 0.1 M HCl. The amount of biochar dosage added to the solution was in the range of 0.1 g to 0.6 g. At room temperature, the mixtures were agitated at the required speed on a shaking incubator for 60 min. Similar agitation time has been applied by other researchers as the equilibrium reaction attained at 60 min (Sidek *et al.*, 2017; Eleryan *et al.*, 2022). The mixtures were then filtered with Whatman 42 filter paper after the flasks were taken out. A UV-VIS spectrophotometer was used to measure the absorbance of the filtrate at the wavelength that corresponded to its highest absorption (664 nm). The concentration of the Cu^{2+} ions was determined using a calibration standard curve constructed from known Cu^{2+} ion concentrations. All adsorption experiments were repeated thrice and the average results with the standard deviation (SD) were reported. The amount of Cu^{2+} ions adsorbed, q_e (mg/g) to the surface of the biochar was calculated using Equation 1.

$$\text{Amount adsorbed, } q_e = \frac{C_0 - C_f}{m} \times V \quad (1)$$

where C_0 and C_f are the initial and final concentrations (mg/L) of Cu^{2+} ions in the solution, respectively, m is the mass of the biochar (g), and V is the volume of the solution (L). q_e is in mg/g.

RESULT AND DISCUSSION

Standard Solution and Calibration Curve

The UV-VIS spectrophotometer showed that the maximum adsorption of Cu^{2+} ions was measured at the highest wavelength of 664 nm. For the calibration curve study, the standard Cu^{2+} solutions were prepared by diluting the stock solution to a set of concentrations ranging from 0.2 to 1.0 mg/L at pH 7. At room temperature, the absorbance of the standard solutions was determined at 664 nm and the calibration curve was obtained by plotting the absorbance versus the solution concentrations, as shown in Figure 1. The linear regression equation was determined to be $y = 0.09x - 0.006$, with coefficient correlation, $R^2 = 0.998$.

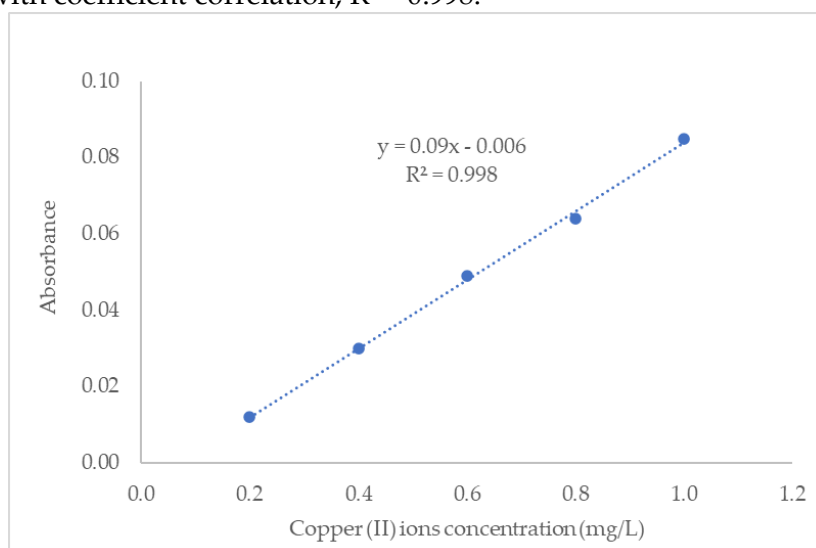
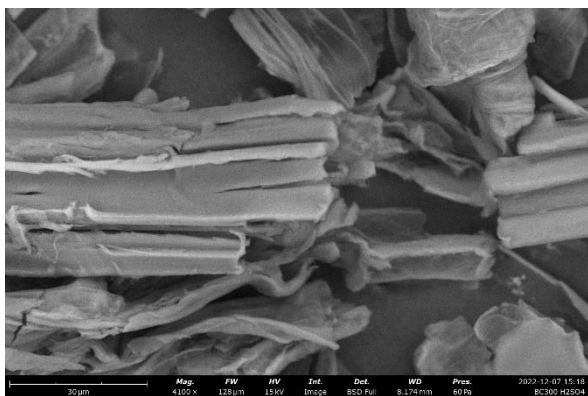


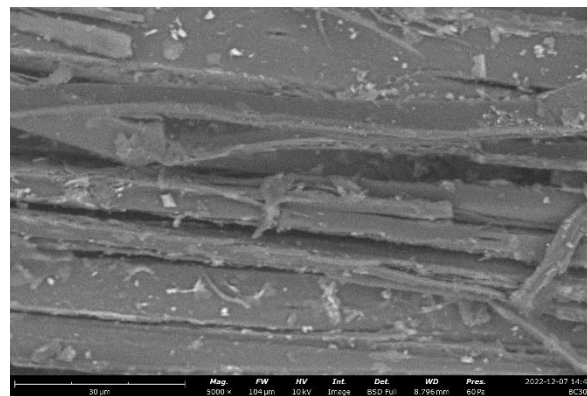
Figure 1. Calibration curve of standard Cu^{2+} ions solution at wavelength = 664 nm, pH = 7 and room temperature.

Surface Morphology and Element Components of Banana Stem Biochar

The SEM images of banana stem biochar before and after adsorption are shown in Figure 2. The surface morphology was observed under magnification of 4000x - 5000x and 30 μm resolution at 10 - 15 kV. The image of biochar before adsorption in Figure 2 (a) shows the rough lignocellulosic surface structure of the biochar, with the opening of irregular micropores of different sizes allowing for the adsorption of Cu^{2+} ions onto various parts of the biochar. It can be observed in Figure 2 (b) where the metal particles were adhered to the biochar surface after the adsorption process. Similar observations using biochar from different agricultural waste for other metals removal were mentioned by Minh *et al.* (2023), Park *et al.* (2019) and Lee *et al.* (2019).



(a)



(b)

Figure 2. SEM image of the banana stem biochar, (a) before adsorption; (b) after adsorption

Table 1 displays the results of the EDX. The banana stem's biochar structure is mainly composed of carbon and oxygen. Chlorine and potassium, two non-essential elements obtained from plants are also found in the biochar (Palapa *et al.*, 2021).

Table 1. EDX results of banana stem biochar.

Element Name	Percentage (%)	
	Atomic	Weight
Carbon	63.093	50.000
Oxygen	29.458	31.100
Chlorine	1.325	3.100
Potassium	6.124	15.800

Effect of Initial Cu^{2+} Ions Concentration

Figure 3 shows the effect of varying initial Cu^{2+} ions concentrations (100, 150, 200, 250, 300, 350 and 400 mg/L) on the adsorption of Cu^{2+} ions was investigated. The adsorption of Cu^{2+} ions onto banana stem biochar increased significantly from 99.10 to 290.00 mg/g with the increasing initial concentration of Cu^{2+} ions solution from 100 to 350 mg/L. The initial concentration of the metal solution acts as the catalyst for resolving metal mass transfer resistances between the aqueous and solid phases. Therefore, the adsorption capability increases with increasing initial concentration of metal ions (Mokkapati *et al.*, 2016). The following Cu^{2+} concentration (400 mg/L) showed the amount of Cu^{2+} ions adsorbed was similar to that at 350 mg/L, indicating the adsorption has achieved equilibrium due to the unavailability of active sites on the biochar surface for more Cu^{2+} ions adsorption (Weng *et al.*, 2014). Since the highest amount of Cu^{2+} ions adsorbed was recorded with the Cu^{2+} concentration of 350 mg/L, thus this concentration was applied for the following adsorption experiments.

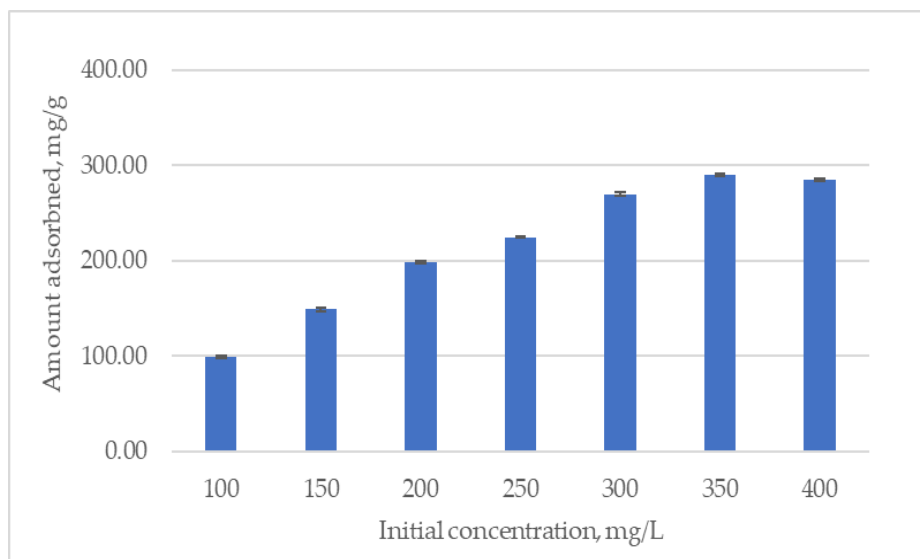


Figure 3. Effect of initial Cu^{2+} concentrations on the adsorption of Cu^{2+} ions onto biochar (biochar dosage 0.1 g; pH 7; temperature 25 °C, stirring speed 120 rpm)

Effect of Biochar Dosage

Figure 4 depicts the effect of biochar dosage variations on the adsorption of Cu^{2+} ions. The amount of Cu^{2+} ions adsorbed is remarkably decreasing with the increase in biochar dosage. The highest amount of Cu^{2+} ions absorbed was 295.25 mg/g with 0.1 g of biochar in 100 mL Cu^{2+} ions (1g/L) and the amount was significantly decreased to 50.03 mg/g when adsorbent dosages were increased to 0.6 g. Thus, the optimum banana stem biochar dosage for this study was chosen as 0.1 g. This could be attributed to a decrease in the effective surface area for adsorption caused by

aggregation of the biochar surface area accessible for Cu^{2+} ions adsorption as dosage increases. The amount adsorbed is getting low because the adsorbent surface has not been chemically or physically modified, it has become saturated with Cu^{2+} ions and is no longer able to absorb additional (Katiyar *et al.*, 2021). A similar decrease in Cu^{2+} ions sorption with an increase in adsorbent dose has been observed in previous studies (Lopičić *et al.*, 2017; Yılmaz & Güzel, 2021).

Effect of pH

The effect of pH solution ranging from pH 2 to pH 12 on the Cu^{2+} ions adsorption onto biochar was examined and the results are presented in Figure 5. The results showed that the amount of Cu^{2+} ions adsorbed was significantly increased by 0.16 mg/g from pH 2 until 6, continued to remain at 299.99 mg/g at pH 8 and then decreased by 0.08 mg/g towards pH 12. The optimum pH was at pH 6 (with 300.00 mg/g amount of Cu^{2+} ions adsorbed), due to the highest adsorption of Cu^{2+} ions onto the biochar surface. The pH value of the solution delimitates the surface charge of the adsorbent and the degree of ionization and the shape of the adsorbate (Saeed *et al.*, 2020). At low pH, the increase in the positivity of the surface charge due of the protonation of the functional groups on the adsorbent surface would prevent the adsorption of Cu^{2+} ions (Boontham & Babel, 2017). As the pH solution increases, a more negatively charged surface becomes accessible for metal uptake, therefore increasing the amount of Cu^{2+} ions adsorbed (Yılmaz & Güzel, 2021). The results were generally consistent with previous studies on Cu^{2+} adsorption (Lopičić *et al.*, 2017).

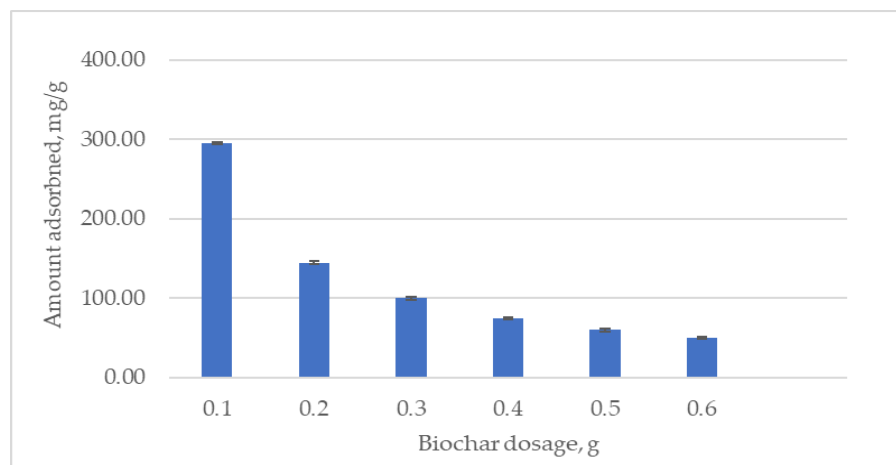


Figure 4. Effect of biochar dosage on the adsorption of Cu^{2+} ions onto biochar (initial Cu^{2+} ions concentration 350 mg/L; pH 7; temperature 25 °C, stirring speed 120 rpm)

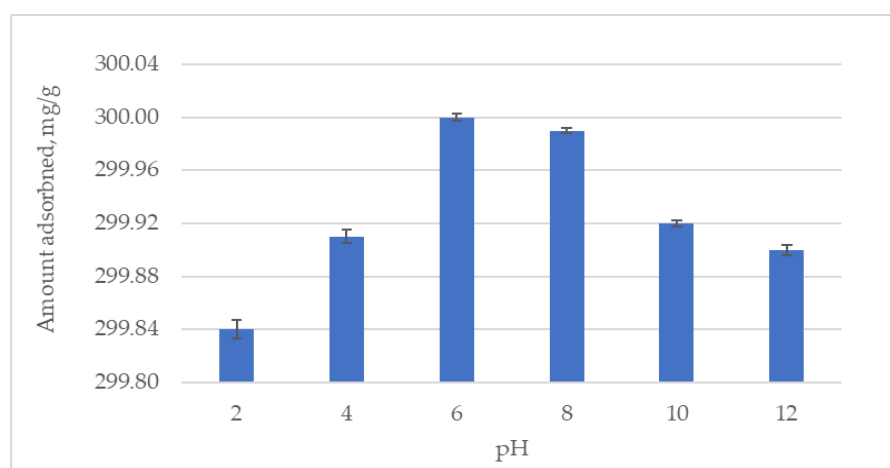


Figure 5. Effect of pH on the adsorption of Cu^{2+} ions onto biochar (initial Cu^{2+} ions concentration 350 mg/L; biochar dosage 0.1 g; temperature 25 °C, stirring speed 120 rpm)

Effect of Stirring Speed

The results of the stirring speed effect on the adsorption of Cu^{2+} ions are shown in Figure 6. The result indicates the adsorption increased by 0.20 mg/g with increasing speed from 60 rpm to 120 rpm and achieved the highest Cu^{2+} ions adsorption of 300.10 mg/g at 120 rpm. This is owing to the optimum stirring speed at which Cu^{2+} ions come into touch with the active porous inner surface regions of banana stem biochar. An inconsistent pattern was seen in the Cu^{2+} ions adsorption when the stirring speed escalated from 150 rpm to 210 rpm. The results showed that the adsorption of Cu^{2+} ions was less than optimal at both low and high stirring speeds. Low speed could not properly disperse the biochar particles in the water to provide active binding sites for Cu^{2+} ions adsorption. It resulted in an accumulation of biochar in the bottom of the water and buried the active binding sites (Bashir *et al.*, 2022). Low stirring speeds are unable to provide the energy required for Cu^{2+} ions to attach to the surface of the biochar (Sezgin & Balkaya, 2016). Conversely, the high stirring speed dispersed the banana biochar particles throughout the water, thus preventing enough time for Cu^{2+} ions to adsorb (Anwar *et al.*, 2010).

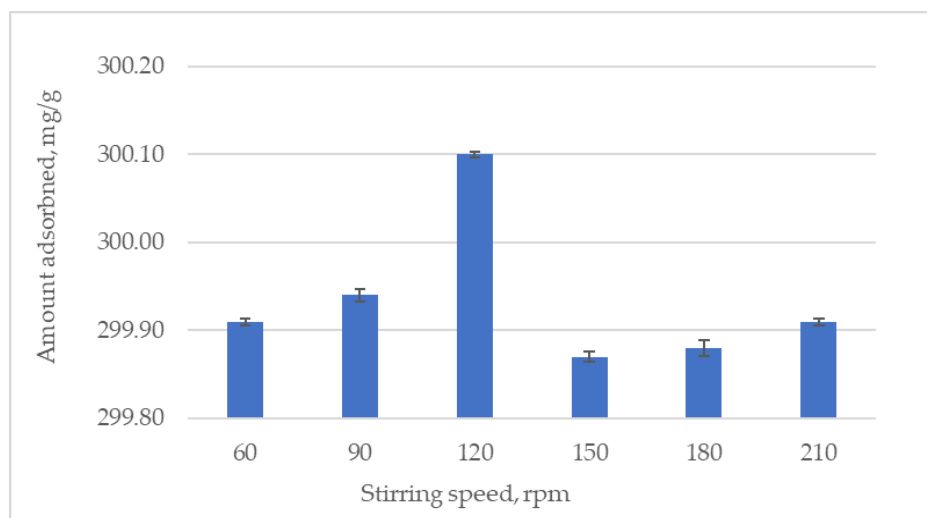


Figure 6. Effect of stirring speed on the adsorption of Cu^{2+} ions onto biochar (initial Cu^{2+} ions concentration 350 mg/L; biochar dosage 0.1 g; pH 6; temperature 25 °C)

CONCLUSION

The banana stem biochar showed great potential to be used as a new adsorbent material for removing copper from an aqueous solution. Banana stems which are usually wasted or left on the plantation can be the feedstock for producing an alternative cheaper adsorbent than commercial activated carbon adsorbent. The adsorbent characterization by the SEM analysis confirmed the rough lignocellulosic and pores surface structure of biochar which signifies a high possibility of copper ions adsorption. The initial Cu^{2+} ions concentration, biochar dosage, pH, and stirring speed significantly influenced the adsorption capacity of the banana stem biochar. The optimum adsorption conditions were 350 mg/L of initial Cu^{2+} ions concentration, pH solution of 6, 0.1 g of biochar dose, and 120 rpm stirring speed, which resulted in the highest Cu^{2+} ions adsorption of banana stem biochar at 300.10 mg/g. The results indicated that the banana stem biochar is effective in the adsorption of Cu^{2+} ions in aqueous solution. As an eco-friendly alternative, this adsorbent could be utilized to solve issues with excessive metal, namely copper ions in wastewater.

ACKNOWLEDGEMENTS

This research was funded by Geran Penyelidikan Negeri Selangor (GPNS/18/01-UNISEL033).

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