Applied Environmental Research
- RESEARCH FOR SUSTAINABLE PLANET -

Research Article

The Effect of Incubation Time and Drainage pH on the Immobilization of Copper in Post Coal Mine Soil Using Sumatera Brown Coal Char

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Abstract

Post coal mine (PCM) activities pose significant environmental challenges. Soils affected by PCM often exhibit increased acidity and elevated levels of heavy metals. In the case of Sungai Buluh PCM in the Jambi Province, Indonesia, copper (Cu) levels in the soil reach 94.79 mg kg^{-1}, surpassing the Indonesian standard of 40 mg kg^{-1}. Sumatera brown coal (SBC), typically is not considered as fuel due to its properties, offers a cost-effective solution for Cu immobilization in such soils. In this study, SBC was subjected to pyrolysis at 400°C for 1 h, sieved through a 10-mesh sieve, and characterized using SEM and FTIR. The soil-char mixture consisted of 250 g of soil and 25 g of char (9:1 ratio), while a control group included 250 grams of soil with no char. The soil's pH significantly influenced Cu adsorption by SBC char. Cu immobility by SBC char was highest at pH 5, with an efficiency of 66.67% from pH 1 to 5. Over a 4-week period, the mobility of Cu in soil containing 10% char decreased by 36.36%. Pseudo-first-order kinetics yielded R^2 values of 0.750 and 0.979 for 0% and 10% char additions, respectively. To inform future research on SBC char's potential for soil amendment, exploring the properties of SBC char produced at different pyrolysis temperatures is advisable. This information can aid in the development of char furnaces with appropriate technology to efficiently produce large quantities of effective SBC char. Additionally, determining optimal incubation times can help predict the frequency of char application in various regions.

ARTICLE HISTORY
Received: 24 Feb. 2023
Accepted: 27 Nov. 2023
Published: 14 Dec. 2023

KEYWORDS
Post coal mine;
Soil amendment;
Pyrolysis;
Copper immobilization;
Low-cost char

Introduction

Mining is an activity of exploring such underground properties as minerals, coal, oil, and natural gas which is carried out to meet human needs. Mining activities have a significant impact on the environment, one of the negative impacts of mining is heavy metal pollution on post-mining land [1–2]. The dismantling and removal of the parent soil material in the process of mining activities cause metals that are initially reductive in the soil to move to the surface and then oxidize. This reductive to oxidative environmental change causes soil conditions in mining areas to become very acidic. Some heavy metal pollutants in the soil include lead (Pb), cadmium (Cd, zinc (Zn), tin (Sn), copper (Cu), nickel (Ni), cobalt (Co), barium (Ba), iron (Fe), and manganese (Mn) [3–5]. The height and weight of plants are severely reduced when the Cu concentration in soil is greater than approximately 250 mg kg^{-1} [6]. Metals that accumulate in the soil can reduce soil microbial activity which affects overall soil fertility, and will later cause the entry of toxic materials

https://doi.org/10.35762/AER.2023023

into the food chain [7–9]. The lignite produced from the ex-coal mining is known as Sumatera brown coal (SBC) and was pyrolyzed to produce char for soil amendment. The use of char in ex-coal mining land is to reduce the activity of heavy metal ions. Char which is formed at a temperature of 400-500°C has various functional groups and relatively contains nutrients [10–11].

Soil conditions are also affected by its acidity level, which can be seen from the pH. Based on research from Houben et al. [12] stated that soil pH significantly increased with the addition of biochar during the incubation experiment, except for day 28 in the 1% biochar treatment where the increase was only significant at $p < 0.064$. This increase in pH is consistent with the alkaline nature of biochar.

In this study, the authors used char derived from lignite coal as a material for making char, due to the characteristics of lignite coal which has a very brittle black color, low calorific value, little carbon content, high water content, lots of ash content, and high sulfur content [13]. This is what causes this type of coal to be unfit as fuel, but it is good to be used as raw material for making char [14]. Char, produced from lignite, was expected to reduce the mobility of copper in thep coal mine (PCM) soils, due to its surface area, negative charge, and porous that can be good candidate for binding to copper ions.

This research aims to investigate the effects of incubation time and drainage pH on the immobilization of copper by SBC char in post-coal mine soil, as well as the acidity of the soil.

Methodology

1) Experimental materials

The soil was carried out in a PCM in Sungai Buluh, Jambi Province, Indonesia [-1.7047901367, 103.3406430922]. The location of the study can be seen in Figure 1.

The sample was taken from the soil layer with 30 cm of depth at the five different spots within the post-mining area [15–16]. Then, the soil samples were cleaned from the impurities (pebble, twig, plastic, leaf, and others), homogenously mixed, sieved by a 10-mesh metal sieve, and dried in an oven at 105°C for 2 h to obtain the constant mass of dry soil.

The acidity of the sample was measured by an electric pH meter based on ASTM D4972 and the amount of the Cu in the samples was analyzed by Atomic Absorption Spectrophotometry (AAS) Perkin Elmer AAS Analyst 100 with EPA 200.7 test method.

2) Char preparation

The char used in this study was Sumatera Lignite with moisture content 15.83% [17], and generated by pyrolysis reactor at 400°C for 1 h. The char was then analyzed by using Fourier-Transform Infrared Spectroscopy (FTIR) Perkin Elmer Spectrum Two to identify the functional groups present in the char. The FTIR spectra were recorded in the range of 4,000–400 cm^{-1} with a resolution of 4 cm^{-1}. The char was also characterized by Scanning Electron Microscope (SEM) FEI Quanta 200, with resolution of up to 1.8 nm. The SEM images were taken at different magnifications to observe the surface morphology and pore structure of the char. The SEM images showed that the char had a porous structure with a large surface area [11, 18–21].

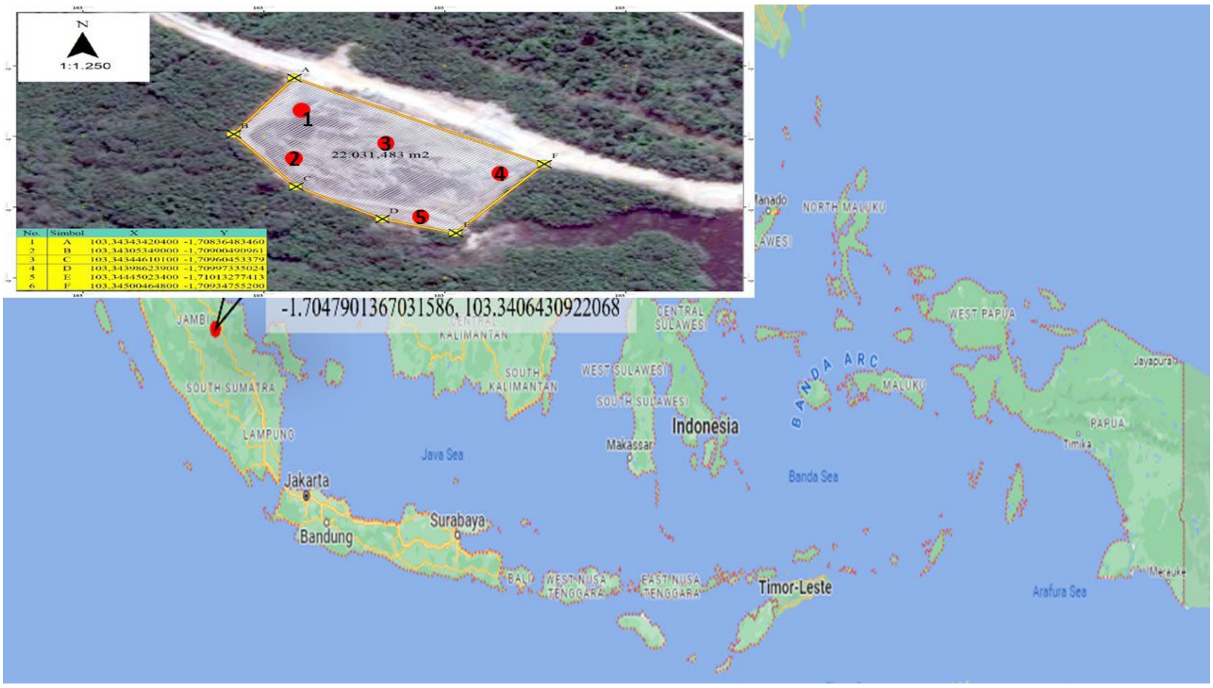


Figure 1 The location of post coal mining in Sungai Buluh, Jambi Province, Indonesia.

3) Soil incubation with various duration

This research was conducted with 10 g of homogenized soil, which had been taken from the PCM area and was serving as the sample of this research. It was then mixed with 0% and 10% of char. This research was carried out for 4 weeks, where the measurement of Cu mobility was performed every week, from week 0 until 4 [22–25]. The soil incubation was performed in a glass bottle. The percentages of char used in this study were 0 and 10. Each glass bottle contained the soil samples obtained in PCM area and char with 250 g of total weight. Then 30 mL of water is added to 50 g of soil [26–28]. The glass bottles were afterwards tightly closed using plastic at the room temperature.

4) Extraction with various pH

Following the water addition, the sample was extracted with an H_2SO_4 solution from Merck with 95.5% of purity and 98% of concentration. The variations of pH were 1, 2, 3, 4, 5, and 6 and the ratio of H_2SO_4 solution to the sample was 10: 1 w/w ratio of H_2SO_4 to soil. The solution was stirred with a magnetic stirrer at 250 rpm for 2 h and afterwards filtered using filter paper called Hawach Medium 110 mm BIO-1-110 [29–31]. The solution obtained from the extraction was then analyzed using AAS so as to compare the Cu content with the 0% and 10% of char additions.

5) Cu adsorption

Absorption (%) is a measure of the efficiency of an adsorbent material (SBC char) in removing Cu from a solution. Analysis with AAS was carried out to determine the concentration of Cu remaining from the sample. The percentage of char adsorption can be determined by calculating the adsorption efficiency through the following formula [32]:

$$\text{Adsorption (\%)} = \frac{[Co - Ce]}{Co} \text{ x } 100 \text{ \%} \qquad \text{(Eq. 1)}$$

Where Co is initial concentration (mg L^{-1}) or the initial concentration of Cu in the soil that has been extracted by a solution of H_2SO_4 with various pH] before incubation and Ce is final concentration (mg L^{-1}) or the final concentration of Cu in the soil after incubation and SBC char treatment.

6) Cu immobilization

Heavy metal mobility is the tendency of heavy metals to move from one place to another, which can be affected by such factors as the type of heavy metal, the pH of the soil, and the presence of organic matter [16]. The mobility of heavy metals is an important factor to take into account when assessing the environmental impacts of heavy metal contamination.

The percentage of immobilized metal is a measure of the amount of metal bound to another substance, such as a mineral or organic matter. Based on Kim et al. [33], the mobility of Cu can be calculated by the Eq. 2.

7) Sorption kinetic model

The sorption kinetic models used in this study was pseudo first order. Linear regression was used to estimate the parameters from kinetic models [2, 34]. The pseudo first order equation can be seen in Eq. 3.

$$\text{Immobilized metal (\%)} = \frac{[Cu\ for\ control - Cu\ for\ treated\ sample]}{Cu\ for\ control} \text{ x } 100\% \qquad \text{(Eq. 2)}$$

Where Cu for control is the mobility of Cu in the soil after incubation and without SBC char addition (mg kg^{-1}) and Cu for treated sample is the mobility of Cu in the soil after incubation and with treatment 10% SBC char addition (mg kg^{-1}).

$$\frac{dq_t}{dt} = k_1\ [q_e - q_t] \qquad \text{(Eq. 3)}$$

Equation. 3 can be linearized and the linearized version can be seen in Eq. 4.

$$\ln [q_e - q_t] = \ln q_e - k_1 t \qquad \text{(Eq. 4)}$$

where q_e is the sorption capasities (mg kg^{-1}) at equilibrium, q_t is the sorption capasities (mg kg^{-1}) at time, and k_1 the rate constant (1/week).

Results and disussions

1) Soil characteristics

The average pH value of the soil from 5 different spots in PCM is 4.7. The texture of the soil is dusty clay. From AAS preliminary analysis, Cu content in soil sample was 94.79 mg kg^{-1}. It indicated that Cu in sample exceed the threshold from Indonesian Government Standard, where the threshold value for Cu in soil is 40 mg kg^{-1} [35–36].

2) Char characteristics

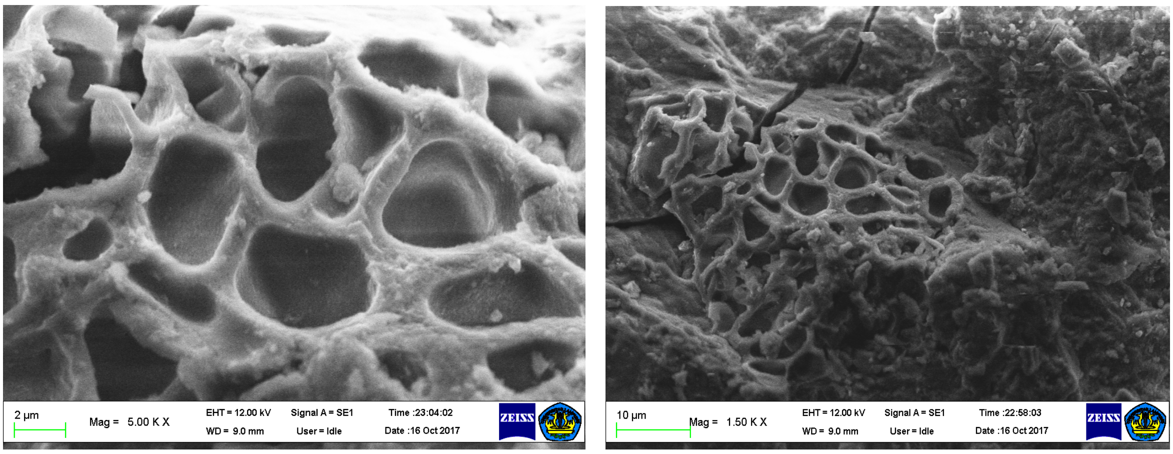
The char used in this result is pyrolyzed SBC at 400°C for 1 h. The acidity of the char was 8.8. The application of the char in contaminated soil was intended to eliminate the activity of heavy metal ions, thus they preventing them entering the food chain system and making them harmless as they would be adsorbed by the char. This phenomenon is closely related to the quality of the produced char. Char production through pyrolysis at high temperatures (>550°C) generally will form high cross-sectional area and is good for physical adsorption [37–38]. However, the char had the char produced in this way has only a few functional groups and nutrients. On the other hand, low temperature pyrolisis (400–500°C) generates char with various functional groups and nutrients [39–40].

The SEM test was carried out to see the surface of the char. The results of the test at magnifications of 2 µm (5,000x) and 10 µm (1,500x) can be seen below.

Scanning Electron Microscope (SEM) FEI Quanta 200, with resolution of up to 1.8 nm, was used to characterize the surface morphology and pore structure of SBC char. SEM images were taken at different magnifications, including 5,000x, to observe the details of the char grains and pores. Figure 2 shows an SEM image of SBC char at 5,000x magnification. The image reveals a high density of open pores. The grains were neatly arranged in a homogeneous morphological form, with a stable framework of approximately 10 µm in area size.

The pores found in char are generally in the form of micropores and mesoporous pores. The availability of pores in char allows the sorption of Cu [41]. According to Al-wabel et al. [42], metal absorption that occurs in micropores and mesoporous char plays a dominant role in the process of soil remediation. The high density of open pores in SBC char indicates that it has a high surface area and pore volume, which can enhance its adsorption capacity for copper. Therefore, the char morphology is an important factor that affects the immobilization of copper in post coal mine soil using SBC char.

The FTIR test was carried out to determine the functional group of organic component [43]. The FTIR analysis results of SBC char showed that the wavenumbers 2,924, 1,600, and 1,100 cm^{-1} were all significantly changed. The decrease in the intensity of the peak at 2,924 cm^{-1} indicates that the amount of C-H stretching vibrations decreased after pyrolysis. This is likely due to the breakdown of aliphatic structures in lignite, which are the main components of lignite that contain C-H bonds. The increase in the intensity of the peak at 1,600 cm^{-1} indicates that the amount of C=O stretching vibrations increased after pyrolysis. This is likely due to the formation of new functional groups, such as carboxylic acids and carbonyl groups, during pyrolysis. The decrease in the intensity of the peak at 1,100 cm^{-1} indicates that the amount of O-H stretching vibrations decreased after pyrolysis. This is likely due to the loss of water from the biochar during pyrolysis.



(a) (b)

Figure 2 The surface morphology of the char from SEM analysis results with magnifications of (a) 5,000 times and (b) 1,500 times.

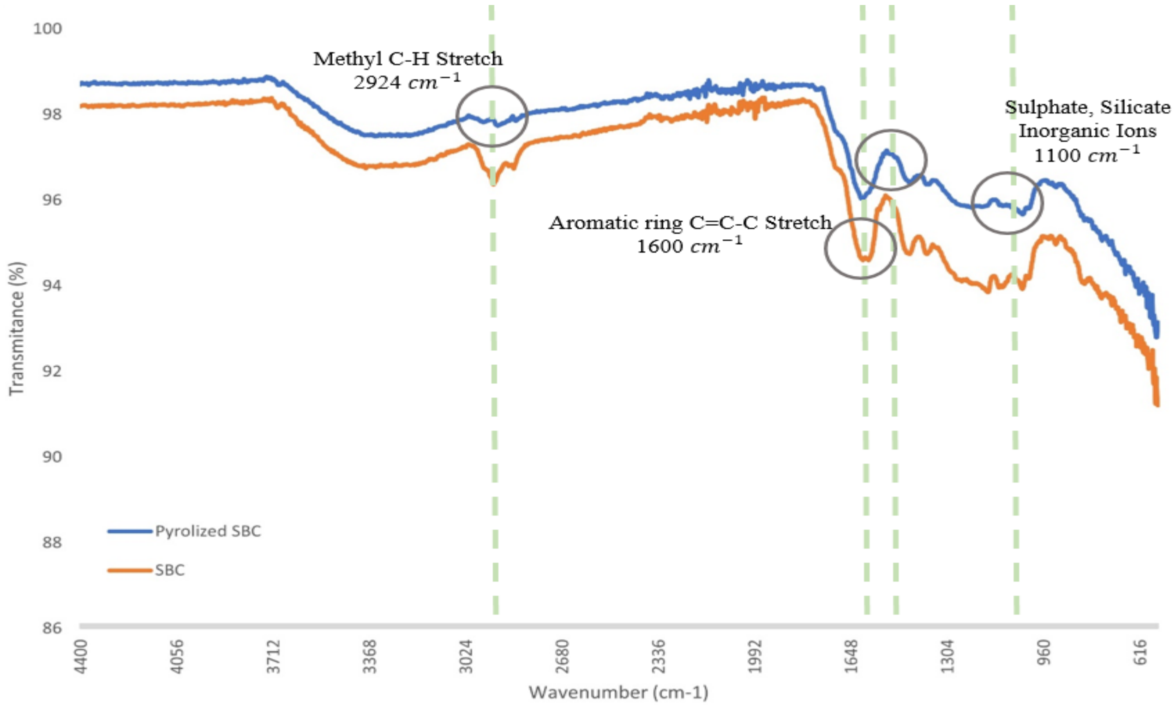


Figure 3 FTIR result for SBC before and after pyrolysis.

3) Soil immobilization

3.1) Effect of pH for Cu mobility

The degree of pH is a major factor on the adsorption process of metal ions in solution, because the presence of H^+ ions in solution will compete with cations to bind to the active site. The pH of the solution is a vital parameter in optimizing the adsorption process. The effect of the pH on the adsorption process depends on the type of char and target contaminants. This affects not only the surface charge of the char, but also the degree of ionization and speciation of the adsorbate [44]. Most studies concerning the adsorption of contaminants to char take into account the effect of the pH.

In this study, the pH values of the extraction solution were varied to determine the mobility of Cu based on the effect of pH. The AAS results the Cu mobility based on variations of the pH are shown in Table 1.

Table 1 Effect of pH for Cu mobility

pH	Cu mobility [mg/kg]		% absorbed Cu
	0% char [control]	10% char	
1	3.37	3.24	3.86
2	0.87	0.83	4.60
3	0.20	0.18	10.00
4	0.08	0.07	12.50
5	0.07	0.03	66.67
6	0.03	0.03	0.00

Table 1 shows, at the pH of 1, the treatment without any char addition, the mobility of copper metal ions was 3.37 mg kg^{-1}, while with the addition of 10% of char, the mobility of copper metal ions increased to 3.24 mg kg^{-1}. It happened because, at a low pH, the surface of the char was positively charged, thus it was capable of absorbing anions [46–48]. In addition, the presence of a number of H^+ and H_3O^+ in solution competed with Cu to interact with the functional groups in the char [49]. Thus, electrostatic repulsion occurred between the cationic contaminants and the positively charged char surface and resulting weak adsorption ability by char at low pH [48, 50–51].

Biochar produced through pyrolysis of biomass for copper adsorption was employed by Chen et al. [28]. In line with Chen et al. and Doskocil et al. [28, 52] who used natural lignite in the removal of heavy metals and found that the adsorption capacity increased with increasing pH and peaked at pH 5. It tended to decrease at pH above 5 due to the formation of hydroxide complexes [53–55].

The results of another study of the adsorption of Cu on three different biochars showed that the adsorption of Cu increased with increasing pH suspension from 3.5 to 6.0 [56]. Based on the description above, it can be seen that the optimum adsorption of Cu occurred at pH 5 with an adsorption percentage of 66.67%.

3.2) The effect of incubation time on the Cu mobility

In this study, various incubation times were carried out. The incubation aimed to prolong the contact time between char and soil. The AAS results of Cu mobility based on the effect of incubation time can be seen in Figure 4.

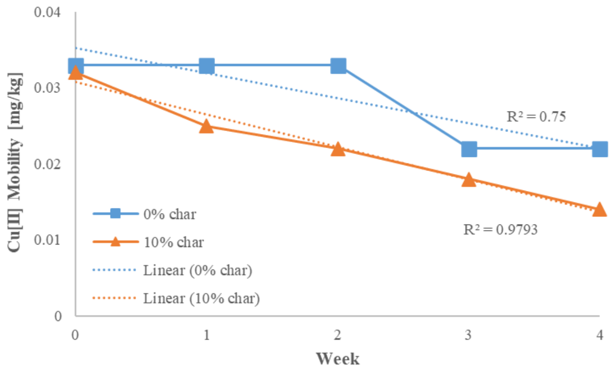


Figure 4 The effects of incubation time on the Cu mobilization.

Figure 4 implies the longer the incubation time, the lower the Cu mobility. The results are in line with the results of the test with the variations of the pH value, which indicate that the Cu mobility with 0% of char was higher than the Cu mobility with the addition of 10% of char.

The mobility of Cu at 0% char (control) in the first and second weeks was constant and slightly decreased in the third and fourth weeks. While the addition of 10% char, in the first week caused the mobility of metal ions to increase and it began to decrease in the second to the fourth week. The longer the incubation period, the more stable the metal ions of the soil. It took place as, over the incubation period, the char decomposed in the soil, which caused the soil to be highly reductive so that the redox potential decreased. Char of acid sulfate soil serves to maintain soil redox, so pyrite oxidation can be suppressed [57–59].

During the incubation period the degree of pH tends to be constant with a slight increase which is still within the normal range [16, 60]. So it is safe to infer that, during the incubation period, char decomposition occurred and it made the soil reductive and there was also an increase in the soil pH so that the Cu mobility was lower. In reference to Figure 4, the Cu mobility percentage in the fourth week decreased by 36.36%. With the addition of 10% of char, the lowest Cu mobility, with a value of 0.0147 mg kg^{-1}, was found in week 4. This is in line with the findings of Hoslett et al. [18] and Ma and Dong [61] that Cu mobility decreases as the length of incubation increases.

3.4) Sorption kinetics

Table 2 Parameters of pseudo first order kinetic models for 0% and 10% char additions

Samples	Pseudo-first order		
	R^2	q_e [mg kg^{-1}]	k_1 [mg kg^{-1}. week]
0% char	0.750	0.033	0.2746
10% char	0.979	0.032	0.1380

The Cu mobility decreased for 4-week incubation period. The equilibrium was happened in the third week for 0% char and fourth week for 10% char addition. R^2 value in pseudo-first order are 0.750 and 0.979, for 0% and 10% char addition respectively.

The potential of lignite to be pyrolyzed into char to immobilize heavy metals is significant and promising. Lignite is a relatively abundant resource, and it is relatively inexpensive to pyrolyze. The char produced from lignite is also relatively low-cost. This makes it a potential low-cost adsorbent for heavy metals [52, 62].

The pyrolysis temperature and the baking time of char are two important factors that affect char characteristic and properties [63]. The pyrolysis temperature is the temperature at which char is produced, and it can affect the properties of char, such as its surface area and its ability to adsorb Cu [18]. Meanwhile, the baking time is the duration of the transformation process [64].

Different baking times can result in different changes in char characteristics and properties [64–65]. For instance, longer baking times can increase the surface area and pore volume of char, which can improve its adsorption capacity for Cu. However, longer baking times can also decrease the organic matter content and functional groups of char, which can lower its adsorption affinity for copper. Therefore, there is an optimal baking time that can produce char with the most suitable characteristics and properties for copper immobilization.

Soil acidity need to consider in future research on the immobilization of Cu by char. The acidity of the soil can affect the ability of char to adsorb Cu, and the optimal acidity level for immobilization may vary depending on the type of char and the Cu concentration in the soil. Therefore, it is important to investigate the effect of soil acidity on Cu immobilization in future studies [16, 49, 64–65].

Short-term analysis can also be used to observe changes in Cu metal over a shorter time span. This would allow researchers to track the immobilization of Cu by char in real time and to identify any factors that affect the immobilization process [5–6]. Short-term analysis could be used to determine the effect of pH on Cu immobilization or the effect of the presence of other heavy metals in the soil.

Incubation time is another important factor to consider in future research. The incubation time is the amount of time that char is in contact with Cu metal [1]. In general, the longer the incubation time, the more Cu will be immobilized by char [2, 16, 68]. However, it is possible that char may reach a point where it is unable to immobilize any more Cu. Therefore, it is important to investigate the effect of incubation time on Cu immobilization in future studies.

https://doi.org/10.35762/AER.2023023

Conclusions

The addition of char from the lignite coal called Sumatra brown coal is able to restrain the mobility of Cu in soil along with the increase in the pH of a solution. The addition of 10% of char decreases the mobility of the Cu by 66.67% at a pH of 5. The longer the incubation time, the lower the mobility of Cu in post-coal mining soil. The addition of 10% of char in the week-4 decreases the mobility of the Cu by 36.36%.

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