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Lead immobilisation in mining contaminated soil using biochar and ash from sugarcane

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Abstract: Immobilisation of lead (Pb) and toxic elements in contaminated soils is of importance due to their persistence in the environment. Herein, we investigated the effects of sugarcane filter cake biochar (SFCB) and sugarcane bagasse ash (SBA) on the extractability of Pb and some toxic and potentially toxic elements (As, Cd, Cu, and Zn) in polluted mine soil samples from Lower Klity Creek, Thailand. The soil was equilibrated with the SFCB and SBA at the respective rates of 0, 1, and 5% (*w*/*w*) for 120 days at field capacity. The results revealed that both SFCB and SBA materials significantly ($P < 0.05$) decreased Pb extractability in the studied soil, and it stabilised after 56 days of incubation. At 120 days, the SFCB and SBA application at the rates of 5% SFCB, 5% SBA, 1% SFCB, and 1% SBA decreased the extractable Pb contents by 50.35, 40.81, 29.42, and 19.27%, respectively, compared to unamended soil. The SFCB and SBA materials also improved soil chemical properties by increasing the soil pH, available phosphorus, and extractable sulfur. At 5%, SFCB decreased As extractability and increased organic carbon in the studied soil. The Zn availability in the studied soil was also improved by SFCB and SBA addition. This study highlights the potential use of biochar and ash from the sugarcane industry to stabilise Pb and As in contaminated soils.

Keywords: *Saccharum officinarum* L.; heavy metal; pollution; soil amendment; trace element

Contaminations of toxic metals (such as Pb, Cd, Cr, and Hg) and metalloids (such as As and Sb) in soils constitute global environmental and human health concerns such as their high resistance to being degraded and high toxicity to biota in ecological and environmental systems. Many toxic elements that polluted soils and environments associated with mining activities have been documented globally, including in Thailand. There have been many severe cases, including Cd and Zn contamination induced by Zn mining in Mae Sot district, Tak province, western Thailand (Simmons et al. 2005, Khaokaew et al. 2011), As contamination caused by gold mining in Dan Chang district, Suphan Buri province on the central plain (Tiankao and Chotpantarat 2018), and Pb contamination from Pb mining in Thong Pha Phum district, Kanchanaburi province, western Thailand (Rotkittikhun et al. 2006, Nobuntou et al. 2010, Poopa et al. 2015). The Pb contamination in western Thailand is the most concerning as it has caused adverse impacts to people residing in the Lower Klity village for over the last 20 years, following pollution from the spillover of the Pb mine tailings into the creek and river sediments (Phenrat et al. 2016). The Pollution Control Department remediated the Pb-contaminated sediment by dredging and allowing natural recovery, which made the contaminants less toxic (Phenrat et al. 2016). Nonetheless, the Pb contamination in surrounding agricultural soils has been affected and poses a vital threat to people involved in plant production and consumption in the polluted area.

Biochar, a stable carbon-rich material, is well recognised as an amendment for improving soil physicochemical and biological properties. It is beneficial for stabilising toxic elements in soils by chemical and physical interactions and precipitation. Biochar also facilitates changes to the soil environment that fa-

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vours the immobilisation of toxic elements. However, biochar properties depend mainly on biomass and pyrolysis conditions (Břendová et al. 2015, Burrell et al. 2016, Speratti et al. 2017, Bandara et al. 2019, Campos and Rosa 2020).

Sugarcane is an important economic crop in many sugarcane-producing countries worldwide, with filter cake and bagasse being the main agro-industrial residues. The sugarcane filter cake is the primary residue from juice filtration in the sugarcane mill (George et al. 2010); it is rich in plant nutrients and is useful as a fertiliser and soil amendment (Prado et al. 2013, Mota et al. 2019). Sugarcane bagasse ash, the end by-product of burning material to generate steam power in the sugarcane mill, is highly suitable for soil amendment as it contains high levels of plant minerals with a liming effect (Webber et al. 2017), but it is still little used in agriculture. Both sugarcane filter cake and sugarcane bagasse ash are easily accessible in Kanchanaburi province and other sugarcane-producing areas. Consequently, the objectives of this study were to determine the effects of sugarcane filter cake biochar (SFCB) and sugarcane bagasse ash (SBA) on Pb, As, Cd, Cu, and Zn extractability from a contaminated mine soil from Lower Klity Creek, Kanchanaburi province, Thailand and to examine their roles on soil chemical properties.

MATERIAL AND METHODS

Soil sampling and analysis. Lead-contaminated mine soil (Haplic Luvisols) was collected from mixed orchards (such as banana, papaya, and jackfruit) in Lower Klity creek, Chalae subdistrict, Thong Pha Phum district, Kanchanaburi province, Thailand (14°52'30.4''N, 98°56'56.9''E). The studied soil represents agricultural soil in this area (in the foothills or hills) that has been affected by Pb leakage from mine tailings into the water body used for both agriculture and human consumption. (Rotkittikhun et al. 2006, Poopa et al. 2015, Phenrat et al. 2016). A composite soil sample was taken from surface soils at a soil depth of 0–25 cm, air-dried, gently disaggregated, and separately sieved through a 2 mm and a 0.5 mm stainless steel sieve. The general soil properties were analysed in the laboratory with three replicates for soil texture (pipette method), pH (1:1 H_2O), cation exchange capacity (CEC; ammonium acetate pH 7.0 method), organic carbon (OC; back titration method), available phosphorus (avail. P; Bray II method), and extractable sulfur (extra. S; Fox et al. 1964). Standard methods were used for the soil analyses as described in Soil Survey Staff (2014).

Preparation and analysis of sugarcane filter cake biochar and sugarcane bagasse ash. Sugarcane filter cake obtained from Rajburi Sugar Co. Ltd, Mueang Kanchanaburi district, Kanchanaburi province, Thailand, was prepared as sugarcane filter cake biochar. Briefly, the sugarcane filter cake (moisture ~75% *w*/*w*) was tightly pressed into a crucible, firmly closed using a lid, and pyrolysed in a muffle furnace, Carbolite (RWF 1200). The temperature was increased at 10 °C/min to reach a peak temperature of 600 °C. After 2 h of slow pyrolysis, the SFCB was produced and allowed to cool down to room temperature overnight, and then was gently crushed through a 0.25 mm stainless steel sieve for sample homogeneity. Sugarcane bagasse ash from the same factory (Rajburi Sugar Co. Ltd) was air-dried (moisture 2.17% *w*/*w*) and gently crushed through a 0.25 mm stainless steel sieve. The SFCB and SBA were generally analysed for physicochemical properties using triplicate samples for specific surface area (SSA) using the BET-N_2 method (Micromeritics 3Flex Surface characterisation), pH (1:5 H_2O), total carbon (TC), and total nitrogen (TN) using a CHNS/O elemental analyser (2400 Series II, PerkinElmer, Buckinghamshire, UK), total phosphorus (TP) and total sulfur (TS) also analysed by an X-ray fluorescence spectrometer (BRUKER S8 TIGER, Bremen, Germany). The available phosphorus, cation exchange capacity, organic carbon, and extractable sulfur contents of the SFCB and SBA were determined based on the same methods used for soil analysis.

Lead and other elemental analysis of soil, SFCB, and SBA samples. The availability of Pb, As, Cd, Cu, and Zn in all samples were evaluated based on DTPA extraction solution, containing 0.005 mol/L DTPA, 0.01 mol/L $CaCl_2$, and 0.1 mol/L triethanolamine buffered at pH 7.3, with a sample-to-extractant ratio of 1:2 (*w*/*v*) (Lindsay and Norvell 1978). The suspension was shaken for 4 h, centrifuged at 4 000 rpm for 10 min, and filtered. The supernatant was analysed for Pb, As, Cd, Cu, and Zn concentrations using an inductively coupled plasma-optical emission spectrometer (ICP-OES; Optima 8300, PerkinElmer, Queenstown, Singapore).

The total Pb, As, Cd, Cu, and Zn concentrations in all samples were also determined using a wet digestion procedure with a mixture of HNO_3:$HClO_4$ (5:2). Briefly, 0.200–1.000 g of sample (0.5 mm sieve) was weighed into a digestion tube; then, 10 mL of the

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mixed acids were added, and the sample was openly digested at 150 °C until a clear solution was obtained. The concentrations of Pb, As, Cd, Cu, and Zn in the digest were also determined using the ICP-OES. For quality control, the stream sediment reference material (STSD-3) (Canadian Certified Reference Materials Project) and blanks were included in each measurement. The accuracy of the STSD-3 measurement was approximately 84.5, 65.6, 99.7, 100.2, and 85.3% for Pb, As, Cd, Cu, and Zn, respectively, relative to the recommended values (Lynch 1990). The available and total concentrations of the elements were determined in triplicate.

Incubation experiment. An incubation experiment was conducted to assess the effects of SFCB and SBA on Pb, As, Cd, Cu, and Zn availability in the contaminated mine soil sample at room temperature (25–28 °C). Each contaminated mine soil sample was weighed into a plastic vessel 16 cm in diameter and 12 cm in height and homogeneously mixed with SFCB or SBA at rates of 0, 1, and 5% (*w*/*w*). The incubation was carried out in triplicate. The soil moisture in each container containing 250 g of mixed soil and amended materials was brought to field capacity (FC; 21.06% *w*/*w*) with deionised (DI) water. The soil moisture at FC was measured using a pressure plate apparatus with a tension of 0.33 bar (Klute 1986). Each container was covered with a hole cap, and the DI water was added weekly to maintain field capacity moisture content. After incubation periods of 1, 14, 56, 90, and 120 days, the mixed soils were retrieved and Pb availability was assessed using the DTPA extraction. Furthermore, the element contents (As, Cd, Cu, and Zn) in the extract, pH, cation exchange capacity, organic carbon, available phosphorus, and extractable sulfur of the soil samples were analysed after the incubation had reached at 120 days.

Statistical analysis. Significantly different means of contaminated mine soil properties and extractable elements based on the DTPA extraction solution after incubation for 120 days were analysed using one-way analysis of variance, and the mean values were compared among treatments using post hoc multiple comparisons followed by Duncan's testing. Pearson's correlation was determined between the extractable Pb in the contaminated mine soil and soil properties after incubation with SFCB and SBA for 120 days.

RESULTS AND DISCUSSION

Properties and Pb, As, Cd, Cu, and Zn concentrations of contaminated soil. The studied soil was classified as sandy loam soil (55.5% sand, 37.6% silt, and 6.9% clay). The soil was moderately acidic (5.71), with a low cation exchange capacity (108.3 $mmol_+$/kg). The soil contained moderate organic carbon content (1.87%) and high available phosphorus (32.52 mg/kg). The extractable sulfur of soil was 13.15 mg/kg.

The contaminated mine soil contained a high Pb content of 250.8 mg/kg, which could have resulted from contamination from mine tailing leaks or from a natural Pb occurrence zone (Phenrat et al. 2016). The Pb content was lower than for the maximum for soil quality standards used for living and agriculture in Thailand at 400 mg/kg (National Environment Board 2004). The Pb content in the studied soil was lower than that reported in other publications in the same contaminated area of values of 325–142 000 mg/kg (Rotkittikhun et al. 2006). Nevertheless, the Pb content was higher than the average Pb content in Thai uncontaminated soil (from agricultural and uncultivated sites, n = 318) of 17.5 mg/kg (Zarcinas et al. 2004). This result suggested there could be a risk of Pb exposure in the environment through crop production. In addition, the studied soil had relatively high contents of Zn (40.57 mg/kg), As (25.99 mg/kg), and Cd (0.23 mg/kg), which were higher than the mean concentrations of uncontaminated Thai soils (23.9, 7.5, and 0.03 mg/kg, respectively) (Zarcinas et al. 2004). The soil total Cu content (4.98 mg/kg) was lower than the mean for uncontaminated Thai soils (14.1 mg/kg). The As content was higher than the

Table 1. Total and extractable concentrations (mg/kg) of Pb, As, Cd, Cu, and Zn of contaminated mine soil, sugarcane filter cake biochar (SFCB), and sugarcane bagasse ash (SBA)

Sample	Pb		As		Cd		Cu		Zn	
	total	extractable	total	extractable	total	extractable	total	extractable	total	extractable
Soil	250.8	124.6	25.99	0.71	0.23	0.06	4.98	0.50	40.57	1.30
SFCB	30.49	0.67	8.45	0.69	0.24	0.01	97.21	3.88	326.6	20.92
SBA	18.02	1.46	8.61	3.93	0.08	0.03	27.82	1.21	148.9	12.07

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soil quality standards used for living and agriculture in Thailand of 3.9 mg/kg. Likewise, the availability of Pb in the soil based on DTPA extraction had the highest value of 124.6 mg/kg. Only small contents of available Zn, As, Cu, and Cd were extracted, as shown in Table 1.

Properties and Pb, As, Cd, Cu, and Zn concentrations of SFCB and SBA. Based on the physicochemical properties, SFCB was slightly superior to SBA as a soil amendment. The SFCB material had higher values for specific surface area, cation exchange capacity, total carbon, total nitrogen, organic carbon, and total and available phosphorus than those in the SBA material, excepted for total and available S (Table 2). Comparison between the properties of the SFCB in this study and other published works showed that the current SFCB had a higher specific surface area but a lower cation exchange capacity, total carbon, and available phosphorus, while the pH and total nitrogen of the SFCB were in the same ranges as other studies, as shown in Table 2 (Eykelbosh et al. 2014, Islami et al. 2016, Speratti et al. 2017, 2018). The SBA in the present work had a higher pH but lower values for cation exchange capacity, total carbon, total nitrogen, and available phosphorus than those in Islami et al. (2016), as shown in Table 2.

The total concentrations of Pb, Cd, Cu, and Zn of the SFCB were higher than those of the SBA, except for the total As concentration (Table 1). The total Zn content in the SFCB and SBA materials was the highest for the metals in this study (326.6 and 148.9 mg/kg, respectively). These Zn-rich materials could provide surplus available Zn, an essential plant micronutrient, to the studied soil having a potentially deficient Zn concentration of 1.3 mg/kg (Sims and Johnson 1991) (Table 1). They contained minor total concentrations of Cu, Pb, As, and Cd in the SFCB (97.21, 30.49, 8.45, and 0.24 mg/kg, respectively) and the SBA (27.82, 18.02, 8.61, and 0.08 mg/kg, respectively). The SFCB and SBA also had small contents of extractable Pb, As, Cd, Cu, and Zn, where the extractable Zn was the highest for both SFCB (20.92 mg/kg) and SBA (12.07 mg/kg) among the studied elements (Table 1).

Changes in soil properties after incubation with SFCB and SBA. After incubating the soil with the SFCB and SBA for 120 days, the results showed that both SFCB and SBA significantly affected soil properties depending on the application rate ($P < 0.05$) (Table 3). The application of the SFCB at 5% significantly induced the highest pH (6.58), organic carbon

Table 2. Physicochemical properties of sugarcane filter cake biochar (SFCB) and sugarcane bagasse ash (SBA) in this and other studies

Material	SSA (m^2/g)	pH (1:5 H_2O)	CEC ($mmol_+/kg$)	TC	TN	OC	TP	Avail. P (mg/kg)	TS (%)	Extra. S (mg/kg)
				(%)						
SFCB	48.62	7.90	194.4	15.11	1.04	14.01	3.81	4 162	0.18	347
SBA	14.36	8.80	61.4	2.98	0.03	0.45	0.55	410	0.27	1 915
Eykelbosh et al. (2014)										
– Filtercake biochar	26.3	9.85 (1:9 H_2O)	na	36.7	1.3	na	na	na	na	na
Speratti et al. (2017, 2018)										
– Filter cake	13.5–41.3	8.5–8.8 (1:2.5 H_2O)	na	19.3–23.3	1.6–1.8	na	na	na	na	na
Islami et al. (2016)										
– Biochar from filter mud	na	7.65 (1% in H_2O)	246.5	29.88	0.08	na	na	20 600	na	na
– Boiler ash	na	7.44 (1% in H_2O)	176.4	26.73	0.08	na	na	19 600	na	na

na – not analysed; SSA – specific surface area; CEC – cation exchange capacity; TC – total carbon; TN – total nitrogen; OC – organic carbon; TP – total phosphorus; avail. P – available phosphorus; TS – total sulfate; extra. S – extractable sulfate

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Table 3. Contaminated mine soil properties after incubation with sugarcane filter cake biochar (SFCB) and sugarcane bagasse ash (SBA) for 120 days

Treatment	pH (1:1 H_2O)	Cation exchange capacity ($mmol_+$/kg)	Organic carbon (%)	Available phosphorus (mg/kg)	Extractable sulfate (mg/kg)
Unamended soil	5.25 ± 0.01^{d}	109.9 ± 11.1^{a}	1.78 ± 0.15^{b}	47.9 ± 2.74^{d}	8.77 ± 2.17^{c}
1% SFCB	5.84 ± 0.11^{b}	103.3 ± 8.4^{ab}	1.78 ± 0.02^{b}	231 ± 30.08^{b}	6.62 ± 0.31^{c}
5% SFCB	6.58 ± 0.04^{a}	112.6 ± 8.5^{a}	2.25 ± 0.20^{a}	1 087 ± 92.71^{a}	35.5 ± 4.08^{b}
1% SBA	5.60 ± 0.04^{c}	94.2 ± 12.1^{ab}	1.55 ± 0.02^{b}	46.2 ± 4.33^{d}	8.41 ± 1.61^{c}
5% SBA	6.53 ± 0.03^{a}	88.6 ± 5.7^{b}	1.56 ± 0.14^{b}	148 ± 8.89^{c}	95.0 ± 3.21^{a}

Means ± standard deviation followed by the same letters are not significantly different at $P < 0.05$

(2.25%), available phosphorus (1 087 mg/kg), and cation exchange capacity (112.6 $mmol_+$/kg) of soil, while the application of the SBA at 5% contributed to the highest soil pH (6.53) and extractable sulfur (95.0 mg/kg). The application of the SFCB and SBA could improve soil chemical properties, as was indicated in many other studies (Jien and Wang 2013, Yang et al. 2016, Webber et al. 2017)

Changes in extractability of Pb and toxic elements in contaminated soil after incubation with SFCB and SBA. The results from incubating soil with SFCB and SBA for 1, 14, 56, 90, and 120 days showed that both SFCB and SBA significantly reduced extractable Pb in soils with time (Figure 1). Furthermore, the extractable Pb of soil was significantly reduced and remained constant after incubation for more than 56 days. Soil incubated with 5% SFCB was the most effective for Pb immobilisation, with a reduction to 50.35% of extractable Pb compared to the unamended soil. The 5% SBA, 1% SFCB, and 1% SBA treatments decreased 40.81, 29.42, and 19.27% of the extractable Pb as compared to the effective soil, respectively; however, a dilution effect may partly affect the reduction of Pb extractability from amended soils compared to unamended soils. Biochar could immobilise the Pb through surface adsorption and complexation processes. The dissolved Pb^{II} in the soils could form complexations with biochar surface functional groups (Jiang et al. 2012, Park et al. 2013, Puga et al. 2015, Yang et al. 2016, Taghlidabad and

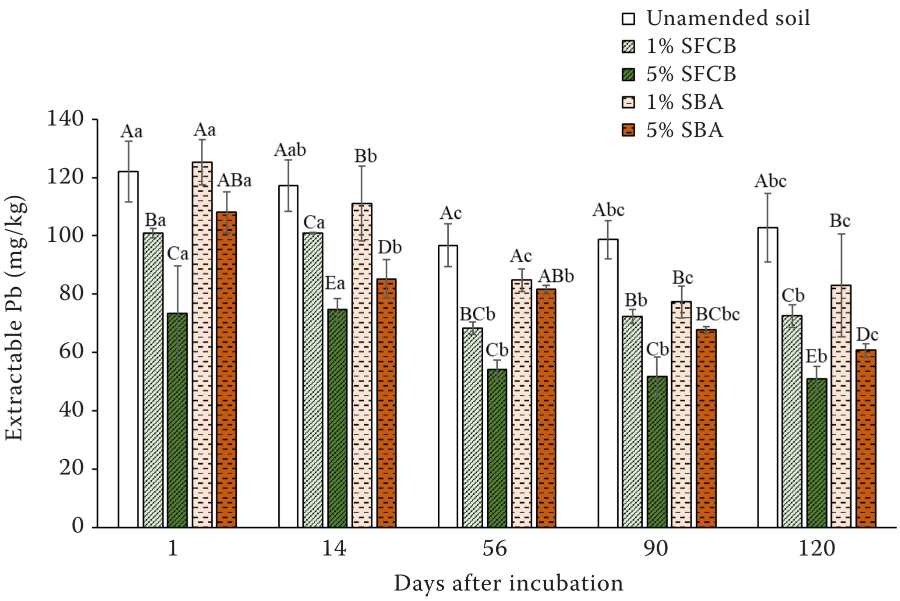

Figure 1. Extractable lead (Pb) in contaminated mine soil after incubation with sugarcane filter cake biochar (SFCB) and sugarcane bagasse ash (SBA) for 1, 14, 56, 90, and 120 days. The bars on the columns indicate ± standard deviation. Uppercase letters denote significant differences ($P < 0.05$) between treatments for one day of incubation. Lower case letters denote significance differences ($P < 0.05$) between days after incubation for one treatment

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Table 4. Pearson's correlation between extractable lead (Pb) in contaminated mine soil and soil properties after incubation with sugarcane filter cake biochar (SFCB) and sugarcane bagasse ash (SBA) for 120 days (n = 15)

Extractable element	pH	Cation exchange capacity	Organic carbon	Available phosphorus	Extractable sulfate
Pb	–0.932**	0.056	–0.389	–0.703**	–0.569*
As	–0.751**	–0.094	–0.483	–0.661**	–0.475
Cd	–0.538*	–0.209	–0.453	–0.648**	–0.155
Cu	0.161	0.358	0.603*	0.450	–0.064
Zn	0.900*	0.124	0.597*	0.845**	0.439

*P < 0.05; **P < 0.01 (2-tailed)

Sepehr 2018). Furthermore, the incorporation of SFCB and SBA induced higher values for soil pH, organic carbon, available phosphorus, and extractable sulfur (Table 3), resulting in Pb in the soil being in unavailable forms as these soil properties are the main factors controlling heavy metal behavior in soil (Park et al. 2013, Yang et al. 2016). This was supported by Table 4 that shows a highly significant negative correlation between the extractable Pb and soil pH (–0.932**), available phosphorus (–0.703**), and a significant negative correlation between extractable sulfur (–0.569*) after incubation for 120 days. Higher soil pH (higher concentration of hydroxide ions) could promote Pb precipitation and complexation with negatively charged hydro(oxides). The increased pH may induce to deprotonation of pH-dependent cation exchange sites on soil surfaces, then Pb sorption may increase (Aldriano 2001, Kumarathilaka

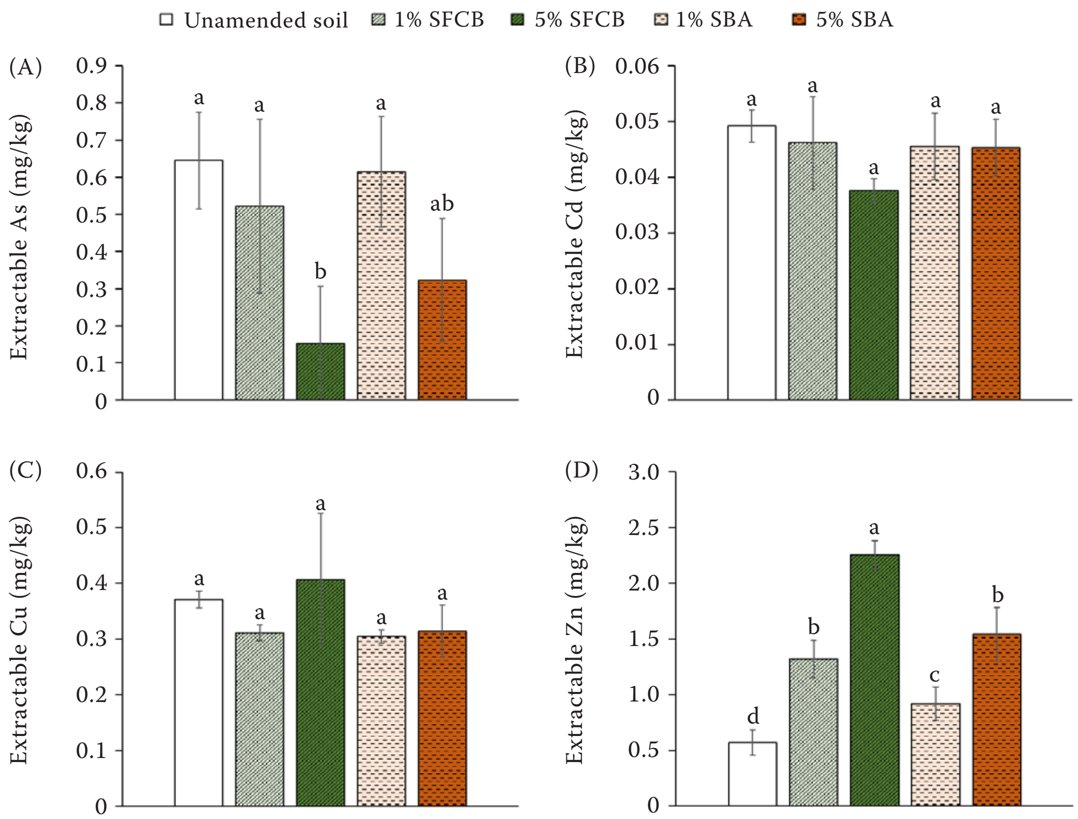

Figure 2. Extractable (A) As; (B) Cd; (C) Cu, and (D) Zn in contaminated mine soil after incubation with sugarcane filter cake biochar (SFCB) and sugarcane bagasse ash (SBA) for 120 days. The bars on the columns indicate ± standard deviation. Lower case letters denote significant differences (P < 0.05) between treatments

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et al. 2018). Furthermore, Pb in soils could be immobilised through the addition of phosphate- and sulfate-rich materials to form a stable Pb phosphate (chloropyromorphite; ($Pb_5(PO_4)_3Cl$)) and Pb sulfate ($PbSO_4$) (Landrot and Khaokaew 2020).

Furthermore, 5% SFCB also affected the extractable As in soil, as it could significantly reduce extractable As up to 76.37% compared to unamended soil (Figure 2A). On the other hand, both the SFCB and SBA significantly increased extractable Zn in the soil (Figure 2D); these results were due to the Zn being highly content in the SFCB and SBA as mentioned before (Table 1). However, the effects of SFCB and SBA were unclear for Cd and Cu availability in the studied soil (Figures 2B, C), which may have been due to the contents of these elements being at concentrations too low to observe effects. However, the extractable Cd in the soil had a significant negative correlation with soil pH (–0.538*) and a highly significant negative correlation with available phosphorus (–0.648**), possibly due to precipitation and complexation as similar to Pb. On the other hand, the extractable Cu had a significant positive correlation with soil organic carbon (0.603*), which is related to Cu contained in the amendment materials as well as Zn (Tables 1 and 4).

Our results suggested that the application of SFCB in the contaminated mine soil should be at the rate of > 5% (*w*/*w*) and pre-equilibrated at least 2 months to effectively decrease the solubility and mobility of toxic elements in the environment. The SBA also was useful for reducing the availability of Pb in soils. Although the SBA was not as good as the SFCB, it is an end by-product of the sugarcane industry and is readily and cheaply available. However, the SFCB had the advantage over the SBA of providing additional plant micronutrients such as Zn and Cu. The selection guidelines for the use of SFCB or SBA should be based on the purpose and cost of production.

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