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Content of Heavy Metals in Various Biochar and Assessment Environmental Risk

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ABSTRACT

Biochar is a product of biomass pyrolysis and has a number of environmentally beneficial uses, but it can also pose risks if not managed properly. These risks are mainly due to the chemical structure of biochar, the content of heavy metals. The aim of this study was to evaluate the mobility and environmental risk of heavy metals in biochar produced from plant biomass (BB), municipal solid waste (MSW), compost (C) and coal refuse (CR). Pollution indices were calculated: geo-accumulation index (GAI), ecological risk (E_r^i), the underlying ecological risk caused by the total pollution (PERI). The total heavy metal concentration is variable and depends on the type of biochar. The results indicate that there is a high risk of cadmium pollution in the environment. The underlying ecological risk caused by the total pollution values indicated that biochar from coal waste was the highest. The results obtained show the importance of mobility analysis in assessing the potential for natural use of biochar.

Keywords: biochar, heavy metals, environmental risk.

INTRODUCTION

Today, there is growing interest in using organic waste to improve degraded soils. Such wastes include biochar. A definition developed by the International Biochar Initiative: "Biochar is a fine-grained carbonaceous material with a high organic carbon content and negligible degradability, produced by the pyrolysis of biomass and biodegradable wastes" [IBI Biochar Standards]. Biochar is not a new material. It was used as a soil amendment in the Amazon basin more than 2,500 years ago. Today, there is growing interest in the use of biochar in many sectors of the economy. The Swiss Ithaka Institute has described 55 possible ways to manage biochar [Gabhane et al, 2020]. Biochar does not have homogeneous physical and chemical properties, they vary depending on the type, the course of the production process and the type of biomass from which it is produced [Malińska, 2012]. Biochar has a high porosity and the specific surface area is generally in the range of 1.5–500 $m^2 \cdot g^{-1}$. These two properties are responsible for the effects of biochar on water uptake, determining its sorption capacity and also nutrient retention [Tomczyk et al., 2020]. The literature review reports that biochar has a high capacity to adsorb herbicides and pesticides, causing their deactivation or accumulation [Cara et al. 2022].

Biochar is primarily composed of carbon, which makes up the majority of its structure. It contains a complex arrangement of carbon atoms, often in the form of aromatic rings and carbon chains [Leng et al. 2019]. The carbon content gives biochar its black color and helps it retain carbon in the soil, making it an effective carbon sequestration method [Sun et al. 2018]. The pH of biochars can vary depending on various factors, including the feedstock used to produce the biochar and the production conditions. Generally, biochars have a near-neutral pH, ranging from slightly acidic to slightly alkaline [Malińska, 2012]. Biochars contain various functional groups that contribute to their chemical properties. Some common functional groups found in biochars include: hydroxyl, carboxyl, phenolic, ketone and aldehyde, ester, amine. These functional groups play a significant role in the chemical reactivity, adsorption capacity, and overall behavior of biochars in different environmental and agricultural applications [Herrero et al., 2018]. Biochar consists of minerals such as calcium, magnesium, hydrogen or nitrogen and small amounts of sulphur. The amount of minerals varies between 0.5% and 5%. Biochar is characterised by a high carbon to nitrogen ratio, which can range from 7 to 500. The C/N ratio is an important indicator of the decomposition capacity of organic matter in the soil. Biochar can also contain volatile compounds, the amount of which can reach up to 40% [Saletnik et al., 2019]. The chemical composition of biochar is important because it determines its management. The higher the amount of carbon with a low amount of minerals, the more biochar can be used for energy purposes. On the other hand, if the amount of minerals is high, the main use of biochar is as a fertiliser or adsorbent for absorbing heavy metals in soils as well as in wastewater [Malińska, 2012; Duwiejuah et al., 2020].

Biochar can contain toxic compounds such as heavy metals and polycyclic aromatic hydrocarbons. Concentrations of heavy metals are variable and depend on the conditions of biochar production [Vassilev et al., 2014]. Heavy metals in biochar are mainly derived from raw materials containing toxic metals, such as industrial solid waste, sewage sludge and residues from biogas production. Heavy metals accumulate in the ash fractions during pyrolysis. The levels of heavy metals in biochar should be tested, particularly if we are introducing biochar into the soil for use as a fertilizer. The introduction of biochar containing heavy metals into soils can have negative effects on the ecosystem. Freddo et al. (2012) studied the content of heavy metals in nine different types of biocarbon (rice straw, maize, bamboo, sequoia and conifer wood) produced at various pyrolysis temperatures from 300 to 600 °C. The results of the comparison of the obtained metal concentrations with the concentrations of these metals in European soils showed that Cd, Ni, Cr metal concentrations in biochar were higher than average concentrations of these metals in European soils. Unfortunately, these studies suggest that biocarbon can contaminate soils with heavy metals. They have also shown that the high concentrations of heavy metals in biochar, they obtained low concentrations in water extracts from biochar, suggesting a low risk of metals leaching from soils [Freddo et al., 2012]. Mendez i in. (2012) studied the effect of the pyrolysis process on the leachability and bioavailability of heavy metals in biocarbon from sewage sludge. They showed that the total concentrations of some metals in biochar from sewage sludge were higher than in sewage sludge, but there was a reduction in Cu, Ni, Pb and Zn concentrations and Cd mobility [Mendez et al., 2012]. Therefore, care should be taken when introducing biocarbon into soils to avoid toxicity problems. Excessive concentrations of heavy metals can cause toxicity to biota or humans, resulting in unacceptable levels of environmental risk [Adriano et al., 2001; Rakshit et al., 2021; Khorram et al., 2016].

It is therefore necessary to study and properly qualify the physical and chemical properties of biochar before choosing a biochar management method. It is also worthwhile to determine the risk of biochar introduction into the environment. The aim of this study is the assessment of heavy metal mobility from biochar and the risk of heavy metal accumulation in the soil.

MATERIALS AND METHODS

Materials

Biochar: BB (biochar form plant biomass), BK (biochar from municipal waste), BP (biochar form compost), BT (biochar from coal refuse) was collected from Fluid Company (now a company in bankruptcy). The characteristics of the biochar used are shown in the Table 1.

Total concentrations and leaching characteristic of of heavy metals in biochar

Leaching

Water extracts of biochar were prepared according to EN 12457-2:2006.

Digestion stage

Biochar samples were weighed into Teflon mineralizer dishes in an amount of approximately 0.1 g (1 ml in water extracts) and 6 ml of HNO_3 and 2 ml of H_2O_2 were added. Mineralization was carried out in a Topex Preekem microwave mineralizer according to the programme given in Table 2.

Table 1. Biochar properties of the biocarbon used in the study [Kujawska et al., 2022]

Biochar	BB	BK	BP	BT
Raw material from which biochar was produced	Plant biomass	Municipal waste	Compost	Coal refuse
Pyrolysis temperature at which biochar was produced	300°C	650°C	650°C	600°C
Total carbon [%]	61.54	55.36	45.50	53.58
Hydrogen [%]	5.16	0.18	4.22	4.57
Total nitrogen [%]	0.22	0.66	0.38	0.12
pH	4.62	10.26	9.96	7.37

Determination of heavy metals

Metals were determined using an Agilent 8900 ICP MS Triple Quad spectrophotometer. Quantitative determination of elements was performed using the external curve method. The following standards were used for the determinations:

- Multi-element calibration standard 2A – Hg Agilent Technologies
- Multi-element calibration standard 2A Agilent Technologies

Table 2. Sample digestion program

Digestagion stage	Temperature (℃)	Time (min)
1	120	3
2	150	3
3	170	3
4	190	20

Indicators of ecological risk

The heavy metal contamination of biochar was assessed using the indicators listed in Table 3. The interpretation of the results of the calculated indicator values is given in Table 4.

Statistical analysis

The analyses were carried out using STATISTICA 13 (StatSoft Poland), licensed from the Lublin University of Technology. To assess the significance of differences between means, statistical analyses were performed based on Tukey's multiple comparison test, with an assumed significance level of $\alpha = 0.05$. Means marked with the same letter indicate that they belong to a statistically homogeneous group, i.e. there is no statistically significant difference between them.

RESULTS

Heavy metals

The results of the metal content of the tested biochar are shown in Table 5. Post hoc statistical analysis using Tukey's test for significant differences showed that the heavy metal content of BT biochar is statistically significantly higher than the other biochars. In the other biochars tested,

Table 3. Ecological risk assessment indicators studied

Ecological risk assessment indicators	Formula	Designation
Geo-accumulation index GAI	$GAI = log_2 \frac{c_n}{1.5B_n}$	c_n is the measured content of a particular heavy metal in PM, SM, PMB, or SMB, and Bn is the background value for the heavy metal. The B_n values of Cd, Cr, Zn, Cu, Pb, Ni used in this study were 0.08, 64, 90, 25, 26, 30 mg·kg^{-1}, respectively [Liu, et al., 2016].
The pollution coefficient c_f^i	$c_f^i = \frac{c_D^i}{c_R^i}$	c_D^i is the content of heavy metal in BBB sample, c_R^i is the background content of heavy metals and defined as B_n [Xu et al., 2017].
Ecological risk E_r^i	$E_r^i = T_r^i \times c_f^i$	T_r^i is the toxicity response factor of heavy metals for Cd, Cr, Cu, Pb, Zn, Ni are 30, 2, 5, 5, 1, 5, respectively [Xu et al., 2017]
The underlying ecological risk caused by the entire pollution $PERI$	$PERI = \sum_{i=1}^{n} E_r^i$	[Xu et al., 2017]

Table 4. Interpretation of the used indicators

Contamination level	Value	
Geo-accumulation index	GAI [Hakanson, 1980]	
Unpolluted	<0	
Unpolluted to moderately polluted	0<GAI≤1	
Moderately polluted	1<GAI≤2	
Moderately to heavily polluted	3< GAI ≤4	
heavily polluted	4< GAI ≤5	
Extremely polluted	GAI >5	
Ecological Risk Index/PERI	Ecological Risk Index [Wan et al., 2019]	PERI [Huang et al., 2011]
Low risk	$E_r^i < 5$	PERI< 30
Moderate risk	$5 \leq E_r^i < 10$	30≤PERI < 60
Considerable risk	$10 \leq E_r^i < 20$	60 ≤PERI < 120
High risk	$300\leq E_r^i < 40$	-
Very high risk	$E_r^i \geq 40$	PERI >120

Table 5. Total concentrations of heavy metals

Biochar	Cr	Ni	Cu	Zn	Pb	Cd	As
	[mg·kg^{-1}]						
BB	23.80a±2.98	16.08a±0.33	11.39a±0.44	9.61a±0.24	35.99b±1.58	0.17a±0.01	1.53a±0.01
BP	5.88a±0.32	4.60a±0.81	3.96c±0.01	18.10c±1.62	1.7a±0.33	0.12a±0.01	0.11b±0.01
BT	69.42b±5.53	33.38b±2.22	40.27b±2.90	138b±7.74	33.87b±2.52	0.71b±0.01	0.61c±0.01
BK	28.78a±1.92	21.35a±2.71	3.45d±1.19	36.36d±0.52	1.71a±0.03	0.15a±0.01	0.12a±0.01
Norm	93–1200	7–420	143–6000	7400	121–300	14336	13–100
Average content of heavy metals in Polish soils [Kabata-Pendias & Kanbata, 1993]	40	7.4	6.3	40	18	0.2	6.2
Compost [Dz.U. 2008 nr 119 poz. 765]	100	60	-	-	140	5	-
Sewage sludge [Dz.U. 2015 poz. 257]	500	100	800	2500	500	10	-

the metal concentrations are not statistically significantly different, except for copper and arsenic. In the case of copper and arsenic, statistically significant differences in its concentration occurred between all the biochars.

The concentrations of chromium (5.88–69.42 mg·kg^{-1}), nickel (4.60–33.38 mg·kg^{-1}), copper (3.96–40.27 mg·kg^{-1}), zinc (9.61–138 mg·kg^{-1}), lead (1.71–35.99 mg·kg^{-1}), cadmium (0.12–0.17 mg·kg^{-1}) and arsenic (0.12–1.53 mg·kg^{-1}) in the biosolids tested did not exceed the limits set by the International Biochair Initiative, which are: for chromium 1200 mg/kg; for nickel 420 mg·kg^{-1}; for copper 6000 mg·kg^{-1}; for zinc 7400 mg·kg^{-1}; for lead 300 mg·kg^{-1}; for cadmium 39 mg·kg^{-1}and for arsenic 100 mg·kg^{-1} [International Biochair Initiative]. A comparison of the chemical composition of the tested biochar with the results of Freddo et al. (2012), who determined the concentration of metals in biochar produced from redwood, rice straw, maize and bamboo, showed that the content of heavy metals was lower. The mean concentrations of these elements in the Freddo study were cadmium 0.03 mg·kg^{-1}; chromium 4.34 mg·kg^{-1}; copper 5.48 mg·kg^{-1}; nickel 0.46 mg·kg^{-1}; lead 0.88 mg·kg^{-1}; zinc 55.63 mg·kg^{-1}; arsenic 0.21 mg·kg^{-1} [Freddo et al., 2012].

Table 5 compares the obtained heavy metal concentrations of biochar with the average values of metals typically found in the top layers of Polish soils. The levels of all the heavy metals studied, except arsenic, in BT biochar exceed the average levels of metals in Polish soils. In BB biochar, higher concentrations than the average content of these metals in Polish soils were found for Ni, Cu, Pb, while in BK biochar only nickel was found. The content of the tested metals in BT biochar was lower than the average content of metals in soils. The levels of heavy metals tested in biochar varied, suggesting that biochar may introduce heavy metals into soils. However, when the obtained metal concentrations in biochar are related to the metal concentrations in sewage sludge and compost, which are legally allowed to be introduced into soils in Poland, the tested biochar does not exceed the values specified for these wastes. The distribution patterns of heavy metals followed the decreasing trends of Pb>Cr>Ni>Cu>Zn>As>Cd for BB; and Zn>Cr>Ni>Cu>Pb>Cd>As for BP, Zn>Cr>Cu>Pb>Ni>Cd>As for BT and Zn>Cr>Ni>Cu>Pb>Cd>As for BK.

The metal content of biochar varies, as confirmed by other work on the metal content of biochar [Liu et al, 2014, Duwiejuah et al., 2020, Wang et al., 2021]. In particular, biochar produced from waste materials such as sewage sludge is characterised by high concentrations of heavy metals. This points to the need for detailed physico-chemical testing of both the feedstocks from which biochar is produced and the biochar itself.

The leaching test

One method for assessing the ecotoxicity of waste is the leaching test. The method used is that described in EN 12457-2:2006. This standard is deficient in that it does not specify limit values (Stiernstrom et al., 2011). Leaching results indicate water-soluble inorganic contaminants that are available to plants and easily transported through the soil. Table 6 shows the heavy metal concentrations and pH of the tested biochar in the aqueous extracts. The metal concentrations obtained from the aqueous extracts of biochar were compared with the limit values and landfill acceptance criteria laid down in the European Directive [Council Decision of 19 December 2002]. Similar to the results presented here, leaching of metals from biochar was obtained by Yargicoglu et al. (2015), who studied leaching from different biochar produced from wood waste. They obtained metal concentration values of: Cd, Cr, Ni and Zn were at the limit of quantification [Yargicoglu et al., 2015]. However, there are studies in the literature reporting on the possibility of heavy metal leaching from biochar. For example, Mancinelli et al. 2016 demonstrated the risk of excessive Ni leaching from biocarbon with digested sewage sludge and lignin [Mancinelli. et al., 2015]. Mellbo et al. (2008) explain the lack of leaching of Cu and Zn from high pH wood biochar as follows (8.76–12.4) [Mellbo et al., 2008]. The eluates tested also showed an alkaline reaction, with the exception of the bio-carbon (BB) produced from plant biomass.

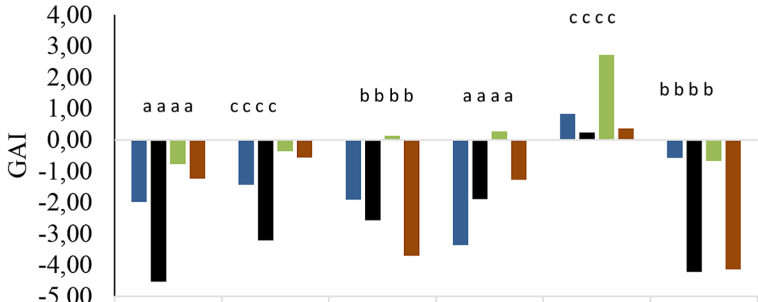
The research results presented show that care should be taken when producing biocarbon from waste contaminated with heavy metals. There is a need for continuous monitoring of biochar before and after its introduction into soils.

Ecological risk assesment

The following indicators have been calculated and analysed geo-accumulation index (GAI), ecological risk (E_r^i) and the underlying ecological risk (PERI). Geoaccumulation indices of selected elements in biochar are shown in Figure 1. Post hoc statistical analysis using Tukey's significant difference test showed that the GAI values for Cr and Zn were not statistically significantly different from each other, a similar relationship occurred for Ni and Cd and Cu and Pb. The GAI values for Cr, Zn, Cu, Pb, Ni in biochar BB, BP,

Table 6. Concentration of metals in aqueous extracts of biochar (1:10)

Biochar	Cr	Ni	Cu	Zn	As	Cd	Pb
	µg/l						
BK	1.74 ± 0.01	1.10 ± .001	1.64 ± 0.04	1.31 ± 0.10	1.78 ± .001	8.14 ± .001	3.59 ± 0.03
BT	3.43 ± 0.02	3.04 ± 0.01	1.69 ± 0.04	2.74 ± 0.06	6.05 ± 0.001	9.51 ± 0.001	5.15 ± 0.15
BB	1.20 ± 0.03	0.99 ± 0.01	0.66 ± 0.01	1.11 ± 0.03	0.93 ± 0.04	7.09 ± 0.001	0.64 ± 0.07
BP	1.97 ± 0.01	1.98 ± 0.001	1.07 ± 0.01	0.79 ± 0.09	3.09 ± 0.001	9.37 ± 0.001	2.52 ± 0.11
Norm according to Directive 1999/31/ EC(2003/33/), ppm	0.50	0.40	2.00	4.00	0.50	0.04	0.50



	Cr	Ni	Cu	Zn	Cd	Pb
BB	-1,98	-1,43	-1,91	-3,36	0,83	-0,57
BP	-4,53	-3,21	-2,57	-1,89	0,24	-4,22
BT	-0,77	-0,36	0,13	0,27	2,72	-0,67
BK	-1,24	-0,56	-3,70	-1,27	0,36	-4,14

Figure 1. Geoaccumulation index of heavy metals in biochar

BK were all below 0, which qualifies them as unpolluted, according to Hakanson. In the case of BT biochar, only the GAIs for Cr, Ni and Pb were below zero, while the GAIs for Cu and Zn were 0.13 and 0.27 respectively, which qualifies them as unpolluted to moderately polluted. However, the GAI for Cd reaches values above zero for all biochars, reaching 0.83 for BB, 0.34 for BP and 0.36 for BK, qualifying them as unpolluted to moderately polluted. The highest GAI value for Cd was obtained for BT biochar (2.72), indicating moderate contamination. The variability in the levels of heavy metal geoaccumulation indices in biochar indicates the need for research, especially before choosing a biochar management method. The potential ecological risk index value of heavy metals in biochars are shown in Figure 2 and 3. A post-hoc statistical analysis using Tukey's significant difference test showed that the E_r^i of Cu for biochar BB and BT are statistically significantly different from the other E_r^i values. E_r^i of Pb of the investigated biochars are statistically significantly different from each other. The E_r^i of Cd, Cr, Ni, Zn for BT biochar are statistically significantly different from the other tested biochars.

The risk indices of Cr, Cu, Ni and Zn for the biochars BB, BP and BK reached a value below 5, which qualifies them as low risk. The E_r^i of Pb in BB was 6.92, Ni was 5.56 and Cu was 8.05 in BT, indicating a moderate risk. The E_r^i values of Cd in all tested biochar reached values above 40, indicating a very high risk. Similarly, E_r^i values above 40 for cadmium were obtained by Wang et al. from biochar produced from pig and sheep manure [Wang, et al., 2020]. There are a number of papers indicating that there is a risk of cadmium transfer from biochar to the environment [Rahi et al., 2022, Azadi and Raiesi, 2021].

For example, Zhang et al. assessed the environmental risk of Cd in biochar produced from

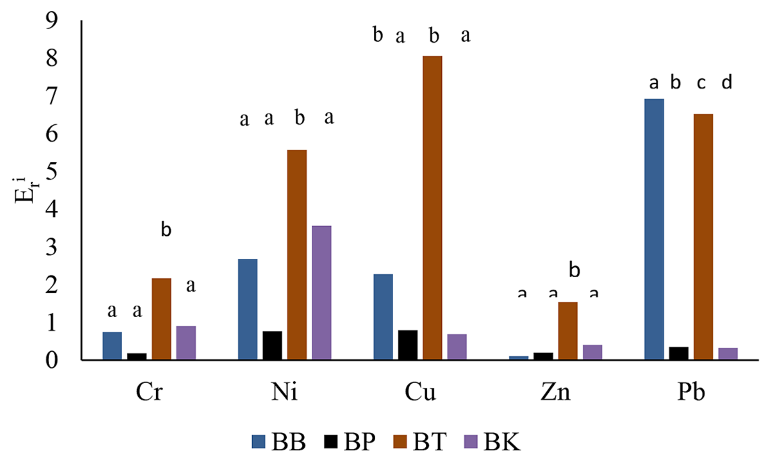


Figure 2. The potential ecological risk index value of heavy metals in biochars

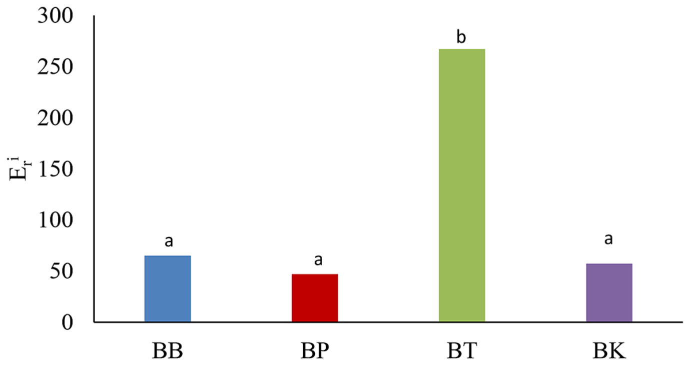


Figure 3. The potential ecological risk index value of Cd in biochars

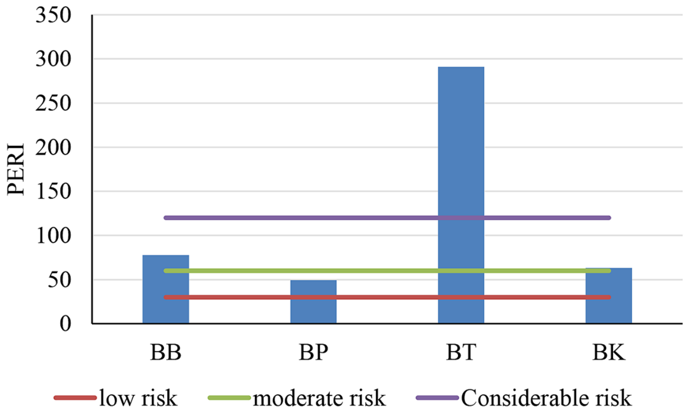


Figure 4. Potential ecological risk index of heavy metals in biochars

the plants *Brassica napus L. Pennisetum sinese* and *Lolium perenne L*. The potential environmental risk (RI) index values for Cd in plant biochars produced at temperatures between 400°C and 600°C showed a very high risk of hazard. When the pyrolysis temperature was higher than 700°C, the index value indicated a low risk. The results of their study showed that Cd contamination of biochar obtained by pyrolysis at temperatures higher than 650°C allows an environmentally acceptable [Zhang et al., 2020].

The values of the potential environmental risk indicators can be ranked as follows:Cd > Pb > Ni> Cu > Cr > Zn for BB; Cd > Cu > Ni > Pb > Zn > Cr for BP; Cd > Cu > Pb > Ni > Cr > Zn for BT and Cd > Ni > Cr > Cu > Zn> Pb > Zn. The sum of all risk factors was calculated to determine the ecological risk of heavy metals in biochar (Figure 4). The PERI index allows a summary assessment of their effects on organisms. Post-hoc statistical analysis using Tukey's significant difference test showed that the PERI values for BT biochar were statistically significantly different from the others. When analysing the results of the potential ecological risk index, it can be concluded that only the BP biochar produced from compost has a moderate ecological risk, while BB biochar and BK biochar show a considerable risk, while BT biochar already represents a high risk of ecological contamination. Of the metals tested, cadmium was found to be the most toxic in all biochar tested.

GAI and PERI values take into account the sum of bound metals that can be assimilated to the F2 fraction in sequential analyses of metal content [Wang et al., 2019]. The carbonate fraction of metals containing metals precipitated with carbonates, sulphates, phosphates; tends to be stable [Mizerna and Król, 2018].

CONCLUSIONS

The levels of heavy metals in the biochar tested vary. However, the metal concentrations in biochar do not exceed the range of acceptable metals for compost and sewage sludge that can be applied to soil. The biochar with the highest heavy metal content was found to be the biochar produced from coal waste. Leaching tests showed negligible leaching of heavy metals from the biochar tested. Analysis of the biochar for the geoaccumulation index showed that the highest risk of contamination was for cadmium. Calculated levels of potential environmental risk indicate that biochar produced from plant biomass and compost has a significant risk of heavy metal contamination. In contrast, biochar produced from coal refuse showed a very high risk of heavy metal contamination.

Due to the high values of potential environmental risks, the properties of biochar should be continuously monitored. The differences in the results of the studies carried out, the determination of the indicators discussed should be integrated to provide a comprehensive assessment of the quality of the environment and a risk analysis of the accumulation of heavy metals in biochar.

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