Soil Pollution by Heavy Metals Near the Lukavac Coke Factory and Models of Its Protection and Remediation

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Abstract. Soil represents a limited and invaluable natural resource that has multiple functions: production, ecological (biotope, transformation, regulatory), filtration-buffering, and provision of raw construction materials. However, industrial, technical-technological development and certain activities that humans carry out today causes numerous forms of soil degradation, including pollution and contamination of the soil with agrochemicals and heavy metals.

Bearing in mind the mentioned functions and importance of soil on the one hand and human activities that can lead to different forms of soil degradation on the other hand, the need to preserve land from degradation processes has taken on a global character.

The aim of this work was to determine the level of contamination of agricultural soils with heavy metals in the narrow zone of the coke industry in Lukavac, (44°49′23"N, 18°41′21"E). After that, in accordance with the obtained results, appropriate remedial measures are taken on the contaminated soils in order to reduce the degree of their contamination.

The results of the research showed that according to the classification of soil contamination with heavy metals, the soil in the vicinity of the Lukavac coke industry is classified as class III. It means that this soil is characterized by a high degree of contamination with heavy metals, but it can be used for growing agricultural crops, with implementation of certain protection measures.

For the purpose of chemical stabilization of heavy metals in this soil and their reduced accessibility for plants, natural aluminosilicate materials zeolite and pyrophyllite were used in this experiment. Research has shown that pyrophyllite had a significant impact on reducing the content of accessible forms of zinc (Zn), cobalt (Co) and manganese (Mn).

Keywords: soil · heavy metals · pollution · contamination · zeolite · pyrophyllite

1 Introduction

Any economic development, especially the one accompanied by the construction of industrial plants, inevitably leads to an increase in the standard of living, but at the same time it leads to an increase in pollution of the land with toxic substances. Through his

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reckless use of soil, the man has brought a large part of the land into a state of high pollution. From numerous pollutants that reach the soil due to human activity, heavy metals represent a significant problem due to their toxic effect on the environment, growth, development and quality of plants. As a result of these circumstances it is possible for humans to develop various diseases in case of exposure to harmful concentrations of heavy metals in the environment (Bates et al., 1995; Vratuša, 1999; Blake & Goulding, 2002; He Z.L. et al., 2005).

Taking into account the aforementioned facts, the preservation of fertile and remediation of agricultural land contaminated with heavy metals is simply imposed as a necessity if one wants to protect land as a primary resource for food production (Jakovljević & Antić-Mladenović, 2000; Malhotra et al., 2014). This need is particularly pronounced in the industrial areas of the Federation of Bosnia and Herzegovina (FBiH), where the possibility of soil contamination with heavy metals is extremely high (Pranjić, 2017). During 2019, the Faculty of Agriculture and Food implemented a project called "Models of protection and remediation of land contaminated with heavy metals in industrial areas of the Federation of Bosnia and Herzegovina", commissioned by the FBiH Environmental Protection Fund. This paper represents part of the results from the mentioned project which are related to the land in the vicinity of the Lukavac coke industry in the Tuzla Canton. The goal of these studies was to determine the degree of soil contamination with heavy metals in the soils in the immediate vicinity of the Lukavac coke industry. Then, on the land found to be the most polluted or contaminated with heavy metals, appropriate remedial measures were implemented with the intention of bringing the content of heavy metals to a level that does not pose a danger to the environment and human health. Through these researches, we tried to confirm the possibility and effectiveness of applying the chemical model of remediation through the use of natural aluminosilicates, zeolite and pyrophyllite. We also tested the effects of using the phytoremediation model through sowing and the cultivation of corn plants on polluted soils.

Previous research shows that the effectiveness of pyrophyllite in the immobilization of heavy metals in the soil depends on the properties of the soil, the pH reaction and the distribution coefficient of heavy metal ions in the soil. Although numerous studies confirm that pyrophyllite has a high efficiency in removing heavy metals from soil and water (Keren and Sparks, 1994; Saxena et al., 2001; Gücek et al., 2005; Kulović, 2017; Halilović, 2018; Panda et al., 2018), the binding mechanisms of heavy metals on pyrophile have not been scientifically fully explained. In the research conducted by Murtić et al. (2019) the use of pyrophyllite showed a significant effect in reducing the mobility of lead and cadmium in the soil. The aluminosilicate mineral zeolite shows similar properties and effects.

2 Material and Method

In the spring of 2019, in the vicinity of the Lukavac coke industry, average soil samples were first taken from three plots of land with an area of 1000 m^2, with the aim of determining the content of heavy metals. Only the plots that are used or can potentially be used for the purpose of agricultural production, which had no major slopes and/or depressions, were selected. On those plots the application of remedial measures is easily feasible.

Analysis of total and accessible forms of heavy metals (Cd, Cr, Pb, Zn, Cu, Mn, Fe, Ni) was performed on average soil samples in the laboratory of the Faculty of Agriculture and Food, University of Sarajevo. Also, within the framework of these studies, the type of land and the basic parameters of fertility were determined (pH, humus content, carbonate content, and the content of accessible forms of phosphorus and potassium). The plot of land, which was determined to have a higher content of certain heavy metals according to the current legislation in the Federation of Bosnia and Herzegovina, was taken into the further focus of the work. Appropriate remediation measures were carried out, such as chemical stabilization of the land using aluminosilicate minerals and phytoextraction/phytoaccumulation.

The first action during the process of chemical stabilization of contaminated soil using aluminosilicate materials (zeolite and pyrophyllite) involved the agrotechnical measure of plowing and harrowing and then setting up a trial using the method of random block arrangement with 7 variants in 3 repetitions. The variants of the experiment were as follows:

1. Variant (control; without addition of pyrophyllite and zeolite).
2. Variant (application of 200 kg/ha of 0–3 mm granulation zeolite).
3. Variant (application of 400 kg/ha of 0–3 mm granulation zeolite).
4. Variant (application of 600 kg (ha) of 0–3 mm granulation zeolite).
5. Variant (application of 200 kg/ha of pyrophyllite with a granulation of 0–3 mm).
6. Variant (application of 400 kg/ha of pyrophyllite with a granulation of 0–3 mm).
7. Variant (application of 600 kg/ha of pyrophyllite with a granulation of 0–3 mm).

The sample plots had an area of 50 m^2, with a 2 m wide distance between all plots to avoid the influence of one variant on another. Seven days after setting up the experiment, corn was sown on the tested plots, with the aim of determining whether the application of aluminosilicate materials had an effect on the reduced uptake of heavy metals by the tested plant. At the moment of technological maturity of the corn, sampling of plant material and soil was carried out on the experimental plots. The content of heavy metals was tested in the plant material (leaf), and the content of accessible forms of heavy metals in the soil.

2.1 Properties of Pyrophyllite

Pyrophyllite is a natural mineral from the group of aluminosilicates with the chemical formula $Al_2[Si_4O_{10}](OH)_2$. It got its name from the Greek words pyr - fire and philon - leaf, because it spreads in fan-shaped manner when heated. It is characterized by the following properties: hardness is between 1 and 1.5 on the Mohs scale, density between 2.7 and 2.9 g/cm^3, hydrophobic, insoluble in water and does not swell, alkaline reactions (pH between 7.5 and 7.8 (Hasanbegović et al., 2020). It has a very high effective cation exchange capacity, between 50 and 70 meq/100 g. It is soft to the touch, visually very similar to talc, and its color varies depending on the proportion of oxides and other components in the pyrophyllite ore, but as a rule it is white to gray white color.

The pyrophyllite material used in this research was created by crushing and sieving pyrophyllite ore from the Parsovići - Konjic site, Bosnia and Herzegovina. Except for silicon dioxide (SiO2) and aluminum oxide (Al2O3), which together make up 80 to 90%

of the chemical composition of pyrophyllite, it also contains various other impurities that can greatly affect its properties, especially the effective cation exchange capacity. The chemical analysis of the prifofilt material used in this research was done in the laboratory of the Faculty of Agriculture and Food Sciences, University of Sarajevo, and the obtained results are presented in Table 1.

Table 1. Chemical analysis of pyrophyllite used in this research

Tested parameter	Unit	Value
pH (H_2O)	pH	8.5
pH (KCl)	pH	8.1
SiO_2	%	67.55
Al_2O_3	%	19.10
Ca	%	3.65
K	%	0.023
Mg	%	0.135
Fe	%	0.337
Cu	mg/kg	1.40
Ni	mg/kg	2.74
Zn	mg/kg	25.68
Co	mg/kg	0.40
Mn	mg/kg	93.14
Cd	mg/kg	n.d.*
Pb	mg/kg	7.97
Cr	mg/kg	0.76

* n.d. - not detected (below the detection limit of the instrument)

The results from Table 1 show that the pyrophyllite used in this research does not deviate significantly from the basic chemical characteristics of pyrophyllite ores, except for the fact that it contains a relatively high proportion of calcium and magnesium. It can be assumed that the pyrophyllite ore from the Parsovići - Konjic site contains a certain amount of dolomite or some other natural material rich in the mentioned elements. A positive characteristic of the pyrophyllite material used in this research is the fact that it contains very low concentrations of toxic heavy metals, which makes it environmentally acceptable for use in remedial purposes.

2.2 Properties of Zeolite

Zeolite is a hydrated aluminosilicate material created by mixing volcanic lava with alkaline groundwater. Its structure is latticed, composed mostly of SiO44- and AlO45-

tetrahedra connected by common oxygen atoms. It contains both monovalent and divalent cations (Na+, K+, Ca2+) and certain amounts of water.

The zeolite used in this research was created by crushing and sieving zeolite ore from the Slanci - Veliko Selo site near Belgrade, Serbia. The basic characteristic of the zeolite used is a high cation exchange capacity (above 180 meq/100 g), which enables it to attract and bind to its structure, as well as exchange positively and negatively charged particles, i.e. cations and anions.

The complete chemical analysis of the used zeolite is shown in Table 2 (results taken from the website of the zeolite manufacturer https://zeolit.rs/).

Table 2. Chemical analysis of the zeolite used in this research

Tested parameter	Unit	Value
pH (H_2O)	pH	8.3
pH (KCl)	pH	7.4
SiO_2	%	64.55
Al2O3	%	14.49
CaO	%	4.9
K_2O	%	1.04
MgO	%	0.88
Fe_2O_3	%	2.3
Cu (bakar)	mg/kg	6.2
Ni (nikal)	mg/kg	21.0
Zn (cink)	mg/kg	42.0
Co (kobalt)	mg/kg	28.0
Cd (kadmij)	mg/kg	3.0
Pb (olovo)	mg/kg	35.0
Cr (hrom)	mg/kg	5.8

The work methods used in this research were as follows:

– a pH meter (ISO 10390, 1994) was used to determine the pH value of the soil,
– humus content in soil samples was determined by the dichromate method (ISO 14235, 1994),
– the content of easily accessible forms of potassium and phosphorus in soil samples was determined by the ammonium lactate (AL) method (Egner et al., 1960),
– the extraction of the total forms of heavy metals from the soil was done with the use of gold dust (a mixture of nitric and hydrochloric acid in a ratio of 1:3) according to the ISO 11466 (1998) method,
– the extraction of accessible forms of heavy metals from the soil was done using the so-called EDTA-solutions (a mixture of 1 M (NH4)2CO3 and 0.01 M EDTA

(ethylene-diamino-tetraacetic acid) where the pH of the solution was adjusted to 8.6 using HCl or NH4OH),

– the extraction of heavy metals from plant material was done with the use of nitric and sulfuric acid (in a ratio of 2.5:1) according to the method specified in the practicum of Lisjak et al. (2009),
– the determination of the content of heavy metals in plant material samples, and the content of total and accessible forms of heavy metals in soil samples was performed using the method of flame atomic absorption spectrophotometry on a Shimadzu 7000 AA apparatus (ISO 11047, 1998).

For the interpretation of the results related to the content of heavy metals in the soil, the Rulebook on determination of permitted amounts of harmful and dangerous substances in the soil for the FBiH (Official Gazette of the FBiH, 72/09) and the classification of soil pollution according to Bašić (1994) were used. Within the framework of the classification of land pollution according to Bašić, land is ranked in several classes, depending on the degree of land pollution with heavy metals (So).

The degree of soil contamination (So) is determined from the ratio of the determined soil contamination with a particular heavy metal to the limit value for the specified heavy metal.

So = content in soil (mg/kg) / limit value.

3 Results and Discussion

A large number of industrial facilities are located in the area of the Lukavac municipality within a distance of a few kilometers, some of which, such as coke ovens and soda factories, are major polluters of the environment. Accordingly, the development of agricultural production in the mentioned area is very questionable, especially on lands located along the Spreča river, into which waste water from various industrial plants flows, not only from the area of the Lukavac municipality, but also from the Tuzla canton as a whole.

The experimental plot was located in the immediate vicinity of the Lukavac coking plant. The type of soil on the mentioned plot is humofluvisol fluvial, meadow - brown non-carbonate clay soil.

After the analysis of the average soil sample from the said plot, the content of heavy metals was determined, which is shown in Table 3.

Table 3. Results of the analysis of the content of heavy metals in the tested soils

No			Content of heavy metals (mg/kg soil)					
	Cu	Zn	Mn	Cd	Pb	Ni	Cr	Co
1	13.3	41.93	249.29	n.d	16.50	54.31	69.91	9.03

Before carrying out the process of chemical stabilization of the tested land, the degree of its contamination with heavy metals was determined based on the ratio of the determined contamination of the soil with an individual heavy metal to the limit value for the specified heavy metal (So), i.e. based on the classification of soil contamination (Bašić, 1994). The threshold values needed to calculate the So factor are taken from the Rulebook on determining the permitted amounts of harmful and dangerous substances in soil for FBiH. According to the obtained results, the examined land is classified in the III class of land pollution, i.e. in the land of heavy pollution with heavy metals (the land belonging to this class can be used for growing plants, but enhanced protection measures are required).

As part of determining the fertility of the tested soil, the values of the following parameters were determined: humus content, carbonate content, content of accessible forms of phosphorus and potassium, and the pH value of the tested soil. The obtained values for selected soil fertility parameters are shown in Table 4.

Table 4. Presentation of the results obtained from the chemical analysis of the tested soil

Tested parameter	Unit	Value	Soil characteristics on basis of measured values
pH (H_2O) pH (KCl)	-	4.8	acidic reaction
	-	3.9	
Humus	%	2.5	moderately humous
Phosphorus (P_2O_5)	mg $100g^{-1}$	12.6	medium
Potasium (K_2O)	mg $100g^{-1}$	14.6	medium
Carbonates	%	0	-

The results of the analysis showed that the tested soil is extremely acidic with a moderate content of organic matter. The content of accessible forms of phosphorus and potassium in the examined soil was at a medium level, and the content of carbonates was not determined.

The average contents of accessible forms of heavy metals in the examined soil (mg/kg soil), depending on the treatment with aluminosilicate materials, are shown in Table 5.

From the results shown in Table 5, it can be seen that the content of accessible forms of Cd and Pb was not determined in any of the examined plots of land. It can also be seen that the content of accessible forms of all other tested heavy metals in the soil, with the exception of Cr, was always lower in the variants where the soil was treated with aluminosilicate materials. With the aim of determining whether the mentioned influence of pyrophyllite and zeolite on reducing the accessibility of heavy metals in the investigated soil located near the coke plant in Lukavac was statistically significant, an analysis of variance (F test) was carried out, as well as testing the significance of the differences between the environments for the content of those heavy metals in which F test proved significant.

The results of the analysis of variance showed that the influence of pyrophyllite and zeolite on reducing the availability of Cu, Pb and Cr in the tested soil near the Lukavac coking plant was not statistically significant. Likewise, zeolite did not show a statistically

Table 5. Results of testing the significance of the differences of the environments for the content of accessible forms of heavy metals in the soil, near the coke oven in Lukavac.

Variant*	Content of accessible forms of heavy metals in soil (mg/kg soil)							
	Cu	Zn	Mn	Cd	Pb	Ni	Cr	Co
V_1 (control)	1.6	0.53^{a}	10.14^{a}	n.d.**	n.d	9.53	0.27	0.042^{a}
V_2 (Z200)	1.59	0.52^{ab}	9.65^{ab}	n.d	n.d	8.88	0.27	0.031^{abc}
V_3 (Z400)	1.47	0.52^{abcd}	7.13^{abc}	n.d	n.d	8.14	0.27	0.027^{bc}
V_4 (Z600)	1.48	0.52^{abc}	7.62^{abc}	n.d	n.d	8.63	0.29	0.038^{ab}
V_5 (P200)	1.46	0.42^{e}	5.60^{c}	n.d	n.d	8.1	0.28	0.000^{d}
V_6 (P400)	1.53	0.43^{e}	6.05^{bc}	n.d	n.d	8.7	0.28	0.021^{c}
V_7 (P600)	1.54	0.46^{bcde}	7.57^{abc}	n.d	n.d	8.98	0.26	0.023^{c}
$LSD_{0.05}$	-	0.069	3.61	-	-	-	-	0.014

* V1 - Control (untreated variant); V2 - Zeolite 200 kg/ha; V3 - Zeolite 400 kg/ha; V4 - Zeolite 600 kg/ha; V5 - Pyrophyllite 200 kg/ha; V6 - Pyrophyllite 400 kg/ha; V7 - Pyrophyllite 600 kg/ha
** (not detected: below device detection level)

significant effect on the reduction of Zn and Mn availability in the tested soil. The only element whose accessibility in the soil is reduced by the application of zeolite is Co, and only when zeolite was applied in a dose of 400 kg/ha.

The use of pyrophyllite, in contrast to zeolite, had a statistically significant effect on reducing the content of accessible forms of Zn and Co in the tested soil, regardless of the applied dose. Varieties of 200 kg/ha and 400 kg/ha of pyrophyllite had a positive effect on the reduction of Mn availability as well.

The average content of the tested heavy metals in the leaves of corn (mg/kg dry matter) grown on the tested soil, depending on the treatment of the soil with aluminosilicate materials, are shown in Table 6.

The content of Cd and Pb was not determined in any sample of corn leaves, regardless of the land plot from which they were collected. When it comes to other elements, it can be seen from the Table 6 that the content of Zn, Mn and Ni in corn leaves was always lower in leaves taken from plots that were previously treated with aluminosilicates, regardless of their type or application dose. This sequence of results was not fully confirmed when the subject of the test was the content of Cu and Cr in corn leaves.

With the aim of determining whether the differences in the content of heavy metals in corn leaves between the tested varieties were significant, the F test was performed, and the significance testing of the differences between the means for the content of those heavy metals in which the F test proved to be significant. The obtained results are also shown in Table 6.

The content of Cd and Pb was not determined in the leaves of corn, which was expected if we take into account the fact that in the land where corn was grown, the content of accessible forms of the mentioned elements was not determined either.

The used aluminosilicate zeolite did not show a statistically significant effect on the reduction of the content of almost any tested element in corn leaves. The only exception

Table 6. Results of testing the significance of the differences between the environments for the content of heavy metals in the leaves of corn grown on the tested soil located near the Lukavac coking plant.

Variant*	Content of heavy metals in corn leaves (mg/kg dry matter)						
	Cu	Zn	Mn	Cd	Pb	Ni	Cr
V_1 (cont.)	7.18	14.53^{a}	29.06^{a}	n.d	n.d	1.77	0.45
V_2 (Z200)	7.29	13.63^{ab}	29.01^{ab}	n.d	n.d	1.63	0.46
V_3 (Z400)	6.82	13.26^{bc}	26.31^{c}	n.d	n.d	1.56	0.47
V_4 (Z600)	6.12	12.12^{cd}	26.58^{bc}	n.d	n.d	1.47	0.36
V_5 (P200)	6.09	11.49^{de}	27.48^{abc}	n.d	n.d	1.51	0.47
V_6 (P400)	6.11	11.27^{de}	26.20^{c}	n.d	n.d	1.56	0.43
V_7 (P600)	6.17	10.21^{e}	23.20^{d}	n.d	n.d	1.62	0.43
$LSD_{0.05}$	-	1.184		-	-	-	-

* V1 - Control (untreated variant); V2 - Zeolite 200 kg/ha; V3 - Zeolite 400 kg/ha; V4 - Zeolite 600 kg/ha; V5 - Pyrophyllite 200 kg/ha; V6 - Pyrophyllite 400 kg/ha; V7 - Pyrophyllite 600 kg/ha

is the content of Zn in the leaves of corn applied at a dose of 400 kg/ha and 600 kg/ha and Mn at a dose of 400 kg/ha, compared to the control variant. Pyrophyllite, regardless of the application dose, showed a statistically significant effect on the reduced Zn content in corn leaves when compared to the control, untreated variant. A statistically significant effect of pyrophyllite in doses of 400 and 600 kg/ha on the reduced Mn content in corn leaves was also recorded.

4 Conclusion

– According to the classification of land contamination with heavy metals, the tested soil in the vicinity of the Lukavac coke plant belongs to the III class of land (So from 0.5–1), which means that agricultural production is allowed on the mentioned soil, but with enhanced protection measures.
– Soil treatment with zeolite and pyrophyllite did not have a statistically significant effect on reducing the accessibility of Cu, Ni, and Cr, regardless of the applied amount of these aluminosilicates.
– Soil treatment with zeolite only showed a statistically significant effect on reducing the content of the accessible form of Co compared to the control variant, but only at the application dose of 400 kg/ha
– Soil treatment with pyrophyllite showed a statistically significant effect on reducing the availability of Zn, Mn and Co, regardless of the applied dose.
– In the conditions of the conducted experiment, the application of pyrophyllite at a dose of 200 kg/ha reduced the accessibility of Zn by 20.7% and Mn by 44.8% in the examined soil, the application of pyrophyllite at a dose of 400 kg/ha reduced the accessibility of Zn by 18.9% and Mn by 40.3%, and the application of pyrophyllite in a dose of 600 kg/ha reduced the availability of Zn by 13.2% and Mn by 25.3%.

– The application of zeolite affected the reduction of the accumulation of Zn and Mn content in corn leaves compared to the control variant and only if it was used in a dose of 400 kg/ha and 600 kg/ha.
– The treatment of soil with pyrophyllite, regardless of the applied dose, significantly reduced the accumulation of Zn in corn leaves when compared to the control (untreated) variant. The application of pyrophyllite also had an effect on the reduction of the accumulation of Mn content in corn leaves, and only if it was used in doses of 400 kg/ha and 600 kg/ha. When it comes to the accumulation of other examined heavy metals in corn leaves, the treatment of soil with pyrophyllite did not show its justification.

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