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Effect of liming of sod-podzolic soils with by-products of steel production on soil acidity and composition of wash water (column experiments)

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Abstract

The waste slag materials from metallurgical plants contain calcareous materials, such as blast furnace (BFS) and converter slags (CS) of the Chelyabinsk Metallurgical Plant. However, the widespread use of these materials is limited by the presence of harmful impurities in their composition that can have a negative effect on soils and plants. The aim of our research is to study the effect of liming of soddy-podzolic soils with metallurgical slags on the pH_{KCl} value and the composition of the wash water. In a model experiment on columns, the migration ability of alkali metals from soils of light granulometric composition was studied after using two phases of steelmaking waste as a lime material. Research results indicate that the ameliorative properties of BFS and converter slags were different. When liming with BFS slag, a month after composting, the pH_{KCl} value increased to 5.1 units. Studied soil from the category of "strongly acidic" moved to the category of "weakly acidic". When liming with the converter slag, the pH_{KCl} value of the soil increased from 4.1 to 4.7 (the soil from the "strongly acidic" category moved to the "medium acidic" category). With an increase in the period of washing, the pH of the infiltration water increases. In the treatments with the use of BFS slag this increase was higher due to the continuing dissolution of ameliorants and the higher chemical activity of BFS slag. The liming led to intensive migration of alkaline earth metals. In the treatments limed with more soluble (chemically active) BFS slag, calcium losses were higher. Empirical estimation of alkaline earth metals leaching from the soil allowed to model the dynamics of the base migration. The dynamics of Ca and Mg migration from the soil were fundamentally different (content of Ca decreased, and of Mg increased). The dynamics of Ca migration from the soil limed with converter slag was most pronounced in comparison with the Ca dynamics for BFS slag and the Mg dynamics in all treatments.

Keywords: blast furnace slag, cation migration, converter slag, industrial waste, liming, steelmaking

Introduction

Utilization of human waste products and industrial wastes is one of the global problems of human development. Slag is the by-product of industrial processes in which mineral iron is reduced, producing high carbon iron and steel. The use of metallurgical solid wastes such as steel slag, in agricultural activity, has become very important in reducing the accumulation of industrial landfills and in increasing crop production (Branca and Colla 2012; Das et al 2019). Industrial slags contain certain plant nutrients (CaO), silicic acid (SiO_2), phosphoric acid (P_2O_5), magnesia (MgO), Mn, and Fe) (Ito 2015), and calcareous materials that can be used to correct soil acidity (Das et al 2007; Ning et al 2016) as well as can stabilize heavy metal activity in soil (Ning et al 2016) This waste material contain calcium and magnesium silicates, which show neutralizing action due to SiO_3^{-2} base (Alcarde et al 2003). Many countries actively recycles metallurgical slag as a fertilizer and liming source. So, in Germany, ¾ of the lime fertilizers represented by various types of metallurgical slag (Shilnikov et al 2008). Notably, steel-making slag have been extensively utilized as raw materials for fertilizer production, mostly in Japan, Korea, and China (Das et al 2019). In the Russian Federation, metallurgical slag dumps occupy about 1 million hectares (Vorobiev 2019).

The Department of Soil Science and Agrochemistry of the St. Petersburg State Agrarian University (Russia), together with the Agrophysical Research Institute (St. Petersburg, Russia) has been dealing with the problem of using non-traditional chemical ameliorants (local lime materials and industrial waste) for liming acidic soils (Litvinovich et al., 2001, 2013, 2016, 2017, 2018, 2019a, 2019b, 2020; Pavlova et al., 2019a, 2019c, 2020; Lavrishchev et al., 2017). The widespread use of these materials is limited by the presence of harmful impurities in their composition that can have a negative effect on soils and plants. These limestone materials include blast furnace (BFS) and converter slags (CS) of the Chelyabinsk Metallurgical Plant. The northwester part of Russia is characterized by very acidic to acidic soils predominantly. Therefore the neutralizing the soil acidity in this region is of a vital importance to produce sustainably crop yields. Shilnikov et al (2011) generalized results of lysimetric studies over 25 years and have shown that to compensate for the natural removal of magnesium and calcium from soils, their annual intake of at least 400-450 kg / ha in terms of CaCO3 is required. Various researchers reported results of using steel slag in agriculture and showed that the correct application of these waste materials increase the pH in acidic soils, increase content of phosphorus, calcium and magnesium, as well as increase the content of silicon (Deus et al 2014; Mantovani et al 2016; Souza and Korndörfer 2010), thus ultimately contributing to the increase in crop yields (Deus et al 2018).

The results of a long-term field experiment of the All-Russian Research Institute of Agrochemistry showed that for 30 years the positive effect of metallurgical slag on the yield of agricultural crops was higher than that of limestone flour. The yield payback of using 1 ton of the active substance of metallurgical slag was 1.45 tonnes of grain units, and of limestone flour - 1.01 (Shilnikov et al 2010). Although there are many papers reporting the ability of metallurgical slag to neutralize soil acidity, the mechanisms of migration of basic cations from soil after addition of slag is neither well understood nor properly studied. Leaching of calcium and magnesium from liming soils is a big challenge worldwide (Adomaitis et al., 2013; Fernández-Sanjurjo, 2014; Goulding, 2016), especially in the humid region with percolation water regime and acidic soils (Bakina 2012; Litvinovich et al., 2012; Pavlova et al 2019b; Litvinovich et al 2019b). The aim of this research was to study the effect of liming of sod-podzolic soils with metallurgical slags on soil pH_{KCl} and the composition of the wash water. The tasks included:

- Reveal the change in pH_{KCl} one month after composting;
- Study the dynamics of pH value of rinsing water depending on the period of washing;
- Determine the content of Ca^{2+} and Mg^{2+} in wash waters
- To construct empirical models of Ca^{2+} and Mg^{2+} leaching from soils limed with steelmaking slags

Materials and methods

The research was conducted on the acidic soddy-podzolic soil of light granulometric composition (Table 1), limed with by-products of the steelmaking production of the Chelyabinsk Metallurgical Plant (blast furnace, BFS and converter slags, CS) as test lime materials (Table 2). Steel making process is conducted by three methods: blast furnace, converter or electric furnace (Nishiwaki 1986). In the paper we tested two type of steel making by-products: blast furnace (BFS) and converter slag (CS).

Table 1. Soil physical and chemical composition

pH_{KCl}	Humus, %	Ca^{2+}	Mg^{2+}	$Ca^{2+}+Mg^{2+}$	CEC	Particles < 0.01 mm, %
		mmol(eq)/100 g				
4.1	3.02	8.2	2.9	11.1	22	18.71

Table 2. Chemical composition of steel slags, %

Element	Blast furnace slag	Converter slags
$CaCO_3$	78.96	81.05
Mg	3.53	4.36
K	0.62	0.58
P	0.14	0.19
S	0.12	0.13
Cu	0.008	0.015

As can be seen from the data presented in the Table 2, the studied slags have a high neutralizing ability and contain a small amount of basic nutrients. The study of the composition of waters seeped from sod-podzolic soils of various humus content was studied on separating funnels. Before filling the funnels, the soil was limed according to the following scheme:

1. Control, no liming
2. Blast furnace slag at a dose calculated by full dose of hydrolytic acidity (1Hy)
3. Converter slag at a dose calculated by full dose of hydrolytic acidity (1Hy)

The limed soil was placed in cups in a composting thermostat. The composting period was 30 days at a temperature of 28˚C and soil moisture was maintained at 60% of full field moisture capacity. After composting, the soil was again dried, crushed and sieved through a 1 mm sieve. Then the soil was placed in separating funnels and soaked with water. The mass of soil in the funnel was 300 g. The height of the soil layer was 17 cm. The density of soil packing in the funnel was 1.0–1.1 g cm^{-3}. After saturation of the soil with moisture, washing was started with a calculated volume of distilled water corresponding to the annual amount of precipitation seeping through the soil stratum. The calculation of the water for washing imitated precipitation was made based on the data of mean annual precipitation in the Northwest of Russia that is around 600 mm, and transpiration by plants and evaporation from the soil surface that is around 400 mm (http://www.meteo.nw.ru/articles/ index.php?id=2; Pestryakov 1977). Thus, annually, 200 mm of precipitation percolates through the soil stratum. The amount of water required for a single wash of one column was calculated from the equation: $V = \frac{\pi * r^2 * 200}{1000}$, where,π = 3.14; r –column radius, mm; 1000 – transition to ml. According to the equation, for one washing, 400 ml of water was used. In total five (5) washings were conducted.

Analytical work was carried out by conventional methods: pH potentiometrically; content of the cations in the eluates was determined by complexometric method after extraction by EDTA (Cantarella et al., 2001); content of humus was determined by Tyurin method; hydrolytic acidity by Kappen method; soil granulometric composition was determined according to Kachinsky method and cation exchange capacity was determined by the Bobko-Askinazi method (Mineev 2001). Chemical composition of the liming materials was determined by X-ray fluorescence spectroscopy using a Spectroscan Max X-ray crystal diffraction spectrometer

Results and discussion

During composting, the studied steel-smelting slags reacted with the soil and reduced the exchangeable acidity (Table 3). Moreover, the BFS slag turned out to be more chemically active. In the treatments limed with this slag, the pH_{KCl} shift was the highest.

Table 3. Effect of addition of steelmaking by-product slags on soil acidity (pH_{KCl})

Treatment	pH (KCl)
Control without liming	4.1±0.2
Blast furnace slag (BFS)	5.1±0.1
Converter slag (CS)	4.7±0.1

Table 3. shows that the ameliorative properties of BFS and converter slags are different. A month after composting, the pH_{KCl} value of the soil increased from 4.1 to 4.7 (the soil from the “strongly acidic” category moved to the “medium acidic” category). When liming with BFS slag, the pH_{KCl} value increased to 5.1 pH units. Soil from the “highly acidic” category moved to the category of “slightly acidic”. Similarly, Suwarno et al (2001) found that the magnitudes of the effects of the two slags were different.

Table 4. represents the data on the content of calcium and magnesium cations in washing waters. The maximum amount of these cations in the eluates was observed during the first period of washing. In the subsequent periods, the concentration of calcium and magnesium in the wash water decreased.

Table 4. Content of Ca and Mg ions in wash waters, mg/ml

Treatment	Cation	Periods of washing				
		1	2	3	4	5
Control	Ca	0.0006	0.0008	0.0009	0.0007	0.0008
	Mg	0.0006	0.0007	0.0006	0.0005	0.0007
Blast furnace slag	Ca	0.0047	0.0018	0.0016	0.0023	0.0018
	Mg	0.0008	0.0012	0.0009	0.0008	0.0024
Converter slag	Ca	0.0044	0.0017	0.0021	0.0009	0.0006
	Mg	0.0004	0.0006	0.0008	0.0004	0.0010

The results of the pH of the wash water (Figure 1.) showed that with an increase in the time of the experiment, the pH of the wash water increases. In the treatments with the use of BFS slag, this increase was higher due to the higher chemical activity of BFS slag. In addition, Suwarno et al (2001) found that both types of slag significantly increased soil pH; exchangeable Ca, and Mg in Andisol.

In our study, the maximum amount of Ca was found in the filtrates of the first wash. This picture did not depend on the type of ameliorant. By the end of the experiment, the concentration of Ca in the eluates in the experiment with MS decreased to 0.0018 mg/ml, i.e. decreased twofold. In the treatment with BFS slag, the decrease was more noticeable. The difference was 0.0042 mg (i.e., it decreased by 8 times).

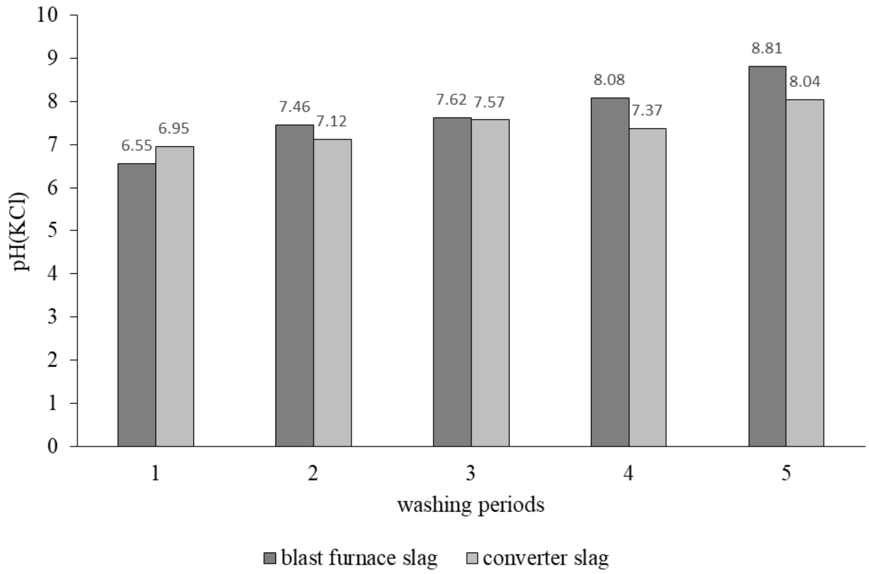

Figure 1. Effect of washing periods on the changes of pH in washing waters

A different picture was established when analysing the Mg content in the wash water. Regardless of the treatment the Mg concentration in the filtrate of the 1st wash was minimal. Until the 5th wash observation period, the concentration of Mg in the wash water fluctuated slightly, while a sharp increase in the concentration of Mg was found in the filtrates of 5th washing. The total amount of leached calcium and magnesium for 5 periods of washing is shown in Figure 2.

Liming enhanced the migration of studied alkaline earth metals. Moreover, in the treatments limed with more soluble (chemically active) BFS slag, calcium losses were higher. In the same treatments, the Mg concentration in the eluates of the 5th washing period increased by 3, and in the treatment with the converter slag by 2.5 times. In total, for the entire period of the experiment, the losses from the soil limed with BFS slag were: Ca - 4.88 mg and Mg - 2.44 mg, while from the soil limed with converter slag Ca – 3.38 mg; Mg – 1.28 mg. The complete removal of these cations from the soil of all treatments was not revealed, implying that the nature of migration of Ca and Mg in the reclaimed waste was different.

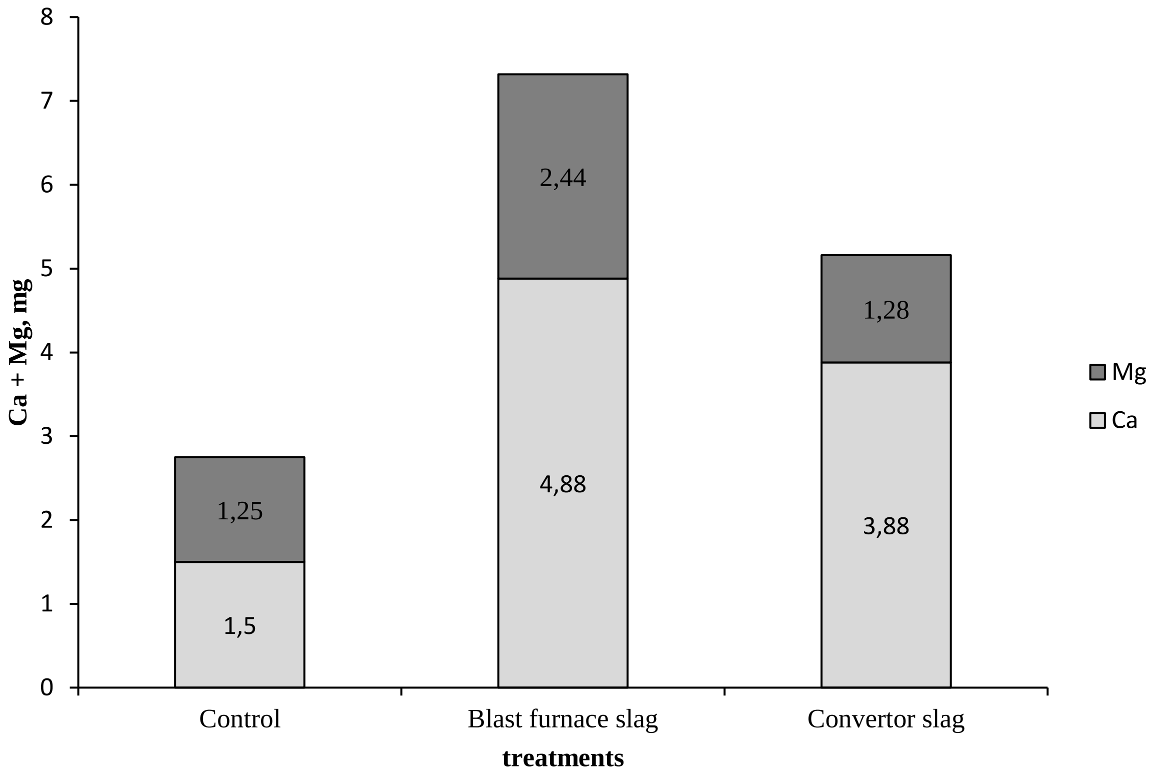

Figure 2. Sum of the calcium and magnesium ions leached from the soil, mg

Empirical models of Ca^{2+} and Mg^2+ leaching and the sum of bases are presented in Table 5 and Figure 3-14.

Table 5. Empirical models of leaching of Ca^{2+}, Mg^{2+} and the sum of bases

No	Model	Average rate of change in metal content (v)	Fishers' criterion *(p)*	Coefficient of determination (R^2)
1.1	$y_{11} = 0{,}004 - 0{,}00053 \cdot t$	– 0.00053	0.23	0.42
1.2	$y_{12} = 0{,}00038 + 0{,}00028 \cdot t$	0.00028	0.23	0.42
1.3	$y_{13} = 0{,}00446 - 0{,}00026 \cdot t$	– 0.00026	0.56	0.11
2.1	$y_{21} = 0{,}00446 - 0{,}00084 \cdot t$	– 0.00084	0.046	0.78
2.2	$y_{22} = 0{,}00034 + 0{,}0001 \cdot t$	0.0001	0.27	0.36
2.3	$y_{23} = 0{,}00495 - 0{,}00077 \cdot t$	– 0.00077	0.047	078

As seen from Table 5 empirical models 2.1 and 2.3 have a high, the models 1.1; 1.2 and 2.2 - not high, and the model 1.3 - a low level of statistical significance according to the Fisher's test. Generally, analysis of the models of migration of bases from the soil, show that the models 1.1, 1.2, 1.3 and 2.1, 2.2 and 2.3 are similar. The dynamics of leaching of Ca and Mg showed different pattern, where Ca content decreased and Mg content increased. The dynamics of Ca migration from the soil limed with converter slag was most pronounced (models 2.1 and 2.3 have a high level of significance) compared to the dynamics of Ca for BFS slag and the dynamics of Mg in all treatments.

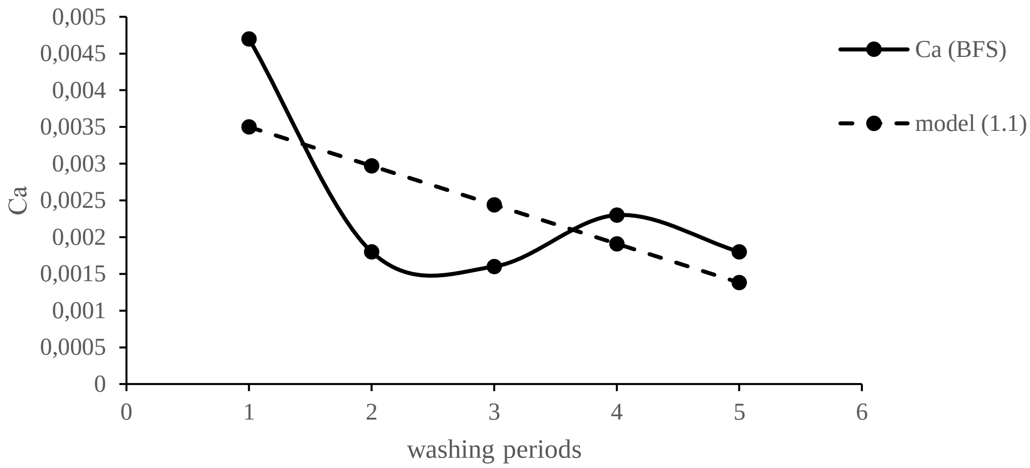

Figure 3. The dynamics of Ca migration from the soil limed with blast furnace slag (BFS)

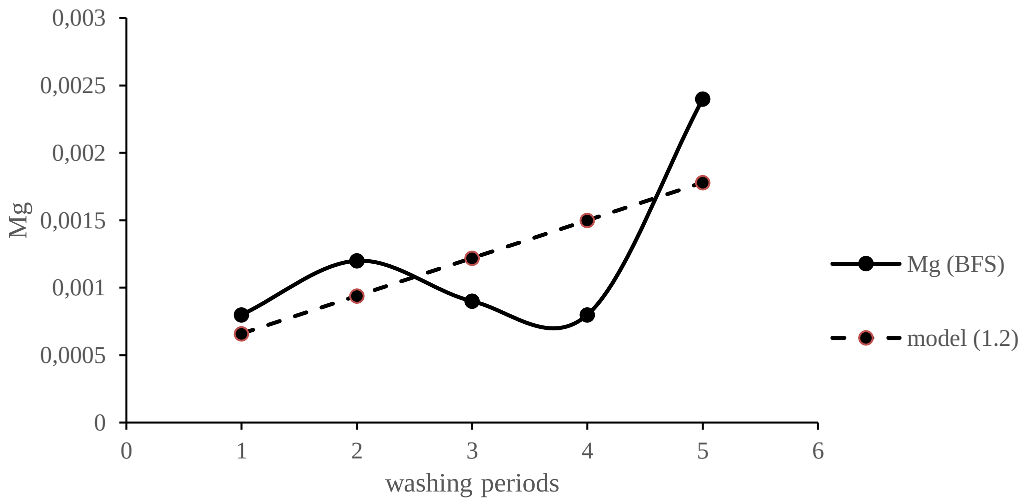

Figure 4. The dynamics of Ma migration from the soil limed with blast furnace slag (BFS)

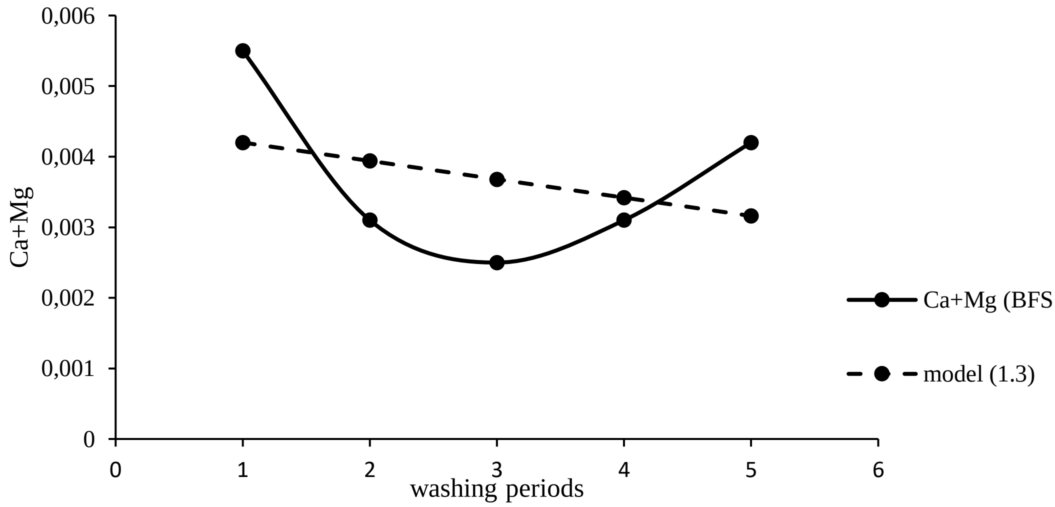

Figure 5. The dynamics of migration of sum of Ca+Mg from the soil limed with blast furnace slag (BFS)

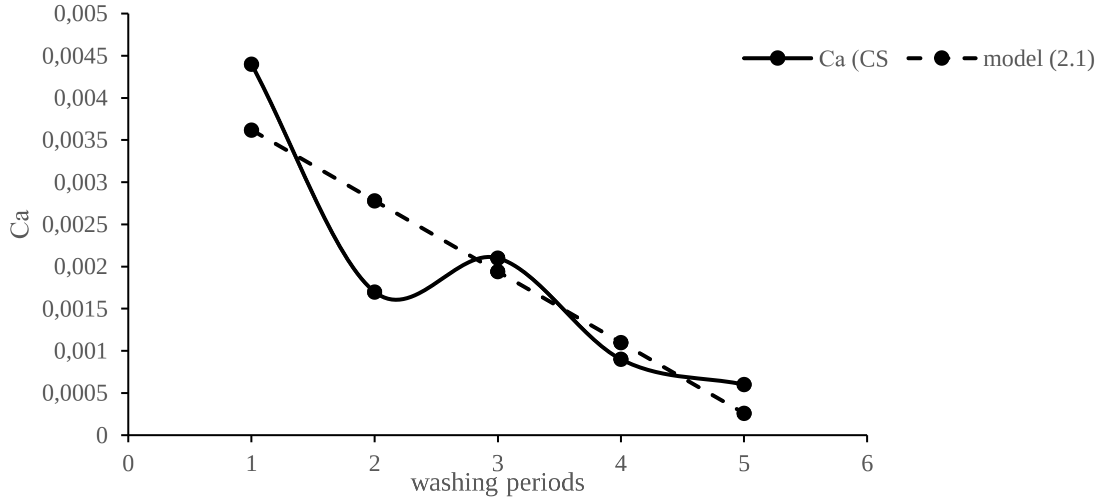

Figure 6. The dynamics of Ca migration from the soil limed with converter slag.

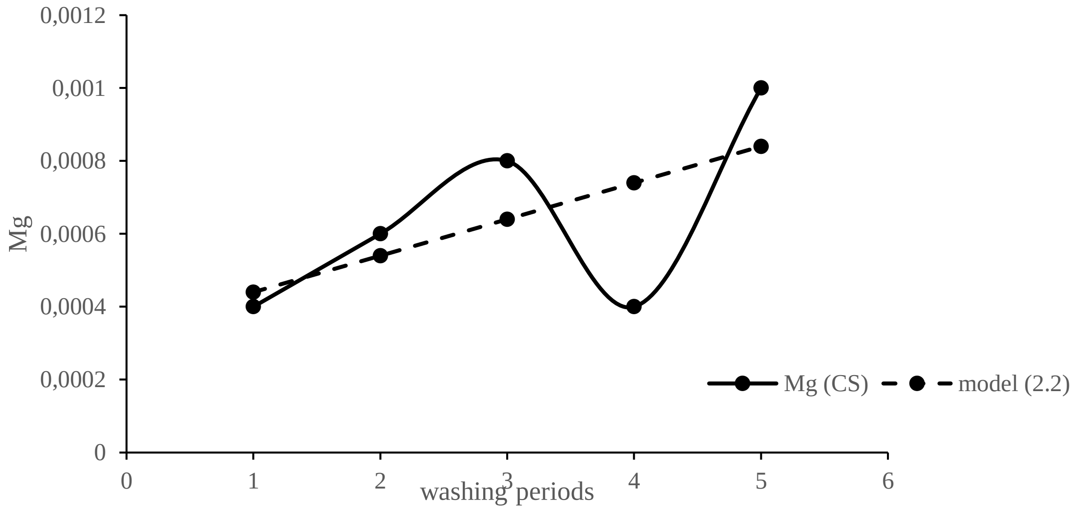

Figure 7. The dynamics of Ma migration from the soil limed with converter slag.

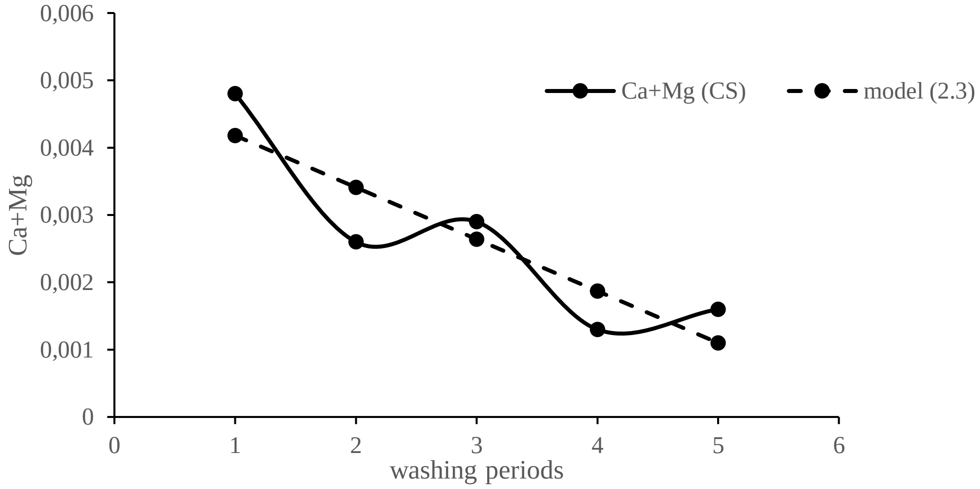

Figure 8. The dynamics of migration of sum of Ca+Mg from the soil limed with converter slag.

Application of slag can neutralize part of the soil acidity due to presence of neutralizing base SiO_3^{2-} (Alcarde 1992) that reacts in water and releases hydroxyl (OH^{-1}) ions, according to the equations showed by Alcarde et al. (2003):

$$CaSiO_3 + H_2O \rightarrow Ca^{2+} + SiO_3^{2-} \quad (1)$$

$$MgSiO_3 + H_2O \rightarrow Mg_2 + SiO3^{2-} \quad (2)$$

$$SiO3^{2-} + H_2O \text{ (soil)} \leftrightarrow HSiO^{-}_3 + OH^- \ (Ci_1 = 1.6 \times 10^{-3}) \quad (3)$$

$$HSiO^{-}_3 + H_2O \text{ (soil)} \leftrightarrow H_2SiO_3 + OH^- \ (Ci_2 = 3.1 \times 10^{-3}) \quad (4)$$

$$OH^- + H^- \text{ (soil solution)} \rightarrow H_2O \quad (5)$$

$$OH^- + Al^{3+} \text{ (soil solution)} \rightarrow Al(OH)_3 \quad (6)$$

The hydroxyl (OH^-) produced neutralizes the H^+ of the soil solution and the phytotoxic Al^{3+}, and consequently, there is an increase in pH and a decrease in the concentration of the potential acidity (H + Al) (Prado et al 2001). Correction of acidity with slag occurs similarly to the use of limestone. In addition to the decrease in acidity levels, Ca and Mg supply also occurs in soil (Torkashvand and Sedaghathoor 2007; Deus et al 2014; Mantovani et al., 2016). Suwarno et al., 2001) found that application of steel slag showed positive effect on plant growth compared to the dolomite liming that had no effect on P uptake by plants.

One of the reasons for the better action of silicates, in comparison with the carbonate forms of limestone fertilizers, is that the harmful effect of active aluminum in the soil is eliminated more completely and for a longer period, since when silicates are used in the soil, aluminosilicates are formed, which have a lower solubility than hydrants of aluminum oxide, obtained by adding Ca $(OH)_2$. In addition, lime-containing industrial waste, such as steel slag is one of the cheapest and highly effective way to combat soil acidity (Osipov 2017). Such slags, in comparison with the standard calcareous carbonate forms of lime fertilizers, have a specific effect on soil and plants, which is associated with the presence of silicic acid in them in the form of calcium silicate. In addition, other components of the slags are also important. There is also experimental evidence of the advantages of metallurgical slags in the process of liming soils.

In the process of steel making the main raw materials are: iron ore, coal, limestone and recycled steel scrap. In the blast furnace phase limestone or dolomite (fluxes) are added where they react with iron ore impurities, such as silica (Branca and Colla 2012). Akanova (2001) in a long-term experiment found that, the payback of 1 ton of $CaCO_3$ by additional crop production during the liming depended on the aftereffect time and the size of particles of the lime material. Shilnikov et al (2011) comparing various types of studied liming materials found that the highest payback was found from the addition of metallurgical slag. Suwarno et al (2001) also found that application of metallurgical slag showed better effect on growing plants than traditional dolomite liming.

X-ray diffraction of the converter slag have shown that the major phases present in this type of slag are dicalcium ferrite, calcium alluminate and wüstite. Although it contains also some reactive mineral phases, such as 2CaO. SiO2, 3CaO. SiO2 and free CaO e MgO (Das et al., 2007), still CS is considered as hard and stable and therefore is actively used in road construction (Branca and Colla 2012). Unlike, the more reactive open- hearth slag can be used in agriculture because of its high sorption capacity of phosphorus, which remains into the available form for the plants (Branca and Colla 2012). The expected negative effects of heavy metal concentrations in these slags supposedly can be controlled and avoided, since heavy metals tend to bound to the slag matrix and thus they are not available for plants (Branca and Colla 2012). Considering the positive effects of steelmaking slags as liming materials, such as better yield of the crops, soil protection and reduction of natural resources consumption (Hiltunen and Hiltunen, 2004), as well as environmental protection by utilization of landfills, the studied blast furnace slag can be successfully applied on studied acidic sod-podzolic soils of north western Russia.

Conclusions

Our study compared the effect of two types of steelmaking by-products (blast furnace slag and convertor slag). Addition of blast furnace slag to acidic sod-podzolic soils showed better performances as liming material, acting faster in increasing the pH of soil solution and converting strongly acidic soils into slightly acidic. In addition, due to higher reactivity of blast furnace slag compared to the converter slag, its aftereffect lasted longer. Liming with both types of slag resulted in an intensive migration of studied cations. The applied empirical models showed that the dynamics of Ca and Mg migrations were fundamentally different: content of Ca decreased and content of Mg increased. The dynamics of Ca migration from the soil limed with converter slag was most pronounced in comparison with the dynamics of Ca for blast furnace slag and the dynamics of Mg in all treatments.

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Утицај кречења земљишта типа подзола са нуспроизводима производње челика на киселост земљишта и састав воде за испирање (колона експеримент)

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Извод

Отпадни материјали као сто су шљака из металуршких фабрика садрже карбонатне материјале, попут сљаке из високе пећи (BFS) и конвертерске шљаке (ЦС) Чељабинске металуршке фабрике. Међутим, широка употреба ових материјала ограничена је због присуства штетних примеса у њиховом саставу које могу имати негативан ефекат на земљиште и биљке. Циљ нашег истраживања је проучавање утицаја крецења земљишта типа подзол металуршким шљакама на вредност pHKCl и састав воде за испирање. У моделном експерименту у колонама, проучавана је миграциона способност алкалних метала из земљиста лаког гранулометријског састава након употребе две фазе отпадног материјала од прооизводње челика као кречног материјала. Резултати истраживања показују да су мелиоративна својства BFS и конвертерске шљаке била различита. При кречењу са BFS шљаком, месец дана након компостирања, вредност pHKCl се повећала на 5,1 јединица. Проучавано земљиште из категорије „јако кисело“ прешло је у категорију „слабо кисело“. При кречењу са конвертерском шљаком, pHKCl вредност земљишта повећала се са 4,1 на 4,7 (земљиште из категорије „јако кисело“ прешло је у категорију „средње кисело“). Са повећањем периода испирања, pH воде за инфилтрацију се повећала. У третманима BFS шљаком ово повећање је било веће због континуираног растварања мелиораната и веће хемијске активности BFS шљаке. Кречење је довело до интензивне миграције земноалкалних метала. У третманима са додавањем растворљивог креча (хемијски активном) - BFS шљаком, губици калцијума су били већи. Емпиријска процена испирања земноалкалних метала из земљишта омогућила је моделирање динамике миграције базе. Динамика миграције Ca и Mg из земљишта била је суштински различита (садржај Ca смањен, а Mg повећан). Динамика миграције Ca из земљишта конвертерском шљаком била је најизраженија у поређењу са динамиком Ca BFS шљаком и динамиком Mg у свим третманима.

Кључне речи: шљака из високе пећи, кретање катјона, конвертер шљака, индустријски отпад, кречење, отпадне материје од производње челика

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