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NEW REMEDIATION TECHNOLOGIES

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CHEMICAL MELIORATION OF ACID SOILS USING METALLURGICAL SLAG IN VEGETATIVE EXPERIMENTS

Aleksandra Stanojković-Sebić, Srboljub Maksimović, Dragana Jošić, Radmila Pivić

Institute for Soil Science, Teodora Drajzera 7, Belgrade;

ABSTRACT

The effect of metallurgical slag application on physico-chemical properties of the soils with limited production capacity (Stagnosol), as well as on yield and chemical composition of cultivated vegetables (spinach and cabbage) as experimental crops, through the vegetative experiments performed in semi-controlled conditions, was studied. The long-term investigations were aimed to define the main parameters for possible wider usage of this secondary raw material (metallurgical slag) for amelioration and fertilization of acid soils in Serbia, as well as to indicate the justification for the use of this material in the wider agricultural practice.

Key words: chemical melioration, metallurgical slag, Stagnosols

INTRODUCTION

Majority of Serbian soils are characterized with high soil acidity. Application of only organic and mineral fertilizer is not enough to sustain soil productivity. On these soils, that occupy about 800.000 ha of the cultivated lands in Serbia, along with regular fertilization, it is necessary to apply Ca-containing fertilizers–calcifies, for improving their physicochemical and biological properties.
Application of lime materials and decrease of soil acidity reduce possibility (due to formation of insoluble forms) of higher uptake of toxic elements, which have tendencies of increased accumulation in soil due to anthropogenic pollution.

In particular neutralizing agents in the form of alkaline materials have been added to acid soils to ameliorate their physical-chemical properties and reduce metal phytoavailability (Bolan and Duraisamy, 2003; Gray et al., 2006). Many alkaline materials are available to neutralize the soil acidity including calcite($CaCO_3$), burnt lime (CaO), slaked lime ($Ca(OH)_2$), dolomite limestone $CaMg(CO_3)$, and slag $CaSiO_3$ (Bolan and Duraisamy, 2003). They can reduce the negative effects of potentially toxic trace elements such as As, Cr, Cu, Pb, Cd, and Zn. Liming of acid soils decreased the soluble fraction and plant uptake of Ni, Cd, Zn, and Cu as well as increased the crop yield (Negim et al., 2012)

The application of traditional alkaline liming materials such as limestone, dolomite and burnt lime to acid soils for the amelioration of acidity consequently improving
crop production is a common practice (Barber, 1984; Foth and Ellis, 1997). Along

with these materials, present in Serbia, Ca -containing metallurgical slag from Steel factory-Smederevo (Serbia) can be of great importance.

However, the alkaline nature and the need for sustainable and environmentally acceptable disposal options for metallurgical slag (Lopez et al., 1995) have prompted its use as a liming material on acid agricultural soils. Although metallurgical slag has the largest quantitative share in the overall metallurgical waste, its physicochemical properties offer a high potential for its utilization in agriculture (National Slag Association, 2001).

Regarding the above mentioned, the aim of this research was to investigate the effect of Ca -containing metallurgical slag from Steel factory from Smederevo, Republic of Serbia, comparing to the effects of selected commercial lime materials and fertilizers, on chemical composition of the aboveground biomass of spinach and cabbage, through vegetative experiments performed in semi-controlled conditions on Stagnosol (WRB, 2006), a type of soil with very acid reaction.

MATERIAL AND METHODS

Greenhouse Experiment

Considering the limited amount of samples of metallurgical slag from Steel factory – Smederevo, at this phase the investigations of its effect on chemical composition of the aboveground biomass of spinach were carried out in pot experiments, under semi-controlled condition in the greenhouse of Institute of Soil Science, Belgrade, from the third decade of February to the beginning of April during 2011 (Stanojković-Sebić et al., 2011) and of cabbage from April to the beginning of June, during. In the experiments the comparisons of the effect of metallurgical slag with other lime materials (ground limestone and hydrated lime) in combination without and with standard mineral fertilizers were studied. The ground limestone (calcium carbonate or calcite, $CaCO_3$) contains 60% of carbonate. Hydrated lime (slaked lime, $Ca(OH)_2$) reacts very rapidly and has a TNV (Total Neutralizing Value) of 135, thus 740 kg of hydrated lime is equivalent to one ton of ground limestone i.e. the TNV = 135 (Culleton et al., 1999).

The experiment was undertaken with a type of soil from central Serbia region that has very low pH and poor physical and biological properties- Stagnosol.

The designed experiments were in three replications: 1. Control – no fertilizer; 2. $CaCO_3$, no standard fertilization; 3. $Ca(OH)_2$, no standard fertilization; 4. Metallurgical slag, no standard fertilization; 5. NPK mineral fertilizer [composite NPK(15:15:15)] + metallurgical slag.

The experiment was performed in plastic pots with 4 kg of homogenized soils. Common spinach, *Spanacea oleracea* (variety Matador) and cabbage, *Brassica oleracea* var. *capitata* L. (variety f.. alba), were chosen as experimental crops. Before planting the crops the amount of fertilizers and slag were measured according to the experiment design and mixed with soil (calculated as for 1 ha): NPK – 15:15:15 = 500 kg ha-1; $CaCO_3$ = 4 t ha-1; $Ca(OH)_2$ = 2,8 t ha-1; Metallurgical slag = 4 t ha-1 (same as the amount of $CaCO_3$, in spite of lower amount of slag). All three lime materials with granulation of 0.2 mm were applied in the experiment.

Slag and Soil Analysis

Before industrial homogenizing and standard grinding the chemical composition of five composite samples of metallurgical slag used from different deposition sites was analyzed. The analyses of chemical characteristics of the study soil were done before the trial was set up.

The following chemical analyses were done: pH in water and 1M KCl was analyzed potentiometrically with glass electrode (SRPS ISO 10390:2007); total N was analyzed on elemental CNS analyzer Vario EL III (Nelson and Sommers, 1996); available P_2O_5 and K_2O were analyzed by Al-method according to Egner-Riehm (Riehm, 1958), where K was determined by flame emission photometry and P by spectrophotometer; Ca and Mg were extracted by ammonium acetate followed by determination on atomic adsorption analyzer SensAA Dual, GBC Scientific Equipment Pty Ltd, Victoria, Australia (Wright and Stuczynski, 1996); determination of effective cat ion exchange capacity (CEC) and base saturation level was done by the standardized method using barium chloride solution (Sumner and Miller, 1996); microelements were determined with an ICAP 6300 ICP optical emission spectrometer, after the samples were digested with concentrated HNO_3 for extraction of total forms, and by DTPA for extraction of soluble forms of the elements (Soltanpour et al., 1996); the total content of $CaCO_3$ in slag studied was determined using the "rapid titration method" by Piper (van Reeuwijk, 2002).

Plant Analysis

The aboveground biomass of spinach and cabbage plants was taken and after drying at 105°C the plant biomass was weighed and expressed in g per pot.
For all the plant samples from all the treatments the analyses of aboveground biomass were done.

The contents of N, P and K in plant samples were determined by so called "wet" combustion, i.e. they were heated to boiling with the mixture of concentrated acids: H_2SO_4 and $HClO_4$. In the obtained solution, nitrogen was determined by the method of alkaline distillation and titration, phosphorus - by spectrophotometer with molybdate, and potassium – by flame emission photometry (Jakovljevic et al., 1985). In the determination of Ca, Mg, investigated trace biogenic elements (Fe, Zn, Cu) and toxic heavy metals (Cd), plant material was converted to a solution by the so-called "dry" combustion, i.e., first by heating at 550°C (for several hours) and then by treating the obtained ash with hydrochloric acid. These elements were determined by AAS method (Miller, 1998).

Statistics

The data shown in Tables are arithmetic means of three replicates of each treatment, namely, of corresponding number of analyzed samples. Standard deviation values are stated with these data in the Tables. Statistical analyses were performed with SPSS version 16 software, 2007.

RESULTS AND DISCUSSION

Chemical Properties of Metallurgical Slag

Properties and composition of metallurgical .slag from Steel factory - Smederevo are shown inTable 1.

Results of laboratory investigations showed that this material has very alkaline reaction (pH =12.50), with the content of Ca in oxide forms (CaO) from 33-45 %, of which about 50 % is easily soluble (in 1 M ammonium acetate). Content of total magnesium is about 0.40 % that was mainly in forms of MgO (0.70%). Total phosphorous contained in the material is about 0.60 % where nearly all the amount was in plant available forms. Content of total iron is expectedly high enough (about 15 %), while the amount of soluble forms is only 0.30 %. The third element (along with Ca and Fe) is Mn, with total amount about 1.8 %, but with low (insignificant) amounts of soluble forms. The studied metallurgical slag contains lower amounts (10-20 mg kg-1) of Zn and a little higher amount of Cu (about 200 mg kg^{-1}). According to previous studies (Yusiharni et al., 2007), metallurgical slag stone (ground steel slag) contains 22-38 % CaO and 3,5-6,5 % MgO. Oxides of calcium and magnesium are partially free, and partially bound to carbonates and silicate that are easily hydrolyzed. Upon the neutralization rate this slag stone material is classified between burned (oxide) slag and ground slag stone (calcium carbonate).

Table 1. *Properties and composition of metallurgical slag (means ± standard deviation).*

Property	Value
pH in H_2O	12.48±0.04
Total content Ca (%)	26.20±3.48
Total content CaO (%)	36.60±4.83
Total content $CaCO_3$ (%)	65.80±8.64
Available Ca (%) (1M amonacetat)	17.18±1.98
Total content of Mg (%)	0.41±0.04
Available Mg (%) (1M amonacetat)	0.07±0.02
Total content P_2O_5 (%)	0.61±0.10
Total content of Fe (%)	15.34±0.79
Available content of Fe(DTPA – mg kg^{-1})	3.38±0.96
Total content of Mn (%)	1.80±0.15
Available content of Mn (DTPA – mg kg^{-1})	3.12±1.04
Total content of Zn (%)	14.60±5.59
Total content of Cu (%)	228.8±15.4

The availability and alkaline nature of some industrial byproduct qualify them as potential alternatives for lime in agriculture and they include metallurgical slag (Das et al., 2006), although limited studies have been carried out on metallurgical steel slag and derivates (Shen et al., 2004). In several researches (Rodriguez et al., 1994; Ali and Shahram, 2007), an increase of pH, exchangeable

Ca and Mg in acid soils by using different doses of metallurgical slag was reported.

Despite the limiting literature on the utilization of processed metallurgical steel slag as lime in agriculture, the results from research conducted have been very encouraging.

Chemical Properties of Soil

In Table 2 the results of soil chemical characteristics and elemental composition of plowed layer of the studied Stagnosol are given.

Table 2. *Chemical characteristics of the studied soil (means ± standard deviation).*

Property	Stagnosol
pH in H_2O	5.48±0.01
pH in 1M KCl	4.45±0.01
The sum of bases -S (cmol kg-1)	11.98±0.95
Potential acidity -Y′	21.66±1.81
CEC (cmol kg-1)	26.06±0.9
Base saturation-V (%)	45.97±2.0
Total N (%)	0.24±0.01
Available P_2O_5 (mg $100g^{-1}$)	3.73±0.28
Available K_2O (mg $100g^{-1}$)	19.8±1.54
Available Ca (mg $100g^{-1}$)	240±19
Available Mg (mg $100g^{-1}$)	35±3.89
Available Fe (mg $100g^{-1}$)	116±5.90
Available Mn (mg $100g^{-1}$)	66±3.00
Available Zn (mg $100g^{-1}$)	1.0±0.1
Available Cu (mg $100g^{-1}$)	1.5±0.1
Available Co (mg $100g^{-1}$)	0.21±0.01

The major factors controlling trace metal concentrations in soil are organic C content, pH, CEC and Fe, Al, Ca, Mg and P concentrations (Chen et al., 1999).
The optimum pH range for growth of most crops in soil is between 5.5 and 7.0, within which most plant nutritive are available (Power and Prasad, 1997). In addition to the aforementioned growth limitations some trace elements may pose a toxicity threat if present at elevated levels as their availability and mobility increases under acidic conditions (Pawlowski, 1997).

The studied Pseudogley had very acid soil reaction, with pH 4.45 in KCl. The soil possessed high potential acidity (Y) and significantly low saturation of CEC (Y =21.7: 11.5; V% = 46:74).

Tested soil had low content of soluble phosphorus and was well supplied with available potassium.Content of soluble calcium is low, while the content of available Mg and microelements are generally within the range of optimal supply.

Experiments in several European countries have demonstrated the ability of metallurgical slag to raise the pH of acid soils, increasing at the same time the Ca and Mg contents of the soils exchange complex. Metallurgical slag use on soils set aside for cereal crops has been studied in previous researches (Coventry et al., 1989), and it has been shown that the slag modified the physical and chemical properties of the soil and lead to an increase in production of between 15 and 40% when 1.6 t ha-1 of metallurgical slag was applied to soils with pH of 4-5.

Chemical Composition of Spinach and Cabbage

Results of the content of main and beneficial biogenic macroelements in aboveground biomass of spinach and cabbage (Table 3) show the significant differences between the treatments, that are due to increased crop yield (their dissolution in plants, especially for some elements: N, K), and due to higher accumulation of some elements and their mobilization from natural soil reserves primarily, as influenced by the additional lime materials. Considering the main goal of this study, the conclusion is that there is a tendency of a little increase of the contents of Ca and P in plants of spinach in thetreatment with metallurgical slag (NPK+manure+metallurgical slag).

The content of biogenic microelements in aboveground biomass of spinach shows that there are significant differences between different treatments (Table 4). The nature of applied upgrading mediums and their concentration have an impact on heavy metal accumulation, their mobility and storing capacity in plant tissues (Riesen and Feller, 2005). It should be noted that there was not found higher accumulation of iron in spinach and cabbage plants in the treatments where metallurgical slag was applied in spite of its significant content in this lime material, except a weak tendency of iron increase in these treatments comparing to those where traditional liming materials (Ca(OH)2 and CaCO3) were applied. The heavy metal concentration in the plant tops is frequently a linear function of total metal concentration in the soil (Chaudri et al., 2001). The content of toxic heavy metals (Cd) in aboveground biomass of spinach were within the allowed concentrations (Kloke et al., 1984) in all the treatments.

Table 3. *Effect of metallurgical slag and selected lime materials use on the content of biogenic macro elements in spinach and cabbage (% of dry mass; means ± standard deviation).*

No. Treatment	N	P	K	Ca	Mg
Spinach					
1.Control	6.26±0.06	1.53±0.02	3.06±0.07	1.27±0.02	0.45±0.03
2.$CaCO_3$	5.67±0.03	1.60±0.02	2.86±0.02	1.25±0.03	0.37±0.01
3.$Ca(OH)_2$	6.18±0.02	1.54±0.02	2.94±0.04	1.25±0.03	0.37±0.01
4.Metallurgical slag	5.64±0.02	1.55±0.03	2.83±0.02	1.24±0.02	0.33±0.04
5.NPK+ Metallurgical slag	6.16±0.02	1.96±0.03	5.16±0.02	1.76±0.02	0.56±0.02
Cabbage					
1.Control	6.78±0.04	1.83±0.01	4.65±0.07	1.74±0.02	0.48±0.02
2.CaCO3	5.93±0.01	1.93±0.01	4.98±0.04	1.70±0.03	0.43±0.01
3.Ca(OH)2	6.33±0.03	1.81±0.03	4.13±0.05	1.63±0.03	0.39±0.03
4.Metallurgical slag	5.61±0.01	1.62±0.02	4.67±0.04	1.61±0.04	0.41±0.01
5.NPK+ Metallurgical slag	6.16±0.02	1.96±0.03	5.16±0.02	1.76±0.02	0.56±0.02

Table 4. *Effect of metallurgical slag and selected lime materials use on the content of biogenic microelements and toxic heavy metals in spinach and cabbage (mg kg^{-1}; means ± standard deviation)*

No. Treatment	Fe	Zn	Cu	Cd
Spinach				
1.Control	12.62±0.25	20.97±0.07	0.13±0.01	0.07±0.007
2.CaCO3	11.25±0.17	20.02±0.03	0.12±0.01	0.06±0.003
3.Ca(OH)2	9.55±0.22	19.80±0.24	0.11±0.02	0.06±0.001
4.Metallurgical slag	12.57±0.10	20.55±0.09	0.13±0.01	0.06±0.002
5.NPK+ Metallurgical slag	12.86±0.11	21.06±0.08	0.10±0.01	0.023±0.002
Cabbage				
1.Control	4.28±0.29	0.42±0.02	0.51±0.01	0.022±0.001
2.CaCO3	4.58±0.23	0.32±0.02	0.49±0.02	0.018±0.003
3.Ca(OH)2	4.81±0.21	0.37±0.01	0.45±0.01	0.021±0.003
4.Metallurgical slag	4.91±0.19	0.31±0.01	0.43±0.02	0.017±0.001
5.NPK+ Metallurgical slag	4.86±0.24	0.36±0.02	0.46±0.02	0.023±0.002

Poniedzialek et al. (1999) found differences between crops in the level of heavy metal accumulation in particular organs. They described that the absorption and transport of metals could be modified by many factors, i.e. cultivar, timing of production and locality. Within the red beet, field pumpkin, chicory, common bean, white cabbage and parsnip the maximum Cd and Pb contents were found in leaves (Sêkara et al., 2005).

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