IMPACTUL ZGURII METALURGICE LF ASUPRA PROCESULUI DE REMEDIERE A SOLURILOR ACIDE ÎN AGRICULTURĂ
IMPACT OF METALLURGICAL LADLE FURNACE SLAG ON ACID SOIL REMEDIATION IN AGRICULTURE

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The paper presents some researches on ladle furnace slag potential to be used in agriculture, as soil amendment. The ladle furnace (LF) slag results from steel secondary refinery. The main components of LF slag used in our experimental trials were: CaO, SiO_2, Al_2O_3, MgO. The LF slag contains high quantity of CaO, which forms different compounds with SiO_2 and Al_2O_3, as well as unreacted CaO too. This is the main reason why this slag can be considered in agriculture as soil amendment to correct the soil acidity. The soil acidity influences the soil fertility and the crop yield. Usually, the soil acidity is corrected by using limestone or dolomite, natural materials. The utilization of LF slag as soil amendment to soil acidity is a novelty in Romania.

It is presented the influence of LF slag admixture on soil remediation, namely on pH, chemical properties and heavy metal content. It is also shown the influence of ladle slag used as soil amendment on the crop yield and on the heavy metal content in the soil and cultivated plants (maize, wheat and soybean) at the experimental sites. The experimental trials indicate that the LF slag can be used for acid soil remediation, having a positive influence on soil chemical properties and increase the crop yield.

Lucrarea prezintă cercetări privind potenţialul utilizării zgurii LF în agricultură, ca amendament al solului. Zgura LF rezultă la rafinarea oţelului in procesele metalurgiei secundare. Principalele componente ale zgurii LF utilizate în studiile noastre experimentale au fost: CaO, SiO_2, Al_2O_3, MgO. Zgura LF conţine o cantitate semnificativă de CaO care formează diferiţi compuşi cu SiO_2 şi Al_2O_3, alături de CaO nereacţionat. Acesta este principalul motiv pentru care această zgură poate fi considerată în agricultură drept amendament al solului in sensul corectării acidităţii solului. Aciditatea solului influenţează fertilitatea acestuia şi producţia culturilor. De obicei, aciditatea solului este corectată prin utilizarea unor materiale naturale, cum ar fi calcarul sau dolomita. Utilizarea zgurii LF ca amendament al solurilor acide este o noutate în România.

Se prezintă influenţa amestecului de zgură LF asupra remedierii solului, şi anume asupra pH-ului, proprietăţilor chimice şi conţinutului de metale grele. De asemenea, este prezentată influenţa utilizării zgurii LF ca amendament al solului asupra producţiei agricole şi asupra conţinutului de metale grele din sol şi din plantele cultivate (porumb, grâu şi soia) pe loturile experimentale. Experimentele au indicat faptul că zgura LF poate fi utilizată pentru remedierea solurilor acide, aceasta având o influenţă benefică atât asupra proprietăţilor chimice ale solului, cât şi asupra creşterii producţiei agricole.

Keywords: *steelmaking, ladle furnace (LF) slag, acid soil, soil amendment*

1. Introduction

Slags are the main by-products generated during iron and crude steel production. The increase of slag recovery and their valorisation in different fields of application, such as in agriculture, allowed to reduce landfills slag and to preserve natural resources, to reduction of CO_2 emissions in cement production, to energy consumption reduction and can lead to a sustainable society founded on recycling practice and environmental friendly. The use of by-products from the iron and steel industry goes back to many centuries ago. In 350 BC, Aristotle already had known the utility of iron slag [1]. In later centuries slag has been used as construction material, cementitious material and/or addition for concrete. The use of steelmaking slag, from Basic-Bessemer or Thomas processes, as phosphating and/or liming agent started in 1880 [2]. Actually, slag coming from both iron and steelmaking processes are consumed at steelworks, used in blended cement production, roadbed material, ground improvement material, civil engineering material and fertilizer and liming material.

Worldwide, it is studied the potential of various steel slag for amending acid soils and to use them as source of important nutrients and growing agents (not only calcium and magnesium compounds, but also other elements such as silicon, providing important beneficial effects for some crops and increasing the plant yields) and its

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use as Fe source for reducing Fe chlorosis in different crops.

Ladle slag (ladle furnace slag – LF slag also) occurs in ladle furnace processing of steel. The ladle furnace is known as a version of electric arc furnace. The addition of silicon and aluminium during deoxidation of steel, leads to silica (SiO_2) and (Al_2O_3) formation and these oxides are later absorbed by the slag generated in the ladle furnace [3]. The generated ladle furnace slag composition is highly dependent on the grade of the produced steel. The most important components of LF slag are: CaO (40-60%), Al_2O_3 (20%), SiO_2 (14%) and MgO (8-12%) [4, 5]. The mineralogical main phases in LF slag are: dicalcium silicate - C_2S ($2CaO·SiO_2$), tricalcium silicate - C_3S ($3CaO·SiO_2$), calcium aluminate-$CaO.Al_2O_3$, free CaO and MgO [3]. Due the high content of free CaO, this slag is very alkaline. LF slag disintegrates into a powder due to instability of the dicalcium silicate, causing an increase of dust emissions to the environment. The most common applications of LF slag are: partial reuse in the steelmaking unit only if the sulphur content of the slag is low, use as substitute for clinker or cement in concrete, as well as for acid mine water treatment, fertilizer in the agriculture and as a chemical trap for CO_2.

As fertilizer in agriculture, the LF slag can improve the soil pH; in addition their siliceous content can improve the soil structure. Negative effects resulting from steel slag uses for soil amelioration, could derive from its heavy metals content, but such metals are bonded in the slag matrix and for this reason they are not available for plants [6,7].

Agricultural soils that are acid, usually have the pH values (in soil solution), less than 7. [8]. Theoretically, soil acidity is largely associated with the presence of hydrogen and aluminium ions in exchangeable forms. The acid soils have low fertility, have poor physical, chemical, and biological properties and seriously affect the crops yield. The soils have a natural buffering capacity, which is dependent on soil physical and chemical properties. This natural buffering capacity of soils can be seriously attenuated when soils are continuously exposed to processes that lead to soil acidification. Soil acidity develops from a combination of natural (weathering and leaching, organic matter decomposition) and anthropogenic processes. The low pH of soil has influence on soil fertility, decreases the availability of essential elements and the activity of soil microorganisms, increases the solubility and negative influence of toxic elements [9]. Soil acidity can cause molybdenum, calcium and magnesium deficiency in plants. The low pH decreases phosphorus availability [9]. Because of the limited land resource, it is necessary a judicious management practice to correct soil acidity and increase the crops yield. One of the most important and particularly feasible management practices in this case is the use of lime and liming materials [10]. Generally, calcite ($CaCO_3$) is used for soil liming but it is expensive and being a natural material it is necessary to save it. From this reason alkaline waste materials are more and more used to ameliorate soil acidity [10, 11].

The aim of the present paper was to investigate the effects of LF slag presence on soil properties, the crops yield and heavy metal content in soil and plants.

2.Materials and methods

For field experiments were collected 6 (six) LF slag samples from the specific storage unit. The slag samples were crushed, grounded, and the rough fraction (>3 mm) was separated. After homogenization, it was obtained, a slag with fine grain size (< 0.09 mm) [4].

The slag was analyzed by ICP-OES (Optic Spectrometry with inductively coupled plasma), for chemical composition. The instrument used was a SPECTROFLAME – ICP model P (Germany). The mineralogical composition of LF slag was determined by the X-ray powder diffraction analysis for the evaluation of the common and predominant phases within the slag, using a BRUCKER D8 ADVANCE difractometer and software package DIFRAC^{plus} BASIC Released 2006 (Brucker) and ICDD PDF-2 database release 2006. The optical microscopy was performed with a Zeiss– Jena– Axio IMAGER Alm microscope.

The leaching tests of LF slag were developed in according with standards and the results were analyzed according to Romanian legislation regarding wastes.

The particle size of soil from the studied area was determined by laser diffraction method using the instrument Laser Particle Size Analysette 22 Compact offering the domain 0.03μ - 300μ. The soil chemical characteristics were determined in agreement with Romanian standards.

3.Experimental

The field experimental studies were carried out in an agricultural area, Moara Domnească (Vlăsia Plain), located in the East side of Bucharest, Romania. In this area the soil is chromic luvisol. The pH of this soil has decreased due to the use of mineral fertilizers with nitrogen content. On the treated soil with LF slag were cultivated maize, wheat and soybean, between years 2013-2015.

On the experimental field, after the autumn tillage, were marked five experimental variants: V1 (reference), V2 (1 t slag/ha), V3 (2 t slag/ha), V4 (3 t slag/ha) and V5 (5 t slag/ha) [12]. The slag was

Table 1

The chemical composition of LF slag (% by weight) / *Compoziţia chimică a zgurii LF(% masă)*

	SiO	CaO	Al_2O_3	Fe_2O_3	MnO	MgO	TiO_2	K_2O	Cr_2O_3	F	P_2O_5	V_2O_5	SO_3	Others
LF slag Zgură LF	19.85	55.12	6.01	1.18	0.89	9.42	0.22	0.031	0.055	4.13	0.034	0.062	1.11	1.89

Table 2

Concentration limits for heavy metals [13, 14] / *Limite de concentraţii pentru metale grele*

Heavy metal *Metal greu*	Concentration of heavy metals / *Concetraţia metalelor grele*		
	Inert waste (RL) *Deşeu inert* (LR)/ L/S=2 l/kg mg/kg	Non hazardous waste (RL) *Deşeu nepericulos*(LR)/ L/S=2 l/kg mg/kg	Leachate *Levigat* L/S=2 l/kg mg/kg
Cu	0.90	25	1.92
Ni	0.20	5.0	2
Zn	2.00	2.0	0.8
Pb	0.20	5.0	1.51
Cd	0.03	0.6	0.46

Table 3

Physical and chemical characteristics of chromic luvisol from experimental sites
Caracteristicile fizice şi chimice ale preluvosolului roşcat din câmpul experimental

Depth *Adâncime* (cm)	Bulk density *Densitate aparentă* (g/cm^3)	Total porosity *Porozitate totală* (%)	Degree of settling *Gradul de tasare* (%)	pH (H_2O)	C_{org} (%)	Humus (%)	Nt (%)	P_{Al} (mg/kg)	K_{Al} (mg/kg)
0-20	1.49	42.42	15.28	5.51	1.21	2.08	0.147	74	230
20-40	1.63	37.19	26.39	5.55	1.23	2.12	0.158	87	235

incorporated in soil and after that maize, wheat and soybean were seeded. The plants were cultivated in the natural climate conditions, without supplying water. In experiments were used seeds: maize hybrid LG 30.489, wheat (Triticum aestivum L-Glosa; a Romanian variety, adapted for climate conditions from this country) and PR92B63 type soybean. Soil samples were collected from all experimental variants from two depths (0-20 cm and 20-40 cm) to follow the influence of slag presence on the soil physical and chemical properties. Before harvesting, samples of maize, wheat and soybean, from all experimental variants, were collected to assess the influence of LF slag presence in soil on the crops yield.

4.Results

The LF slag composition, used in field experiments, determined by the chemical analysis is given in Table 1.

The main components of LF slag used in field experiments are: CaO, SiO_2, Al_2O_3, MgO. The LF slag contains high quantity of CaO, which forms different compounds with SiO_2 and Al_2O_3, but the slag contains CaO free too. Small quantity of iron oxide is present (1.18%). The F (4.13%) is bonded in the slag matrix (Fluorite and Calcium Fluoride Silicate).

The leaching test results show that the LF slag used for field experiments is a non- hazardous waste (according to Ministerial Order no.95/2005 [13]). The values for heavy metals are higher than the values for inert slag but are lower than for non-hazardous waste. In Table 2 are presented the concentration limits for heavy metals for inert and non-hazardous wastes according to Romanian regulations and the previous leaching test results. [14]

The low content of heavy metals in leachate shows that these metals are strongly bonded in the slag matrix.

The XRD analysis results have shown a mixture of crystalline and amorphous phases in LFS. The identified crystalline phases were: Calcium-olivine (45%), Mayenite (12%), Periclase (11%), Calcite (3.5%), Fluorite(6.5%) and Calcium Fluoride Silicate (22%).

The optical microscopy analysis have shown the presence of microcrystalline and vitreous phases as given in Figure 1.

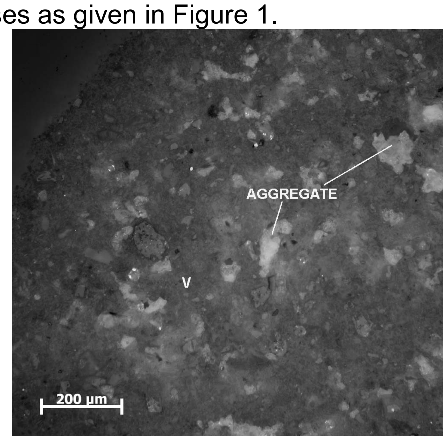

Fig. 1 - LF Slag sample. N+ Reflected light. V- Vitreous phase / *Proba de zgură LF N+ Lumina reflectată. V- fază vitroasă* .

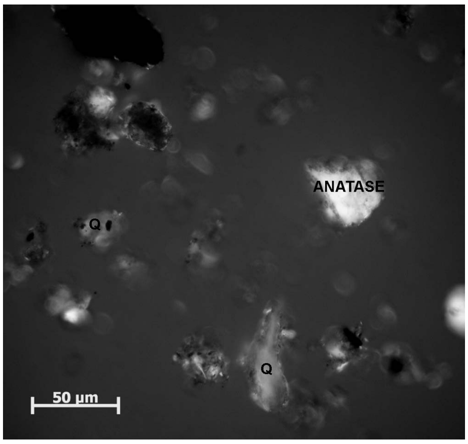

Fig. 2a - Soil sample. N+ Transmitted light. Quartz crystals -Q , Anatase, covered by fine soil particles (micro-crystals) / *Proba de sol. N+ lumină transmisă. Cristale de cuarţ-Q, Anatas, acoperite de particule fine de sol (microcristale).*

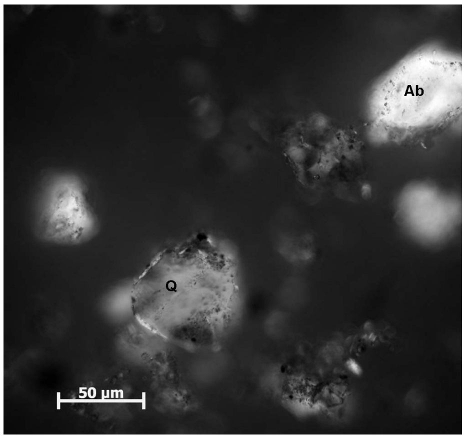

Fig.2b - Soil sample. N+ Transmitted light. Quartz crystals -Q , Albite - Ab, covered by fine soil particles (micro-crystals) / *Proba de sol. N+ lumină transmisă. Cristale de cuarţ-Q, Albit-Ab, acoperite de particule fine de sol (microcristale).*

Table 4a

The influence of LF slag admixture on physical and chemical properties of soil (depth 0-20 cm)
Influenţa prezenţei zgurii LF în sol asupra proprietăţilor fizice şi chimice (adâncime 0-20 cm)

Treatment *Tratament*	Bulk density *Densitate aparentă* (g/cm^3)	Total porosity *Porozitate totală* (%)	Degree of settling *Gradul de tasare* (%)	pH	C_{org} %	N_t %	P mg/kg	K mg/kg
V1(reference *martor*	1.49	42.42	15.28	5.51	1.21	0.147	74	230
V2(1t /ha)	1.44	44.35	11.44	5.68	1.35	0.157	106	225
V3 (2t /ha)	1.58	38.93	22.25	5.80	1.28	0.153	97	298
V4 (3t /ha)	1.49	42.75	14.62	5.78	1.25	0.151	85	299
V5 (5t /ha)	1.55	40.05	20.00	6.01	1.32	0.157	78	311

The soil samples collected from two depths were analyzed to assess the chemical and physical characteristics. The soil has a clay-loam texture. Some physical and chemical characteristics of soil samples from the experimental sites are presented in Table 3 (depth: 0-20 and 20-40 cm).

The data presented in Table 3 suggest that the soil from the selected area has the pH= 5.51 corresponding to a medium acid soil, which does not sustain a high fertility and is not able to offer proper conditions for crops. The low pH of soil has influence on soil fertility, decreases the availability of essential elements and the activity of soil microorganisms, can determine calcium and magnesium deficiency in plants and decreases phosphorus availability.

The optical microscopy analysis show that the soil samples contain quartz and albite micro-crystals (Fig. 2a and Fig. 2b). The samples contain amorphous iron oxy-hydroxides (limonite) too. The crystals are covered by very fine soil particles (probably microcrystalline clay minerals, iron oxides and mica).

The XRD analysis identified the following crystalline phases: Quartz, Anatase, Muscovite, Albite and Montmorillonite, being in good agreement with the optical microscopy analysis results.

The influence of presence of LF slag on soil chemical properties is presented in Table 4a and Table 4b, for two depths and different LF slag quantities.

The data given in Table 4a (depth 0-20 cm) and Table 4b (depth 20-40 cm) show that the admixture of LF slag in soil improves the soil pH. The soil pH increases with the increase of the added slag quantities. The pH increases, after LF slag application at the depth 0-20 cm, from 5.51 in the reference variant (V1) to 6.01 in case of V5. At depth 20-40 cm the pH increases from 5.55 in the reference variant (V1) to 6.35 when 5t/ha slag (V5) was applied.

The presence of LF slag in soil improves the soil chemical characteristics too. At depth 0-20 cm the organic carbon, total nitrogen and mobile potassium are increased with increasing the LF slag quantities. The highest content of phosphorus was achieved at both depths, when the soil was treated with 1 t/ha LF slag (V2). The presence of LF slag in soil influences the soil physical properties too.

Table 4b

The influence of LF slag admixture on physical and chemical properties of soil (depth 20-40 cm)
Influenţa prezenţei zgurii LF în sol asupra proprietăţilor fizice şi chimice (adâncime 20-40 cm)

Treatment *Tratament*	Bulk density *Densitate aparentă* (g/cm^3)	Total porosity *Porozitate totală* (%)	Degree of settling *Gradul de tasare* (%)	pH	C_{org} %	N_t %	P mg/kg	K mg/kg
V1 (reference *martor*)	1.63	37.19	26.39	5.55	1.23	0.158	87	235
V2(1t /ha)	1.59	38.94	23.27	5.77	1.31	0.153	108	270
V3 (2t /ha)	1.59	38.59	23.62	5.92	1.23	0.155	105	287
V4 (3t /ha)	1.64	36.76	27.24	5.95	1.21	0.157	87	326
V5 (5t /ha)	1.63	37.10	26.58	6.35	1.25	0.132	89	317

Table 5

The influence of LF slag admixture in soil on maize, wheat and soybean yields
Influenţa prezenţei zgurii LF în sol asupra producţiei de porumb, grâu şi soia

Treatment *Tratament*	Maize *Porumb* t/ha	Wheat *Grâu* t/ha	Soybean *Soia* t/ha
V1(reference *martor*)	7.215	4.63	2.415
V2(1t /ha)	7.628	4.98	2.656
V3 (2t /ha)	7.780	5.28	3.014
V4 (3t /ha)	7.845	6.07	3.118
V5 (5t /ha)	7.966	6.32	2.876

Table 6

Heavy metals content of the soil samples from Moara Domnească before LF slag use (depth: 0-20 and 20-40 cm) [15, 16]
Conţinutul de metale grele al solului de la Moara Domnească înainte de tratament (adâncime 0-20 și 20-40 cm)

Heavy metals *Metale grele*	**Cd**	**Co**	**Cu**	**Ni**	**Mn**	**Pb**	**Zn**
	$mg \cdot kg^{-1}$						
Soil samples Moara Domnească, Depth 0-20cm *Probe de sol Moara Domnească, adâncime 0-20 cm*	0.66	14.3	27.1	38.0	797	22.6	66
Soil samples Moara Domnească, Depth 20-40cm *Probe de sol Moara Domnească, adâncime 20-40 cm*	0.54	15.2	25.2	43.3	761	21.9	66
Order 756/1997, Alert Limit – for agricultural soils *Ordinul 756/1997 Pragul de alertă- utilizare sensibilă*	3	30	100	75	1500	50	300
Maximum Admissible Limit (MAL) for soil Order 344/2004 *Limita Maximă Admisă (LMA) pentru sol Ordinul 344/2004*	3	-	100	50	-	50	300

On the treated soil with different quantities of LF slag admixtures were cultivated maize, wheat and soybean in 2013-2015 years climate conditions. In Table 5 is presented the variation of maize, wheat and soybean yield with the LF slag quantities applied on soil.

The data from Table 5 show that in case of maize and wheat the crop yield increases with the LF slag quantities admixed in soil. The highest yield was obtained when was used 5t/ha LF slag (V5) for soil treatment. In the case of soybean the highest yield was obtained when was used for treatment 3t/ha LF slag (V4).

The LF slag used for acid soil treatment, as was previously presented, can be classified, in agreement with Romania regulations, as a non-hazardous waste. Although in the field experimental work at Moara Domnească, can be noticed a positive influence of the LF steel slag on soil properties and crop yield, but this aspects should be assessed in conjunction with their environmental influence, mainly the soil heavy metal content. For this reason soil samples, from the experimental field were analyzed to assess the heavy metal content before and after LF slag application. The results of ICP analysis were compared with Romanian legislation regarding to the soil pollution with heavy metals (Order 756/1997 and Order 344/2004). The results obtained for heavy metal concentration in soil samples before LF slag use in comparison with Romanian legislation, are presented in Table 6.

From Table 6 results that the soil from Moara Domneasca is not polluted with heavy metals, and can be used in agriculture for crops.

In Table 7 are presented the heavy metal concentration in soil samples after the admixture of different quantities of LF slag.

The use of LF slag for acid soil treatment does not influence the heavy metals accumulation in soil, even at 5 t/ha the heavy metal concentration in soil was in national limits. These

Table 7

Heavy metal concentration in soil after treatment with LF slag (depth 0-20 and 20-40 cm)
Concentraţia de metale grele în sol după tratament cu zgura LF (adâncime 0-20 şi 20-40 cm

Heavy metals *Metale grele*	**Cd**		**Co**		**Cu**		**Ni**		**Mn**		**Pb**		**Zn**	
	$mg \cdot kg^{-1}$													
Depth, cm *Adâncime,* cm	0-20	20-40	0-20	20-40	0-20	20-40	0-20	20-40	0-20	20-40	0-20	20-40	0-20	20-40
V1(reference *martor*)	0.66	0.54	14.3	15.2	27.1	25.2	38.0	43.3	797	761	22.6	21.9	66	66
V2(1t /ha)	0.66	0.62	16	16.5	26.2	26.4	36.0	36.4	816	785	21.0	22.3	66	68
V3 (2t /ha)	0.65	0.64	16.3	13.0	26.0	25.8	37.8	35.8	778	805	20.5	19.4	68	69
V4 (3t /ha)	0.7	0.52	14.0	13.8	25.2	25.8	37.6	34.7	780	792	21.5	19.1	67	69
V5 (5t /ha)	0.61	0.49	10.5	14.3	25.8	24.6	35.0	34.9	816	820	20.4	17.4	65	66

Table 8

The heavy metal content of wheat and soybean (p.p.m.)
Conţinutul de metale grele în grâu şi soia (p.p.m.)

Treatment/Heavy metals *Tratament/Metale grele*	Wheat *Grâu*			Soybean *Soia*		
	Pb	Cu	Zn	Pb	Cu	Zn
V1 (reference *martor*)	<2	6.5	34	02	13.6	55
V2(1t /ha)	<2	5.7	28	0.3	11.8	50
V3 (2t /ha)	<2	7.0	34	0.28	13.6	54
V4 (3t /ha)	<2	5.7	35	0.26	13.8	54
V5 (5t /ha)	<2	5.0	25	0.25	11.6	51

data confirms the results obtained with a portable XRF instrument and presented in a previous paper [17]

In the field experiments it was studied, the influences of LF slag admixtures on cultivated plants capacity to uptake heavy metals from soil too. For this reason, before the harvest, was collected wheat, soybean plant samples. The heavy metal concentration in wheat and soybean is presented in Table 8.

The low content of copper in wheat from site V5 (soil treated with 5 t slag/ha) is explained by the presence of a high content of LF slag in soil. In this case the soil pH at depth 0-20 cm has the highest value pH=6.01. It is known [1] that the Cu stability in soil is pH dependent, its mobility decreases with increasing pH. The lead solubility in clay-loam soils decreases with pH increasing [18]. The clay minerals have a good affinity to adsorb Pb cations and reduce its mobility in soil solution and availability for plants. The present of Zn in wheat and soybean is in normal limits for usual wheat [19] and soybean species.

5.Conclusions

It was studied the possibility to use LF slag from the steel refining for acid soil remediation. The slag chemical, mineralogical compositions were determined. It was made leaching tests too. The results of leaching tests were compared with the limits for heavy metals from Romanian legislation. The LF slag is a non-hazardous waste according with national laws.

The acid soil (chromic luvisol) chemical, physical and mineralogical characteristics were determined. The initial pH of soil was 5.51. The field experiments were carried out in an experimental site from Moara Domnească, located in the east side of Bucharest, Romania, in Vlăsia Plain.

Different quantities of LF slag were incorporated in acid soil, to improve the soil pH and chemical characteristics. On the treated soil were cultivated: maize hybrid LG 30.489, wheat (*Triticum aestivum* L-Glosa; a Romanian variety, adapted for climate conditions from this country) and PR92B63 type soybean. It was studied the influence of LF slag admixtures in acid soil on its physical, chemical characteristics, as well as on the crops yield and on the heavy metal content in the soil samples and plants.

The presence of LF slag in acid soil improves the soil chemical characteristics (increase the soil pH to 6.01) and increases the crop yield. The crop yields increase registered on chromic luvisol under natural climate conditions were significant due to the better uptake of nutrients. The highest yields were achieved when the soil was treated with 5 t slag/ha, in case of maize and wheat. In case of soybean the highest yield was obtained when the soil was treated with 3t/ha LF slag. The presence of LF slag in soil, does not influence the natural content of soil.

The results highlighted that the use of LF slag for acid soil remediation is effective, having a positive influence on soil characteristics, leading to increased crop yield.

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