STABILIZAREA ZGURILOR METALURGICE (LF) PENTRU UTILIZAREA ÎN AGRICULTURA SUSTENABILĂ

STABILISATION OF THE METALLURGICAL LADLE FURNACE SLAGS (LF) FOR APPLICATION IN SUSTAINABLE AGRICULTURE

LAVINIA GABRIELA POPESCU [1], ENIKÖ VOLCEANOV[1,2 *], ILDIKO ANGER [3]
ALEXANDRA GEORGIANA MOISE [3], ADRIAN VOLCEANOV [2], ECATERINA MATEI [2]
[1] Institute for Metallurgical Research, 39 Mehadiei street, Bucharest
[2] University POLITEHNICA of Bucharest, 313 Splaiul Independentei
[3] National R&D Institute for Nonferrous and Rare Metals, 102 Biruintei Blvd, Pantelimon, Ilfov

Present paper presents a comparative assessment of environmental factors by using an industrial waste from secondary metallurgy, such as white slag. LF slag generated at two sources as powder and subsequently as pelletized form were investigated. The chemical composition of the investigated slags were analysed by x-Ray Fluorescence (XRF).

Leachability tests and the chemical analysis were carried out by various methods. Consequently, the chemical composition of the leachate for various particle size fractions of the waste LF slag was determined by using flame atomic absorption spectrometry (FAAS) and by induction coupled plasma optical emission spectrometry (ICP-OES) to determine the content of heavy metals - Ni, Cu, Pb, Zn, Cd, brought by slag.

The values of pH and electrical conductivity on leachate samples, prepared with solid / liquid ratio of 1:2 and 1:10 were also measured.

The advantage of LF slag using as pellets consist in decrease the LF slag instability, comparatively with the initial powdered slag, which leads to dispersion as dust in the environment. This is possible by processing the slag to an optimal grain size distribution in conjunction with physical and chemical characteristics to obtain a homogeneous product. Their use as acid soil amendment in agriculture is effective.

Lucrarea prezintă o evaluare comparativă a factorilor de mediu la utilizarea unui deşeu industrial – zgura albă provenită din metalurgia secundară. Au fost investigate zguri LF din două surse sub formă de pulbere, prelucrate ulterior sub formă de pelete.

Compoziţia chimică a zgurilor evaluate a fost determinată prin spectrometrie de fluorescenţă cu raze X (XRF).

Au fost efectuate teste de levigabilitate şi analize ale compoziţiei chimice prin diverse metode. Astfel, compoziţia chimică a levigatului rezultat pentru diferite fracţii granulometrice ale deşeului de zgură LF a fost determinată cu ajutorul spectrometriei de absorbţie atomică în flacără (FAAS) și prin spectrometrie de emisie optică în plasmă cuplată inductiv (ICP-OES), în special pentru determinarea conţinutului de metale grele, antrenate din zgură.

De asemenea, a fost investigată evoluţia pH-ului şi conductibilităţii electrice pe probele de levigat, preparate la raportul solid / lichid de 1:2 și 1:10.

Avantajul folosirii zgurilor LF sub formă de pelete constă în reducerea instabilităţii zgurii LF, spre deosebire de zgura iniţială care are caracter prăfos, fapt ce duce la dispersia prafului în mediul înconjurător. Acest lucru este posibil prin prelucrarea zgurii la o granulaţie optimă, care conduce la obţinerea unui produs mai omogen şi din punct de vedere al caracteristicilor fizico-chimice. Acest lucru permite utilizarea efectivă ca amendament al solurilor acide în agricultură.

Keywords: *pellets, LF slag, leachability, acid soil amendment, agriculture*

1. Introduction

LF slag (ladle furnace slag) is a secondary metallurgy slag resulting by the refining of steel slag generated in the different primary unit (electric arc furnace).

The most usual applications of dusty LF slag are currently focused on the following areas [1, 2]:

- reintroduction into the steelmaking units: electric furnace slag frothing, converter with oxygen blast-furnace desulfurization or ladle [3 - 7];
- use as addition of the clinker in cement industry [8 - 18];
- used for acid mine water treatment [19];
- use as a fertilizer in the agriculture, because of trace elements in slag, which may act as micronutrients [20 - 22], or a neutralizer for the acidic soils [20, 21];
- use as a possible trap for chemical sequestration of CO_2, the LF slag having the potential 14 folds greater than the classical slag [20].

As regarding the uses of LF slag, in powder form, as a fertilizer in agriculture, or as a neutralizer for acidic soils. Our preliminary tests have been shown that it can get an increase in soil pH from 5 to over 8, using only 1% slag LF [2].

* Autor corespondent/*Corresponding author*,
E-mail: evolceanov@gmail.com.

LF dusty slag shows as inconvenience during application the fact that it can be dispersed by the wind, and thus controlled release of slag micronutrients in the soil is affected. Due to this fact, it is necessary a LF slag processing by agglomeration, in order to stabilize it, using the method of pelletization.

In this context, our work deals with a comparative analysis of LF slag leachability – in powder form, and of pellets and the pH and electrical conductivity values for the leachate were analyzed.

2. Experimental Procedures

2.1. Materials

For experiments two sources of slag were identified. They come from secondary metallurgy steel, suitable to make possible the development of a mixed type acidic soils amendment to replace the natural materials used today:

- steel producer for the energy industry – P1 slag;
- medium - alloy steel manufacturer for rolling stocks – P2 slag.

The chemical composition of the layers was determined by X-ray Fluorescence (XRF) with Panalytical AXIOS X-ray fluorescence spectrometer operating at 60 KV and current of up to 160 mA with dual multi-channel analyser (DMCA) and commercial software Super Q, analytical programs (IQ +, WROXI).

The CaO_{free} was determined according to ASTM E 1621-13 by wet chemistry.

Phase analyses re done by X-ray diffraction, in parallel beam, scan axis 2Θ/Θ on powder samples, using SHIMADZU XRD 6000 diffractometer with CuK_{α} radiation.

The pH of leachates was measured against time, using the HACH sensION156 portable pH/conductivity meter. The meter can measure absolute mill volts (mV). Absolute mill volts are displayed with 0.1 mV resolution in the range of -2000 to 2000.

Characteristics of these slags are discussed as follows.

The grain size distributions of slags powder are presented in Table 1. It is to be noticed the advanced fineness of both LF slags, meaning that 71.3 % is under 0.044 mm for P1 slag and 81.6 % for P2 slag, respectively.

Chemical composition

The XRF analyses determined on typical LF slags from various Romanian steel producers are presented in Table 2.

The most significant oxide components of the LF slag are CaO (usually 50 – 62.5%), Al_2O_3 (4-8%), SiO_2 (12- 24%) and MgO (7- 15%). LF slag generated in the furnace has a lower content of Fe_2O_3 and higher of Al_2O_3.

The results of analyzed ladle slag has showed that free CaO contents were from < 0.52 to 4.60 %, determined by wet chemistry [1].

Mineralogical composition

The analyses of the mineralogical phases determined through X-ray diffraction method confirm the results obtained by chemical composition determination, emphasizing as crystalline constituents, the typical oxide compound expected for this class of materials, such as: γ-calcium silicate $Ca_2(SiO_4)$ calcio-olivine; mayenite - $12CaO.7Al_2O_3$ with fluor inclusion like $Ca_{12}Al_{14}O_{32}(O,F_2)$, periclase MgO, calcite $CaCO_3$, fluorite CaF_2 and calcium fluoride silicate $Ca_{6-0.5x}Si_2O_{10-x}F_x$.

The dominant compound is the γ- calcium silicate $Ca_2(SiO_4)$, about 45% for the slag P1 and 39.8% for the slag P2, as shown in the X-ray

Table 1

The grain size distribution of LF powder form

Distribuţia granulometrică a probelor de zgură LF sub formă pulverulentă

processed amount = 100 g / sample *cantitate prelucrată = 100 g / probă*	Grain size distribution *Distribuţie granulometrică* Rest (%) on sieve mesh size (mm) *Rest (%) pe sita cu ochiurile (mm)*				
	0.2	0.09	0.063	0.044	< 0.044
P1	12.6	2.2	13.2	0.7	71.3
P2	0.7	1.2	15.3	1.2	81.6

Table 2

Chemical composition of typical LF slags produced in Romania

Compoziția chimică tipică a zgurilor LF produse în România

Sample	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	MnO	K_2O	P_2O_5	F
A	22.44	0.24	5.45	1.62	8.12	56.64	0.82	0.02	0.01	3.20
B	16.85	0.22	6.85	0.95	14.85	50.47	1.41	0.03	0.06	7.20
C	23.54	0.21	7.40	1.45	7.45	55.85	0.31	0.04	0.01	2.20
D	12.90	0.21	4.10	0.55	9.75	62.52	1.62	0.04	0.06	5.14

Compound Name	Formula	S-Q	SS-VVV-PPPP
γ-Ca2SiO4, Calcio-olivine	$Ca_2(SiO_4)$	45	01-080-0941 (A)
Calcium Aluminum Oxide Fluoride (~Mayenite)	$Ca_{12}Al_{14}O_{32}(O,F_2)$	11.5	00-053-1232 (*)
Periclase	MgO	9.1	01-071-3631 (*)
Calcite	$CaCO_3$	2.8	00-005-0586 (*)
Fluorite	CaF_2	7	00-035-0816 (*)
Calcium Fluoride Silicate	$Ca_{6-0.5x}Si_2O_{10-x}F_x$	24.6	00-038-0627 (C)

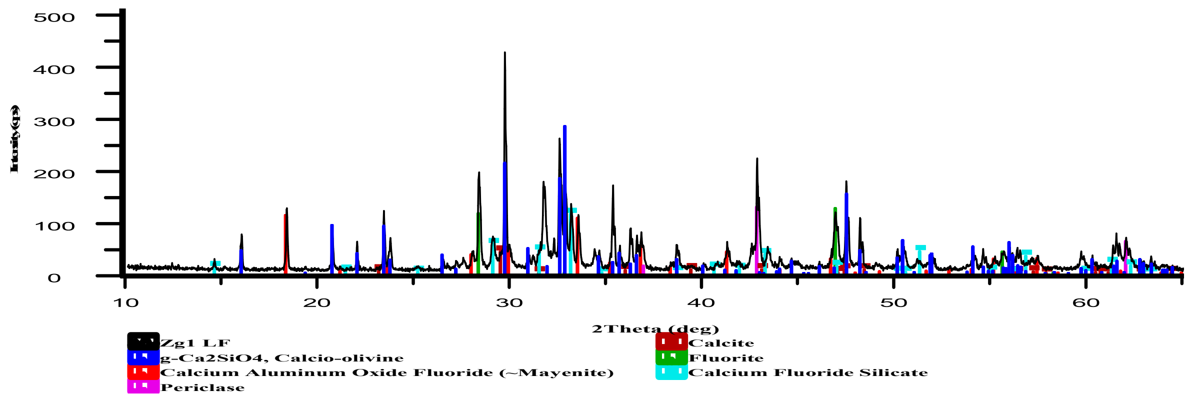


Fig. 1 - X- ray diffraction pattern of the P1 slag/ *Difractograma de raze X a zgurii P1*.

Compound Name	Formula	S-Q	SS-VVV-PPPP
γ--Ca_2SiO_4, Calcio-olivine	$Ca_2(SiO_4)$	39.8	01-080-0941 (A)
Calcium Aluminum Oxide Fluoride (~Mayenite)	$Ca_{12}Al_{14}O_{32}(O,F_2)$	3.7	00-053-1232 (*)
Periclase	MgO	13.8	01-071-3631 (*)
Calcite	$CaCO_3$	2.4	00-005-0586 (*)
Fluorite	CaF_2	11.3	00-035-0816 (*)
Calcium Fluoride Silicate	$Ca_{6-0.5x}Si_2O_{10-x}F_x$	29	00-038-0627 (C)

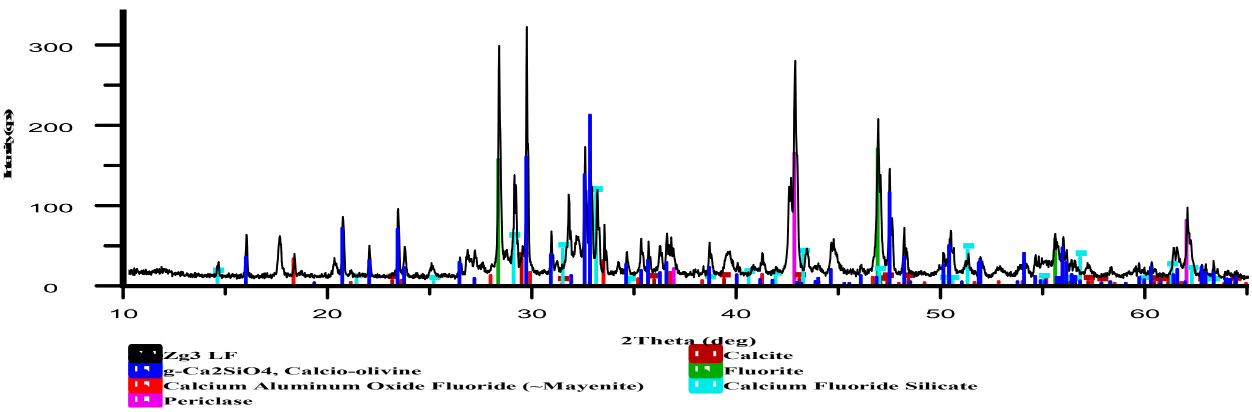


Fig. 2 - X- ray diffraction pattern of the P2 slag/ *Difractograma de raze X a zgurii P2*.

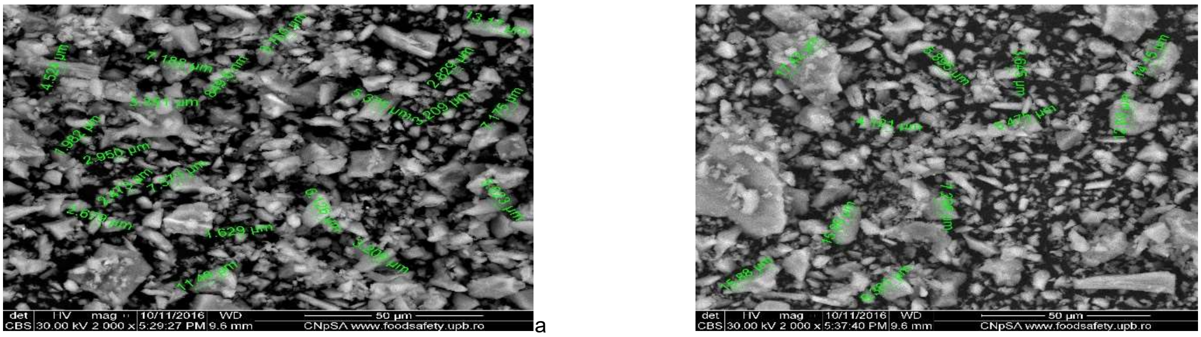
pattern given in Figure 1 for the P1 slag and respectively in Figure 2 for the P2 slag.

The currently appearance of LF dusty slag obtained in the steel industry in Romania is shown in Figure 3.

Fig.3 **-** Aspect of a representative LF slag sample /*Aspectul unei mostre reprezentative de zgură LF* .

2.1.1. Microstructure

The scanning electron microscopy (SEM) images of P1 slag and P2 slag are given in Figure 4 (a, b).



a b

Fig.4 - SEM image of LF slags / *Aspectul unei mostre reprezentative de zgură LF* (a) P1 slag; (b) P2 slag

2.2. Procedures for obtaining LF slag in pellets form

Pelletization of the two slag samples was performed at laboratory scale by using a horizontal drum, with the following technological parameters:

Drum speed = 40 rpm;
The feeding rate of drum = 0.2 kg / min;
Initial humidity = 1.05%;
Final humidity of pellet = 11.92% for sample no. 1
Final humidity of pellets = 12.56% for sample no. 2.

The amount of water used in pelletizing process ranged between 14-17%. The laboratory drum productivity was 32 kg/h. The total amount of water absorbed during the pelletization was 0.569 g $/g_{pellet}$ for the P1 slag, respectively for the P2 slag was 0.564 $g_r/g_{pellet.}$

The duration of saturation of water was 16.6 min for P1 slag, respectively 14.6 min for P2 slag.

2.2.1. Pellets characteristics

The diameters of pellets were made using a laser granulometer type Mastersyzer 2000. Thus, for P1 slag, D^{100}_{max} = 0.032 mm and for P2, D^{100}_{max} = 0.041 mm were determined. The chemical composition of LF slag in pellet form as analyzed by XRF method, is shown in Table 3.

The results have showed, for both slags, a pronounced basic character; CaO/SiO_2 ratio = 2.5.....3.0 and useful micro-nutrients for plants such as: Fe, Mn, Cr, P.

The appearance of the material obtained in pellets form, is illustrated in Figure 5.

2.2.2. Levigability analysis of slags LF

To assess soluble fraction of wastes, its quality and leaching behavior of polluting elements from the waste, the levigability test was performed. LF slag samples in powdery and pelletized form were analyzed.

The procedure for leachability analysis consists in extraction of metals, metal compounds or a material with a solvent (normal water, acidic or alkaline water) which crosses the crumbled material.

The pelletized material was sieved and the grain size with the fraction of 0.2 - 0.09 mm has been retained to leachability test, comparatively with the non-pelletised LF slag sample. In such circumstances, the test leads to the identification of those components (especially heavy metals) that can be taken off by repeated washing of water by the analyzed material.

Table 3

The chemical composition of the LF slags in the pellet form (%) / *Compoziţia chimică a zgurilor LF sub formă de pelete (%)*

Proba Sample	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	SO_3	V_2O_5	Cr_2O_3	IL*/ PC
P1	16.72	0.19	4.37	29.50	0.53	5.97	41.63	0.01	0.01	0.03	0.18	0.01	0.06	0.74
P2	15.50	0.23	8.19	8.21	1.32	12.82	46.20	0.01	0.02	0.06	0.30	0.01	0.08	6.90

IL- Ignition loss/ *PC Pierdere la calcinare*

Sample of the P1 slag / *Proba de zgură P1*
Apparent density = 1.055 g / cm^3 on dried sample
Densitate aparentă = 1,055 g/cm^3 în stare uscată

Sample of the P2 slag / *Proba de zgură P2*
Apparent density = 0.99 g / cm^3 on dried sample
Densitate aparentă = 0,99 g/cm^3 în stare uscată

Fig.5- Aspect of the pelletized LF slags / *Aspectul zgurilor LF peletizate*

The leachability tests are based on the extraction method which involves stirring for 24 hours a sample of solid material in a volume of leaching agent, at a ½ solid/liquid extraction ratio.

It was used a ratio of liquid/solid (l/s) – 2:1 (or 10:1) / kg dry material and distilled water as leaching agent was used.

After leaching and filtering, the leachate was analyzed by atomic absorption spectrometry to determine the content of heavy metals - Ni, Cu, Pb, Zn, Cd.

Also, after filtering the suspension, the material was dried in oven to constant mass and analyzed by XRF method.

The evolution of pH and electrical conductivity for the leached LF slag was investigated by using a multi-parameter portable Hach sension^{TM}156.

3. Results and discussions

The chemical composition of LF slag after leaching for the 1:2 and 1:10 ratio is shown in Table 4a for the non-pelletized slag samples and in Table 4b for pelletized ones, respectively.

The results of the leaching determinations are interpreted in relation with the "*Order no. 95/2005, of the* Ministry of the Environment, *establishing acceptance criteria and preliminary procedures for the acceptance of waste storage and the national list of waste accepted in each class of landfill*".

The data obtained by this method could be compared with the regulations of the Ministry of the Environment (ME) no. 95/2005 and show that this type of slag does not fall in the category of "inert" waste, because a content of Cd > 0.04 mg/kg dried material and Pb > 0.5 mg kg dried material, comply with the category of "non hazardous" waste.

Additionally to the requirements of Romanian regulations the content of Fe in leachate was determined, as the iron is an important fixing element in plant tissues. It was noticed the lack of Fe in leachate.

It is of vital importance to be familiar with the technical significance of the secondary application of waste materials, but also with their possible environmental effects because some waste materials might contain increased concentrations of substances harmful to human health or the environment, especially to the water. The environmental conformity of the ladle furnace slags has been investigated for years, which is normally to be judged by the leachability of the slags. Due to the very low solubility of the most mineral phases of the LF slags in water, the LF slags do not affect the environment.

In Table 5 is shown the oxide composition (determined by XRF method) for LF slag pelletized samples of dry residue after leaching.

Levels of only 4 - 6 mS / cm for the electrical conductivity, for the highest solid/liquid ratio at this stage represent a good premise for LF slag usage in the process of acid soil improvement.

XRF analyses performed on pelletized LF slag samples after the leachability test on the filtered and then dried material showed a ratio CaO/SiO_2= 2.27 for the P1 sample at a dilution of 1:2 and CaO/SiO_2= 2.43 at 1:10 dilution, respectively. For the sample P2 of pelletized LF slag, the ratio CaO/SiO_2 was 2.94 for a dilution to 1:2, and CaO/SiO_2= 2.93 at 1:10 dilution, respectively.

Table 4*a*

The results of leachability test for initial samples (nonpelletized) determined by FAAS
Date de levigare pentru probele inițiale (nepeletizate) de zgură LF determinate prin FAAS

Sample / s/l ratio / *Probă / raport s/l*	Cu		Ni		Zn		Pb		Cd	
	mg/l	mg/ kg	mg/l	mg/kg	mg/l	mg/kg	mg/l	mg/ kg	mg/l	mg/ kg
P1 /1:2	0.096	1.92	0	0	0.01	0.2	0.064	1.28	0.01	0.2
P1 /1:10	0.079	0.79	0	0	0	0	0.151	1.51	0	0
P2 /1:2	0.062	1.24	0.1	2	0,02	0.4	0,013	0.26	0.023	0.46
P2 /1:10	0.071	0.71	0	0	0	0	0.054	0.54	0	0

For a ratio of 1:2. 100 g were dissolved in 200 ml distilled water / *Pentru raport 1:2 au fost dizolvate 100 g la 200 ml apă distilată*
For a ratio of 1:10, 10 g were dissolved in 100 ml distilled water / *Pentru raport 1:10 au fost dizolvate 10 g la 100 ml apă distilată*

Table 4b

The results of leachability test for pelletized samples determined by FAAS
Date de levigare pentru probele peletizate de zgură LF determinate prin FAAS

Sample / s/l ratio / *Probă / raport s/l*	Cr_{total}	Ni	Zn	Pb	Cd
	mg/l	mg/l	mg/l	mg/l	mg/l
P1 /1:2	SLD	SLD	0.100	0.135	0.004
P1 /1:10	SLD	SLD	0.100	0.229	0.003
P2 /1:2	ND	ND	0.097	0.167	0.002
P2 /1:10	ND	ND	0.97	0.195	0.001

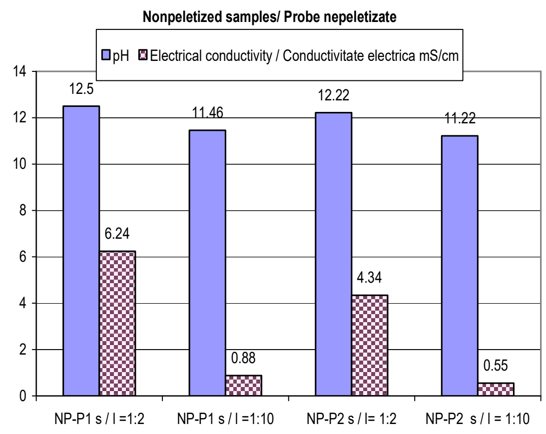
SLD= bellow the FAAS detection limit /*sub limita de detecție FAAS, Ni: 0.37 mg/l, Cr_{tot}: 0.28 mg/l*
ND= non-detectable/ *nedetectabil*

Table 5

Oxide compositions (determined by XRF method) for LF slag pelletized samples of dry residue after leaching / *Compoziţii oxidice (determinate prin metoda XRF) pentru probele peletizate de zgură LF sub formă de reziduu uscat după levigare*

Sample / s/l ratio / *Probă / Raport s/l*	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O
P1 /1:2	16.77	0.19	4.44	15.00	0.40	5.43	38.08	0.04	0.01
P1 /1:10	15.09	0.18	3.30	21.12	0.44	5.38	36.67	0.01	0.01
P2 /1:2	13.35	0.21	6.60	15.17	1.16	11.02	39.30	0.02	0.01
P2 /1:10	14.95	0.24	7.19	8.90	1.24	12.25	43.80	0.03	0.01

Sample / s/l ratio / *Probă / Raport s/l*	P_2O_5	SO_3	PC	CuO	NiO	ZnO	PbO	Cr_2O_3
P1 /1:2	0.02	1.19	18.77	0.127	0.010	0.007	na	0.041
P1 /1:10	0.02	1.20	16.64	0.042	0.024	0.008	na	0.052
P2 /1:2	0.06	0.58	12.43	0.120	0.013	0.019	na	0.101
P2 /1:10	0.06	0.80	10.58	0.082	na	na	0.003	0.091



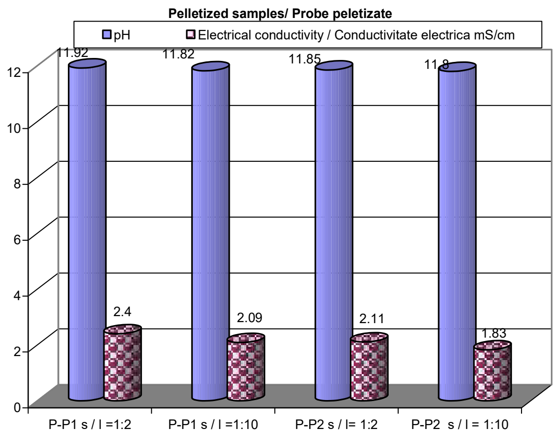


Fig. 6 - pH and electrical conductivity values for leachate samples from : a) non-pelletized, b) pelletized LF slag /*Valori ale pH-ului şi conductivităţii electrice pentru levigate provenite din a) probele nepeletizate b) peletizate de zgură LF.*

The data highlight a massive decrease of CaO content in dry residues after leaching in both samples, which supports its leaching and utility in neutralizing of acidic environments. Also, on can notice a decrease of CaO/SiO_2 ratio for both samples and dilutions compared to the same ratio for the initial pellets, ranging from 2.5 to 3 (see Table 2).

As the slag LF will be used as amendment for acid soils, the pH and electrical conductivity of the leachate was measured.

Figure 6 (a, b) presents the values of pH and electrical conductivity for leaching liquids from non-pelletized, and pelletized LF slag, respectively.

From Fig. 6 a, b results that the pH values of the non-pelletized LF slag samples are in the range of 12.50 to 11.22 and LF slag samples pelletized registers a slight decrease of pH between 11.92 and 11.80. It can be noticed that for the nonpelletized sample, the pH values are higher comparatively with the pelletized samples for the same solid/liquid ratio.

Regarding the electrical conductivity of the non-pelletized LF slag samples, it belongs to the range 6.24 - 0.55 mS/cm, while for the pelletized LF slag samples it registers a decrease of their conductivity at values between 2.40 -1.83 mS /cm. Consequently, we can conclude that the release of various ions in distilled water is slower in case of the pelletized samples. The electrical conductivity of leachate (shows actually the salinity level) must be interpreted in this case as the risk of salinization of soil where LF slag will be used, rainfall regime / vegetation cycle and hence, the choosing of plants for which the salinization effect will be the as low as possible.

4. Conclusions

LF slag is a waste occurring in secondary metallurgy, resulting from the refining of steel, with a profound dusty feature that has no applications in constructions. It is generally deposited in landfills, but it has a high improvement potential for acid soils.

Regarding the pelletized LF slag used in experimental works it can be claim that the leaching in the ground is harder and its grain size has no negative implications on soil and plants.

The behavior of the LF slag submitted to leachability tests revealed a small variation of the pH, while the electrical conductivity showed a significant reduction which emphasize the salts content in the leachate of pelletized samples and consequently, allowing a controlled release of them in acid soils.

The advantage of LF slag use as pellets consist in diminishing of the LF slag instability, comparatively with the initial powdered slag, which leads to dispersion of dust in the environment. The pelletisation implies agglomeration of the slag to an optimal grain size distribution having as consequence, a more homogeneous product, concerning also, physical and chemical characteristics. Their use as acid soil amendment in agriculture is effective.

The use of steel slags in agriculture produces not only economic but also ecological advantages. A more effective exploitation of natural resources can be achieved in both the steelmaking processes and in the agriculture as acid soil amendment which obviously can supply nutrients in soil and consequently to be used also as fertilizer, but should not have negative effects on the environment.

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