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Waste Materials in Construction: Putting Theory into Practice

ASSESSMENT OF CHEMICAL SENSITIVITY OF WAELZ SLAG

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1. Abstract

In the recycling industry, the recuperation of zinc from Electric Arc Furnace dust by the Waelz process generates important quantities of slag. This slag presents good mechanical properties, and for the most siliceous slag, a high stability which would enable its use by total or partial substitution of certain granulates in civil engineering. Our study (within the framework of a european programme, cofunded by the European Commission - DGXII) concerns the physico-chemical and mineralogical characterization and leaching behaviour of several types of Waelz slag. The leaching tests used are regulatory tests and specific characterization tests of leaching behaviour. They take into account the influence of several main parameters of the valorization scenarios envisaged for the slag (e.g. pH, Redox potential, chemical nature of the leachant, type of contact - liquid/solid etc).

2. Introduction

2.1. Objective

In the European Union, the steel industry produces about 700000 tons/year of Electric Arc Furnace (EAF) dusts. The EAF dusts can be recycled by the Waelz process which converts the dusts into an impure zinc oxide, called Waelz oxides, which is reprocessed in metallurgical plants (i.e. Imperial Smelting Process). A slag is also produced. The recycling of all the EU EAF dusts could lead to the production of about 500000 tons/year of Waelz slag. This slag is basically an iron reduced slag. In order to reuse it, i.e. as aggregates or as filling material in civil engineering (specific road and construction applications), in total or partial substitution of natural materials, important research programmes concerning its characterization and long term behaviour will have to be carried out.

Our study is a contribution to the chemical and mineralogical characterization of different Waelz slags. We have also observed the influence of different parameters (i.e. pH, EH, leachant composition,...) on the release of different chemical species during different leaching tests. There is a wide choice of parameters due to the variety of disposal and reuse scenarios of the slags. The aim of our paper is to gain a better understanding of the slag properties in relation to different reuse scenarios.

2.2. General presentation of the programme partners

The research programme was carried out with the participation of two main industrial companies operating the Waelz process within the European Union : BUS Metall GmbH and METALEUROP. Two independant research laboratories, POLDEN-Insavalor and ECN carried out the programme in the laboratory and on a pilot scale, coordinated by Metaleurop Research (MER).

2.3. Description of the Waelz process and identification of the most relevant technological parameters

The Waelz process allows to treat EAF dust (polluting waste from steel industry) in order to recycle zinc and to obtain a steel residue. After mixing of EAF dusts with coke breeze (reducing agent) and other additives (e.g. siliceous sand), this feed is continuously introduced into a rotary kiln. The temperature in the furnace, about 1100°C, allows reduction and vaporization of zinc and other volatil metals (lead, cadmium) to recover ofter oxidization-condensation an impure zinc oxide (Waelz oxides). The molten residue, containing low zinc and lead contents, is water cooled at the outlet of the kiln to form granular Waelz slag. According to the mixture and composition of the material on entering the furnace, the operating conditions of the furnace (e.g. temperature or residence time, etc) and the characteristics of the slag cooling, two different types of slag are obtained: silica rich slags and lime rich slags.

2.4. Case history of local valorization of slags

For many years, low amounts Waelz slags have been used locally as basic material for road construction, sportsgrounds and dykes. However, for use in compliance with the ever stricter environmental regulations, it has become necessary to characterize the slag more precisely. Particular attention must be paid to long term leaching behaviour of slags in order to estimate their long term behaviour under the conditions of different valorization scenarios.

2.5. General presentation of the experimental study

The experimental study can be divided into three parts. The first part is to determine the constituents and the structure of the slags (chemistry and mineralogy). The second part is to apply the French and Dutch regulatory tests (results not presented here). Finally, the third part is to characterize the leaching behaviour of slags using tests specially designed and adapted to the objectives of the study presented below.

3. Experimental study

3.1. Presentation of the studied reference slag samples

In the following table we present the elementary composition (% mass), basicity index (BI) and the codes used for the five Waelz slags under study. The slags come from five different plants. The technological operating conditions used for sampling of the slags in each installation was characterized. Homogenization, carried out at the end of the process ensured the representative character of the slag sampling. The granulometric fraction studied was between 0-20mm.

Code	1-OK	1-FQ	2-DU	2-FG	3-FG
B.I.	0.39	0.48	0.4	2.6	3.5
Zn	2.38	0.24	3.77	3.2	0.35
Pb	1.07	0.36	1.55	1.6	4.22
Cd	0.0007	0.0011	0.0002	0.0015	0.0015
As	0.0141	0.0141	0.089	0.04	0.0107
Cu	0.49	0.32	0.4	0.29	0.32
Fe metal	17.2	24.9	23.2	23.3	6.21
FeO	6.62	3.47	6.75	21.5	39.7
SiO2	31.4	37.3	26.9	6	7.77
CaO	7.58	15.35	7.7	13.1	23.5
K2O	0.29	0.11	0.22	0.23	0.23
Na2O	0.41	0.39	0.51	0.42	0.92
S	2.03	0.75	0.8	0.97	1.12

Table 1 : Chemical composition of slags (% mass)

The slags 2-DU, 1-OK and 1-FQ are slightly alkaline (BI about 0.4 to 0.5) whereas the slags 2 and 3-FG are more alkaline (BI greater than 2.5). The pollutant content, especially Zn and Pb, varies within quite large limits (more than ten fold) according to the slag characteristics.

3.2. Characterization of leaching behaviour

3.2.1. Chemical sensitivity test: pH 5 and pH 12.5 (controlled with NaOH or with $Ca(OH)_2$ saturated solution)

The leaching tests at controlled pH aim to estimate pollutant release (at equilibrium and dynamically) from slags which undergo prolonged aggression as far as pH is concerned. The choice of pH 5 corresponds to acidic aggression which is quite classical (due, for example, to acid rain or oxidized sulfur compounds). pH 12.5 corresponds to alkaline aggression (for example, water having been in contact with a material containing hydraulic binders). In the case of acid aggression, the effect of pH is evaluated as such without using acids whose anion would influence the solubility (such as CO2 or organic acids). As for the alkaline medium, we have tried to evaluate the effect of pH alone controlled by a simple species (NaOH) and also the effect of a calcium containing medium close to the hydraulic binder scenario.
The choice of this type of test involves the use of a pH regulation loop, consisting of a pH electrode, an electronic regulator and a pump.
The main experimental parameters of the controlled pH test are presented in table 2. Particular care was taken to avoid carbonation of the leachates.

parameter	conditions
initial mass ratio L/S	10
particle diameter	<4 mm
stirring	continuous
renewal time	after 1 day, 2, 3, 4 and 7 days
pH	5, controlled using HNO3 1N 12.5, controlled using NaOH 1N or saturated solution of Ca(OH)2
filtration	0.45 µm

Table 2 : Experimental parameters of chemical sensitivity test

In the following table we present the cumulated release (mg/kg of slag) after 7 days of leaching for certain elements (Pb, Zn, As and Fe) according to the pH.

		1-OK	1-FQ	2-DU	2-FG	3-FG
Pb	pH=5	128.5	20.9	14.9	19.6	97.6
	pH=12.5,NaOH	4.1	ND	91.7	111.3	2294.3
	pH=12.5,$Ca(OH)_2$	2416.4	634.6	1372.5	3353.8	2316.2
Zn	pH=5	201.7	6.9	420.1	2336	650
	pH=12.5,NaOH	5.2	ND	33.6	25.9	18.1
	pH=12.5,$Ca(OH)_2$	141.8	8.1	24.7	125.2	14.7
As	pH=5	ND	ND	ND	ND	ND
	pH=12.5,NaOH	22.1	4.1	0.5	3.7	0.3
	pH=12.5,$Ca(OH)_2$	8.1	6.3	ND	ND	ND
Fe	pH=5	258.9	3235.7	255.9	7376.2	8401.2
	pH=12.5,NaOH	10.8	ND	6.0	ND	3.0
	pH=12.5,$Ca(OH)_2$	ND	ND	3.2	ND	3.3

Table 3 : Cumulated release (7 days) for different types of leaching

The following graphs show the cumulated release of lead, zinc, arsenic and iron after 7 days of leaching for each type of slag and for each test (controlled pH 5, pH 12.5 by NaOH or in saturated lime solution).

In a general manner, it can be noted that there is a greater cumulated release for the most **alkaline slags**, 2 and 3 FG, which are therefore less stable.

According to the **specificity of metals**, two types of behaviour can be demonstrated (figure 1) :

- metals whose release is greater under acidic conditions (controlled pH 5) : Zn and Fe for example ;
- metals whose release is greater in alkaline conditions : Pb and As for example.

The influence of the chemical nature of the alkaline leachant (lime or NaOH) is particularly important in the case of release of lead (strongly leached by a lime solution), of zinc and of arsenic. The behaviour of arsenic shows that it is not solubilized at pH 5 and very slightly solubilized at alkaline pH (saturated lime solution). This phenomenon could be explained by the integration of certain solubility data (Nishimura) : arsenic is only slightly soluble at acid pH in the presence of certain heavy metals (Zn, Cu, Ni and Co) and also only slightly soluble at alkaline pH in the presence of alkaline earth metals (Ca, Mg, Sr and Ba).

As regards lead and zinc, a distinct destabilization of these elements in a saturated lime solution can be noted.

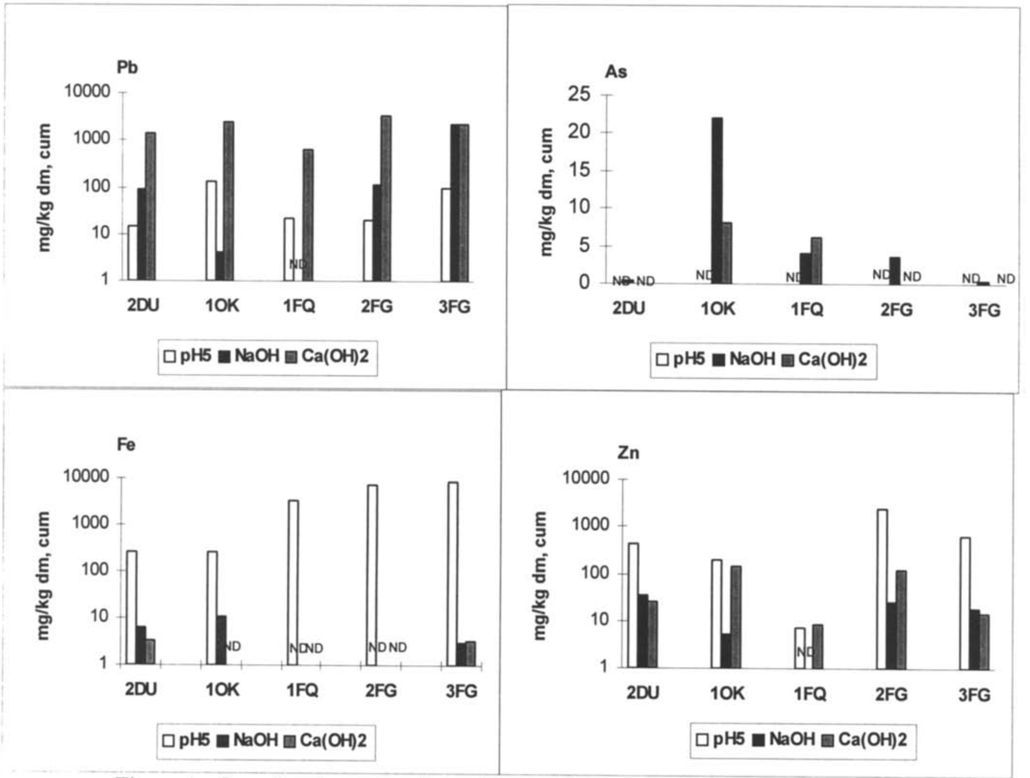


Figure 1 : Cumulated release of metals according to pH during 7 days leaching

The release dynamics are represented as the evolution of the cumulated extracted mass against the cumulated contact time. Three types of behaviour of the leached species can be distinguished :

- slow dissolution without apparent saturation of the solution (see figure 2). The concentration is therefore proportional to contact time. This is the case for zinc (slags 2-DU and 2-FG at pH 12.5_{NaOH}, 2-DU at pH 5), of copper (2-DU at pH 5 or pH $12.5_{Ca(OH)2}$), of lead (slags 1-FQ at pH $12.5_{Ca(OH)2}$).

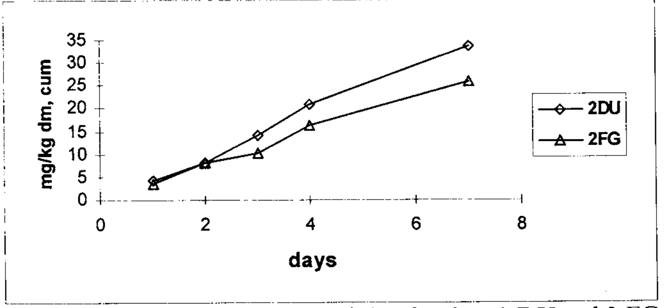


Figure 2 : Dissolution behaviour of zinc for slags 2-DU and 2-FG at pH 12.5_{NaOH} after successive leachings

- rapid initial dissolution with a slow progression of the phenomenon, asymptotic (figure 3). his may be due to a mass dissolution of certain grains but also to a surface dissolution of the grains, followed by low mass transport towards the solution. This type of behaviour can be observed for lead in the case of slag 1-FQ at pH 12.5Ca(OH)2 and for arsenic in the case of slag 1-OK at pH 12.5Ca(OH)2 .

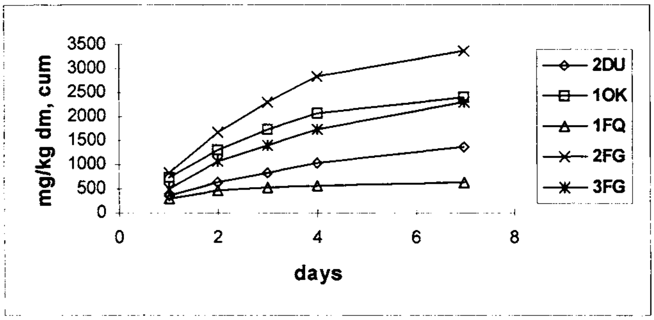


Figure 3 : Dissolution behaviour of lead in slags at pH 12.5Ca(OH)2 during the successive leachings

- dissolution with apparent control by saturation of the solution. This is the case for lead at pH 12.5Ca(OH)2 for slags 2-FG, 1-OK (figure 3) and of zinc for slags 1-OK and 2-FG at pH 12.5Ca(OH)2 (figure 4).

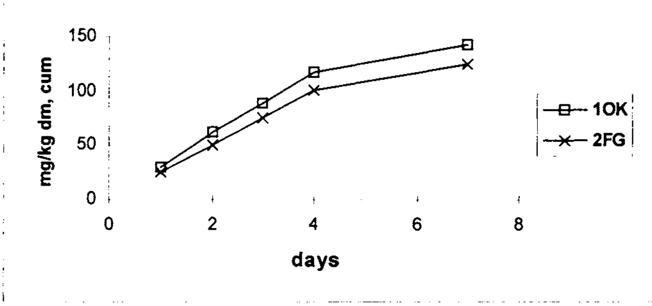


Figure 4 : Zinc dissolution behaviour for slags 1-OK and 2-FG at pH 12.5Ca(OH)2 during successive leachings

3.2.2. Redox properties of Wealz slag

Using a recently developed test procedure (Draft NVN 7348, 1995) to assess the reducing properties of a material, the Wealz slags have been tested. The first step in the procedure is to identify, whether the material being tested has reducing properties (conditions : L/S=2, particle size < 1mm, degassed demineralized water, 24 hours contact time; measurement of redox potential against redox potential for normal oxidized water at same end pH). If this proves to be the case the release of reducing substances and the reducing capacity of the solid phase is assessed. The reducing substances in the leachate and the reducing substances in the solid phase are oxidized by an excess of CeIV, the surplus is back-titrated with Fe II (Hoede et al, 1993). From the tests, it follows that the change in redox potential is less than 50 mV at the normal pH of slag leachate (pH 7-12).

This indicates that the slags do not affect their surrounding by imposing reducing conditions (less than 0.04 Mol O_2/kg). Still the slag do possess reducing properties as Fe(0) and Fe(2+) and Mn(2+) are present. This is illustrated by the oxygen consumption measured after Ce titration 1.8-2.0 Mol O_2 /kg.

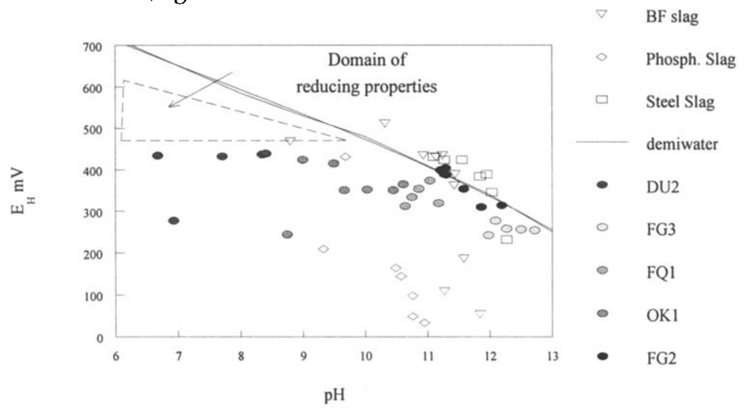


Figure 5 : Redox potential in function of the pH

The measured redox potential in leachates has been placed in perspective to other slags by plotting the redox potential measured as a function of pH in comparison with oxidized, demineralized water (figure 5). The Wealz slag is reducing, but it is only appaerent at pH < 6. This is related to the solubility of reduced Fe and Mn below pH 6. The release of reducing substances above pH 6 is however limited. This is probably caused by the rather low leachable S content. From other work S -species have been identified as the most important carriers for imposing reducing conditions on the slag surroundings. The presence of metallic Fe can lead to stresses in the slag matrix, when water can reach these iron phases through cracks.

3.2.3. ANC test

The objective of the study was to follow the concentration modifications at equilibrium of the main elements in the slags according to the different levels of acid attack of the leachant. The study also allows characterization of the slags according to their acid neutralizing capacity. The protocol of the test is as follows (table 4) :

parameter	conditions
mass ratio L/S	10
particle diameter	<4 mm
stirring	continuous
temperature	23±2°C
time to reach equilibrium	48 hours
pH control	solutions of HNO_3 (from 10^{-4} M to 2 M)
filtration	0.45 µm

Table 4 : Experimental parameters of ANC test

The range of acid concentration allowed us to obtain, at the end of the contact time, a pH between (denoted final pH) 2 and 12.

Two types of graph present the test results ; the evolution of the final pH of the leachates according to the acid added (fig 6) and the species solubility according to final pH of the leachates (fig 7).

The dependance of the final pH of the slag solutions on the quantity of nitric acid added (following figure) shows that the slags have different neutralizing capacities, in logical agreement with the progression of the basicity index :

2-DU < 1-OK < 1-FQ < 2-FG < 3-FG.

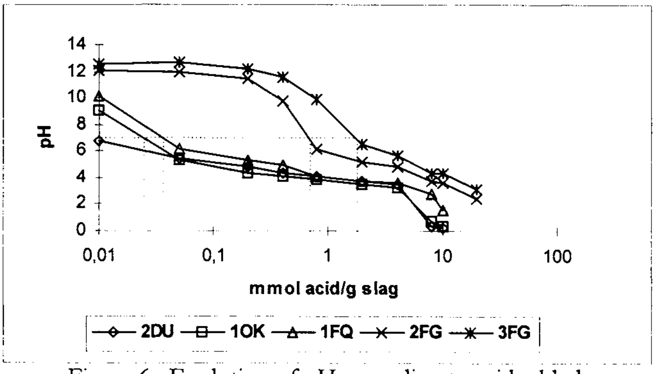


Figure 6 : Evolution of pH according to acid added.

The decrease in pH for acid leaching of the slags 2-DU, 1-OK and 1-FQ is relatively linear with increased acidity of solutions until about 5 mmol. A more important decrease then occurs for 2-DU and 1-OK, which could indicate a chemical transformation of a phase.
The two most alkaline slags of the series (2-FG and 3-FG) show an important decrease in their buffering capacity especially in the interval 0.4 to 2 mmol HNO_3/g of slag.

The following figures present the concentrations (mg/l) of certain elements (Pb, Zn, As et Na) in the extraction solutions according to their final pH.

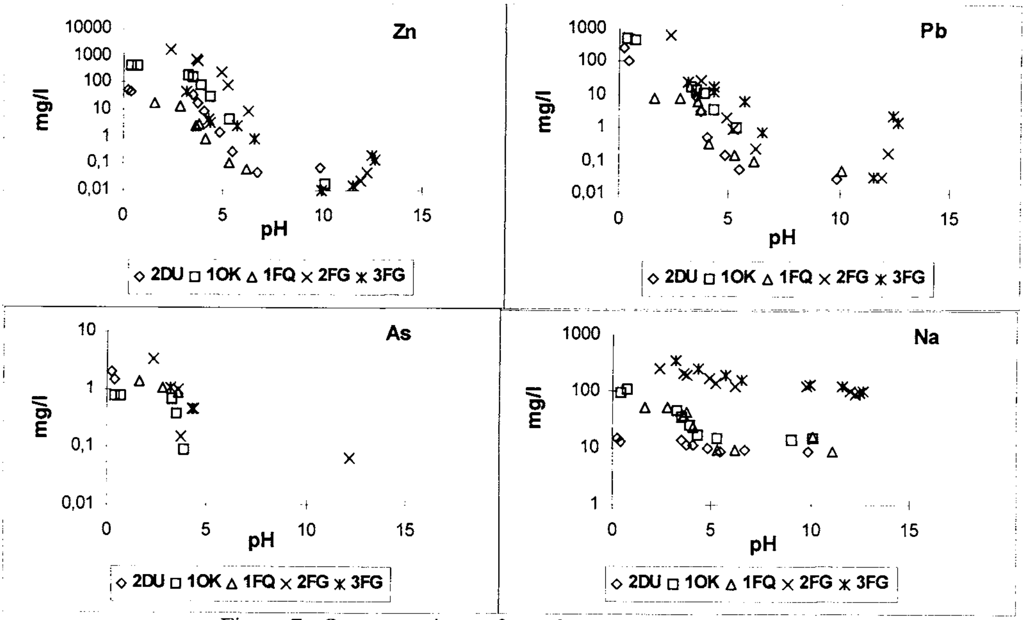


Figure 7 : Concentrations of metals at equilibrium against pH

Solubilization of the species according to the nitric acid added shows characteristic behaviour :

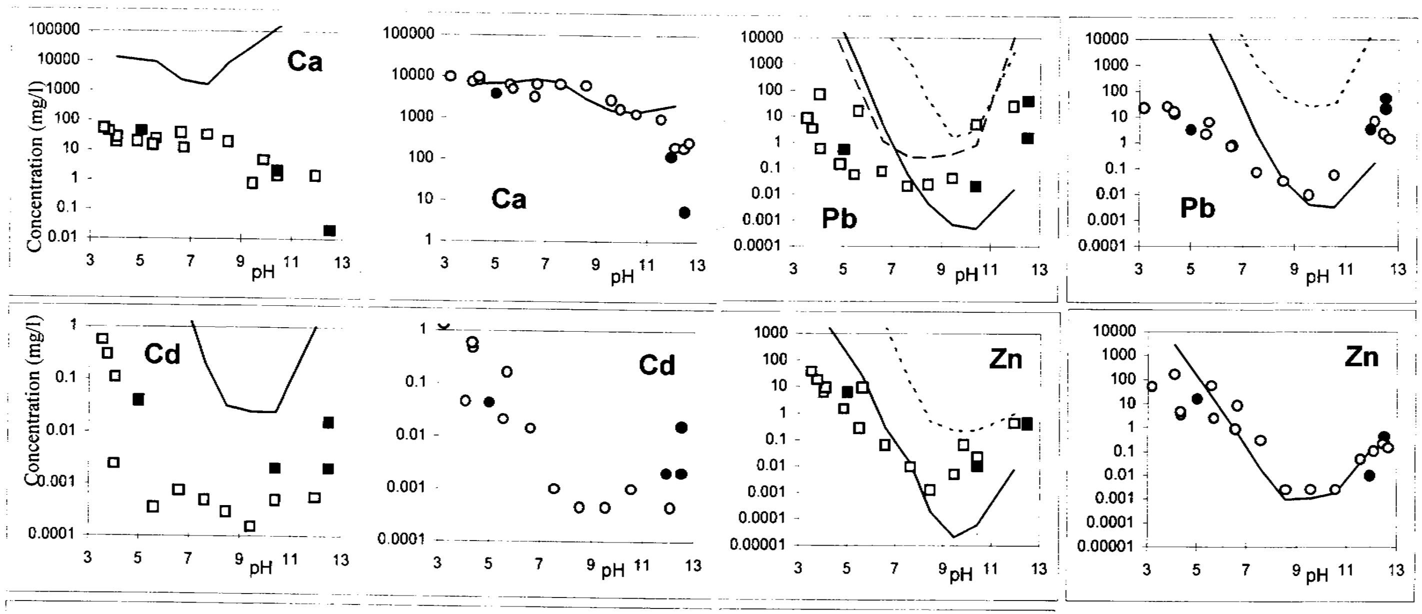
- amphoteric behaviour for solubilization of zinc and lead. Their release is low for pH 7 to 11, which are in fact the pH values obtained by application of the test X31-210.
- greater dependance on the slag specificity for the solubilization of arsenic and certain (Ca, Cu and Cd). A greater dependance on slag nature can be noted in an acid medium.
- low dependance solubility/pH for alkaline elements (Na and K).

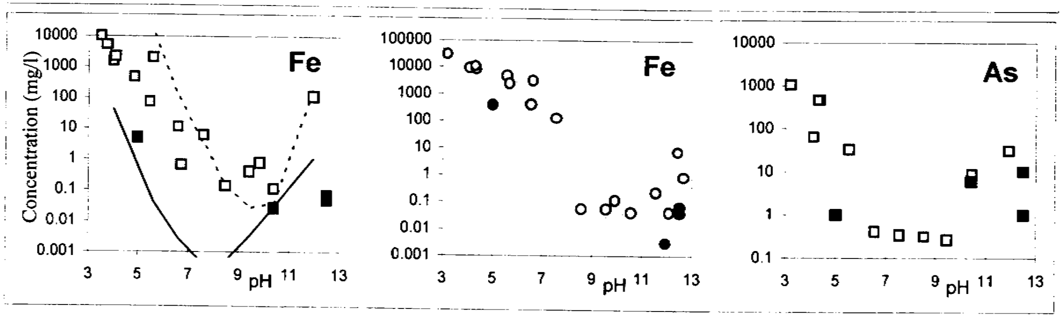
The agreement between ANC test results and controlled pH test results can be noted, especially at pH 5 and pH 12.5 controlled by NaOH.

3.2.4. pH stat

This pH static test (Van der Sloot et al, 1993....) provides information on the pH sensitivity of leaching behaviour of the slag. This information is important as it forms the basis to identify the solubility controlling phases. Furthermore the test gives information on the sensitivity to pH in specific field scenarios, where the pH of the slag may change due to external influences. The acid neutralization capacity (ANC) derived from the test is a useful additional property in this respect as it allows a quantification of the buffering of the system when exposed to externally imposed pH changes. Finally, data from many different leaching tests can be placed in perspective using the pH test data as a reference.

To assess the behaviour of slags as a function of pH, a pH stat test is carried out on granular material ($d < 2mm$) using a liquid to solid ratio of $L/S = 10$ and pH control in the range from pH = 4 - 12. A pH controller is steered by computer dosing HNO_3 or NaOH depending on whether acid or base is required. The dosing equipment is developed in such a way as to minimize the volume increase. Results of pH stat tests on Wealz slag are presented in figures 8.





Slag code	symbol	pH
DU2	square	8.8
FG3	circle	12.1

Element	Mineral phases	
	Line	Dotted line
Ca	Gypsum	
Cd	Otavite	
Fe	Ferrihydrite	Siderite
Pb	Pb(OH)2	Cerrusite
Zn	ZnSiO3	Zincite

In figures 8 the data are given. Results are presented for a silica rich slag (2 DU) and for a lime rich slag (3 FG). To illustrate the agreement between pH stat information and other test methods, the results of the single pH controlled tests and the ANC test data are included for comparison.

As can be seen from the graphs there is no relation between chemical composition and leachability, as leachability is largely controlled by pH in solution. The agreement between different tests is reasonable to good with some specific exceptions, where the addition of for instance Ca or CO2 affects directly the solubility of a specific element (e.g. Cd, As).

3.2.5. Leaching modelling: identification of the solubility controlling phase

Using the geochemical speciation model MinteqA2, the leaching data obtained from the pH-stat experiments on Wealz slags have been modelled to identify the potential solubility controlling phases. This information is relevant for decisions on possibilities for modification to improve slag leaching characteristics. It also helps to identify the influence of basicity and the degree of amorphous glass versus crystaline phases. The leaching data obtained for major, minor an trace elements have been transformed to mol/l concentrations and used to calculate saturation indices for all possible mineral phases. Based on the evaluation of the saturation indices, those phases that are likely controlling phases due to a good match between measured concentrations corrected for activity (Debye-Huckel) and solubility of specific phases over all or a part of the pH range studied have been selected. Below the behaviour of a few individual elements is discussed:

Cadmium - The leachability of Cd is apparently not controlled by otavite (CdCO3), as the actual solubility is 2 orders of magnitude less than predicted for otavite. Cd-silicate does not appear to be a relevant controlling phase either. In this case, solubility may be correlated with matrix dissolution. The Cd/Si ratio of leached concentrations amounts to 0.0008 (st dev. 0.0006), when a few extreme values are omitted. This is remarkably constant for a trace element, so this mechanism may apply here.

Calcium - The solubility of Ca is reasonably well explained for the lime rich slags, where gypsum is a controlling phase. There are many possible calciumsilicate phases that may be controlling release in the silicate rich slags.

Iron - In the low pH region Fe II minerals are relevant solubility controlling phases. The mineral siderite proves to be a likely controlling phase. In the neutral to alkaline pH domain Fe leachability can be controlled by ferrihydrite - $Fe(OH)_3$. This will be particularly relevant upon aging of the slag, when due to environmental influences (oxidation and carbonation) a coating of ferrichydroxide is formed on the slag surface.

Lead - The most relevant solubility controlling phase for Pb solubility from Wealz slag is $Pb(OH)_2$. $PbSiO_4$ is not of importance. In the longer term the important controlling phase appears to be cerrusite - $PbCO_3$. Only in case of very fresh slags, which have not undergone sufficient aging - conversion of Pb to PbO will occur, then upon contact with water this will change to $Pb(OH)_2$ and finally upon contact with the atmosphere to $PbCO_3$. Depending of the state of aging control of Pb solubility by one of the intermediate phase may be most prominent. The leachability of Pb as function of pH is very similar for all slags. The silica rich slag show generally a higher leachability at high pH than the lime rich slags.

Zinc - The solubility of Zn is different for the different slags. In some cases the $ZnSiO_3$ solubility matches quite well with the measured data points (3 FG, 2 DU for part of the pH range). In case of 2 FG and the high pH range for 1 OK, willemite (Zn_2SiO_4) appears to be the major solubility controlling phase. The leachability of the lime rich slags is significantly higher than that of the silica rich slags. This is particularly relevant in the low pH domain.

The geochemical modelling has shown that for the majority of the elements possible solubility controlling phases can be identified. The lime rich and silica rich slags can be distinguished in terms of their leaching properties for several elements. Based on the modelling it can be concluded that slag surface mineralogy controlling leachability is different from bulk slag mineralogy as obtained by XRD and similar techniques. This is particularly important when the slag can be considered as a stable material with very limited susceptibility to dissolution. The changes in the slag surface due to exposure to environmental conditions will largely control what the environmental impact from slags in a given scenario will be. From this work it can be concluded that application in an acidic environment (e.g. peat soils, acid sandy soils) should be avoided.

4. Conclusions

The experimental programme aims to characterize the intrinsic physico-chemical properties of slags studied as well as their leaching behaviour. It aims to demonstrate the influence of certain main parameters (such as pH). Several important points can be made :

- The release of polluting elements during leaching tests using the X31-210 standard for example is below the regulatory thresholds for slags 2-DU, 1-OK, 1-FQ and 2-FG. This is not the case for 3-FG.

- The data obtained with different leaching tests compare generally well with the data obtained with the pH stat test and ANC test, which implies that these tests can be regarded as a basic characterisation test of leaching behaviour. The pH stat data can be used as a basis of reference for quality control testing. It also provides crucial information on potential risks of exposure to other pH conditions in the field, which can not be obtained from a single step leaching test.

- The geochemical modelling has been placed in relation to slag characteristics such as lime-rich and silica-rich properties. This has revealed interesting general characteristics of Wealz slags and leads to a better identification of possible solubility controlling phases. The changes in the slag surface due to exposure to environmental conditions will largely control what the environmental impact from slags in a given scenario will be.

    - The leaching behaviour of the different Wealz slags is largely solubility controlled in the pH domain ranging from pH 5 to 12. This can be concluded from the similarity in leaching as function of pH in spite of differences in slag basicity and level of crystalization.

    - Complex interactions can be observed between the chemical nature of the leachants and the physico-chemical specificity of each slag. The valorization scenarios by partial or total substitution of granulates by slags in cement with high limestone content may pose the problem of the high availability of lead, observed experimentally.

- It can be noted that more alkaline slags such as 2-FG and 3-FG are more sensitive to the influence of pH than slags 1-FQ, 1-OK and 2-DU. The acid-base capacity and the release of elements are dependant on the characteristics of each slag. The low buffer capacity of the silica rich-slags implies that application or disposal of untreated slag in acid environments (pH 4-6) should avoided.
- To optimize integration of slags in materials, it will be necessary to orient research towards less alkaline binders than classical CPA cements, while offering sufficient protection against acid waters. A concrete with low limestone content (slag, aluminates), where the alkalinity in the porewater would have a less corrosive effect on the vitreous phases of slags, would seem more appropriate.

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