

## Long and short-term performance of a stabilized/solidified electric arc furnace dust<sup>☆</sup>

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### Abstract

The application of class F fly ash, cement and lime to the Stabilization/Solidification (S/S) of electric arc furnace dust containing hazardous metals such as Zn, Pb, Cd, and Cr is described. The aim of the study was to determine the influence of the setting conditions during the S/S treatment and to know the behaviour of an aged solidified and stabilized waste. In order to determine the efficiency attained by the S/S process, USEPA TCLP, and other leaching tests have been accomplished. In addition, the compressive strength of the solidified waste at different times has been determined. In order to study the influence of the environmental conditions in which setting occurs, experiments were carried out with samples of the same composition, under different setting conditions: laboratory environment, stove at a temperature of 40–60 °C and setting in a hermetically sealed plastic bag at room temperature. All the samples were subjected to the TCLP test at 28 days, and the metal content of the resulting leachates was analysed. The results show that in some cases the setting conditions of the mixtures have a noticeable influence on the characteristics of the leachate. The evolution with time of some S/S solids, one month after their manufacture and more than 9 years after that has also been evaluated, by means of their leaching behaviour. The results obtained in this work have shown, in all the laboratory cured samples that the leachate pH decrease in the course of time, and consequently the leaching behaviour is in general worse. This could be due to the carbonation of the S/S solid and the subsequent loss of alkalinity.

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### 1. Introduction

Stabilization/solidification (S/S) is, according to the US Environmental Protection Agency (EPA), one of the best demonstrated available technologies, to treat certain industrial wastes and contaminated soils. S/S technologies are widely used for treatment of hazardous wastes that are mostly inorganic and contaminated soils before final disposal [1–3].

The agglomerating agents used in S/S processes for the treatment of hazardous wastes that contain heavy metals are usually alkaline materials that reduce the leachability of the metals in a high-pH matrix. Coal fly ash is also one of the most com-

mon binders in waste stabilization formulations, mainly in two kinds of mixtures: Portland cement + fly ash or lime + fly ash [2]. The use of appropriate agents can help control the long-range leachability of the stabilized solid, releasing alkaline substances such as hydroxides that are capable of maintaining a high pH level in the water or in the extraction fluid (normally an acid solution) that is put into contact with the treated waste [4].

Despite the increased use of S/S technologies, there is little published information on the long-term leaching performance for this type of treatment. Recent studies recognize as a major problem in this area that there is a fundamental lack of field data related to the performance of solidified products [5]. In general, although many studies appear in the literature that refer to the long-term behaviour of different S/S solids, generally it is the result of accelerated aging tests [6] or an estimate based on some mathematical models [7] that allows to predict how the long term leaching behaviour and the evolution with the time of the stabilized solids will be. Real studies are much scarcer and

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leaching data of S/S solid samples are rarely known more than 6 years after their preparation.

In a study carried out on some commercially stabilized waste, toxicity characteristic leaching procedure (TCLP) extraction [8] was performed at 28, 90, 200, 470 and 650 days after treatment [9]. The results demonstrated a highly waste-dependent effect of time on the TCLP results. Leachate values for some metallic wastes remained stable and for others increased over time. The study did not include exploration of the mechanisms causing the increased leaching in stabilized wastes over time.

Also little research has been conducted on the weathering and degradation of solidified and stabilized wastes once the treated materials have been buried. Mineralogical alterations of seven metal-bearing solidified and stabilized wastes that have been landfilled or stored aboveground for up to six years have been published [10]. The wastes were treated with Portland cement and stored outdoors, archived in the laboratory or buried on site. Results show that although the extent of degradation after 6 years is considered slight to moderate, the same environmental factors that affect the durability of concrete also must be considered when evaluating the durability and permanence of cement-stabilized and solidified wastes.

In an interesting study [11], a metal-plating waste treated by S/S using Portland cement (OPC) and coal fly ash has been characterised after exposure to the environment in SE England for approximately 10 years. In that study the surface region of the S/S waste was extensively carbonated and had reduced acid neutralization capacity compared to bulk samples. In addition, the leaching properties of material tested after 28 days were very different from leaching determined in environmentally exposed S/S waste.

Carbon dioxide interacts with cementitious wasteforms in a process known as carbonation. Carbonation occurs when Portlandite and other calcium bearing phases in the wasteform react with  $\text{CO}_2$  to form calcite ( $\text{CaCO}_3$ ). It has been reported that carbonation may directly alter the chemical and physical properties of solidified waste forms [12], but contradictory results regarding metal leachability have been published. According to Lange et al. [13,14], the effects of carbonation appear to be beneficial on the leachate production of cement-solidified hazardous wastes, showing a significant reduction in leachable metals extracted.

The application of carbonation treatment to cement-based waste forms as a means to reduce the leachability of entrained undesirable substances has been the subject of much study over the past decade. The carbonation process has been shown to reduce the pore size distribution and permeability of the cementitious materials since portions of the original pore network become sites for precipitation of secondary carbonate minerals [15].

However, other studies show that carbonation produces the opposite effect. Thus, in a study to evaluate the role of carbon dioxide on the long-term performance of cementitious waste forms, carbonation increased the release of Cd, Pb, and Co [16]. In a paper concerning the S/S of municipal solid waste incineration (MSWI) residues, the pastes were exposed to an accelerated carbonation procedure. In contrast with other studies, the authors

mention that the carbonation process increased the leachability of heavy metals such as Zn and Cr [17].

Both carbonation and storage effects have been recently studied by Sánchez et al. [18] and Gervais et al. [19] in scenarios with a variable relative humidity. According to the authors S/S solids in many field or management scenarios are not continuously wetted but experience cyclic wetting and drying under varied environmental conditions (humidity and  $\text{CO}_2$ ). In addition, the extent of carbonation increased with lower relative humidity during storage intervals. Other studies show that the concentration of specific metals or other hazardous components within waste might be important in governing the rate of carbonation reaction [5]. In these changing conditions different processes can influence the release of inorganic constituents from the solid matrix and depending on the case cause increases or reductions in the metal release.

This paper refers to the stabilization of arc furnace dust waste (EAFD). EAFD is a dust that results from the collecting systems of particulate material in carbon-steel electric arc furnaces. The hazardous metals normally found in electric arc furnace dust are Pb, Cd, and Cr. In addition, the dust usually has a high zinc content which may reach up to 40% resulting from the use of galvanized scrap [20].

Although EAFD is considered a waste difficult to stabilize, the use of different cementing systems containing Type F coal fly ash, lime, dolomitic lime, Type I Portland cement, blast furnace slag or silica fume may be found in the literature [21–23]. Other alternative treatment for EAF dust is vitrification [24].

Fuessle and Taylor [23] have investigated the effect of cure age for the stabilization of lead and cadmium in an electric arc furnace dust. They have found that, although the TCLP pH values decreased with increasing cure age after one year the rank of Cd and Pb results in TCLP leachates is (basically) established by mix design.

EAFD has been treated using different formulations containing coal fly ash, cement and lime, and the results are published elsewhere [25]. Many of the S/S solids obtained after treatment met some short-term regulations stated in order to evaluate the efficacy of the S/S process. In this paper the evolution with time of some of these well stabilized EAFD S/S solids, which were archived in the laboratory, and the influence of the ambient conditions maintained during the setting period have been evaluated, by means of the leaching behaviour of the solids.

## 2. Material and methods

ASTM class F fly ash from the combustion of high quality pulverized coals in one of the biggest coal power plants in the south of Spain, Los Barrios (550 MW), was used as the main aluminosilicate agent. Other stabilization agents used in this work were lime and Portland cement (Types I and II). Chemical composition of these agents has been carried out after disgregation at  $750^\circ\text{C}$  and analysis using Atomic Absorption Spectrometry (AAS) according to ASTM D-3682 and is included in Table 1.

The heavy metal content of the waste (EAFD) has also been determined after disgregation at  $750^\circ\text{C}$  using AAS and is shown in Table 2. Average concentrations are shown since

Table 1  
Chemical composition (major species) of S/S agents (% w/w, dry basis)

Agent	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO
Fly ash (FA 1)	50.0	8.8	20.8	4.0	1.3
Fly ash (FA 2)	45.3	2.4	34.4	8.4	1.9
Fly ash (FA 3)	63.9	5.9	21.5	3.9	1.8
Portland cement (OPC) (Type II)	23.0	3.8	2.5	64.7	1.3
Portland cement (OPC) (Type I)	22.5	3.6	4.9	67.3	1.7

Table 2  
Average hazardous metal content in waste (% w/w, dry basis)

Waste	As	Cd	Cr	Ni	Pb	Zn
EAFD	0.29	0.08	0.95	0.20	1.30	26.0

the metal contents varied with time, as different batches were used.

### 2.1. Quality criteria

To evaluate the degree to which the S/S objectives were met, some specific criteria were defined. The criteria refer to some physical (setting time range: 5–72 h and compressive strength  $\geq 0.34$  MPa) and chemical properties of the S/S solids, measured at 28 days. The chemical criteria include the determination of the pH after mixing the crushed solid with distilled water in a liquid (water)/solid (stabilized waste) ratio, *L/S*, of 10 (w/w) and the analysis for the most important metals in the TCLP leachates of EAF dust S/S solids.

All metal analyses were performed using AAS. The chemical criteria are summarized in Table 3.

The limits for the concentration of Cd, Cr and Pb in leachates are those used by the USEPA to characterize a waste not listed as toxic and to evaluate S/S processes. The limits of these values were chosen by multiplying the maximum permissible concentrations for potable water by one hundred. This gives a safety factor of 100 supposing that the leachates of the treated waste, once they are in a well-designed landfill, will be diluted in the water courses at least one hundred times before reaching a potable water intake. Following the same criterion, but based on the Spanish guidelines for pre-potable water, a limit was set in this study for the concentrations of Zn in the TCLP leachates.

### 2.2. Preparation of S/S solids

The methodology used for obtaining solids was described as follows [25]:

Table 3  
Quality criteria for S/S solids (chemical properties)

Parameter	Range/value			
pH	2–12.5			
Metal concentrations in TCLP leachates (ppm)	Cd	Cr total	Pb	Zn
	0.5	5	5	300

Table 4  
Composition of S/S mixtures (proportions by weight)

Sample	EAFD	FA	Lime	OPC (type II)
M1	1	1.4	0.2	0.2
M2	1	1.4	0.2	0.25
M3	1	1.4	0.2	0.33
M4	1			2
M22	1	1	0.2	0.25
M26	1	1.4	0.2	0.2
M27	1	1.4	0.2	0.2 (type I)

1. Enough water is added to the waste-fly ash mixture to make the paste castable; then Portland cement is sometimes incorporated. Finally, if necessary, some lime is added so that the paste will have a pH near 12. The resulting mixture, with a water/solid ratio of around 0.25, is agitated in a planetary mixer. The pH of the paste is measured in the solution obtained by mixing 5 g of paste with 100 ml of distilled water after agitating the mixture. In some cases, the mixing process can be improved by adding the water of the formulation as part of a waste or fly ash slurry.
2. With the mass obtained, cylindrical plastic moulds are filled and compacted. During setting, the samples obtained are placed in a controlled-moisture chamber (90% RH).
3. After 28 days, the samples are crushed and sieved to between 0.25 and 4 mm, and are then extracted using leaching tests.

After the 28-days curing period most of the samples were stored in the laboratory in ambient conditions (average temperature: 20–25 °C and relative humidity of 45–55%).

For this study, four mixtures, M1 to M4, were selected. The first three are mixtures of EAF dust with fly ash (FA 1), lime and increasing quantities of cement (OPC). The M4 is a mixture of EAF dust with OPC only. For the study of setting conditions, other samples M22, M26 and M27 were also studied, in which different fly ash batches (FA 2 and FA 3) were utilised. The composition of the blends is shown in Table 4.

### 2.3. Compressive strength test

Compressive strength test was performed as per ASTM D-1633-84 using two cylinders of each sample and averaging the experimental values obtained. A Suzpecar, mod. MEM-102/50 t, compressive strength testing apparatus was used.

### 2.4. Other leaching tests

In 1986, Environment Canada published the Acid Neutralization Capacity (ANC) test [26] to measure the capacity of different binders and wastes to resist pH reduction by exposure to a dilute nitric acid solution. The GANC test [27] is, as the ANC, a single-batch procedure that uses from 0 to 6 equivalents of acetic acid (the same acid used in the TCLP test) per kilogram of solids. The GANC test was adapted as described by Vale Parapar [4] to study the effect of leaching on the different components used in S/S processes and on their mixtures. From

the data recorded, we obtain the acetic acid consumption for pH values ranging from approximately 12.5 to 5.5 and the corresponding GANC curves. The NEN 7345 diffusion test (Tank Leaching Test) [28] is a monolith-type leaching test developed to assess the leaching rate of solids as the stabilized/solidified wastes over the long term. Metal analysis in NEN leachates was carried out using AAS and Inductively Coupled Plasma Emission Spectroscopy (ICP-ES) techniques.

### 3. Results and discussion

#### 3.1. Carbonation of S/S solids

The four blends selected, M1 to M4, gave good results after 28 days, with respect to the degree of compliance with the quality criteria adopted. Test pieces of the M1-M4 solids were then subjected to TCLP leaching test, determining the leachate pH in the test (after 30 days, three months, 6 months, 1 year, 5.5 years and 9 years). Different aliquots of the same mixtures were then subjected to the GANC test 28 days, 3 months, 5.5 years and 9 years after preparation.

As can be seen in Fig. 1, the leachate pH in the TCLP test decreases rapidly in mixtures M1 to M3 over time, showing an initial resistance to pH reduction, which was greater in those samples with a higher OPC content. With regard to mixture M4, this reduction is much lower, but in all the mixtures it was observed that the pH was near neutrality after 5.5 years. In water (with a  $L/S=10$ ), a reduction in pH is also observed, but the reduction is in no case as great as with the TCLP test, and even after 9 years, the leachate pH is above 12 in all the cases.

In general, as can be seen in Table 5, the metal content in the TCLP leachate varies along the time with the decrease in pH,

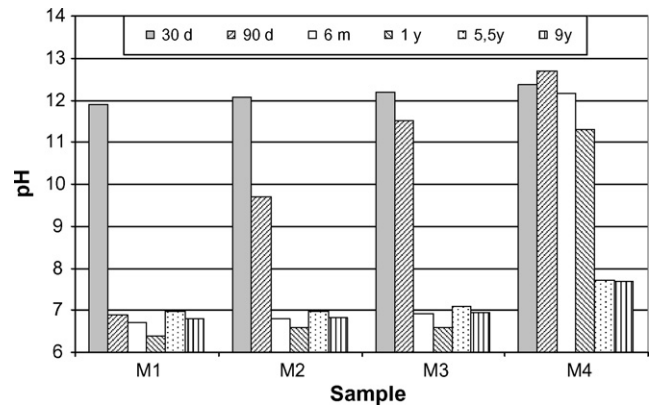


Fig. 1. Evolution with time of pH in TCLP leachates.

according to the dependence relationship observed between the leachability of Zn, Cd, Pb and Cr and pH in the S/S solids [25]. Thus, after 9 years, only the M4 sample meets the quality criteria for the metal TCLP leachates, as M1, M2 and M3 exceed the limits for Zn and Cd.

Fig. 2 shows the results of the GANC tests for samples M2 and M4, which may be considered as representative of the behaviour shown by the other samples. The figures reveal a change in the behaviour of the solids in the period of time considered. This fact results more evident in the right graph showing the evolution with time of the GANC curves of the M2 sample. The comparison between M2 and M4 also evidences the greater acid neutralization capacity of M4, as consequence of its greater alkaline character.

Upon studying the behaviour of different mixtures over time, it seems that there are some reactions, which can bring about the almost total disappearance of the alkaline equivalents con-

Table 5  
Metal concentrations in TCLP leachates (mg/L)

Sample	Zn			Pb			Cd			Cr		
	18m	5.5y	9y	18m	5.5y	9y	18m	5.5y	9y	18m	5.5y	9y
M1	280	572	501	2.4	1.8	1.2	2.3	2.2	1.74	0.1	–	0.15
M2	320	514	450	2	2	<0.2	2	2	1.66	0.1	–	0.16
M3	280	458	320	1.9	1.6	<0.2	1.9	1.6	1.01	0.2	–	0.15
M4	0.3	65	17	0.4	0.3	0.5	0.1	1.2	0.42	1.0	–	0.41

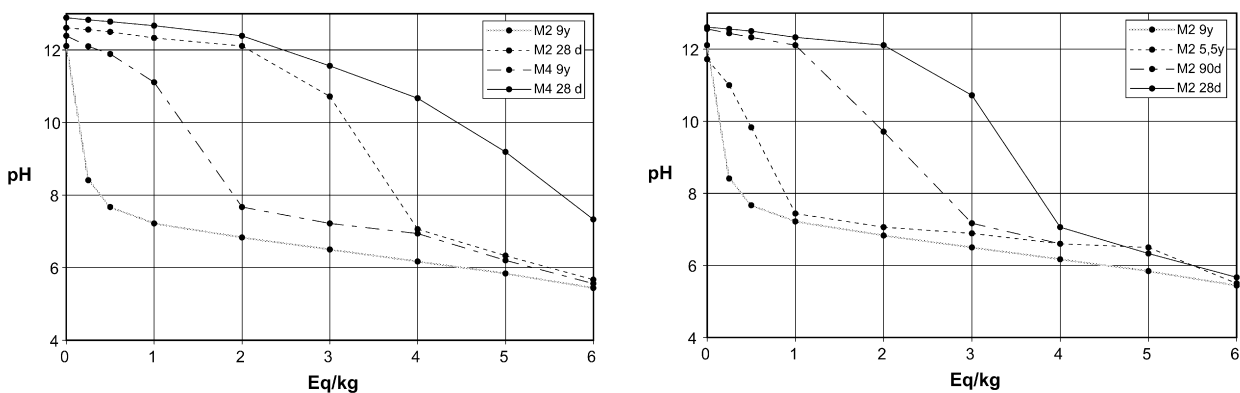


Fig. 2. GANC curves of M2 and M4 solids. Evolution with time.

tributed and consequently release the metals from the solid matrix. In addition to the reaction due to the setting of the cement and the pozzolanic reaction of the fly ash, there may be other reactions, probably due to a carbonation process of the samples cured at ambient conditions, which may explain their evolution with time.

Apart from the carbonation phenomenon, which in many cases has a slower development, another potential consequence of storage in an unsaturated environment is the drying of the matrix caused by gradients in relative humidity between the saturated matrix and the surrounding atmosphere. Moisture loss during storage may be expected to influence precipitation and redistribution of contaminants within the pore structure.

### 3.2. Influence of the setting conditions

In order to study the influence of the environmental conditions in which setting occurs, experiments were carried out with samples of the same composition, under different environments. The M22, M26 and M27 mixtures were selected and test pieces were prepared and allowed to set in different environmental conditions: ambient setting in the laboratory (A), setting in a stove at a temperature of 40 or 60 °C (E), with or without air renewal (E40 R and E40 respectively), and setting in a hermetically sealed plastic bag at ambient temperature (B). All were subjected to the TCLP test at 28 days (and 9 months–9m–for M26 and M27), and the metal content of the resulting leachates was analysed. The results of these tests are shown in Table 6.

The results of the TCLP leaching test show that the setting conditions of the mixtures have a notable influence on the characteristics of the leachate. As can be seen, there is a considerable difference in the pH of the mixtures which set in the bag and those which set in a stove or at ambient temperatures (the bag samples always have a higher pH), results which have also been confirmed with other compositions. This shows that temperature and humidity during setting play an important part, at least in the short term, not only on the physical properties but also on the chemical stability of the final S/S solid. The differences between ambient and stove setting appear lower.

Table 6  
Influence of the S/S setting conditions

Sample	pH	Zn	Pb	Cd	Cr
M22 A	11.85	0.26	2.32	0.03	2.81
M22 B	12.34	3.12	20.2	0.04	5.79
M22 E40	11.89	0.31	2.46	0.03	3.36
M22 E40 R	11.96	0.34	3.30	0.03	3.41
M26 A	11.18	0.1	0.6	0.03	2.7
M26 B	11.78	0.3	8.8	0.04	5.1
M26 E60	11.61	1.2	4.0	0.04	2.8
M26 A 9m	7.25	350	0.5	2.13	0.2
M26 B 9m	8.04	24	1.0	0.72	0.7
M27 A	7.85	32	1.0	0.7	0.50
M27 B	12.31	1.7	9.2	0.04	5.9
M27 A 9m	7.15	114	0.2	1.7	0.21
M27 B 9m	7.30	156	0.5	2.0	0.21

TCLP results (mg/L).

In the case of the M26 and M27 samples a clear evolution is observed in the period that goes from 28 days to 9 months. Thus, both the A and the B samples show pH values in the TCLP leachates at 9 months close to neutrality, which supposes in general an increment of the concentrations of Zn and Cd, and on the other hand a decrease of the levels of Pb and Cr. One can also observe in the case of the sample M27 A an anomalous pH value at 28 days (already observed in other similar samples), possibly due to a quick carbonation. This behaviour could be the result of the existent conditions of temperature and humidity in the laboratory at the time of the year in which the samples were prepared. In this case, the pH of the TCLP leachates at 28 days presented similar values to those shown by other samples at 9 months, and TCLP metal concentrations also similar.

If the data shown in Table 6 are analyzed in the light of the metal limits stated before, you can observe that, at 28 days all the bag samples present leachability problems of Pb and Cr, probably due to their high pH values, while the air-cured (and stove-cured) samples, almost always meet the established criteria. At 9 months the leaching problems of Pb and Cr disappear, but on the other hand, these are then shown by Cd.

In the case of the M26 samples a GANC study has been carried out at 28 days and 9 months. The results are shown in the Fig. 3. As can be seen, there is an evolution over time that is particularly important in the air-cured samples (as it happened in the samples M2 and M4), and is less important in the bag samples that better maintain their alkalinity.

Regarding the mechanical properties we can clearly observe in Fig. 4 how the curing process in a saturated atmosphere implies a considerable improvement of the properties. Thus, the compressive strength that is already greater in the B samples than in the A or E samples at 28 days, reaches at 9 months values in between 13–22 MPa, when the air-cured samples have a compressive resistance of 2–4 MPa and those of the stove are around 1 MPa. The values at 17 months seem to stabilize, also showing small decreases in some cases.

As it is generally recognized, the TCLP is a leaching test carried out in unfavourable conditions, not only because of the high amount of acid used, but also because the test requires a size reduction so that the encapsulation effect attained by the treat-

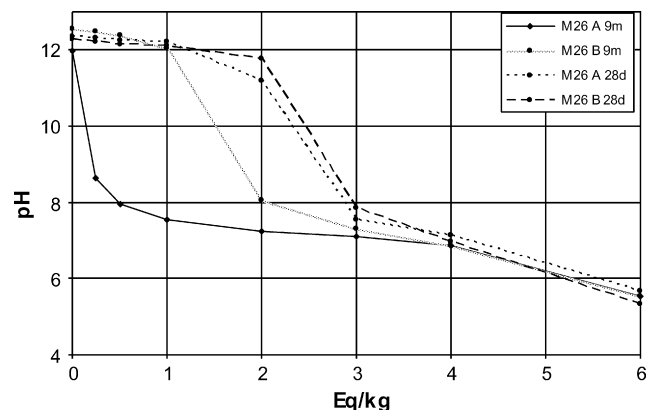


Fig. 3. GANC curves of M26 solids. Evolution with time.

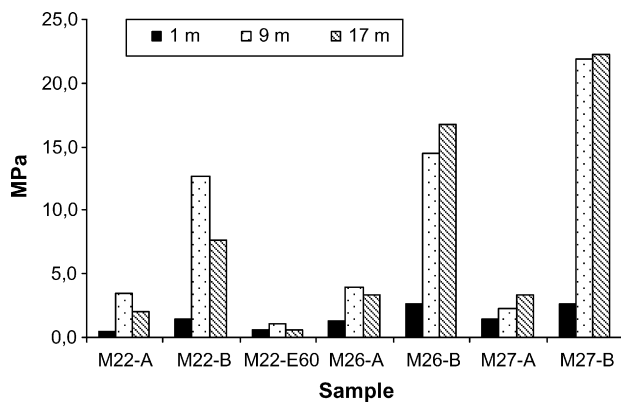


Fig. 4. Evolution with time of the compressive strength of M22, M26 and M27 solids.

ment is lost. With the aim of obtaining a better understanding of the waste stabilization degree reached by the S/S solids, some of the samples previous described (M26) were submitted to the NEN 7345 test [28], a tank leaching test for bound or shaped materials. Table 7 shows the NEN 7345 leachate concentrations of M26 test pieces.

Many metals are under detection limits, as for example As, Cd, Cu, Ni and V in all the cases and Pb and Zn in some cases, showing a high degree of immobilization in the S/S matrix. The most important leaching problem is that of Cr. After 64 days the accumulated Cr concentrations were in all the samples above 100 mg/m<sup>2</sup>. One of the M26 samples (M26B) also showed a high Pb concentration probably related to a high pH (values in the range 12.0–12.8).

As it has been indicated previously, the stabilization of Zn, Cd and Pb in the metallurgical waste studied depend clearly on the alkalinity of the matrix, and for this reason a direct relationship between the pH of the TCLP leachate and its metal concentrations is observed. Cr exhibits different behaviour to the other metals, although Cr leachability also depends on the leachate acidity. Thus, in the S/S of this waste when the pH of the TCLP leachate is very high (pH > 11.5), its Cr concentrations are usually also high, exceeding the established limit of 5 ppm, but the concentration-pH data are more disperse than those of Cd, Pb or Zn. Cr is a metal difficult to stabilize in some wastes, mainly when this element is in hexavalent form, which is much more soluble and mobile than the trivalent form. This behaviour has been described in a recent study related to the leachability of EAFD waste [29]. For this reason, in some chromium containing wastes, to guarantee their stabilization, a previous reduction stage of the metal, by means of the treatment of the waste with some appropriate reducing agent becomes necessary [30].

Table 7  
NEN 7345 Accumulated 64 days leaching test results (mg/m<sup>2</sup>)

Sample	As	Cd	Cr	Cu	Ni	Pb	Zn	Ba	V
M26A	6.2	0.6	168.4	3.7	1.2	6.2	3.6	9.8	1.2
M26B	6.2	0.6	181.6	3.7	1.2	225	6.8	93.4	1.2
M26A/40	9.5	0.9	124.5	5.7	1.9	9.5	2.0	18.1	1.9

#### 4. Conclusions

The results here presented show that the alteration of cement-based materials during storage may influence the leaching potential and release rate of inorganic contaminants by changing the chemical and physical properties of the treated material that control constituent release. More specifically, the carbonation and the rapid loss of moisture produced in stabilized wasteforms cured under the laboratory environment could be responsible of the observed leachability increase along the time. In consequence, regulations concerning waste stabilization should have to consider the curing conditions of the solidified solids in more detail and the potential evolution with time of the S/S solids.

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