



# Solidification/stabilization of fly and bottom ash from medical waste incineration facility

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## ABSTRACT

In the present work, the stabilization/solidification of fly and bottom ash generated from incinerated hospital waste was studied. The objectives of the solidification/stabilization treatment were therefore to reduce the leachability of the heavy metals present in these materials so as to permit their disposal in a sanitary landfill requiring only a lower degree of environmental protection. Another objective of the applied treatment was to increase the mechanical characteristics of the bottom ash using different amounts of Ordinary Portland Cement (OPC) as a binder. The solidified matrix showed that the cement is able to immobilize the heavy metals found in fly and bottom ash. The TCLP leachates of the untreated fly ash contain high concentrations of Zn (13.2 mg/l) and Pb (5.21 mg/l), and lesser amounts of Cr, Fe, Ni, Cu, Cd and Ba. Cement-based solidification exhibited a compressive strength of 0.55–16.12 MPa. The strength decreased as the percentage of cement loading was reduced; the compressive strength was 2.52–12.7 MPa for 60% cement mixed with 40% fly ash and 6.62–16.12 MPa for a mixture of 60% cement and 40% bottom ash. The compressive strength reduced to 0.55–1.30 MPa when 30% cement was mixed with 70% fly ash, and to 0.90–7.95 MPa when 30% cement was mixed with 70% bottom ash, respectively.

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## 1. Introduction

Incineration, as a method of hospital waste management, generates solid residues, such as bottom and fly ash as well as off-gas cleaning residues with high levels of heavy metals, inorganic salts and other organic compounds. Many references in international literature characterize the bottom ash as either dangerous, not dangerous at all, or inert, in an effort to diagnose its proper management and disposal. According to bibliography, fly ash possesses a high content of heavy metals, dioxins and furans [1]. For this reason it requires special management. Bottom ash was only recently included (2003) on the list of dangerous waste materials according to the Council of the European Union, while fly ash and solid waste coming from the flue gas control system have already found their place on the list of dangerous waste materials with codes 19.01.13 and 19.01.07, respectively [2].

The limited space and the high cost of land disposal led to the development of recycling technologies and the reuse of bottom ash in structural and construction materials [3]. In this case the bot-

tom ash must be examined for its toxicity and all the necessary measures must be taken in order to minimize the leaching of its hazardous components into the environment [4]. Bottom ash and fly ash – generated from incinerated hospital waste – are used for the production of concrete and bricks, after mixing at a ratio of 50:50 with the remaining materials [5]. In several European countries high quantities of ash are reused for the manufacture of pavements, bridges and structural stones but also as a sublayer in the construction of motorways and as a daily cover for landfills. By contrast, while there is a general interest in USA and Canada for the potential utilization of such ash in construction materials thus is not a common practise until now.

In Germany, 50% of the ash produced from incinerated waste is used for the manufacture of sound insulation in walls along highways, as well as for sublayers of city roads. In the Netherlands, 60% of the bottom ash is used for the construction of asphalt and for a sublayer of roads. The aim is to reuse 80% of the bottom ash. In Denmark, over 72% of the ash is reused for the construction of parking lots, cycling tracks and other roads [6].

Technologies for bottom ash treatment are continuously scrutinized and controlled in order to minimize the potential for environmental pollution. In this paper, before the stabilization/solidification experiments were conducted, the nature of the fly ash and bottom ash was characterised by determining the heavy

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metals, particle size distribution, morphology and mineralogy of several samples [7].

## 2. Materials and methods

### 2.1. Materials

Several amounts of fly and bottom ash were sampled from a Medical Waste Incineration Facility (MWIF). This MWIF had a double lined feeding system which led to two rotary kiln systems with a capacity of 625 kg/h each. The operational temperature of the combustion chambers was recorded between 1100 and 1200 °C using an oxygen access of  $\lambda = 1.68$ .

The bottom ash was collected from the combustion chamber, while the fly ash was collected from the filter system (fabric filters) after cooling down to room temperature and lime pre-treatment. The weight of each sample was approximately 20 kg. The samples were collected from a pile of fly and bottom ash, respectively, according to the ASTM D6009 Standard Guide for sampling waste piles [8].

The fly ash particle size was below 120  $\mu\text{m}$  and had a greyish colour. The bottom ash was of black colour and its particle size ranged from 9.5 mm to 63  $\mu\text{m}$ . Only the particle size class below 2 mm was used for solidification/stabilization experiments with OPC.

In order to determine the nature of the fly and bottom ash, and to evaluate the efficiency of the applied technique, the following investigations were carried out before and after each solidification/stabilization process:

- Chemical composition—XRF analysis of fly and bottom ash and OPC;
- Morphology and mineralogical determination—XRD pattern of fly and bottom ash;
- Hardening time of the stabilized cement/ash matrix;
- Compressive strength test of the stabilized cement/ash matrix;
- Leaching test for heavy metals coming from the stabilized cement/ash matrix.

### 2.2. Chemical composition determined by X-ray fluorescence spectrometer

Atoms fluoresce at specific energies (keV) when excited by X-rays. The X-rays emitted are characteristic of the atom, and provide a qualitative identification of the element. Comparing the intensities (cpm) of the X-rays from an unknown sample to the measured values of known standards provides the basis for quantitative analysis of the element. The samples were prepared by grinding them down to a particle size <60  $\mu\text{m}$  and then pressing to a tablet. An S2 Ranger EDS (Bruker Ltd.) was then used for quantitative chemical analysis of fly ash, the bottom ash and the OPC sample.

### 2.3. Morphology and mineralogical determination by X-ray diffraction

X-ray diffraction (XRD) was utilized to determine the mineralogical properties of the fly and bottom ash samples. The bottom ash tablets (the test samples) were placed in a holder which were then placed in a Rigaku XRD machine with a copper target ( $\lambda = 15.406 \text{ nm}$ ). A diffraction angle ( $2\theta$ ) between 5° and 80° and a scanning rate of 4°/min was applied to analyse the crystal phases of the fly ash and bottom ash samples. Diffraction patterns were manually analysed using the Joint Committee on Powder Diffraction standards.

### 2.4. Stabilization/solidification process with OPC—sample preparation

During the stabilization/solidification process, fly and bottom ash were homogenized with OPC by using a blender for 2–3 min. Water was added slowly into the dry mix to promote hydration. Then the cement/ash mixture was mixed at high speed for 5 min. After the mixing procedure the matrix was placed into blocks and was let to dry. The blocks were left undisturbed for 24 h at room temperature (20–25 °C) and high relative humidity.

After defined periods of time, leaching tests were conducted and physical characteristics such as hardening time and compressive strength were determined for the stabilized matrix. In the present work four different ratios of cement/fly ash and cement/bottom ash mixtures were examined (60:40, 50:50, 40:60 and 30:70 w/w). Four replicates were analysed for each trial.

### 2.5. Hardening time

Hardening time was determined by visual observation and hand-pressing the stabilized cement/ash matrix specimens every hour.

### 2.6. Compressive strength test

The physical strength of the solidified matrix is significant since it determines the suitability of the solids to be used as construction material and for secure landfill stacking. Cubic test specimens (50 mm), prepared according to the protocols of the ASTM Test Method for Compressive Strength of Hydraulic Cement Mortars [9], were used for the compressive strength test. Total maximum loads were recorded at the point of fracture, and the compressive strength was determined using the formula  $f_m = P/A$ , where  $f_m$  is the compressive strength (MPa),  $P$  is the total maximum load (N) and  $A$  is the area of loaded surface ( $\text{mm}^2$ ).

Compressive strength was determined on duplicate specimens after 1, 7, 14 and 28 days of curing (98% room humidity, 25 °C) using a calibrated hand-operated hydraulic compression apparatus.

### 2.7. Leaching test for heavy metals

Landfill disposal of solid waste can lead to environmental impacts associated with the leaching of pollutants to surface and ground water. Therefore, leaching tests play a major role in assessing the possibility of landfilling within regulatory limits [6,10].

The Toxicity Characteristic Leaching Procedure (TCLP) is a soil sample extraction method for chemical analysis employed as an analytical method to simulate leaching through a landfill. The extract is analysed for substances appropriate to the protocol.

Manually crushed material (<1 cm) was leached using an extraction buffer of acetic acid and sodium hydroxide ( $\text{pH } 4.93 \pm 0.05$ ) at a liquid/solid ratio of 20:1 [11]. The extraction (at  $25 \pm 2$  °C) was performed by shaking the material for 18 h. Subsequently, the leachate samples were filtered through a 0.8  $\mu\text{m}$  borosilicate glass fibre filter, and the resultant TCLP extract (filtrate) was analysed for heavy metals using Inductively Coupled Plasma-Mass Spectrometer (ICP-MS) Agilent Technologies, model 7500cx. The laboratory quality control procedures included sample duplicates.

## 3. Results

### 3.1. Chemical composition of samples analysed by X-ray fluorescence spectrometer

EDXRF-Spectrometer analysis of fly and bottom ash samples before treatment revealed that the major elements of the investi-

**Table 1**  
Chemical composition of fly and bottom ash and OPC.

Chemical composition (wt%)	Fly ash	Bottom ash	OPC
SiO <sub>2</sub>	6.0	39.74	18.52
CaO	89.2	27.77	57.22
Al <sub>2</sub> O <sub>3</sub>	–	5.16	2.80
Na <sub>2</sub> O	2.5	9.13	9.04
MgO	1.0	2.92	2.74
Fe <sub>2</sub> O <sub>3</sub>	0.3	4.53	4.11
BaO	–	2.25	–
TiO <sub>2</sub>	–	2.24	–
SO <sub>3</sub>	–	1.36	3.12
K <sub>2</sub> O	–	0.49	1.32
ZnO	0.6	–	–
PbO	0.3	–	–
Other	0.1	4.41	1.13

gated fly ash were CaO (89.2%), SiO<sub>2</sub> (6.0%) and Na<sub>2</sub>O (2.5%), while the major elements of the bottom ash were SiO<sub>2</sub> (39.74%), CaO (27.77%), Na<sub>2</sub>O (9.13%), Al<sub>2</sub>O<sub>3</sub> (5.16%) and Fe<sub>2</sub>O<sub>3</sub> (4.53%), respectively.

The quantitative analysis of the fly and bottom ash samples, obtained by the X-ray fluorescence spectrometer, is shown in Table 1., together with the results for a 100% OPC sample.

Comparing the obtained data with previous studies [12–14] regarding the composition of fly ash generated, the content of CaO is very high due to its lime pre-treatment. With the exception of CaO, the results concerning the percentages of the other main oxides present in fly ash are in close agreement with the previous studies. The percentages of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> were substantially less.

Comparing the presented results with previous studies on the composition of bottom ash generated at another MWIF in Greece [7] and several other countries, such as Italy [15], Malaysia [16] and China [17], a complete agreement was observed concerning the type of oxides that were present in the bottom ash of MWIF. The results regarding the percentages of various oxides present in bottom ash are also in close agreement with the previous studies. The percentage of Na<sub>2</sub>O was higher in the bottom ash of this study, while the percentages of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> were again substantially less.

### 3.2. Morphology and mineralogical determination by X-ray diffraction (XRD)

According to X-ray diffraction analysis, the investigated fly ash sample had a highly complex mineralogy. The mainly crystalline compounds were calcite (CaCO<sub>3</sub>), halite (NaCl), quartz (SiO<sub>2</sub>) and anhydrite (CaSO<sub>4</sub>). This result is in total agreement with the literature [13,18–20]. In addition, the presence of crystal structures,

such as calcium chlorite hydroxide (CaClOH), zinc chloride hydroxide (Zn<sub>4</sub>Cl<sub>4</sub>(OH)<sub>4</sub>) and copper aluminium oxide (CuAlO<sub>2</sub>) were also verified.

On the other hand, the bottom ash sample contained a considerably high percentage of amorphous mass. As a result, a high background signal was present in the sample. The main components in this sample, according to the diffraction pattern obtained by XRD, were halite (NaCl) and hematite (Fe<sub>2</sub>O<sub>3</sub>). The presence of several oxides, such as calcite (CaCO<sub>3</sub>) and lime (CaO) were also verified.

Previous studies have demonstrated that the main minerals present in bottom ash are quartz, halite, anhydrite, anorthite, calcite, and hematite [14,18,20]. In conclusion, these results are in agreement with these studies.

The solidification process of the fly and bottom ash samples was observed using a scanning electron microscope (SEM). Fig. 1(a) shows an image of the fly ash sample before the solidification process while Fig. 1(b) shows an image of the solidified fly ash sample.

Fig. 2(a) shows an image of the bottom ash sample before the solidification process while Fig. 2(b) shows an image of the solidified bottom ash sample.

### 3.3. Hardening time

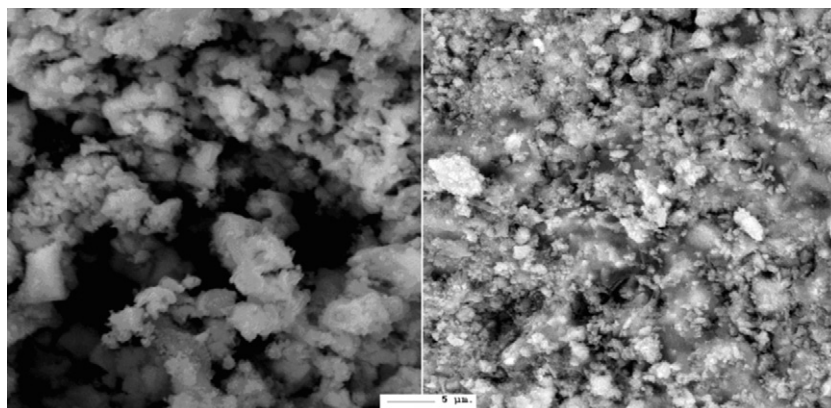
Hardening time ranged between 4 (cement with fly ash) and 7 h (cement with bottom ash). The cement control samples that did not contain fly or bottom ash loose longer and hardened within a day.

### 3.4. Compressive strength

Cement-based solidification samples exhibited a compressive strength of 0–16.12 MPa. The strength decreased as the percentage of cement loading was reduced, the compressive strength being 2.52–12.70 MPa for 60% cement mixed with 40% fly ash and 6.62–16.12 MPa for 60% cement mixed with 40% bottom ash, respectively. The compressive strength were reduced to 0–1.30 MPa when 30% cement was mixed with 70% fly ash and to 0.90–7.95 MPa when 30% cement was mixed with 70% bottom ash. The control sample of pure OPC recorded a compressive strength of 9.13–32.30 MPa.

In Figs. 3 and 4 the compressive strength values of the cement-based fly and bottom ash matrices after 1, 7, 14 and 28 days of solidification, respectively, are presented.

The compressive strength of the solidified matrix, including the control samples, increased with time. All values of compressive strength of the test matrices exceed the standard stipulated for solidified waste forms, which is 0.414 MPa after 28 days of solidification [21].



**Fig. 1.** Pictures from a scanning electron microscope of a sample of fly ash before (a) and after (b) the solidification process.

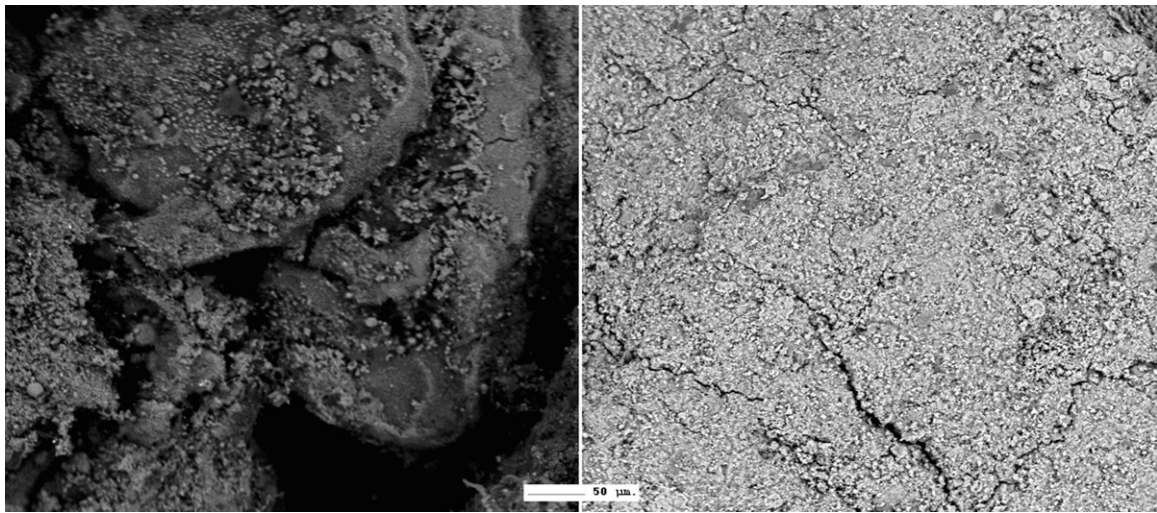


Fig. 2. Pictures from a scanning electron microscope of a sample of bottom ash before (a) and after (b) the application of the solidification process.

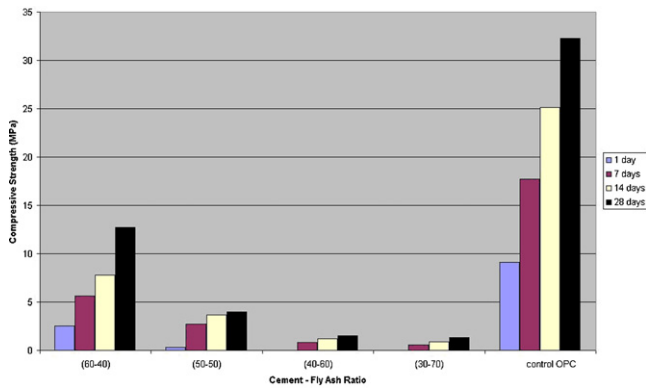


Fig. 3. Compressive strength of solidified fly ash with cement additive samples after 1, 7, 14, 28 days of solidification.

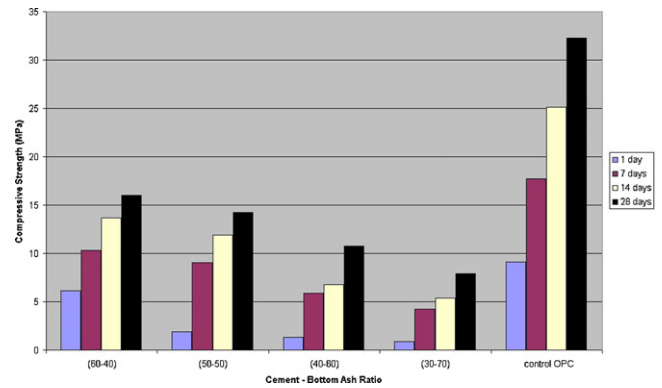


Fig. 4. Compressive strength of solidified bottom ash with cement additive samples after 1, 7, 14, 28 days of solidification.

Table 2  
Summary of TCLP leaching values after 1, 7, 14 and 28 days for solidified fly ash samples.

	Concentration of metals in leachate (mg/l)							
	Cr	Fe	Ni	Cu	Zn	Cd	Ba	Pb
Untreated fly ash	0.0855	0.8600	0.0762	1.0300	13.2000	0.0171	1.8404	5.2162
Cement	0.0144	0.4300	0.0803	0.0044	0.0657	<DL	0.1458	0.0012
Cement/fly ash								
60–40								
1st day	0.0676	0.6549	0.0643	0.3651	3.0231	0.0049	1.2264	0.8465
7th day	0.0523	0.4562	0.0614	0.3019	2.9651	0.0041	0.9562	0.6328
14th day	0.0494	0.3276	0.0567	0.2926	2.3549	0.0038	0.6961	0.5845
28th day	0.0481	0.3345	0.0446	0.3012	2.3718	0.0021	0.5154	0.3368
50–50								
1st day	0.0791	0.7592	0.0687	0.7937	2.9873	0.0097	1.4936	0.9581
7th day	0.0773	0.6439	0.0694	0.7651	2.3510	0.0074	1.4427	0.7541
14th day	0.0719	0.5821	0.0613	0.5619	2.4861	0.0051	1.1146	0.6734
28th day	0.0707	0.4254	0.0546	0.5507	1.7828	0.0056	0.9858	0.5244
40–60								
1st day	0.0686	0.7543	0.0609	0.7756	3.9138	0.0102	1.3595	1.0862
7th day	0.0675	0.6549	0.0563	0.7727	3.6902	0.0098	1.1204	0.9581
14th day	0.0653	0.3728	0.0401	0.6281	2.8904	0.0084	1.1124	0.6198
28th day	0.0562	0.3216	0.0469	0.5962	1.5200	0.0071	0.9873	0.3861
30–70								
1st day	0.0784	0.6734	0.0623	0.8952	5.0862	0.0099	1.3492	1.3993
7th day	0.0636	0.6198	0.0306	0.6742	4.8601	0.0065	1.2751	0.7829
14th day	0.0591	0.5932	0.0202	0.4265	3.1825	0.0044	1.0592	0.6332
28th day	0.0419	0.5234	0.0112	0.4358	2.8	0.0038	1.0130	0.5440

**Table 3**  
Summary of TCLP leaching values after 1, 7, 14 and 28 days for solidified bottom ash samples.

	Concentration of metals in leachate (mg/l)							
	Cr	Fe	Ni	Cu	Zn	Cd	Ba	Pb
Untreated bottom ash	0.1280	2.4012	0.6258	1.5500	0.1191	0.0006	2.4393	0.0050
Cement	0.0144	0.4300	0.0803	0.0044	0.0657	<DL	0.1458	0.0012
Cement/bottom ash								
60–40								
1st day	0.0996	1.3843	0.0982	0.4723	0.0598	0.0004	1.4134	0.0023
7th day	0.0663	1.0350	0.0973	0.3913	0.0479	0.0004	1.3963	0.0014
14th day	0.0592	0.6852	0.0962	0.3658	0.0403	0.0003	1.3952	0.0002
28th day	0.0567	0.5673	0.0932	0.3401	0.0255	0.0003	1.2067	<DL
50–50								
1st day	0.1095	1.5843	0.1169	0.4061	0.0093	0.0005	1.8825	0.0044
7th day	0.0992	1.0963	0.1046	0.4001	0.0073	0.0004	1.6538	0.0040
14th day	0.0898	0.8752	0.0834	0.3698	0.0061	0.0004	1.2745	0.0038
28th day	0.0495	0.7624	0.0763	0.3621	0.0056	0.0004	1.2145	0.0035
40–60								
1st day	0.1165	1.9851	0.1397	0.7782	0.1042	0.0006	1.8932	0.0004
7th day	0.1118	1.5438	0.1281	0.6831	0.0943	0.0006	1.2945	0.0002
14th day	0.1095	0.9531	0.1127	0.6023	0.0732	0.0006	1.0535	<DL
28th day	0.1091	0.8786	0.1071	0.5919	0.0703	0.0005	0.8997	<DL
30–70								
1st day	0.1259	2.0438	0.1824	1.2165	0.1008	0.0006	1.9842	0.0067
7th day	0.1232	1.8653	0.1239	1.1283	0.9252	0.0006	1.6320	0.0063
14th day	0.1242	0.9935	0.1048	0.7627	0.9423	0.0005	0.7392	0.0055
28th day	0.1161	0.9921	0.0973	0.5123	0.0935	0.0005	0.6894	0.0050

Comparing the compressive strength of the solidified matrix with previous studies [12,22], a general agreement was observed concerning the values of compressive strength of the test matrices and the decrease in strength as the percentage of cement loading was reduced.

### 3.5. Leaching test for heavy metals

The leaching test results for the solidified matrices show that cement addition was able to immobilize the heavy metals found in fly and bottom ash. The TCLP leachates of the untreated fly ash contained high concentrations of Zn (13.2 mg/l) and Pb (5.21 mg/l), and lesser amounts of Cr, Fe, Ni, Cu, Cd and Ba.

Among these metals, Zn concentration exceeded the regulatory limit for the TCLP test which is 5 mg/l. Cement-based solidification reduced all the metals in the leachate from the solidified matrices. The values for all the metals measured were within the permitted level for the TCLP test suggested by the US EPA. These results are in agreement with several other studies [12,19,22].

In Table 2 the TCLP leaching values after 1, 7, 14 and 28 days of solidification are displayed for the fly ash samples

The TCLP leachate from cement (OPC) alone also contained these elements in various proportions, in some cases exceeding the regulatory limit for the TCLP test. These metals originate mostly from natural raw materials used in the cement manufacturing process [23].

In Table 3 the TCLP leaching values after 1, 7, 14 and 28 days of solidification of the bottom ash samples are presented.

Reduction of heavy metals leaching potential was observed as a function of the stabilization time. Most of the samples showed significant stabilization rates of heavy metals from the 1st day of solidification. On the 28th day stabilization rates reached the maximum value for all samples. As was expected (optional), increase of cement/ash ratio in a sample also increased the stabilization rate of heavy metals in it.

## 4. Conclusions

This study demonstrates the feasibility of using fly and bottom ash in cement matrices in order to dispose them safely in non-hazardous landfills, or even to reuse these materials in the construction industry. Acceptable mechanical strength levels were reached for both types of ash after the 1st day, while an acceptable performance in the leaching test was reached too. TCLP conducted on all samples showed concentrations of heavy metals lower than the regulatory limits. Compressive strength tests, performed on the stabilized material after 28 days of solidification, revealed values which satisfy the regulatory limit of 0.414 MPa. More tests are needed to study the long-term behaviour (more than 1 year) of solidified samples to evaluate possible changes in the matrix and leachability of heavy metals, as well as time depending changes of medical wastes and their influence on the composition of ashes after incineration.

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