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# Properties of stabilized/solidified admixtures of cement and sewage sludge

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**Abstract**

The objective of this work is to investigate a viable alternative for the final disposal of sewage sludge from urban wastewater treatment plants by using it as an additive in order to develop new construction materials. For this purpose, several mixtures of sludge–cement–calcium chloride and calcium hydroxide were prepared and stabilized/solidified. Calcium chloride and calcium hydroxide were used as accelerating additives. X-Ray Diffraction (XRD) analysis as well as Thermogravimetry – Differential Thermal Analysis (TG-DTA) and Scanning Electron Microscopy (SEM) were used to determine the hydration products. The specimens were tested in order to determine their setting time and compressive strength after 28 days. Furthermore, in order to investigate the environmental compatibility of these new materials, Toxicity Characteristics Leaching Procedure (TCLP) and CEN/TS 14405 tests were carried out for the determination of heavy metals.


*Keywords:* Sewage sludge; Cement; Stabilization/solidification; Heavy metals; Setting time; Leaching test

## 1. Introduction

Stabilization/Solidification (S/S) is a widely accepted treatment process for the immobilization of hazardous substances, such as heavy metals contained in wastes. It entails mixing these materials with binders and reagents in order to reduce the leaching of contaminants. The most commonly used media in the S/S process is Portland cement [1].

The disposal of hazardous waste is a vital step of an effective plan of waste management. The main reason why the S/S treatment process is so widely accepted is because it immobilizes and stabilizes the hazardous substances (such as heavy metals that are found in the sewage sludge).

It has been found that the presence of organic materials in the sludge may cause some interference in the solidification process. This happens mainly because the organic material affect the cement setting [2].

Several authors have reported the interactions of many inorganic substances on the setting and mechanical properties of cement based systems [3].

It was found that metals could react with cement paste and affect the hardening and strength development during the early stage of cement hydration. Several types of interactions may occur simultaneously in the solidified systems. The waste component may react in one or more of the following ways: adsorption, chemisorption, precipitation, ion exchange, passivation, surface complexation, inclusions and chemical incorporation into the cement system [4].

Since heavy metals cannot be destroyed as is the case with organic contaminants there are two major options for dealing with heavy metals contamination: (1) Chemical

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transformation into a compound that reduces the potential hazard to tolerable levels for the human health and environment (stabilization) and (2) reduction of the metal mobility through physical encapsulation in a low-permeability material in the solidification process. Moreover, ettringite due to his needle-like structure, can develop extensive crystal interlocking, thus inducing significant strength gains when it forms. According to literature [5], it is suggested that heavy metal compounds bound themselves strongly with ettringite during its formation and are therefore permanently immobilized. It is also reported that the stabilized matrixes exhibited high strength values owing to ettringite formation.

The simple use of a binder for the S/S process can make it difficult to determine whether true stabilization has taken place. That is why a minimum compressive strength of 350 kPa at 28 days is recommended. It is also suggested that in order to determine whether the above process is successful, leaching tests should be carried out, with a target leachate pH value between 7 and 9. Furthermore, the concentrations of the heavy metals in this leachate should not exceed a minimum value. These values are determined for each heavy metal separately by governmental agencies [6,7].

The disposal of sludge from wastewater treatment presents highly complex problems to any municipality, due to increasingly stringent environmental regulations and industrial growth that have markedly increased the disposal requirements. Most of the sludge is disposed off by landfilling or spreading on reclaimed land, while incineration only reduces the volume of the sludge and the remaining residues would still require ultimate disposal in the landfill. All these disposal methods have varying degrees of environmental impact. Hence there is a need for alternative methods of sludge disposal to be developed. Various researchers into the possible applications of sludge have carried out studies as building and construction material. The reuse of wastewater sludge into construction materials not only alleviate disposal problems but also has economic, ecological and energy saving advantages. The properties and applications of the products derived from sludge and their potential applications as novel materials are discussed [8].

In the current work the waste to be treated is sewage sludge. Depending on its origin, sewage sludge can be harmful to the environment. Here we consider the possibilities of developing a construction material containing this waste.

The study consists of including wastewater sewage sludge in a building material bound with Portland cement–calcium hydroxide and calcium chloride as accelerating additives thus guaranteeing the stabilization and solidification of this waste because the wastewater sewage sludge is a waste with toxic potential [9].

The long-term safety of using sewage sludge in mortars needs to be reviewed. Due to the high organic content of the sludge and the presence of heavy metals, the durability of the specimens should be examined and for this reason long-term experiments have been programmed.

## 2. Experimental

Sewage sludge is mixed with various percentages of Portland cement, calcium chloride, calcium hydroxide, and sand.

### *2.1. Type of sludge*

The sewage sludge used is a mixed primary and secondary biological sludge generated in the Wastewater Treatment Plant of *Metamorphosis*, in Athens.

#### *2.1.1. Sludge characteristics*

The sludge was dried and its moisture content and pH were measured according to the standard methods [10]. The Total Organic Carbon (TOC) was determined by a titrimetric method [11] and the concentrations in heavy metals were measured by means of Atomic-Absorption Spectrometry (Perkin Elmer 3300) [12]. The results of the analyses are shown in Table 1.

From Table 1, it can be noted that the pH values measured were in the neutral area of pH. This was expected because the sludge is subjected to neutralization before its disposal.

#### *2.1.2. Grain size distribution*

Particle size distribution was measured by a laser scattering particle size distributor analyzer (CILAS). In Fig. 1, the grain size distribution of the sludge is shown. It can be remarked that the sludge has a cumulative percentage passing of 100% at 96 μm.

### *2.2. Other materials*

For the preparation of the referred specimens Portland cement type CEM I 42,5 was used as binder. Standard Sand (according to EN 196-1) was used as an aggregate [13].

Table 1
Sludge characteristics

| Parameters | Metamorphosis sludge |
|---|---|
| Moisture (%) | 74 |
| TOC (%) | 10 |
| pH | 7.00–7.50 |
| Heavy metals (mg/g)[a] | |
| Cr | 0.470 |
| Cu | 0.460 |
| Fe | 13.160 |
| Ni | 0.230 |
| Pb | 1.090 |
| Zn | 2.400 |

[a] Concentration of heavy metals (mg/g dry sludge).

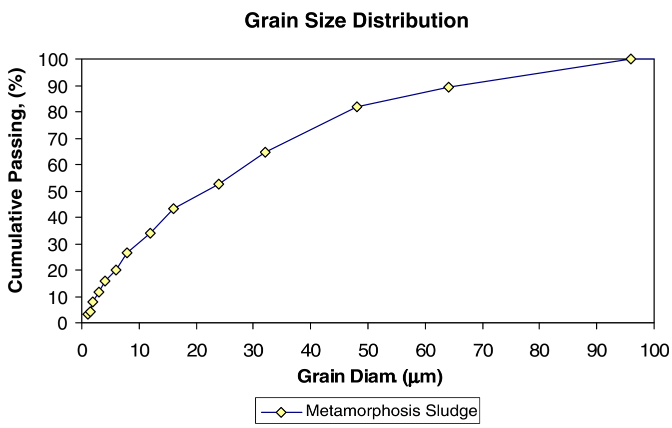


Fig. 1. Sludge grain size distribution.

Table 2
Calcium hydroxide characteristics

| $Ca(OH)_2$ (%) | $CaCO_3$ (%) | Specific weight ($g/cm^3$) |
|---|---|---|
| 89.1 | 5.98 | 0.47 |

Calcium chloride and calcium hydroxide were also used as accelerating additives (see Table 2).

*2.2.1. Sample preparation*

The mixing procedure was the same for all mortars. First the amount of sludge was mixed with the water and then the cement or the mixture of cement–calcium chloride and calcium hydroxide was added.

Various ratios of sludge solids/binder were used, from 0.30/1 to 0.55/1 [14]. The ratio 0.30/1 was used in order to facilitate the comparison of the samples and expresses the highest percentage of the use of cement. The amount of water was calculated according to previous work [14] so that the mixtures would be workable. Based on that, the composition of the mixtures produced is given in Table 3.

## 3. Results and discussion

### *3.1. Compressive strength results*

Various percentages of the sludge samples in wet condition were mixed with cement–calcium chloride–calcium hydroxide-sand and the $4 \times 4 \times 16$ cm specimens produced were cured and tested by determination of compressive strength according to the methods described by European Standard EN 196.1 [13].

The results of the compressive strength tests at 28 days are shown in Table 4.

From Table 4, it can be noticed that 4 of the 7 mixtures exceed the minimum limit compressive strength of 350 kPa at 28 days. Moreover the mortars with the lower amount of solids content have given the best results in compressive strength.

Table 3
Composition of the mortars

| Samples | Sludge/binder | Sludge solids/binder | Sludge (g) | Dry sludge | Cement (g) | $CaCl_2 2H_2O$/binder (%) | $Ca(OH)_2$/binder (%) | Sand (g) | Water (ml) |
|---|---|---|---|---|---|---|---|---|---|
| WM | 1.16/1 | 0.30/1 | 262.3 | 68.2 | 225 | 0 | 0 | 1350 | 180 |
| WM | 1.49/1 | 0.39/1 | 335.0 | 87.1 | 225 | 0 | 0 | 1350 | 180 |
| $WMC_1H$ | 1.49/1 | 0.39/1 | 335.0 | 87.1 | 222.75 | 1 | 2 | 1350 | 170 |
| $WMC_2H$ | 1.49/1 | 0.39/1 | 335.0 | 87.1 | 218.25 | 3 | 2 | 1350 | 155 |
| WM | 1.73/1 | 0.45/1 | 389.4 | 101.25 | 225 | 0 | 0 | 1350 | 175 |
| WM | 1.92/1 | 0.50/1 | 432.7 | 112.50 | 225 | 0 | 0 | 1350 | 110 |
| WM | 2.11/1 | 0.55/1 | 475.8 | 123.70 | 225 | 0 | 0 | 1350 | 62.5 |

M: *Metamorphosis*, W: Wet, $C_1$, $C_2$: Calcium chloride 1% and 3%.
H: 2% Calcium hydroxide.

Table 4
Compressive strength tests results

| Samples | Sludge solids/binder | $CaCl_22H_2O$/binder (%) | $Ca(OH)_2$/binder (%) | Compressive strength (kPa) |
|---|---|---|---|---|
| WM | 0.30/1 | 0 | 0 | 1426 |
| WM | 0.39/1 | 0 | 0 | 1000 |
| $WMC_1H$ | 0.39/1 | 1 | 0 | 1050 |
| $WMC_2H$ | 0.39/1 | 3 | 2 | 1100 |
| WM | 0.45/1 | 0 | 2 | 161 |
| WM | 0.50/1 | 0 | 0 | 115 |
| WM | 0.55/1 | 0 | 0 | 80 |

M: *Metamorphosis*, W: Wet, C1, $C_2$: Calcium chloride 1% and 3%.
H: Calcium hydroxide 2%.

Table 5
Results of speed setting process

| Samples | Sludge solids/binder | $CaCl_22H_2O$/binder(%) | $Ca(OH)_2$/binder (%) | Beginning of setting time (h) | End of setting time (h) |
|---|---|---|---|---|---|
| WM | 0.39/1 | 0 | 0 | 142 | 171 |
| $WMC_1H$ | 0.39/1 | 1 | 2 | 94 | 121 |
| $WMC_2H$ | 0.39/1 | 3 | 2 | 72 | 93 |
| $WMC_2HS$ | 0.39/1 | 3 | 2 | 40 | 60 |

M: *Metamorphosis*, W: Wet, $C_1$, $C_2$: Calcium chloride 1% and 3%.
H: Calcium hydroxide 2%, S: High alumina cement.

### 3.2. Setting time results

The inclusion of sewage sludge in the cement paste increases the setting time. For this reason it is recommended, depending on the applications, to add an accelerating additive such as $CaCl_2$.

To determine the effectiveness of the additive, tests were performed on various percentages of $CaCl_2$ in the cement pastes. Tests were also performed using High Alumina Cement.

The results of the speed of the setting process of the mortars according to the standard EN196.3 [15] are shown in Table 5.

Only specimens with a sludge solids/binder radio of 0.39/1 that showed satisfactory compressive strengths were investigated for setting times. From Table 5, the following remarks can be made:

- The inclusion of sewage sludge in the cement paste affects the setting (time) speed. For this reason, it is recommended, depending on the applications, to add $Ca(OH)_2$ and an accelerating additive such as $CaCl_2$.
- The setting time reduces as the percentage of $CaCl_2$ added increases.
- It is also observed that the sample containing High Alumina Cement presents a significant reduction in the setting time.

### 3.3. XRD Analysis results

XRD analysis results are given in Fig. 2 for the specimens that met the limiting compressive strength of 350 kPa. The pastes containing cement–sewage sludge *Metamorphosis*, $Ca(OH)_2$ 2% and $CaCl_2$ 3% with a sludge solids/binder ratio of 0,39/1 were studied.

The XRD analysis (Fig. 2) has shown that there are characteristic peaks of $Ca(OH)_2$.

$Ca(OH)_2$ is produced simultaneously with C–S–H when the reactions of $C_2S$ and $C_3S$ with water take place. The reaction of the hydration of $C_3S$ is fast and in the period of 28 days a great quantity of $C_3S$ is consumed, forming hydration products; finally, the formation of C–S–H occurs which, along with ettringite, leads to the hardening of the cement. $C_2S$ hydrates more slowly. $Ca(OH)_2$ is one of the main hydration products of the sample.

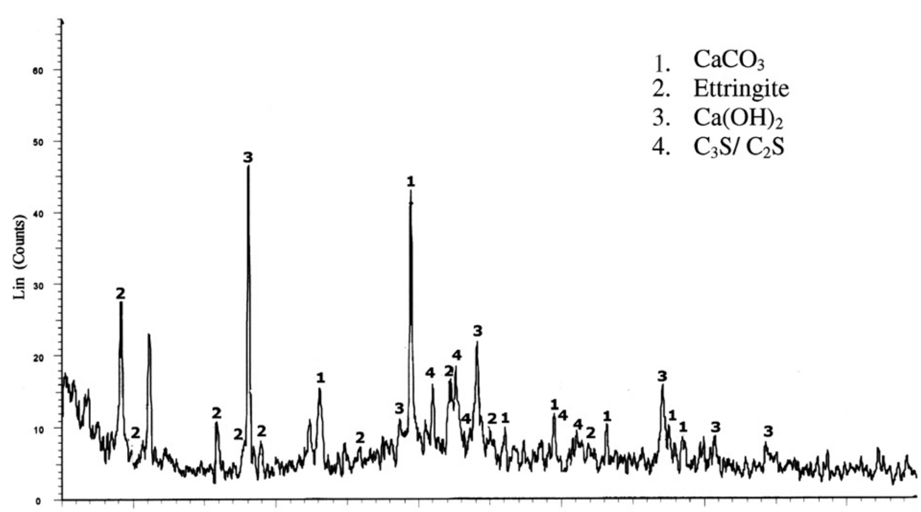


Fig. 2. X-ray diffraction–sample $WMC_2H$ (28 days).

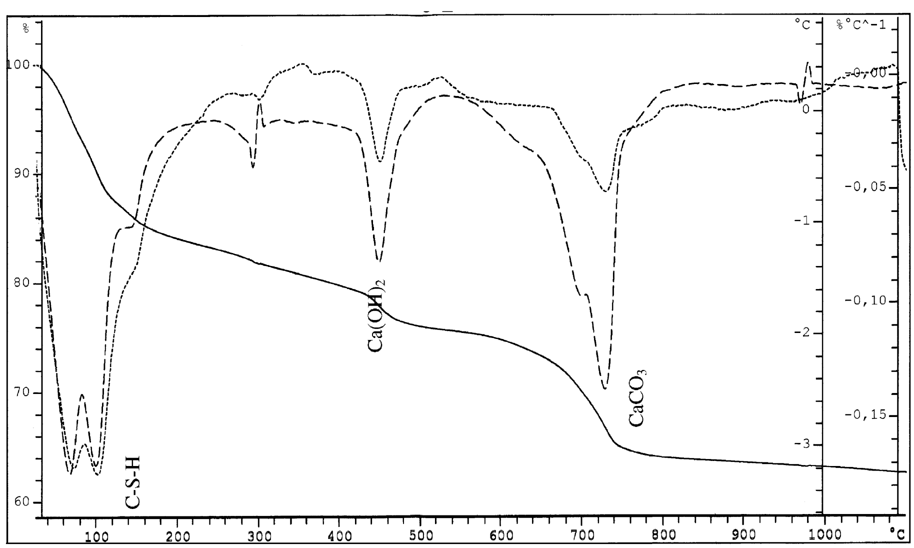


Fig. 3. TG – DTG – SDTA curves of sample $WMC_2H$ (28 days).

### 3.4. Thermal analysis results

Thermogravimetry – Differential Thermal Analysis (TG-DTA) was also used. The specimen containing sludge–cement-2% $Ca(OH)_2$ and 3% $CaCl_2$, with ratio sludge solids/binder: 0.39/1 were subjected to TG, DTG and SDTA.

As shown in Fig. 3, the curves can be divided into three major parts representing three different kinds of reactions:

Up to 300 °C: removal of water from hydrated products which are likely to include among others most of C–S–H.

The endo-peak at 450 °C is due to the dehydroxylation of calcium hydroxide which reveals the presence of $Ca(OH)_2$ according to the following reaction:

$$Ca(OH)_2 \rightarrow Ca^{2+} + 2OH^-$$

The endo-peak at 750 °C is due to the decarbonation of calcium carbonate

$$CaCO_3 \rightarrow CaO + CO_2 \uparrow$$

### 3.5. Scanning electron microscopy (SEM)

The study using a scanning electron microscope illustrated the details of the sample microstructure and confirmed the results of XRD analysis. XRD analysis (characteristic peaks in Fig. 2) and Scanning Electron Microscopy (characteristic needles in Fig. 4) both confirm the presence of the ettringite. In the next figure the most characteristic photos are presented, using the scanning electron Microscope Jeol JSM – 5600 with Oxford Link Is is 300.

In the photographs (Fig. 4), crystals of $Ca(OH)_2$ can be seen confirming the XRD analysis results as far as the hydration process is concerned, for the samples using cement as binder.

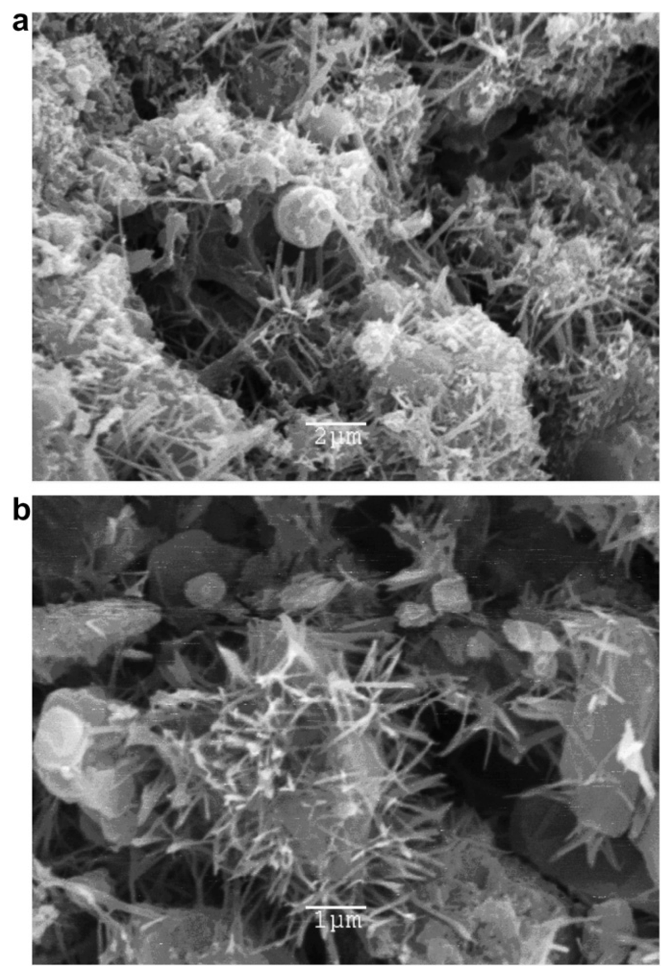


Fig. 4. SEM photographs of $WMC_2H$ sample: (a): 500X, (b): 1000X.

In the photographs, the needles of ettringite are characteristic. The ettringite fibers are also observed around the $Ca(OH)_2$ crystal. Ettringite is one of the main products of the cement hydration process.

Table 6a
TCLP test results and heavy metal retention percentage for *Metamorphosis* sludge

| Sample | C (%) | H (%) | Cu (ppm) | (%) | Fe (ppm) | (%) | Zn (ppm) | (%) | Ni (ppm) | (%) | Cr (ppm) | (%) | Pb (ppm) | (%) | pH |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| M | 0 | 0 | 2.43 | 0 | 9.40 | 0 | 4.20 | 0 | 2.22 | 0 | 0.30 | 0 | 7.40 | 0 | 7.40 |
| WM | 0 | 0 | 0.54 | 78 | 0.20 | 97.8 | 0.03 | 99.3 | 0.07 | 97 | 0.29 | 5 | 0 | 100 | 8.68 |
| $WMC_1H$ | 1 | 2 | 0.24 | 90 | 0.15 | 98.4 | 0.02 | 99.5 | 0.12 | 95 | 0.18 | 40 | 0 | 100 | 9.85 |
| $WMC_2H$ | 3 | 2 | 0.11 | 95 | 0.04 | 99.5 | 0.01 | 99.7 | 0 | 100 | 0.13 | 56 | 0 | 100 | 10.2 |

M: *Metamorphosis*, W: Wet, $C_1$, $C_2$: Calcium chloride 1% and 3%.
H: Calcium hydroxide 2%.

Table 6b
CEN/TS 14405 test results and heavy metal retention percentage for *Metamorphosis* sludge

| Sample | C (%) | H (%) | Cu (ppm) | (%) | Fe (ppm) | (%) | Zn (ppm) | (%) | Ni (ppm) | (%) | Cr (ppm) | (%) | Pb (ppm) | (%) | pH |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| M | 0 | 0 | 2.43 | 0 | 9.40 | 0 | 4.20 | 0 | 2.22 | 0 | 0.30 | 0 | 7.40 | 0 | 7.40 |
| WM | 0 | 0 | 0.42 | 83 | 0.17 | 98.2 | 0.013 | 99.7 | 0.01 | 99.5 | 0.06 | 80 | 0 | 100 | 12 |
| $WMC_1H$ | 1 | 2 | 0.22 | 91 | 0.02 | 99.8 | 0.004 | 99.9 | 0 | 100 | 0.085 | 72 | 0 | 100 | 12.5 |
| $WMC_2H$ | 3 | 2 | 0.17 | 93 | 0 | 100 | 0.04 | 99 | 0 | 100 | 0.081 | 73 | 0 | 100 | 12.8 |
| Limits[a] | | | 0.6 | | | | 1.2 | | 0.12 | | 0.1 | | 0.15 | | |

M: *Metamorphosis*, W: Wet, $C_1$, $C_2$: Calcium chloride 1% and 3%.
H: Calcium hydroxide 2%.
[a] EC Council Decision 2003/33/EC of 19 December 2002 "Establishing criteria and procedures for the acceptance of waste at landfills pursuant to Article 16 of and Annex II to Directive 1999/31/EC".

### 3.6. Leaching tests results

Leaching tests were carried out for the samples with wet *Metamorphosis* sludge–cement–$Ca(OH)_2$ 2% and $CaCl_2$ 1% and 3%. The TCLP [6] and CEN/TS 14405 tests [16] for heavy metal leachability were carried out only for the samples which complied with the minimum limiting value of compressive strength. The raw waste sample was also subjected to the TCLP extraction procedure and CEN/TS14405 tests in order to determine the amount of heavy metals that can be extracted from the sample.

The results of the TCLP and CEN/TS 14405 tests are given in Tables 6a and 6b.

- The heavy metals with a high percentage of retention are Pb, Zn and Ni.
- The TCLP and CEN/TS 14405 tests have shown high retention percentages of heavy metals in the cement phases.

This retention of heavy metal molecules in the hydrated Portland cement appears to be a combination of more than one process.

It is suggested that some of those are ionic adsorption to the hydrated C–S–H in the hydrated cement paste, ionic incorporation into the crystalline network of some compounds of the hydrated cement such as sulfates in the ettringite hydrate and, finally, physical retention in the porous structure. More investigation needs to be done in this field.

- It is observed that the sample containing sewage sludge–$CaCl_2$ 3% and $Ca(OH)_2$ 2% gives the best results of the TCLP and CEN/TS 14405 tests as far as the heavy metal retention is concerned. Furthermore, it can be concluded that the greater the percentage of the $CaCl_2$ added, the greater the percentage of heavy metal retention.
- Concentrations of heavy metals in the leachate of the CEN/TS 14405 tests were considerably lower than the limit values set by the European Council decision 2003/33 for the acceptance of a waste in an inert landfill.

## 4. Conclusions

The following conclusions can be drawn from this investigation:

1. Sewage sludge from Wastewater treatment Plants can be solidified and stabilized in a matrix of Portland cement.
2. Calcium hydroxide and ettringite, the main products of cement hydration, are characteristic products of the early ages of the curing process, where ettringite formation favors also the compressive strength.
3. It can be noted that the samples containing calcium chloride improve the compressive strength; therefore its addition has a positive effect. The best results were observed for the samples containing 3% $CaCl_2$ and 2% $Ca(OH)_2$.
4. The TCLP and CEN/TS 14405 tests have shown high percentages of retention of heavy metals in the hydrated cement phases. This retention of heavy metal molecules in the hydrated Portland cement appears to be a combination of more than one

process. This solidified sludge can be characterised as an inert material, as far disposal to a landfill is concerned.

5. It is observed that the sample containing sewage sludge–$CaCl_2$ 3% and $Ca(OH)_2$ 2% gives the best results of TCLP and CEN/TS 14405 tests as far as the heavy metal retention is concerned.
6. It is well known that the addition of $CaCl_2$ in substitution of the Portland cement in the paste reduces the setting time. It is observed that the optimum mix of additive $CaCl_2$ is 3% and $Ca(OH)_2$ 2% of the weight of cement. An addition of 3% $CaCl_2$ by the weight of cement reduces significantly the setting time of the cement pastes. A decrease in the setting time is observed however, it is not satisfying therefore further investigation needs to be done in this field.
7. As far as setting time is concerned, the sample containing $CaCl_2$ 3% and $Ca(OH)_2$ 2% shows the best results, where the behavior is quite similar to when High Alumina Cement is used.
8. Sewage sludge contains high organic substances and heavy metals, and when it is directly used in mortar samples, then their durability is concerned. Therefore further investigation needs to be done and long term experiments have been programmed.
9. Heavy metal compounds bound themselves strongly with ettringite during its formation and were therefore permanently immobilized therefore the stabilized matrixes exhibited very high retention of heavy metals and very low leaching concentrations owing to ettringite formation.
10. The ultimate goal of this study is the reuse of wastewater sludge into construction materials. From the first results of this investigation, this goal seems to be achievable. However, the final results from the long term experiments could give the definite answer to this question.

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