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The application of Solidification and Stabilization technology for the treatment of harbour sediments

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Summary

The aim of this study was to investigate the effect of cement on the geotechnical properties of contaminated harbor sediments with the use of S/S technology. Treatment techniques such as Solidification and Stabilization are becoming increasingly important in the contaminated land sector while they increase awareness of the environmental impact arising from its activities and resulting materials. This study was conducted on harbour sediments derived from Le Havre Harbour (France). The main objectives of this study were to determine the basic geotechnical and chemical characteristics of the harbour sediments, to mix harbour sediments with alcalosynthetic zeolites and ordinary Portland cement (OPC) at 5, 7.5 and 10 percent, by dry weight of the sediments, to determine the compressive strength of the stabilized sediments after curing for 7, 14 and 28 days and 90 days and to examine the leaching characteristics (in accordance with the norm EA NEN 7375:2004 protocol) of the stabilized material with emphasis to alcalosynthetic zeolites Heavy metal concentrations.

Keywords: Harbour Sediments; Solidification/Stabilization; Polluted sediments; Heavy metals; Mechanical properties; Leaching

1. Introduction

Shortages of natural resources in combination with the sustainable development approach adopted by several countries, led different domains such as civil engineering, agriculture, and manufacturing to plan and perform more efficient strategies in relation to the beneficial use of dredged sediments. Stabilization is a pre-landfill waste treatment process, which has been used for different types of industrial wastes, but is particularly suited to those containing heavy metals. The solidification/stabilization (S/S) process utilizes chemically reactive formulations that, together with the water and other components in sludges and other aqueous hazardous wastes, form stable solids. The material used for solidification/stabilization (S/S) not only solidifies the hazardous waste by chemical means but also immobilizes, encapsulates, destroys, sorbs, or otherwise interacts with selected waste components. The result of these interactions are solids that are non hazardous or less hazardous than the original waste.

In this context, this study is aimed at developing a solidified/stabilised material based on fine dredged sediments in combination with hydraulic binders. The amount of binders used ranged from 5% to 10% increasing thus the economic viability of contaminated harbour sediments treatment. To evaluate the influence of binders on the mechanical behaviour of the designed materials, mixes were treated with cement (at different proportions) and alcalo-synthetic zeolites, and compared. Moreover, the environmental impacts of the raw fine sediments and the

designed mixes are investigated by performing leaching tests.

2. Results

Between curing ages 7 and 14 days a decrease in the compressive strength of Mix Design No3 (10% cement) is observed. This could be characterized as an unexpected progression since the amount of Portland cement in that specific mixture is greater in comparison to the other two Mix Designs. The intensive decrease in the compressive strength of main specimens could have both a chemical and a physical explanation. The chemical reason, which is the main cause of an intensive decrease in compressive strength at the early curing ages of 7 and 14 days, could be attributed to heavy metal contamination.

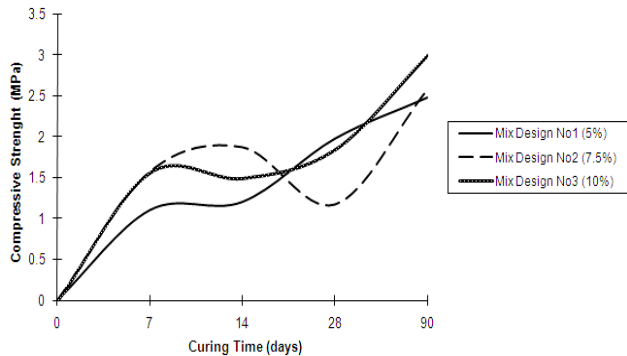


Figure 1. Compressive strength of specimens as function of curing time (days).

Extending the curing period of solidified sediments, especially after 28 days, the hydration reactions of the cement started and resulted in the formation of main products, which bear high compressive strength. However Mix Design No2 (7.5% of cement) presents an intensive decrease in its compressing strength from 1.87MPa to 1.17MPa. This value is even lower than the value obtained after the first stage of the curing period (7 days). This decrease could be attributed to the presence of heavy metal oxides, as mentioned earlier, however a physical explanation could also support this decrease.

3. Discussion

The increase of cement quantities resulted in an increase, in the appropriate mix designs, in strength as measured by the unconfined compression test. The final strength obtained is higher in Mix designs No3 and No2 respectively, and this was achieved during the maximum curing period (90 days). However, significant decreases in compressive strength were observed in both mix designs during the curing period. The presence of heavy metals acts as a hydration retarder and reduces the strength in Mix Design No2 and Mix Design No3 after 28 and 14 days respectively. In general, the strength increases as the percentage of cement increases and as the curing period increases. Mix Design No1 shows a homogeneous performance in strength increase and it is more efficient for landfill designated wastes.

In relation to leaching performance and consequently environmental impact, all mix designs studied in this project have shown to be capable of reducing heavy metal concentrations by up to 98%. Alcalo-synthetic zeolites in combination with Portland cement seem to be capable of incorporating heavy metals in the solidified matrix while cement at minimum quantities can produce a quick alternative to the problem of long-term storage of contaminated sediments in designed containment facilities. Finally, it cannot be assured that the overall effectiveness in heavy metal retention by the three (3) different mix designs will be maintained when applying different test conditions. On the basis of this study, the developed mixes could be studied with more polluted sediments in order to evaluate the efficiency of the proposed solution and the influence of other pollutants in the matrix.

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Keywords: Harbour Sediments; Solidification/Stabilization; Polluted sediments; Heavy metals; Mechanical properties; Leaching

1. Introduction

Sediments, which result from natural processes, are an essential, integral and dynamic part of river basins, estuaries and coastal zones. The primary sediment input into an estuary generally comes from the river, with the grain sizes potentially ranging from gravel through sand, to fine silt and clay. Another important sediment source is sand carried into the estuary from the marine environment (from the ocean beach) transported into the estuary by tidal currents flowing through the inlet. In some estuaries and bays this marine source of sediment can dominate over the river supply [1]. The mixing of river and marine derived sediments within the estuary is controlled by a number of different parameters such as the sediment transport resulting from the continued river flow, reversing tidal currents and the particular circulation pattern of water within the estuary. These processes vary hourly to seasonally and for this reason sediment distribution and composition is the result of a long-term integration of sediment-transport processes.

Navigation in waterways together with harbour access can only be ensured by dredging operations. Each year, several 100 millions of tons of materials are dredged around the world [2]. After dredging, traditional solutions such as ocean dumping are constrained by national and

international regulations. European directive number 75/442/CE (JOCE, 1975 [3]), classifies dredged sediments as waste under section 17 05 05 (polluted sediments) and section 17 05 06 (other sediments). Discharges of industrial and domestic waste water, shipping and agriculture are the main sources of pollution in harbour sediments. These activities can alter the initial sediment composition leading thus to a material containing harmful components such as organic compounds and heavy metals. Most organic pollutants are micro-biologically decomposed by different pathways, and which are finally converted into CO₂ and water. However, the behaviour and fate of heavy metals is governed by various physico-chemical processes, which dictate their availability and mobility in soil and sediment systems. Depending on their binding forms, sediment-associated metals like chromium, cadmium, zinc, copper, arsenic and lead, are more or less available for mobilization and subsequent uptake by plants and living organisms. Sediments containing a concentration of up to several hundred mg/kg of heavy metals represent one of the most challenging, contentious, and politically charged environmental issues involving heavy metal-contaminated materials. The extensive, worldwide contamination of riverine and harbour sediments contaminated by heavy metals and the large potential costs involved with the remediation of these materials has heightened debate amongst regulators, responsible parties, the scientific community, and community members [4].

Long-term storage of contaminated sediments in designed containment facilities does provide a relatively inexpensive remedial option. However, terrestrial disposal, is costly and requires large areas (Jones et al, 2005 [5]). For example, Brouwers *et al*, 2005 [6] indicates that ripening, which is the most common treatment technique for dredging material, has reached its limitations since it is time consuming (it takes 1.5 to 2 years for the soil to be suitable as a construction material) and also, it cannot cover the constant supply of dredging sludge. The development of beneficial use strategies for dredged sediments is therefore necessary, since it can provide affordable methods to remove the contaminants and beneficially use the decontaminated material.

Shortages of natural resources in combination with the sustainable development approach adopted by several countries, led different domains such as civil engineering, agriculture, and manufacturing to plan and perform more efficient strategies in relation to the beneficial use of dredged sediments. Stabilization is a pre-landfill waste treatment process, which has been used for different types of industrial wastes, but is particularly suited to those containing heavy metals [7]. The solidification/stabilization (S/S) process utilizes chemically reactive formulations that, together with the water and other components in sludges and other aqueous hazardous wastes, form stable solids. The material used for solidification/stabilization (S/S) not only solidifies the hazardous waste by chemical means but also immobilizes, encapsulates, destroys, sorbs, or otherwise interacts with selected waste components. The result of these interactions are solids that are non hazardous or less hazardous than the original waste.

Previous research on the use of solidified/stabilised raw fine sediments in construction has shown that treatment by hydraulic binders could satisfy the needed mechanical and chemical characteristics [2]. However, the proportion of hydraulic binders needed to meet prescribed specifications is important. For sediments from Le Havre Harbour (France), about 15% of a hydraulic binder was necessary [8]. However, the need for a large amount of hydraulic binder makes the use of raw dredged sediments unlikely from an economic point of view. Moreover, the presence of organic matters can constitute a problem with regards to cement hydration [8].

In this context, this study is aimed at developing a solidified/stabilised material based on fine dredged sediments in combination with hydraulic binders. The amount of binders used ranged from 5% to 10% increasing thus the economic viability of contaminated harbour sediments treatment. To evaluate the influence of binders on the mechanical behaviour of the designed materials, mixes were treated with cement (at different proportions) and alcalo-synthetic zeolites, and compared. Moreover, the environmental impacts of the raw fine sediments and the

designed mixes are investigated by performing leaching tests.

2. Materials and Methods

After description of the site from where the sediments are dredged, the basic characteristics of the studied materials are discussed and a developed methodology is implemented to identify mixes with optimum mechanical and environmental behaviour.

2.1 Site description

The marine sediments used in this study were dredged from Le Havre Harbour (Fig. 1). The port district extends 27 kilometres from west to east (from the breakwaters to Tancarville) and at its maximum point runs 5 kilometres from north to south. The port area of Le Havre includes the zones directly related to sea traffic and a vast industrial and port complex, covering approximately a 50 million m³ area. The environmentally-protected land covers a total surface area of more than 1,600 hectares, primarily lying within the nature reserve of the Seine



Figure 1. Location of Le Harve (left) and Le Havre Harbour (right).

estuary. This harbour is well known for its intensive industrial activities (e.g. petroleum, gas, and steel) and can process every type of cargo, whether in liquid or dry bulk format including hazardous waste and goods.

The dredged fine sediments presented in this study were obtained from the sea-bed at about 15 m in depth. Samples of the dredged materials were stored in sealed containers of 0.04 m³ in volume.

2.2 Characterization of Harbour Sediment

2.2.1 Chemical Characterization

In order to characterize the environmental impacts of the dredged sediments, leaching tests were performed on three samples from the same batch according to European test standard EN 12457-2 [9]. In the leaching tests, a liquid-to-solid ratio of ten (L/S 10) was adopted. Eluates from waste samples were analyzed via Inductively Coupled Plasma Mass Spectrometry (ICP-MS) and the average values from the three tests are shown in Table 1. According to the prescribed limits establishing the criteria and procedures for the acceptance of waste at landfills [10, 11], the disposal of the material on a terrestrial site would require treatment due to the high levels of Chromium, Zinc and Nickel.

Table 1. Analysis of physico-chemical elements in leachate after leaching test

Tests	ICRCL Trigger Values			After Leaching
As (mg/kg)			22	« 2 »
Cd (mg/kg)			12	« 4 »
Cr (mg/kg)			25	§ 131 §
Cu (mg/kg)			100	« 64 »
Ni (mg/kg)			130	§ 135 §
Pb (mg/kg)			300	« 101 »
Zn (mg/kg)			300	§ 433 §
References	Pollution Level			
European directive 2003/33/CE (JOCE,2003)	Low	Class III	Class II	Class I
		Inert waste	Non dangerous	Dangerous Waste
			« »	§§

2.2.2 Physical Characterization

The main physical characteristics measured in the dredged sediments are reported in Table 2. Using oven drying at 105°C, the measured initial water content was 66.03%. This high water content is typical of dredged sediments and depends mainly on the type of sediments, but also on the type of equipment used for dredging. The liquid limit (W_L) of the sediments, which represents the limit between the liquid state and the plastic state as measured by the percussion cup method, was found to be 78%, while the plastic limit (W_P), which represents the limit between the plastic state and the solid state as measured by the rolling test method, was found to be 31%. According to the plasticity index (I_p), defined as the difference between the liquid and the plastic limits, the sediments exhibit similar characteristics as very cohesive clays with a very low permeability [2]. The specific gravity, measured with the application of Proctor test was found to be 1680 kg/m³. This value was lower than values adopted for standard materials and this indicated the presence of organic matter in the sediments.

Table 2. Physical characteristics of Harbour Sediments

Parameters	Value
Water Content (%)	66.03
ρ_s (kg/m ³)	1680
Atterberg Limits	
W_L (%)	78
W_P (%)	31
I_P (%)	47

2.3 Mix designs

The experimental program for solidified and stabilized harbor sediments consisted of the combination of one primary binder (Portland cement-type I 35/A) at variable quantities (5%, 7.5% and 10% by dry weight) and one inorganic secondary binder (alcalo-synthetic zeolites) at a stable quantity of 1.3% by dry weight. Previous studies on heavy metal contaminated soil [12] indicated that heavy metal retention capability of alcalo-synthetic zeolites is optimised when used at range of 1.3% to 2.6% by dry weight of the contaminated soil. In order to enhance the

economic viability of the project, the minimum amount of secondary binder (1.3%) was adopted. Table 3 summarizes the mix design of the total 3 admixtures with Portland cement being the only variable component.

Table 3. Mixture proportions of each element

Mixtures	Contained Materials	Proportions	No of samples
Mix No1	Harbour sediments (g)	7196	7
	Portland Cement (g)	360 (5%)	
	Zeolites (g)	93.5 (1.3%)	
	Water (l)	1.8 (25%)	
Mix No2	Harbour sediments (g)	7196	7
	Portland Cement (g)	540 (7.5%)	
	Zeolites (g)	93.5 (1.3%)	
	Water (l)	1.8 (25%)	
Mix No3	Harbour sediments (g)	13746.6	7
	Portland Cement (g)	720 (10%)	
	Zeolites (g)	93.5 (1.3%)	
	Water (l)	1.8 litres (25%)	

In total 21 moulds of S/S treated material, were produced (7 moulds for each mix design) while the Unconfined Compressive tests and the Leaching tests were performed in triplicate. Following the production of the S/S material, samples remained in the mould for an additional 24 hours (solidification stage) and then were placed in transparent polyethylene bags in order to prevent moisture loss.

2.3.1 Unconfined Compressive Strength Test

The compressive strength of the cement mortar in solidified sediments was tested in a conventional method according to the standard ASTM D2166 [13] test method, using 100x80mm cylindrical moulds. Compressive strength of specimens was measured after 7, 14, 28 and 90 days, using a hydraulic type machine (Press MTS insight 100) for concrete testing with a loading rate of 1.0 k-N/s. Previous studies [14] indicated that in order for a material to be suitable for use in the construction industry a minimal strength of 0.34MPa is required, but this largely depends on the construction domain.

2.3.2 Leaching Test

The heavy metal retention capability (treatment performance) of each mix design was determined via the Static diffusion test [15]. In total 9 moulds of S/S treated material, were assessed for heavy metal leaching behavior. The solidified samples, were immersed in nine different clear plastic containers, with W (mm): 240, H (mm): 180 and L (mm): 290 dimensions, which were filled with distilled water at a Liquid to Solid (L/S) ratio equal to 5. According to the standard protocol, the water in each tank needed to be replaced eight times and at specified intervals. The times at which the water had to be replenished were 0.25, 1.0, 2.25, 4.0, 9.0, 16.0, 36.0 and 64.0 days. Before, the scheduled replenishments of water, leachant samples were obtained and the pH of the leachant was measured. The samples collected for chemical

analysis, were preserved after collection in Envirobottles and at a temperature between 18 and 22°C.

In total 72 samples were collected for further chemical analysis. 24 samples corresponded to each of the three different mix designs, which in their turn represented 8 samples for each selected S/S specimen. All sample leachates were filtered for the removal of fine suspended solids. In order to avoid metal precipitation from the dissolved liquid phase into the solid phase, which causes error in the accuracy of the metal concentration measurement, the pH of the samples was reduced to approximately 1.0 by the addition of concentrated nitric acid before the metallic analysis, because metals remain dissolved in pH less than 2.0. The presence of Zinc and Nickel in the leachate was measured by Flame Atomic Absorption Spectrometry (FAAS). In order to detect the presence of Hexavalent Chromium (Cr(VI)) in the eluates, UltraViolet-Visible (UV-Vis) Spectrophotometry was used. The spectrophotometric approach that was chosen relied on the specific reaction of Cr (VI) with 1.5-diphenylcarbazine (1.5-DPC).

3. Results and discussion

3.1 Compressive Strength Results

The compressive strength of the specimens is shown in Table 4, while Figure 2 gives a graphical representation of the results facilitating thus their interpretation. Between curing ages 7 and 14 days a decrease in the compressive strength of Mix Design No3 is observed. This could be characterized as an unexpected progression since the amount of Portland cement in that specific mixture is greater in comparison to the other two Mix Designs. The intensive decrease in the compressive strength of main specimens could have both a chemical and a physical explanation. The chemical reason, which is the main cause of an intensive decrease in compressive strength at the early curing ages of 7 and 14 days, could be attributed to heavy metal contamination [16].

Table 4. Unconfined compressive strength of the three Mix Designs (MPa)

Mix Design (Cement %)	Curing Time (days)			
	7	14	28	90
Mix No1 (5%)	1.10	1.20	1.97	2.47
Mix No2 (7.5%)	1.57	1.87	1.17	2.57
Mix No3 (10%)	1.57	1.50	1.85	3.00

Heavy metal oxides such as zinc, nickel, lead and chromium may cause the retardation of cement paste and prevent the formation of main cement hydration products such as Calcium Silicate Hydrates (CSH) and portlandite (Ca(OH)_2) crystals, which bear the mechanical forces [17] such as compressive forces in a solidified cement matrix. This could also be explained by the high alkaline environment of the eluates (pH=12). More specific in the case of Zn, the Zn hydroxy anions Zn(OH) and Zn(OH)_4^{-2} are reported to be present between pH 12 and 13. These anions are transformed into $\text{CaZn}_2(\text{OH})_6 \cdot \text{H}_2\text{O}$, which completely cover the cement grains and inhibits further hydration reactions [16, 17].

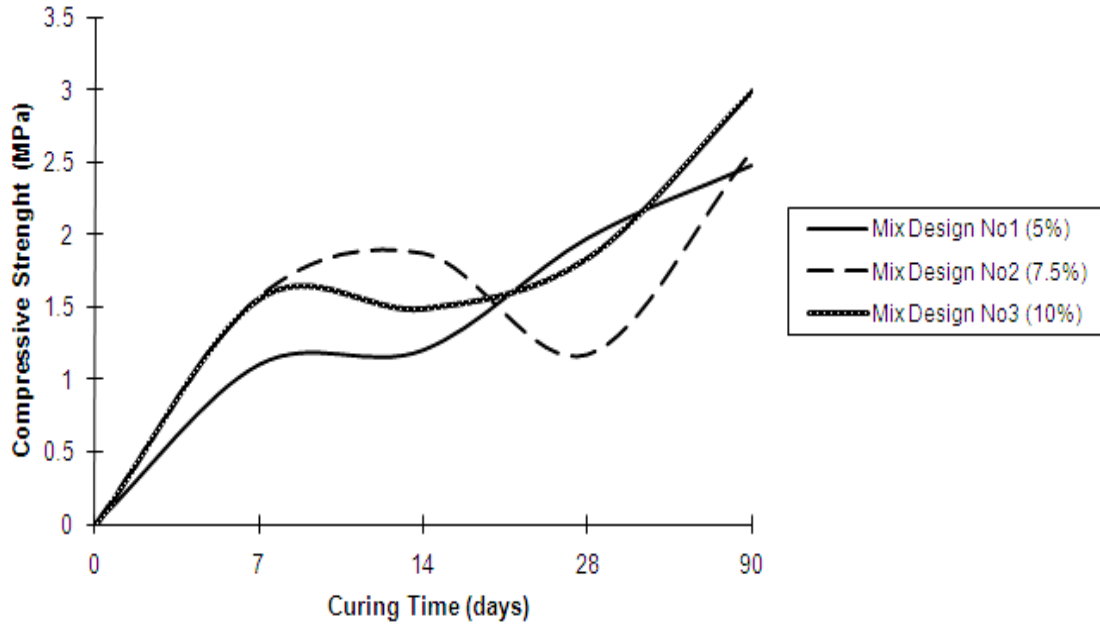


Figure 2. Compressive strength of specimens as function of curing time (days).

Extending the curing period of solidified sediments, especially after 28 days, the hydration reactions of the cement started and resulted in the formation of main products, which bear high compressive strength. However Mix Design No2 (7.5% of cement) presents an intensive decrease in its compressing strength from 1.87MPa to 1.17MPa. This value is even lower than the value obtained after the first stage of the curing period (7 days). This decrease could be attributed to the presence of heavy metal oxides, as mentioned earlier, however a physical explanation could also support this decrease. The presence of fine particles of soil with high specific surface areas, which might effectively reduce the amount of cement available for binding the fine and coarse aggregates required to provide adequate strength. In addition, soil might cover the surface of aggregates and prevent the adhesion of the cement paste and the aggregates [17]. The compressive strength of specimens cured for 90 days has increased in all Mix Designs, but negative effects, especially chemical effects, on cement hydration limited this increment strongly in Mix Design No2 and Mix Design No 3. The only Mix Design that shows a slow but progressive increase in compressive strength with no negative effects is Mix Design No1. This leads to the conclusion that effective and rapid treatment of contaminated sediment could be achieved with the application of Mix Design No1 (5% cement+1.3% of Zeolites). However, if the application of the solidified products extends into the construction industry then longer periods setting and curing time are required in order to achieve a satisfactory compressive strength performance. But before any application, the leaching results of the solidified soil should satisfy the environmental limits.

3.2 Leaching Results

Regulations and guidelines regarding heavy metal contaminated land have evolved as part of a more integrated environmental approach. One type of regulation seeks to contain the limit of addition of heavy metals in the soils, while the other imposes upper heavy metal concentrations in soils destined to receive wastes (landfill) and in soils designated for future development. It could be concluded that overlapping issues can easily arise among the different powers that control contaminated land since there is more than one regulator with concurrent powers. For the purposes of this study, the recommendation value of the Interdepartmental Committee on

the Redevelopment of Contaminated Land (ICRCL) together with the acceptance criteria for hazardous waste of the European Landfill Directive (99/31/EC) was adopted. However, this study is not aimed at performing a risk assessment research. If a risk assessment study is required, then information from this work could be used to derive a site specific soil guideline value.

3.2.1 Results

Figures 3 to 5 show Zn, Ni and Cr concentrations in leachates in the specific time intervals of the tank test.

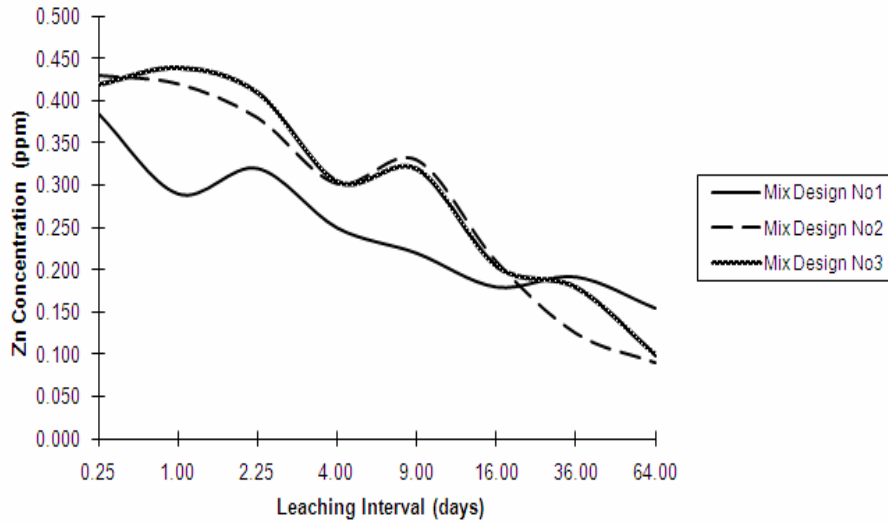


Figure 3. Relation between the amount of leached Zinc (ppm or mg/l) and test duration (days)

It should be noted that leaching tests were performed in each monolithic bloc after 28 days of curing. It is generally observable that extending the curing age of solidified soil decreases the metal concentration in leachates.

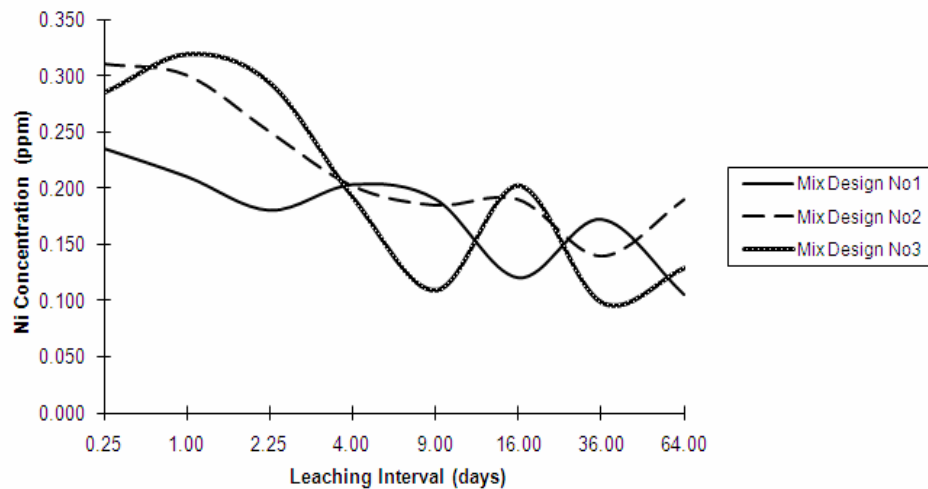


Figure 4. Relation between the amount of Nickel (ppm or mg/l) and test duration (days)

This indicates that the treatment has been enhanced by the hardness achievement and formation of main crystalline phases, which increased the acid neutralization capacity of the matrix and prevented metal solution in acidic leachants.

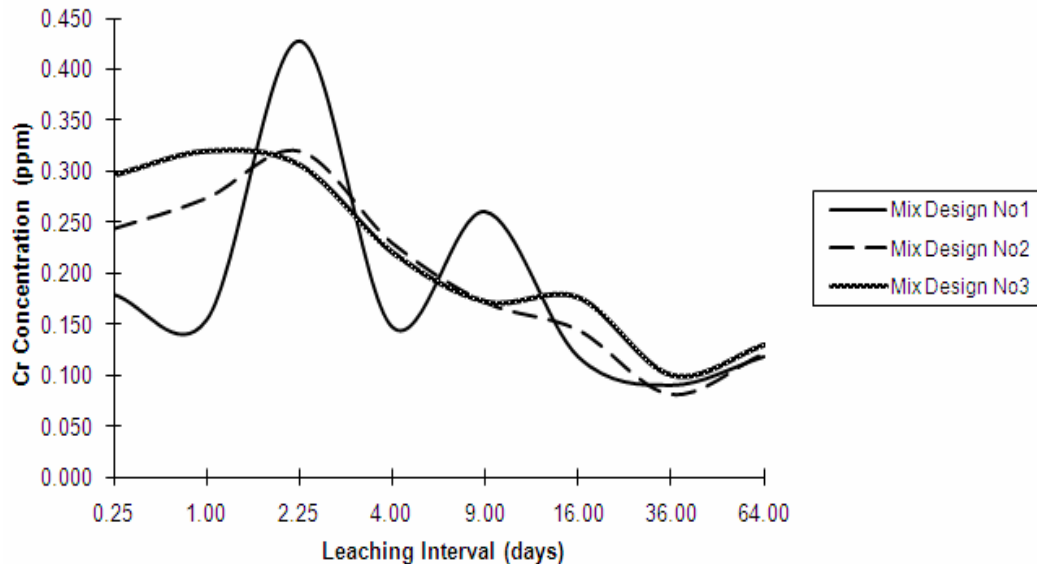


Figure 5. Relation between the amount of leached hexavalent chromium (ppm or mg/l) and test duration (days)

Additionally, due to aging (28 days), more hydration reactions took place and, as a result, more metals stabilized in the form of metal hydroxide precipitations, which presented themselves in the developing crystalline phases of hydrated cement or were absorbed into the CSH gel or zeolite crystals. As a significant result, the leaching concentrations of all metals at any curing age were considerably lower than ICRL and European regulatory limits according to Table 1.

4. Conclusion

The increase of cement quantities resulted in an increase, in the appropriate mix designs, in strength as measured by the unconfined compression test. The final strength obtained is higher in Mix designs No3 and No2 respectively, and this was achieved during the maximum curing period (90 days). However, significant decreases in compressive strength were observed in both mix designs during the curing period. The presence of heavy metals acts as a hydration retarder and reduces the strength in Mix Design No2 and Mix Design No3 after 28 and 14 days respectively. In general, the strength increases as the percentage of cement increases and as the curing period increases. Mix Design No1 shows a homogeneous performance in strength increase and it is more efficient for landfill designated wastes.

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