Budownictwo i Architektura 22(1) 2023, 25-35
DOI: 10.35784/bud-arch.3407
Received: 05.01.2023; Revised: 13.02.2023;
Accepted: 10.03.2023; Available online: 30.03.2023

Orginal Article

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Efficiency comparison of mixture formulations in the stabilisation/solidification of the loess silt contaminated with zinc in terms of mechanical properties

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Funding

This research was supported by “Fly ash as the precursors of functionalized materials for applications in environmental engineering, civil engineering and agriculture” no. POIR.04.04.00-00-14E6/18-00 project carried out within the TEAM-NET programme of the Foundation for Polish Science, co-financed by the European Union under the European Regional Development Fund.

Abstract: The effectiveness of various types of binders in stabilizing/solidifying (S/S) contaminated soils is strongly dependent on the type of soil and contaminants present. The literature abounds with studies of stabilisation/solidification of clayey soils, which provides a background for initial assumptions in design of the method application for contamination of this type of soil. However, studies on the stabilisation/solidification of loess silt contaminated with heavy metals are not available. Filling this deficiency is important in order to ensure the rapid adoption of the most effective remedies in case of contamination and their immediate implementation in the subsoil. This paper has enabled the determination of the most effective mixture among the examined for the remediation of loess silt contaminated with zinc in terms of compressive strength. Strengths were determined with the implementation of 30% Portland cement (2.63 MPa), 30% of fly ash-cement mixture (2.21 MPa), an incinerated sewage sludge ash-cement mixture (0.93 MPa) and mixtures in which cement was replaced by an MgO activator (0.18 MPa for fly ash and 0.63 MPa for incinerated sewage sludge ash). In addition, the determination of strength was carried out for samples containing a mixture of fly ash, activator and cement (0.26 MPa) and incinerated sewage sludge ash, activator and cement (0.26 MPa), with weight ratios of 5:4:1 respectively. In summary, fly ash and cement in a 2:1 ratio can be considered the most effective binding mix in terms of unconfined compressive strength increase.

Keywords: loess silt, alternative binder, activator, unconfined compressive strength

1. Introduction

Stabilisation/solidification (S/S) is a process that is recognised as a leading remediation method of soil contaminated with heavy metals [1]–[4]. The relatively low cost of its application [5]–[7], time efficiency [8], [9], and most importantly, effective immobilisation of contaminants while increasing the strength characteristics of the subsoil contribute to this. Implementation of various types of binders into the soil in the presence of water causes the hydration process to begin. Simultaneously, various chemical reactions take place, as a result of which heavy metals contaminating the soil precipitate in a less soluble and mobile form, undergo oxidation, chemical reduction as well as mechanical encapsulation in the soil matrix. The above processes immobilising heavy metals can occur individually or collectively, depending on the binder used and the type of contaminant [10]–[12]. Currently, the most commonly used binder in the S/S process, which is cement [13]-[23], has been replaced in significant part by various type of amendments. The reason for these developments is the well-recognised disadvantages of cement, including the uncertain long-term effectiveness of soil binding due to the sensitivity to ageing factors. The aforementioned factors mainly include sulphate erosion [24], the effect of acid rain [25], freeze-thaw cycles [26], drying-wetting and the usual reduction in properties resulting from the passage of time [27]. The negative impact of cement production on the environment manifested in the consumption of natural resources and the formation of a high carbon footprint [28]–[31] is also not ignorable. The above prompts the use of environmentally friendly substitutes for cement, especially in the form of waste materials with pozzolanic properties and various activators and additives that increase the efficiency of the S/S process. It should be kept in mind that no one type of binder can be considered the best for remediating all types of soil contaminated with any heavy metal [32]. In order to obtain the best possible results, binder and additive mixtures should be designed with appropriate proportions that are aimed at the type of soil and the contaminant present, based on existing knowledge from numerous studies reported in the literature [33]. Meanwhile, loess is a very under-researched soil for stabilisation/solidification. There are hardly any data in the publications on the results of studies on the leachability of heavy metals from treated subsoil of this type and its strength. One of the very few studies is the work of Akhter et al. [34], in which the authors determined the leachability of arsenic, cadmium, chromium and lead from loess stabilised with various types of binders, including fly ash, slag, lime and cement. Studies of the strength of stabilised loess [35]–[37], but without the crucial presence of heavy metal contaminants for stabilisation/solidification, do not compensate for this deficiency. Therefore, the authors of the present study attempted to perform stabilisation/solidification of loess contaminated with a selected heavy metal using cement and alternative green binders. The aim of the study was to evaluate the strength of the S/S product after 28 days of treatment using different types of binders and their proportions, as well as to select the most optimal mixture, in terms of mechanical properties. In addition, the performed analysis was used to establish the future research of zinc-contaminated loess silt.

2. Materials and methods

2.1. Raw materials

The selection of loess as the soil material for the stabilisation/solidification process was determined by the insufficient results of studies of this type in the worldwide scientific literature and the fact that the authors concentrated their attention on testing this type of soil in various geotechnical aspects. The loess (natural water content samples) for the tests was obtained from the area of the left-bank basin of the Bystrzyca River, near Czuby Południowe district in Lublin, from the depth of about 1.5 m below the surface. The location of the sampling point is shown in Figure 1.

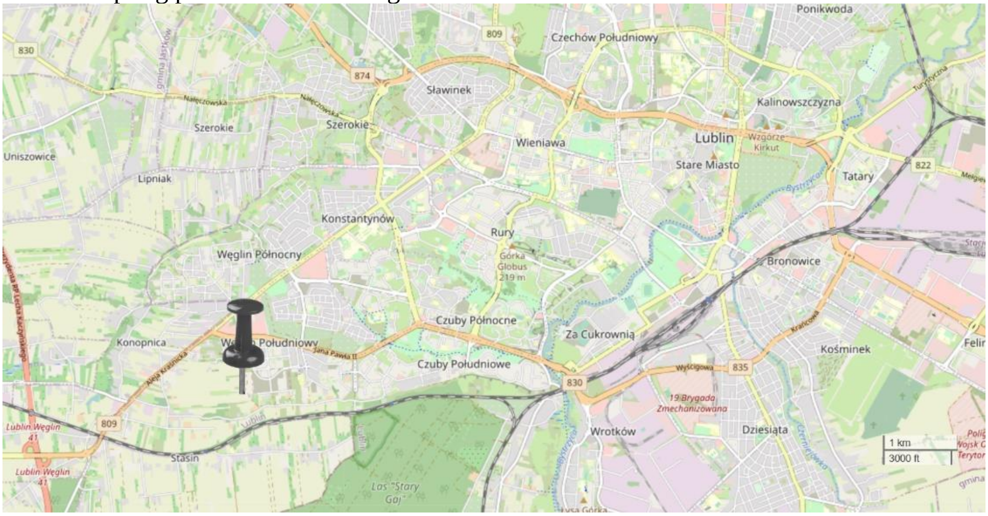

Fig. 1. Location of the loess sampling point [based on https://geolog.pgi.gov.pl/].

The loess used for the test (denoted as Si) represents a formation of aeolian facies [38]. According to the Unified Soil Classification System (ASTM (2011) D2487), the soil is classified as a low plasticity silty clay (SC-SM) occurring in the subsoil in a solid state, with the grain size and physical properties shown in Table 1. The properties were determined according to ASTM method guidelines [39].

Table 1. Engineering and geo-environmental properties of loess tested.

Property	Values measured	Reference for measurement method
Specific gravity ρ_s [g/cm^3]	2.66	ASTM D854
Plastic limit w_P [%]	23.3	ASTM D4318
Liquid limit w_L [%]	29.8	ASTM D4318
Grain size distribution [%][a]		ASTM D422
Clay (< 0.002 mm) [%]	2.24	
Silt (0.002 – 0.063 mm) [%]	78.64	
Sand (0.063 – 2.0 mm) [%]	19.12	
Soil classification	SC-SM	ASTM D2487
Optimum moisture content [%]	11.82	ASTM D698

The most common heavy metal in contaminated soils [20], while also occurring in the highest concentrations in the natural subsoil [40], is zinc. It represents one of the most soluble metals, with ions characterised by high mobility [41]. Although an essential nutrient for living organisms, it has been shown to be toxic in excessive concentrations [42]. For these reasons, this type of contaminant, introduced in the form of zinc chloride ($ZnCl_2$) of analytical grade purchased from WarChem, was adopted for the study of loess stabilisation/solidification. The assumed zinc concentration was 0.2% of the dry weight of the soil, i.e. 2000 mg/kg. The feed of the contaminant into the soil-binder mixture was in the form of a solution of $ZnCl_2$ inserted in distilled water. The binders used were CEMI cement of 42.5R class (marked with the symbol PC), fly ash from a thermal power station located in Gdańsk (labelled with the symbol FA) and incinerated sewage sludge ash obtained from a sewage treatment plant in Kraków (denoted with the symbol ISSA). The activator used was magnesium oxide (MgO, in mixture formulations denoted as M). The chemical composition of all materials is listed in Table 2.

Table 2. Main chemical compositions of the loess, used binders and activator.

	Percentage in weight [%]									
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	P_2O_5	SO_3	TiO_2	LOI
Soil	72.47	8.26	2.84	4.62	1.22	2.90	nd	nd	0.69	3.77
PC	15.39	2.73	4.18	69.11	0.35	0.57	nd	3.62	0.26	3.28
FA	50.00	20.36	8.00	4.56	1.16	1.89	0.58	1.23	1.44	10.36
ISSA	31.60	6.29	19.62	12.11	3.12	2.11	18.68	1.70	1.17	2.28
MgO	0.99	0.38	12.04	9.42	71.58	nd	nd	0.57	nd	4.27

* LOI: Loss of ignition; nd: not detected.

A control sample, i.e. loess silt contaminated with Zn not treated with S/S, and test samples to undergo stabilisation/solidification process were prepared for the study. The dry weight addition of binding materials and additives to the dry weight of the soil was determined at 30%. Due to the fact that binder and additive content of the soil dry matter strongly influences the unconfined compressive strength of cured soils further optimisation of the soil/binder ratio need to be performed in future S/S studies. Soil-cement mixes at a ratio of 7:3, soil-cement-ash mixes at a weight ratio of 7:1:2 and soil-ash mixes with an activator at a ratio of 7:2:1 were used for comparison. In addition, a soil-ash mix with an activator and 3% cement content was prepared. The symbols of the samples along with their compositions are summarised in Table 3.

Table 3. Mixture formulations (wt%) of soil, binders and activator samples

	Percentage in weight [%]				
	Loess	PC	FA	ISSA	MgO
100Si	100	–	–	–	–
FA_M_P	70	3	15	–	12
ISSA_M_P	70	3	–	15	12
FA_M	70	–	20	–	10
ISSA_M	70	–	–	20	10
FA_P	70	10	20	–	–
ISSA_P	70	10	–	20	–
30PC	70	30	–	–	–

The mixtures shown above were prepared according to the procedure described in section 2.2 Sample preparation.

2.2. Sample preparation

Materials adopted for testing were dried to a constant weight at 105°C for min. 24 h. After drying, the loess was grounded in a ball mill and then sieved through a 2.0 mm screen. The obtained loess silt was combined with selected binders in the weight proportions described in Table 3, and then manually stirred until a homogeneous mixture was obtained. A zinc chloride solution was fed into the mixtures at the assumed concentration until a moisture content of 15% was achieved. All ingredients were again mixed manually until a homogeneous paste was attained. The resulting material was placed in cylindrical moulds (38 mm in diameter and 76 mm high) in three layers with compaction to achieve a final uniform bulk density of the soil skeleton of 1.55 g/cm^3. After compaction and weighing, the samples were demoulded using a hydraulic press, immediately wrapped in foil and placed in a chamber with a constant temperature of 22°C (±1°C) and humidity of 95% (±1%). The process of stabilisation/solidification of zinc-contaminated loess silt should be considered to have started exactly when the sample was placed in the environmental chamber. The samples were cured for 28 days, which is the most commonly curing time assumed in the literature for comparison purposes. Nevertheless, due to the large influence of the time factor on the final strength of the S/S product, there is a need to analyse the influence of time for the selected mixture formulations in further studies. The above-described procedure, shown in the diagram in Figure 2, was repeated for all presumed formulations. All samples were prepared in quadruplicate.

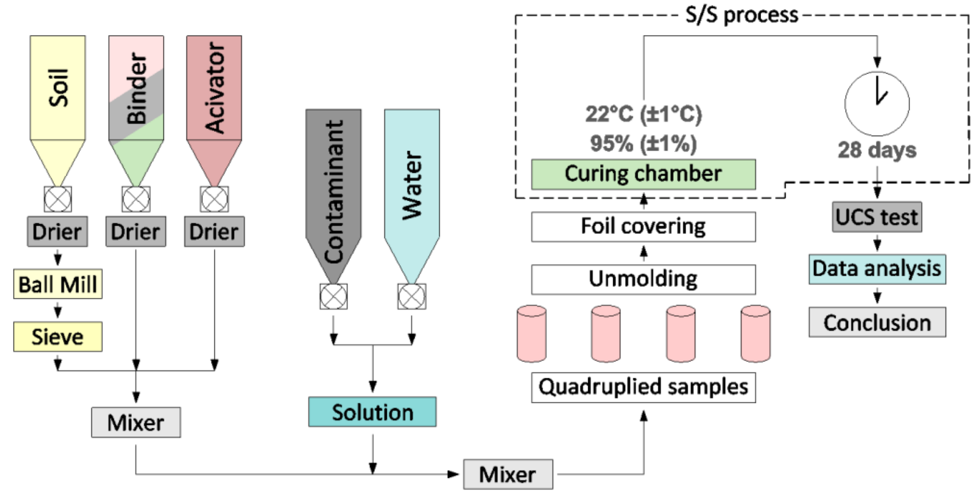

Fig. 2. A scheme of the testing procedure including sample preparation, treatment process and compilation of results.

2.3. Test methods

A half hour before the test commenced, the specimens were removed from the climatic chamber and stripped of their foil cover.

The unconfined compression test (UCT) was carried out using the MTS 809 Axial/Torsional Test System at a constant shear rate of 0.2 mm/min, which equates to approximately 0.26 %/min. Each mixture was prepared and tested in quadruplicate, resulting in a final 32 examined samples. After testing, the destroyed specimens were dried to constant weight to determine the change in moisture content, which decreased from the initial moisture content by 0.8 - 1.4%. Due to the relatively small volume of the samples, it was very important to strictly adhere to the preparation procedure. Exposure to air for an excessive period of time without foil film before being placed in the humidity chamber could have resulted in a reduction in moisture content in the near-surface sections of the sample, with a consequent disruption of the hydration process and subsequent reduction in compressive strength.

3. Results and discussion

3.1. Unconfined compressive strength

The performed tests resulted in the compressive strength of the S/S process products using various types of binders after 28 days of treatment. As shown in fig. 3 the obtained values reveal considerable variation.

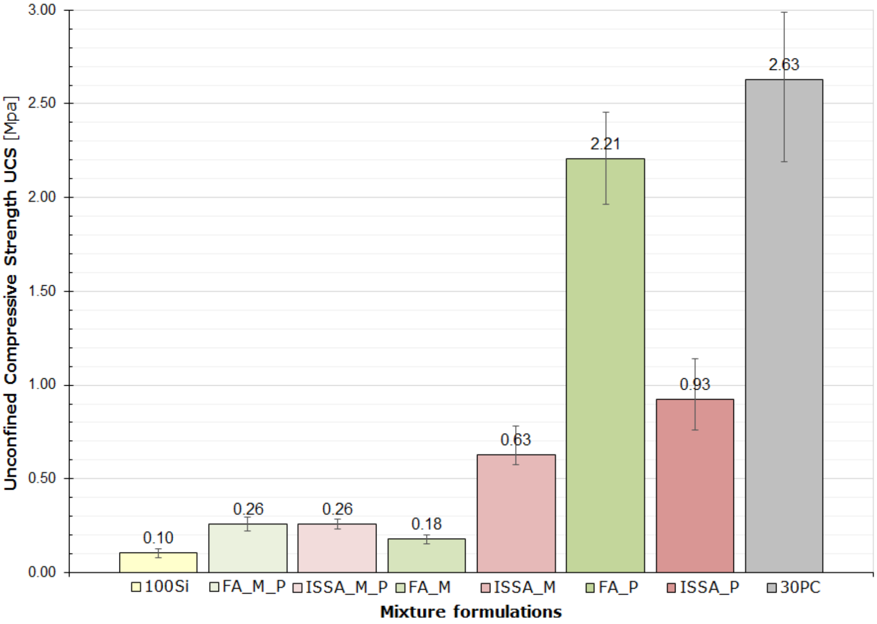

Fig. 3. Unconfined compressive strength of the mixture formulations with use of different binders mixtures after 28 days of stabilisation/solidification process.

All applied mixtures have shown a higher unconfined compressive strength than the untreated soil contaminated with zinc. Product of the S/S process with use of cement presented the highest values, with average of 2.63 MPa. Samples treated with 10% of cement and 20% of fly ash have also demonstrated competitive values, with average of 2.21 MPa. The mixture composed of 70% contaminated soil, 20% ISSA and 10% cement (ISSA_P) showed a strength 58% lower than the FA_P mixture, reaching an average UCS value of 0.93 MPa. In light of the aforementioned strengths of the soil-ash-cement mixtures (30PC, FA_P and ISSA_P), the results of the soil-ash mixtures with 10% MgO activator appear surprising.

The ISSA_M mixes, in which the activator was combined with incinerated sewage sludge ash, have shown a strength 32% lower than the ISSA_P mixes, but at the same time up to 71% higher than the FA_M mixtures. The FA_M blends, composed of 70% soil, 20% fly ash and 10% MgO achieved an average UCS strength of only 0.18 MPa, which is less than 2 times the strength of the zero samples. Samples composed of soil, 15% green binder (FA or ISSA), 12% MgO and 3% cement (i.e. FA_M_P and ISSA_M_P) have indicated an average UCS value of 0.26 MPa.

3.2. Stress-strain behaviour.

By analysing the strain-stress relationship curves, it is possible to determine the type of behaviour of the materials in terms of their brittleness. Figure 4 depicts the stress- strain curves of all tested mixtures.

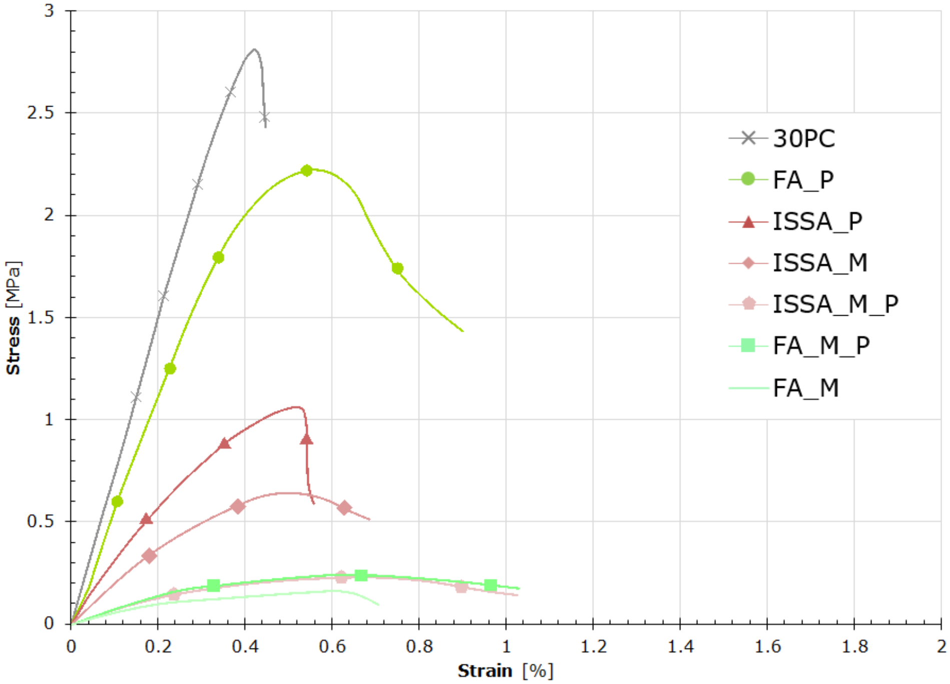

Fig. 4. Stress-strain curves of S/S product with use of different binders mixtures.

It should be noticed, that mixtures present different stress-strain behaviour. The most brittle are mixtures of loess silt with PC and with a composition of incinerated sewage sludge ash and cement. This can be observed in rapid post-peak stress decreasing with any increase in strain. Conversely, mixtures containing MgO are proving to be ductile materials, regardless of the other components. Mixes containing fly ash also exhibit ductility, although in the combination with cement creep is not prominent.

With regard to the results obtained, there is a need to repeat the tests with the ISSA_M and FA_M mixes, which showed disproportionate variations in strength with respect to the ISSA_P, ISSA_M_P, FA_P and FA_M_P samples. In order to accurately analyse the results obtained, it is also necessary to determine the chemical processes occurring during stabilisation/solidification with the individual mixes. This will enable the strength results of the S/S product to be contrasted in the light of microstructural effects. In addition, it is necessary to determine the leachability of zinc from the soil after 28 days of treatment, which, in addition to geomechanical parameters, is the essential part of a stabilisation/solidification process. The introduction of ageing factors (freeze-thaw, drying-wetting, sulphate attack cycles) is also required to determine the long-term effectiveness of the stabilisation/solidification of all designed mixes.

4. Conclusions

The article provides a summary of basic and mechanical laboratory studies carried out for loess silt contaminated with zinc subjected to a stabilisation/solidification process. For the purpose of remediation, the contaminated soil was mixed with different types of binders and the optional use of an activator. According to the results obtained, the following conclusions can be drawn:

1. The highest unconfined compressive strength was demonstrated by contaminated loess silt samples treated with cement. Competitive values were achieved by mixtures with replacement of 2/3 of the cement with fly ash. The unconfined compressive strength obtained were 2,63 MPa and 2,21 MPa respectively.
2. The use of incinerated sewage sludge ash for the S/S process of zinc-contaminated loess silt has been found to be less effective. With combination of cement (in weight ratio 2:1) achieved UCS value was 0,93 MPa.
3. The composition of ISSA and MgO has proven 3,5 times more efficient than FA and MgO in the unconfined compression test performed.
4. The addition of 10% cement per dry weight of all soil additives resulted in an increase in strength for the FA and MgO samples, but a parallel decrease for the ISSA and MgO samples. The strength of the FA_M_P and ISSA_M_P mixtures averaged 0.26 MPa in both cases.
5. A good correspondence between ISSA and MgO is apparent, and at simultaneously a poor one between FA and MgO. On the other hand, the mechanical properties in the presence of FA are very positively affected by even a minor addition of cement.
6. In terms of unconfined compressive strength with environmental consideration, fly ash and cement in a 2:1 ratio can be regarded as the most effective binder mixture in the light of the tests performed. The reasonably good results of mixtures using incinerated sewage sludge should also be highlighted.

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