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Research Paper

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Zagreb clay improved with various binders

The paper presents the results of laboratory testing of clay samples from two localities in the city of Zagreb, mixed with fly ash, slag and cement. Under laboratory conditions, composite samples of clay mixtures with binders were prepared, where the binders are added to the clay in ratios of 5 %, 10 % and 20 %, and tested at different maturation time intervals of 7, 14 and 28 days. The influence of different type and amount of binder and age of the composite sample on moisture and uniaxial compressive strength was examined and analysed. The obtained results show that the utilization of cement, fly ash and slag can improve the physical and mechanical properties of Zagreb clay, depending on the amount of binder and the maturation time of the composite.

Key words:

Zagreb clay, fly ash, slag, cement, soil improvement, moisture, uniaxial compressive strength

Prethodno priopćenje

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Zagrebačka glina poboljšana različitim vezivima

U radu su prikazani rezultati laboratorijskog ispitivanja uzoraka gline s dva lokaliteta u gradu Zagrebu, pomiješane s letećim pepelom, zgurom i cementom. U laboratorijskim uvjetima su pripremljeni kompozitni uzorci mješavina gline s vezivima koji su u omjerima od 5 %, 10 % i 20 % dodani glini, te ispitani u različitim vremenskim intervalima sazrijevanja od 7, 14 i 28 dana. Ispitan je i analiziran utjecaj različitog tipa i udjela veziva te starosti kompozitnog uzorka na vlažnost i na jednoosnu tlačnu čvrstoću. Dobiveni rezultati pokazuju da se primjenom cementa, letećeg pepela i zgure mogu poboljšati fizikalna i mehanička svojstva zagrebačke gline, u ovisnosti o količini veziva i vremenu sazrijevanja kompozita.

Ključne riječi:

zagrebačka glina, leteći pepeo, zgura, cement, poboljšanje tla, vlažnost, jednoosna tlačna čvrstoća

1. Introduction

Cement and lime are still dominantly used in geotechnical practice as traditional binders for improving quality of fine-grained soil, the aim being to enhance mechanical properties such as strength and stiffness of soil, to reduce plasticity, moisture or potential shrinkage / swelling of soil, to increase bulk density of soil, etc. [1-3]. However, the potential for use of industrial waste materials, such as fly ash and slag, as a means to ensure sustainable improvement of soil, is becoming increasingly evident, especially in the deep soil mixing methods, where machines are used to break the structure of soil and then mix the soil with an appropriate binder [4], and also in shallow stabilisation of soil in transportation engineering. In fact, great quantities of natural raw materials and fossil fuels are currently consumed to enable production of standard binding materials, cement and lime. Huge quantities of CO₂ are released into the atmosphere as a result of chemism, i.e. decarbonisation of CaCO₃. The cement and lime participate with 2-5 % by mass in the soil improvement procedure. Any reduction in the consumption of cement and lime would certainly contribute to lower consumption of natural raw materials and fossil fuels, and hence to the abatement of CO₂ emissions. For instance, to improve one tonne of soil it would be necessary to consume at least 20 kg of cement, 30 kg of natural raw material, and 96 MJ of fossil fuel. At that, 18 kg of CO₂ is released into the atmosphere [5]. The consumption of cement and lime as binding agents can be reduced by using waste materials from other industrial processes [6, 7]. The trend of reusing industrial waste as secondary raw material in soil improvement has increasingly been present for quite some time. This is due to two reasons: on the one side, the price of natural resources is increasing because of their rapid consumption and, on the other side, the industrial waste disposal sites are expanding at high rate which is why a necessity to recycle such materials is prominent [8].

Considerable research has been conducted worldwide with the purpose of improving properties of various types of clay by adding fly ash [9-24] and slag [25-33]. The results obtained by testing physicomaterial properties of improved clays are for the most part consistent, but at the same time they reveal that different types of clays, improved with the same type of binder under similar conditions, can influence mechanical properties quite differently. For instance, the strength and stiffness of improved clay does not only depend on the type and quantity of a binder, but also on a number of other factors such as moisture, degree of saturation, stress state, and drainage conditions in soil. That is why, considering their specificities, research efforts are mostly focusing on the analysis of the influence of binder on physicomaterial characteristics of clay at particular microlocations. Thus, Ahnberg et al. [34] investigate the effects of improvement of some Swedish clays by various binders, while Ahnberg and Johansson [35] study time-related increase in strength of Swedish clays improved by various binders, considering the type and quantity of binder. Gupta et al. [36] present improvement of mechanical characteristics of some Indian clays after addition of slag, while Yadu et al. [37] present improvements noted after addition of slag and fly ash. Literature offers a number of studies on the contribution of industrial by-products to the improvement

of some Egyptian [32], Iraqi [38], Italian [39], Irish [40], English [41], German [42], Turkish [43], Canadian [44], Chinese [45] clays, etc.

This paper presents results obtained by laboratory testing of strength and moisture of Zagreb clay improved with various binders. The investigations were conducted in geotechnical laboratory of the Faculty of Civil Engineering of the University of Zagreb, and in companies IGH d.d. and Geoekspert d.o.o., both based in Zagreb. The principal material used was Zagreb clay received from two localities: Zagreb crematorium and Dolac open-air market in Zagreb. The aim of this research is to obtain information that will enable better understanding of mechanical behaviour of Zagreb clay improved with cement, fly ash and slag as binders, for various proportions and conditions of maturation of the mix. Based on test results, a correlation can be made between an increase and value of the uniaxial compressive strength and the quantity of binder and curing period of tested samples, while a relation can also be established between the reduction and value of natural moisture on the one side, and the quantity of binder and curing period of tested sample on the other. New findings enable determination of mechanical parameters needed in the design of sustainable improvement of Zagreb clay based on the results of standard laboratory tests in soil. The significance of this study also lies in the possibility of reducing significant quantities of industrial by-products both in Croatia and its surroundings [8], e.g., fly ash in Bosnia and Herzegovina and Serbia, and slag in Croatia, Bosnia and Herzegovina and Serbia, through their use in the improvement of fine-grained types of soil.

2. Material subjected to testing

2.1. Zagreb clay

The city of Zagreb generally lies on two basic geological units: Medvednica mountain in the north, and the Sava depression in the south of the city. These two structures are divided by a reverse fault spreading in the northeast-southwest direction, and passing through the centre of the city. Geological unit to the south of the fault is formed, in the top part, of Quaternary alluvial deposits of the first and second Sava terrace, which are composed of gravel and sand down to the depth of approximately ten meters, and are covered with a thin layer of sandy and silty clays. The substratum is formed of overconsolidated stiff clay layers of similar mechanical properties, reaching to greater depths [46]. According to the Basic Geological Map, sheet: Zagreb and Ivanić Grad, the Podsljeme district of Zagreb is formed of various types of soil among which a dominant place is taken by the clay of Neogene and Quaternary origin, and so it can be named as Zagreb clay. This Zagreb clay is characterised by typical structure with plate morphology and large specific area, which is why Zagreb clay minerals exert a very strong influence on physical and hence also on mechanical properties of soil in the wider urban area of Zagreb.

Zagreb clay originating from two localities is used in this research. One locality is the Podsljeme city district next to the city cemetery, i.e. next to Crematorium, and the other locality is Dolac – an open-air market situated in the centre of the City of Zagreb. As to mineral composition, these clays consist of quartz, muscovite and lightweight



Figure 1. Sampling locations – Dolac open-air market and Crematorium

mineral montmorillonite. According to the USCS standard, the material from these localities is classified as clay of low (CL) to high (CH) plasticity, with typical mild swelling potential.

2.2. Cement

According to chemical composition, cements can be divided into silicate cements and aluminat cements. Silicate cements are obtained by burning marl and limestone. The most significant cement belonging to the silicate cement group is Portland cement, which is also used as a basis for manufacturing metallurgic, pozzolanic, and supersulphated cements. White Portland cement is a type of Portland cement that is obtained by burning kaolin and limestone. Aluminat cements are obtained by burning bauxite and limestone. They are used in the preparation of refractory

concrete, or when concrete is placed at very low temperatures. Silicate cement manufactured by HOLCIM d.o.o. in Koromačno Plant under the name of Holcim Expert cement is used in this research. It is characterized by moderate need for water, low loss of initial consistence, optimum cement setting time, usability in a variety of structures, medium development of strength, very moderate development of hydration heat, and good resistance to moderately aggressive influences. What needs to be emphasized is that this cement is not a "pure" cement but rather it is composed of 65-79 percent of Portland cement clinker, 21-35 percent of mixed additions (granulated blast furnace slag and siliceous fly ash), and up to 5 percent of filter dust. This cement was selected in this study because it is a commercial type of cement that is used on an everyday basis in civil engineering. Basic physicochemical properties of this cement are presented in Table 1.

Table 1. Physicochemical properties of cement used in this study and comparison with relevant standards [47]

Physical (F) / chemical (K) properties	Holcim Expert cement CEM II/B-M (S-V) 42.5 N	Standard requirements HRN EN 197-1; BAS EN 197:1
F: Volume consistency (Le Chatelier)	0 mm	≤ 10 mm
F: Start of setting	180 min	≥ 60 min
F: Compressive strength at 2 days	23.0 MPa	≥ 10 MPa
F: Compressive strength at 28 days	50.0 MPa	≥ 42.5 ≤ 62.5 MPa
K: SO ₃	2.5 %	≤ 3.5 %
K: Cl	0.02 %	≤ 0.1 %

Table 2. Chemical properties of slag samples

Content	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Cl	Na ₂ O	LOI
[%]	37.83	12.63	3.09	42.96	7.00	0.61	0.125	0.52	0.33

Table 3. Chemical properties of fly ash sample

Content	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Cl	Na ₂ O	K ₂ O	LOI	R45
[%]	55.88	16.07	5.39	4.5	1.67	0.54	0.01	1.65	1.71	3.85	9.19

2.3. Slag

Slag is obtained as a residue, i.e., as a byproduct of the steel manufacturing process in blast furnaces. It is estimated that approximately 1,8 million tonnes of slag are easily available in the territory of the Republic of Croatia from ironworks in Sisak and Split [8]. Approximately 150–200 kg of slag is generated during production of one tonne of steel. Liquid slag floating on molten iron is rapidly cooled down and thus transformed into clinker-shaped grains. There are two forms of slag: basic and acidic. Basic slag contains at least 50 % of basic oxides of CaO and Al₂O₃, while the rest is mostly SiO₂. Acidic slag contains much less than 50 % of basic oxides of CaO and Al₂O₃, and the SiO₂ is dominant. Only the basic slag is used as a binder. In fact, slag by itself has no binding properties, i.e. it gains these properties after catalysts (cement, lime, gypsum) are added. The main mineral of the basic slag is belite and its hydration products are almost identical to belite hydrates from cement clinker. In other words, the slag hydration process and the Portland cement clinker hydration process are of the same basic type.

The slag provided by the Croatian company HOLCIM d.o.o., and originating from ILVA steelworks situated in Taranto, Italy (Gruppo Rive), was used in this research. The slag was supplied to the laboratory in a metal container in granular form, with an average grain size of approximately 2.0 mm, and then the grain size of this slag was reduced by grinding before mixing, and sieved to the grain size of 0.425 mm. Chemical properties of the slag sample were determined in HOLCIM d.o.o. laboratory (Table 2). An average specific density of the slag sample is 2.90 g/cm³.

2.4. Fly ash

Fly ash is a byproduct, i.e. a secondary product formed by incineration of coal in thermal power plants. The only thermal power plant in the Republic of Croatia that uses coal as fuel is the Plomin plant located at the eastern coast of Istrian peninsula. While the supply and demand relationship is well balanced in Croatia and Slovenia, for the greater part because of smaller generation of fly ash, there are significant quantities of disposed fly ash in Bosnia and Herzegovina that could be used for soil improvement purposes. Approximately 7 tonnes of fly ash and slag are generated in Serbia each year, out of which only 3 percent is used in cement production. The rest is disposed at landfill site of approximately 300 million tonnes of fly ash, which occupies an area of approximately 1600 hectares [8].

Fly ash originating from Serbian coal-powered thermal power plants has pozzolanic properties but, due to low concentration of calcium compounds (less than 10 % CaO), it does not have self-binding properties.

According to ASTM C618, fly ash can be classified into two dominant categories: class F fly ash and class C fly ash. This classification is based on chemical composition. Class C fly ash is created as a byproduct of incineration of younger lignite coal or subbituminous coal which, in addition to their pozzolanic properties, also have self-binding properties. Due to its self-binding properties, this fly ash does not need an activator. It contains more than 20 % of lime. In the presence of water, class C fly ash will harden, and its strength will increase over time. Alkali and sulphate content is greater than in the case of class F fly ash. Class F fly ash generally occurs as a product of incineration of harder and older anthracite and bituminised coal. Class F fly ash has pozzolanic properties and contains less than 15 % of lime. Silicates and aluminium silicates of class F fly ash require a cement agent (such as Portland cement, lime, or hydrated lime) so that they can react in the presence of water and produce a cementitious compound. An addition of chemical activator to class F fly ash leads to formation of the so called geopolymer.

For the purposes of this research, a fly ash sample was supplied from company HOLCIM d.o.o. (Croatia) which uses in its production process fly ash from the Plomin thermal power plant located in Croatia. Chemical properties of this fly ash are defined in the HOLCIM company laboratory (Table 3). As the proportion of SiO₂+Al₂O₃+Fe₂O₃ is greater than 70 %, while the content of sulphur trioxide (SO₃) is smaller than 5 %, such fly ash is classified, according to ASTM C618-94a, into class F. An average specific density of the sample is 2.11 g/cm³.

3. Experimental testing

3.1. Preparation of samples for laboratory testing

Natural moisture was previously determined on all clay samples to be used for the preparation of the mix. Samples that are made of the clay and binder mix were named composite samples. Clay material was previously dried to 105 °C and then crushed and ground to pass through the 0.425 mm sieve. Clay material treated in this way was mixed with water to natural moisture, and the binder was added during this mixing process. The following binders were added to the clay mix: cement, fly ash



Figure 2. Keeping composite samples in desiccator and preparations for testing

and slag at 5 %, 10 % and 20 % of the clay weight, respectively. The mix prepared in this way was shaped and placed into a cylindrical mould approximately 36 mm in diameter and 80 mm in height. Such prepared composite samples were kept in desiccator to prevent moisture loss, and were tested after 7, 14 and 28 days of curing. The clay used in the experimental part was taken from two localities in Zagreb, i.e. from Crematorium locality and from Dolac market locality situated in the centre of the city. The mixing and composite sample forming program was prepared for each of the mentioned clays. Figure 2 shows the clay and binder mixing, and preparation of the composite sample.

The composite samples for which clay for Crematorium locality was used were marked with the initial mark „Kr”, followed by the composite number and the number of days of curing, while samples for which the clay from Dolac locality was used were marked with the initial mark “Do”. At that, a total of 24 samples for each clay composite group and for the defined binder percentage (5, 10 and 20 %) and so, taking into account the curing times of 7, 14 and 28 days, the testing for each location involved

a total of 72 samples of the composite with fly ash, 72 samples of the composite with slag, and 72 samples of the composite with cement. Overall, 432 composite samples were prepared for this testing, and they were tested separately in two cycles, separately for the Crematorium locality (216 samples) and separately for the Dolac locality (216 samples)

3.2. Testing samples in laboratory

The testing involved preparation of the clay and binder composite samples, with binder consisting of cement, slag and fly ash added at 5 %, 10 % and 20 % of the total clay content by weight, respectively. The samples were kept in desiccator for 7, 14 and 28 days. Each sample was subjected to compressive strength testing in the jack apparatus until failure, and the sample moisture at failure was determined after the testing. A sample during the testing and after failure is shown in Figure 3. Test results were grouped according to mass properties of individual composites, and the values were taken as average values of all samples belonging to the same group.



Figure 3. Testing compressive strength of composite in laboratory of Department of Geotechnics, Faculty of Civil Engineering, Zagreb

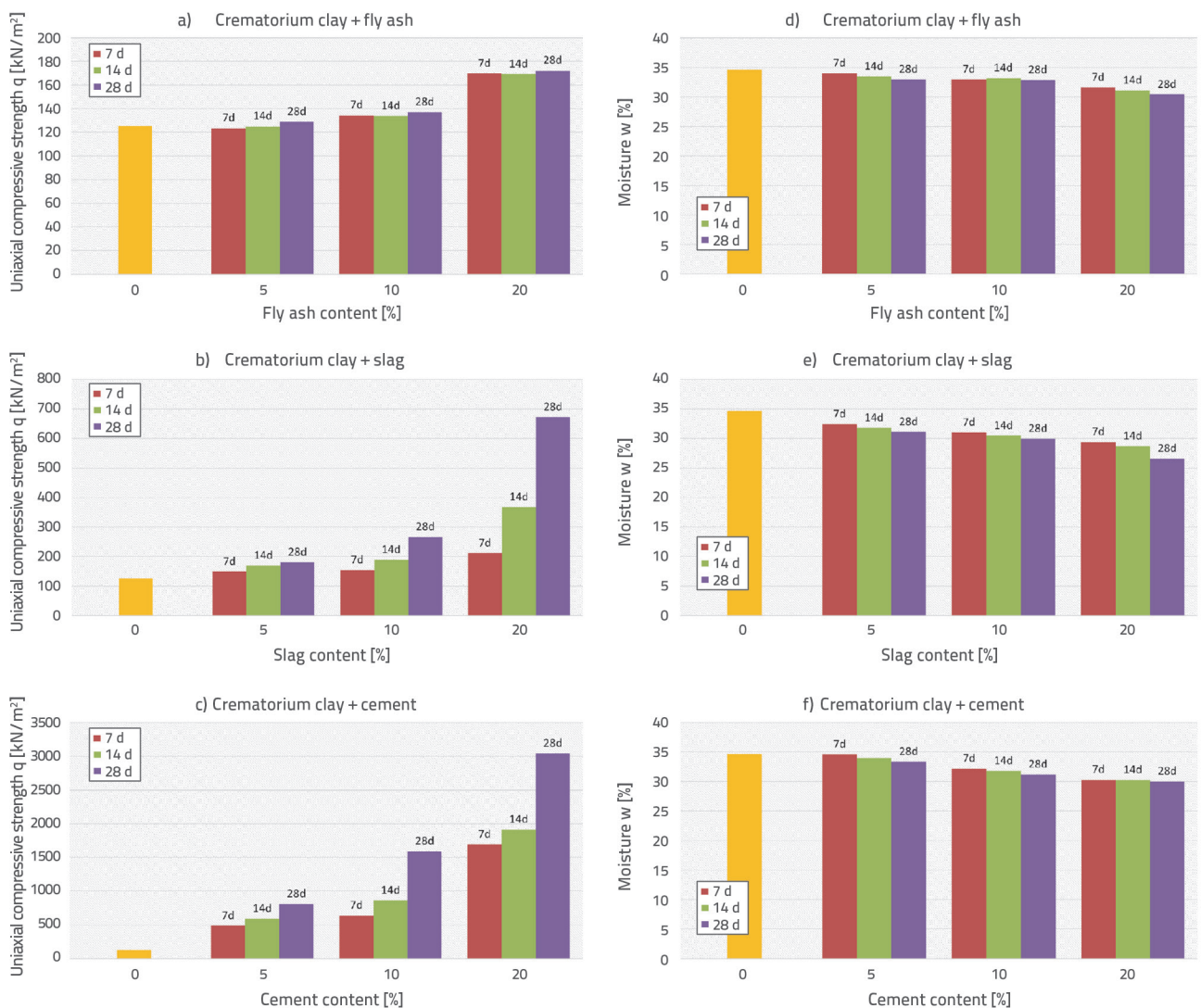


Figure 4. Testing uniaxial compressive strength of clay samples from Crematorium locality with the addition of: a) fly ash; b) slag; c) cement, and testing natural moisture of samples with the addition of: d) fly ash; e) slag; f) cement

3.3. Composite test results

Uniaxial compressive strength results of individual clay composites for the Crematorium locality, and moisture values determined after the testing, are shown in Figure 4.

Results obtained by testing uniaxial compressive strength of individual clay composite samples from Dolac market, and moisture values obtained after the testing, are presented in Figure 5.

4. Analysis of results and discussion

The uniaxial compressive strength of clay (q_0) determined in laboratory, without binder, is 125 kPa for the Crematorium locality, and 155 kPa for the Dolac market locality. On both localities, the greatest increase of uniaxial compressive strength can be observed in composites with cement as

binder, followed by the composites with slag as binder, while the lowest increase is observed in composite with fly ash as binder. Expectedly, a greater percentage of binder, whether it is fly ash, slag or cement, results in greater uniaxial compressive strength values.

In order to gain a better insight into the influence of the content of individual binder type on uniaxial compressive strength and moisture of improved clay, the results are also presented in the form of normalised diagrams. Figure 6 shows the ratio of the normalised uniaxial compressive strength (q) to uniaxial compressive strength of sample without binder (q_0), as related to the proportion (percentage) of individual binders. Figure 7 shows the ratio of the normalised moisture of composite sample (w) to natural moisture of clay sample (w_0), as related to the proportion (percentage) of individual binders.

Crematorium clay and Dolac clay composites, improved with 5 % of fly ash (Figure 6a) do not show an increase of uniaxial

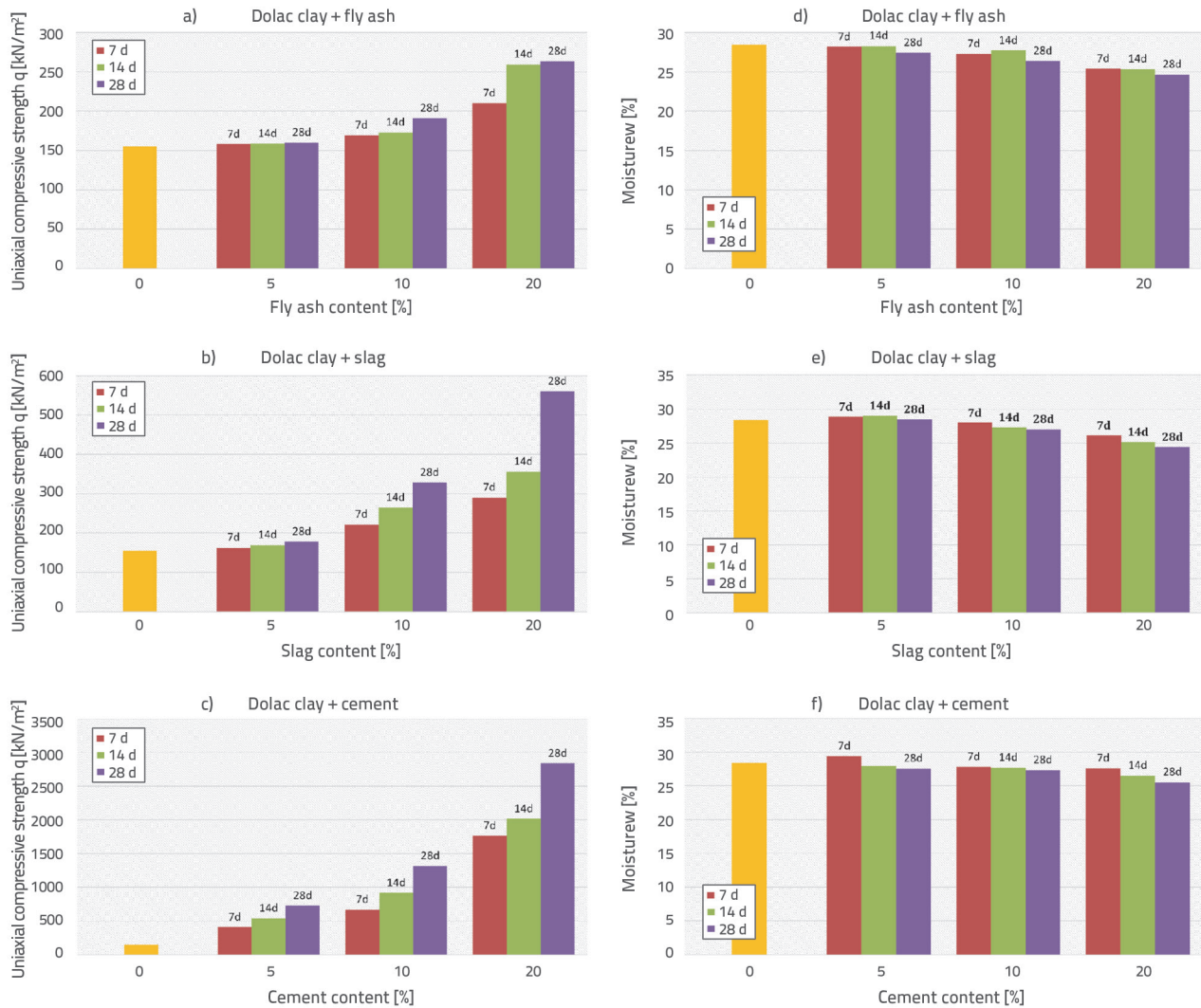


Figure 5. Testing uniaxial compressive strength of clay samples from Dolac market locality with the addition of: a) fly ash; b) slag; c) cement, and testing natural moisture of samples with the addition of: d) fly ash; e) slag; f) cement

compressive strength when compared to samples without binder, regardless of the age of samples. When 10 % of fly ash is added, the uniaxial compressive strength increases for about 7-10 % as related to samples without fly ash, while for the Dolac locality, the uniaxial compressive strength increases by 23 % after 28 days. When 20 % of fly ash is added to the mix, Crematorium clay samples achieve approximately 35 % increase in strength, while the age of samples does not have any influence on an increase in strength value. On the other hand, Dolac clay samples at 7 days of age achieved a 35 % increase for mixes with 20 % of fly ash, samples at 14 days of age achieved an increase of approximately 67 %, while samples at 28 days of age achieved an increase of 70 %, compared to samples without fly ash.

Crematorium clay and Dolac clay composites, improved with 5 % of slag (Figure 6b), unlike samples with the same percentage of fly ash, show an increase of uniaxial compressive strength when compared to samples without binder. Further, an increase is

evident with an increased age of samples, where Crematorium clay samples have a 18 % increase of uniaxial compressive strength (7 days), 35 % (14 days) and 44 % (28 days), while Dolac clay samples show a somewhat smaller increase of uniaxial compressive strength of 5 % (7 days), 9 % (14 days) and 15 % (28 days), all compared to uniaxial compressive strength of samples without binder. When 10 % of slag is added, Crematorium clay samples have an increase of uniaxial compressive strength of 23 % (7 days), 51 % (14 days), and 112 % (28 days), while Dolac clay samples have an increase of uniaxial compressive strength of 43 % (7 days), 70 % (14 days), and 111 % (28 days), all compared to uniaxial compressive strength of samples without binder. Just like in the case of improvement with fly ash, samples with the greatest percentage of slag (20 %) also show the greatest increase of uniaxial compressive strength – 68 % (7 days), 193 % (14 days) and 436 % (28 days) for Crematorium clay samples, and 87 % (7 days), 129 % (14 days) and 261 % (28 days) for Dolac clay samples.

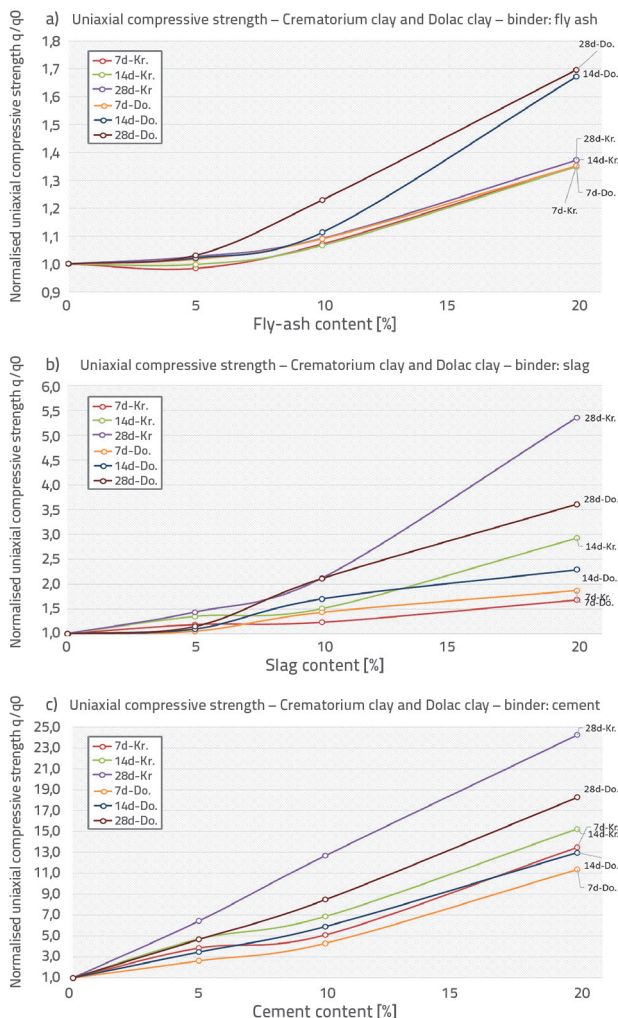


Figure 6. Normalised value of uniaxial compressive strength of composite with: a) fly ash; b) slag; c) cement, for different binder content

In the case of improvement of clay samples with cement (Figure 6c), a significant increase of uniaxial compressive strength can be observed when compared to the samples without improvement, i.e. it is greater at smaller quantities of added cement, and an increase is evident with an increase in the age of samples. Thus for Crematorium composite samples, an increase of uniaxial compressive strength amounts to 3.87 times (7 days), 4.72 times (14 days), and 6.44 times (28 days) for samples with 5 % of cement, while it is 5.1 times (7 days), 6.86 times (14 days), and 12.67 times (28 days) for samples with 10 % of cement and, finally, 13.48 times (7 days), 15.24 times (14 days), and 24.23 times (28 days) for samples with 20 % of cement. For Dolac composite samples an increase of uniaxial compressive strength amounts to 2.63 times (7 days), 3.47 times (14 days), and 4.68 times (28 days) for samples with 5 % of cement, while it is 4.31 times (7 days), 5.91 times (14 days) and 8.47 times (28 days) for samples with 10 % of cement and, finally, 11.36 times (7 days), 11.97 times (14 days)

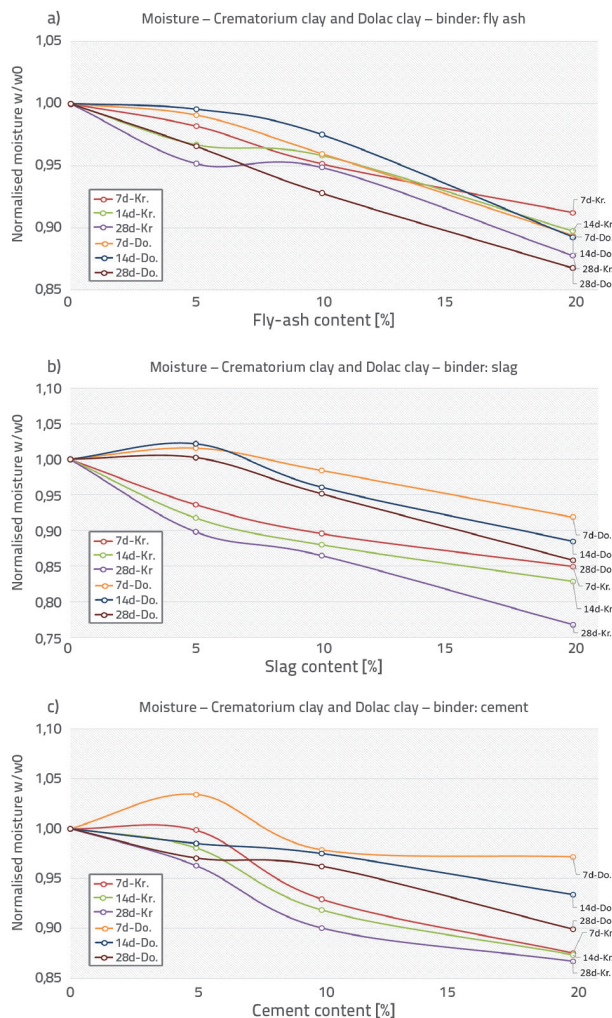


Figure 7. Moisture of composite samples with: a) fly ash; b) slag; c) cement, for different binder content

and 18.27 times (28 days) for samples with 20 % of cement. It is evident that samples with cement exhibit the greatest increase of uniaxial compressive strength over maturation time of samples, when compared to samples to which fly ash and slag is added.

Figure 7 shows reduction in moisture of tested samples of clay mixed with fly ash (Figure 7a), slag (Figure 7b) and cement (Figure 7c), when compared to natural moisture of clay samples without binder. It can be seen that the greatest reduction in moisture occurs in the case of composite samples with slag, while this reduction is similar in the case of samples with fly ash and cement. In all composites, regardless of the type of binder, the reduction in moisture is consistent with higher percentage of binder, as well with an increase in the sample maturation time. Dolac locality samples mixed with slag show an increase in composite moisture even at lower binder content (5%), while moisture decreases in the case of greater binder content (10% and 20%).

5. Conclusion

The paper provides an overview of laboratory test results for Zagreb clay samples, taken from Crematorium cemetery locality and Dolac market locality, and improved with various binders:

- cement as a binder that is traditionally used for soil improvement
- fly ash
- slag as industrial by-products that are increasingly used in soil improvement studies worldwide.

A total of 432 composite clay samples with the mentioned binders were tested for both localities for curing period of 7, 14 and 28 days. Results obtained by testing influence of the type and quantity of each binder, as well the curing period, on mechanical property, i.e. uniaxial compression strength of the composite, and on physical property, i.e. composite moisture, are analysed and discussed in the paper. The results show that clay and cement composites exhibit the greatest increase in the uniaxial compressive strength with an increase in cement content. These composites are followed by clay and slag composites, while the lowest increase in uniaxial compressive strength was noted in the case of clay mixed with fly ash. Also, the composite with cement exhibits the increase in uniaxial compressive strength with an increase of curing period, while this influence is the lowest in the

case of samples with fly ash. Although it can be expected that average normalised strengths of geopolymer samples from Dolac are greater compared to those of Crematorium samples, because of somewhat greater uniaxial compressive strength of Dolac clay (without binder), it was nevertheless observed that this was the case only when fly ash is used as binder. In the case when slag or cement is used as binder, normalised strengths of geopolymer samples were greater in the case of Crematorium clay samples. This may be explained by the fact that Dolac clay samples are mostly characterised by high plasticity (CH), while Crematorium clay samples are characterized by low plasticity (CL). Moisture of tested samples consistently reduces with an increase in binder content, and with an increase in the sample maturation time. Laboratory test results presented in this study provide a preliminary insight into behaviour of physico-mechanical parameters of composites of clay improved by various binders. Further research will focus on the determination of stiffness properties of clay composites, in order to gain a more comprehensive insight into parameters needed for the design of sustainable improvement of Zagreb clay. The significance of this research also lies in the analysis of the influence of industrial by-products binders on the improvement of Zagreb clay, which might potentially result in the reduction of significant quantities of industrial by-products currently disposed in Croatia and the wider region.

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