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The Effect of Nanomaterials Injection on the Mechanical Characteristics of Coarse-Grained Soils

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Modern technologies offer nowadays a great variety of solutions for soil stabilization, as low- or high-pressure injections of fluid mixtures. There are many different products suitable for this purpose, depending on the properties of the soil to be treated: for sand, in particular, suspensions of nanoparticles, made available by modern industrial technologies, can be considered among the fluid mixtures potentially suitable for its treatment. This work presents the preliminary results of a wider study developed to verify the potential beneficial effects of the injection of natural based micro- and nano-materials on the mechanical properties of coarse-grained soils. To verify the improvement in sand strength, standard geotechnical tests, such as direct shear tests, were performed on soil samples before and after the injection of micro- and nano-particles of natural materials (sand, clay and graphite). The results proved that the injection in sand of a mixture of water and micro-sized sand improves the mechanical characteristics of the samples. By adding clay nanoparticles and graphene to this mixture the results showed an even greater increase in the shear strength. The results of this work proved that natural based micro- and nano-materials seems to have promising properties to become a competitive resource in the field of geotechnical engineering as soil stabilizers, as a valid alternative to widely used materials.

1. Introduction

In the past few decades many different techniques have been used to improve the physical properties and the mechanical characteristics of the soil to prevent natural and anthropogenic undesired phenomena. Thanks to technological developments (Bavasso et al., 2016), nowadays it is possible to choose from a great variety of solutions for soil stabilization, as low- or high-pressure injections of fluid mixtures: in literature there are several examples of studies on the improvement of the mechanical characteristics of soils through injections of mixtures of nanomaterials. According to the recommendation adopted by the European Commission in 2011, a nanomaterial is "a natural, incidental or manufactured material containing particles, in an unbound state or as an aggregate or as an agglomerate, and where for 50% or more of the particles in the number size distribution, one or more external dimensions is in the size range 1 nm – 100 nm". Nanomaterials typically show different behaviours or properties when compared to larger particles of the same material (Nohani & Ezatolah, 2015). In literature, most of the works refers to a clay matrix in which mixtures composed of different nano-sized elements are injected, as nano-aluminum oxide (Luo et al., 2012), SiO_2 nanoparticles with both cement (Bahmani et al., 2014) and recycled polyester fiber (Changizi & Haddad, 2015), nanosized silica (Eswaramoorthi et al., 2017) and nanosized flyash (Sanchin Prabhu et al., 2017). Few studies have been conducted on coarse-grained matrices instead (Ghasabkolaei et al., 2017) and the suspensions of nanometric particles can be included among the fluid mixtures suitable for the treatment of medium-sized sand (Proia et al., 2017). In all these studies, the results of the experiments demonstrate that the addition of nanomaterials increases the values of the strength parameters investigated, such as the uniaxial compressive strength and the shear strength. Next future technologies will allow to micronize materials, that is to produce particles with diameters smaller than 100 μm, or to synthesize them (Bavasso et al., 2018) at increasingly lower costs and times (Vilardi et al., 2017), hence nanoparticles in the next future will become a competitive resource in geotechnical engineering compared to the mixtures obtained with other suspensions, i.e. cement mortars or ultra-fine cements.

The aim of this work is to analyse the effects of the injection of micro- and nano-metric particles, obtained from the micronization process of natural materials such as sand, clay and graphite, on the strength characteristics of coarse-grained soils. This work is aimed at supporting the use of these types of mixtures, with low- or high-pressure injections, to solve geotechnical problems related to soil improvement such as stabilization of existing foundations, improvement of the stability of natural and artificial slopes or soil stabilization within the framework of tunnelling and underground works.

2. Methodology

2.1 Materials

To evaluate how liquid mixtures of micro- and nano-sized particles and water modify the strength parameters of a coarse-grained soil, the first step was the characterization of the materials used in this work: London clay, Colleferro sand (described also in Guida et al., 2019) and graphene. Both original and micro- and nano-sized clay and sand have been studied through the granulometric analysis, the Atterberg limits, the specific weight of the grains and the mineralogical analysis (X-ray diffraction). Graphene, instead, has not been characterized since it is already well known from literature (Boehm et al., 1994).

The granulometric analyses were carried out according to AGI 1994 (Figure 1) and through laser diffraction analysis. Table 1 shows the values of the mass-median-diameter (D_{50}) obtained with all the different types of analysis, in the presence or absence of sodium hexametaphosphate, a dispersing agent. The results obtained show that micro-sand and micro-clay belong to the range 0 – 100 μm and are therefore micromaterials, however nano-clay and nano-sand do not belong to the range 0 – 100 nm, therefore they cannot be considered nanomaterials.

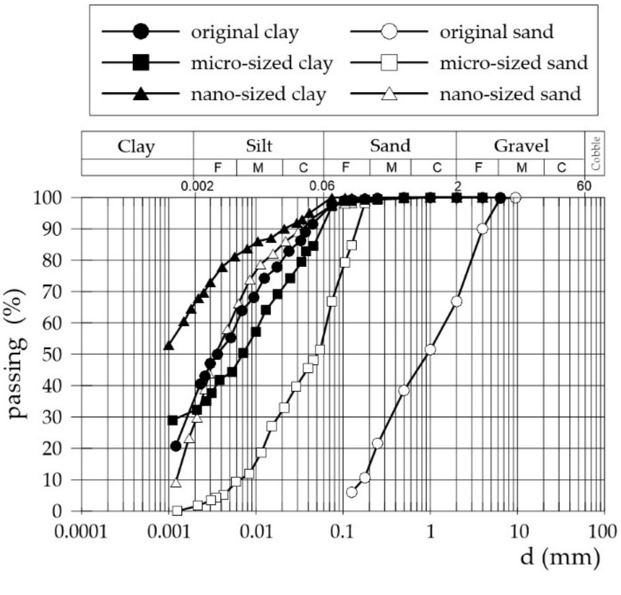

Figure 1: Granulometric curves of London clay and Colleferro sand obtained through sieves and hydrometer analysis

Atterberg limits were determined, according to ASTM D 4318-17, to complete the London clay characterization. Moreover, from the granulometric analysis it can be observed that the micro- and nano-sized sand reach dimensions typical of fine-grained soils. Therefore, their liquid limits were also determined, excluding the one of the original Colleferro sand which, being a coarse-grained soil, does not have Atterberg limits (Table 2). It can be observed that nano-sand has actually a liquid limit, while micro-sand has not. This result suggests that despite the small dimensions, micro- and nano-sized sand have not changed their mineralogical nature and, as their original configuration, does not present any plasticity. For London clay, instead, as the grain size decreases, an increase in the PI, LL and PL is observed.

The helium pycnometer was used to determine the value of the specific weight of the grains and the density of the materials, according to ASTM D 854-92. The results (Table 2) show an increasing trend in specific weight as the grain sizes decrease. This behaviour could be explained by the decrease of the interparticle voids present on the individual grains, due to the micronization process.

Table 1: Values of the mass-median-diameter (D50) of London clay and Colleferro sand, obtained in the presence or absence of a dispersing agent through sieves and hydrometer analysis and laser diffraction analysis

materials		ASTM D_{50} (mm)	 $D_{50\ disp}$ (mm)	laser D_{50} (mm)	 $D_{50\ disp}$ (mm)
London clay	original	-	0.004	0.007	0.011
	micro-sized	0.008	0.007	0.006	0.013
	nano-sized	-	<0.0010	0.003	0.007
Colleferro sand	original	0.91	-	-	-
	micro-sized	0.05	0.031	0.017	0.014
	nano-sized	-	0.003	0.003	0.004

Table 2: Atterberg limits (liquid limit LL, plastic limit PL, plasticity index PI and activity A), density (ρ) and specific weight of the grains (γ_S) of London clay and Colleferro sand

materials		LL (%)	PL (%)	PI (%)	A (-)	ρ (g/cm^3)	γ_s (kN/m^3)
London clay	original	55.38	24.89	30.49	0.76	2.68	26.28
	micro-sized	53.05	22.18	30.87	1.01	2.70	26.44
	nano-sized	104.24	-	-	-	2.85	27.92
Colleferro sand	original	-	-	-	-	2.71	26.54
	micro-sized	-	-	-	-	2.75	26.97
	nano-sized	36.25	-	-	-	2.85	27.94

The mineralogical analysis was carried out by the X-ray diffraction spectroscopy on micro- and nano-metric sand, while it was not possible to characterize the mineralogy of the clay due to some limitations of the instrument used. The results show that the percentages of the minerals in the two configurations of the sand are different (Table 3). Since the micronization process is based on the breakdown of the grains due to the collision between particles or particle and solid surface, it is possible that, in a certain interval of time, the materials characterized by a lower hardness first reached the nanometric dimensions. Therefore, it is possible to deduce that the minerals have not been equally divided: there are higher percentages of the softer minerals in nano-sand and higher percentages of harder ones in micro-sand.

Table 3: Percentages of micro- and nano-sized sand minerals

	calcite (%)	quartz (%)	albite (%)	phengite (%)	leucite (%)	aragonite (%)	dolomite (%)
micro-sized Colleferro sand	36.29	29.67	17.21	10.19	3.3	2.09	1.25
nano-sized Colleferro sand	51.15	19.28	11.27	9.45	2.01	6.84	-

2.2 Laboratory test

The laboratory tests selected for the determination of the strength parameters of a coarse-grained soil treated with micro- and nano-sized materials are the direct shear tests, according to AGI 1994. The applied stresses were the axial load, chosen equal to 0.5, 1 and 2 kg/cm^2, and the horizontal force deriving from the application of a constant speed equal to 0.165 mm/min or 0.180 mm/min, depending on the machine used.

2.3 Samples preparation

The samples preparation began with the choice of the liquid mixtures to be used. The materials making up the starting mixture are micro-sized Colleferro sand, micro-sized London clay, graphene and water. Their percentages (mixture 1 in Table 4) have been obtained from previous experiments. Starting from these values it was decided to create new mixtures obtained by subtracting one or more materials, to study how and how much each element modifies the strength of the coarse-grained soil. For this reason, new mixtures have been created, as shown in Table 4 where they are identified by the numbers 1, 2, 3, 4 and 5.

Table 4: Materials percentages of the different mixtures

mixture	micro-sized sand (%)	micro-sized clay (%)	graphene (%)	water (%)
1	35.3	21.2	1.1	42.4
2	57.6	-	-	42.4
3	35.9	21.7	-	42.4
4	56.5	-	1.1	42.4
5	-	56.5	1.1	42.4

All the mixtures were obtained by gently mixing the solid particles in water. Monogranular Colleferro sand, with a diameter between 1 and 2 mm, was used as soil matrix for all the samples.

The specimens for direct shear tests (**Errore. L'origine riferimento non è stata trovata.**) were obtained by pouring each mixture into a shear box on which the test was subsequently carried out, and then slowly inserting by pluviation the monogranular matrix. In this way the standard geometry of the sample has been obtained, the sample was not disturbed during the extrusion phase and the excess water of the mixture was removed from the holes on the box. Three specimens were prepared for each mixture. Furthermore, in order to make comparisons, specimens of loose and dense monogranular sand without any mixture were also prepared. Since the samples were packaged in the shear boxes, it was not necessary to extrude them. For all the samples, except for those without mixture, 4 days were awaited to complete the maturation and then proceed with the test.

3. Results

To determine any improvements induced by the presence of mixtures in the monogranular sand, direct shear tests were carried out first on samples of loose and dense sand without mixtures, then in the ones with mixtures injected. The results are shown in Table 5, the curves obtained for a vertical stress equal to 50 kPa are shown in Figure 2a (the results are similar for the other vertical stresses).

Table 5: Peak stress (τ_P) and critical state stress (τ_{CR}) values obtained with the direct shear test at three vertical stress (σ_N)

mixture	σ_N (kPa)	τ_P (kPa)	τ_{CR} (kPa)
loose sand (no mixture)	50	46.9	29.6
	100	97.6	65.9
	200	144.9	121.8
dense sand (no mixture)	50	61.5	33.8
	100	114.1	70.2
	200	207.4	125.1
1	50	112.3	53.4
	100	176.9	108.8
	200	304.9	179.1
2	50	84.7	32.4
	100	173.6	77.4
	200	242.7	176.9
3	50	102.5	34
	100	137.8	73.7
	200	306.8	160.5
4	50	70.4	35.3
	100	125.4	81.2
	200	169.9	132.7
5	50	103.9	47.8
	100	134.1	80.3
	200	202.9	175.6

4. Discussion

As shown in the previous figure, the values of the strength obtained through the direct shear test of the loose and the dense sand in the critical state are approximately coincident; in fact, as it is known from literature (Lancellotta, 1993), for high levels of deformation the observed trends converge. The same trend is followed by the specimens treated with graphene-free mixtures, while the results obtained from all the other tests show greater values. For both peak and critical state condition, the mixture that allowed the achievement of the highest values is mixture 1 (Figure 2a), which is made up of all the micronized elements (micro-sand, micro-clay, graphene and water). In addition to the peak strength, the critical state one also undergoes a relevant increase: as deformations increase, a decrease in the strength values for mixture 1 is observed, however it never reaches the values obtained for the untreated samples.

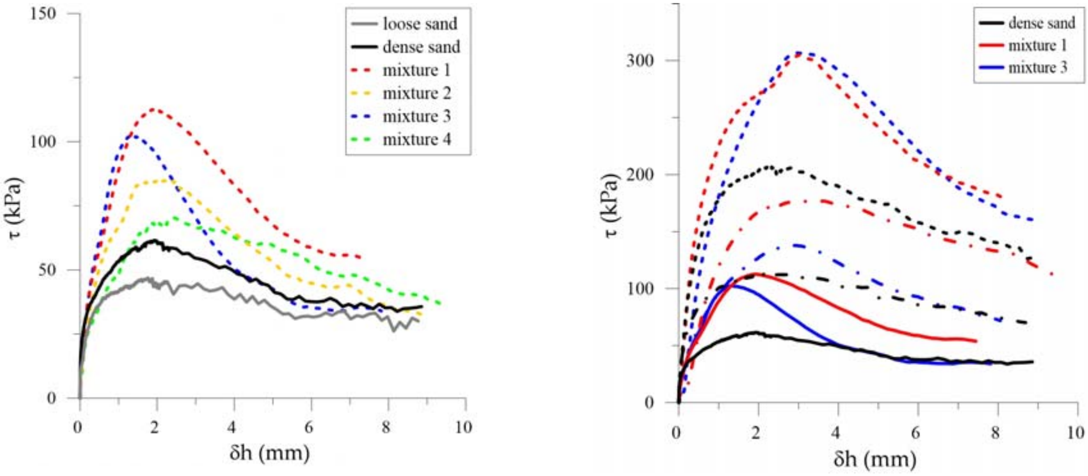

Figure 2: a) curves obtained through direct shear tests for a vertical stress equal to 50 kPa; b) curves obtained through direct shear tests for a vertical stress equal to 50 kPa (solid line), 100 kPa (dash-dotted line) and 200 kPa (dashed line)

At the same time, interesting results have been obtained with the mixture 3, made up of micro-sand, micro-clay and water (Figure 2). In fact, it is possible to observe that the values of the peak strength obtained from the treated samples are much higher than the untreated ones but, unlike the mixture 1, while approaching the critical state the trends assume increasingly similar values for the same vertical stress imposed (Figure 2b). By comparing all the results previously reported (Table 5) it can be deduced that the improvement of the critical state strength is linked to the presence of graphene in the mixtures.

5. Conclusions and future developments

The preliminary results obtained from this research activity suggest that the injection of a mixture composed by micro- or nano-sized sand and water (mixture 2) in a monogranular sand increases its shear strength (84.7 KPa,173.6 kPa and 242.7 kPa for a vertical stress equal to 50, 100 and 200 kPa respectively). The addition to this mixture of micro-sized clay (mixture 3) leads to an even more evident increase in the strength parameters (102.5 kPa, 137.8 kPa and 306.8 kPa for a vertical stress equal to 50, 100 and 200 kPa respectively), which however are slightly lower compared to those obtained with a mixture made up of micro-sand, micro-clay, graphene and water (mixture 1). The results of the direct shear tests, in fact, showed that the latter composition, thanks to the action of graphene, allows to reach highest peak strength values (112.3 kPa, 176.9 kPa and 304.9 kPa for a vertical stress equal to 50, 100 and 200 kPa respectively) and, for large deformations, the critical state conditions of the untreated specimens are never reached.

To verify that the results obtained are also valid for soils below the water table or crossed by water, the samples were immersed in water: the cancellation of the effect of the mixtures on the samples was observed and the final configuration has returned to the one of loose sand.

The results obtained and the considerations reported in this work, lead to conclude that the high- or low-pressure injections of micro- and nano-materials into a coarse-grained soil allow to improve the mechanical properties of the soil. Thanks to technological development, it will be soon possible to obtain large quantities of micronized materials, especially natural-based ones, quickly and at low costs. The natural micro- and nano-materials can be widely used, with low environmental impacts, in the engineering field to solve geotechnical problems related to soil improvement such as the stabilization of existing foundations, the improvement of the stability of natural and artificial slopes or soil stabilization within the framework of tunnelling and underground works.

As future development of this work it would be useful to deepen the characterization of the materials used in the mixtures using equipment suitable for the analysis of micro- and nano-particles.

Furthermore, it would be interesting to test other mixtures made up by the same materials with different solid/liquid ratio: the decrease of the s/l ratio of the mixtures, for example, will make it easier to inject it into the soil but will also lead to a change in the strength parameters.

It will be also interesting to investigate the potential change in the strength parameters of the specimens with the increasement of their maturation time, obtaining a correlation that allows to determine the most appropriate choice for each mixture, or with the increasement of their temperature: graphene, in fact, is an excellent heat conductor and could lead to different results in terms of sample strength if subjected to high temperatures or microwaves.

Since the results reported in this work are valid only in water absence, it would be interesting to identify a new element to add to the mixture to be injected in order to achieve a permanent modification of the strength parameters even for grounds under an aquifer or crossed by water.

Finally, the design and the construction of a testing site would be extremely important to observe whether the laboratory results are replicable on a large scale and, in this case, which is the best injection method for the chosen mixture.

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