Activation of marble industry waste for sustainable stabilisation of clays

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Abstract. Environmental and economic sustainability is an increasingly important priority in civil engineering. In this context, the stabilisation of clayey soils offers an effective strategy for reusing excavated materials, thereby reducing the consumption of natural resources and minimizing transport-related environmental impacts. Typically, excavated geomaterials are compacted in situ under unsaturated conditions and employed in the construction of infrastructure such as roads and railway embankments, levees, dams, and backfills. However, enhancing the mechanical behaviour of low-quality soils often requires chemical binders such as lime or cement, which undermine sustainability due to the high environmental impact of their production processes. Therefore, alternative solutions involving the use of industrial by-products need to be explored. This study investigates the potential of marble sawing sludge, a by-product of marble production with significant disposal challenges, as a novel chemical binder for the stabilisation of high-plasticity clays. After characterizing the sludge, its transformation through calcination was examined. The resulting binder was then used in clay-binder mixtures, which were subjected to compaction tests, swelling and oedometer compression tests, unconfined compression strength (UCS) tests along with total suction measurements. Furthermore, UCS tests over time combined with suction monitoring were conducted to evaluate the binder's long-term effectiveness in soil stabilisation. The results demonstrate that stabilisation using this sustainable binder is very promising for geotechnical applications.

1 Introduction

The pursuit of design solutions that reduce the consumption of non-renewable raw materials and promote environmental sustainability has become an increasingly primary objective in civil engineering. For example, during the construction of road and railway infrastructure, earthmoving operations lead to a significant increase in CO_2 emissions, considerable costs, and a high demand for natural resources [1].

In this context, the use of unsaturated earthen geostructures - such as embankments, levees, or compacted fills - offers a promising alternative, as they can often require fewer resources and can be constructed with locally available soils. These geostructures, operating in unsaturated conditions, can achieve adequate mechanical performance while contributing to the reduction of environmental impacts. Nonetheless, surficial soils frequently exhibit low strength, high deformability, and high sensitivity to water interaction, which may compromise structural integrity. Therefore, it is crucial to develop design methodologies and construction technologies that enable the construction of high-performance geostructures, while meeting the requirements of cost-effectiveness and sustainability. Lime stabilisation has been demonstrated to significantly enhance the geomechanical properties of excavated clayey soils, thus representing a sustainable solution that has been successfully adopted for decades [2,3]. This technique enables the direct reuse of otherwise unsuitable excavated materials in the construction of embankments and subgrades, thus reducing the demand for new construction materials and the disposal of waste in landfills [4]. The chemical and fabric modifications resulting from the interaction between lime and clay minerals can be broadly categorised into short-term and long-term effects [5]. Short-term effects include improvements in the geotechnical properties of clayey soils, such as reduced plasticity and water content, enhanced workability, and increased strength. Long-term effects arise from pozzolanic reactions triggered by interactions among free calcium ions and the phyllosilicates present in the clay. Under highly alkaline conditions (pH>12.4), these minerals solubilize, promoting the formation of cementitious gels that bind clay particles together, thereby ensuring high mechanical performance over time [6,7]. Despite efforts to promote circularity and sustainable development, the use of conventional binders entails significant consumption of non-renewable resources, contributing directly to the depletion of raw materials [8]. Consequently, there is an urgent need to explore viable alternatives to traditional commercial binders, with a particular focus on calcium-based industrial by-products [9].

This study aims to reassess a by-product from the Sicilian marble industry as a potential substitute for

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industrial binders in soil stabilisation applications. The investigation focuses on its effects when applied to a natural high-plasticity clay obtained from excavation activities. The extraction of Perlato di Sicilia marble in the Custonaci area (Sicily, Italy) holds a prominent position in the global market for high-grade ornamental stone. The distinctive characteristics of this carbonate rock have driven substantial demand, with an estimated annual turnover of €100 million and approximately 90% of the extracted marble being exported worldwide [10]. Of the total extracted volume (3×10^6 tons per year), only 20-25% is processed for market sale, while the remainder is classified as waste. The marble cutting sludge, which is primarily composed of calcium carbonate in an aqueous suspension, is generated from the cutting of rock using diamond blades and water jets, producing approximately 2×10^5 tons of waste annually. Disposal of this waste is both costly and environmentally detrimental [11]. Conversely, previous studies have demonstrated that this by-product can function as a viable alternative to conventional binders in cementitious mortars, developing similar properties as a hydraulic binder when properly calcined [12]. A few researchers have conducted experimental studies on the effectiveness of marble by-products for soil stabilisation in road construction [13]. However, simple addition of calcium carbonate to fine-grained soils has been found to result in a granulometric modification, without inducing substantial microstructural changes. In contrast, calcination proves to be a critical step when calcium-based by-products are involved as substitution for conventional binders [14].

In the present study, marble sludge was thermally treated by calcination to produce a suitable binder, i.e. a powder mainly composed of calcium oxide. Three distinct soil-binder mixtures were selected for further testing to assess their potential application. Their performance was evaluated by comparing the results with those obtained from untreated clay samples. Given that chemically stabilised geomaterials are typically compacted in situ under unsaturated conditions for use in embankments, levees, dams, and backfilling, the selected mixtures were subjected to Proctor Standard test to determine their optimal compaction conditions in terms of dry density, water content, and degree of saturation. Swelling behaviour induced by saturation under low total stress of unsaturated compacted samples at optimum condition was investigated in conjunction with oedometric compression tests. Unconfined compression tests, suction measurements and water content checks on samples cured for a period up to 28 days were conducted to investigate the role of unsaturated conditions on the improved mechanical behaviour.

2 Materials and methods

The clay investigated in this study was obtained from excavation operations in the province of Trapani (Sicily, Italy). Its particle size distribution consists of sand (f_{sand} = 2%), silt (f_{silt} = 40%), and clay (f_{clay} = 58%), with a liquid limit, plastic limit of w_L = 59% and w_P = 29%, respectively, and a plasticity index (PI) of 30%. According to EN 11531-1 [15], the soil is classified as A7-6. Mineralogical analysis conducted via X-ray diffraction (XRD) revealed that the material is mainly composed of kaolinite, quartz and calcite, as shown in Figure 1. The marble sludge collected from a quarry in Custonaci (TP), had an initial water content of 40% and was subjected to air-drying in the laboratory. It was then manually crushed in the laboratory using a mortar, and sieved at 425 microns, resulting in a fine powder, hereafter referred to as marble powder (MP). X-ray fluorescence (XRF) analysis revealed a predominant presence of calcium carbonate in MP, with a calcium oxide (CaO) content of about 57% and a loss on ignition (LOI) of 42%, corresponding to the CO_2 mass undetectable by this technique. The remaining 1% is composed of trace elements. These values closely align with the composition of pure calcium carbonate and were further confirmed by mineralogical investigation using the XRD technique, which revealed major peaks corresponding to calcite.

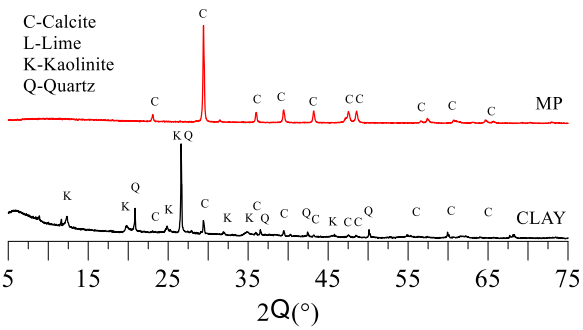

Fig. 1. X-ray diffraction patterns of MP and clay.

The binder was activated by calcining marble powder (MP) at 900°C for three hours in a furnace, using a heating/cooling rate of 10°C/min. This process produced calcined marble powder (CMP), which exhibited a free calcium oxide (CaO) content sufficient for use as a binder. The free CaO content was estimated at 84% based on XRF analysis, and 84% based on thermogravimetric analysis (TGA) of the residue. Following the method proposed by Eades and Grim (1966), the minimum binder content required to create an alkaline environment with pH > 12.4 - capable of activating cation exchange and initiating pozzolanic reactions - was determined. This value, referred to as the Initial Consumption of Lime (ICL), was found to be ICL = 2.5%, and based on this value, three mixtures were prepared with increasing dosages (ICL, 1.5×ICL, and 2×ICL). The effect of the binder on soil plasticity was then evaluated by determining the Atterberg limits of the mixtures. The findings revealed that the plasticity index decreased to 7% at the ICL dosage, with no substantial changes observed for higher values of ICL.

Proctor compaction tests were carried out in accordance with the standard energy specification (E = 0.6 MJ/m³), as defined in EN 13286-2 [16].

Prior to testing, samples were subjected to 24 hours of hydration through the addition of water to the disaggregated clay. CMP was then mixed mechanically to ensure homogenous distribution. Following this, the

samples were subjected to sieving through a 16 mm sieve, placed into airtight containers, and allowed to rest for one hour prior to compaction. Samples statically compacted at the optimum condition of the three mixtures were tested for one dimensional swelling and oedometric compression after 1 hour from mixing with binder. Oedometer tests were conducted on cylindrical specimens (h=20 mm, d=56 mm), including one from the untreated clay and three of the treated mixtures. Initially, the specimens were subjected to a total vertical stress of σ_v=10 kPa, and after 120 seconds, they were submerged in distilled water. Swelling was monitored for 24 hours. Thereafter, a progressive loading sequence was applied up to 4915 kPa, followed by unloading back to the initial stress level (10 kPa). Unconfined compression strength (UCS) tests were carried out on cylindrical specimens (h=76 mm, d=38 mm) statically compacted in three layers. Stabilised mixtures were tested at different curing times after 0, 7, 14, and 28 days of curing. To prevent moisture loss, samples were wrapped in plastic film and stored in sealed conditions. Also, the untreated clay was tested to provide a strength reference parameter.

UCS tests were performed applying a strain rate of 1.70%/min until failure. Suction measurements were conducted using a WP4-T dew-point potentiometer on samples that had been compacted directly within small containers (h=0.86 mm, d=3.74 mm). The samples were then hermetically sealed in these containers for curing until 28 days. This ensured controlled curing by preventing moisture loss. Water content was determined by calculating the mass difference of the samples before and after each curing period.

3 Results

Figure 2 shows the results of the Proctor Standard compaction tests conducted on the three formulated mixtures and the untreated clay. The data show a gradual reduction of the maximum dry unit weigh with increasing dosages of CMP at optimum compaction conditions. When compared with the untreated clay (γ_{dmax} = 15.29 kN/m^3), the highest CMP dosage (5.00%) results in a lower dry unit weight of γ_{dmax} = 14.75 kN/m^3, as indicated at the peaks of the interpolated curves derived from experimental data. Additionally, the optimum water content increases with the addition of CMP, as typically observed in lime-treated soils, due to binder hydration processes and short-term microstructural changes [17]. In particular, the addition of the binder induces an immediate reduction in the initial water content of the clay, with a reduction of approximately 1.50% for each percentage point of binder added. This reduction must be taken into account during in-situ mixing, to ensure that the target optimum water content is achieved. All mixtures studied exhibited degrees of saturation between 70% and 80% under optimum conditions. Therefore, the samples for subsequent tests were prepared to reflect these unsaturated conditions.

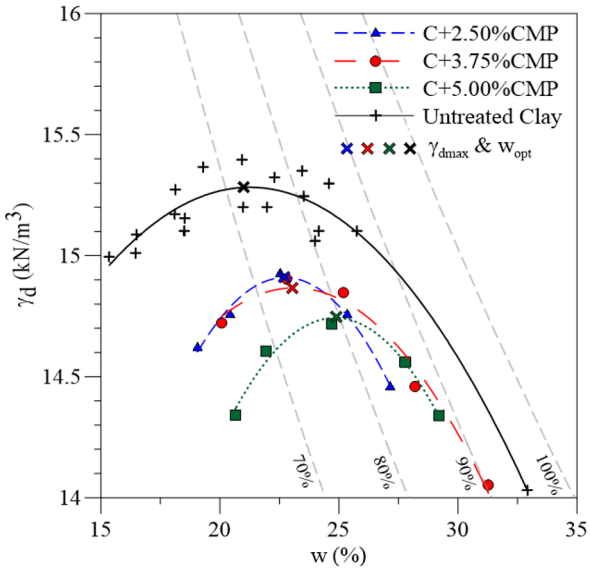

Fig. 2. Experimental Standard Proctor curves.

The strength and deformability results reflect the short-term effects of treating the clay with CMP, highlighting its effectiveness in improving the clay's behaviour. Figure 3 shows the swelling upon saturation curves for untreated clay and the three mixtures investigated. During the saturation phase at a total vertical stress of σ_v = 10 kPa a significant reduction in uniaxial swelling is observed in CMP treated samples, compared to untreated clay. The untreated material demonstrated significant swelling, exhibiting an axial strain of 5.24%. Conversely, the treated mixtures exhibited considerably reduced levels of swelling indicated by progressively reduction in axial strain with increasing addition of CMP, as shown in table 1. This indicates a significant reduction in water susceptibility.

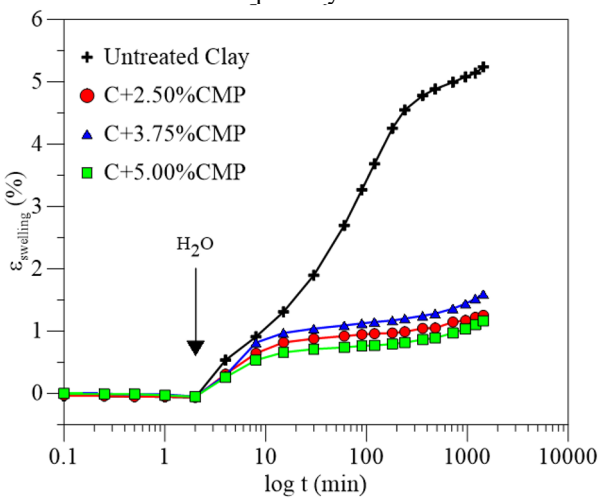

Fig. 3. Swelling response in the saturation phase under oedometer conditions (σ_v = 10 kPa).

The oedometric curves given in Figure 4 show that the CMP treatment has an immediate effect on the volumetric behaviour of the soil. The difference in void ratio at a vertical stress of 10 kPa, before and after the

swelling phase, decreases in the mixtures when compared with untreated clay.

This reduction indicates a slight change in volume in the presence of water, particularly at the highest concentration of CMP. One of the most notable effects is the reduced change in void ratio during the loading phase, indicating that the treated soils undergo significantly less volumetric strain than the untreated one. More in detail, the difference between void ratio, e, at maximum vertical stress (4915 kPa) and at initial vertical stress (10 kPa), after the swelling stage, decreases with increasing CMP content, as reported in Table 1.

Table 1. Results of swelling and oedometer test.

	Untr. Clay	+2.50% CMP	+3.75% CMP	+5.00% CMP
$\varepsilon_{swelling}$ (%)	5.24	1.25	1.59	1.16
$De_{swelling}$ (-)*	0.095	0.047	0.054	0.042
De_f (-)**	-0.432	-0.185	-0.163	-0.135
$\sigma'_{v,y}$ (kPa)	126	1660	1820	1906
Cc (-)	0.248	0.301	0.246	0.200
Cs (-)	0.076	0.023	0.017	0.014

* Void ratio variation due to the initial swelling.
** Void ratio variation due to the loading path (4915 kPa)

These findings indicate that the addition of CMP contributes to the improvement in soil stability, probably due to the progressive formation of cementitious bonds between clay particles during the test, which started immediately after the addition of CMP.

The effectiveness of the CMP treatment is also evidenced by the yielding stress values ($\sigma'_{v,y}$), calculated by the Casagrande method and shown in Table 1.

The untreated sample has a yielding stress of 126 kPa, markedly lower than the treated mixtures, which reach a maximum value of 1906 kPa at the highest CPM dosage. The increased yielding stress is a direct consequence of the bonding mechanisms induced by CMP, which increase the stiffness and reduce the compressibility of the soil. However, despite the marked increase in yielding stress, it is observed that above this value, the slopes of the straight portions of the oedometric curves remain almost unchanged. This indicates that while CMP treatment significantly enhances the yield behaviour of the soil, the post-yield compressibility of the treated mixtures remains comparable to that of the untreated soil. The compression index (Cc) values for the treated samples ranges between 0.200 and 0.301, showing only minor variation compared to the untreated clay, which has a Cc value of 0.248.

A more pronounced difference appears during the unloading phase, where the swelling index (Cs) gives an indication of the ability of the soil to recover volumetric strain after unloading. The untreated clay exhibits a swelling index of Cs = 0.076, more than three times higher than that of the treated samples, which range from 0.023 to 0.014.

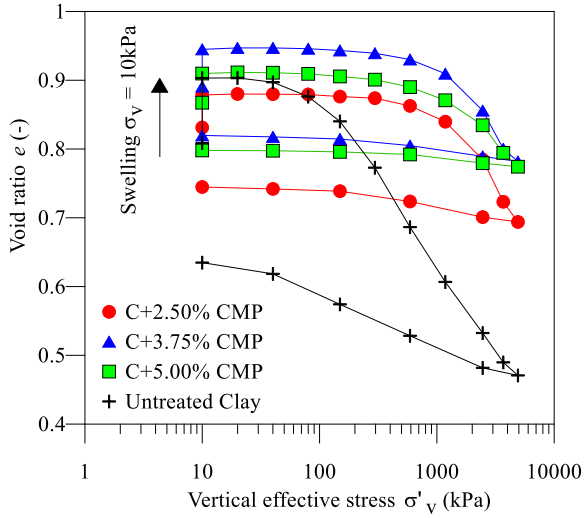

Fig. 4. Oedometric curves for untreated clay and treated mixtures.

Unconfined compressive strength (UCS) testing is a rapid and widely used method for assessing the shear strength of soils. For each mixture, two specimens were tested at each curing time. Time zero (t = 0) is referred to the time at which the mixtures were first prepared. The primary objective of this study was to evaluate the effectiveness of the binder in improving the mechanical strength and long-term stability of the soil. Mechanical strength is a critical factor, as stabilised soils must withstand significant loads without excessive deformation or structural failure. The stress-strain curves of untreated clay and treated mixtures are shown in Figure 5. Each curve corresponds to a representative sample of each mixture at curing times of 0 and 28 days, except for the untreated clay, which is shown only at t = 0. The data indicate that the untreated clay exhibited ductile behaviour, characterised by a gradual decrease in post-peak stress as axial strain increased and by low stiffness. At the time of mixing and compaction (t = 0), the treated mixtures showed a slight increase in strength, followed by a rapid decrease in post-peak stress, indicating a transition from ductile to more brittle behaviour. In particular, the strain at failure of the untreated clay was significantly higher than that of the treated samples, confirming that the addition of CMP induces a change in the mechanical behaviour of the clay from ductile to brittle, even at early curing stages. After 28 days of curing, the stress-strain curves of the treated mixtures show a clear increase in both strength and stiffness. The increase in strength is more pronounced at higher CMP contents, suggesting that a greater amount of pozzolanic products is generated with increased binder dosage. The improvement in stiffness is reflected in the slope of the linear elastic portion of the curve before the peak stress, especially after 28 days of curing. In addition, the treated mixtures at 28 days exhibited a distinctly brittle behaviour, with a rapid decrease in stress after the peak and failure at relatively low axial strains compared to the mixtures at t = 0. These observations confirm that curing time fosters the progression of pozzolanic reactions, resulting in stiffer and brittle material behaviour.

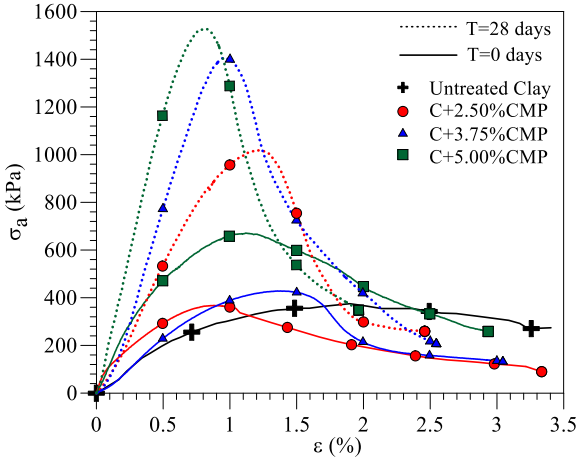

Fig. 5. Axial stress vs axial strain for UCS tests on untreated clay and mixtures at t=0 and t=28 days.

Figure 6 shows the UCS test results for untreated clay and CMP-treated mixtures cured for up to 28 days.

The ultimate unconfined compressive strength was measured on two samples per curing time and only the average is reported. The test results indicate that CMP dosage and curing time have a significant effect on the strength behaviour of the treated clay.

The maximum UCS for untreated clay was recorded at 375 kPa. As no chemical reaction takes place in the untreated samples, the curing time has no effect on its strength; therefore, the UCS value for untreated clay remains constant over the curing times for comparison purposes. In contrast, the addition of 5.00% CMP to the clay resulted in a substantial increase in initial strength at t = 0, reaching 672 kPa.

This immediate increase in strength observed at the highest dosage suggests a rapid interaction between CMP and the clay particles in the presence of water.

However, at lower dosages of CMP, the initial UCS values are comparable to those of the untreated clay. This interaction is closely related to the amount of CMP used: for example, at the ICL and intermediate dosages, the increase in strength is relatively small.

The average ultimate strength values for the 2.50% and 3.75% CMP mixtures at t = 0 are 431 kPa and 427kPa, respectively. After 7 and 14 days of curing, all treated mixtures show a significant increase in strength due to the ongoing pozzolanic reactions.

Between 14 and 28 days, the strength development trend for the 2.50% CMP mixture is almost constant, reaching a maximum UCS of 983 kPa. This suggests that - at this dosage - the available binder is sufficient to create the alkaline environment necessary for pozzolanic reactions, but no additional free calcium ions are available to form new pozzolanic products over the longer period of 28 days. Conversely, mixtures treated with 3.75% and 5.00% CMP continued to develop strength over time, indicating a greater availability of free calcium ions to facilitate further bonding with clay minerals.

This progressive increase in strength validates the effectiveness of the binder in improving the mechanical properties of the treated soil.

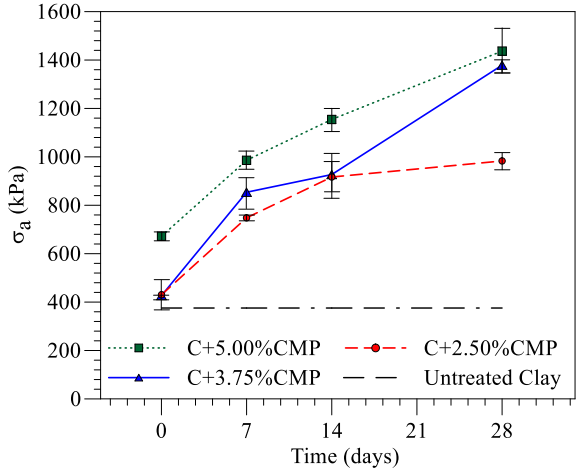

Fig. 6. Evolution of unconfined compressive strength over time for treated samples.

The variation in total suction and water content over the curing time are shown in Figure 7. It is evident that the suction of all samples increased with time. For the untreated clay compacted at optimum conditions, the suction value was approximately 0.8 MPa at 21.6% water content. As it was expected, at the time of compaction, the CMP-treated exhibited suction values similar to those of the untreated clay (ranging between 0.7 and 1.1 MPa), although water content increased with higher CMP dosage. As curing progressed, suction gradually rose to values between 7.6 and 9.1 MPa at t = 28 days. It is notable that these changes in suction occurred with only a minimal loss of water content. For instance, the mixture containing 5.00% CMP had a final water content of 21.3% at t =28 days - comparable to that of the untreated clay - yet it exhibited a total suction of 9.1 MPa. The formation of pozzolanic compounds leads to an increase in micropores and a concurrent reduction in macropore size and connectivity. These changes in pore structure enhance the soil's water retention capacity, thereby increasing its total suction over time.

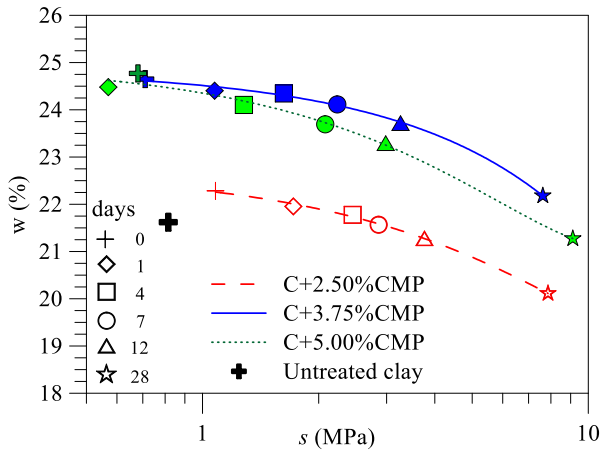

Fig. 7. Suction and water content measurements of the untreated clay and the mixtures over curing time.

The increase in suction can be attributed to the pozzolanic reactions induced by the CMP treatment, which alters the soil's microstructure. The formation of porous pozzolanic compounds contributes to an increase

in the number of micropores. Additionally, these compounds might also bind clay aggregates, resulting in a reduction in macropore size and a modification in their interconnectivity. Such microstructural changes could help explain the observed improvement in the soil's retention behaviour [18], although further investigation is needed to confirm this mechanism.

4 Conclusions

The experimental study carried out in this research investigated the feasibility of using marble cutting sludge, appropriately pre-treated and thermally activated, as a sustainable alternative to traditional industrial binders in the stabilisation of clayey soils derived from excavation activities. The objective is to enable the construction of unsaturated earthen geostructures that better align with the principles of sustainability and efficiency. The results obtained have highlighted the effects of immediate modifications induced by short-term reactions in the treated mixtures. In particular, it was found that the addition of CMP promotes the development of an alkaline environment, which is essential for the first cation exchange and then pozzolanic reactions, leading to a significant improvement in the clay behaviour. With regard to the characteristics of compaction, the behaviour of CMP-treated mixtures is analogous to that observed in lime-stabilised soils, exhibiting a decrease in maximum dry unit weight and increase in optimum water content. The reduction in compressibility evaluations confirmed that compacted CMP-treated samples at their optimum conditions exhibit an improvement in the volumetric behaviour compared to untreated soil, and an immediate reduction in swelling potential. Oedometric tests further revealed a marked improvement in compressibility, with an increase in yielding stress in CMP-treated mixtures. This enhancement is attributed to cementitious bond formation between soil particles, which occurs during the oedometric tests and is accelerated due to a saturated state. In fact, the highest dosage of CMP resulted in the most significant compressibility improvement, confirming the effectiveness of the binder at higher concentrations. The unconfined compressive strength (UCS) results further validated the effectiveness of CMP in improving mechanical properties. The results showed a significant increase in the Unconfined Compressive Strength (UCS) of the CMP treated clay compared to the untreated sample. The progressive increase in UCS over time are indicative of ongoing pozzolanic reactions, which lead to the formation of cementitious compounds that enhance the strength of the clay. This improvement, particularly at higher CMP doses, highlights the effectiveness of the method for soil stabilisation. Changes in suction over the curing period revealed insights into the microstructural evolution due to these reactions and emphasise their role in long-term mechanical behaviour. Although only marginal reductions in water content were observed, the increased suction suggests stronger internal bonding, improving both moisture retention and mechanical performance over time. Consequently, this leads to an improvement in both water retention capacity and overall stability. Collectively, the findings of this study support the potential of CMP as a sustainable alternative to conventional lime-based stabilisers. The binder demonstrated notable enhancements in soil strength, reduced swelling potential, and volumetric strain under load. The observed increase in yielding stress and UCS values suggest that CMP enhances both the short-term and long-term stability of clayey soils. However, in order to provide a comprehensive evaluation of the long-term effectiveness of this innovative binder for stabilisation, further investigation is necessary. Future work should focus on monitoring the evolution of geomechanical properties over time, coupled with detailed microstructural analysis of the treated soils.

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