

Article

Peculiar Features of Lime-Treated Pyroclastic Soils through a Multi-Scale Experimental Investigation

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Abstract: Soil-improvement techniques with binders are used in several geotechnical engineering applications as a sustainable solution for the reuse of waste soils. Due to their inherent complexity and their mechanical behaviour, pyroclastic soils are generally considered waste geomaterials in their natural state. Lime treatment of pyroclastic soils can be considered a viable solution for their reuse in geotechnical applications. In this paper, some peculiar features of the chemo-physical evolution and mechanical behaviour of lime-treated pyroclastic soils are evidenced through a multi-scale experimental investigation. While, for clayey soils, the fine fraction is mainly responsible for ion exchange and pozzolanic reactions induced by lime, for pyroclastic soils, pozzolanic reactions are dominant processes due to the low quantity of clay minerals along with the abundance of aluminates and silicates as the main constituents of their amorphous phase. The link between the phenomena detected at the microscale level, the mineralogical composition, and the macroscopic behaviour of two lime treated pyroclastic soils of different origin is explored through a multiscale approach.

Keywords: pyroclastic soils; lime treatment; mineralogy; microstructure; shear strength; soil dilatancy



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1. Introduction

Soil treatment with lime addition is certainly one of the most widespread methods used worldwide to improve the geotechnical properties of land soil waste, mainly clayey soils. This technique appears to be very effective [1–12] and durable [13–16] for the reuse of soils that are not suitable for earthworks in their natural state. During the construction of large infrastructure and geotechnical works, the surplus of unsuitable soil is a common occurrence leading to the serious problem of disposing of excavated material in dedicated areas. This issue is crucial and timely, since, from a sustainability perspective, there is an increasing and urgent need to reuse “local soils” in geo-environmental and geotechnical projects. Therefore, the reuse of excavated soil can be considered a sustainable opportunity if an appropriate technology, based on the geotechnical properties of the excavated waste soil, is adopted.

The possibility of using lime treatment technique for natural pozzolanic soils of pyroclastic origin, although rather unsuitable for earthworks in their natural state, has represented the first goal of a wide experimental research carried out by the authors in the last decade on three Italian volcanic soils, each one different in its geological origin and geotechnical properties [17–20]. The results of these previous studies has clearly enhanced the effectiveness of the treatment in terms of soil improvement, and they are motivating the current research, focusing on considering natural pozzolanic soils as resources for innovative materials and acting as precursors for alkaline activated binders in soil treatment.

It is certainly well known that volcanic soils and rocks are tricky and complex soils due to their origin, mineralogical composition, structure, and fabric, which together lead to a definite hydro-mechanical behaviour upon loading, in both saturated and unsaturated

conditions. In particular, pyroclastic soils deposits, whose “*origin is a consequence of rapid energy changes whereby magma moves and is violently erupted from a deep higher temperature and higher pressure to the earth’s surface*” [21] are found all over the world, and their characteristics are strictly correlated to the eruptive mechanisms. The attention of geologists and geotechnical engineers to the hydro-mechanical properties of such materials has increased in recent years, as also testified by international workshops on volcanic rocks held in representative volcanic fields, i.e., Madeira and Azores (Portugal), Tenerife (Spain), Ischia island (Italy), and very recently Fukuoka (Japan, 2022). In Italy, natural pyroclastic soils and rocks form the deposits in the subsoil of large and highly populated cities in Central and Southern Italy, like Rome and Naples. In addition to their inherent complexity as natural soils, some lithotypes are considered waste geomaterials since some specific aspects of their mechanical behaviour, such as the well-known structural collapse upon wetting, may represent a limitation to their use and possible reuse as construction geomaterials.

Italian natural pozzolanic soils of pyroclastic origin are very diffuse in Italy, covering a total extent of 8000–9000 km². The adjective “pozzolanic” originates from the Latin word *lapis puteolanus*, and it is generally associated with the pyroclastic deposits that are widespread in the Campania region (Pozzuoli, Naples–Italy). The term was only later extended to encompass a broader meaning, and in the literature, it now refers to materials rich in silica or aluminosilicates that react strongly with calcium ions dissolved in pore water. However, due to their distribution over large areas that have been densely settled since historic times, natural pozzolanic soils have been exploited since the Roman times and have been used from those ancient times to the present day as building blocks to produce hydraulic mortars, cements, and road pavements. Persistent mining activities in pozzolanic deposits have resulted in complex and intricate networks of underground cavities and tunnels in urbanized areas. In several cases, the formation of subsidence troughs or, in the most severe cases, sinkholes at the ground surface occurred and still occur at present. These materials are generally considered problematic soils due to their nature, mineralogical composition, intrinsic heterogeneity, and microstructure features. Studies focusing on the lime treatment of pyroclastic soils for geotechnical applications are rather recent. Cecconi et al. [22], Cecconi and Russo [17], and Russo et al. [18] proved the suitability of pyroclastic soils to lime treatment and showed the effects of treatment parameters (i.e., lime per cent by weight, compaction energy, and initial water content) on their mechanical performances. Guidobaldi et al. [19] investigated the mechanical improvement induced by lime addition on a chabazite-rich pyroclastic soil coming from the Orvieto cliff, in Central Italy, highlighting the development of a structured behaviour linked to the chemo-mineralogical evolution of the system and showing the effectiveness of the improvement technique for coarse-grained soil. While, for clayey soils, a fine fraction is mainly responsible for ion exchange and pozzolanic reactions induced by lime [23], for coarse-grained pyroclastic soils, pozzolanic reactions are prevailing processes due to the low quantity of clay minerals in the fine fraction and, contextually, the abundance of aluminates and silicates, especially when exchanging minerals such as zeolites are absent.

In the present paper, the peculiar features of the chemo-physical evolution and the mechanical behaviour induced by the addition of lime to pyroclastic natural soils are discussed. The experimental results, as representative of typical effects induced by lime on pyroclastic soils, refer to two lime-treated Italian pozzolanic soils of different origin and nature. The first one belongs to the fallout deposits from the Somma–Vesuvius volcanic complex (Naples, Italy), while the second one is a black coarse-grained pozzolana derived from the Colli Albani volcanic complex (Rome, Italy) and belonging to a pyroclastic flow deposit (see their location on the map in Figure 1). The link between phenomena taking place at different scales—namely particle level, microstructure, and element of volume—is explored through a multi-scale experimental investigation. This represents the main goal of the paper, and the thorough study which is based on the results obtained from a very wide experimental campaign on pyroclastic soils typical of Central and Southern Italy, here revised upon a different view and scale of observation.



Figure 1. Location of investigated pyroclastic deposits: (a) volcanic districts in Central Italy; (b) Monteforte Irpino site (adapted from [24]); (c) Pozzolana Nera outcrops.

2. Lime Treatment of Pyroclastic Soils

Main typical behaviours of pyroclastic soils as a consequence of lime addition are discussed following an experimental multi-scale approach. Some aspects related to the chemo-physical evolution of the solid-lime-water system are considered, with a higher prevalence of pozzolanic reactions than ion exchange reactions for these materials. At the microstructure scale level, it is shown that the addition of lime does not lead to the re-organisation of grains in the solid skeleton, as generally observed for natural soils rich in clay minerals: the amorphous phases do not change their state of aggregation as a result of variations in the pore water chemistry. The direct link between the chemo-physical evolution of the system and the mechanical behaviour, whose features are those typical of “structured” material, is highlighted by means of triaxial testing on lime-treated samples. These peculiar features are exemplified and highlighted through some experimental results from two different Italian pyroclastic soils. Chemical and microstructural analyses were performed on these two pozzolanic soils, in both raw and treated conditions. Micro-X-ray Fluorescence (μ -XRF), atomic absorption spectrometry (AAS), X-ray Diffraction (XRD), Thermogravimetric Analysis (TGA), mercury porosimeter intrusion (MIP) tests, and scanning electron microscopy (SEM) analyses were performed to investigate the chemo-physical evolution and microstructure of the pyroclastic soils.

2.1. Investigated Soils

Monteforte Irpino pyroclastic soil (MF) is a weathered and humified ashy soil belonging to the stratigraphic succession of Somma–Vesuvius eruptions products on the north side of Mount Faggeto. This pozzolana characterizes the upper layers of the succession of about 40 km northwest of the Somma–Vesuvius volcano (Figure 1b) and belongs to its shallower more recent unit, i.e., fallout deposits including millimetric greenish pumices, which are monogranular and strongly porphyritic with leucite and biotite crystals, as well as lavic and carbonate lithic fragments. The relevant hydro-mechanical properties of this

soil are discussed in [24]. The bulk mineralogy of MF soil was determined by XRD on randomly oriented samples (Figure 2a). Monteforte soil mainly contains amorphous phases with small amounts of quartz, kaolinite, anorthite, and muscovite/illite. In terms of grain size distribution, MF soil is a silty sand (Figure 2b). At SEM, the pores' and grains' shape and size variability are observed (Figure 2d).

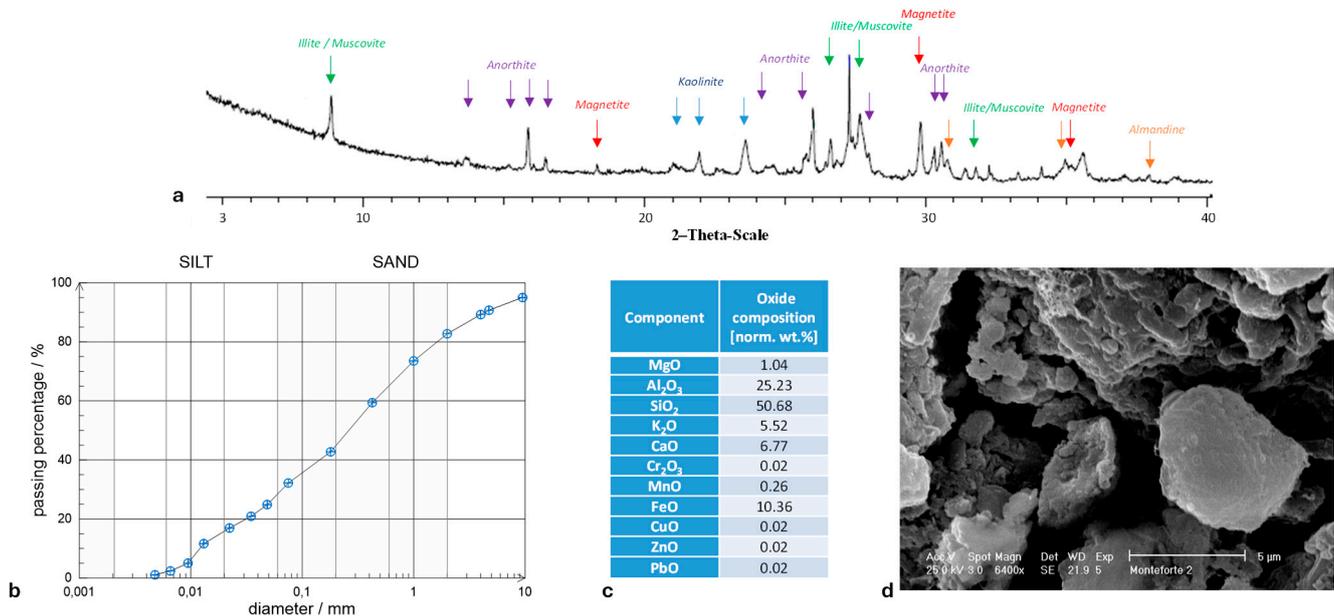


Figure 2. Monteforte pyroclastic soil: (a) X-ray Diffraction pattern; (b) grain size distribution; (c) elemental composition; (d) SEM micrograph 6400×.

Pozzolana Nera (PN) is a coarse-grained pyroclastic weak rock from the area south-east of Rome, which originated from the third cycle of intense activity of the volcanic complex of the Colli Albani, still quiescent, dating to the Upper-Middle Pleistocene [25,26], and belonging to the Vulcano Laziale lithosome unit [27]. The geotechnical properties of this material have been extensively investigated [17,20,22,28,29]. Most of the Southern part of the city of Rome was built on these pyroclastic products, whose deposits are generally massive and chaotic, with a maximum thickness of approximately 20 m.

As shown in Figure 3, XRD analyses allowed us to define the mineralogical composition of Pozzolana Nera from Rome (PN soil), consisting mainly of crystals of leucite and augite, with albite and muscovite, whereas a small amount of zeolites (analcime) has been found [29]. Rietveld analysis of X-ray Diffraction results allowed a quantitative evaluation of the amorphous phases, which were present in about 27% of the minerals. According to the grain size distribution shown in Figure 3b, the selected material is classified as medium sand. High values of the surface area, which vary from 35 to 25 m²/g as grain size increases [17], enhance the reactivity of PN soil.

2.2. Chemo-Physical Evolution and Impact on Microstructure

Despite the scarcity of clay minerals, the high reactivity of pyroclastic soils is ensured by the relevant amount of amorphous phases that are rich in silicon and aluminium. In an alkaline environment, this favours the development of pozzolanic reactions in the short term. Moreover, no significant ion exchange phenomena are expected soon after the addition of calcium due to the scarcity of exchanging minerals (i.e., clay minerals, zeolites).

Cambi et al. [29] have demonstrated that pozzolanic reactions develop in the short term for lime-treated PN soil, thus highlighting the remarkable reactivity of this pyroclastic soil–lime–water system by means of atomic absorption measurements. PN samples at increasing lime percentages were prepared by adding 0.4 g of lime and dry PN soil such that the lime percentages were equal to 2%, 5% and 10%; 500 mL of distilled water was

added to each mixture so that the calcium concentration in water is fixed at the same value of 294 ppm, lower than the concentration upon saturated conditions. Figure 4 shows the consumption of calcium over time, which is at a maximum for the sample treated at the lowest percentage of lime, i.e., the maximum amount of solid phase. The reduction in calcium concentration after one hour clearly reveals the development of pozzolanic reactions in the very short term.

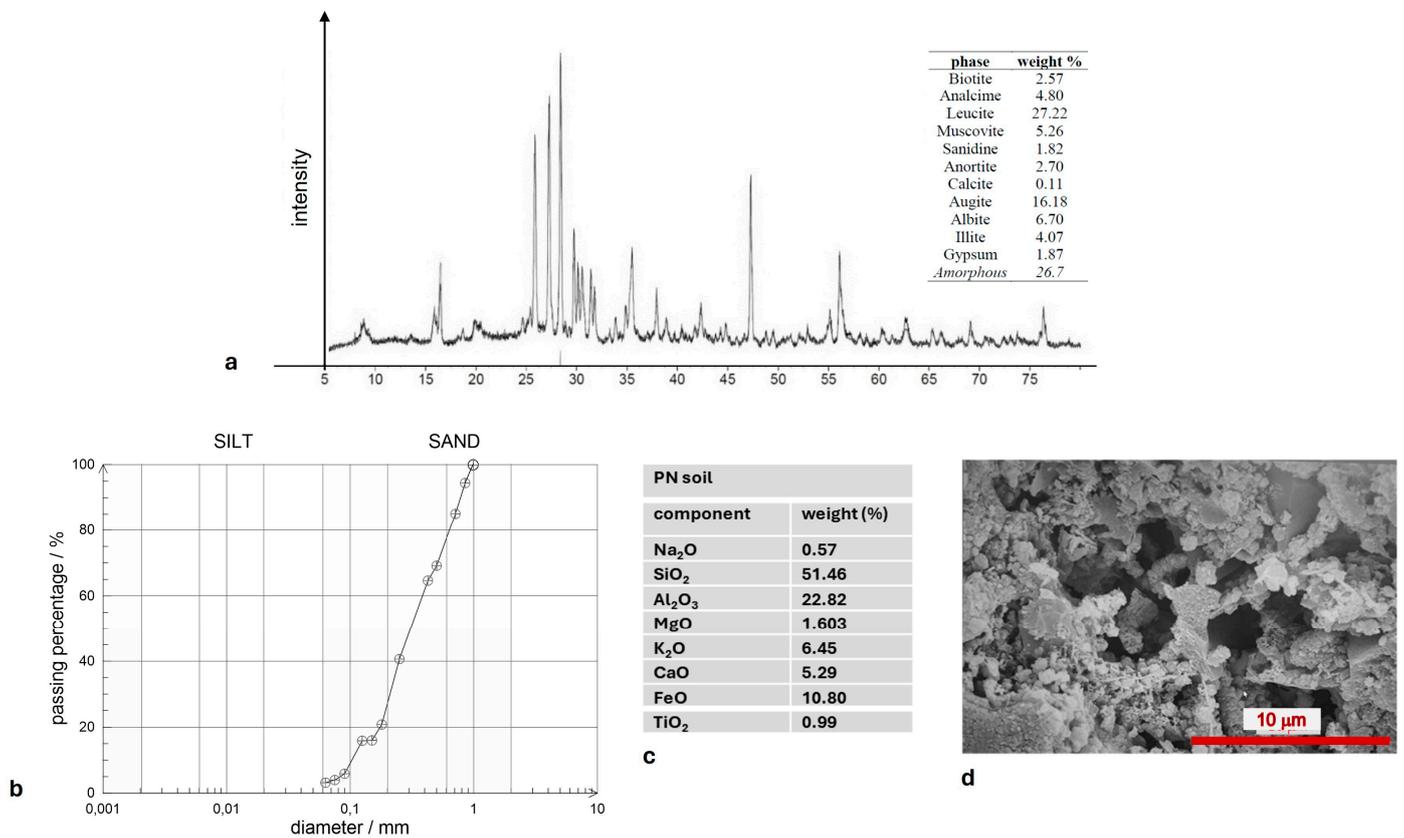


Figure 3. PN soil: (a) X-ray diffraction pattern; (b) grain size distribution; (c) elemental composition and (d) SEM micrographs at 4500×.

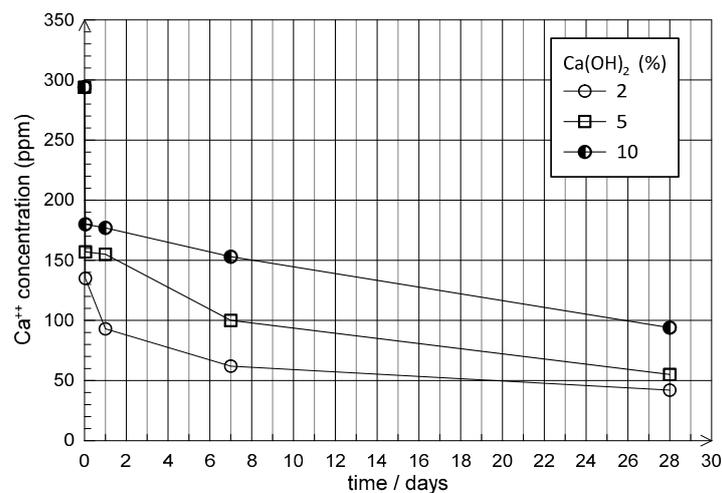


Figure 4. Atomic absorption measurements on lime-treated PN soil (adapted from Cambi et al. [29]).

The evidence of the high reactivity of pyroclastic soil can be deduced from soil pH measurements performed on lime-treated samples. On PN samples prepared according to

the ASTM D6276-99a standard [30] with increasing lime content, Cambi et al. [29] measured the pH values with increasing curing time (1 h, 24 h, 2 d, 4 d, 6 d, 14 d).

The amounts of lime for sample preparation ensure over-saturated calcium samples. The pH value of calcium-saturated solution, equal to 12.4 [31], is never reached in the first hour of reaction for any of the samples (Figure 5a). As no ion-exchanging minerals (i.e., clay minerals and zeolites) are present, the consumption of calcium ions is consistent with the development of pozzolanic reactions in the very short term. The subsequent attainment of the target pH value results from the ionic dissociation of excess lime in the system. Measurements of pH at increasing curing times (Figure 5b) show that pozzolanic reactions induce calcium consumption with a consequent reduction of pH, which is greater for samples treated with lower lime percentages.

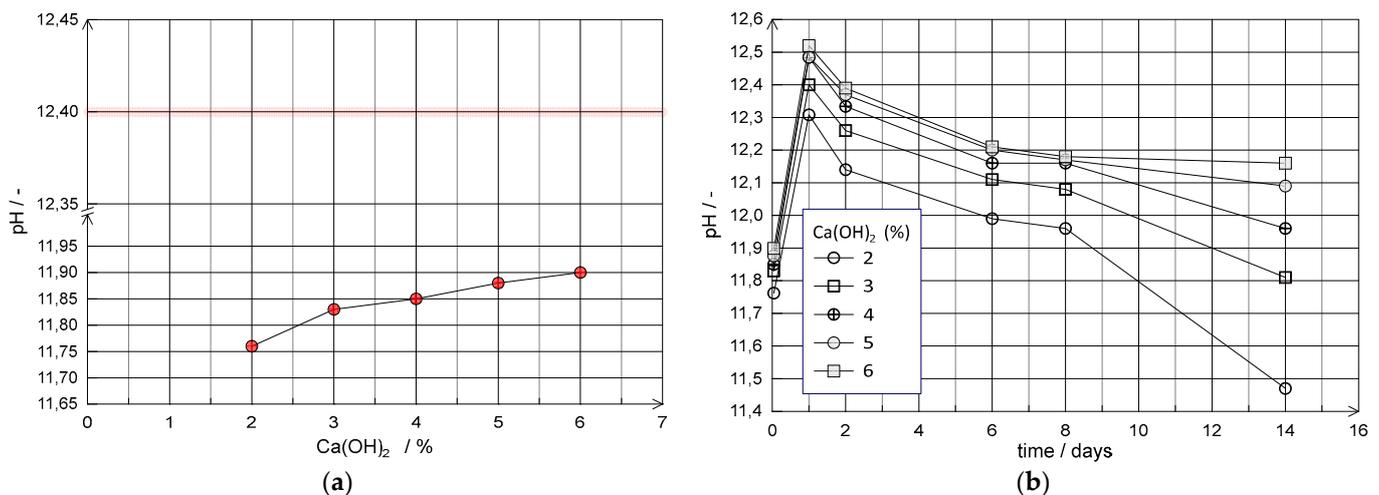
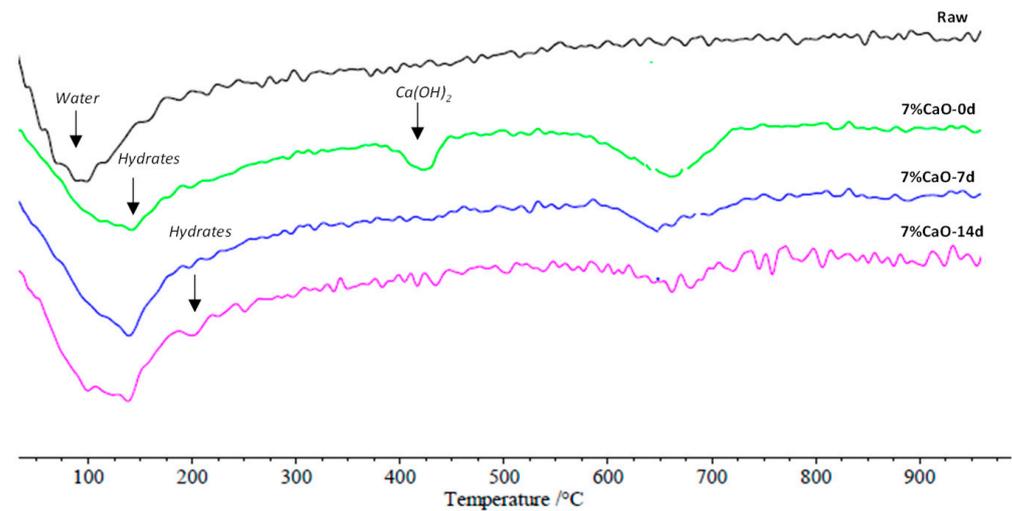


Figure 5. pH measurements on lime-treated PN soil: (a) 1 h pH measurements at increasing lime content (the red line represent the target pH value after lime addition); (b) pH trends over time for samples with increasing lime contents (adapted from Cambi et al. [29]).

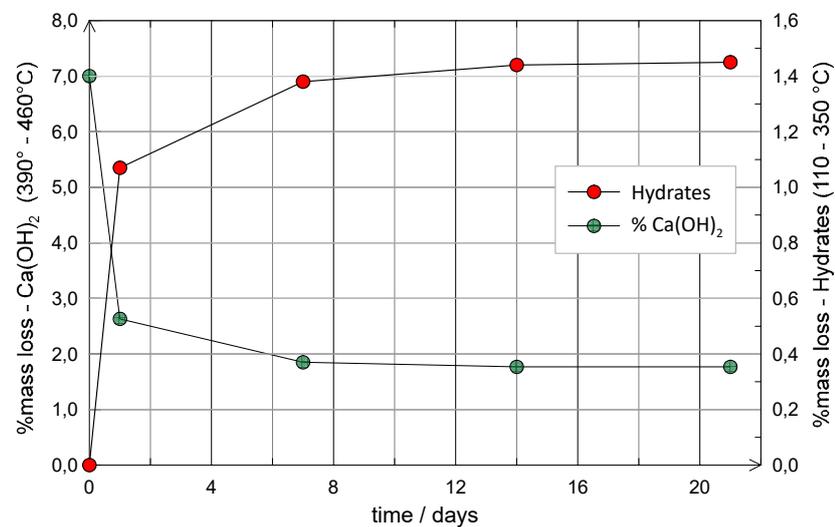
Russo et al. [18] subjected MF samples prepared at 7% CaO to thermogravimetric analyses at increasing times (i.e., 24 h, 7 d, 14 d). From the DTG results, shown in Figure 6a, the progressive dissolution of portlandite (390–460 °C), already relevant after 7 days of curing, and the formation of hydrated phases (130–350 °C) are observed in the short term. The rapid development of the pozzolanic reactions over time is proven by the trends of portlandite dissolution and secondary hydrated phase precipitation over time, shown in Figure 6b by the quantitative interpretations of the thermogravimetric analyses.

Pyroclastic soils do not undergo substantial microstructural reorganisation as a result of the addition of lime, as generally observed for soils rich in clay minerals. While in the presence of clay minerals, which are electrochemically active, the interaction with calcium ions causes a redistribution of the net charge on the particle surface and consequently a different aggregation [32], due to the absence of clay minerals, the aggregation of particles within the solid skeleton is not altered.

The effect of gel formation resulting from the development of pozzolanic reactions can be detected by mercury intrusion porosimetry analyses at increasing curing times. Figure 7 shows the results of two analyses performed on PN samples, prepared at the same initial dry density and water content, treated with 5% Ca(OH)₂, and left to cure for 1 day and 90 days, respectively. The distribution of pores over time remains bimodal (Figure 7a), indicating the absence of microstructural reorganisation of the samples. The lower pore frequency observed in the long term for each of the modal dimensions is consistent with the precipitation of hydrated gels on the surface of solid particles, reducing the pores' accessibility. The reduction in total porosity in the long term is also shown by the cumulative intrusion curves plotted in Figure 7b.



(a)



(b)

Figure 6. Thermogravimetric analyses of lime-treated MF soil: (a) DTG at increasing curing times; (b) quantitative interpretation of TGA analyses.

2.3. Soil—Dilatant Behaviour upon Shearing

In this section, the attention is focused on the behaviour upon shearing of raw and lime-treated PN soils, which was investigated through triaxial tests by adopting a conventional triaxial apparatus [33]. Lime-treated samples were prepared by hand-mixing the soil with a fixed amount of quicklime powder, precisely 10% CaO and distilled water ($w/c = 20\%$). Samples were tested at different curing times, namely 0 (raw, i.e., not-treated), 7, 14, days and 105 days. The soil-lime–water mixture was then “dynamically” compacted in thin layers inside special aluminium cylindrical moulds (internal dia. = 38.1 mm) made of two split parts and bolted together during sample preparation (see Figure 8). A hollow cylindrical mallet of mass 850 g sliding along a vertical bar was used for the compaction of each soil layer, with the mass being allowed to drop from a target height through a fixed number of consecutive blows in order to obtain a target voids ratio in the range of 1.0–1.1, i.e., a typical average value for the investigated material; see, for example, [28]. Sample extrusion was made after 5 days from initial preparation and compaction. The specimens were then cured inside sealed containers to avoid variations in water content.

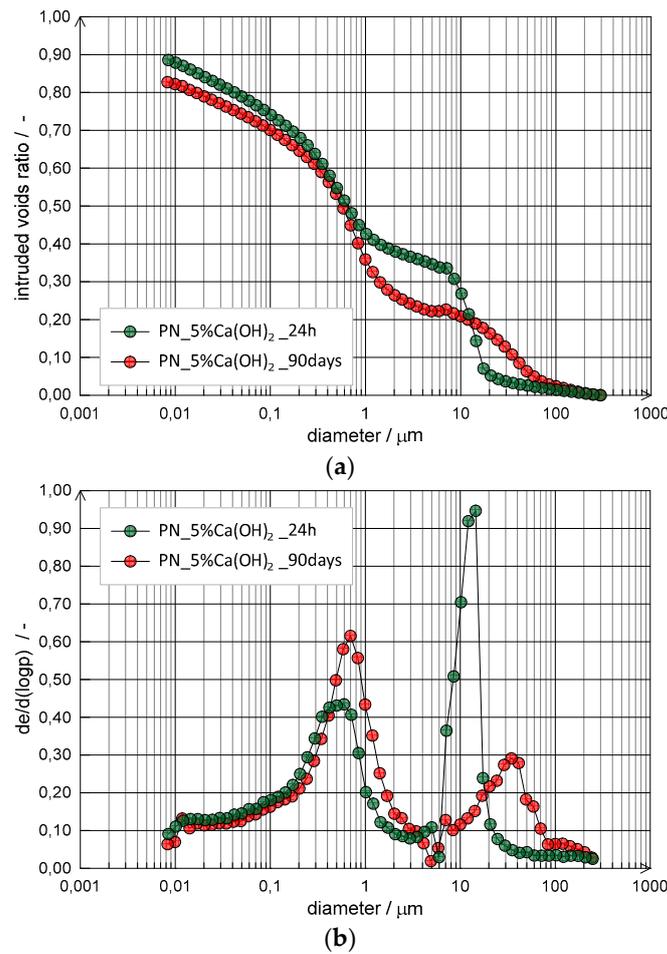


Figure 7. Mercury intrusion porosimetry on lime-treated PN soil: (a) cumulative intrusion; (b) pore size distribution function.

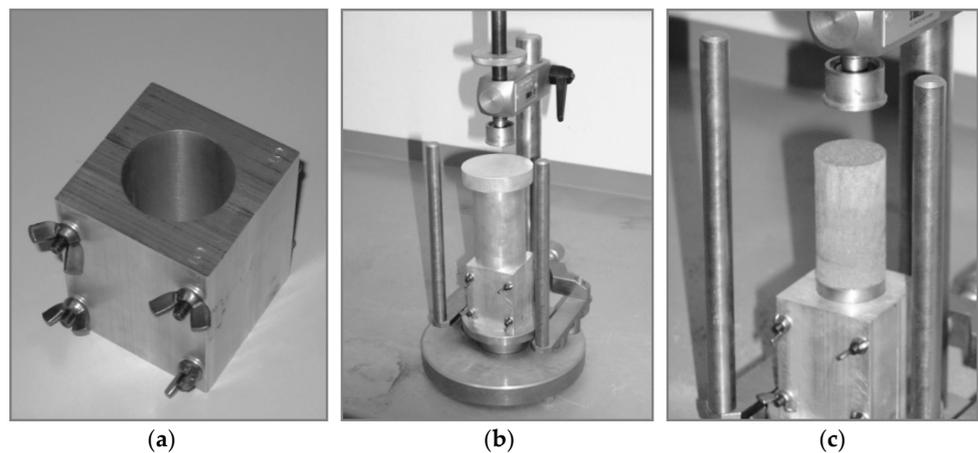


Figure 8. Equipment for triaxial samples preparation: (a) split mould; (b) sample extrusion device; (c) PN-treated soil specimen after extrusion.

Conventional consolidated drained triaxial tests were performed at different increasing stress levels (50–400 kPa) on treated PN samples that were previously saturated under a vacuum. The stress–strain behaviour observed for PN soil is shown in Figure 9 in terms of deviator stress, q vs. axial strains ϵ_{ax} (Figure 9), and measured volume strains ϵ_{vol} vs. ϵ_{ax} (Figure 9).

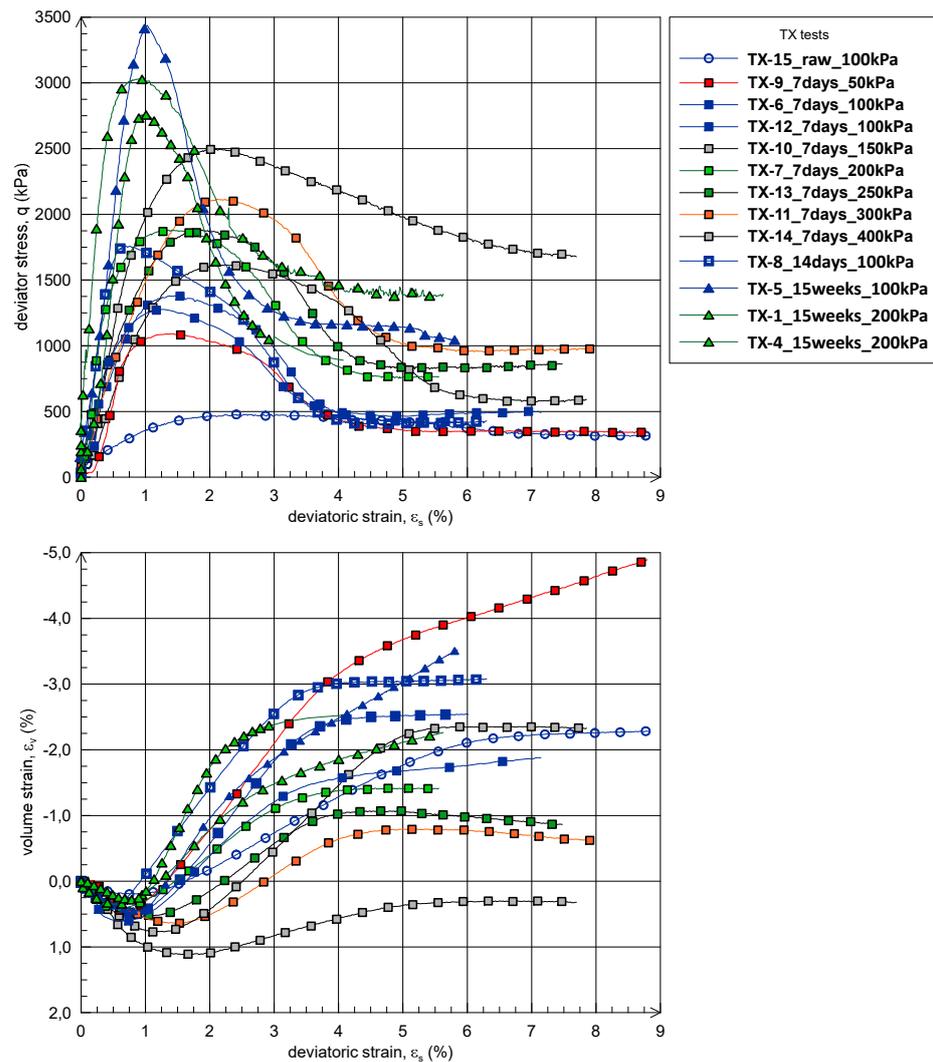


Figure 9. Triaxial compression tests on raw and treated PN samples: deviator stress, q ; deviatoric strain, ε_s (**top of figure**); volume strain, ε_v ; deviatoric strain, ε_s (**bottom of figure**).

The results clearly highlight the expected effect of confining stress and curing time on the stress–strain behaviour. At low stress level (50 kPa) and a 7-day curing time, the brittle behaviour of PN soil—associated with soil dilatancy (i.e., negative volume strains)—is indicated by an abrupt decrease in deviator stress, which occurs immediately after the peak strength mobilization. The observed brittle behaviour is reduced as confining stress increases. On the other hand, the stress–strain behaviour for raw, not-treated PN soil is undoubtedly ductile, even if it is still associated with positive volume strains (blue open circles in Figure 9). For samples cured for very long times (i.e., 105 days), whose shear strength is certainly increased due to the formation of cementitious compounds in the long term, the behaviour is systematically fragile and dilatant, independent of the applied stress level.

A selection of results obtained from TX tests on raw samples and samples cured at 7, 14, and 15 weeks is plotted in Figure 10 in terms of the mobilised stress ratio q/p' . It is noted that even if volumetric strains still develop, the value attained in “end-of-test” conditions falls within a narrow range of values ($M = q/p'_{\text{eot}} = 1.5\text{--}1.8$) corresponding to a soil friction angle $\phi'_{\text{eot}} = 37\text{--}40^\circ$; i.e., both the raw samples and the treated samples exhibit—approximately—the same shear strength at large strain levels. This is true for all the investigated stress levels within 7–14 days after lime addition. This is consistent with

the results obtained from different laboratory investigations on natural and reconstituted PN raw samples [17,20,29].

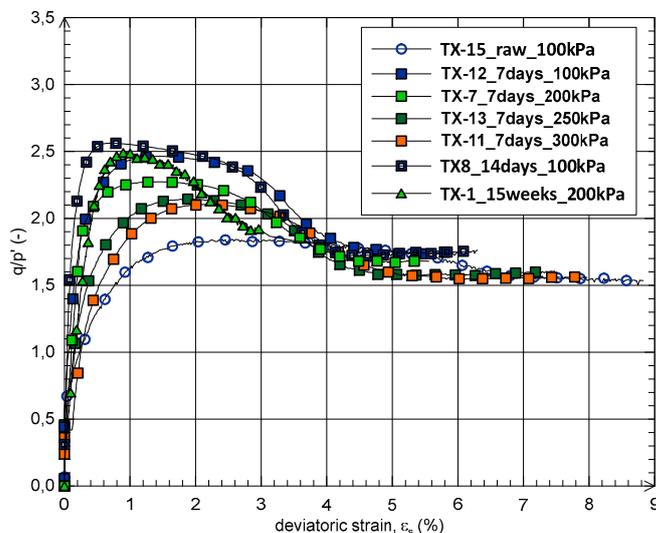


Figure 10. Triaxial compression tests on raw and treated PN samples: mobilised stress ratio vs. deviatoric strain for raw and treated PN samples for different curing times and applied stress levels.

For the same set of results, the maximum value of stress ratio is also plotted in Figure 11a as a function of the effective radial stress, σ'_r . As mentioned before, the results unquestionably suggest that the brittleness of the material decreases with the applied stress level. As expected, for a given stress level, the larger the curing time, the larger the mobilized peak friction angle. The role of curing time is also investigated in terms of soil dilatancy behaviour (see Figure 11b). Here, dilatancy is simply calculated as the ratio $-\delta\varepsilon_{vol} / \delta\varepsilon_s$; i.e., negative values of volume strains lead to positive values of dilatancy. The maximum stress ratio increases with soil dilatancy calculated at the same test condition. However, the effect of curing time on soil dilatancy does not prevail on the effect of confining stress.

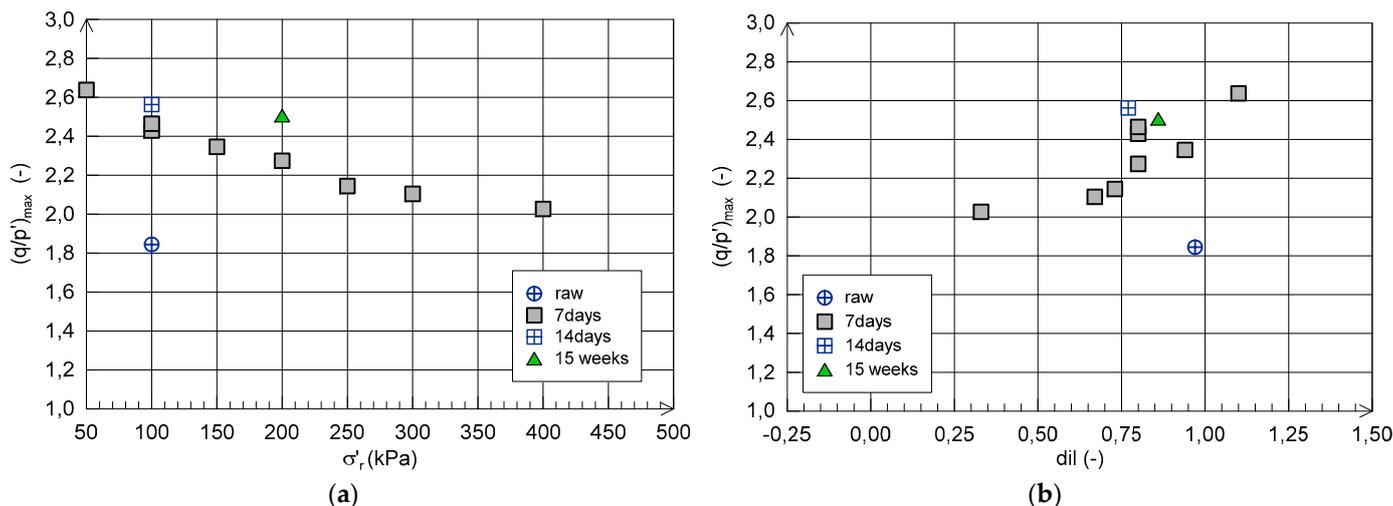


Figure 11. Triaxial compression tests on raw and treated PN samples. (a) Maximum mobilised stress ratio vs. radial effective stress; (b) maximum mobilised stress ratio vs. soil dilatancy @ $(q/p')_{max}$.

3. Concluding Remarks

Due to some peculiar aspects of their mechanical behaviour, such as structural collapse upon wetting, pyroclastic soils in their natural state might be considered waste geomaterials.

Lime treatment, which is recognized to be a widely used technique for the mechanical improvement of fine-grained clayey soils, has proved to also be effective for pyroclastic soils. From this perspective, pyroclastic soils represent a natural resource for sustainable civil infrastructures.

The results of a recent extensive experimental investigation on the effects of lime addition on the microstructure and geotechnical features of pyroclastic soils from Central and Southern Italy, different in nature and mineralogical composition, allowed to draw some conclusions on the peculiar aspects of their behaviour after the addition of lime at different scale of observation. In this paper, these peculiar aspects are evidenced with reference to experimental results of two pyroclastic soils, coming from different volcanic districts. The experimental research using a multiscale approach allowed to understand the link between chemo-physical evolution, microstructure modifications, and the mechanical behaviour of lime-treated pyroclastic soils.

The main peculiar aspects can be listed as follows:

- (a) The chemo-physical evolution is mainly due to the pozzolanic reactions, promoted by the dissolution of silica and alumina in a highly alkaline, calcium-rich environment, whereas low ionic exchange is due to the low percentage of exchanging minerals;
- (b) The hydration products of pozzolanic reactions precipitate in the very short term in the system, highlighting the relevant reactivity of the mainly amorphous solid phase;
- (c) The aggregation of particles forming the solid skeleton is marginally altered by the addition of lime due to the low amount of clay minerals;
- (d) The formation of cementitious compounds in the long term favours the tendency of the material to exhibit a brittle and dilatant stress–strain behaviour independently of the applied stress level.

The improved mechanical behaviour of the investigated pozzolanic soils, observed after the addition of lime, represents an interesting result for the practical application of lime-treatment techniques in the fields of engineering and geology/geotechnical engineering. Through a wide experimental research, the study has shown the effectiveness of the lime treatment technique—originally devoted only to fine-grained materials—and its applicability to medium/coarse-grained pozzolanic soils.

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Data Availability Statement: Data are contained within the article. The authors are able to share their research data.

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Conflicts of Interest: The authors declare no conflicts of interest.

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