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# The role of intrinsic soil properties in the compressive strength and volume change behavior of unstabilized earth mortars

Nikiforos Meimaroglou · Charalampos Mouzakis

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Abstract Despite the growing interest in earthen construction, there is critical lack of reliable experimental data on the soil properties which mostly affect the engineering characteristics of the dried building material. Therefore, the main objective of this research was to explore the influence of some of these properties, namely clay fraction content, Specific Surface Area (SSA), Cation Exchange Capacity (CEC), chemical and mineralogical composition and various forms of iron and calcium carbonate on earth mortars. The initial trigger for this research was the extraordinary compressive strength of four earth mortars prepared with different soils. So, these four soils, along with seven others from previous research, were thoroughly examined using soil science techniques to investigate the link between soil properties and compressive strength and linear shrinkage of earth mortars. A relationship between the compressive strength to CEC ratio and dry density was found, highlighting the decisive role of clay activity as expressed by CEC, in earthen materials properties. According to linear regression and dominance analysis, the strongest correlation was exhibited by SSA followed by CEC, demonstrating that compressive strength is largely dependent on these two properties. Less

N. Meimaroglou  $\cdot$  C. Mouzakis ( $\boxtimes$ )

Laboratory for Earthquake Engineering, School of Civil Engineering, National Technical University of Athens, Zografou Campus, 9 Iroon Polytechniou str., 15780 Zografou, Greece e-mail: harrismo@central.ntua.gr strong correlation was found for clay fraction content, while poorly ordered/amorphous iron oxides were found to correlate with strength and shrinkage, but their contribution requires further research. Regarding the mineralogical properties, it was found that the mortars that achieved the highest strengths contained poorly crystalline smectite clays. Finally, even significant differences in soil chemical composition did not necessarily lead to different mortar properties.

**Keywords** Soil specific surface area (SSA) · Cation exchange capacity (CEC) · Iron oxides · Earth mortars · Compressive strength · Dominance analysis

### 1 Introduction

Raw earth, processed with various techniques, has been the principal building material of mankind for millennia, as testified by historical sources and prehistoric archaeological remains [1–4]. Various earth building techniques were widespread throughout the world and even today it is estimated that one third of the human population resides in earthen houses [5, 6]. However, steel and concrete dominance in twentieth century construction led to the marginalization of earth building and other traditional construction methods.

In the recent past, environmental and sustainability concerns have renewed interest in earth building, resulting in a tenfold increase of scientific studies



on earth construction in a single decade [7]. The subsequent development of the earth construction sector has so far led more than 20 countries to introduce normative documents and a legislative regulatory framework for various earth building techniques [8, 9].

This increased attention. along with the heterogeneous, multiphase and multimineralic nature of the fundamental raw material, the soil, has attracted scientists from different scientific disciplines, making earth construction an interdisciplinary field. Despite the accelerated progress in many aspects of earth construction, research is hampered by a lack of insight regarding the inherent properties of soil that contribute to the mechanical and physical properties of building materials, the most important of which are compressive strength and volumetric shrinkage upon drying. Even clay fraction content, one of the most frequently cited properties in standards and papers, is implemented as a soil suitability index mostly through empirical thresholds rather than systematic research. As a result, only few studies [10-13] have dealt with the relation between clay fraction content and physical and mechanical properties of the building material. Other intrinsic soil properties that are postulated to affect earthen building materials properties, such as Soil Specific Surface Area, iron and calcite content, are rarely assessed in literature and insufficiently, if at all. studied.

The main objective of the present research is to explore the influence of some intrinsic soil properties on the dry strength and volume change of earth mortars. The starting point for this research was the need to explain the extraordinarily high compressive strength (over 10 MPa) exhibited by mortars fabricated with four soils from northern Greece, after failing to find a conclusive answer in literature, along with further questions arising from the authors' previous research [12]. The decision was thus made to employ a wide range of soil science techniques in order to gain a better understanding of which soil properties could be associated with the engineering properties of earthen building materials.

The properties examined in this study are primarily those of the clay fraction, as this soil fraction can be considered the binding phase of earthen building materials that imparts cohesion and strength. Various studies such as those of Lima et al. [14], Meimaroglou and Mouzakis [12] and Lagouin et al. [13] have demonstrated that clay fraction properties exert a strong influence on the properties of earth mortars and this is the main reason why this particular material was selected as the reference earthen building technique. Thus, the results primarily refer to earth mortars but, considering the limitations of the material's characteristics and testing methods, general trends could be extended to other earth building techniques. Emphasis was placed on the role of SSA and the different forms of iron and calcite, properties believed to have a deep impact on the mechanical behavior of earthen materials, although there are few reliable data in literature to confirm this.

1.1 Compressive strength of earth building materials

# 1.1.1 Interparticle forces and mechanisms of strength gaining

Compressive strength is considered to reflect earthen material quality and is one of the basic properties in design codes and standards. While considerable progress has been made in understanding the mechanisms of cohesion, no scientific consensus has been reached on the effect, positive or negative, of various factors and soil properties on strength. Even the testing procedures for assessing compressing strength are a matter of dispute, leading to inconsistencies and erroneous results [15–18]. According to "traditional" geotechnical engineering, cohesion is attributed to a variety of interparticle attractive forces, which are:

- (a) Electrostatic forces. The preservation of electrical neutrality of clay particles, which have a residual negative charge caused primary by isomorphous substitution, requires the adsorption of cations onto their surfaces. Divalent and trivalent cations like Ca<sup>2+</sup> and Fe<sup>3+</sup> can form a network binding adjacent particle of clay, while monovalent ions such as Na<sup>+</sup> can cause their repulsion [5, 19, 20].
- (b) Electromagnetic forces. Another source of attraction is the fluctuating dipole bonds, the Van der Waals forces, which, in conjunction with double layer repulsions, describe the interaction of double layers according to the Gouy-Chapman theory.
- (c) Aggregation and cementation, caused by the precipitation of iron oxides, aluminum, calcium



carbonate etc., and involving chemical bonding between constituents [21], are also considered soil particle binding mechanisms [22].

(d) Capillary forces in partially saturated soils. Negative pore water pressure also generates an effective compressive stress among particles.

According to unsaturated soil mechanics, a clear demarcation exists between the forces to be considered in a saturated and an unsaturated soil. Saturated soil can be treated as an equivalent continuum medium under the combined action of forces propagating through the grains and counterbalancing forces, e.g. Born's repulsion, acting at the interatomic level. Contrarily, local interparticle forces must also be considered for an unsaturated soil, with forces arising from surface tension and negative pore-water pressure having a significant contribution [23]. The adaptation of concepts from unsaturated soil mechanics for the testing and understanding of earthen building materials' mechanical behavior constitutes a major step forward in earth construction. This novel perspective is particularly applicable to rammed earth, which is treated as a manufactured unsaturated soil [24-26]. In this context, the role of water and moisture is highlighted, suction plays a key role in strength gaining and the prevalent attractive forces are those due to capillary action [27–29]. Capillary forces are affected by the clay platelets orientation and recent studies have demonstrated that their rearrangement with the use of clay dispersants can significantly increase strength [30-32]. Finally, regarding the strength mechanism and the predominant attractive forces, Bui et al. [27] suggested that cohesion is attributed to capillary forces for sandy soils, while for clayey soils, additional forces such as the Van der Waals forces also contribute.

### 1.1.2 Soil properties related to strength

Regardless of the approach to the strength gaining mechanism of earthen building materials and the principal attractive forces, there is consensus that the mechanical behavior depends greatly on the properties of the raw material, the soil. In this respect, this research followed a soil science approach, focusing on soil properties which are widely believed, but not yet categorically proven, to influence mechanical properties. These properties, besides clay fraction content and CEC which were investigated in Meimaroglou and Mouzakis [12] and more recently in Lagouin et al. [13], are:

- (a) Soil Specific Surface Area, a property which in the field of earthen building materials, is considered a property of particular importance, associated with clay-water interaction and the building material's required clay content, modulus of elasticity, compressive strength and drying shrinkage [33, 34]. However, a direct correlation between SSA and building materials properties has not yet been established in literature, and the link is based on CEC and methylene blue value measurements [34, 35].
- (b) Iron oxides which are considered an effective cementing and stabilizing agent of natural soils, inducing soil aggregation and promoting stability against water [36, 37]. The use of iron-rich lateritic soils in the production of building materials is associated with enhanced strength and resistance to meteorological agents [5, 38]. For certain types of laterites, the rapid and irreversible induration of the building material is attributed to the formation of a water insoluble matrix by metallic oxides [3]. Finally, iron-stabilization has been found to considerably increase the strength of rammed earth [39].
- (c) Calcite is also considered a cementing, stabilizing agent, especially when present in particles with a few  $\mu$ m diameter [20, 40]. In earth construction literature, emphasis is placed on calcium hydroxide Ca(OH)<sub>2</sub>, the most common stabilizer, and the soils' intrinsic CaCO<sub>3</sub> content is generally ignored. To our knowledge the only literature report is that of the earth blocks from Shibam, Yemen, whose high compressive strength of over 8 MPa was attributed to the high (upwards of 20%) calcite content of the soil [3]. However, further research is required to confirm this assumption.
- (d) Chemical and mineralogical properties. The chemical composition of the soil has been associated with important properties as CEC and texture [41, 42]. In earthen construction it can indicate the presence of contamination in the raw material that could be harmful for the health. Furthermore, the SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> has been found to



be indicative of the quartz and the clay minerals content, especially when soils with the same clay minerals are assessed [43].

The type of clay minerals and their content as can be expressed for example with the kaolinite/smectite ratio [43], also have a significant influence on the mechanical properties of earthen building materials. Three-layer clays have higher SSA, generating higher suctions resulting in materials with higher bonding capacity and strength [26]. For earth plasters in particular, illitic clays that have intermediate values of SSA and CEC and exhibit balanced properties of strength and shrinkage, have been found to be the most appropriate [14]. Nevertheless, drawing straightforward conclusions based on XRD results is rather challenging due to the difficulty in identifying and quantifying clay minerals and even more so, due to the mixed-layer clays and the poorly crystalline/amorphous clays, which recent studies have shown that they can constitute a significant part of the soil's clay fraction with a huge impact on its properties [44].

1.2 Volume change behavior of earthen building materials

Moisture is a perquisite for the activation of the binding strength of clays, but the evaporation of water during the drying process leads to a volumetric shrinkage that is sometimes of a different order of magnitude compared to other commonly used building materials. Volume change of clayish materials is the result of complex interactions between the solid skeleton and the pore fluid [45]. The loss of water during drying leads in an increase in suction and capillary forces and the tighten of clay platelets [33]. The final amount of swelling and shrinkage largely depends on the quantity and type of clay minerals present as well as the grain size distribution of the silt and the aggregates [6]. In general, the greater the plasticity of the clay minerals, the greater the shrinkage upon drying [19].

Kaolinite subgroup minerals present the lower volumetric shrinkage among clay minerals. The bonding between successive layers (van der Waals forces and hydrogen bonds) is of sufficient strength and there is no interlayer swelling. Smectite group minerals undergo a large volume contraction and



are considered expanding clays. They have a weak interlayer bond by loosely held cations. Clays of the illite group have intermediate values of volume change due to the firmly fixed potassium ions between layers [19, 20]. Vermiculite is considered to be a clay mineral with a medium shrink-swell capacity. In its interlayer space there are double molecular layers of water as well as Al-hydroxy ions and cations which act as bridges holding the units together and thus, limiting the expansion. Finally, chlorites are considered to be non-expansive clays. The structure of chlorite is similar to vermiculite, but instead of the double layer of water in the interlayer space, there is a magnesium-dominated trioctahedral hydroxide sheet which forms strong hydrogen bonds with the adjacent sheets, prohibiting expansion [19, 20].

Other parameters that affect the volume change behavior of earthen materials according to Delinière et al. [46] and Moevus et al. [33] are:

- (a) The water content during manufacture. When the soil is saturated, a higher water content usually results in a higher linear shrinkage.
- (b) The SSA and the CEC. These two properties are interrelated. Generally, the greater the SSA and especially the external surface, the greater the amount of water retained [20]. Also, a higher CEC usually results in a higher volume change [12]. The negative charges of the soil colloids attract the positively charged end of the polar water molecule. The cationic content of the water is also important.
- (c) The drying procedure.

The volume change of earthen materials is usually assessed through the linear shrinkage index. A humid soil is placed in a box of standardized dimensions and when dry the change in length is measured and divided by the initial length. Unfortunately, the box dimensions, the water content, the compaction applied and the proposed thresholds vary significantly among standards, handbooks and normative documents making it difficult to compare the obtained results [8]. For earth mortars and plasters, DIN 18946-47 [47, 48] define the use of the dried 40 mm  $\times$  40 mm  $\times$  160 mm prisms to assess the linear shrinkage, but Delinière et al. [46] argued that for plasters, the real shrinkage in the field is different than the one measured due to the support preventing contraction.

# 2 Materials and methods

# 2.1 Soil selection

A total of 11 soils from 5 different regions of Greece (Fig. 1) and corresponding mortars were selected for examination in this study. These 11 soils were those 4 newly collected (S1–S4) presenting extremely high strength, plus 7 more from a previous investigation [12], so as to cover a wide range of soil variabilities and answer some secondary research questions. More specifically:

 For D1 and D4 further investigation was necessary as they also presented high values of strength, 6 and 7 MPa respectively.

- L1 and L2 were sourced from nearby fields and their relatively small differences in texture and CEC could not account for their large difference in strength.
- Soils P3, B2 and B4 were chosen as reference materials, since they presented average values of strength, clay fraction content and CEC. Additionally, they came from different regions than the rest, providing a better geographic distribution of the samples.

### 2.2 Soil assessment methods

Soil properties were assessed after sieving all soil samples with a 2 mm sieve. SSA, amorphous iron oxides, free iron oxides, active carbonates and chemical composition were determined for all 11 samples, while CEC, clay content and mineralogical properties were determined for soils S1–S4 using the



**Fig. 1** Soil map of Greece depicting the sampling locations

same methods and laboratories as for the other seven soils in [12].

The SSA of these 11 soils was measured after vacuum degassing using the multipoint BET-N2 adsorption method with a Quantachrome NOVA 1200 instrument. The values of specific surface are highly dependent on the method used, with some methods measuring the external surface and others measuring the total one (external and internal) [49]. The nitrogen adsorption method, which measures the external surface, was selected for this study, because the measurement of the total surface yields very high values, especially when smectite clays are present [50], but also because the contribution of the internal surface to interparticle bonding and compressive strength is very unlikely.

In order to determine the content of amorphous iron oxides and of free iron oxides, selective dissolution analyses were carried out. More specifically, soil samples were extracted for free iron (Fe<sub>d</sub>) oxides using the sodium–dithionite–citrate (DCB) method [51]. In addition, acid ammonium oxalate (pH=3.0–3.5) extractable Fe (Fe<sub>o</sub>) was measured using the method of Blakemore et al. [52]. Ammonium oxalate is the extractant for amorphous or poorly ordered colloid components and extracts iron (Fe<sub>o</sub>) from ferrihydrite and Fe–humus complexes. Fe<sub>d</sub> and Fe<sub>o</sub> were determined by atomic absorbance spectrometry.

The total CaO and  $Fe_2O_3$  content of the cohesive part of the soils and their chemical composition were determined with X-ray fluorescence spectrometry (XRF). The soils were sieved with a 63 µm sieve and pressed into pellets. The bulk chemical compositions were obtained by energy dispersive X-ray fluorescence (ED-XRF), using a Spectro X-Lab Pro 2000.

Active carbonates, i.e. the finely divided and chemically active part of carbonates, were determined according to the standard NF X31-106 [53], by adding ammonium oxalate to the soil and titrating the part that has not reacted with KMnO<sub>4</sub>.

For the 4 newly collected soils, CEC, texture, organic matter and mineralogy were assessed using the same methods as for the other 7. Specifically, CEC was determined with the sodium acetate method performed in three steps repeated three times each. The first step was to mix 5 g of soil with sodium acetate solution in order to replace the exchangeable



cations with sodium cations, the second to use absolute ethanol to remove the excessive sodium cations and the third to add ammonium acetate to replace sodium cations with ammonium cations. Finally, a flame photometer was used to measure the displaced sodium. Soil texture was measured with the Bouyoucos hydrometer method for which soil was dispersed with sodium hexametaphosphate  $((NaPO_3)_6)$  [54]. Organic matter content was determined with the Walkley-Black method in which organic carbon of the soil is oxidized by potassium dichromate in concentrate sulfuric acid [55]. Mineralogical properties were assessed by X-Ray Diffraction (XRD) analysis on pulverized samples with a Bruker D8 advanced diffractometer with a Cu K $\alpha$  source. All samples were scanned between 2 and 70°  $2\theta$  but selected samples were also scanned after heat treatment at 550 °C and ethylene-glycol treatment for the better identification of clay minerals. For the evaluation EVA and Xpowder software were used.

#### 2.3 Preparation and assessment of earth mortars

Mortars were prepared according to DIN 18946/47 as follows: At first, clumps were crushed with a plastic hammer, the soil was sieved with a 2-mm sieve and was dried at a drying chamber. Then earth mortars were produced with the use of a laboratory mortar mixer by slowly adding water to the dry, sieved soil at low speed, mixing at high speed, stop the procedure for a few minutes and finally mixing again at high speed. The water to soil ratio was kept constant for all samples at 35% by weight and the mixing time was set at 10 min. At this point, consistence of fresh mortars was assessed according to EN 1015-3 [56] with a flow table. Afterwards, the mortars were poured in  $40 \times 40 \times 160$  mm molds in two layers with the use of a spatula and 25 strokes of a 50 g wooden tamper with metallic edge, covered with a damp cloth to avoid fast drying and then placed in standard atmospheric conditions  $(23 \pm 5 \text{ °C}, 50 \pm 15\%)$ RH) per DIN 18946-47 [47, 48]. Three mortar prisms were prepared for each soil and so, six compressive strength tests and three flexural strength tests were carried out. Constant weight (no more than 0.2% mass deviation of two consecutive measurements taken at 24 h intervals) was achieved in less than 2 weeks for all samples, but in order to comply with

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both DIN 18946-47 [47, 78] and EN 1015-11 [57] standards, dried mortar properties were assessed after a period of 28 days.

Compressive and flexural strength were measured with a TONIPRAX 1540 testing machine. The loading rate was defined so as failure occurs at approximately 60 s. Using the same prisms, just before the flexural strength tests, linear shrinkage and dry bulk density were also measured according to DIN 18946-47 [47, 48]: the former as the change in length of the prisms compared to the initial length measured with a digital caliper, the latter as the mass in relation to the volume of the prisms.

In order to determine the most dominant feature related to compressive strength, dominance analysis [58, 59] that is based on multivariate regression and the examination of  $R^2$ , was performed. Multivariate regression analysis including all four features (SSA, CEC, Clay and Fe<sub>o</sub>) was deemed inappropriate since multicolinearity can cause several issues in interpreting the results, mainly the ones related to the regression coefficients estimates. Among several statistical methods, dominance analysis yields weights that can be used to measure the relative importance of predictors in multivariate regression analysis and can be used in the presence of multicolinear predictors [60]. It is relatively easy to perform and the results are

Table 1 Properties of soil samples (percentages per mass)

straightforward to interpret, allowing the comparison of importance between predictors. Hence, dominance analysis is both robust and intuitive. For the purpose of this analysis, a Python library implementing dominance analysis was used [61].

# **3** Results and discussion

# 3.1 Physical, chemical and mineralogical properties of examined soils

The physical, chemical and mineralogical properties of the examined soils are summarized in Table 1. The examined soils displayed a wide range of natural variability, with clay fraction content ranging from 12.5 to 55.2%, SSA from 6.44 to 72.98 m<sup>2</sup>/g and CEC from 11.8 cmol(+)/kg to 37.1 cmol(+)/kg. Active CaCO<sub>3</sub>, Fe<sub>d</sub> and Fe<sub>o</sub> also covered a wide range, from 0 to 7.0%, from 0.29 to 3.68% and from 0.07 to 0.60% respectively.

The clay fraction content of soils S1-S4, which were the trigger for this study, was between 38.1 and 55.2%, exceeding the upper limit reported in literature for some earthen construction techniques. However, apart from the fact that some studies on earth construction used soils with clay fraction content higher than 40% [62, 63], the objective of this research was to investigate the properties that have

| Soil sample | Clay (%) | Sand (%) | CEC<br>(cmol(+)/<br>kg) | SSA (m <sup>2</sup> /g) | CaCO <sub>3</sub><br>active<br>(%) | Organic<br>Matter<br>(%) | Fe <sub>d</sub> (%) | Fe <sub>o</sub> (%) | Mineralogy <sup>a</sup>               |
|-------------|----------|----------|-------------------------|-------------------------|------------------------------------|--------------------------|---------------------|---------------------|---------------------------------------|
| D1          | 21.4     | 50.4     | 15.7                    | 18.18                   | 0.0                                | 1.87                     | 1.81                | 0.07                | Qz, Ilt/Ms, An, Kln, Clc              |
| D4          | 26.0     | 25.7     | 21.2                    | 24.16                   | 2.9                                | 2.20                     | 1.01                | 0.32                | Qz, Ms/Ilt, Cal, Lab, Kln Clc,<br>Act |
| S1          | 45.9     | 25.8     | 31.9                    | 43.50                   | 7.0                                | 2.41                     | 0.73                | 0.18                | Qz, Cal, Sme,                         |
| S2          | 38.1     | 21.2     | 30.5                    | 32.99                   | 0.8                                | 2.27                     | 1.32                | 0.44                | Qz,, Sme, Ms/Ilt, Ano                 |
| <b>S</b> 3  | 55.2     | 20.6     | 37.1                    | 65.66                   | 1.3                                | 1.90                     | 1.75                | 0.45                | Qz, Sme, An                           |
| S4          | 49.9     | 25.9     | 35.8                    | 72.98                   | 1.2                                | 1.94                     | 3.68                | 0.60                | Qz, Sme, Mcc, An                      |
| L1          | 25.7     | 36.4     | 16.0                    | 15.97                   | 1.2                                | 1.54                     | 1.81                | 0.10                | Qz, Cal, Ilt/Ms, Clc, Kln, Ano        |
| L2          | 26.2     | 40.8     | 20.8                    | 18.94                   | 4.1                                | 1.80                     | 1.97                | 0.16                | Qz, Cal, Ilt/Ms, An                   |
| P3          | 12.5     | 66.5     | 11.8                    | 6.80                    | 3.9                                | 0.61                     | 0.29                | 0.06                | Cal, Qz, Ilt/Ms, Sme, Mcc             |
| B2          | 18.6     | 59.1     | 14.4                    | 6.44                    | 2.4                                | 1.84                     | 1.61                | 0.04                | Qz, Cal, Ms/Ilt, Clc, Kln, An         |
| B4          | 18.3     | 56.8     | 15.9                    | 6.39                    | 0.0                                | 1.85                     | 2.10                | 0.11                | Qz, Ms/Ilt, Sme, Kln, An              |

<sup>a</sup>Mineralogy: *Qz* quartz, *Ilt* Illite, *Ms* Muscovite, *Cal* Calcite, *Kln* Kaolinite, *Sme* Smectite, *Clc* Clinochlore, *An* Anorthite, *Ano* Anorthoclase, *Mcc* Microcline, *Lab* Labradorite, *Act* Actinolite

an impact on the mechanical properties of earthen building materials. In this context, it was considered essential to evaluate a broad range of characteristics at the extreme material boundaries in order to identify both possible influences on mechanical properties and a possible upper limit of linear shrinkage, SSA, CEC or clay fraction content beyond which strength is reduced.

The diffractograms of XRD analysis are presented in Fig. 2 and the minerals in Table 1. In all the examined soils there is a strong presence of quartz (SiO<sub>2</sub>) which is the major phase except for P3. In the latter, calcite (CaCO<sub>3</sub>) was the major phase as the highest intensity peak was at 3.04 Å. Calcite was also identified in soil S1 in significant quantity as well as in soils B2, D4 L1, L2 as a minor phase, something that agrees well with the CaO content measured through XRF analysis. In addition, all the soils besides S1 also contained small amounts of either alkali or plagioclase feldspars.

Illite/Muscovite were detected in all soils except S1, S3 and S4. It is very difficult to distinguish these minerals with XRD analysis and so, I/Ms or Ms/I in Table 1 were based on the relative intensity of the main peak of illite at 4.43 Å and the peak of muscovite at 9.95 Å. Kaolinite was also identified in soils B2, B4, D1, D4 and L1 by the characteristic peaks at ~7.2 and 1.5 Å. Its presence was also verified with heat treatment of the soils at 550 °C, a

temperature at which the peak at 7.2 Å disappears due to dehydroxylation.

Soils S1, S3 and S4 didn't present the reflections corresponding to either illite or kaolinite clay minerals, despite their very high clay fraction content. Interestingly enough, they exhibited a broadening of XRD reflections between 17 and 15 Å, characteristic of poorly crystalline clays, most likely smectite with very small particle size. The higher intensity of the broadened peak was observed for S4 soil, followed by S3 and S1. This finding corresponds to the high SSA and CEC observed in these three soils.

The concentration of the major oxides, determined by means of XRF analysis, is presented in Table 2. The widespread values of total SiO<sub>2</sub> (28.89–54.14%), Al<sub>2</sub>O<sub>3</sub> (8.85–19.89%) and CaO (0.59–26.04%) confirmed that the selected soils covered a broad spectrum of soils. In the same table, some trace elements were included based on their correlation with important soil properties [41, 42]. Comparing the values of Fe<sub>d</sub> and Fe<sub>o</sub> from Table 1 with total Fe<sub>2</sub>O<sub>3</sub> from Table 2, it can be assumed that a considerable portion of the total Fe is bound in other minerals, such as silicates.

#### 3.2 Properties of earth mortars

The mechanical properties of the earth mortars are presented in Table 3 and Fig. 3. Mortars from soils



Fig. 2 X-ray diffraction patterns of the soils. Cl: clinochlore, S: smectite, M: muscovite, I: Illite, K: Kaolinite, Q: quartz, C: calcite, A: anorthite, Mi: Microcline, An: anorthoclase, Ac: actinolite, S(pc): poorly crystalline smectite



| Table 2   Chei | mical composi        | ition of examine                   | ed soils (perce | intages per mas               | s)      |                       |                      |                                     |           |           |                         |
|----------------|----------------------|------------------------------------|-----------------|-------------------------------|---------|-----------------------|----------------------|-------------------------------------|-----------|-----------|-------------------------|
| Soil sample    | SiO <sub>2</sub> (%) | Al <sub>2</sub> O <sub>3</sub> (%) | CaO (%)         | ${\rm Fe}_{2}{\rm O}_{3}$ (%) | MgO (%) | Na <sub>2</sub> O (%) | K <sub>2</sub> O (%) | V <sub>2</sub> O <sub>5</sub> (ppm) | CuO (ppm) | ZnO (ppm) | Rb <sub>2</sub> O (ppm) |
| DI             | 52.89                | 19.15                              | 0.71            | 7.27                          | 1.62    | 1.35                  | 2.46                 | 378                                 | 156       | 187       | 118                     |
| D4             | 46.47                | 15.92                              | 4.94            | 6.15                          | 3.50    | 1.01                  | 3.08                 | 145                                 | 90        | 127       | 171                     |
| S1             | 28.89                | 8.85                               | 26.04           | 3.99                          | 1.58    | 0.68                  | 1.28                 | 264                                 | 70        | 85        | 84                      |
| S2             | 50.87                | 18.31                              | 1.81            | 6.91                          | 3.67    | 0.97                  | 3.53                 | 170                                 | 69        | 153       | 194                     |
| S3             | 53.41                | 15.73                              | 1.81            | 6.57                          | 1.58    | 0.56                  | 1.16                 | 206                                 | 58        | 96        | 124                     |
| S4             | 54.14                | 16.10                              | 2.00            | 6.43                          | 1.65    | 0.69                  | 1.80                 | 332                                 | 73        | 113       | 141                     |
| L1             | 48.17                | 14.00                              | 6.90            | 6.11                          | 2.60    | 0.97                  | 2.24                 | 298                                 | 146       | 305       | 90                      |
| L2             | 45.10                | 13.04                              | 11.26           | 5.81                          | 2.10    | 0.82                  | 1.90                 | 335                                 | 124       | 275       | 82                      |
| P3             | 32.95                | 10.48                              | 19.26           | 4.10                          | 2.50    | 1.32                  | 2.60                 | 100                                 | 89        | 95        | 125                     |
| <b>B</b> 2     | 40.32                | 19.89                              | 9.54            | 6.87                          | 1.98    | 1.42                  | 3.19                 | 346                                 | 196       | 156       | 126                     |
| B4             | 49.91                | 18.31                              | 0.59            | 8.00                          | 2.90    | 0.88                  | 3.29                 | 186                                 | 174       | 169       | 143                     |
|                |                      |                                    |                 |                               |         |                       |                      |                                     |           |           |                         |

S1–S4 exhibited tremendously high compressive strength, in the range of approx. 10–16 N/mm<sup>2</sup>. Such high values have rarely been reported in earthen materials literature, e.g. for extruded bricks [64] and earth blocks [15]. These high strength values were accompanied by proportionally significant linear shrinkage, between 10.4 and 15.6%, demonstrating that there is no upper limit of volume change, as expressed by the linear shrinkage index, beyond which compressive strength is reduced. On the contrary, strength and linear shrinkage are correlated linearly even at such high values (Fig. 4a). It should be noted, though, that the obtained strength values may have been affected by specimen size, as well as by the aspect ratio of 1 used for earth mortar testing.

Even though compressive strength was unaffected by volume change and the subsequent development of hairline cracks, the same was not true for flexural strength. As illustrated in Fig. 4b, compressive and flexural strength were linearly correlated up to a certain point (approximately 10 N/mm<sup>2</sup>), as reported before [12, 13], with the ratio between them typical for earth blocks and earth mortars in the range of 0.26–0.37. Beyond this point, both flexural strength and flexural-to-compressive-strength ratio decreased dramatically due to the detrimental effect of cracking and volumetric shrinkage. Therefore, it can be suggested that the flexural strength of earth mortars is more sensitive to macro- and microscopic cracks than the compressive strength, as is the case with most building materials.

In Table 3 it can be seen that for the same water to soil ratio the consistency/workability of the fresh mortars presented significant differentiations. According to EN 1015-6 [65] mortars S1–S4, D4 and L2 can be classified as stiff, mortars D1, L1, B2, B4 as plastic and mortar P3 as soft. Thus, the stiff mortars are those with the high strength, while mortars S1–S4 with the highest strength also had the lowest flow values. But in needs to be mentioned that consistency was measured according to a standard designed for lime/cement mortars and not with quasistatic methods that have been found to be reliable for earthen materials [66, 67].

Based on the work of Ardant et al. [68] and Audren et al. [69], a noteworthy relationship was found in this study, which is presented in Fig. 5. These researchers, recognizing that the mechanical

| Mortar     | Dry<br>density<br>(kg/dm <sup>3</sup> ) | Mean<br>compressive<br>sStrength (N/<br>mm <sup>2</sup> ) | Compressive<br>strength SD<br>(N/mm <sup>2</sup> ) | Mean flexural<br>strength (N/<br>mm <sup>2</sup> ) | Flexural<br>strength SD<br>(N/mm <sup>2</sup> ) | Mean linear<br>shrinkage<br>(%) | Linear<br>shrinkage<br>SD (%) | Consistence<br>(flow values in<br>cm) |
|------------|---|---|--|--|---|---------------------------------|-------------------------------|---------------------------------------|
| D1         | 1.82                                    | 6.02  | 0.27   | 1.86   | 0.14  | 7.48                            | 0.30                          | 14.5                                  |
| D4         | 1.81                                    | 7.27  | 0.46   | 2.14   | 0.39  | 8.75                            | 0.77                          | 12.0                                  |
| <b>S</b> 1 | 1.90                                    | 11.47   | 0.52   | 1.53   | 0.21  | 12.83                           | 0.51                          | 11.3                                  |
| S2         | 1.85                                    | 9.83  | 0.49   | 2.70   | 0.37  | 10.44                           | 0.56                          | 11.0                                  |
| <b>S</b> 3 | 1.97                                    | 12.85   | 0.28   | 0.73   | 0.18  | 14.39                           | 0.53                          | 10.8                                  |
| S4         | 1.99                                    | 15.82   | 0.74   | 0.91   | 0.11  | 15.56                           | 0.71                          | 10.5                                  |
| L1         | 1.76                                    | 3.27  | 0.10   | 0.97   | 0.08  | 6.04                            | 0.47                          | 14.3                                  |
| L2         | 1.92                                    | 6.94  | 0.35   | 1.79   | 0.13  | 9.67                            | 0.50                          | 13.4                                  |
| P3         | 1.80                                    | 3.28  | 0.15   | 1.08   | 0.04  | 6.17                            | 0.22                          | 20.2                                  |
| B2         | 1.69                                    | 2.36  | 0.17   | 0.86   | 0.07  | 4.42                            | 0.37                          | 16.4                                  |
| B4         | 1.67                                    | 2.50  | 0.16   | 0.79   | 0.06  | 4.35                            | 0.58                          | 15.3                                  |

 Table 3
 Properties of the earth mortars



Fig. 3 a Compressive strength, b flexural strength and c linear shrinkage of earth mortars

properties of earthen materials are also dependent on clay activity, and to overcome the problem of density being correlated with strength only for a given soil, they divided the compressive strength by the Methylene Blue Value (MBV) and found that there is a link between this ratio and dry density. MBV has been used to determine CEC [70], which is an inherent soil property that is also representative of the clay activity. Similar to the Activity (A) introduced by Skempton [71] as the ratio of plasticity index (PI) to clay fraction content, Cerato and Lutenegger [72] introduced CECA defined as the ratio of CEC to clay content. As can be seen in Fig. 5, a relationship can be established between dry density and both compressive strength/CEC and compressive strength/CECA, indicating that these properties are interrelated and that CEC plays an important role the in the dried building material properties.

# 3.3 Correlation between soil properties and engineering properties of mortars

# 3.3.1 Influence of SSA

The relationship between the SSA of the soils and the compressive strength of the mortars is





Fig. 4 Relation between compressive strength and a linear shrinkage, b flexural strength



Fig. 5 Relation between dry density and the ratio of a compressive strength to CEC and b compressive strength to CECA

demonstrated in Fig. 6a and that between SSA and linear shrinkage in Fig. 6b. A strong linear relationship between SSA and these properties was found  $(R^2=0.94$  for compressive strength,  $R^2=0.92$  for linear shrinkage), in agreement with the earlier findings in soil science literature that SSA determines or is related with many physical, chemical and engineering properties of soils, especially the fine-grained ones [73, 74]. The high correlations obtained here, in combination with studies from this field relating SSA with CEC and the elastic modulus of unsaturated soils [75], as well as with the soil water retention curve and thus with soil suction [76], show that SSA plays an important role in strength gaining of earthen materials. Of great importance in this context is the correlation of SSA with water retention at very low matric potentials [77, 78], i.e. at the dry state of the soil, in which the compressive strength of earthen building materials develops.

SSA was also assessed in the study of Lagouin et al. [13], but there was no linear correlation





Fig. 6 Relation between SSA and a compressive strength, b linear shrinkage

with compressive strength, only a trend. This can be explained by the relatively narrow range of SSA values between ~2.5 and 14.5 m<sup>2</sup>/g and the measurement of 4 SSA values from 4 soils of different mineralogy and origin, (2 were natural soils and 2 sludges from the washing of aggregates) with which mortars were prepared. In particular, the mortars with the calcite-rich sludge deviated significantly from the rest in [13] and therefore further investigation is required to assess whether the relationship between SSA and strength can encompass all the different soil mineral suites. Walter et al. [79] also attempted to correlate SSA as well as the clay fraction content and the methylene blue values with strength and found a much higher  $R^2$  for SSA. However, the strength of mortars made with tropical soils was highly dependent on the metal oxide content as discussed in Sect. 3.3.3.



Fig. 7 Relation between CEC and a compressive strength, b linear shrinkage



Fig. 8 Relation between clay fraction content and a compressive strength, b linear shrinkage

#### 3.3.2 Influence of CEC and clay fraction content

CEC and clay fraction content were also correlated with the compressive strength and linear shrinkage of the produced mortars, as shown in Figs. 7 and 8. The correlation between clay fraction content and compressive strength, although reasonably good  $(R^2=0.87)$ , was lower than that between SSA and strength and CEC and strength. This was anticipated, albeit not indubitably, as CEC and SSA capture the combined effect of clay fraction content, clay size and clay type. The high coefficients of determination obtained in this study are augmented by the elevated values of strength, SSA, CEC and clay fraction content of S1-S4. However, even excluding these 4 soils from the regression analysis, the correlation of SSA  $(R^2=0.82)$  and CEC  $(R^2=0.63)$  with strength is much higher than that of clay fraction content ( $R^2 = 0.41$ ).

These correlations of CEC and clay fraction content with strength were also observed earlier [12, 13], but here it is proven that this correlation has no upper limit: the higher the clay fraction content, CEC and SSA, the higher the strength and the linear shrinkage. The obtained results regarding clay fraction content align with the results of previous studies. Hamard et al. [10] also found that clay fraction content correlates with shrinkage and flexural strength with high  $R^2$  but also with shear strength, while Emiroğlu et al. [11] showed that a higher clay content leads to a higher compressive strength.

Lagouin et al. [13] found a correlation between clay fraction content and compressive strength with soils with a much lower clay content than in this study, up to  $\sim 14\%$ . Considering these results and those in Table 1, where clay content ranges from 12.5 to 55.2%, it can be assumed that the correlation between clay fraction content and compressive strength is valid for a wide range of different textures.

CEC has been found to be correlated with strength and volume change of earthen materials prepared with soils of varying mineralogy and chemical composition [12, 13]. Recently it was used as one of the proxies to assess soil suitability for light earth building at large scale [80]. Nonetheless as shown in [79], these correlations and applications are not valid for materials made with highly weathered tropical soil, which can be positively charged due the high content of iron oxides and the dominance of kaolinite.

#### 3.3.3 Influence of iron oxides and calcium carbonate

From Fig. 9, a correlation between poorly ordered/ amorphous iron oxides with compressive strength and linear shrinkage can be deduced.

Amorphous/poorly ordered iron oxides, despite being of small presence, their very small particles (3-7 nm) and their very high specific surface  $(200-500 \text{ m}^2/\text{g})$  suggest that they might contribute to the total SSA of the examined soils to some extent [81]. Moreover, it has been proved that





Fig. 9 Relation between amorphous iron oxides and a compressive strength, b linear shrinkage

the lower the crystallinity of Fe oxides, the more effective their aggregating capacity: Schahabi and Schwertmann [82] found that a mere 1% addition of ferrihydrite in a loess soil resulted in 250% increase in the formation of water-stable aggregates, while the addition of the same quantity of hematite yielded no such increase. The aggregates in the abovementioned research were formed through the attraction between positively charged Fe-oxide particles and negatively charged matrix particles, a mechanism that could be associated with compressive strength of earthen building materials. This is in agreement with the fact that no correlation between Fe<sub>d</sub> and compressive strength or shrinkage was detected in this study, but the complexity of free iron oxides doesn't permit a clear assumption.

In temperate climatic zones, as is the one of the collected samples, the content of iron oxides is usually low. Zhang et al. [83] suggested that there are three occurrence types of free iron oxides, of which the "cladding form" (the formation of relatively large aggregates by structural units coated with free iron oxides) found in lateritic soils has the strongest effect in increasing strength and inhibiting the swelling–shrinking capacity. This is consistent with the research of Walter et al. [84] on iron-rich soils collected from lateritic quarries, who showed that for these highly weathered tropical soils, compressive strength is correlated with iron oxides and especially goethite. Therefore, according to the



findings of Walter et al. [79, 84] and the results of this study, iron oxides are a critical factor in the mechanical properties of earthen materials with iron-rich tropical soils, whereas for moderately weathered soils, SSA, CEC and clay fraction content are of profound importance and the role of iron oxides is rather unclear.

Concerning the role of calcium carbonate, no solid conclusion could be drawn as only S1, L1 and P2 had substantial enough amounts of calcite that could have an influence on mortar properties.

# 3.3.4 Influence of chemical and mineralogical properties

Finally, the determination of the chemical composition of the soils proved of limited value. The great diversity in the chemical composition of soils, demonstrated in Table 2, differentiates it as a building material from other commonly used materials such as cement and lime. Thus, no direct relationship was found between the content of either the major oxides, the SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> ratio or the trace elements of the soils and the engineered properties of the mortars. A useful example is the comparison between soils and mortars S1 and S3. These two soils were found to have similar, very high values of strength, 11.5 and 12.9 N/mm<sup>2</sup> respectively, corresponding with their values of CEC, SSA and clay fraction content. Yet despite these similarities, their chemical composition was completely different: SiO<sub>2</sub> was almost double in S1 than S3, Al<sub>2</sub>O<sub>3</sub> in S1 was approximately half that of S3 and CaO of S1 was equal to almost 14.5 times that of S3. Lastly, the compressive strength of reference mortar B2 was 25% of that of mortar S2, with which it had an almost identical chemical composition.

Regarding the XRD results, it was not possible to draw clear conclusions since only qualitative analysis was performed. In order to have reliable quantitative assessment of the clay minerals thermo-gravimetric analysis would also be required as proposed by Lagouin et al. [13], but again the mixed clay mineralogical properties of the examined soils would make the task rather uncertain. Nonetheless, from the obtained results it is shown that the mortars with the extraordinary strength (S1-S4) contained poorly crystalline smectite clays, a finding that accords well with their high SSA and CEC values. Due to the expanding nature of smectite these mortars are also those that presented the higher linear shrinkage. Illitic clays that have been found by Lima et al. [14] to be more adequate for plasters are very common in Greek soils and were found in most samples, while in some of them they were the predominant clays.

#### 3.3.5 Dominance analysis results

The results of dominance analysis over all predictors related to compressive strength indicate (Table 4) that SSA dominates in all level of dominance defined by Azen and Budescu [85] (general, conditional and complete). In addition, CEC appears to be the second most important predictor in the model, followed by clay fraction content. Table 4 describes in a qualitative manner the relation of importance among the four variables. Furthermore, to accompany the statistical analysis with a quantitative metric, two additional measures are presented in Table 5: k=3 conditional dominance that is the contribution to  $R^2$ of each predictor to the complete model (the model that includes all four variables), and the measure of

Table 5 Quantitative metrics of dominance for the four soil properties related to compressive strength

| Predictors      | K=3 Conditional dominance | Overall average |
|-----------------|---------------------------|-----------------|
| SSA             | 0.056                     | 0.282           |
| CEC             | 0.034                     | 0.258           |
| Clay            | 0.023                     | 0.237           |
| Fe <sub>o</sub> | 0.008                     | 0.198           |

general dominance, i.e., overall average as described in [85]. The results of Table 5 confirm what was observed through linear regression of each individual soil property and the corresponding  $R^2$ , showing that SSA and CEC are the most important parameters related to compressive strength.

# 4 Conclusions

The presented research data aimed to provide an insight into the correlations between the intrinsic properties of the soils and the physical and mechanical properties of the corresponding earth mortars. Based on 11 soils and mortars that were examined, the following conclusions were drawn:

- The compressive strength of unstabilized earth mortars was linearly correlated with the proportionally increasing linear shrinkage even at very high values, up to 15.8 N/mm<sup>2</sup>. Compressive and flexural strength were also linearly correlated but up to approximately 10 N/mm<sup>2</sup>. At higher values of compressive strength, flexural strength dropped dramatically due to excessive volume change and cracking.
- A correlation was established between dry density • and both the ratio of compressive strength to CEC and the ratio of compressive strength to CECA (the ratio of CEC to clay fraction content). This

| <b>Table 4</b> Comparison ofdominance level of the four | Predictors | Generally dominating                | Conditionally dominating            | Completely dominating               |
|---|------------|-------------------------------------|-------------------------------------|-------------------------------------|
| soil properties related to                              | SSA        | ['CEC', 'Clay', 'Fe <sub>o</sub> '] | ['CEC', 'Clay', 'Fe <sub>o</sub> '] | ['CEC', 'Clay', 'Fe <sub>o</sub> '] |
| compressive strength                                    | CEC        | ['Clay', 'Fe <sub>o</sub> ']        | ['Clay', 'Fe <sub>o</sub> ']        | ['Clay', 'Fe <sub>o</sub> ']        |
|   | Clay       | ['Fe <sub>o</sub> ']                | ['Fe <sub>o</sub> ']                | ['Fe <sub>o</sub> ']                |
|   | Feo        | -                                   | -                                   | -                                   |



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confirms previous researches that clay activity is a critical factor in the mechanical behavior of earthen building materials and can be expressed by CEC, which is an important intrinsic soil property.

- Statistical analysis (linear regression and dominance analysis) revealed that the most dominant soil property with the highest correlation to compressive strength was SSA, followed closely by CEC, showing that strength is largely dependent on these soil properties. The robust correlation of CEC and SSA with strength and linear shrinkage could provide a better understanding of the mechanisms governing strength gaining. Clay fraction content did not correlate with strength as strongly. No upper limit of these three properties was found beyond which strength is reduced.
- The vast differences in the chemical composition (major oxides, trace elements) of the soils did not allow for a direct correlation between mechanical properties and chemical composition. On the contrary, soils of entirely different chemical composition had similar values of CEC, SSA and strength, while soils with completely different properties had almost identical chemical composition.
- The mortars with the highest compressive strength exceeding 10 N/mm<sup>2</sup>, were prepared with soils with the highest SSA, CEC and clay fraction content. Furthermore, these soils didn't contain illite or kaolinite clays but only poorly crystalline smectite clays.
- No relationship could be established between total iron oxides or free iron oxides and compressive strength. A correlation was found between amorphous iron oxides and strength and linear shrinkage, a finding in agreement with the soil science literature; however, the fairly low  $R^2$  values for the present data set of wide-ranging strength values, coupled with the measured low contents of Fe<sub>o</sub>, render the contribution of amorphous iron to compressive strength still unclear.

Overall, the findings of this study provide further evidence for the claim of previous researches that the clay fraction content and CEC of the soils are correlated with the compressive strength and linear shrinkage of earth mortars. SSA was also proved to correlate with these mortar properties. Given the limited existing research data, further research with a variety of soils is required to assess the exact contribution of each soil property to the mechanical and physical properties of not only earth mortars, but also of the various earthen materials.

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