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Influence of chemical, mineralogical and geotechnical characteristics of soil on earthen plaster properties

Méryl LAGOUIN, Jean-Emmanuel AUBERT, Aurélie LABOREL-PRÉNERON,
Camille MAGNIONT

Highlights

- LL, CEC and SSA seem relevant for the characterisation of clayish materials
- water requirement of plasters is found to be correlated to the LL of soils
- correlation between % passing at 2 μm and linear shrinkage is strong
- plaster mechanical resistances are linked to CEC of soils and to % passing at 2 μm

Abstract

Earth is perfectly suited for indoor plastering purposes thanks to its numerous technical, ecological and economic assets. The quantity of clay is frequently evoked to explain fresh and mechanical performances of earthen coatings but this indicator fails to give full account of the diversity of earth behaviour encountered in earth construction. This paper aims to examine plaster properties from the point of view of their geotechnical and mineralogical composition. Four types of soil were employed to design earthen plasters with variable earth to sand ratios. The behaviour of coatings was assessed through their water requirement, linear shrinkage, and flexural and compressive strengths. A noteworthy relationship was detected between the water requirement of earth plasters and the liquid limit of the raw earth employed. The mechanical resistances of plasters were found to be linked to the cation exchange capacity of the soils. The correlations between linear shrinkage, or compressive strength, and percentage passing at 2 μm were strong, justifying the widespread use of texture and clay content for soil classification, and assessment of the properties of earth-based building materials.

Keywords

Earth plaster; mineralogical composition; liquid limit; cation exchange capacity; specific surface area; mechanical behaviour.

1 INTRODUCTION

To address environmental issues, the building industry must innovate to reduce its energy consumption and CO₂ emission by developing materials that are environmentally friendly while ensuring the comfort of users. Along with new ecological materials such as lightweight concretes, traditional construction materials, including earth, have been increasingly investigated over the last twenty years.

After years of being marginalized from conventional construction in highly industrialized societies by mass-produced building materials, earth is still the most widely used construction material in many countries. Nowadays, around one third of human beings live in earthen houses and, in developing countries, this figure is more than one half [1].

Earth exhibits numerous technical, ecological and economic advantages that explain its recent upsurge. Although not renewable on a human time scale, it is a local resource, available in abundance, affordable, reusable and indefinitely recyclable (when used without chemical stabilization). Earthen building materials also have low embodied energy: little energy is required to transport and process the raw resources into a building material (no calcination and minimal transportation distances). Moreover, earth-based building materials are able to buffer

indoor moisture variations and to improve the comfort of the building's users [2–6]. Clayish earth is therefore very suitable for indoor plastering purposes.

If a building is to have little impact on the environment, the resources employed must be found locally. Therefore, soils for earth construction are excavated directly on site or in the immediate vicinity. This requirement is, however, challenged by the local soil variability. Unlike most conventional construction materials, soils exhibit highly variable properties because of the diversity of their compositions.

Soil is a multiphasic medium composed of solid particles, liquid and gas in variable proportions. The solid particles vary in size and shape, from millimetric elements (gravel) to particles that are visible only with the aid of an electron microscope (clay tablets), and from nearly spherical grains to flat plates or long, slender needles [7]. The specificities of a soil depend on the different amounts and types of clay, silt, sand and gravel composing it. The coarser elements (gravel, sand, non-plastic silts) constitute the skeleton of the material, while the ultrafine grains contain the cohesive parts: the clay minerals. The cohesive clay minerals perform the task of holding the grains of the soil's skeleton together. However, these very fine particles ($< 2 \mu\text{m}$) are not all clay minerals.

Soil is also multimineral: it may contain virtually any of the elements present in the Earth's crust, organized into various crystalline forms to yield common minerals: quartz (silicates), feldspar, mica, calcium carbonates, gypsum, clay minerals, etc. Among them are phyllosilicates, which form clay minerals. These elements have a layered structure, with each layer composed of two, more or less continuous, tetrahedral and octahedral sheets. The differences in structure among clay minerals fundamentally affects the swelling/shrinking potential of the soil. There are three important groups of clay minerals: the kaolinite group (generally has no swelling potential), the mica-like group (illites, low swelling potential) and the smectite group (montmorillonites, very high swelling potential) [8]. The stability of the mineral structures greatly influences the soils' and clays' characteristic properties, such as plasticity, shrinkage and swelling [9]. In this context, clear differences can be observed between two clay-based building materials of different compositions.

Many methods have been used in earth construction research to assess the quantity, type and activity of clay. The particle size distribution (through sieving and hydrometer testing or laser diffraction analyses), Atterberg limits, and methylene blue values are among the most commonly used indicators to assess a soil's suitability [10].

Considering an application as interior coatings, percent passing the $2 \mu\text{m}$ sieve is frequently used to explain the fresh and mechanical behaviour of plasters by itself [11–17]. When the amount of clay involved in a plaster increases, regardless of its nature, the kneading water requirement increases to activate clay binding properties and to achieve the targeted consistence. The mechanical performances are also impacted: the higher the clay content, the higher the resistance.

However, the clay content of materials has failed to fully account for the diversity of earth behaviour encountered in earth construction [18,19], since this indicator only focuses on particle sizes, which does not allow the clay mineral content and type to be fully taken into account. Therefore, the relevance of this indicator must be challenged and another approach must be considered to assess the behaviours of mortars. Beside texture (i.e. balance between clay, silt, sand and gravel contents), mineralogical identification and quantification, the methylene blue value [10,14,20–22], cation exchange capacity [18,19,22], and specific surface area can be regarded as good indicators to characterize the colloidal properties of earth materials.

The present study aims to examine the correlation between the physical properties (texture, plasticity, cation exchange capacity, specific surface area, chemical and mineralogical compositions) of four soils and the properties of their earth mortars. The first part of the paper

focuses on the chemical, mineralogical and geotechnical characterisation of the four soils studied. The better understanding gained of the properties of the raw materials then enables further examination of earth plasters in terms of water requirement, linear shrinkage, flexural and compressive strengths.

2 MATERIALS AND METHODS

2.1 Raw materials

2.1.1 Earth and sand

Four soils from distinct sources were selected for sampling for this study.

Two of them, referred to as “soil B” and “soil N”, came from brickworks in the southwest of France. Soil B had a rust colour linked to the presence of iron oxides material. Soil N had an ochre colour typical of clayey soil from the Garonne river valley.

The other two soils were composed of quarry fines from aggregate washing processes. These fines, smaller than 0.1 mm, were by-products generated by the washing of coarser aggregates. The sludge created was left in sedimentation basins. One of the fines, brownish-grey and referred to as “soil C”, was collected directly from the sedimentation basins of a local quarry, in the southwest of France. The second type, coming from a calcareous aggregates quarry located in the north of the country, was taken in sedimentation basins and was then reduced to powder to be used in different applications. This ochre soil is referred to as “soil F”.

A siliceous river sand (0-2 mm) locally produced was employed for plastering formulations. The granulometric curve is presented along with the soils grain size analysis, in Section 3.2.1. According to the product description provided by the manufacturer, the sand employed has the following characteristics:

- percent passing the 63 μm sieve of $1.8 \pm 0.41\%$;
- methylene blue absorption value: $0,38 \pm 0,10 \text{ g}/100 \text{ g}$;
- solid dry density: 2,66 t/m³;
- water absorption : 1,86%.

2.1.2 Mortar preparation

The earth plasters studied were composed of a clayish earth (soil or quarry washing fines), sand and tap water. Various proportions of sand and water were mixed with each type of earth to design six mortars, with different mass ratios of clayish earth to sand: 1:1 (earth content of 50%), 1:1.5 (40%), 1:2 (about 33%), 1:2.5 (about 29%), 1:3 (25%), and 1:4 (20%). In view of its behaviour, an extra formulation with 15% earth content was mixed for soil C (Table 1).

Table 1. Mix designs of the formulations

Earth to sand ratio (%)	Water content (%)			
	Soil B	Soil C	Soil F	Soil N
15	-	19.40	-	-
20	22.00	21.50	19.24	21.98
25	23.19	25.16	19.09	23.39
29	23.31	24.10	19.70	22.99
33	22.86	25.02	19.90	24.12
40	27.93	27.93	23.30	26.06
50	44.82	25.84	24.55	28.80

In all cases, mortars were prepared with an automatic programmable mortar mixer (Controls) by using the following mixing procedure:

- mix the solid phase at low speed;
- add water to the solid phase and adjust the amount to ensure a flow table value of 175 ± 5 mm, while mixing at low speed;
- mix the mortar at higher speed;
- stop the mixer and leave the mixture to rest for a few minutes;
- complete mixing at high speed.

To obtain similar consistency among the materials, ensuring good workability, the water content was adjusted. The German standard [23] proposes fixing the consistency at 175 ± 5 mm. The water content of fresh plaster was measured by drying at 60°C .

2.2 Procedures

2.2.1 Earth characterisation

2.2.1.1 Chemical and mineralogical characterisation

Major oxide composition was evaluated on the basis of macroelemental analysis performed by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) on crushed samples smaller than $80 \mu\text{m}$.

The crystalline compounds of the clay fraction of the soils were identified using an X-Ray Diffractometer (XRD) and Thermo-Gravimetric Analysis (TGA) techniques.

A Bruker D8 Advance X-ray diffractometer, equipped with a SOLLER monochromator having a copper anticathode ($\lambda = 1.54 \text{ \AA}$), was used on powdered samples of crushed raw soils ($< 80 \mu\text{m}$). This analysis was completed, when necessary, by a specific characterisation of the clay minerals contained in the raw soils, which was performed on oriented aggregates using three preparations: air dried or natural, after glycolation, and after heat treatment at 500°C . When the oriented aggregate method was used, platy clay particles were precipitated onto a glass slide by drying a deflocculated suspension. With most particles oriented parallel to the slide, the first-order reflections were intensified, whereas reflections from higher atomic planes were minimized. Specific treatments were applied to soils to distinguish certain swelling-type clay minerals:

- smectites expand after treatment with ethylene glycol: a drop of this solvent was placed on one of the oriented slides and left to evaporate under an extractor hood for 4 hours before analysis by X-ray diffraction spectrometry;

- clay mineral samples were analysed after enough heating to collapse any expandable layers: another the oriented slide was calcined at 500°C for 1 hour, then analysed.

TGA was carried out on crushed samples of raw soils heated to 1000°C at a constant rate of $10^\circ\text{C}/\text{min}$ using a Netzsch SATA 449 F3 Jupiter apparatus.

2.2.1.2 Geotechnical characterisation

2.2.1.2.1 Particle size distribution

The size distribution of the soils was determined using two complementary techniques. The coarser fraction ($> 80 \mu\text{m}$) was analysed by wet sieving according to standard XP P94-041 [24]. The finer fraction was characterized by means of a densimeter measurement of the sedimentation time of solid particles in suspension in water, as described in NF P 94-057 [25].

2.2.1.2.2 Atterberg limits and methylene blue absorption test

The Atterberg limits were determined according to standard NF EN ISO 17892-12 [26]. The liquid limit (LL) was obtained using the Casagrande method. The numerical difference between the liquid limit and the plastic limit (PL) of a soil enabled the plasticity index (PI) to be calculated.

The methylene blue adsorption test was conducted in accordance with NF P 94 068 [27] to quantify the water absorption capacity of soils on the 0/5 mm fraction.

2.2.1.2.3 Cation exchange capacity

The Cation Exchange Capacity (CEC) test determined the exchangeable sodium, potassium, calcium and magnesium contents in soil at its pH. It was conducted using barium chloride solution (NF EN ISO 11260). First, 2.5 g of each soil, without previous treatment, were saturated with respect to barium by treating the soil with a 0.1 mol/L barium chloride solution. After this, the concentrations of sodium, potassium, calcium and magnesium were determined by ICP-AES. Then, the soil was equilibrated with a 0.01 mol/L barium chloride solution and a known excess of 0.02 mol/L magnesium sulfate was added. All the barium present, whether in solution or adsorbed, was precipitated in the form of highly insoluble barium sulfate and, consequently, the sites with exchangeable ions were readily occupied by magnesium. Finally, the excess magnesium was once again determined by ICP-AES.

2.2.1.2.4 Specific surface area

The specific surface areas (SSA) were calculated according to the Brunauer-Emmett-Teller (BET) theory. The samples were analysed under nitrogen atmosphere (adsorption-desorption isotherms at 77.350 K) in a volumetric working device. The moisture content in the samples was removed by drying them at 90°C for 15 h prior to the analysis. The Flowsorb 2300 equipment (Micromeritics) provided single-point BET surface area measurements.

2.2.2 Utilisation properties of plasters

As no European standard exists for the characterisation of clay plasters, the procedures used were adapted from the German standard for the characterisation of ready-mixed clay plasters [23].

2.2.2.1 Characteristics of fresh state plasters

Based on standard EN 1015-3 [28], the consistency of fresh plaster was determined using a flow table. The consistency was characterized by measuring the mean diameter of a test sample of fresh plaster that had been given 15 vertical impacts by allowing it to fall freely through a given height. A truncated conical mould (60 mm high with internal diameters of 100 mm at the bottom and 70 mm at the top) was used to shape the specimen, which was placed at the centre of a flow table disc.

To obtain similar consistency among the materials, ensuring good workability, the water content was adjusted. The German standard [23] proposes fixing the consistency at 175 ± 5 mm. The water content of fresh plaster was measured by drying at 60°C.

2.2.2.2 Shrinkage

As the shrinkage of earth mortars was significant not only along their length, volumetric shrinkage was evaluated. Once stabilized, the dimensions of the specimens were measured using a calliper. For each formulation, the volumetric shrinkage was expressed as the mean percentage volumetric variation between the fresh and hardened state of three $40 \times 40 \times 160$ mm³ prismatic specimens of mortar.

2.2.2.3 Flexural and compressive strength

The tests of flexural and compressive strengths were carried out in accordance with EN 1015-11 [29] on prismatic specimens of earthen mortar prepared following the same standard. Once demoulded, the samples were kept for at least 21 days. For the last few days, samples were stored at $(23 \pm 2)^\circ\text{C}$ and $(50 \pm 5)\%$ relative humidity until mass stabilisation was reached.

The tests were performed using an MTS bending press equipped with a 10 kN sensor. Three $40 \times 40 \times 160$ mm³ samples per formulation were provided for the three-point bending test. The

two halves of 40 x 40 x 160 mm³ samples obtained from the flexural test were used to provide six half specimens for the compressive strength test.

The samples failed between 30 and 90 s in both tests. The loading speeds used were 10 N/s when testing the flexural strength and 50 N/s when testing compressive strength.

3 RESULTS

3.1 Chemical and mineralogical characteristics

3.1.1 Chemical compositions

Table 2 presents the chemical compositions of the raw materials studied, obtained by ICP-AES and loss on ignition (L.O.I.).

Table 2. Chemical analysis of the four earths (in percentages)

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	L.O.I	Total
B	61.71	17.24	5.59	0.07	1.34	1.87	0.16	3.84	0.73	0.12	7.52	100.16
C	56.70	21.60	6.60	0.19	2.22	3.20	1.80	3.07	0.86	0.19	5.48	101.90
F	18.73	7.47	2.39	0.03	1.27	35.27	0.09	0.90	0.39	0.09	31.92	98.55
N	55.18	13.98	4.69	0.06	2.28	7.27	0.66	2.94	0.54	0.16	11.72	99.47

Soils B, C and N are mainly composed of silica and alumina. These compounds are distributed between clay and detrital minerals in soils, like quartz and feldspars. Quantities of silica and alumina vary among the soils: B has a higher SiO₂ content than C and F, while C exhibits the highest Al₂O₃ content, followed by B and N.

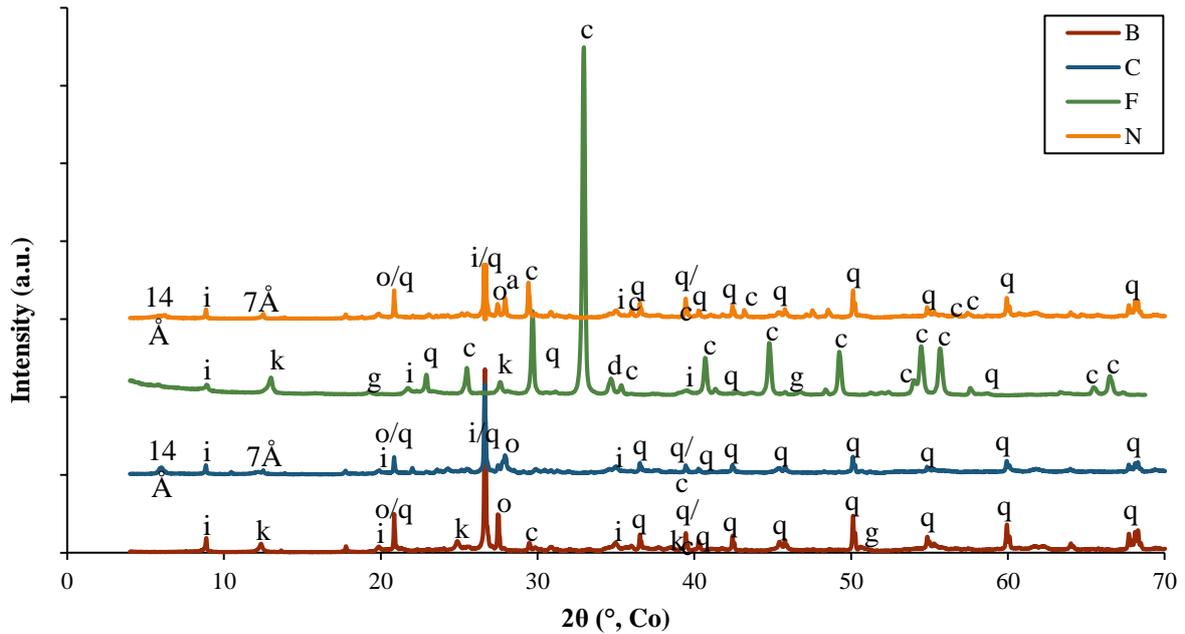
The presence of other important elements: iron (about 5%) and potassium (about 3%) oxides can be highlighted. The proportions of these elements are similar among the three earths B, C and N. Magnesium oxide and especially calcium molecules are present in significant quantities. The calcium content of soil N is much higher than that of soils B and C.

The chemical composition of soil F differs radically from that of the other soils. It can be noticed that soil F contains a large amount of calcium. This finding is in accordance with the soil origin: fines F come from the washing process of limestone aggregates, so the calcium content is high. Silica and alumina components are also present but in smaller quantities. In addition, unlike the other three soils, soil F contains small amounts of iron and potassium.

The loss on ignition measured at 1000°C on soil F is much greater than on earths B, C and N. This may be explained by the decarbonation of the calcite, which is contained in greater quantities in this soil. Sample N also exhibits a high loss on ignition. This could be due to the larger amount of hydration water and of calcite contained in the soil.

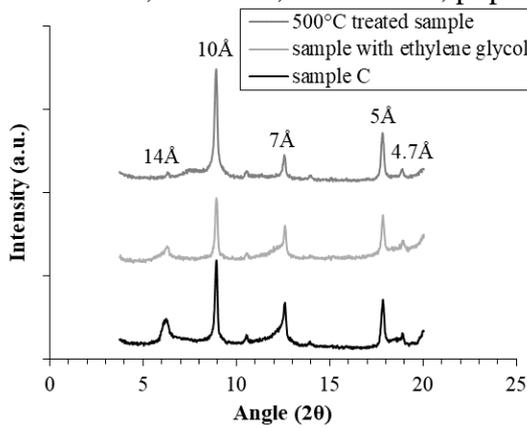
3.1.2 X-ray diffraction

The X-ray diffractograms are presented in Figure 1.

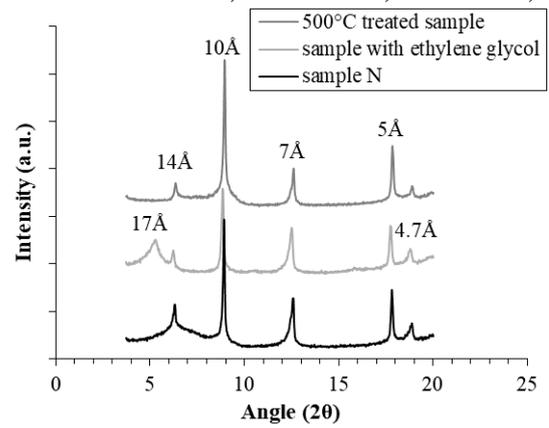


a.

a: albite, c: calcite, d: dolomite, p: pargasite, g: goethite, i: illite/muscovite, k:kaolinite, o: orthose, q:quartz



b.



c.

Figure 1. X-ray diffraction patterns of the four earths (a.), and of soils C (b.) and N (c.) following the oriented aggregate method

The diagram of F quarry fines reveals the strong presence of calcite (CaCO_3) and shows the presence of other minerals in smaller quantities: kaolinite ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$), quartz (SiO_2), illite ($\text{KAl}_2(\text{AlSi}_3)\text{O}_{10}(\text{OH})_2$), goethite ($\text{FeO}(\text{OH})$) and dolomite ($\text{CaMg}(\text{CO}_3)_2$).

The X-ray diffractograms reveal that soils B, C and F are mainly composed of quartz. The presence of illite minerals is also evidenced. In the soils B and C, glittery grains are visible to the naked eye, indicating the presence of muscovite minerals. In addition to these constituents, the diagrams show that the three soils contain feldspars. However, the nature of feldspars in the soils varies: soils C and N contain albite ($\text{NaSi}_3\text{AlO}_8$) and orthoclase (KSi_3AlO_8), while soil B only contains orthoclase. Moreover, goethite is identified in soil B and calcite elements are more significant in the composition of soil N than of soils B and C.

In addition to all these minerals that are usually found in clay soils, soil C contains pargasite ($\text{KCa}_2\text{Fe}_4\text{AlSi}_6\text{Al}_2\text{O}_{22}(\text{OH})_2$), which is an amphibole. Amphiboles are present in rocks but are absent from conventional soils resulting from the weathering of rocks. Since soil C is not an excavated soil but the residue from the washing of aggregates, it is possible to find fine elements in this residue that come from rocks containing amphiboles.

The X-ray diagrams of soils C and N do not allow the nature of the clays involved to be identified because of the peaks at 7 Å and 14 Å, which may correspond to different types of clay minerals. The oriented aggregate method was thus used; the corresponding X-ray diagrams are shown in Figures 1.b. and c.

The diagrams obtained are assessed considering the evolution of the first four peaks (from left to right) during the various preparations of the earth samples. Illite exhibits a first-order reflection value of about 10 Å, which remains fixed both in the presence of polar liquids and after drying. Thus, for both soils, the peaks at 10 Å and 5 Å show the presence of illite. On the untreated samples of soils C and N, the peaks at 7 Å and 14 Å could correspond to the reflections of montmorillonite, chlorite or vermiculite. Specific pretreatments are thus required to ensure proper identification [7,30]. Heating at 500°C destroys montmorillonite and vermiculite but not chlorite. Moreover, the expansive character of montmorillonite minerals provides the basis for their positive identification: when air dried, these minerals may have basal spacings of 12 to 15 Å. When they are oven dried, this value drops to about 10 Å as a result of the removal of interlayer water. After treatment with ethylene glycol, the montmorillonites expand to a first-order basal reflection value of 17 to 18 Å. Finally, vermiculite, does not expand in presence of ethylene glycol: it looks much the same after such treatment. One hour of heating above 300 °C destroys vermiculite and, as for montmorillonite, the basal reflection drops to 10 Å.

Therefore, the oriented X-ray diagram for soil C shows the disappearance of the 14 Å peak along with a strong growth of the 10 Å peak in presence of ethylene glycol. This behaviour is typical of the presence of vermiculite. The broad peak at 7 Å disappears to a large extent, but a thin peak is still visible, attesting the presence of kaolinite (first-order reflection peak).

When the samples of soil N were heated up to 500°C, the intensity of the peaks at 14 and 7 Å decreased and the peak at 10 Å widened. The presence of chlorite is proved by the fact that these peaks did not disappear completely. The treatment with ethylene glycol shifted the peak from 14 Å to 17 Å, showing the presence of montmorillonite.

This assessment enables to conclude that fines C contain kaolinite, illite and vermiculite, while illite, montmorillonite and chlorite minerals compose soil N.

3.1.3 Thermo-gravimetric analysis

To complete the mineralogical characterisation, soils were subjected to thermo-gravimetric analysis, the results of which are presented in Figure 2.

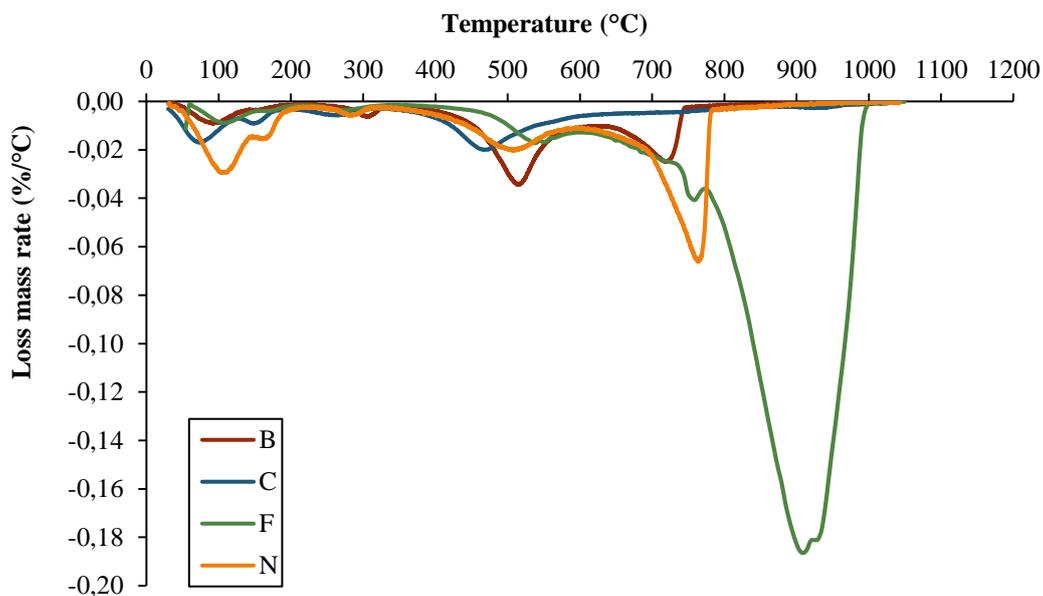


Figure 2. Derivative thermo-gravimetric curves

The first loss of mass with rising temperature, which occurs between 80 and 160°C, is due to the evaporation of water. The amount of hygroscopic water that evaporates is greater for soil N, followed by soil C, than for the other earths. This observation is consistent with the swelling potential of the clay minerals in these soils. Montmorillonite and, to a lesser extent, vermiculite have a high capacity to store humidity in ambient air.

Lower intensity peaks, around 300°C, correspond to the dihydroxylation of goethite elements. In the range of 470-550°C, the constitutive water is removed. As several clay minerals are present simultaneously in soils and as their dihydroxylation temperatures overlap, it is not possible to identify the type of clay minerals present from these curves.

In the case of soil F, the greatest loss of weight occurred around 730-930°C and was due to the decarbonation of calcite and dolomite. This loss was about 50% for soil F, demonstrating the high proportions of these elements in the sample.

3.1.4 Mineralogical compositions

By combining both mineralogical and chemical characterisations, a mineralogical composition that was as consistent as possible with the experimental results was established. The X-ray diagram, using the oriented aggregate technique if necessary, allowed the nature of the minerals composing the soils to be identified.

The quantitative assessment of the minerals present relied mainly on the chemical composition. This step required strong assumptions on the chemical formulae of minerals, knowing that some of them have highly variable chemical compositions. The thermo-gravimetric analysis was also used to establish the mineralogical composition, since it was possible to estimate the amount of FeO(OH) and $\text{CaMg}(\text{CO}_3)_2 / \text{CaCO}_3$ from the mass losses due to dihydroxylation of goethite and decarbonation of calcite and dolomite.

Finally, the derivative thermo-gravimetric curves were employed to validate the previously established potential mineralogical composition. Based on the weight loss due to dihydroxylation of clay minerals, the consistency of the estimated clay content was checked.

To meet most of the experimental requirements, an iteration processed was employed. The most satisfactory compositions are presented in Table 3.

Table 3. Potential mineralogical composition of soils B, C, F and N

	B	C	F	N
Carbonate minerals				
calcite/dolomite [%]	5	2-4	66	12
Oxide minerals				
goethite [%]	2	5	3	1
haematite [%]	3	-	-	-
Tectosilicates minerals				
quartz [%]	33-35	18	10	22-23
feldspars				
<i>albite</i> [%]	-	15	-	5
<i>orthoclase</i> [%]	10-14	5	-	3
<i>muscovite</i> [%]	5-13	13	*	0-3
Inosilicate minerals				
pargasite [%]	-	5	-	-
Phyllosilicate minerals				
chlorite [%]	-	-	-	8
illite [%]	9-12	10	11*	19-23
kaolinite [%]	23	15-17	11	-
montmorillonite [%]	-	-	-	19-22
vermiculite [%]	-	9	-	-
Total of clay minerals [%]	32-35	34-36	22*	46-53

*It was not possible to make the distinction between illite and muscovite minerals for F fines.
The estimation of 11% corresponds to the sum of the two types of minerals.

3.2 Geotechnical characterisation of soils

Based on the potential mineralogical compositions of soils, this section will focus on the interpretation of the measured geotechnical characteristics.

3.2.1 Particle size distribution

The particle size distribution curves from the various samples are shown in Figure 3, which includes both sieving and hydrometer testing. The passing percentages along with the median grain diameter values (D50) and the diameter for which 90% of the distribution lies below this value (D90) are noted in Table 4.

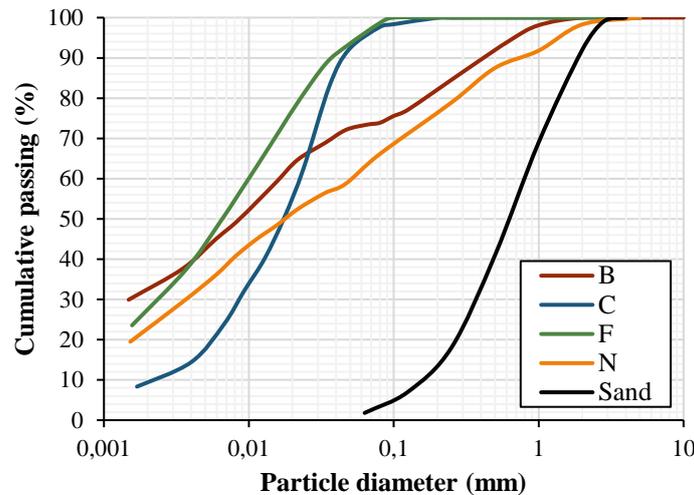


Figure 3. Particle size distribution of sand and soils B, C, F and N

The curves show two types of grain assemblies, which is not surprising given the distinct origins of the materials. Extracted directly from natural earths, soils B and N present a broader particle size distribution than quarry fines C and F. About 30% of sand grains are encountered in the former soils while the latter are composed for more than 90% of fine particles (clay and silt). All samples contain little or no gravel and a proportion of clay sized particles ranging from 10 to 33%.

Table 4. Characteristic dimensions of soils

Soils	Clay < 2 μm (%)	Silt 2-63 μm (%)	Sand 63-2000 μm (%)	Gravel > 2 mm (%)	D90 (μm)	D50 (μm)
B	33	40	27	0	413	9
C	10	88	2	0	44	17
F	28	66	6	0	37	7
N	23	37	38	2	750	18

Soil C has the lowest proportion of particles smaller than 2 μm (only 10%). Referring to the same particle size category, soil B appears to be the most clayish soil (33%), followed by fines F (28%) and soil N (23%). Even though the amount of material finer than 2 μm is often used to approximate the amount of clay mineral, these clay size percentages are far from the estimations given in Section 3.1.4 **Erreur ! Source du renvoi introuvable.** This was to be expected since not all clay particles are smaller than 2 μm, and not all non-clay particles are coarser than 2 μm. More particularly, the washing fines are likely to contain fine non-argillaceous particles, such

as limestone fines for soil F. Thus, the percentage of clay minerals is three to four times higher than the percentage of clay sized particles in fines C. Results for the excavated soils are not satisfactory either: soil N contains 46 to 53% of clay minerals, twice the percentage of clay sized particles.

For this reason, a soil's grain composition alone does not allow a comprehensive assessment to be made of its suitability as a construction material. Although criteria and threshold proportions of clay, silt and aggregate have been set in various references [31,32], studies have shown that it is possible to manufacture earthen materials with a huge variety of earths, including some that are in contradiction with the grading recommendations stated in the literature [10,33,34]. Moreover, these quantitative limits vary significantly among references and the indicated partial size thresholds are not always comparable (the percentage of sand and gravel may be considered together and different documents often refer to different test procedures, in which the sieve apertures are not necessarily equivalent) [32]. Because granularity fails to evaluate the specific quality of clay minerals, clay-water interactions are always assessed alongside grain size parameters.

3.2.2 Atterberg limits and methylene blue value

The argillaceous content and clay activity were qualified through two different techniques: Atterberg limits and the methylene blue (MB) test. The results from these two tests, along with the percent passing the 2 μm sieve ($P_{2\ \mu\text{m}}$), are brought together in Table 5.

Table 5. Atterberg limits (LL, PL, PI), activity (I_A) and methylene blue (MB) absorption values

Soils	B	C	F	N
$P_{2\ \mu\text{m}}$ (%)	33	10	28	23
LL (%)	39.9	42.8	32.9	44.4
PL (%)	17.5	30.4	21.2	21.7
PI (%)	22.4	12.4	11.7	22.7
I_A	0.69	1.24	0.42	0.99
MB (g/100g)	2.65	0.78	1.64	4.10

Atterberg limits of the investigated earths were between 32.9 and 44.4% for the liquid limit and in the 17.5-30.4% range for the plastic limit. The higher the liquid limit, the higher the clay content; hence, the LL results imply that soil N had the highest clay content, followed by C, B and finally F, which is quite consistent with the potential mineralogical composition.

Calculating the difference between the two standardized moisture content levels determined the plasticity index. It represents the range of the plastic domain (Table 6): the higher the plasticity index, the more pronounced the colloidal properties of the clay.

Table 6. Correlation between the plasticity index and a soil sample's level of plasticity (Brown, 2016)

PI (%)	Level of plasticity
0-3	non plastic
3-15	slightly plastic
15-30	medium plastic
>30	highly plastic

The soils investigated showed two distinct plasticity levels: fines C and F exhibited similar values of PI, about 12%, and the soils extracted at brickworks were grouped around the value of 22.5%. Hence, C and F are classified as slightly plastic materials while B and N are

considered as medium plastic soils. According to Gee [35], the level of PI enables conclusions to be drawn about the presence of swelling-type clays: swelling clays, such as smectites, are typically more plastic than non-swelling clays (e.g. kaolinites), so soils B and N are slightly more likely to contain swelling-type clays than fines C and F. It is difficult to relate this statement to the potential mineralogy compositions. On the one hand, B and F contain illite and kaolinite and can thus be expected to behave in roughly the same way, even if B is slightly richer in clay mineral than F (33% and 14-18% respectively). On the other hand, B and C are also close in composition, with some kaolinite content being replaced by vermiculite, a mineral with higher swelling properties than kaolinite. Hence, a similar, or even higher, plasticity level would have been expected from soil C than from soil B.

These inconsistencies might stem from the PL determination, especially regarding soil C. Unlike the LL test, which seems quite reliable and reproducible, the PL determination depends heavily on the operator's appreciation and the results are therefore open to discussion.

Furthermore, Skempton [36] proposed a relationship between the plasticity index and the clay fraction in clayey soils to determine the activity of clays, I_A ($I_A = PI/P_{2\mu m}$). High activity signifies a large volume increase in a soil that is wetted and large shrinkage when it dries. Soils with high activity are very reactive chemically. Consequently, C seems to be more sensitive to drying shrinkage than soils N, B and, lastly, F.

Even though some authors have based their clay mineralogical analysis on typical values of LL, PL or I_A for kaolinite, illite and montmorillonite [37], I_A should be used cautiously for clay identification. On the one hand, the relevance of the percent passing the 2 μm sieve as a parameter for the identification of clay minerals can, once again, be questioned. On the other hand, in some cases, soils in which expanding clays, such as montmorillonite and vermiculite, are predominant, have proved to have much lower values, less than half of the 1.25 figure that is proposed for active clays [19,22,38].

The methylene blue test consists of measuring the quantity of methylene blue that can be absorbed by a clayey soil. The water sensitivity evaluation is largely related to the amount and nature of the clays contained in the earth. The higher the MB value, the higher the plasticity of the soil.

In this study, the MB values ranged from 0.78 to 4.10 g/100 g. With the lowest MB value, C fines were in the typical range for silty soils ($MB < 1.5$ g/100 g) which is consistent with the texture analysis previously presented for this soil. Soil F was classified, according to the French management system for road earthwork materials named "Guide des terrassements routiers" [39], as a silty soil with low plasticity, while soils B and N were considered of medium plasticity. The findings from the MB test are not fully consistent with the previous characterisation elements. This might be because the MB spot test contains operator-related errors (e.g. in judging the end-point), the method of sample preparation is not adapted, the dispersion time is insufficient or even because these parameters depend on the type of clay minerals present in the soil [22,40,41].

Moreover, the French classification of soils states that, when $PI > 12$, the MB value should not be preferred to Atterberg limits as a criterion, and vice-versa [39].

These two commonly used methods for earth classification measure the water sensitivity of soils, which is largely related to the amount of the finest elements present in the earths, in particular clay particles [19]. The relationship between Atterberg limits, MB values and the percentage of particles passing the 63 μm sieve is investigated in Figure 4.

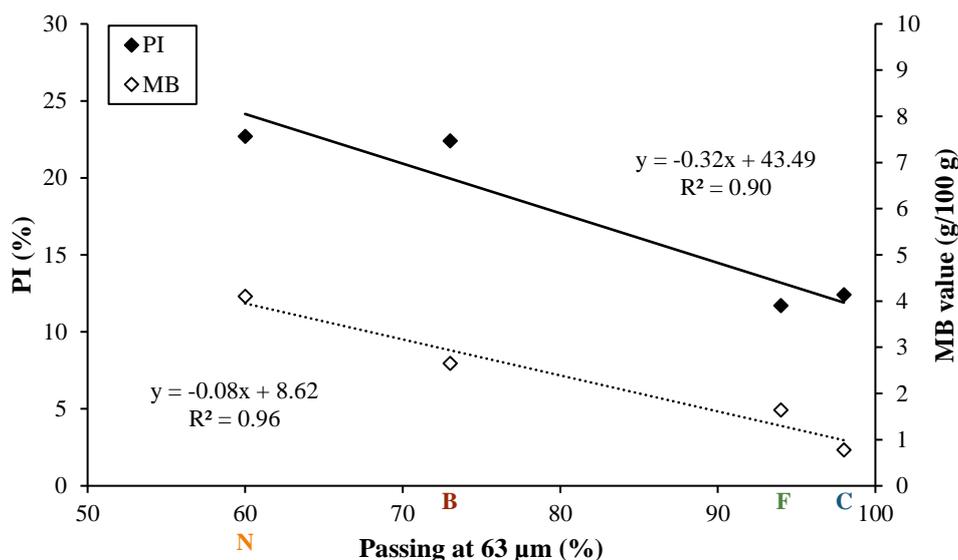


Figure 4. Correlation between plasticity index (PI) or methylene blue (MB) value and the percentage of particles smaller than 63 μm in soils

A strong linear relationship can be noted between the plasticity index and the percentage of particles passing through a sieve of 63 μm mesh. Similar observations can be made regarding the MB value. When the fine elements content of a soil increases, the plasticity index and methylene blue value are lowered, evolving toward values corresponding to a siltier soil.

3.2.3 Cation exchange capacity and specific surface area

Soils containing clay minerals possess negatively charged particle surfaces that hold exchangeable cations, generally calcium (Ca), potassium (K), magnesium (Mg), and sodium (Na). These cations can be exchanged between the mineral surface and the pore water solution without a mineral structure change. The amount of exchangeable cations a soil possesses is referred to as its effective Cation Exchange Capacity (CEC). The cations are required to balance the charge deficiency of clays. CEC depends on clay type and clay content, as well as organic matter content. Table 7 gives the specific surface area, effective CEC and exchangeable cations of the four soils studied.

Table 7. Effective CEC, exchangeable calcium, potassium, magnesium and sodium, and specific surface area results

Soil	B	C	F	N
Effective CEC (cmol ⁺ /kg)	10.26 ± 0.67	12.05 ± 0.76	19.51 ± 0.79	24.81 ± 0.54
Exchangeable Ca (cmol ⁺ /kg)	29.81 ± 1.29	24.96 ± 1.31	165.81 ± 14.59	94.70 ± 3.21
Exchangeable K (cmol ⁺ /kg)	0.65 ± 0.01	0.78 ± 0.03	0.36 ± 0.00	0.48 ± 0.01
Exchangeable Mg (cmol ⁺ /kg)	6.40 ± 0.13	3.40 ± 0.05	4.24 ± 0.19	9.44 ± 0.03
Exchangeable Na (cmol ⁺ /kg)	0.18 ± 0.01	0.59 ± 0.06	0.35 ± 0.03	0.14 ± 0.01
Specific surface area (m ² /g)	21.18	16.98	14.25	28.33

CEC values range from 10.26 to 24.81 cmol⁺/kg. The higher values, 19.51 ± 0.79 and 24.81 ± 0.54 cmol⁺/kg, were found for F and N soils respectively. These results are the highest because of the large amount of exchangeable Ca²⁺ available in the two soils.

The results for the sum of exchangeable cations are greater than the actual CEC, due to the dissolution of salts present in the soil. Preliminary washing of the soil with water, not performed

on the studied soils, must remove these salts and, hence, change the relative proportions of cations in the CEC.

In a previous study, the commonly used geotechnical engineering indices, such as Atterberg limits and methylene blue values, were found to be closely correlated with the CEC [42], which does not appear to be the case here. According to Laribi et al. [8], the MB value is, in fact, correlated with the cation exchange capacity of soil particles. No clear link between CEC and methylene blue results could be established either. Finally, unlike the statement of Woodruff and Revil [43], the CEC was not observed to be linearly related to the specific surface area of clay minerals. The absence of obvious correlation between these parameters might come from the amount of calcium in the specimens tested. The correlation observed when considering the sum of exchangeable cations, excluding Ca^{2+} , is presented in Figure 5.

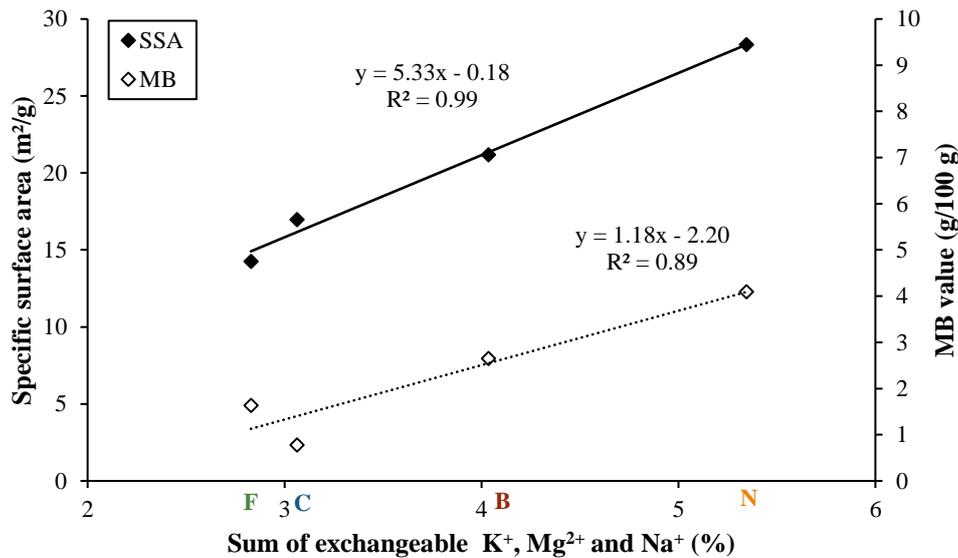


Figure 5. Correlation between both specific surface area (m^2/g) and methylene blue (MB) value and the sum of exchangeable cations, except for Ca^{2+} in soils

Thus, the sum of exchangeable K^+ , Mg^{2+} and Na^+ cations appears to be linearly linked to both specific surface area and MB value.

The specific surface areas, measured by gas absorption, are quite close for the soils studied, ranging from 14 to $28 \text{ m}^2/\text{g}$. C and F fines show quite low values, respectively 16.98 and $14.25 \text{ m}^2/\text{g}$, while B and N exhibit higher values (21.18 and $28.33 \text{ m}^2/\text{g}$ respectively). The small crystal size yields a high specific surface area, which promotes high surface reactivity.

According to Meimaroglou and Mouzakis [19], three-layer clays such as smectite and vermiculite have a large specific surface, providing a higher bonding capacity than two-layer clays (e.g. kaolinite). This observation implies that soils B and N contain clays with a stronger swelling potential than C and F do, which is inconsistent with B and C potential mineralogical compositions, where C contains expandable clays that are not present in B. On the contrary, findings for N are consistent since the soil contains montmorillonite minerals.

It should also be noted that, if the nitrogen absorption method is used, the molecules of the gas cannot cover the interlayer surface in expansive layer-silicates, which remain tightly bound under dry conditions, giving low values of specific surface [44].

Some typical values of cation exchange capacities and specific surface areas for fundamental soil colloids are given in Table 8.

Table 8. Characteristic properties of soil colloids (Brady and Weil, 2016)

Colloid	Cation exchange capacity (cmol ⁺ /kg)	Specific surface area (m ² /g)
Humus	100-500	variable
Chlorite	10-40	70-100
Illite	10-40	70-150
Kaolinite	1-15	5-30
Smectite	80-120	80-150
Vermiculite	100-200	70-120

Based on the potential mineralogical composition and Table 8, and using variable CEC and SSA values within the specific range of each colloid, it was possible to estimate theoretical value ranges for both cation exchange capacities and specific surface areas of the four soils studied. The theoretical and the experimental values are given in Table 9.

Table 9. Theoretical estimations and experimental results for cation exchange capacities and specific surface areas

Soil	Cation exchange capacity (cmol ⁺ /kg)		Specific surface area (m ² /g)	
	Theoretical estimations	Experimental results	Theoretical estimations	Experimental results
B	1 - 8	10.26 ± 0.67	7 - 23	21.18
C	7 - 19	12.05 ± 0.76	12 - 27	16.98
F	0 - 2	19.51 ± 0.79	3 - 13	14.25
N	18 - 39	24.81 ± 0.54	34 - 76	28.33

The roughly estimated theoretical values are quite satisfactory compared with the experimental values, except for the CEC estimation of F, which is far from the experimental measurement. The large calcite content in the washing fines could be responsible for the CEC overestimation. To a lesser extent, the experimental SSA value for N is somewhat different from the calculated one.

Finally, the specificities of the studied soils might explain the divergence between literature findings and results from the present study. Compared to types of soils commonly used for construction purposes, C and F washing fines exhibited very fine elements and, particularly, relatively low ratios of clay mineral grains to fine particles. The mineralogical composition is of great importance in the water sensitivity of earths. An attempt could have been made to relate the behaviour of earthen materials to their mineralogical clay content. However, since the nature of clay minerals, and therefore their expansive abilities, vary considerably depending on the soil, it would not have been possible to establish a correlation between clay mineral content and plasticity of soils.

The analysis of the geotechnical characteristics in conjunction with the potential mineralogical composition of four soils enabled us to identify some geotechnical parameters that seemed more relevant than others for the characterisation of clayish materials. The selected indicators were chosen on the basis of consistency between the potential mineralogical composition and the estimated argillaceous content and clay activity quantified by the different geotechnical tests. Therefore, the liquid limit, the cation exchange capacity and the specific surface area appeared as the most reliable properties to explain the water affinity and cohesive capacity of a soil. To discuss this assessment, these three physical soil characteristics, along with the most commonly used 2 µm passing percentage parameter, will be used as criteria for the evaluation of earth-based plaster in both the fresh and hard states. This item will be the subject of the last part of this article.

3.3 Soil properties and behaviour of plasters

This part of the article aims to evaluate the relationship between some physical characteristics of soils and the properties of earth-based plasters in the fresh and hardened states. The fresh state was assessed by determining the water requirement. The mechanical behaviour was measured through shrinkage, and flexural and compressive strengths.

The percent passing the 2 μm sieve, the liquid limit, the CEC and the specific surface area were estimated for the 25 studied plasters based on the measured characteristics of the four soils. The properties of the plasters are attributed to the soil fraction, without considering the added sand. This very strong hypothesis is supported by the low fraction passing the 63 μm sieve (less than 2%) as well as the low methylene blue absorption value ($0.38 \pm 0.1 \text{ g}/100 \text{ g}$). Indeed, the little sand fraction passing the 63 μm sieve was neglected, enabling the estimation of the clay size content based on the soil fraction only. Since the MB and the water absorption values of sand were quite low, the CEC and liquid limit were attributed entirely on the clay particles of the soil fraction.

3.3.1 Water content

To establish a potential relationship with the soil properties, the water required to achieve the flow table value of $175 \pm 5 \text{ mm}$ is plotted versus the percentage of particles passing at 2 μm , the liquid limit, the CEC and the specific surface area in Figure 6.

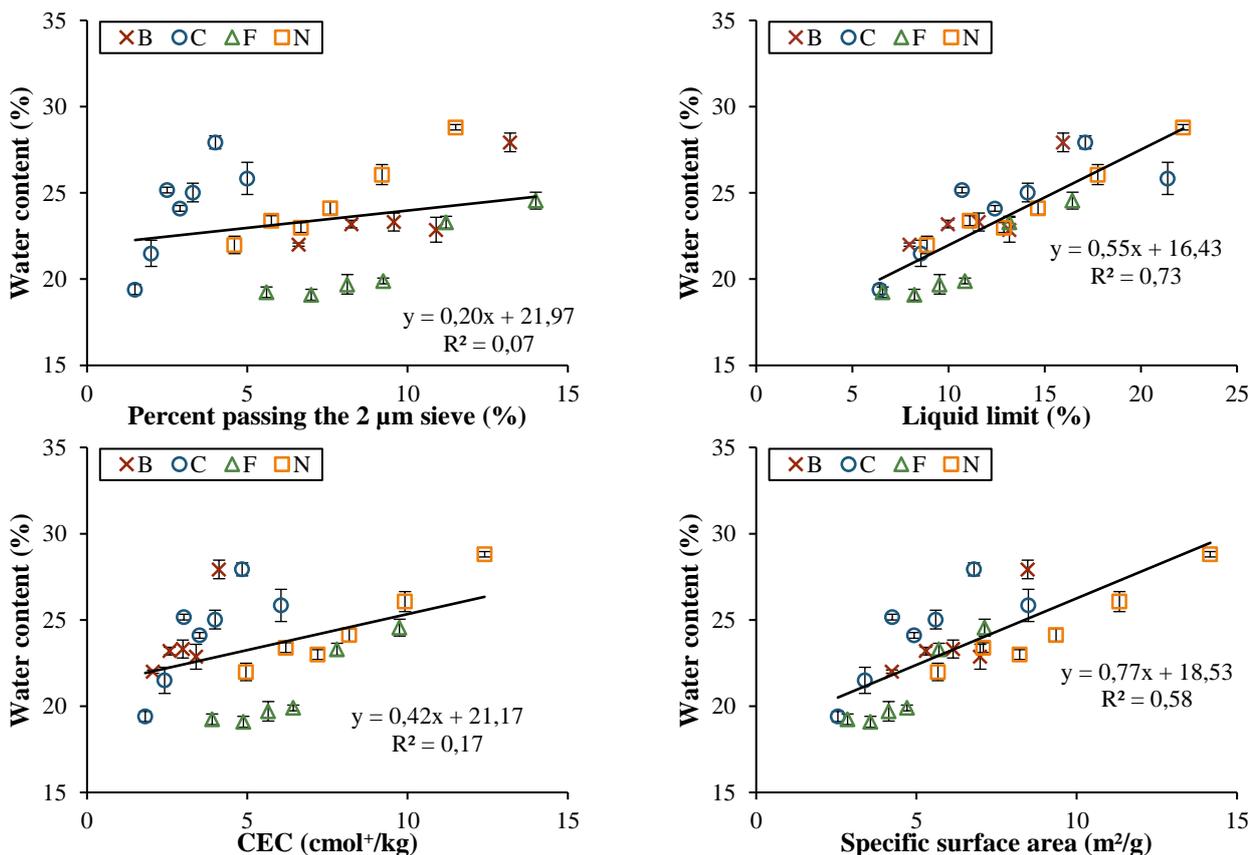


Figure 6. Potential dependency of water content in the percent passing the 2 μm sieve, the liquid limit, the CEC and the specific surface area of earthen materials

The water needed to achieve the target consistency does not appear to be dependent on the percent passing the 2 μm sieve, the CEC or the specific surface area. In contrast, the required moisture content is strongly correlated to LL, as would be expected. The higher the liquid limit, the greater the amount of water required. It is true that, in a very wet state, a fine-grained soil

acts as a viscous material, ideal for application as a coating. Thus, the moisture content must be adapted to be close to the liquid limit, which is the minimum moisture content at which a soil flows.

With a lower target consistency (140 ± 0.3 mm at the flow table), Meimaroglou and Mouzakis [19] found that nine out of the twenty-six soils they tested had only $\pm 2\%$ difference between LL and the water added. Therefore, the flow table test could appear to be a promising method for determining the plastic range of soils.

3.3.2 Shrinkage

Linear and volumetric shrinkage were measured and examined in accordance with all the physical soil properties previously evaluated (Figure 7).

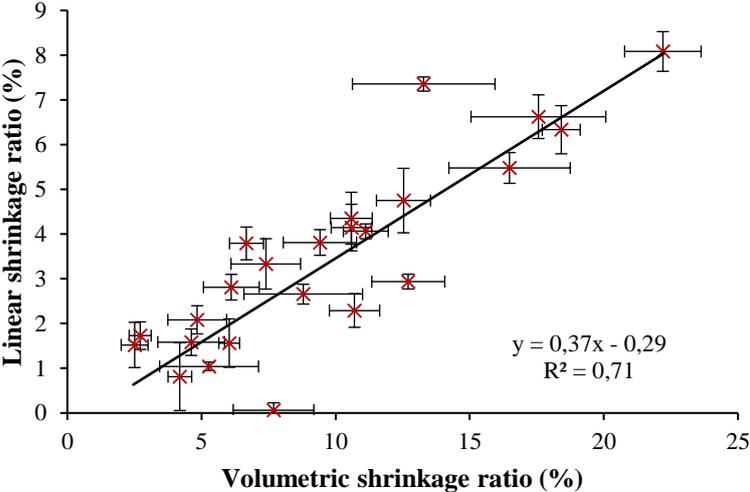


Figure 7. Correlation between linear and volumetric shrinkage ratios

Although the two parameters appear to be linearly dependent on one another and the volumetric shrinkage ratio seems to be the more sensitive parameter, more representative of the total shrinkage than the linear ratio, the relationship between shrinkage ratio and the characteristics of clayish soils was clearer when the linear ratio was considered. The relationship between linear shrinkage ratios of plasters and four geotechnical characteristics are presented in Figure 8.

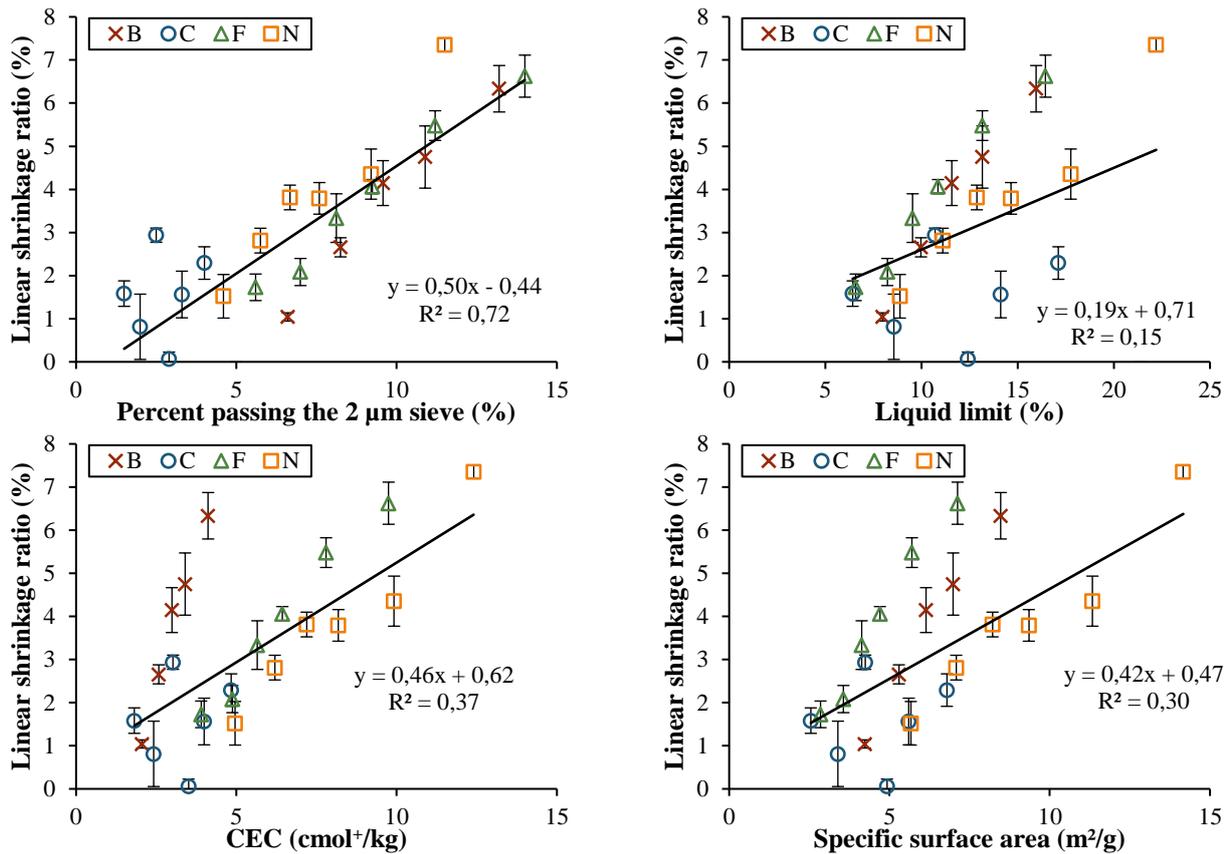


Figure 8. Potential dependency of linear shrinkage in the percent passing the 2 μm sieve, the liquid limit, the CEC and the specific surface area of earthen materials

First, it can be noted that, based on the German standard [23], only 8 out of 25 formulations meet the linear shrinkage requirements, with values lower than 2%. When considering a thin coat application, this threshold value can be increased to 3%, for which 13 out of 25 mix designs are satisfactory.

The shrinkage ratio on prismatic specimens rises linearly with the clay size fraction, confirming the trend reported by Delinière et al. [11], Gomes et al. [13], Hamard et al. [14] and Meimaroglou and Mouzakis [19]. However, it cannot be explained by the amount of binding agent since clay minerals and clay sized grains refer to two distinct types of elements. Yet, this result could be due to other physical phenomena, such as capillary pressures: the capillary forces might be more likely to increase with the increase in smaller grains, bringing the fine soil particles closer to each other and leading to a greater contraction causing mortar shrinkage.

The shrinkage ratio might indicate if the clay is reactive or inert. However, this study revealed no clear correlation between the shrinkage of the plasters and the soil properties linked to their mineralogical composition (Figure 8). Research findings of Meimaroglou and Mouzakis [19] reached the opposite conclusion: their work demonstrated a stronger dependence of linear shrinkage ratio on CEC than on clay size fraction.

3.3.3 Flexural and compressive strengths

The flexural and compressive strengths measured on prismatic specimens of earth mortars are gathered together in Figure 9.

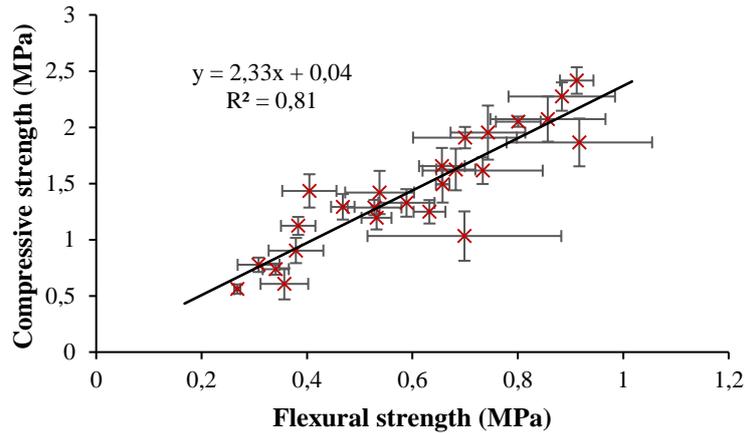


Figure 9. Relationship between flexural strength and compressive strength

The compressive strength of earth-based plasters is strongly related to their flexural strength. Compressive strengths are about 2.4 times higher than flexural resistances. Compressive strength consistently increases as flexural strength increases. A similar order of magnitude of compressive strength to flexural strength ratio have been found in previous works on earth mortars [19,45]. Other earth building materials, such as extruded earth bricks and compressive earth blocks also exhibit this correlation [45–47]. This behaviour is specific to earth-based materials and is very different from values encountered for cementitious materials (ratio of approximately 10). In the case of compressive earth blocks, the three-point-bending test is of special interest for the indirect measurement of compressive strength for in situ quality control. Forces required to induce failure are typically 80-150 times lower than that required to induce failure under uniform compression. Consequently, this indirect measurement of compressive strength is quite achievable under site conditions without resort to sophisticated equipment [46].

Hereafter, only the compressive strength data are presented, but similar analysis regarding flexural strength can be carried out. To investigate the impact of texture as well as the mineralogical composition of soils on the mechanical resistance of earth-based plasters, the compressive strength is plotted as a function of clay size, liquid limit, CEC and SSA in Figure 10.

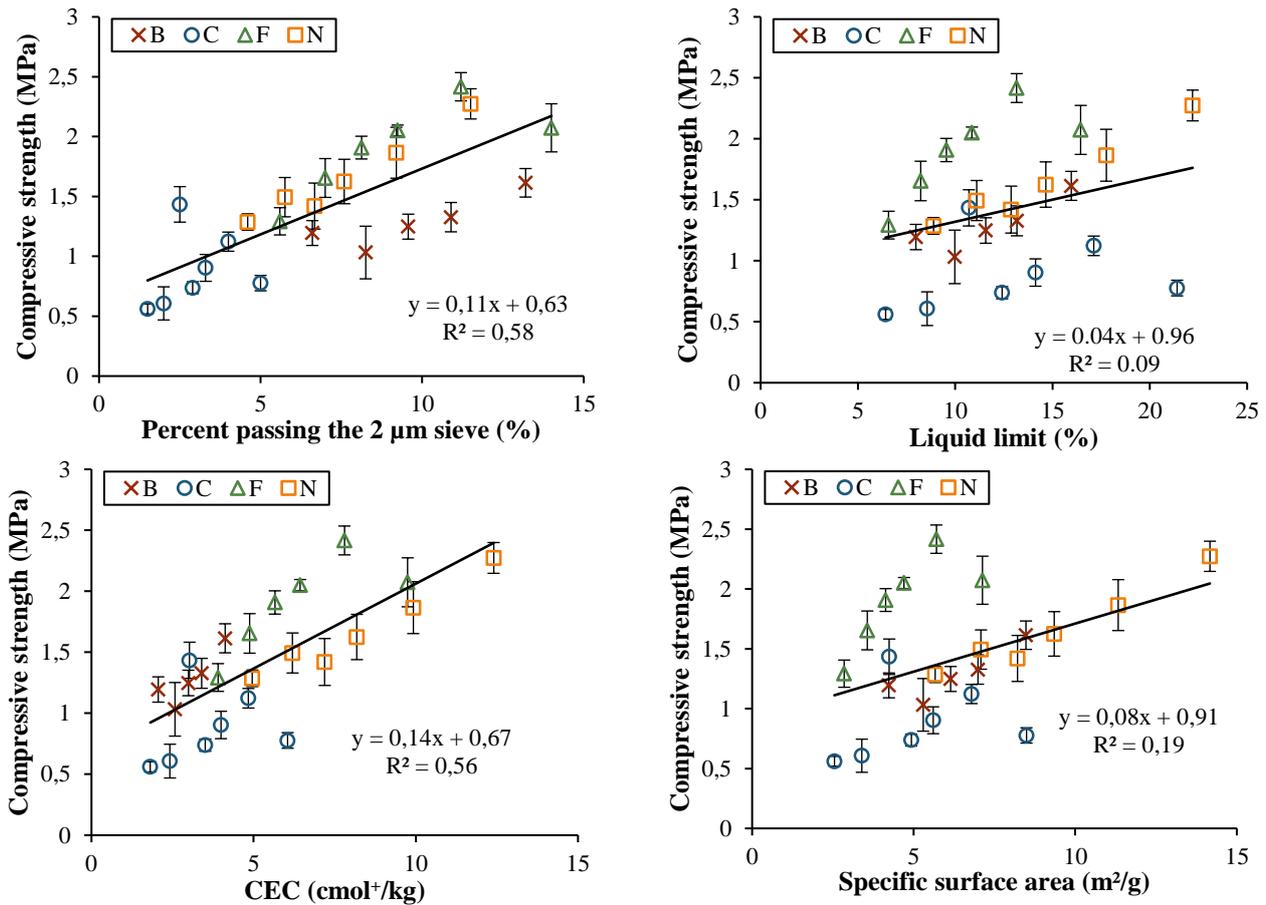


Figure 10. Potential dependency of the compressive strength in the percent passing the 2 μm sieve, the liquid limit, the CEC and the specific surface area of earth-based plasters

The compressive strength increases along with the percent passing the 2 μm sieve. A similar correlation has also been established by Delinière et al. [11], Emiroğlu et al. [12], Hamard et al. [14], Lima et al. [15] and Meimaroglou and Mouzakis [19]. Consequently, the mechanical behaviour of plaster appears to be dependent on the granular arrangement, since the finest fraction of soil lowers the volume of voids within the material, yielding a more resistant specimen. These results are consistent with the linear relationship previously established between percent passing the 2 μm sieve and shrinkage ratios: the more compact the grain arrangement, the more resistant the plaster.

The dependence of compressive strength on CEC appears to be just as strong as on its percentage of clay sized grains. Thus, the amount of exchangeable cations enables the binding potential of the clay particles to be quantified. The higher the CEC (i.e. the quantity of cations), the higher the electrostatic attraction. When two clay particles are in proximity with each other, divalent and trivalent cations in the space between them attract the negative charges on the clay surfaces, thus forming bridges that hold the particles together [19]. These findings have also been reported by Meimaroglou and Mouzakis [19].

4 CONCLUSION

Earth exhibits numerous technical, ecological and economic assets that explain its recent upsurge as a building material. Its ability to buffer indoor moisture variations and to improve the comfort of the building's users makes clayish earth suitable for indoor plastering purposes.

The quantity of clay is frequently employed to explain both the fresh and the mechanical performances of earthen coatings. However, not all clay minerals are smaller than 2 μm , and

not all non-clay particles are coarser than 2 μm . Therefore, this texture indicator consistently fails to give full account of the behavioural diversity of soils encountered in earth construction.

This paper has aimed to examine the properties of plasters considering their geotechnical and mineralogical compositions. Four earths were employed to design earthen plasters with variable earth to sand ratios. The behaviour of coatings was assessed through water requirement, linear shrinkage, and flexural and compressive strength.

The first part of the study gave an interpretation of the soils' geotechnical specificities considering the potential mineralogical composition that was established. It became apparent that soil F was mainly composed of ultra-fine particles, including calcite elements, while soils C and N contained high-swelling-potential clay minerals: vermiculite and montmorillonite respectively.

The analysis of the geotechnical characteristics, in conjunction with the potential mineralogical composition, of four soils enabled us to identify three geotechnical parameters that seemed more relevant than others for the characterisation of clayish materials. On the one hand, the clay size percentages did not match any characteristics from the estimated mineralogical composition, especially when very fine soils were used; clay minerals and clay sized grains refer to two distinct types of elements. The size and mineralogical specificities of washing fines might explain the poor correlation between some geotechnical indicators and the mineralogical composition of soils C and F. On the other hand, the PL and MB value determinations are partly based on methods involving the operator's appreciation and the results are therefore debatable. Thus, the liquid limit, the cation exchange capacity and the specific surface area appear as the most reliable properties to characterise clayish materials.

The second part of the article, providing the relationship between soil properties and plaster performances, showed that the moisture content required to attain the target consistency is strongly correlated with the liquid limit determined in the Atterberg test. The correlations between linear shrinkage, or compressive strength, and percent passing the 2 μm sieve were strong, justifying the widespread use of grain size analysis and clay size content, in particular for soil classification, and the assessment of earth-based building materials' properties. In addition, the mechanical resistances were found to be linked to the cation exchange capacity of soils. The earth suitability for plasters should therefore be analysed by crossing granulometric information with clay mineralogy and water sensitivity.

Further investigations should be undertaken on a greater number of samples with more marked differences in composition to clearly confirm the correlations established here. Future efforts should attempt to establish correlations between the mineralogical composition of earth and the hygrothermal properties of earth-based plasters. This could enable raw materials to be selected for the development of a good/excellent moisture buffering plaster based on the soil characterisation. Finally, the soil composition may be of interest to explain the variable effects of organic adjuvants on the properties of earth-based plasters.

5 DATA AVAILABILITY

Some or all data, models, or code that support the findings of this study are available from the corresponding author upon reasonable request.

6 ACKNOWLEDGEMENT

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