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Research Article

Clay composition patterns and their influence on the adhesive strength of earthen plasters

García Villar Gonzalo[1], **Marcial Enzo**[2], **Rolón Guillermo**[3]

[1] Consejo Nacional de Investigaciones Científicas y Técnicas (CONICET), Centro Regional de Investigaciones de Arquitectura de Tierra Cruda (CRIATiC), Facultad de Arquitectura y Urbanismo (FAU), Universidad Nacional de Tucumán (UNT), Tucumán (Argentina); arqgonzalogv@gmail.com

[2] Consejo Nacional de Investigaciones Científicas y Técnicas (CONICET), Instituto Superior de Correlación Geológica (INSUGEO), Centro Integral de Microscopía Electrónica (CIME), Universidad Nacional de Tucumán (UNT), Tucumán (Argentina); enzomarc7@gmail.com

[3] Consejo Nacional de Investigaciones Científicas y Técnicas (CONICET), Instituto de Investigaciones Territoriales y Tecnológicas para la Producción del Habitat (InTePH), Facultad de Arquitectura y Urbanismo (FAU), Universidad Nacional de Tucumán (UNT), Tucumán, (Argentina); guillerolon02@gmail.com
*Correspondence: arqgonzalogv@gmail.com

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Abstract: The clay fraction of earthen plasters is the part responsible for the acquisition of cohesion and adherence that they possess against deterioration factors. Adherence is the property responsible for keeping the plaster together with the wall and is influenced by the percentage content of clay as well as by its mineralogy and the heterogeneity of minerals that may be present. However, it is still unknown in depth how clay minerals perform in the adherent properties of earthen plasters when the composition is heterogeneous in the material. The objective of this study is to evaluate the incidence of the mineralogical complexity of clay mineralogy in the variability of the adherence of earth plasters. To evaluate the variability, considering that the mineralogy of the soils depends directly on the place and the formation processes, eight soils from Tucumán (Argentina) corresponding to different physiographic units were analyzed. A methodology was designed for sample preparation that allows soils to be compared through adhesion tests. They were characterized by XRD to determine their mineralogical composition and by the hydrometric method to determine their granulometry. To evaluate the adherence of mixtures made with the respective soils, it was proposed in the first instance to compensate the granulometry of the soils to equate them and, once the plasters were made, this property was evaluated through shear and pull-off tests. The results showed that they allowed us to identify that the soils presented a pattern of mineralogical composition common to all the physiographic units, made up of the Ill and K pair, the former being predominant. For this pattern, it was observed in particular that there is a positive correlation between the increase in Ill content with the increase in the adhesive strength of the plasters. Clay minerals from the Sm group also contribute to the increase in adherence when the percentage is greater than or equal to 11%. On the contrary, K and Cl do not influence the increase in adhesive strength.

Keywords: shear test, pull-off test, granulometric compensation physiographic units, mineralogical composition

1. Introduction

The most important function of plaster is to protect the walls against the erosive action of natural and anthropogenic factors. In the case of earthen plasters, atmospheric agents play a significant role in the deterioration process by affecting their durability, while they can alter the internal cohesion of the material and its ability to adhere to the wall. One of the characteristics that allow the plaster to remain attached to the wall is the adhesive strength of the first, understood as the ability to resist normal and tangential stresses that affect the interface between the first and the second (Faria et al., 2019). Earth plasters are mainly composed of soil with clay content, additional sand, vegetal fibers, and water. The fraction of clay contained in the soil is a key factor as it is responsible for the internal cohesion (Jia et al., 2024) and adherence of the plaster; then there are the fractions of sand and silt, which make up a granular skeleton in charge of providing resistance and regulating porosity. The fibers, for their part, collaborate in absorbing the retraction stresses that may appear in the sample during the drying of the renders immediately after their application (Stazi et al., 2016). The incorporation of water is carried out to activate the adherent capacity of the clays, reach the plastic state of the mixture, and grant workability to it during the preparation and execution of the plaster. Earthen plasters, like traditional cement or lime-based plasters, are generally executed in two layers, thick for leveling and thin for finishing. For their application, it is necessary to moisten the wall and place them in a plastic state.

The term clay has a double meaning. On the one hand, it indicates the granulometric range of a fraction of the soil, which is made up of a material of natural origin formed by very small mineral particles. From a geological perspective, clays are considered to be particles smaller than 0,002 mm -2 μm-, and from an engineering perspective smaller than 0,005 mm -5 μm. On the other hand, it refers to phyllosilicates, a name assigned to a group of minerals within the silicate family. These minerals are hydrated aluminum silicates that are formed by the alteration of pre-existing minerals and are characterized by their layered structure that forms sheets. Clays can be divided into families based on their physicochemical properties and atomic structure. This structure depends on the arrangement of tetrahedral and octahedral layers that compose them, formed mainly by Si, Al, Fe, and O atoms in the first case or by Al, Mg, Fe, and O in the second (Christidis, 2011; Huggett, 2013). Species from 4 large groups are generally found in the soils used for construction: smectites (Sm), illites (Ill), kaolinite (K), and chlorites (Cl) (Delinière et al., 2014; Randazzo et al., 2016; Lima, Faria and Santos Silva., 2020; Lagouin et al., 2021a). The atomic structure of Sm consists of a sheet composed of two tetrahedral layers and an octahedral layer in the middle, plus an interlayer space that allows water atoms to be accommodated inside it. As a result of this, they have a high specific surface in the order of 700 to 800 m^2/g. Illites have the same structural unit as Sm, but are differentiated by their smaller interlaminar space that does not allow the entry of water atoms between the layers. This characteristic results in a significantly lower specific surface area, from 80 to 120 m^2/g. As for the Cl sheets, they are also composed of two tetrahedral layers and one octahedral layer, plus an octahedral layer in the interlaminar space strongly linked to the previous sheets. They have a specific surface area similar to that of Ill which is between 70 and 90 m^2/g. Lastly, K sheets are made up of a tetrahedral and an octahedral layer with no interlaminar space and only an external surface; this aspect means that they can have an even lower specific surface around 10 to 20 m^2/g (Van Olphen and Fripiat, 1979; Hugget, 2011). Another property that derives from the structure of clays is the cation exchange capacity (CEC), which is defined as a measure of the concentration of non-fixed cations in the interlaminar sites and on the surface of the clay sheets. The CEC of the soil refers to the number of cation exchange sites that it may have. A greater number of available exchange sites implies greater cation storage capacity and greater availability (Bueno Buelvas and Fernández Lizarazo, 2019). This property is directly related to the specific surface area, which is why Sm has a higher CEC than Ill and Cl, and these, in turn than K (Christidis, 2011). In this way, the properties of each soil such as its compressive strength, rheology, and its linear shrinkage are associated with this aspect (Delinière et al., 2014; Jia et al., 2024).

It is known that each family of clays presents distinctive mineralogical and chemical properties that are used in many different technological applications (Odom, 1984; Sheng et al., 2001; Ekosse, 2010; Muhammad Faheem et al., 2013), appreciating their adherent activity in the construction field. It is precisely the bonding property between clays, due to electrochemical charges, that allows the development of cohesion and adhesion properties of earthen materials (Van Olphen, 1979). In recent times, research has emerged that focuses on the properties of the clay fraction of soils and their mineralogical composition for their application in plasters (Lima et al., 2020; Delinière et al., 2014; Randazzo et al., 2016; Lagouin et al., 2021a). Regarding plaster adhesion, it is a property that the Argentine standard IRAM 1764 (2013) defines as the maximum tensile strength between a mortar and a defined support. For its part, Callister y Rethwisch (2014) and the *Instituto de Promoción Cerámica* (IPC, 2022) state that adhesion is a phenomenon that occurs when facing a system made up of two materials that are intended to be joined, called adherents, and a third material called a joint or adhesive bond. In the case of earthen plasters, the joint is made up of the internal face of the plaster mix (adherent). Adhesion can be mechanical or chemical. The first is a physical phenomenon and occurs due to a link between the component materials in the plasters and the supports, where the presence of pores and micropores on the surfaces favors the process. The second is produced by the formation of compounds and chemical interaction between molecules. In earthen plasters, both types of adhesion are combined: mechanical is made possible by the materials used and the shape and roughness of the surfaces, be they fibers, sand, or silt contained in the plaster or in the substrate, which provide a bond at the time of drying; the chemistry is given by the interaction between the materials of the substrate, the clay and other cementing materials contained in the plaster, which can react establishing unions during the drying process.

There are two types of tests for assessing adherence to earthen plasters: the pull-off test (breaking by tensile stress) for mortars and substrates standardized in British Standard EN 1015-12 (2000) and extended for earthen plasters in the Deutsche Standard DIN 18947 (2013), which establishes two categories based on the adhesive strength, class 1 ≥ 0.05 N/mm² and class 2 ≥ 0.10 N/mm²; and the shear test proposed by Hamard et al. (2013). Both tests try to evaluate the same adherent property of the plasters considering different efforts, but there is no consensus on the performance of one compared to the other. Some authors (Delinière et al., 2014; Lima et al., 2016; Lima et al., 2020; Faria et al., 2019) carried out pull-off tests following the British Standard EN 1015-12 (2000), to physically and mechanically characterize, and evaluate plasters with illitic soils to contribute to the performance and sustainability of buildings, find out the influence of mineralogy, and study the adhesive force on different substrates. For his part, García Villar (2022) carried out laboratory tests with rectangular prismatic samples to evaluate earthen plasters with bonding layers in experimental walls of light earth, while Quiñónez and Ayala (2014) also did so with rectangular samples but in prototypes of adobe walls, to understand the adhesive performance of plasters with different dosages. Regarding the shearing test, Stazi et al. (2016) applied the method proposed by Hamard et al. (2013) to study exterior protective plasters of earthen constructions, while Faria et al. (2019) modified the size of the samples in experimental laboratory tests carrying out tests on hollow bricks and adobes, establishing differences in the adhesive forces according to the type of previous paints and the samples were molded dry or wet; Cavicchioli et al. (2022) and Lagouin et al. (2021b), also carried out laboratory tests but with cylindrical samples and with improved instruments to eliminate the friction of the instruments on the substrates; the first with a chemical approach for the selection of soils for restoration, and the second in pursuit of understanding the effect of organic additives in earthen plaster. Both conclude, among several things, that the clay content and the mineralogy are key parameters in the physical and mechanical properties of the soils for plastering.

It is known that the heterogeneity of environmental and geomorphological factors that affect the formation of soils is high and variable, so it is common for them to contain a complex composition of minerals in their matrix, particularly in the types of clay present. In addition, each type of clay contained in the soil performs differently in the plaster mortars that are made with it. In this sense, this research aims to evaluate the incidence of clay mineralogy in the variability of the adherence of earthen plasters. For this research, samples of eight soils of the province of Tucumán (Argentina) from different quarries commonly used in construction were taken. These several soils are used intensively for construction, including their use in the production of earthen plaster for new works as well as in the restoration of existing and patrimonial architecture. The knowledge of the local builders has made it possible to identify the soil quarries necessary for their works, paying special attention to those that they consider to be more "loamy" or clayey.

2. Materials and methods

To evaluate the variability of the adherence of earthen plasters, considering that the mineralogy of the soils depends directly on the place and the formation processes, a comparative methodology was used through adherence tests of the eight selected soils. They were characterized by different analyses to determine their mineralogical composition and particle size distribution. To evaluate the adherence of mixtures made with the respective soils, it was proposed in the first instance to compensate for the particle size distribution of the soils to match them and, once the plasters were made, this property was evaluated through shearing and pull-off tests.

2.1. *Soil selection*

The physiography of the province of Tucumán (Argentina) can be divided into five general units. To the east, the relief is characterized by an extensive grassland that is divided into *Chaco* and depressed plains, while to the west, the mountainous relief and intermontane valleys predominate. Finally, there is the foothill plain that represents the transition between the previously mentioned units (Puchulu and Fernández, 2014). The soils studied are located mainly in these last three units.

The mountainous region presents soils developed on steep slopes and a rocky substrate almost on the surface (Puchulu and Fernández, 2014). Two subunits are differentiated: a) the soils of the humid mountainous sector, located on the eastern slopes of the mountain ranges, corresponding to sectors with high slopes protected by a jungle cover between 800 and 1,700 mamsl. The soils of this unit are characterized by being little evolved, of little depth conditioned by the slope of the place, with a dark superficial horizon rich in organic matter and, on some occasions, they develop horizons rich in clays; and b) the soils of the dry mountainous sector, located in the western mountainous region that develops between 1,700 and 3,500 mamsl. The typical vegetation of this sector is characterized by the presence of high-altitude grasslands that alternate with remnants of montane forest and shrublands. The subunit presents an arid moisture regime and the soils present abundant stoniness and thick accumulations of coarse sandy-stony sediments and a lower proportion of clayey material Fernández et al. (2008).

The intermontane valleys covered by the soil study include the Tafí and Santa María valleys. The soils of the Tafí Valley constitute soils developed on loess sediments (mainly made up of silt) and detrital deposited during the Pleistocene and Holocene. The Santa María Valley, located in the northwestern sector of the Tucumán province, is characterized by an environment strongly conditioned by the relief and a semi-arid climate with dominant xerophytic vegetation. The soils are generally of incipient development, shallow, without differentiation of horizons, with predominantly sandy textures, poorly structured, and with very homogeneous light colors (Puchulu and Fernández, 2014).

Finally, the foothill plain represents the transition between the mountainous area and the plain, it is distributed in a strip that extends parallel to the mountain relief. The soils are developed on the sediments produced by the weathering and erosion of the mountainous area deposited on the plain by an abrupt change in slope. The soils are characterized by being evolved, deep, and practically horizontal, with predominantly fine textures, that is silt and clay (Puchulu and Fernández, 2014).

The eight soils used in this work come from quarries usually used to extract construction material and correspond to three different physiographic units located to the northwest of the province of Tucumán: two soils in mountainous areas, one dry and one humid; five soils in areas of the intermontane valley sector: Santa María Valley and Tafí Valley; and a soil in a foothill plain area. The characteristic soils of each unit for the province of Tucumán, according to Puchulu and Fernandez (2014), and those selected for this study can be seen in Figure 1 and Table 1. The samples were identified as:

Table 1. Soils identification. The ID and the Unit ID correspond to the abbreviated names of the soils and the physiographic units respectively.

N°	Denomination	ID	Physiographic unit	Unit ID
1	Río Jorge	RJ	Intermontane valley (Valle de Santa María)	VISM
2	Barranca Lara	BL	Dry mountain	MS
3	Quilmes	QU	Intermontane valley (Valle de Santa María)	VISM
4	Amaicha del Valle	AV	Intermontane valley (Valle de Santa María)	VISM
5	San Miguel	SM	Piedmont plain	LLP
6	Cachamay	CA	Intermontane valley (Valle de Tafí)	VIT
7	Potrerillo Rincón	PR	Intermontane valley (Valle de Tafí)	VIT
8	Las Mesadas	LM	Humid mountain	MH

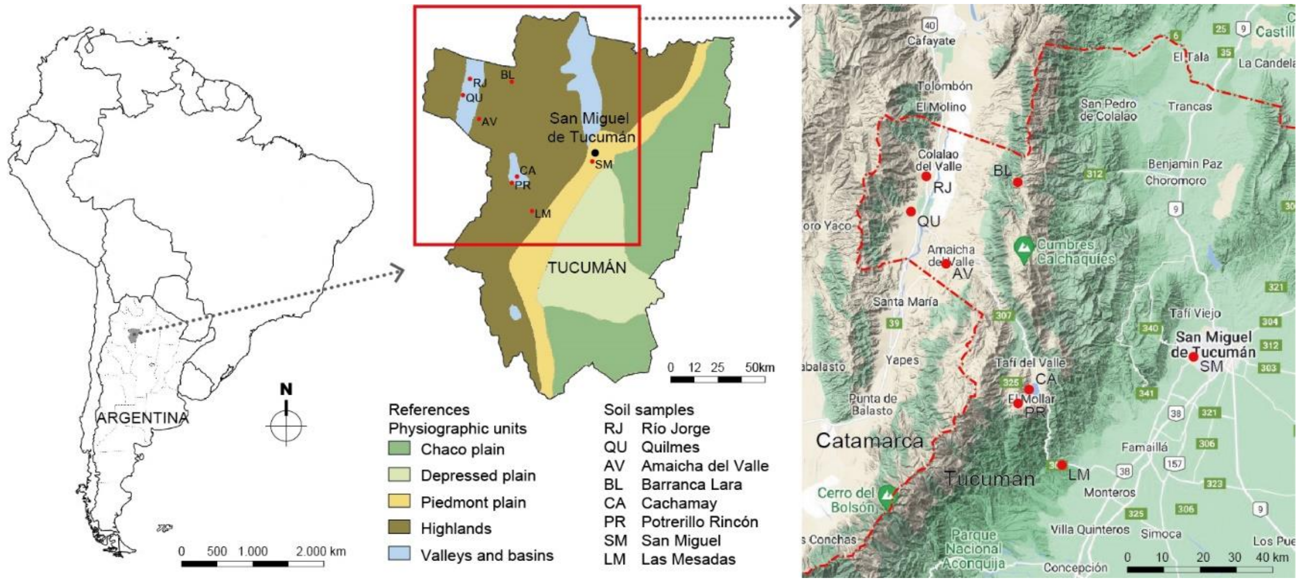

Figure 1. Satellite image extracted from Google Earth with the location of the sampled soils and soil map (modified from Puchulu and Fernandez, 2014).

2.2. *Characterization of soils*

The collected soils were characterized according to the following analyses:

2.2.1 XRD analysis

Samples of each soil were placed in milliRo water and sonicated for 15 minutes. Then, a dispersant (sodium metaphosphate) was added to each sample, shaken for 15 minutes, and, using the centrifugation procedure, the clay fraction was separated. Each fraction obtained was placed on two slides (original and duplicate) to obtain ordered aggregates (OA) and allowed to air dry. The original OA was analyzed by XRD in a PANalytical X'Pert Pro diffractometer at 40 kV and 40 mA with Cu-Kα radiation, between angles 4º and 35º 2θ (time per step 35,015 s and step 0,02º), for each sample, 3 diffractograms were made corresponding to 1) air-dried AO, 2) glycolate AO and 3) calcined AO. For the glycolate, the samples were placed in a desiccator with ethylene glycol and left in an oven at 50º for 12 h. On the other hand, calcining was carried out at 500ºC for 2 h.

Identification of minerals in the clay fraction: The study of clays consists of reading three diffractograms, which represent the sample under the three treatments before obtaining the spectrum. The different types of clays are identified according to their behavior against the mentioned treatments.

The diffractograms obtained were analyzed using the PANalytical X'Pert HighScorePlus 4.0-4.7ª program. Subsequently, based on the relative intensities (Moore and Reynolds, 1997). of the main reflections of the previously identified mineralogical components, they are semi-quantified.

2.2.2. Granulometric analysis

The particle size determination of the soils was determined by the hydrometric method (Bouyoucos, 1962). By sedimentation in a test tube, the distribution of particles less than 0.075mm -75µm- (silt and clay) was established using a hydrometer 152H; then, by sieving, the distribution of the largest particles (sand) was obtained according to the ASTM D-422 63 (2007) standard, after washing the sample on an N° 200 sieve. For this research, as established by the same standard mentioned above, particles less than or equal to 0,005 mm -5µm- are considered clays.

2.3. *Granulometric compensation*

To reduce the analysis variables in the comparison of the adherence between the soil samples, the percentage of the coarse fraction (sand) was equated using compensation with graduated sand. That is, the objective was to measure the adherent performance of the clay types of each soil, minimizing the incidence due to granulometric variability. Although the fine fraction of the soil, ≤ 0.075mm, is also composed of a small aggregate called silt, the separation of the clay fraction is not a simple method, which is why it was not considered in the proposed compensation.

To carry out the adjustment, the soil with the highest percentage of a sand fraction of the eight samples (RJ for this case) was started and the curves of the other soils were adjusted about it. This soil was taken as a reference because it had the highest sand content and, at the same time, the percentage of the clay fraction was within the ranges suggested in the bibliography as suitable for use in plasters (Minke, 2000; Stazi et al., 2016). To achieve a proportional equalization to the fine fraction (clay and silt) contained in the sample, the Thales theorem was used. A relationship factor was established between each soil and RJ by calculating a quotient between the fine fractions of both (1) for each case. Then the coarse fraction (sand) was compensated by considering separately the weight of the grains of each range of the same sieves used for the coarse fraction in the granulometric analysis (section 2.2.2, sieves N° 4, 10, 20, 50, 100, and 200) and multiplying the values by the factor obtained (2). In this way, the different sand ranges of the soils were equated to those corresponding to RJ.

$$\mathrm{F} = \frac{\mathrm{ff_n}}{\mathrm{ff_{RJ}}} \qquad (1)$$

$$\mathrm{X_{nT_n}} = \mathrm{Y_{nT_n}} * \mathrm{F} \qquad (2)$$

where:
F: Relationship factor between the fine fraction of soils
ff_RJ: Fine fraction of RJ (g)
ff_n: Fine fraction of n soil (g)
Y_n T_n: Fraction of n soil of n sieve (g)
X_n T_n: Compensatory fraction for n soil of n sieve (g)

Below is an example of obtaining the relationship factor between Sm and RJ, and Table 2 shows the compensation of the coarse fraction (sand) for each sieve.

F= (ff SM) / (ff RJ) = 45.45 g / 16.85 g = 2.697

Table 2. SM compensation example.

Sieve No,	Soil SM (g)	Soil RJ (g)	Soil RJ (g) * F (2,697)	SM equated to RJ
4	0.00	1.65	4.45	4.45
10	0.00	4.84	13.06	13.06
20	0.00	4.38	11.81	11.81
40	0.05	5.21	14.05	14.00
50	0.23	2.15	5.80	5.57
100	2.19	7.52	20.28	18.09
200	2.08	7.40	19.96	17.88

The sand used for compensation is commercial sand from the La Aguadita quarry (San Miguel de Tucumán).

2.4. *Plaster execution*

For plaster elaboration, 600 g of compensated soil was placed in a plastic tray and 30 ml of water was added to the eight cases to prepare the corresponding mortars. The amount of water incorporated had the objective of achieving plastic states and workability in the mortars. The decision to use the same amount of mixing water for all mortars was made to reduce the analysis variables, achieve even more similarity during preparation and considering that the compensated soils were remarkably similar to each other. Furthermore, the plasticity index through the Atterberg limits, which could provide the amount of water to achieve a plastic state in each of the soils, could not be achieved, mainly because all the soils, when compensated, were formed as sandy.

The moistened soils were mixed with a spatula for 2 minutes, the trays were covered and left to rest for 24 h at a constant temperature so that the clay fraction could be moistened. After the rest period, the back of a tile that presented a patterned and rough surface was used as a support for the plastering. The surface was previously treated with earth and water-based paint to condition it, and each paint corresponds to the earth of the plaster to be analyzed. Then, the mortars were manually mixed for one minute and placed in PVC molds 5 cm in diameter by 1,5 cm wide for the shear test, and 0.5 cm wide for the pull-off test arranged directly on the tile surface in a horizontal position. The molds were removed immediately and the samples were left to dry for 28 days in an environment with a temperature of 25°C and constant humidity.

2.5. *Pull-off test*

For this test, the methodology indicated in the British Standard EN 1015-12 (2000) with adjustments was applied. The instruments consisted of a 24 cm x 20 cm tile in which the renderings were made. For the execution of the test, the tile was placed vertically on a support, and a thin metal piece 5 cm in diameter was glued to the plaster samples with Sikadur 31 epoxy glue. The plate contains a hook to which a steel cable is attached, which goes through a pulley and ends in a container (figure 2, a1-3). Then, the test was carried out by incorporating batches of 300 g of sand into the container every 10 seconds until the sample was torn off. 5 samples per soil were tested and the mean value and standard deviation were estimated for each series.

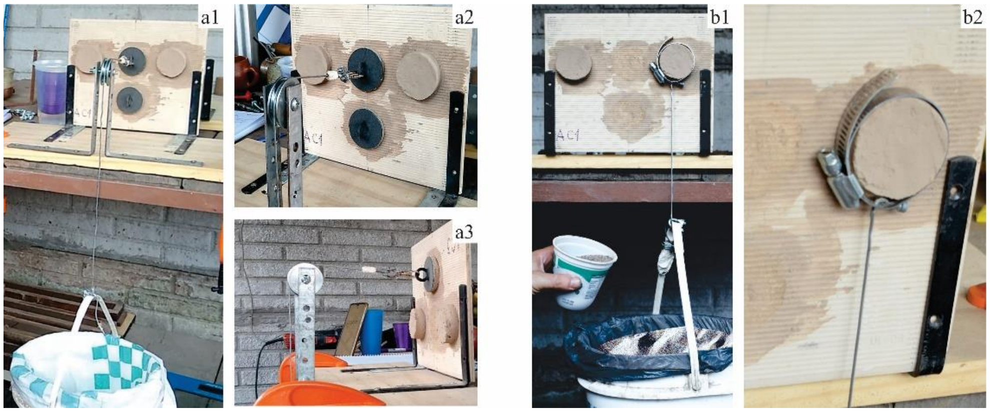

Figure 2. a1-3) Pull-off test instrumental; b1-2) Shear test instrumental.

2.6. *Shear test*

For this test, the methodology described by Hamard et al. (2013) was followed; modifications were incorporated into the device to reduce the forces of overturning and friction of the instrument on the wall or substrate. The instruments consist of a 24 cm x 20 cm tile to which the plasters are applied. To carry out the test, the tile was placed vertically on a support, a metal rack clamp on the plaster, from which a steel cable and a container were hung (Figure 2, b1-2). Then the test was carried out incorporating batches of 300 g of sand into the container every 10 seconds until the cut in the sample was produced. Once the rupture was produced, the total aggregate sand was weighed. 5 samples per soil were tested and the mean value and standard deviation were estimated for each series.

2.7 *Statistical treatment of the results*

The measurements of the series tested were analyzed using the RStudio software to verify the normality of the distributions using the Shapiro-Wilk test. In addition, the homogeneity of the variances was analyzed using the Levene test, and the ANOVA test of one factor was applied to verify if the differences were statistically significant; then, the post hoc test was applied using the Tukey test.

3. Experimental results and analysis

3.1 Identification and semi-quantification of clays by XRD

Through XRD, species of the four families of clays mentioned above were identified (Figure 3, table 3). After semi-quantification, it was observed that Ill clays are present and are the majority in all soils. In addition, in CA, BL, AV, and SM soils they are in proportions greater than 80%. Clays from the K family also make up all the soils, but in a range between 10 and 37%, with RJ and PR having the highest content. On the other hand, the identification of clays of the Sm group did not occur in all the soils. In RJ, QU, CA, BL, and SM it was observed in proportions equal to or less than 4% and 11% only for the LM soil. On the other hand, only two soils contain species of the Cl family, RJ with 2% and QU with 18%. In general terms, it can be noted that the mineralogical composition of the clays of the selected soils follows the same pattern of the predominance of the Ill / K pair.

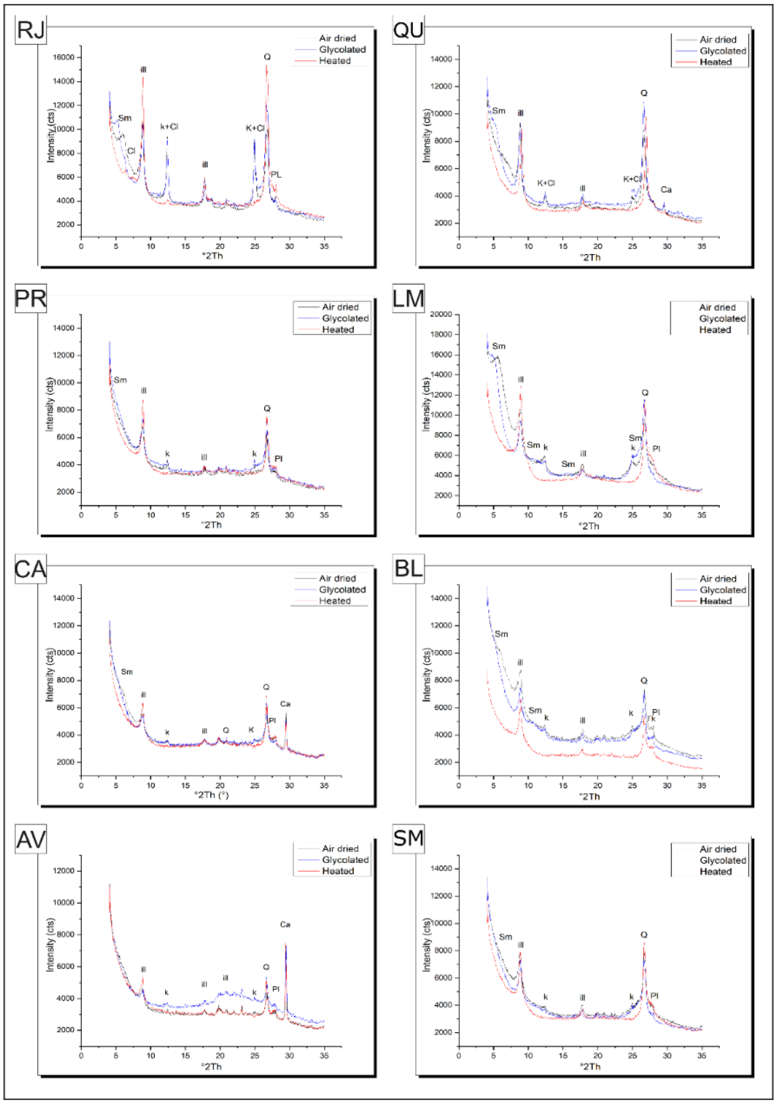

Figure 3. XRD diagrams of each of the samples.

Table 3. Identification and semi-quantification of clays.

Soil	Clays type (%)			
	Illite-Mica (Ill)	Smectite (Sm)	Kaolinite (K)	Chlorite (Cl)
RJ	58	3	37	2
QU	61	4	17	18
PR	69	0	31	0
LM	69	11	20	0
CA	83	2	15	0
BL	83	3	14	0
AV	85	0	15	0
SM	87	3	10	0

3.2 Granulometric analysis and soil compensation

The granulometric analysis showed important variability between the selected soils (Figure 4 above). The greatest range of variability occurred in the sand fraction, then in the silt, and finally in the clay (Table 4). Considering the USDA classification, silt loam soils are the most represented (4), the rest are loam soils (2), silt clay loam (1), and sandy loam (1). After compensating the soils with the addition of sand, the granulometric curves of the sand fraction were equated, nullifying the existing variability, and the dispersion of the percentages of both the silt fraction and the clay fraction was significantly reduced to 8.03% in both cases (figure 4 below). All the soils were modified to sandy loam (figure 5).

The percentages of the compensated clay fraction were adjusted to values equal to or less than 12%. However, since the total percentage of clay in the soil is central to the adherence of earthen plasters, it was sought to limit the incidence itself in the comparative analyses as much as possible, grouping the soils according to the greatest similarity. In this way, it was proposed for the analysis to separate the compensated soils into two groups considering similar silt/clay ratios: on the one hand there are SM, RJ, BL, PR, L, M, and QU, which presented a ratio of 2 (except QU with 3), within a range of 4,22 of variability in the clay content; and on the other hand, AV and CA with a ratio of 7 within a variability range of 0,44.

Table 4. Percentages for each original and adjusted soil fraction. Data in descending order according to compensated clay content.

Soils ID	Unit ID	Sand (%)		Silt (%)		Clay (%)		Silt / Clay (entire)
		Original	Comp.	Original	Comp.	Original	Comp.	
SM	LLP	9.10	66.30	58.49	21.68	32.41	12.02	2
RJ	VISM	66.30	66.30	21.80	21.80	11.90	11.90	2
BL	MS	51.66	66.30	32.38	22.57	15.96	11.13	2
PR	VIT	18.98	66.30	55.46	23.07	25.56	10.63	2
LM	MH	36.66	66.30	43.41	23.10	19.93	10.60	2
QU	VISM	20.24	66.30	61.29	25.90	18.47	7.80	3
AV	VISM	26.26	66.30	64.04	29.27	9.70	4.43	7
CA	VIT	37.68	66.30	54.93	29.71	7.39	3.99	7
Range		57.20	0.00	42.24	8.03	25.02	8.03	-

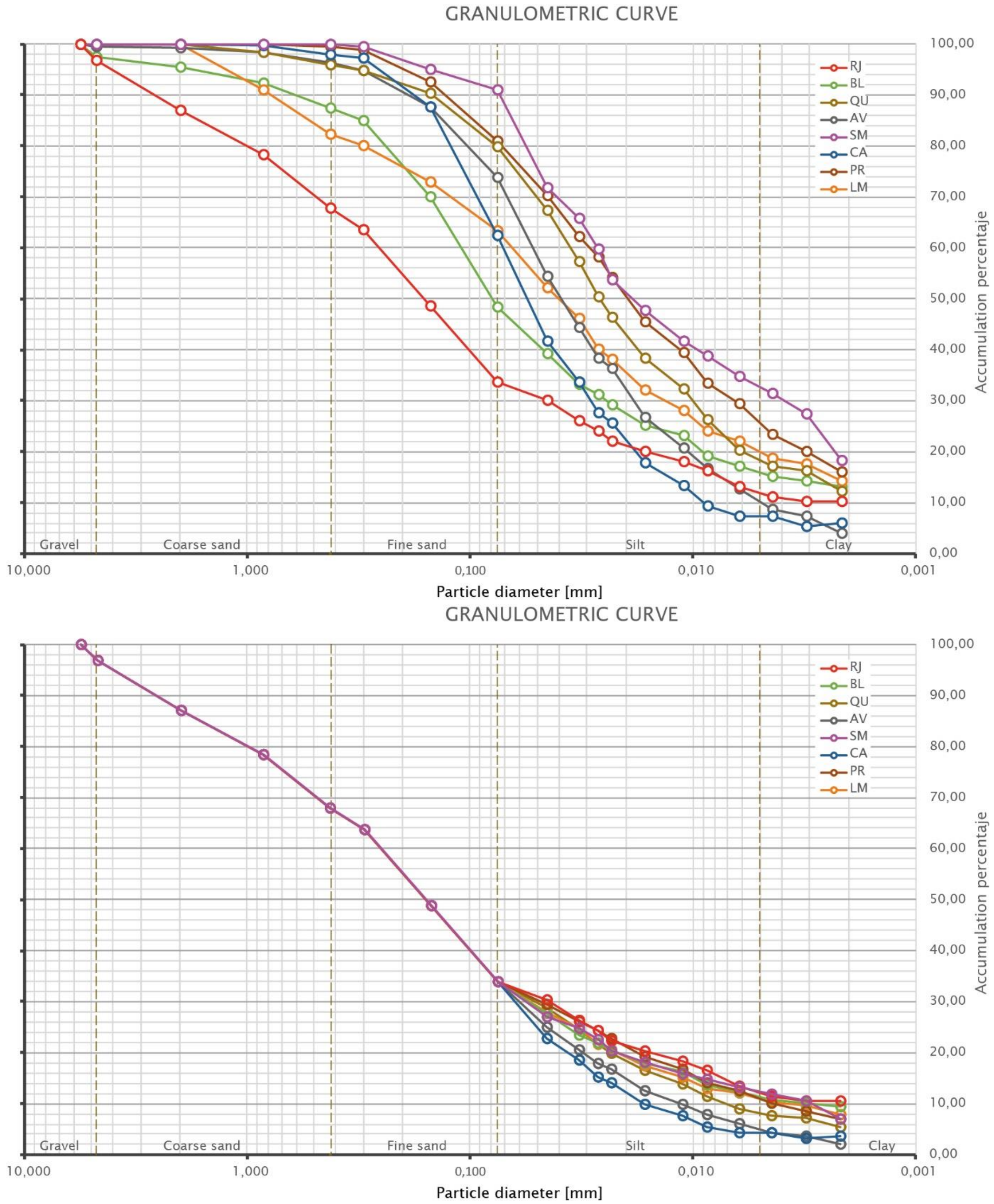

Figure 4. Granulometric curves of the eight original (upper) and compensated (lower) soils.

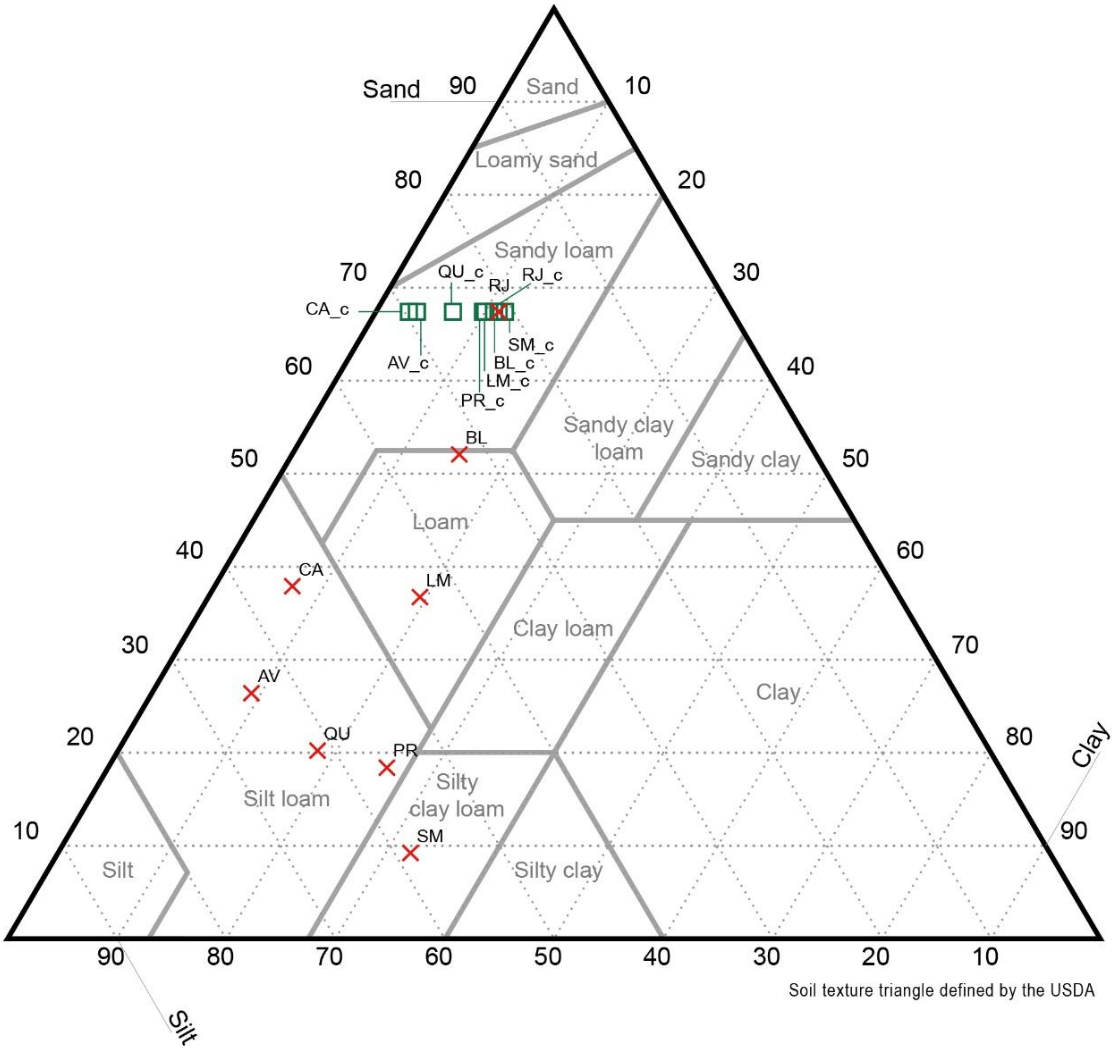

Figure 5. Distribution of original soil (red cross) and compensated (green square) according to the classification defined by the United States Department of Agriculture.

3.3 *Adhesion tests*

The results of the pull-off and shearing tests are shown in Table 5. The values obtained present a normal distribution of the data, except for the CA plaster sample, which presents an atypical value. In a general analysis of the two tests, the differences are statistically significant between the results, however, the significance is greater in shearing.

The plaster samples with the highest adherence were SM, LM, and BL for the two types of tests, with values ≥0.05 N/mm^2 for the direct tear test, and ≥0.11 N/mm^2 for the shear test. In both cases, they verify the requirements of the DIN 18947 standard for earthen plasters, on adherence for class 1. Although these three soils tend to present the highest values of standard deviations, they do not have statistically significant differences among themselves, but they do with the others. five soils, especially in shearing.

Table 5. Results of the pull-off and shear tests. 1 N/mm^2 = 10 kgf/cm^2. Data in descending order according to pull-off test, shearing, and silt/clay ratio.

Soils ID	Unit ID	[1] Pull-off test(N/mm2)	Standard deviation	[2] Shear test (N/mm^2)	Standard deviation	Magnitude ratio [2]/[1]	% Compensated silt	% Compensated clay	Silt / Clay (entire)	Clay type (%)			
										Ill	Sm	K	Cl
SM	LLP	0.06	0.016	0.15	0.017	2.50	21.68	12.02	2	87	3	10	0
LM	MH	0.06	0.005	0.13	0.031	2.17	23.10	10.60	2	69	11	20	0
BL	MS	0.05	0.033	0.11	0.044	2.20	22.57	11.13	2	83	3	14	0
PR	VIT	0.04	0.005	0.06	0.007	1.50	23.07	10.63	2	69	0	31	0
QU	VISM	0.03	0.007	0.07	0.022	2.33	25.90	7.80	3	61	4	17	18
RJ	VISM	0.03	0.007	0.06	0.028	2.00	21.80	11.90	2	58	3	37	2
			Range				4.22	4.22					
AV	VISM	0.03	0.009	0.08	0.014	2.67	29.27	4.43	7	85	0	15	0
CA	VIT	0.03	0.013	0.05	0.027	1.67	29.71	3.99	7	83	2	15	0
			Range				0.44	0.44					

3.4 *Comparative analysis of mineralogy and adherence*

When comparing the results of the mineralogy of the soils with the adhesive force values obtained from the plaster samples (Table 5), the following aspects could be observed:

- There is a first group of soils that acquired high adherence. The SM soil, corresponding to the piedmont physiographic unit, made it possible to obtain the plaster sample that presented the highest adhesive strength for the two types of tests. It should be noted that this soil contains the highest percentage of Ill clay minerals of the entire series and, at the same time, the lowest percentage of K, with a small percentage of Sm. For its part, the LM soil belongs to the humid mountainous zone and is part of the plaster samples that also present high values of adhesive force. The mineralogical composition of clays in the soil does not contain an amount of Ill as in SM, to the extent that it is partially replaced by clays of the K and Sm groups, the latter in 11%, the highest value in the series. The BL soil, corresponding to a dry mountainous area, is the third soil that completes the group that provided the plaster samples with the highest values of adhesive strength. It turned out very similar to SM, but with a slight imbalance of Ill clays in favor of K.
- In the case of RJ and QU soils, from the Santa María Valley, and PR, from the Tafí Valle, they presented intermediate adherence values in the series. This condition is probably due to a smaller content of group Ill clays in the entire series. In the case of RJ and PR, Ill are fundamentally replaced by K clays, with the highest values of the series, above 30%. In the case of QU, with a silt/clay ratio of 3, this replacement occurs evenly between K and Cl clays.
- If the results of adhesive strength of these plaster samples from this commented group are compared with the Ill content, a positive correlation is observed between both values of 77.9% in the pull-off test and 78% in the shear test.
- The plasters sample AV and CA, from the Valle de Santa María and Valle de Tafí physiographic units, respectively, although they have proportions of the Ill/K pattern similar to the samples with the highest bond strength, the data indicate that their lower performance adherent is attributed to the lower percentage of clay present in the soil, around 7 in the silt/clay ratio.

4. Discussion

The main objective of the study was to determine how clay mineralogy affects the adherence of earthen plasters. For this, it was necessary to make comparisons between different soils that presented an important variability in their granulometric composition, especially in the clay fraction. For this reason, a simple granulometric compensation procedure applied to the coarse fraction was developed, which allowed adjusting the soils in terms of the composition of the sand, silt, and clay fractions in such a way as to reduce as much as possible the variability of the analyzed set. The procedure made it possible to cancel out the differences in the coarse fraction (sand) with the controlled addition of additional sand and, as a consequence, to resemble the percentages of the fine fractions (silt and clay). As a result, two groups of well-marked soils were obtained: 6 soils that presented a silt/clay ratio with an index between 2 and 3, and a range of variability of the clay fraction of 4,22%, and 2 soils that presented an index of 7 with a range of 0,44%. Until now, the different studies on plaster performance did not require applying this granulometric correction to the original soils, before the preparation of test samples, because the nature of the objective did not demand it (Hamard et al., 2013; Emiroğlu et al., 2015), the original soils are already similar (Delinière, 2014), or clay minerals were studied without correcting for the clay fraction in a larger study (Lagouin et al., 2021b).

Regarding the results obtained, the main aspect observed was that the family of phyllosilicates corresponding to Ill is the dominant group in all the soils analyzed with values greater than half the percentage in their composition. These values are accompanied in all cases by a lower and variable percentage of clay minerals in the K group. This Ill / K composition pattern seems to be common for the study area considered and is characterized by being minerals that do not produce swelling with the incidence of water, which favors its use in construction; Fratini et al. (2011), as Emiroğlu et al. (2015), also report a similar relationship of these minerals in studies for sites in Italy and Turkey respectively, however, it is clear that the pattern may change in other regions as reported by other authors (Costa et al., 2013; Duarte et al., 2015; Randazzo et al., 2016; Cavicchioli et al., 2022; Lagouin et al., 2021a).

Directly linked to this aspect, a positive correlation was observed between the percentage of Ill in the soils and the adhesive force values of the plaster samples. In terms of clay mineralogy, the adherent force in the earthen plasters tends to be higher with the increase in the percentage amount of Ill for samples with similar percentages of the clay fraction. This correlation seems to be explained, in part, because the Ill group clays, although they have a specific surface area and CEC that is lower than the Sm group, are still higher than the K and Cl group of clays (Meimaroglou and Mouzakis, 2019). This condition confirms its ability to provide better adherent performance in earthen plaster, an issue also indicated in a general way in the investigation by Lima et al. (2020) regarding the mineralogy of clays and its influence on different aspects of earthen plaster. Houben and Guillaud (2008) suggest that kaolinitic soils are preferable for earthen plaster, however, from the results of this investigation, illitic soils would perform better. Due to the scarcity of data to reinforce this affirmation, new research would be necessary.

Following this line of analysis, the presence of clays from the Sm group complementing the Ill ones is not very high percentages (around 11% for this study) help to improve adherent properties –in addition to cohesive ones, as suggested by Lagouin and colleagues (2021a)– of the plaster samples, this issue is observed in the performance of the LM soil. The greater specific surface that Sm has and the high CEC available to this class of clay seem to be one of the factors linked to this issue (Mitchell and Soga, 2005). Sm may act favorably only in moderate or low presence ranges because, it is known in the field of earthen construction, that soils with high contents of expansive clay minerals end up being harmful due to the volumetric instability that they present due to swelling against the presence of water (Guerrero Baca, 2007; Elert, 2014; Elert et al., 2022).

Considering the other types of clay identified, K and Cl, it was observed that as their percentage content in the soils increased, the values of adhesive forces of the plaster samples decreased for both tests carried out. For the data obtained, this aspect is notoriously observed when the percentage of K clays or the sum of K and Cl is above 30%. Inversely to what occurs with Sm, the lower specific surface that K and Cl have and the low CEC available to this class of clays explain the lower adherence of the plaster samples as their presence increases.

The samples in this study were not subjected to compressive and flexural stress tests as proposed in DIN 18.947 (2013) and as observed in various current studies on earthen plaster because the objective was to evaluate the adhesive force induced by the clay minerals and not the cohesion of the material. On the other hand, Faria et al. (2016) point out that a render without high compressive strength can perform well in its adhesive strength. However, the three plaster samples with the best performance verify category 1 of DIN 18.947 (2013) in both tests.

Another aspect that could be analyzed was the relationship between the mineralogy of the clays and the percentage of clay fraction in each soil. This results from comparing such characteristics between the two established groups (6 and 2). The AV and CA soils presented contents of the Ill/K pattern similar to the SM and BL soils, however, the adhesive force values obtained in the plaster samples of the former are among the lowest values in the series. This is explained by the lower percentage content of the clay fraction of the former compared to the latter. This is certainly an expected result given that several studies have already pointed out the important incidence of the clay fraction in the cohesive performance of plastering materials. Hamard et al. (2013) consider that percentages of 9% in the clay fraction achieve optimum adhesive strength in renders without generating cracks and that the adhesive strength doubles about percentages of 6%. However, with values greater than 12%, the adhesive forces begin to decrease due to shrinkage and detachment; coinciding, Minke (2000) indicates an optimal range of 5 to 12% –range of the first group of plaster samples in this study– while Stazi et al. (2016) optimize the conditions and reach higher values with 16% clay fraction. The least adherent soils turned out to be precisely those that contained around 4% clay.

A significant finding of the research is the confirmation of a high correlation between the results obtained in the two adherence evaluations carried out. It was observed that shear tests showed superior performance than pull-off tests, with a difference ranging between 1.50 times greater (in the case of PR) and 2.67 times greater (in the case of AV). This notable difference can be attributed to the intrinsic properties of the material against different types of stress. When the material is subjected to shear stresses, it responds by activating its internal cohesion and friction forces to counteract slippage and maintain its structural integrity. On the other hand, during tensile tests, the applied forces tend to separate the particles of the material, acting perpendicular to the contact surfaces between them. In this scenario, friction plays a secondary role, which explains the lower resistance observed compared to the shear tests. In particular, the case of AV, which shows the greatest disparity in terms of resistance between shear and tensile, suggests that the friction generated by the greater amount and specific nature of the inert materials present (sand and silt) could be a determining factor in the cut resistance values obtained. However, this hypothesis requires further investigations for confirmation.

Lastly, about the physiographic units, the formation conditions of the soils in each one of them do not give indications of having an impact on the percentage variation of the Ill / K (majority) pattern, at least for this study, but on the clay content of the respective soils. Thus, after compensation, the SM, LM, and BL soils from piedmont plain, humid, and dry mountain physiographic units respectively showed the best adherent performances, coincidentally with high values of clay content in the series. Only the QU and RJ soils correspond to the physiographic unit of intermontane valleys, particularly the Santa María Valley, present Cl, so this mineral is scarce in the study area. The Sm was observed to be present in all the physiographic units studied but a prominent presence in the humid mountain.

5. Conclusions and comments

1. This research allows us to conclude that the mineralogy of the clays influences the variability of the adherence of earthen plasters, increasing or decreasing it according to the composition patterns, and that this can be decisive between soils with similar percentage contents of the clay fraction. When this condition does not occur, the percentage value of the clay fraction present in the soil better explains the adherent performance; however, this last aspect was not central to the study and requires further research to obtain greater precision;
2. For this study, an *ad hoc* compensation method was tested to be able to analyze the effect of clay minerals by restricting the incidence of particle size composition in the analysis. This proposed method provided a way to homogenize the granulometric curves of soils, allowing comparisons to be established between them with an added degree of similarity, an issue that has not yet been addressed in other research. He is pending to apply this method to new data sets to evaluate its performance and refine it;
3. In particular, it was observed that the Ill / K composition pattern can be common to different physiographic units of a large geographical area and, in terms of its application for plastering, it is positively correlated with the increase in adherence as the percentage content of Ill increases in the pattern. Thus, soils with the presence of Ill are preferable for use in earthen plaster, especially if it is the dominant mineral in the mineralogical composition. In addition, the presence of Sm in a sufficient but not abundant quantity (11% for this study) favors an increase in adherent strength

without adding retraction forces. In contrast, the clay minerals K and Cl do not improve the adhesive strength of plastering mortars. Future studies with soils that contain other mineralogical composition patterns that include Sm in particular are necessary to know the adequate ranges of this clay mineral for use in construction.

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Rolón carried out tests, analyzed the results, worked on the discussion, finally corrected the article, and edited Figure 1.

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