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Stabilization of earthen plasters: Exchange of knowledge and experiences between Italy and Morocco

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Abstract
The preservation of earthen architectural heritage and earthen contemporary architecture asks for the experimentation and the development of proper materials and intervention techniques in order to prevent decays which may compromise the conservation of earthen heritage over time. This research program, thanks to a bilateral cooperation between the CNRST-UCA and CNR-ICVBC, aims to design and test earth-based plasters stabilized with traditional products (gypsum or lime), in order to acquire information necessary to develop more durable earthen mixtures to be applied both on existing and new buildings. The experimental campaign intends to assess the capacity of these stabilized plasters to guarantee an appropriate protection of earthen walls. The plasters have been tested at first in laboratory, then they have been applied on an earthen building in the outskirts of Marrakesh. The performances of the on site plasters are still under study and will not be presented in this paper.

Keywords: Earthen plasters, Stabilization, Traditional products, Lime, Gypsum

Introduction

Widely spread in many European, American, Asian and African countries, earth structures bear important and significant witness of knowledge, building technics, technological culture that holds unique landscape values besides historical ones (Jaquin, 2008; Guillaud, 2012). Ecologic material par excellence, earth had been used since antique times to build architectural structures; then it went into a period of complete oblivion as a consequence of the wide diffusion of industrial products. Recently, however, its use has become the object of renewed interest not only for developing countries, but also for a new emerging market. In fact earth is widely available in nature at an extremely low price; it is used without the necessity of special transformations; it requires a reduced energy amount for its processing and allows to work with a minimum impact on the environment; it gives the opportunity to build high living comfort structures, thanks to its breathability and thermal insulation. The only drawback of these architectures is the limited durability of the material against the aggressive action exerted by external agents (such as rainwater, capillary rise, erosion, wind, anthropic action) that makes it necessary a constant maintenance (Guettala et al., 2006; Avrami et al., 2008; Boussalh , 2005). In order to mitigate the decay of the earthen structures, the choice of appropriate materials and techniques is required, thus allowing the preservation of the earthen architectural heritage and promoting the use of earth in modern architecture. This choice should be dictated by the knowledge gained through retrieval and study of the traditional building techniques together with new experimentation. To this purpose a bilateral research project between Moroccan CNRST (Department of Geology of the University of Cadi Ayyad, Marrakesh) and Italian CNR (Institute for Conservation and Promotion of Cultural Heritage, Florence), has been developed in order to experiment and develop different types of earthen plasters through the evaluation of their performances both in laboratory and in situ. Although the experimental activities conducted in recent years by the Italian research group have been designed to test plaster made both with traditional stabilizers and with less common products (cement kiln dust, enzymes, geopolymeric solution), in this case it was considered appropriate to perform the experimentation making use exclusively of materials easily available on the Moroccan territory (such as lime and gypsum).

2. Materials and methods

All the plasters have been made using an earth coming from Douar Oulad Mezzoug, 22 km East of Marrakesh, along the road to Sidi Rahal. The earth was taken from a trench 2.20 m deep. The facies encountered in the trench are the following:

- on the surface a topsoil layer, about 0.20 m thick;
- more or less compact clayey silt up to 2.20 m depth.

The earth has been taken excluding the topsoil layer. The bulk density of the earth is 1.88 KN/m^3 and the water content is 8.8 %. According to the Atterberg limits (WL=31% and IP=11 %) and to the grain size analysis (Table 1) the material can be classified as silty sand [SL] [classification of the central laboratory of Ponts et Chaussées (LCPC)].

Table 1: Granulometry

Granulometry through sieving [NF P 94 056]						
Sieve ϕ (mm)	*>50*	*50-5*	*5-2*	*2-0.63*	*0.63-0.08*	*< 0.08*
% of grain fractions	12	8.2	18.2	28.9	20.2	12.5

Table 2 shows the composition of the different plaster mixtures tested. The additives' percentages refers to the weight of the dry earth. The samples were prepared by providing a first layer (approximate 1.5 cm thick) using earth with coarser particle size, subsequently coated by a finishing layer (approx. 0.5 cm thick) made of an earth with a smaller granulometry (passing through a Ø 1 mesh sieve).

Table 2: Composition of the plaster mixtures

Plaster	***First layer***			***Finishing layer***		
	Earth (g)	***Gypsum (g)***	***Lime (g)***	***Earth (g)***	***Gypsum (g)***	***Lime (g)***
A	7300	-	-	2600	-	-
B	7300	-	1095*	2600	-	390*
C	7300	1460**	-	2600	520**	-

*=15% w/w of the earth; **20% w/w of the earth

Other samples (A1, B1, C1) were prepared adopting the same plaster mixtures but adding a little quantity of straw (four handfuls of straw in the first layer and one handful in the finishing layer).
Three samples have been realized for each type of plaster and their performances and characteristics have been compared with those resulting from tests conducted on the samples of the non-stabilized earthen plaster, commonly used in the traditional architecture.
The following kind of samples have been realized to carry out the different tests: 25x25x2cm for the erosion and capillary absorption tests; 5x5x2cm for the colour measurements and water vapour permeability test; 5x4x2cm for the shear test.

2.1. Investigation techniques

The following investigation techniques were used to assess the composition of raw materials and performances of the stabilized earthen plasters.
The composition of the starting materials (lime, gypsum, earth) and of the new phases formed in the plaster earth + lime and earth + gypsum, have been investigated by X ray diffraction analysis with a X'Pert PRO diffractometer by PANalytical equipped with X'Celerator detector and HighScore software for acquisition and interpretation of the data (Cu Kα1= 1.545 Å radiation, 40 KV, 30 mA, 2Θ = 3-70° and 2Θ = 3-35°for clay minerals).
A deeper study has been carried out in the case of the plaster made of earth + lime to identify possible presence of pozzalanic reaction products (hydraulic phases) between lime and the clay minerals of the earth, utilizing the following investigation methods: infrared spectrometry analysis with Bruker Optics ALPHA Spectrometer in the mid-infrared region (4000-400cm^{-1}). Spectra were acquired in total reflection mode collecting 16 scans, with 4 cm^{-1} resolution on KBr pellet; thermogravimetric analysis with a Pyris 6 TGA by Perkin Elmer equipped with

Pyris TGA software for acquisition and elaboration of the data. The sample (3-4 mg) was placed in an ceramic crucible under N2 gas atmosphere with a flow rate of 20 mL/min and analysed from 50°C up to 900ºC at a rate of 10ºC/min.
The durability performances of the earthen plasters have been investigated through: water erosion test (Geelong and spray test); water absorption tests (Karsten pipe); shear test; permeability test; colorimetric test.
The erosion tests were conducted, according to New Zealand Standards NZD 4298, in two different ways: Geelong test (NZS 4298E, 1998) and spray test (NZS 4298D, 1998).
The Geelong test is based on the measurement of the samples' erosion caused by the repeated impact of a water drop(from an height of 400 mm and for a total of 100 ml) on the tested surface which is placed at an angle of nearly 30° to the horizontal (Figure 1). The evaluation is done by measuring the cavity depth due to the impact of the water drop on the surface.

Figure 1: The Geelong test (Manuela Mattone).

The erosion spray test involves the measurement of the erosion of the sample caused by exposition to a water jet projected from a distance of 470 mm and with a pressure of 0.5 bar. The test lasts up to one hour, or until complete erosion of the sample, and it is interrupted at regular intervals of 15 minutes to evaluate the depth of the erosion caused by the water jet.
The water absorption tests have been carried out using the Karsten pipe (Karsten, 1983) and measuring, for a maximum of 15 minutes and at regular intervals of one minute, the rate of water absorbed by the plaster from the graduated pipe.
In order to evaluate the bond of the plasters to the wall, shear tests were conducted according to what suggested in recent researches (Hamard et al., 2013). Three specimens (size 40x50x20 mm) of each kind of plaster were prepared. The shear test measures the load necessary to make the specimen fail. Each specimen was loaded with little lead balls until fail, using a special device (Figure 2). The load at which the sample fails was recorded. It was possible to evaluate the average shear stress (τ, N/mm^2) of each plaster considering the failure load (m_f, Kg) and the surface (S, mm^2) of the samples since$\tau = m_f \times g/S$, where g = 9.81 m/s^2.
The water vapour permeability test was carried out according to the procedure and instrumentation contained in the European standard EN15803 (2010). The permeability is expressed as the Residual Permeability in percent. The tests have been carried out on 3 specimens for each type of plaster mixture.
The colour variations induced on the earthen plasters with and without additives is a purely aesthetic parameter that may be interesting to know in case integration of missing parts of plaster becomes necessary. The Konica Minolta spectrophotometer CM 700d adopting the CIE L* a* b* method (CIELAB, 1976), where L* is the lightness, a* is the red- green axis and b*is yellow-blue axis, has been utilized. The total change of colour is summarized by ΔE^* ($\Delta E^*=5$ is the detection limit of human eye) (EN15886: 2010). For each type of earthen plaster (non-stabilized and stabilized), a total of 30 measures were carried out.

Figure 2: The shear test device (Manuela Mattone).

3. Results and discussion

3.1. Composition of raw material and identification of new phases in stabilized plasters

The composition of the Moroccan starting materials carried out by XRD analysis is described in Table 3 and Figure 3.

Table 3: Mineralogical composition of the Moroccan starting materials*

Material	***Composition***
Earth	quartz, feldspars, traces of iron oxides and clay minerals (illite 45%, kaolinite 30%, chlorite 25%)
Gypsum	calcium sulphate hemihydrate (bassanite), calcium sulphate (anhydrite), traces of carbonates (calcite and dolomite) and quartz.
Lime	calcium hydroxide (portlandite), β-calcium silicate (larnite), traces of calcite and quartz

*concerning clay minerals the data are semiquantitative

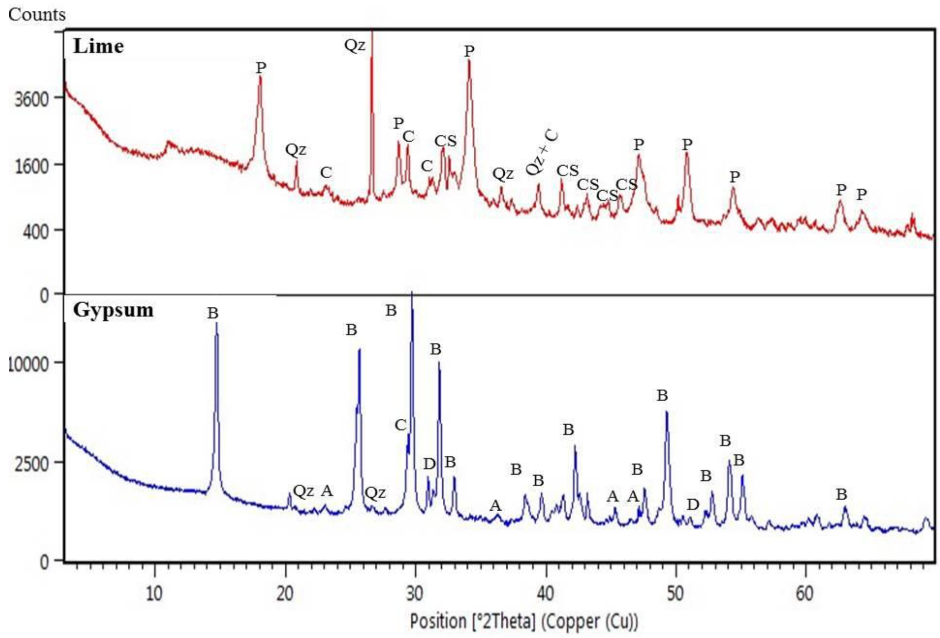

Figure 3: XRD spectra of Moroccan lime and gypsum. Calcium silicate (larnite) is a typical compound of hydraulic lime (P=portlandite; CS=calcium silicate; B= bassanite; A= anhydrite; Q=quartz, C=calcite, D=dolomite).

The mineralogical investigation made it possible to verify that the gypsum can be ascribed to a “soluble anidrite” while the lime is a natural hydraulic lime due to the presence of β-calcium silicate (larnite).
After 28 days of curing, as new mineralogical phases, we have evidenced the presence of calcite in earthen plaster stabilized with lime and presence of gypsum in earthen plaster stabilized with gypsum (Fig.4).
Hydraulic phases due to hydration of larnite (CS) or pozzolanic reaction between calcium hydroxide (portlandite) with clay minerals have not been identified (may be due to amorphous state or low crystallinity).

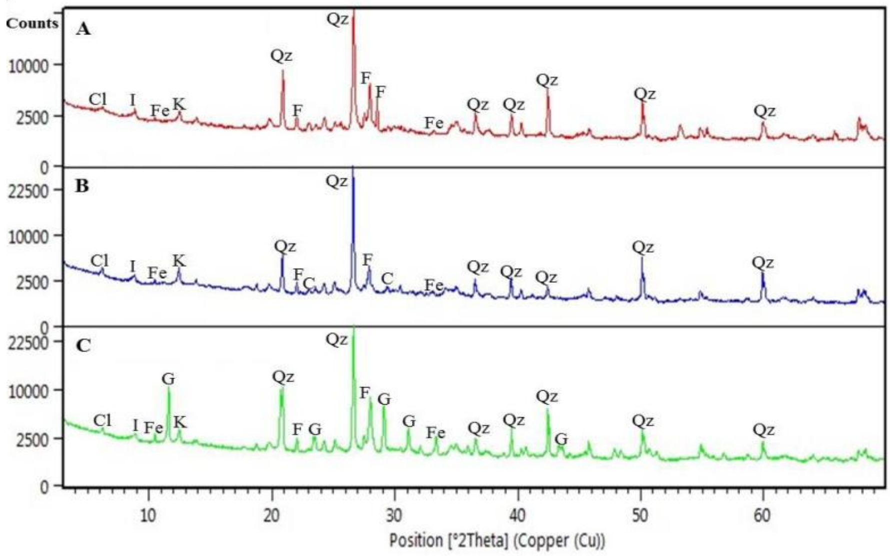

Figure 4: Comparison among XRD spectra of earthen plaster made with only earth (A) earth + lime (B) and earth + gypsum (C). Qz=quartz, F=feldspars, C=calcite, D=dolomite, P=portlandite, I=illite, K=kaolinite, Cl=chlorite; G= gypsum, Fe= iron oxides.

In Figure 5 the results of mid –IR investigation carried out on earth - lime samples (graph A) compared to those obtained from the earth ones (graph B), are shown.

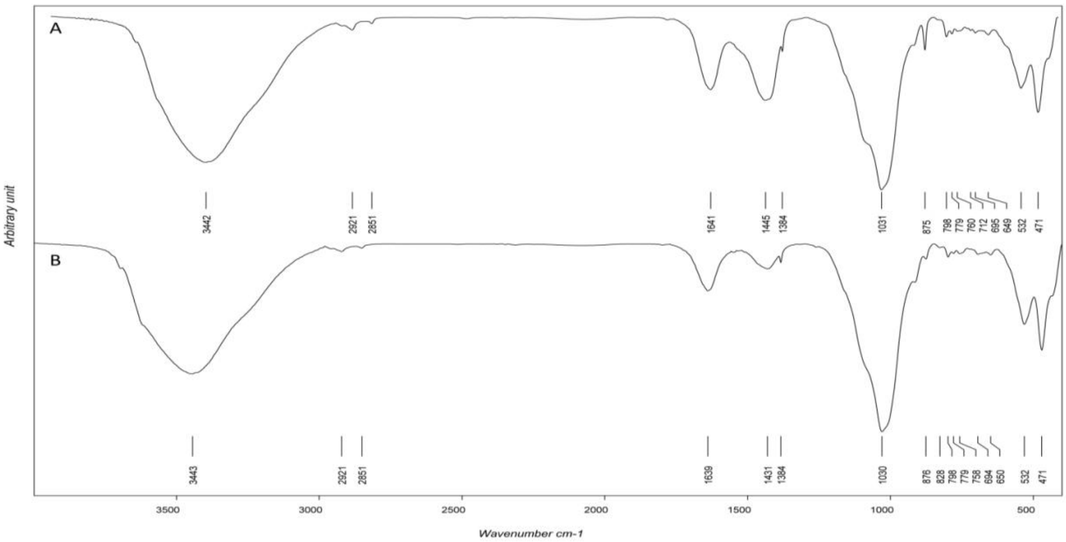

Figure 5: Mid –IR spectra: comparison between sample of earth with lime (graph A) and sample of only earth (graph B).

The spectra are very similar and IR bands are comparable. In particular, in both spectra, the following peaks are present: symmetric and asymmetric stretching of Si–O (800-1000cm^{-1}, silicates) and, specifically, a high peak at 1030cm^{-1}attributable to stretching of Si-Al-O (alumin̆cosilicates); deformation of SiO_4 tetrahedron (400–500cm^{-1}, silicates) (Hanke, 1986); asymmetric stretching of CO_3^{2-} (1400–1500cm^{-1}, carbonates) and a shoulder

of bending out-of-plane of CO_3^{2-} ion (875cm^{-1}, carbonates) (Hunt 1971); bending H-O-H of molecular H_2O (1640cm^{-1}) and –OH stretching vibration of H_2O (2800–3700cm^{-1}) (Williams, 1966). It should be noted that, the main vibrations characteristics of C-S-H compounds (peak at ~970cm^{-1} and two shoulders at ~1060cm^{-1} and 900cm^{-1}), related to Si–O stretching vibrations of the Q2 tetrahedra (Trezza, 2007, Yu, 1999), are not visible (probably they are hidden by the broad band of silicates at 800-1000 cm^{-1}).
The result of the thermal analysis carried out on the sample of earth - lime plaster (B) is shown in Figure 6.

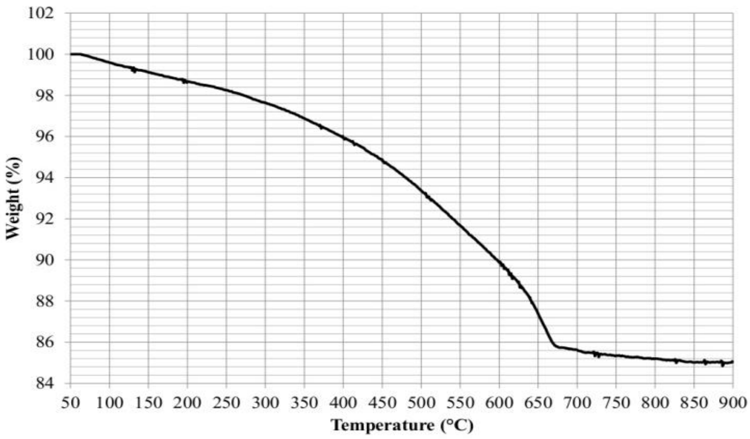

Figure 6: Thermogravimetric curve of sample of earth with lime (plaster B).

The thermogravimetric curve (Figure 6) indicates that the dehydration of the earth + lime sample occurs in more stages: 60-110°C loss of absorbed water; 130-190°C loss of water from calcium silicate hydrates (CSH) (Eskander et al, 2012); 250-450°C loss of water from different stages of dehydration of C-S-H and calcium aluminate hydrates, and loss of water from dehydration of interlayer water of clay minerals (Eskander et al,2012; čč et al, 2013; Plante et al., 2009); 450-600°C expulsion of OH-ion from the clay mineral lattice (dehydroxylation) (Plante et al., 2009); 600-680°C deydroxylation of clay mineral and initial stage of decomposition of carbonates ion associated with calcite (Eskander et al, 2012); 680-900°C decomposition of carbonate ions of calcite (Eskander et al, 2012). It should be noted that this analysis is the only one that allows to evidence the presence, as expected, of products of larnite's hydration.

4.2. Performances of earthen plasters

The Geelong test shows that, except for the earthen plasters (A and A1) which failed, all the other tested samples were not eroded at all (Figure 7). Concerning the Spray test as well (Figure 8) higher erosion resistance is evidenced for the stabilized earths. In particular, the erosion is none for the plasters stabilized with lime (B and B1).

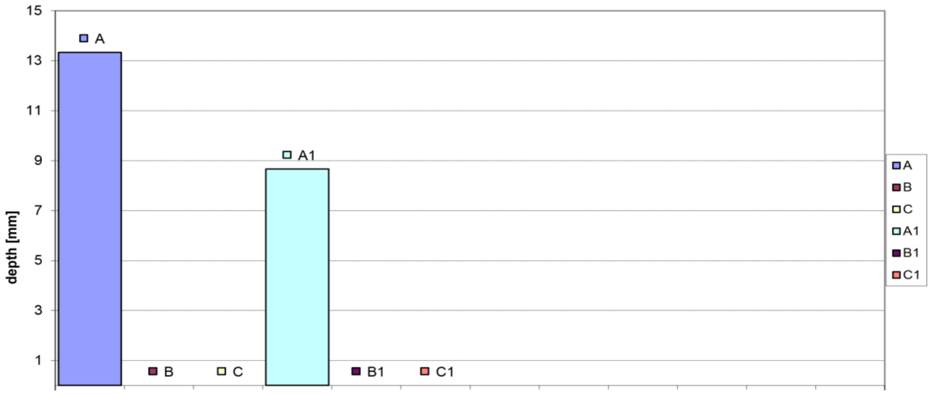

Figure 7: Diagram showing Geelong test results (in ordinates is the depth of erosion)

The water absorption test shows that, while gypsum (C and C1) reduces the rate of water absorption, the lime (B and B1) determines an increase of absorption with respect to the earth plaster (A) and the earth + straw (A1) (Figure 9).

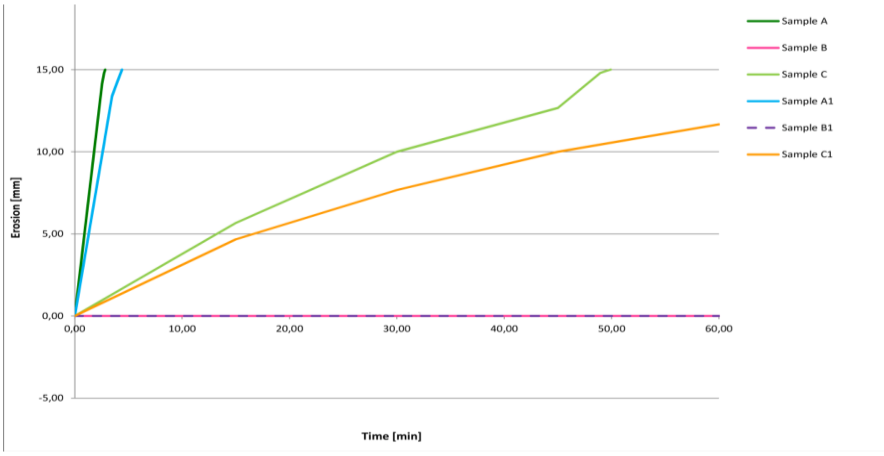

Figure 8: Diagram showing the erosion spray test results

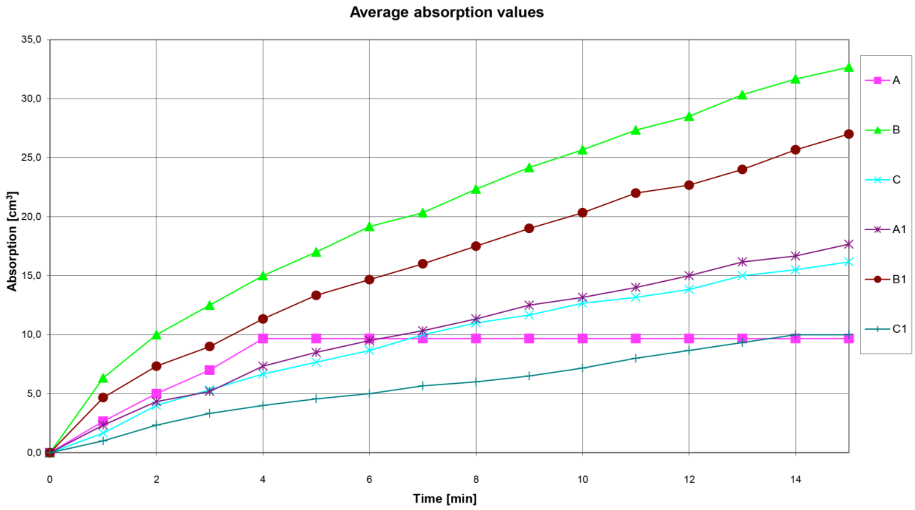

Figure 9: Diagram showing the water absorption test results.

The shear test point out that all the plasters made with straw (A1, B1, C1) are characterized by a lower shear strength compared to the others (Figure 10). The higher shear strength was obtained for the plaster stabilized with gypsum (C). However the results are quite scattered, which could be due both to heterogeneities of the wall and to the dimensions of the samples that makes it difficult to guarantee a good application of the coating. Therefore, the shear strength should be evaluated preparing more samples and increasing their size.

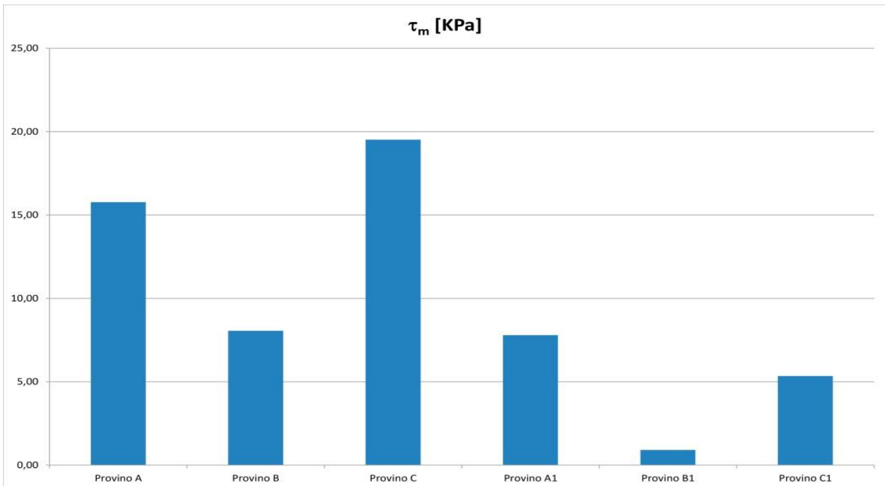

Figure 10: Shear test to evaluate the bond of the plasters to the wall (τ_m[KPa]).

The results of the water vapour permeability test, expressed as the residual water vapour permeability in percent, shows that: in the case of plaster A1 (earth +straw) with respect to plaster A (only earth), the residual permeability has a very little decrease (P. Res = 97%); plasters B and C with respect to plaster A, both have a very little decrease of residual permeability, respectively 96% and 97%; concerning plasters B1 and C1 with respect to plaster A1, the first has a decrease of the residual permeability slightly higher than the second, respectively 92% and 98%, but both are almost unchanged. In general the plasters keep their water vapour permeability unchanged.
The results of the colorimetric measurements are reported in Table 4 as average values of the coordinates L*, a* and b*. The ΔE* values between the following couples, are calculated: plaster without straw (A) and plaster with straw (A1); plaster with only earth (A) and plaster with earth + additive (B; C); plaster with earth + straw (A1) and plaster with earth + straw + additive (B1, C1). Also the parameter ΔE* related to the previous differences is reported.

Table 4: Colour parameters

Sample ID	*L**	*a**	*b**
A	50.92±0.24	10.26±0.07	16.15±0.13
A1	49.11±0.87	10.05±0.08	15.90±0.21
B	51.53±0.51	12.13±0.12	20.04±0.21
B1	53.40±0.70	11.97±0.09	20.83±0.17
C	54.83±0.64	10.37±0.04	17.08±0.11
C1	55.18±0.40	10.38±0.04	17.02±0.06

The data of the parameter ΔE* show that the addition of straw to the plaster without additive (A1) causes a little change (ΔE*=1.87) which, in particular, is manifested by a slight decrease of L*(darkening), not perceptible by the human eye (detection limit of the human eye ΔE*=5). In the case of plasters without straw, the following aspects have been pointed out: the plaster with lime shows a ΔE* equal to 4.35 and the higher contribution is due to an increase of b*(yellowing); the plaster with gypsum shows a ΔE* equal to 4.02 and the higher contribution is due to an increase of L*(whitening) (Table 4). In the plasters with straw the following aspects have been pointed out: the plaster with lime +straw shows a ΔE* equal to 6.81 and the higher contribution is due to an increase of b*(yellowing) and L*(whitening); the plaster with gypsum + straw shows a ΔE* equal to 6.18 and the higher contribution is due to an increase of L*(whitening).
In general the plasters with straw and stabilized with lime or gypsum show a higher change in colour, which can be detected by the human eye, with respect to those without straw.

Conclusion

The performance tests carried out in laboratory show that, with regard to water resistance (determined with the Geelong test and spray test), the addition of lime gives the best results. To be noted however that the plaster with no additives has not withstood the test.
With regard to the capillary absorption capacity, the values obtained show that gypsum has reduced it while the addition of lime has made the plaster more absorbent. With regard to shear tests, the presence of straw generally worsens the behaviour, while for gypsum and lime, the best performance has been obtained by the first one. Regarding the permeability to water vapour, it keeps almost unchanged in all the plasters.
With regard to colour, the presence of straw causes the higher chromatic variations both with gypsum and lime. In summary we can say that gypsum is the additive that allows the larger performances' improvement. It remains to determine the in situ plasters' behaviour, with respect to the results obtained in laboratory.

Author Contributions

To be noted: all the authors should be considered as principal authors

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