Effects of organic admixtures on the fresh and mechanical properties of earth-based plasters

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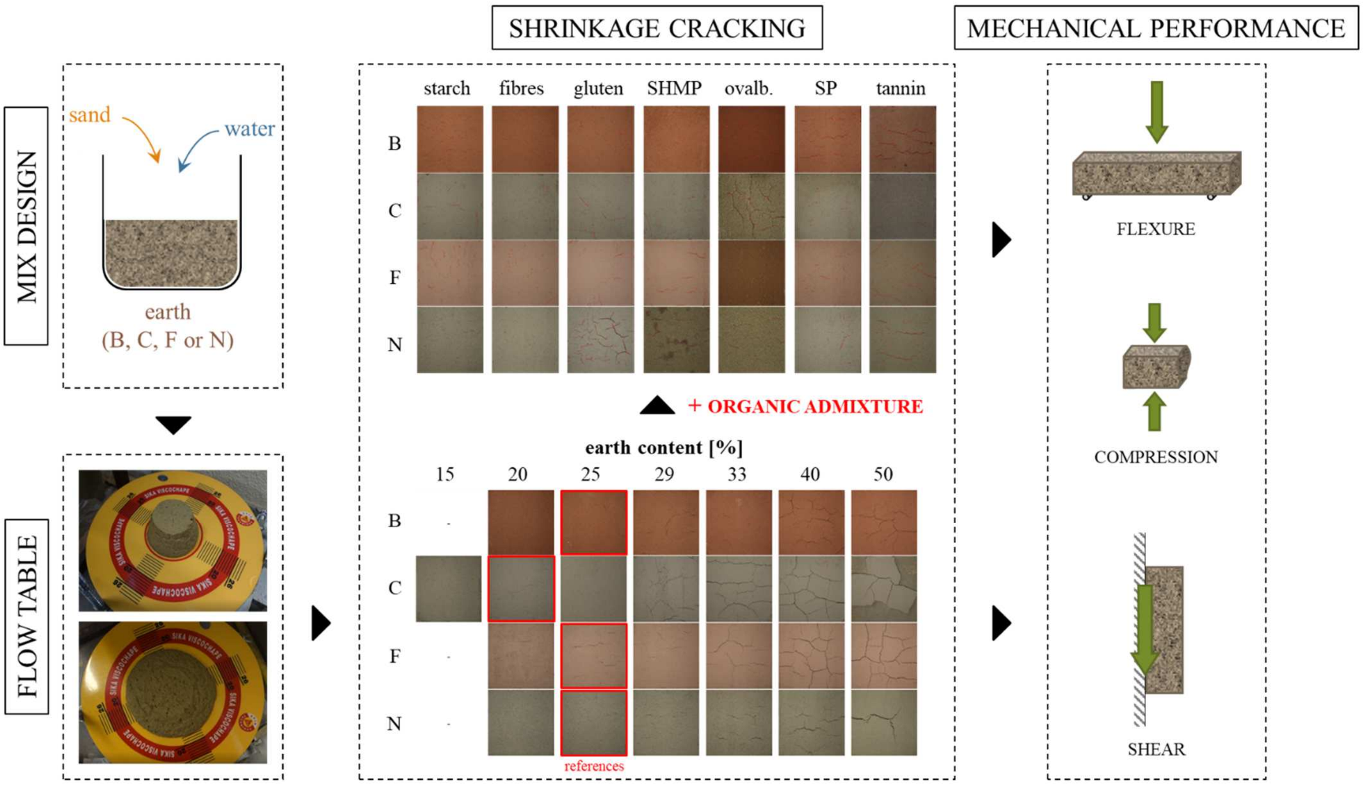
HIGHLIGHTS.

- Tests on four earths with distinct characteristics were conducted;
- Potential of 3 deflocculants, 1 fibre addition and 4 strengtheners were evaluated;
- Consistency, shrinkage, flexural, compressive and adhesive strengths were measured;
- Clay content is a key parameter on fresh properties and mechanical performances;
- Swelling potential and mineralogical compositions impact performances of plasters.

ABSTRACT. As a building material, earth is known to be a natural humidity regulator and to improve comfort inside buildings, making it a good choice for indoor plastering. The clayey phase ensures the sorption capacity of the material and its global cohesion by acting as a binder for the sand grain skeleton. However, clay also induces drying shrinkage of the mortar, leading to cracking of the plaster. Additives or stabilizers are typically used to address issues such as mechanical resistance and drying shrinkage in earthen building materials. To promote the development of eco-building materials, this paper pays particular attention to the potential of organic admixtures (vegetal or animal). The effectiveness of such admixtures could depend largely on the type of soil used and on the amount and nature of the clay minerals involved. Four types of soils were investigated. Based on consistency, shrinkage, visual and numerical detection of cracking, and flexural, compressive and shear tests, the characteristics of mortars in fresh and hardened states were evaluated. The results show that the clay content is a key parameter of both fresh properties and mechanical performances of plasters but is not sufficient by itself, especially when organic admixtures are involved. The potential of seven admixtures was evaluated. Although the deflocculants enabled a water reduction in mixes as well as a mechanical improvement, and the cohesion strengtheners were able to enhance mechanical properties at the hardened state, only flax fibre-based mortars demonstrated sufficient adhesion to their application substrate and, above all, no cracking as they dried.

GRAPHICAL ABSTRACT.



KEYWORDS. earth plaster; organic admixtures; flow table value; shrinkage; flexural and compressive strengths; shear stress.

1. Introduction

In an effort to reduce its environmental footprint, the building industry is looking for eco-friendly materials and construction techniques for the development of sustainable buildings. Earthen materials are in line with this perspective and, as a building material, earth offers many advantages that make it perfectly suited for indoor plastering.

First of all, as the visible envelope of the building, coatings must be able to meet aesthetic constraints. The finished texture and colour of clayey plasters can be defined at will, depending on the raw materials and the application techniques used [1], [2].

From an environmental point of view, as underlined by the Life Cycle Methodology implemented by Melià *et al.* [3], earth plasters outperform conventional industrial mortars. Found almost everywhere and in great quantities, earth can be extracted directly from natural soils or can be a by-product of the transformation of aggregates in quarries (washing fines). The raw materials for earth construction, primarily soil and sand, are endlessly reusable and recyclable (when not stabilized with chemical binders). Earth does not need energy for calcination like conventional binders, it only requires limited energy for grinding and it is often available locally (on or near the project site). Thus costs and energy for production and transportation are reduced [4].

Since clay acts as a passive ozone removal material, it can help to improve indoor air quality and effectively control indoor pollution. Experiments have shown that clay substantially reduces indoor ozone concentrations. The perceived air quality is then more acceptable and concentrations of aldehydes are lower [5], [6].

Furthermore, earthen plasters can contribute to comfort in indoor environments. Due to the high hygroscopicity of the clay minerals, earth-based materials are able to absorb and desorb humidity faster and to a greater extent than any other building material [7]–[9]. This capacity to balance indoor climate allows earth-based plasters to act as moisture buffers [7], [10]–[13]. The moisture buffering capacity is mainly linked to the first few centimetres of materials. The penetration depth of earthen materials has been shown to be lower than 10 mm [12], [14] and there is no apparent additional benefit to moisture buffering beyond this thickness of clay-based material. Hence, even a thin clay plaster plays a part in moisture diffusion, helping to limit the variations of humidity in a room. The use of earthen materials may also passively promote the energy efficiency of buildings, since it may help to decrease the need for mechanical ventilation and air conditioning [15].

Thermal inertia is also one of the main advantages of earth material. Like all heavy materials, earth-based building materials store heat. As a result, they allow the external temperature cycling to be smoothed so that relatively small temperature fluctuations are observed indoors [7], [16].

The characteristics of a soil vary with its origin, the site from which it was dug. Depending on its specificities, it could be composed of different amounts and types of clay, silt and sand. In order to prepare the correct mix for a specific application, the design and implementation method needs to be adapted according to the properties of the raw materials and, when necessary, it must be possible to select and apply the adequate additives efficiently to guarantee the desired behaviour.

Additives or stabilizers are typically used to address issues with earthen building materials and, in general, to improve particular properties of the material for specific applications: mechanical resistance, cracking during drying and abrasion (strengthen cohesion), protection against rainwater or erosion (preserve cohesion). A wide range of admixtures have, in fact, been commonly used in earthen construction. They include: mineral binders (cement, lime, gypsum) [17]–[21], sand or larger aggregates [7], [22]–[24], animal and vegetal stabilizers (alginates, casein, sugars) [25], [26], and natural fibres and aggregates [27]. To promote the development of eco-building materials, particular attention is paid to the potential of organic admixtures (vegetal or animal) in this paper.

Regarding the effect of organic stabilization encountered in the literature, one type of admixture can exhibit opposite results. Sorgho *et al.* [28] and Vissac *et al.* [26] used tannins to provide the waterproofing of earthen materials while Ouedraogo [21] only succeeded in improving water resistance time by a few minutes. Alginic acid-based bricks from Pinel *et al.* [29] also enhanced the water resistance of clayish specimens, which lasted three days in water and showed damage due to cracks only. Specimens admixed with alginic acid, on the other hand, completely disintegrated in 2 to 4 hours of immersion [21]. Although the nature of biopolymers or the amount incorporated could impact the effect of stabilization on earth-based materials, results from Ouedraogo [21] question the impact of the amount and nature of the clay minerals involved on the effectiveness of the admixtures. Considering two soils, F and N, distinct in terms of clay minerals, divergent effects were observed on similar admixed mix designs. Waxy maize had a beneficial effect on the water resistance of soil N and a detrimental one on the second earth type. Alginic acid was more effective on the F than on the N soil, improving its water resistance twice as much.

The effectiveness of these admixtures could depend largely on the type of soil used and on the amount and nature of the clay minerals involved. Yet, little research has been conducted on the impact of the mineralogical nature of soils and their compatibility with biopolymers. The work reported here investigated the effects of organic admixtures on the fresh and mechanical properties of earth-based plasters. Four types of soils were used and the effects of adding five different organic stabilizers to earth plasters were studied. The effects of these stabilizers on the workability in the fresh state were evaluated. After drying, the shrinkage was assessed in free and restrained conditions. The cracking analysis concerned both visual and numerical detection of the fissures on the surface of the specimens. The flexural, compressive and adhesive strengths of the mixtures were also measured.

2. Materials and methods

2.1. Materials, mortars and specimens

2.1.1. Soils

Four soils were used for this study: two of them came from brickworks and the other two were composed of quarry fines from aggregate washing processes. These soils were chosen because of their distinct natures (grain sizes, mineralogy, etc.).

The two brickwork soils, referred to as “soil B” and “soil N” had a typical rust colour linked to the presence of iron oxides, similar to a laterite and an ochre-coloured, clayey soil of the Garonne river valley, respectively.

The fines, smaller than 0.1 mm, were a by-product of the washing operation of coarser aggregates and were collected from settling ponds. Quarry fines are often considered as clayey materials that are too fine and too wet to be sold. Thus, using quarry washing fines is a way to add value to this type of waste. The sludge by-product was left in sedimentation basins. One of the fines, 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 left to dry before being reduced to powder. This soil is referred to as “soil F”.

The soils used in this study had been characterized in other studies [30], [31]. The main characteristics of the four soils are summarized in **Table 1**.

Soils	B	C	F	N
Clay < 2 µm [%]	33	10	28	23
Silt 2-63 µm [%]	40	88	66	37
Sand 63-2000 µm [%]	27	2	6	38
Gravel > 2 mm [%]	0	0	0	2
D90 [µm]	413	44	37	750
D50 [µm]	9	17	7	18
Liquid limit [%]	39.9	42.8	32.9	44.4
Plasticity index [%]	22.4	12.4	11.7	22.7
Main clay minerals	illite, kaolinite	illite, chlorite, vermiculite	kaolinite, illite	illite, chlorite, montmorillonite

Table 1. Soil characteristics.

2.1.2. Organic admixtures

Seven admixtures were tested here: five organic stabilizers, one type of fibre addition and a deflocculant largely employed by the ceramic industry. The latter was intended to serve as a benchmark for the organic admixtures.

On the one hand, the use of dispersants and superplasticizers was considered. Deflocculating the micro-sized clay-based structures would enable the interaction force between clay particles to be reduced, improving the earth material’s workability and the organization of the particles without adding water.

Sodium hexametaphosphate (SHMP), a conventional admixture in the ceramic industry, was used as a dispersant in this study to make the mortar more fluid. Previous works have shown that the use of this complexing agent reduces the viscosity and thus the quantity of water used for the dispersion, at constant workability. It therefore leads to a decrease of the drying shrinkage [32]–[34].

According to the French national research project PaTerre+ [26], tannins are also able to disperse clay particles under appropriate conditions of pH and ionic strength. They are especially useful to form iron tannate complexes, releasing multivalent iron ions. These metallic ions exhibit a load density so high that they glue clay platelets to one another strongly [28], [35]. Consequently, a tannic acid solution was tested in this study.

A "green" superplasticizer, synthesized from agricultural waste raw materials, was used. This superplasticizer, designed for a cement-based mix, is composed of more than 90% bio-based carbon and has a lower carbon footprint than conventional superplasticizers. Some authors, e.g. Alhaik *et al.* [36] and Perrot *et al.* [33], have also employed superplasticizer intended for cement formulations in earthen materials.

On the other hand, organic molecules such as starch, ovalbumin and gluten were chosen for their ability to strengthen the soil by holding the clay particles together. They are long enough to adhere to several clay platelets at once and connect them. Hence, those molecules are assumed to contribute to the tensile strength improvement.

Starch is a polysaccharide of vegetable origin, maize in our case, which provides a potential solution as a rheology modifying admixture. Starch, in powder, is usually used as a thickening agent because of its capacity to gel when in contact with water. In the fresh state, the earth mortar is thus more flexible during application. During drying, the polysaccharide gel causes clay layers to stick together, strengthening the plaster properties [26], [36], [37].

Proteins, generally amphiphilic, interact strongly with the clay particles, forming a kind of surface film that repels water. Protein adhesive sticks clay particles together and maintains the cohesion of the material even under water. Surface characteristics of proteins, like the egg white protein ovalbumin, are highly dependent on the environmental conditions: pH and ionic strength. Ovalbumin has proved its hydrophobic and strengthening effects on earthen surfaces [21], [25], [26], and this is why it was selected for this study.

The protein of gluten has also been employed to enhance earth-based materials [21], [38], [39]. In particular, Guerrieri (in Mileto *et al.* [38]) found that stabilizing mixtures with gluten led to a significant improvement regarding mechanical strength and superficial erosion resistance.

Finally, several authors have studied the effect of plant fibres to enhance the mechanical properties of plasters [10], [11], [19], [40]–[46]. As advised by Laborel-Préneron *et al.* [27], to reduce shrinkage cracking in plasters, it is preferable to use plant particles in fibre shape. Because of the small thickness applied, the fibres used were short and the particle content was low in order to obtain homogeneous mixtures that were easy to apply to the wall. Flax fibres, scutched before being cut mechanically, were chosen for this study. Their particle size distribution is given in **Figure 1**. It was obtained by image analysis [47]. The fibres were between 1 mm and 12 mm long with a median length of 6.1 mm. The aspect ratio (ratio of the major to minor axis of the particle's fitted ellipse) of 95% of the particles was lower than 30.

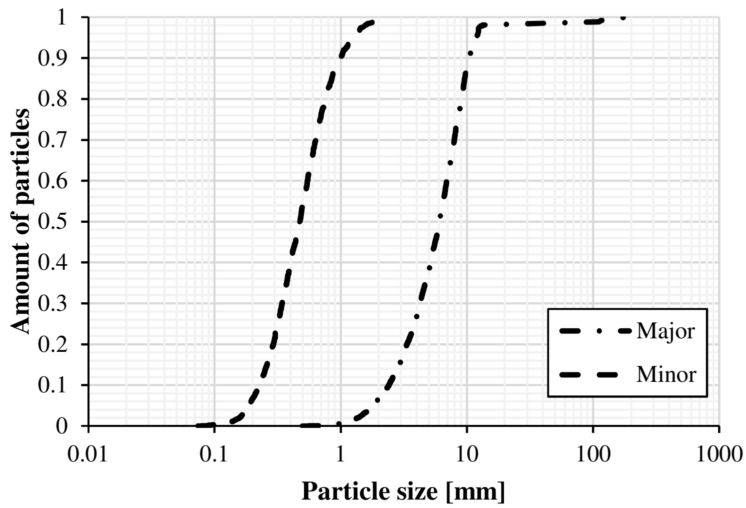


Figure 1. Particle size distribution of flax fibres incorporated within mortars, based on their major and minor axis lengths.

2.1.3. Design and manufacturing of specimens

The earth plasters studied were composed of a clayish earth (soil or quarry washing fines), a siliceous river sand (0-2 mm) and tap water. By virtue of its extremely small particle size and high surface-to-volume ratio, clay exhibits glue-like properties in the presence of water [48]. Thus, the clayey phase acts as a binder while sands form the granular skeleton. During drying, the finer fraction shrinks, causing plaster rich in clay minerals to crack. To prevent this phenomenon, earth is admixed with sand.

Earth plasters were prepared from earth to which various proportions of sand and water had been added. Six mortars were formulated for each type of soil, 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%), 1:4 (20%). In view of its behaviour, an extra formulation with 15% earth content was mixed for soil C. For each type of soil, the plaster with the lowest earth to sand ratio that exhibited cracks was selected as the reference formulation (**Table 2**).

Earth type and ratio [%]	Reference	Starch	Fibres	Gluten	SHMP	Ovalbumin	Super-plasticizer	Tannin
B - 25	23.2	22.2	24.3	24.5	16.7	24.1	22.5	21.5
C - 20	21.5	21.0	25.4	25.9	17.9	28.1	18.8	17.3
F - 20	19.1	18.1	19.0	21.4	14.7	19.4	16.9	19.0
N - 25	23.4	22.1	25.2	31.5	20.5	26.9	22.2	22.8

Table 2. Water content (in %) of the reference and admixed formulations.

One of the objectives of this article is to compare the admixtures with each other without trying to optimise their use. To assess the effect of stabilization on the physical and mechanical properties of plasters, admixtures of 1% of the total solid dry mass (i.e. considering both earth and sand) were added to a reference mix design, except for flax fibres. Fibre particle additions were restricted to 0.5% of the solid dry mass to maintain a satisfactory workability for the implementation of the coating. The effect of stabilizers and additions were assessed with respect to the reference formulation and a deviation value was calculated (in %) as follows:

$$deviation = \frac{value_{admixed\ mortar} - value_{reference\ mix}}{value_{reference\ mix}} * 100$$

In all cases, mortars were prepared by using the mixing procedure:

- mix the solid phase at low speed over a period of 30 seconds;
- if necessary, incorporate the admixture into water;
- 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 for 1 min;
- stop the mixer was and leave the mixture to rest for at least five minutes;
- complete mixing at high speed for a further 30 seconds.

Specimens applied to the wall panels

A commercial fibrewood rigid panel was used as the substrate. This material was intended for use as facade/wall/roof insulation. It is described as a fire-resistant (class E), thermal and acoustic insulation board ($\lambda = 0.044$ W/(m.K)). The board was 35 mm thick and had a density of 180 kg/m³. The fibrewood panel was soiled and sprayed with water in advance. Once dry, the substrate was lightly and evenly water sprayed again immediately before the application of a plaster coating.

For the preparation of the test specimens, the fresh mortar was directly applied to the prepared substrate with light impulsion. The plaster was easily levelled using a wooden frame and a trowel. The thickness of all plasters was around 1 cm.

The shrinkage test required two 250 x 250 mm² specimens of each formulation to be applied to the commercial fibrewood panels. A wooden frame was used once again to ensure the dimensions of the specimens.

Circular test areas approximately 66 mm in diameter were cut through the mortar layer while it was fresh, for the shear test. After application of the mortar layer on the substrate, a clean cup was pressed, with slight rotation, through the fresh mortar layer until full contact with the substrate was achieved. The outer edges of the cup were then carefully detached and the cup was removed, still slightly rotating. At least five test areas were produced per formulation.

The coated panels were placed in an upright position and stored until they were in equilibrium with the ambient conditions (18 ± 2°C and 57 ± 8% RH).

Prismatic specimens

In accordance with the German standard [49], prismatic specimens were prepared following EN 1015-11 [50]. The mixing procedure in this standard is the same as that used for the preparation of cement-based plasters [51]. Once the mixing procedure had been completed, the mortar was introduced into each of the mould compartments, directly from the mixing bowl as stated in procedure EN 196-1 [51].

The mould was filled with mortar and compacted in two layers. The surplus mortar was removed to level the surface and the mortar was left to dry in ambient conditions. Once the specimens were dry enough to permit proper demoulding, generally after three to four days, they were completely demoulded, taking care not to damage them. The specimens were kept for at least 21 days. For the last few days, specimens were stored at (23 ± 2)°C and (50 ± 5)% relative humidity until mass consistency was reached and the test could be performed.

2.2. Testing Procedures

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 [49] and inspired by the methodology developed by various authors [42], [45], [52].

2.2.1. Characteristics of fresh state plasters

Based on standard EN 1015-3 [53], the consistency of fresh plaster was determined using a flow table. The consistency was characterized by measuring the mean diameter of a test specimen 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 ensure similar consistency among the materials, ensuring good workability, the water content was adjusted. The German standard [49] proposes fixing the consistency at 175 ± 5 mm. The water content of fresh plaster was measured by drying at 60°C.

2.2.2. Mechanical performance of earthen materials

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 $40 \times 40 \times 160$ mm^3 prismatic specimens of mortar.

The shrinkage was also evaluated on two plaster specimens, 25 x 25 cm^2 and 1 cm thick, per formulation. The presence or absence of cracks in the specimens after drying was detected by both visual and image analyses. The cracking assessment was processed by numerically isolating of the fissures using Matlab and a specific algorithm. Regarding the segmentation of the images into grey levels, the same Particle Swarm Optimisation (PSO) segmentation algorithm as described by Blanc *et al.* [54] was implemented in the Matlab program. An improved form of the segmentation method developed by Kennedy and Eberhart [55] was employed: the Fractional-Order Darwinian Particle Swarm Optimisation (FODPSO) [56]. In addition to the PSO algorithm, a large number of filters and statistical calculations were added to increase the precision of the cracking detection: background adjustment, morphology, filtering on both circularity factor and minimum area. The various steps of the analysis are summarized in **Figure 2**. This work enabled the cumulative developed length of cracks to be characterized quantitatively.

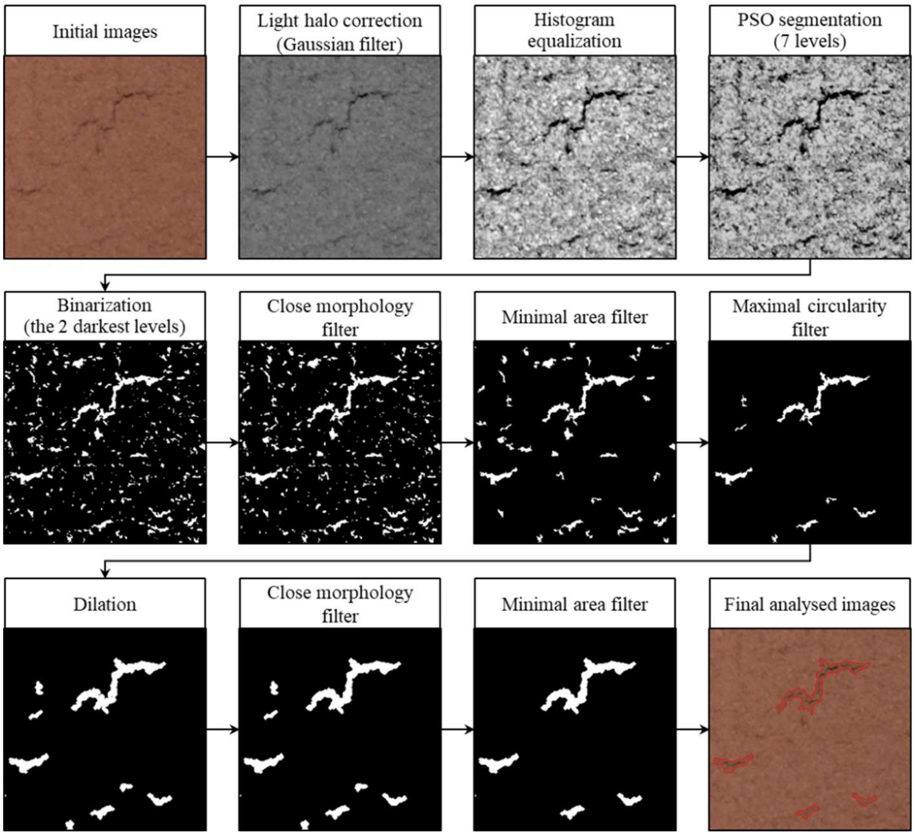


Figure 2. Matlab software analysis steps.

The shrinkage test was conducted according to the procedure proposed by Hamard *et al.* [42]: an earth plaster is of acceptable mechanical quality if, after shrinkage, there are no cracks through which water can penetrate into the wall and if the plaster is not detached (even partially) from the substrate.

Flexural and compressive strengths

The tests of flexural and compressive strengths were carried out in accordance with EN 1015-11 [50] on prismatic specimens of earthen mortar, using an MTS bending press equipped with a 10 kN sensor. Three 40 x 40 x 160 mm³ specimens per formulation were provided for the three-point bending test. The two halves of a 40 x 40 x 160 mm³ specimen obtained from the flexural test were used to provide six half specimens for the compressive strength test.

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

Adhesive strength

The shear test aims to evaluate the bond of an earth plaster with the wall. The adhesive strength is determined as the maximum tensile stress applied by a direct load perpendicular to the surface of plastering mortar on a substrate. The tensile load is applied on a circular specimen by means of a bucket suspended on the test area of the mortar surface by a cord. This system ensures a good contact with the upper part of the specimen without any contact with the wall (**Figure 3**).

First of all, specimens were loaded by a 2 kg device. If, after 30 seconds, all specimens had resisted the load, the substrate was considered to have been well prepared and the mortar was judged to be of sufficient quality [57].

Once this first test had been validated, specimens were loaded with about 1 kg increments every 15 seconds until failure. The mass at which the specimen broke was recorded. The adhesive strength obtained was the quotient between the failure load [kg] multiplied by the gravitational acceleration (= 9.81 $m.s^{-2}$) and the test area [m^2].

Figure 3. Shear test assembly.

3. Results

3.1. Characteristics of plasters in the fresh state

In the fresh state, all mortars achieved a consistency of 175 ± 5 mm in the flow table test. The water needed to ensure such a workability is given for the various clay contents and types of soils in **Figure 4** and **Figure 5**.

For plasters without admixture, the water content needed to achieve the desired workability ranged from 19.1 to 28.8%. On the one hand, for a given type of earth, the water amount increased with the clay content in nearly all cases. The more binder particles there were, the greater was the amount of water required to reach yield stress.

On the other hand, the nature of the earth had a significant impact on the water demand. In **Figure 4**, the water content needed is plotted against the clay content of each mixture, i.e. the content of particles smaller than 2 µm in each type of earth. Nevertheless, fine particles, identified as silt in **Table 1** could also play a role in the water demand of the soil, especially for soil C, in which this size fraction represents almost 90% of the particles. The same results were then represented in **Figure 5** against the fines content of each soil, namely the total percentage of particles smaller than 63 µm.

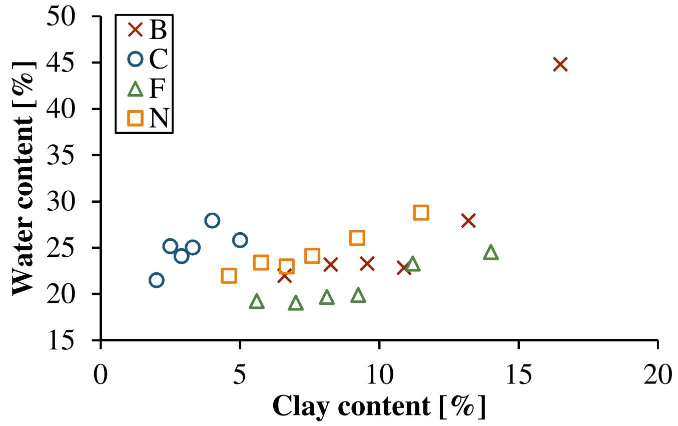


Figure 4. Water content of the four soils depending on the clay content.

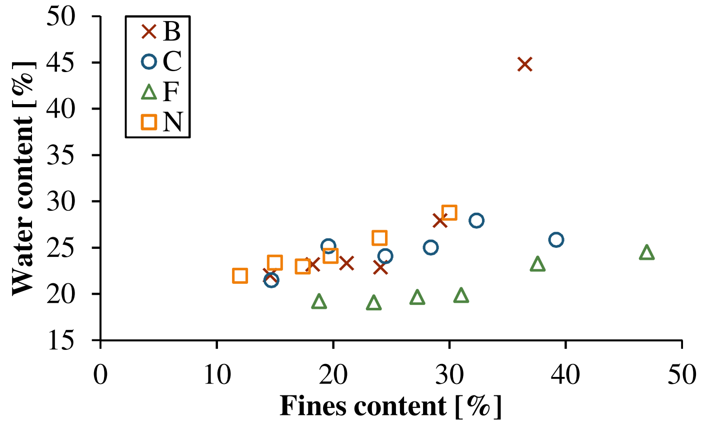
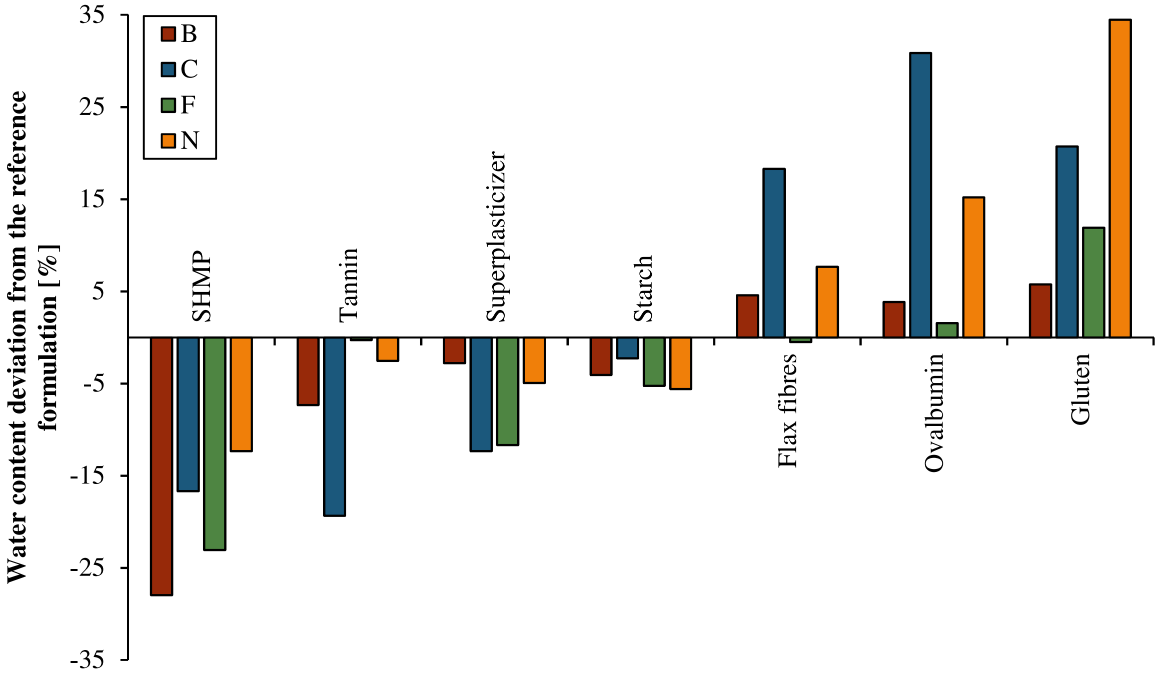


Figure 5. Water content of the four soils depending on the fines content.

This method of representation evidences the comparable behaviour of soils B, N and C and confirms the effect of the silt fraction on the water demand. The significantly lower values obtained with soil F could be linked to its specific mineralogical composition (high amounts of non-clayey fractions such as calcite and quartz). These results support the idea that analysing the properties of earthen materials by equating clayey fraction to size fraction below 2 µm is not sufficient. The mineralogical composition of the soil appears to be a key parameter and will be explored in further work.

All the water contents needed to achieve the desired workability were of the same order of magnitude as results from Delinière *et al.* [52], Fabbri *et al.* [58], Faria *et al.* [44], Hamard *et al.* [42], Randazzo *et al.* [59] and Santos *et al.* [2], [60].

Figure 6 brings out the effect of stabilization on mixing water requirement, in comparison with the reference formulations (addition of 1% wt. of admixtures or 0.5% of fibre particles (flax fibres)).



The kneading water for admixed mortars is found to be between 15 and 31%. The capacity of SHMP, tannin, superplasticizer and starch to reduce the water to binder ratio is clearly highlighted for the same quantity and nature of clay minerals and for similar consistency of plasters. They all act as dispersants, reducing the quantity of water needed to activate clay binding properties. SHMP is a well-known dispersant used mostly in the ceramic industry. Its ability to modify the rheology of the binding phase, making clay-based mortars more flowable regardless the type of clay minerals involved, is highlighted here. This property was already underlined and exploited in the works of Moevus *et al.* [34] and Perrot *et al.* [32], [33].

The addition of tannin also enables a reduction of water to binder ratio. According to Roux [35] and Vissac *et al.* [26], tannin is able to disperse clay particles and increase plasticity but only under adequate pH and ionic strength conditions. Tannin reacts with cation to form tannin-cation complexes. The transformation of the environment linked to the appearance of negative charges can lead to the dispersion of clay particles. This difference of conditions between the four earths might explain the difference of water content required when tannin is incorporated. Hence, earths B and C seem to be more suitable than F and N.

The capacity of superplasticizer to reduce the water-to-binder ratio in clay-based materials is clearly highlighted. This was also observed by Alhaik *et al.* [36] and Perrot *et al.* [33]. The superplasticizer appears to have a more pronounced effect on soils containing smaller particles. This could be explained by the fact that repulsive potential caused by the superplasticizer depends on the diameter of the grains: the smaller the grains, the stronger the electrostatic repulsion.

The addition of starch has a thinning effect which, although moderate, is verified regardless of the nature of the soil. These results differ from the findings from Alhaik *et al.* [36], who observed a large increase of water-to-binder ratio on reinforced plaster mixtures admixed with starch compared to non-admixed mortars, for the same consistency. This might be due to the quantity of starch incorporated. In the latter work, the starch/binder ratio was set at 1%, which is about 4-5 times higher than the quantity involved here. In the work of Ouedraogo [21], when the wheat starch/binder ratio was reduced from 4% to 1%, the effect on water requirement was seen to be slight.

Regarding the impact of flax fibre, ovalbumin and gluten additions, the mixing water need can be seen to increase compared to the reference formulations. Several studies have also shown that plant aggregates and fibres are hydrophilic materials that absorb manufacturing water [27]. Their highly porous structure and water sensitivity lead to a greater water requirement to ensure similar workability. Flax fibres, like most ligno-cellulosic materials, have a great capacity to absorb water [61], [62].

Ovalbumin and gluten additions also increase the water required to achieve the targeted consistency. The behaviour of ovalbumin depends strongly on the pH and ionic force conditions: it can either flocculate or disperse clay minerals. The clay-based environments studied seemed to favour flocculation of clay particles by ovalbumin protein. The effect was more pronounced for earths C and N than for B and F. Results regarding ovalbumin and gluten admixtures are consistent with water content measurement in Ouedraogo study [21].

3.2. Mechanical performance of earthen plasters

3.2.1. Shrinkage

The clay fraction ensures the cohesion of the material thanks to its colloidal properties. During drying, the water required to achieve workability evaporates. Internal stresses are thus generated causing the earth-based mortar to shrink.

This shrinkage is expressed as a volumetric variation between the fresh and the hardened states. The results of non-admixed plasters are brought together in **Figure 7**.

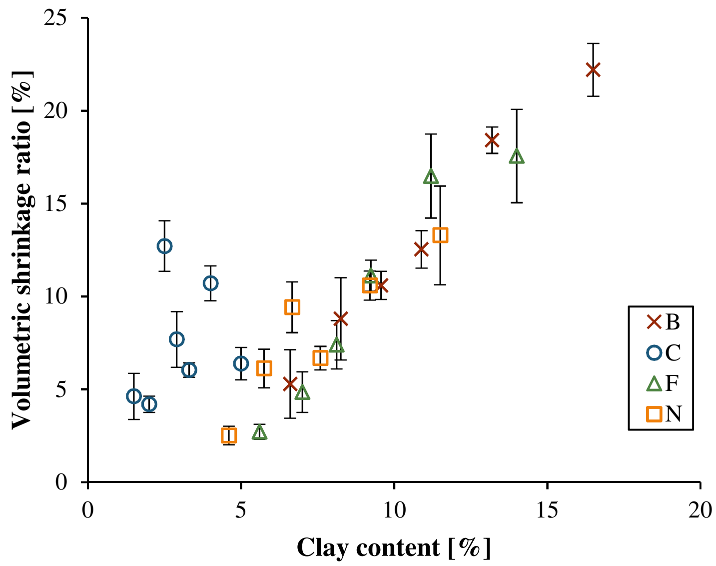


Figure 7. Volumetric shrinkage of mortars depending on clay contents and types of soil.

The amount of clay significantly impacts the shrinkage of 40 × 40 × 160 mm³ specimens. The volumetric shrinkage ratio rises with clay content. In fact, higher clay content in mortars results in an increase of binder quantity, leading to a greater volumetric variation of the specimens. This observation is in accordance with findings by Delinière *et al.* [52], Gomes *et al.* [19] and Hamard *et al.* [42].

Shrinkage of prismatic specimens of soils B, F and N follow the same trend. Thus, no noteworthy link is detected between the type of clay minerals and the level of shrinkage. Plaster designed with earth N, mainly composed of montmorillonite clay, does not appear more likely to suffer shrinkage than the others. This observation is in accordance with Delinière *et al.* [52] but inconsistent with findings from Lima *et al.* [63]: montmorillonite mortar showed the highest shrinkage, about nine times higher than kaolinite mortar and almost four times higher than illite mortar. Nevertheless, soil C exhibits a distinct behaviour. In spite of their reduced amount of particles smaller than 2 µm (below

5%), the mortars made of soil C suffered a large volumetric shrinkage. This could be linked to the contribution of particles with sizes between 2 and 63 µm.

The effect of admixtures and additions on the volumetric shrinkage compared to the reference formulation for each type of soil is presented in **Figure 8**, while **Figure 9** summarizes the link between water requirement and volumetric contraction for the admixed mixes.

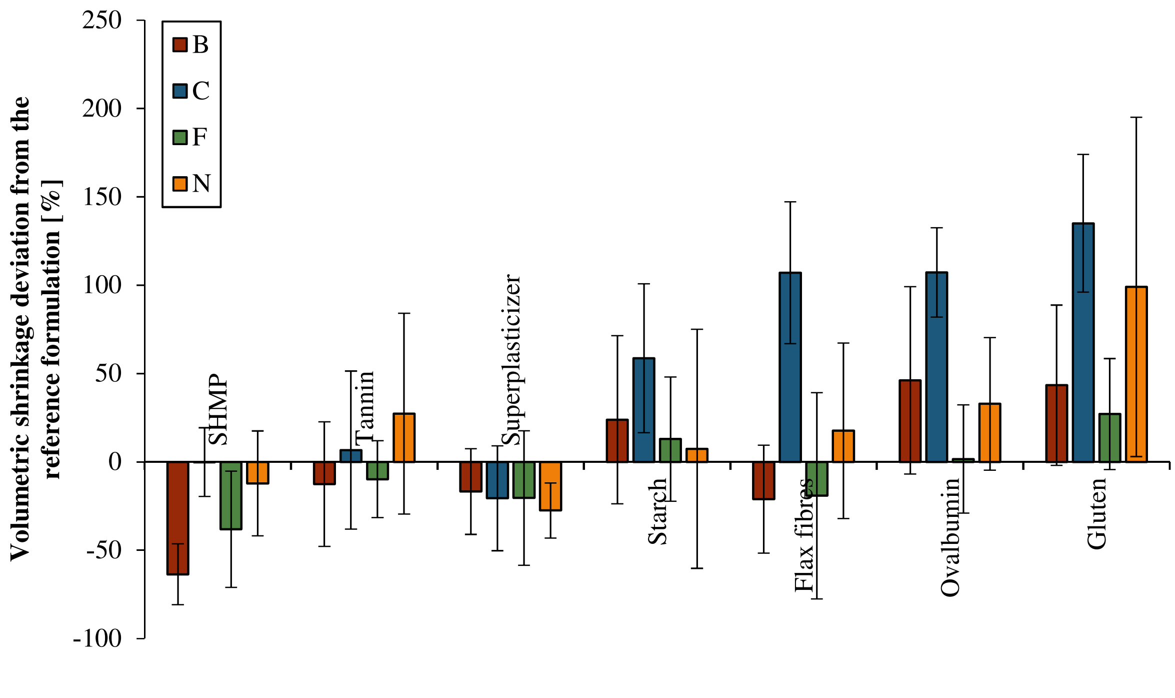


Figure 8. Volumetric shrinkage ratio deviation from the reference formulations.

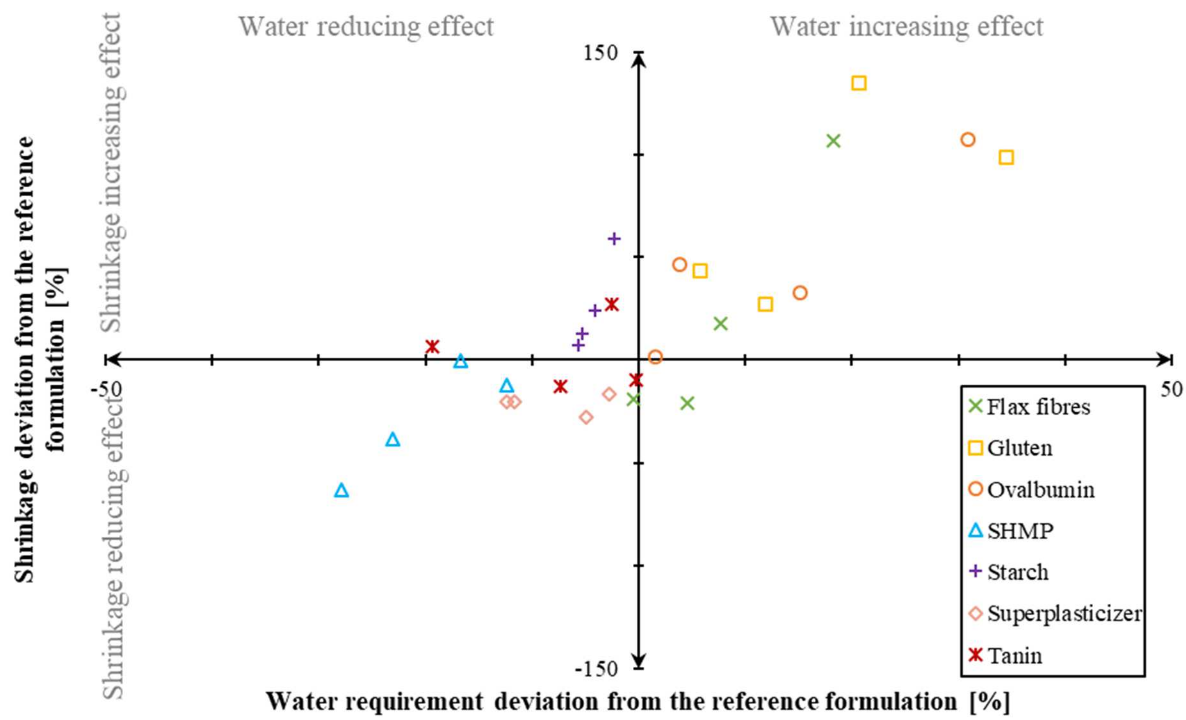


Figure 9. Link between water requirement and volumetric contraction of admixed mixes.

Figure 9 highlights prevailing trends. Thus, it can be seen that both SHMP and superplasticizer reduce the kneading water need, ultimately leading to a slight tendency towards shrinkage reduction for all earth specimens.

Gluten and ovalbumin additions show the opposite effect: these biopolymers increase the need for water, accentuating the volumetric contraction, regardless of the nature of the earth.

The effect of starch, given the margin of error, is not obvious. The slight increase in volumetric contraction might come from the fact that the organic admixtures enhance the cohesiveness of clay minerals, by increasing the adhesion forces involved. The activity of clay minerals could be connected to this property of admixtures: the greater the activity of the clays, the more effective the adjuvants would be in strengthening the bonds and thus the greater the shrinkage.

Tannin and fibre addition effects cannot be interpreted solely on the basis of water requirement; they depend on the type of clay involved. The shrinkage is reduced for C and N formulations while it is worsened for B and F soils. As far as tannin is concerned, this might be due to the fact that C and N environments are more suitable, in terms of pH and ionic forces, for the formation of iron tannates. These metal ions have such a high charge density that they stick the clay platelets together strongly. Regarding fibre additions, several studies observed an improved behaviour of plasters regarding shrinkage when plant aggregates and fibres were incorporated [27], [42], [43], [64]. However, with soils B and F, the opposite situation occurred. The positive effect of fibres on shrinkage might be counterbalanced by the greater amount of water needed to ensure a good workability of the plaster.

The volumetric shrinkage ratios of this study ranged from 2.5 to 22.2%. This significant discrepancy can be explained, first of all, by the parameter considered. The shrinkage ratio employed here is volumetric whereas other authors referred to linear variations. As underlined by Gomes *et al.* [19], results are quite variable between volumetric and linear shrinkage. Volumetric shrinkage seems to be a more sensitive parameter than the linear one according to Gomes *et al.* [19], values being much higher (globally, more than doubled).

The volumetric shrinkage values correspond to linear variation ratios varying between 0.1 and 8.1%. These values are in accordance with those given by Minke [7], who stated that the linear shrinkage ratio is usually between 3 and 12% with mixtures used for mortar and mud brick. The results obtained are, however, considerably higher than the ones available in other studies. Most experimental measurements report shrinkage values lower than 1.5% for prismatic specimens [2], [22], [43], [44]. Delinière *et al.* [52] obtained linear shrinkage ratios between 1.5 and 2.5% while specimens made by Ashour and Wu [64] reached up to 4.6% of dimensional variation (cohesive soil only).

This deviation from literature findings can be justified by the absence of stabilizers or additions within the mortars, by the higher water content required to achieve the desired consistency, and the curing under ambient conditions. Moreover, as mentioned by Delinière *et al.* [52] shrinkage is a complex phenomenon involving several parameters, such as the content and nature of clay minerals, the cationic content of water, and the drying procedure used (which may cause various suction conditions within the material). The study by Ashour and Wu [64] highlighted the impact of temperature on shrinkage: an increase in the curing temperature from 30 to 70°C led to a significant increase in the shrinkage ratio.

However, this test on prismatic specimens can only be used as an indicator. The plastic shrinkage evaluated by the two tests is distinct in nature. On prismatic specimens, the shrinkage is free with unrestricted deformation. The behaviour of plasters applied to walls is quite different. Walls restrict contraction of the mortars to their immediate vicinity, possibly leading to cracks: restrained shrinkage occurs. The different drying kinetics between the two sides of the plaster is also responsible for a change in drying conditions [52]. Consequently, it is necessary to carry out shrinkage tests in realistic conditions. The shrinkage profiles observed on non-stabilized plaster specimens laid on fibrewood panels are gathered in **Figure 10**.

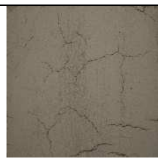
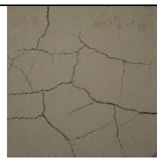
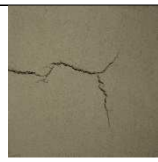
Earth content [%]	15	20	25	29	33	40	50
B	-						
cracked	-	No	Yes	Yes	Yes	Yes	Yes
bowed out	-	No	No	No	No	Yes	Yes
fallen	-	No	No	No	No	No	No
C							
cracked	No	Yes	Yes	Yes	Yes	Yes	Yes
bowed out	No	No	No	No	Yes	Yes	Yes
fallen	No	No	No	No	No	No	Yes
F	-						
cracked	-	No	Yes	Yes	Yes	Yes	Yes
bowed out	-	No	No	No	Yes	Yes	Yes
fallen	-	No	No	No	No	No	Yes
N	-						
cracked	-	No	Yes	Yes	Yes	Yes	Yes
bowed out	-	No	No	No	Yes	Yes	Yes
fallen	-	No	No	No	No	No	Yes

Figure 10. Shrinkage test results for different earth to sand ratios.

The behaviours of plasters applied on walls are consistent with the results of shrinkage measurements made on prismatic specimens. The increase of earth content induces cracks or bowing on the plasters. These observations have already been made on plaster surfaces [42], [45], [65]. With a smaller amount of earth, the specimens remained flawless: those formulations (20% B, 15% C, 20% F and 20%N) appear to be the best regarding shrinkage. Hence, for each type of soil, the plaster with the lowest earth to sand ratio that exhibited cracks was selected as the reference mortar to compare the effect of admixtures.

Regarding the admixed formulations, none of the plaster specimens bowed out or fell off the substrate. Almost all coatings cracked. The cracking results make it clear that the reduction in free shrinkage thanks to admixtures was not sufficiently marked to enhance the shrinkage of plasters significantly. Only the four flax-fibre-admixed mortars exhibited flawless surfaces. This underlines the reinforcement properties of vegetal fibres epitomized by previous studies [27], [42], [43], [64].

The Matlab algorithm can quantitatively assess the impact of admixtures on cracking. Although cracks are still visible on the surface of most specimens, some positive effects are encountered (**Figure 11** and **Figure 12**).

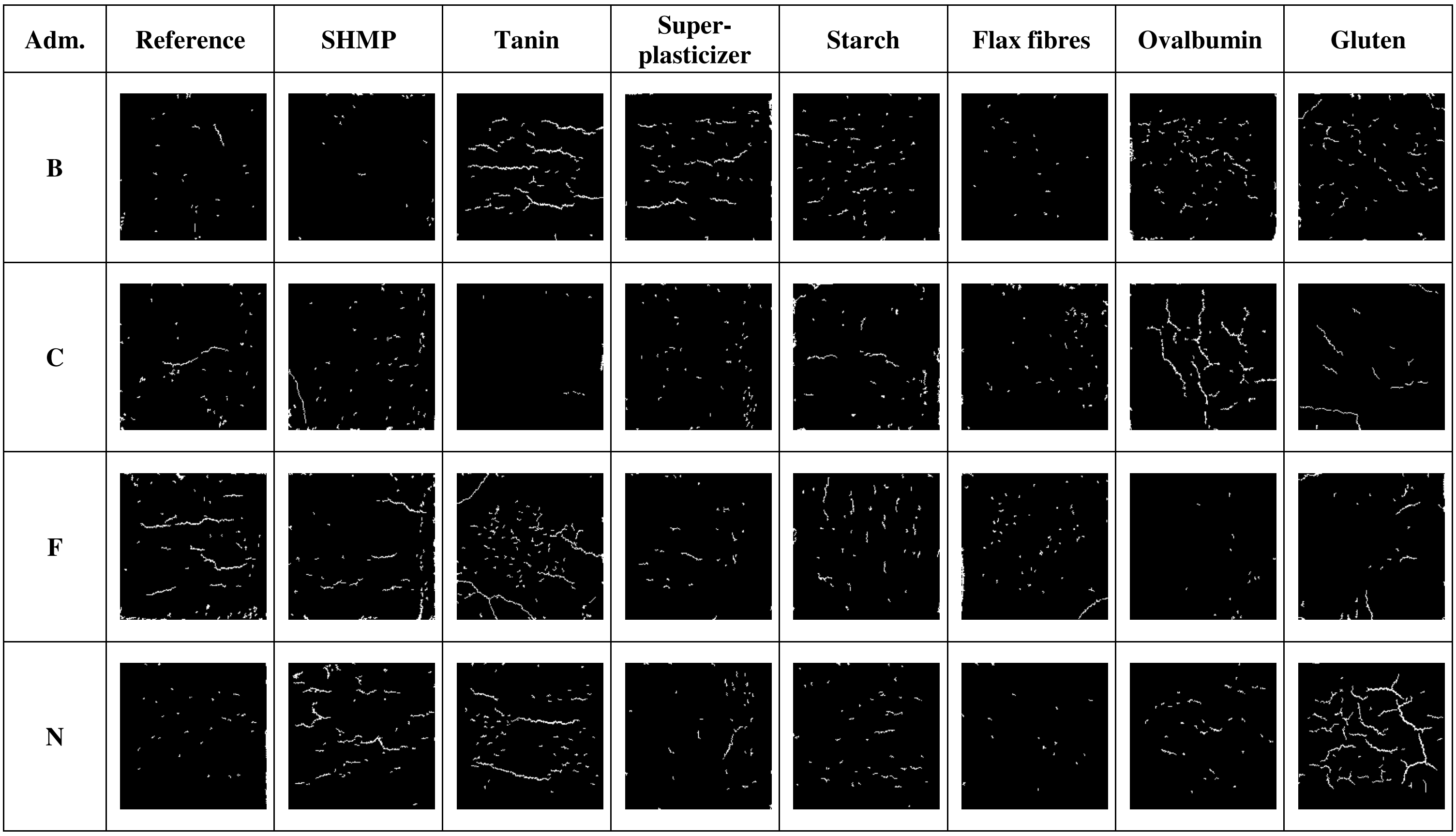


Figure 11. Shrinkage test binarised images of the admixed mortars.

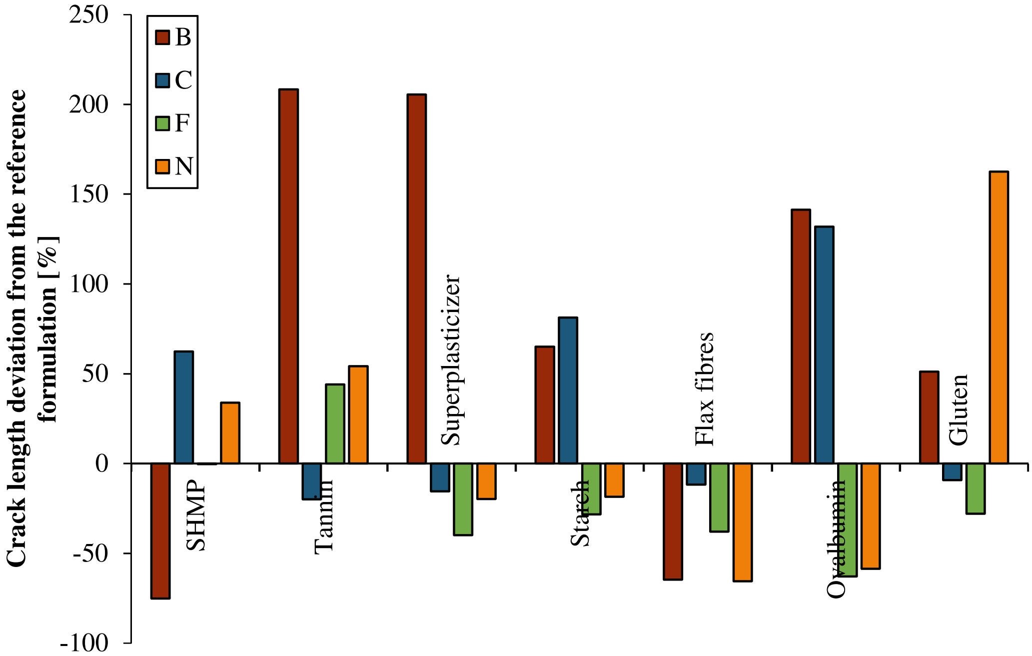


Figure 12. Comparative effect of admixtures on the cumulative length of shrinkage cracks.

Despite a significant reduction of free shrinkage, the effect of SHMP and of the superplasticizer is limited regarding shrinkage cracking in restrained conditions and depends on the nature of the clay minerals involved. The other biopolymers exhibit mixed results depending on the type of earth used. It seems that cracks are likely to be reduced for soils F and N, while their number and cumulative length increase for the other two soils. It is also worth noting that restrained shrinkage is a complex phenomenon: it involves not only the nature and amount of clay together with the cationic content of the water and the drying procedure used but also the drying kinetics of both the plaster and the substrate [52]. Consequently, the mortars might detach themselves slightly from the substrate when drying, allowing freer shrinkage and reducing flaws on the specimen surface. The protocol employed must be developed further to identify the different contributions to restrained shrinkage and to prevent the specimen detaching from the wall.

3.2.2. Flexural and compressive strengths

Figure 13 gives the evolution of both flexural and compressive strengths with the increase in clay content.

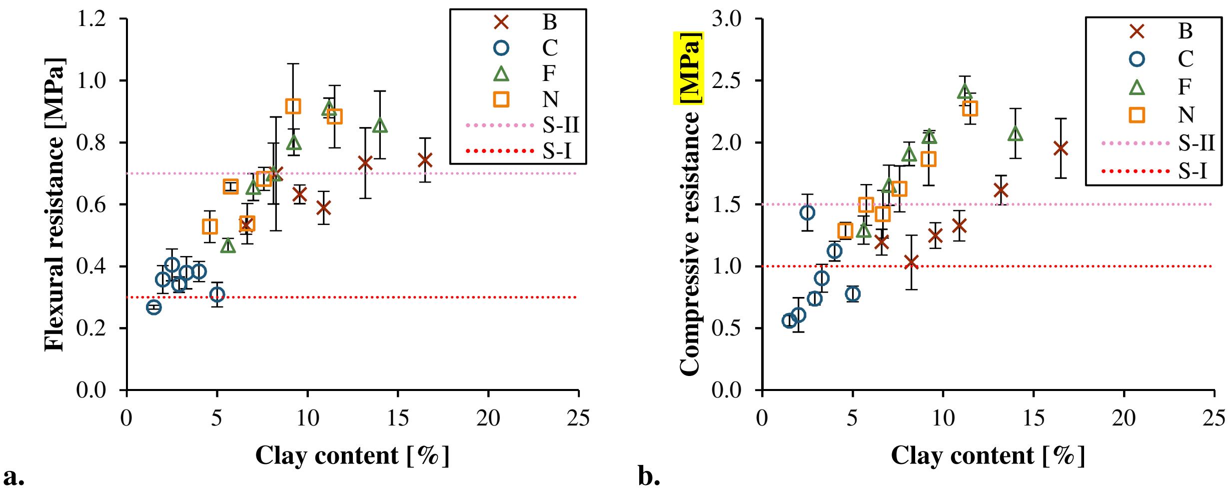


Figure 13. (**a.**) Flexural and (**b.**) compressive resistances of prismatic specimens of mortars depending on their clay contents.

The flexural strength ranges from 0.27 to 0.92 MPa. According to the German standard [49], except for the 15% C earth formulation, all specimens reach the S-I resistance class (≥ 0.3 MPa). 46% of them also meet requirements imposed for the S-II class from the flexural point of view (≥ 0.7 MPa).

The compressive strength values are between 0.56 and 2.42 MPa. Once again, only C-based formulations are lower than the S-I minimum value imposed by DIN 18947 (≥ 1.0 MPa): this is the case of 15%, 20%, 29% and 33% earth content mixes. Eleven of the 25 plasters designed fall into the S-II category.

Figure 13 demonstrates that clay content strengthens the mixes from both a flexural and a compressive point of view, as also established by Delinière *et al.* [52]**,** Emiroğlu *et al.* [65], Hamard *et al.* [42] and Lima *et al.* [22]. Results from soils C, F and N are in line on both graphs. The nature of the main clay minerals contained in these three soils does not seem to influence the strength of the clay plasters, results being very close at similar clay content. Similar findings have been obtained by Delinière *et al.* [52]**.** However, earth B's behaviour is quite offset. This might come from its mineralogical nature, the activity of illite and kaolinite being rather low compared to that of other clay minerals [66]. Colloidal properties of the material are thus impacted. The clay content in B-based plaster must be higher than in other plasters to achieve comparable resistances.

The impact of admixtures on mechanical strengths is assessed in **Figure 14.**

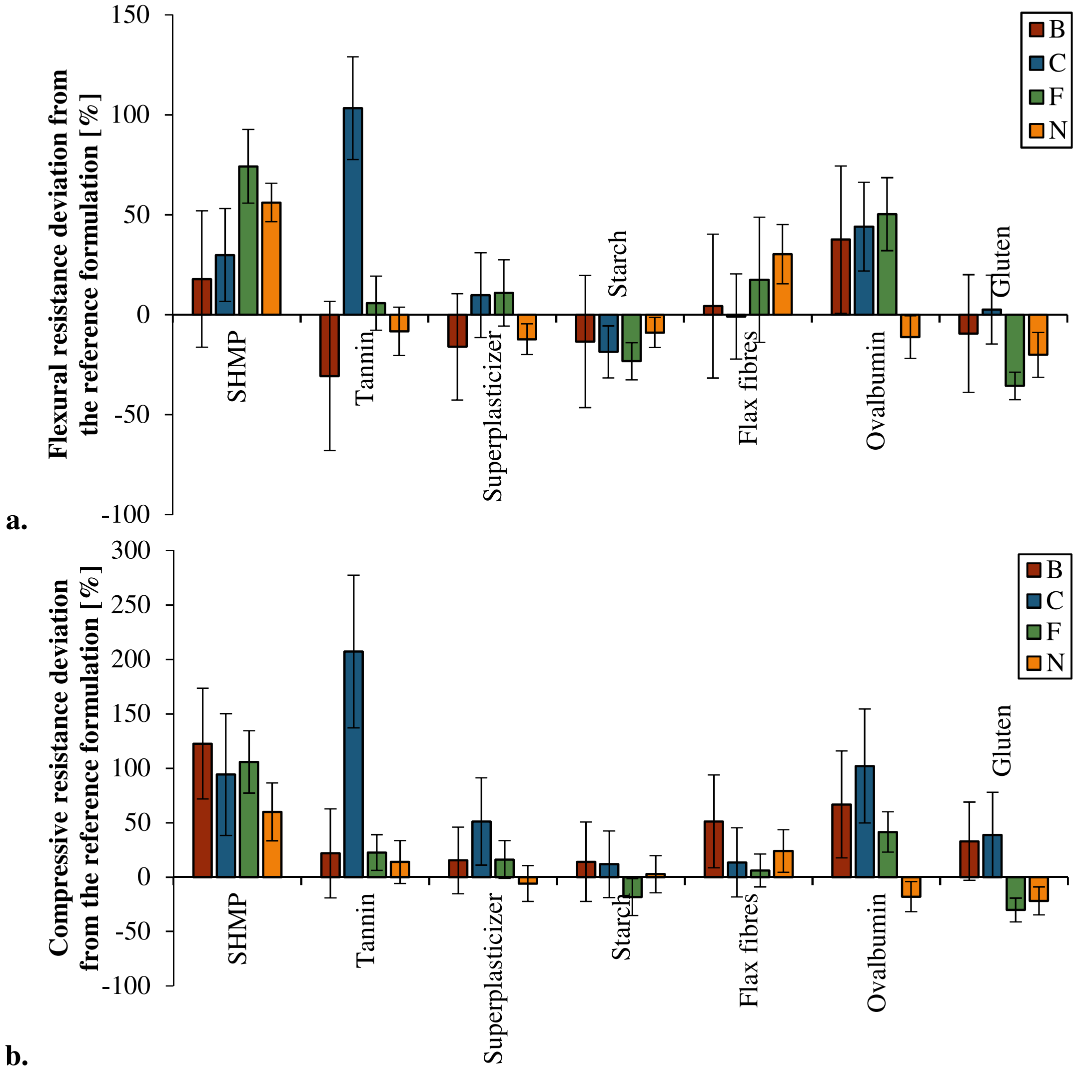


Figure 14. Deviation of (**a.**) flexural and (**b.**) compression resistances of admixed plasters from reference formulations' resistances.

The flexural and compressive strengths of hardened plaster specimens follow the same trend, given the large uncertainty of the results. Adding SHMP and, to a lesser extent, superplasticizer and tannin, enhances the mechanical performances of mortars. Thanks to their deflocculant properties, they densify the granular organization and reduce the kneading water requirement to ensure sufficient workability. Moevus *et al.* [34] also found a significant increase of the strength in the SHMP-admixed mortars resulting from an increase of the cohesivity of binding bridges, i.e. their cohesive force and their packing density.

Alhaik *et al.* [36] reported that adding starches to earthen mixes increased flexural and compressive strengths. It is not the case here. The compressive performances are similar to the reference, within the margin of error for the experiments. The flexural strengths, in contrast, have worsened.

The addition of flax fibres is more likely to enhance both the flexural and compressive strengths of mixtures, impact on flexural resistance being more limited. Millogo *et al.* [67] also witnessed the strength increase of fibre reinforced specimens. They explained that the incorporation of kenaf fibres reduced the propagation of cracks in the blocks, because of the good adherence of fibres to the clay matrix (shown on SEM micrographs), and therefore improved their mechanical properties. Other studies [27] did not report any influence for plant fibre additions.

Results on ovalbumin admixtures are consistent with those of Ouedraogo [21]: mechanical strength improved except for the N soil. The swelling behaviour of this type of earth is not compatible with the protein molecules, annulling and even counteracting their strengthening effects. On the contrary, performance improvement is detectable for the plasters made of earths B, C and F.

Gluten incorporation shows mixed results. The effect of this biopolymer seems to be dependent on the type of clay minerals involved. It has a clearly negative impact on soils F and N. The swelling behaviour of montmorillonite could also explain this finding for soil N. On the other hand, gluten addition is not critical as far as soils B and C are concerned. It even has a slight positive effect on compressive strength for both those soils thanks to the ability of gluten to strengthen the soil by holding the clay particles together. Gluten adheres to several clay platelets at once and connects them.

3.2.3. Shear stress

The bond of earthen plasters with their application substrate is of some importance. This bond depends strongly on the type of support that is coated. The tests performed here were for comparison between the different plaster formulations. Regarding adhesion strength of plasters without stabilization or addition, the results are presented in **Figure 15**.

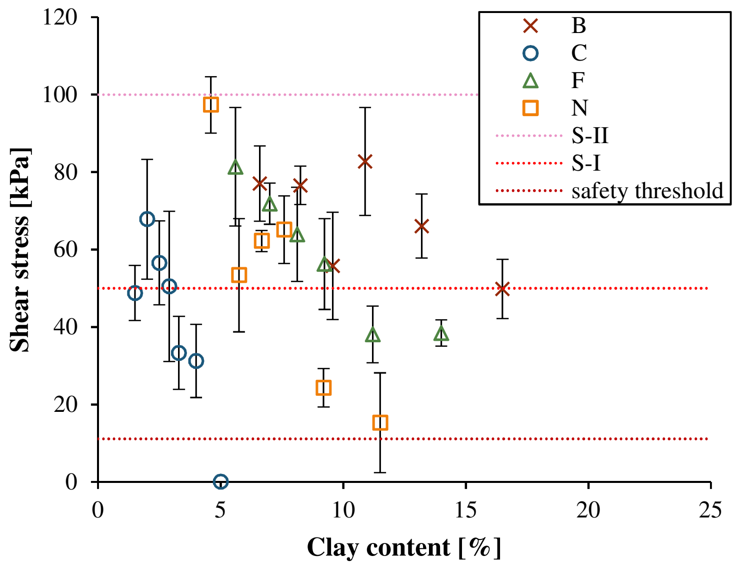


Figure 15. Shear stress of unstabilized plasters.

The shear stress values of mortars without admixture ranges from 0 to 97 kPa. It appears that most plasters achieve the minimum values of adhesive strength defined in the class S-I of the German standard [49] (≥ 50 kPa). However, none of them comply with the minimum value of the S-II class resistance (≥ 100 kPa). This is also the case for most plasters found in the literature [22], [43], [52] even though some mix designs are able to surpass the S-II minimum value [43], [44], [52]. In the work of Hamard *et al.* [42] and of Stazi *et al.* [45] the plasters were unable to reach the 50 kPa limit.

Another criterion can be chosen to validate the use of a plaster: as suggested by the FFB [57] and applied by Hamard *et al.* [42] and Stazi *et al.* [45], a plaster is of adequate adhesive strength if it is able to bear its own weight with a safety coefficient of 10. Considering a thickness of 60 mm, which could be the maximum thickness for in situ applications, and a density of 1887 kg/m³ (average value of the specimens studied), a threshold adhesive strength of 11 kPa (10×60.10^{-3} kg x 1887 kg/m³ x 9.81 m/s² ≈ 11 kPa) is assumed. In that case, all plasters except for 50%C and 50%N (not all specimens fulfilled the criteria) are validated.

No dependence of adhesive strength on clay content can be drawn from the results in **Figure 15**, regardless of the nature of the clay minerals.

Furthermore, the results can be seen to be highly dispersed. According to Hamard *et al.* [42], this is attributable to the heterogeneities of the substrate. In their study, Delinière *et al.* [52] highlighted the effect of surface preparation on the scattering of adhesive strength values. Discrepancies of results were also reported by Stazi *et al.* [45]. These elements are in accordance with the observations of the rupture profiles in our study: some specimens of a given mix tore off a part of the substrate when falling, while others did not.

Regarding each earth type individually, the relationship between the shear strength and the clay content of the plaster show similar patterns. Globally, as the clay content increases, the values of shear strength decrease. Hamard *et al.* [42] and Stazi *et al.* [45] point out the existence of an optimum clay content corresponding to the maximum shear strength for plasters. These optima are reported to be between 6 and 9% by Hamard *et al.* [42] depending on the earth used, and about 16% by Stazi *et al.* [45]. In our study, the existence of a maxima can only be considered for the quarry-fines-C-based plaster.

The effects of admixtures evaluated in comparison with an admixed formulation with the same earth to sand ratio are presented in **Figure 16**.

As far as the admixed formulations are concerned, the adhesive strength measured is between 28 and 138 kPa. 80% of these plasters achieve S-I class resistance (except soil B admixed with tannin, soil N with ovalbumin, soil C with SHMP and soils C, F and N with gluten) and three of them are in the S-II class (soil F admixed with the green superplasticizer and soils F and N with SHMP). However, all admixed formulations satisfy the criteria of the FFB [57].

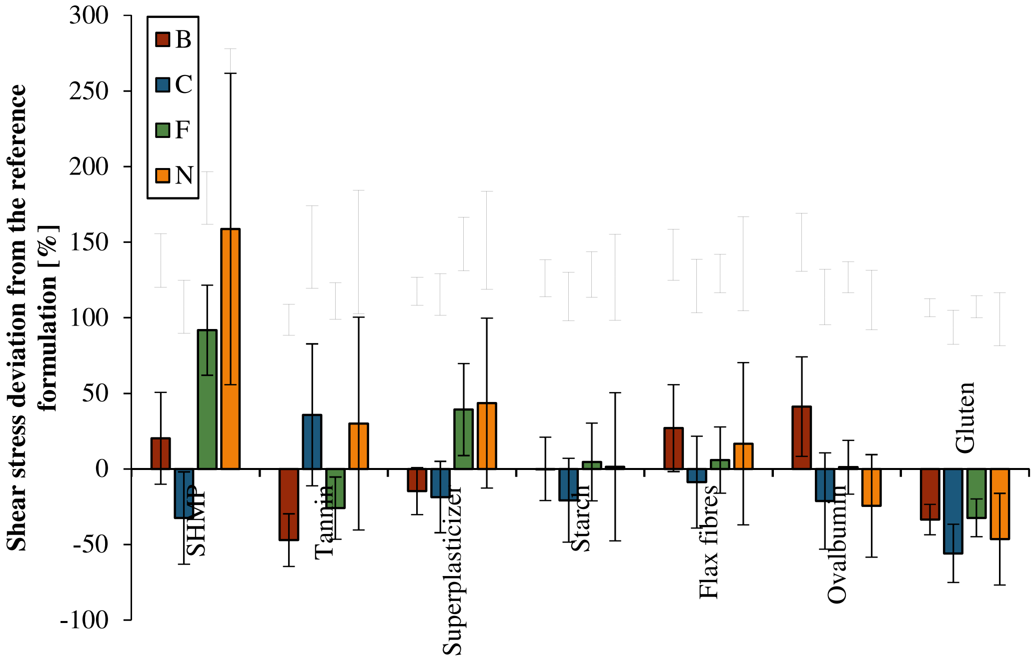


Figure 16. Effect of admixtures on shear stress.

The shear stress results, like those of the previous tests, do not reflect a clear trend regarding the effect of each admixture, independently of the clay minerals involved. Likewise, no soil exhibits improved performance regardless of the admixture. As for the shrinkage cracking results, the shear stress values depend strongly on the drying kinetics. On the one hand, if temperature and humidity conditions promote quick drying, coatings can detach slightly from the substrate, allowing a freer shrinkage and reducing the cracking but degrading the adhesion. On the other hand, when slower drying conditions are applied, plasters adhere strongly to the wall, which restrains shrinkage and allows greater cracking to occur.

Soil N responded rather positively to all deflocculants. Their water-reducing effect was more beneficial for this earth as swelling clays were involved. The smaller the amount of water required, the smaller the volumetric contraction of the specimens when drying and the better the adhesion to the substrate.

The shear strengths of the plasters with starch and flax fibre additions are of the same order of magnitude, given the large uncertainty of results, as those of their counterparts without admixtures. Similar findings have been reported for fibre reinforcement by Hamard *et al.* [42].

Ovalbumin exhibits quite negative impacts on adhesion except for soil B. The enhancement of mechanical properties of this soil counterbalance the negative effect of ovalbumin on shrinkage.

The results of the gluten-based specimens are consistent with the volumetric contraction findings. Gluten admixture accentuates the drying shrinkage effect, encouraging the coating to free itself from the wall constraint, i.e. to detach slightly from the substrate. The adhesion of the plaster to the wall is therefore degraded.

4. Conclusion

Tests on four earths with distinct geotechnical and mineralogical characteristics were conducted in the fresh and hardened states. Results show that the clay content is a key parameter for both the fresh properties and the mechanical performances of plasters. When the amount of clay involved in a plaster increased, regardless of its nature, the kneading water requirement increased to activate clay binding properties and to achieve the targeted consistence. The drying shrinkage is hence greater when the plaster is applied to a substrate, and leads to cracks or poorer wall-plaster adhesion. The mechanical performances are also impacted: the higher the clay content, the higher the resistance.

The quantity of clay is not sufficient to systematically explain the fresh state and mechanical behaviour of plasters by itself. The nature of the clayey phase can also be noted. The swelling potential and mineralogical compositions impact the performances of plasters, especially when organic admixtures are involved. The type of clay mineral must also play a role in defining compatibility with the wall, but no clear trend emerged from our results.

The potential of seven admixtures was evaluated: three deflocculants, one fibre addition and four cohesion strengtheners. Even though the deflocculants enabled a water reduction in mixes and also a mechanical improvement resulting from a more compact granular arrangement, the plasters still presented flaws regarding drying shrinkage: cracks were reduced thanks to SHMP and superplasticizer biopolymers but were not totally removed.

The cohesion strengtheners increased the amount of water needed to achieve the right consistency in the fresh state and no positive effect was recorded concerning drying shrinkage. However, when used with active clay minerals, regarding their affinity with water molecules, and in an adequate environment in terms of ionic strength and pH, they were able to enhance mechanical properties in the hardened state.

For use as plasters, only formulations with flax fibre additions are validated in accordance with Hamard *et al.* [42]. They demonstrated sufficient adhesion to their application substrate and, above all, no cracks appeared as they dried.

Once again, it is worth recalling that this work aimed to compare the effect of admixtures with each other and not to optimise their use. The prospects, in terms of optimisation, are hence very important.

Considering an application as interior coatings, it seems necessary to assess the hygrothermal properties of such materials. It can thus be noted that the high hygroscopicity of earthen components is conferred by the finer fraction of soils [11], [12], [15], [22]. Presumably, plasters with a high clay content are to be preferred. However, this ability to absorb and give off moisture decreases with the addition of plant particles [68]. The potential of other organic admixtures is hence worth exploring. Bio-based surfactants could limit the shrinkage cracks by reducing the surface tension of plasters. These elements will be further investigated in order to develop an eco-friendly, efficient plaster satisfying comfort and health requirements.

5. References

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