



# Article Effect of Different Surface Treatments on the Performance of Earth Plasters

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Abstract: Earth plasters have several advantages. Nevertheless, they are vulnerable when in contact with liquid water. For that reason, they have low durability when applied as an outdoor coating or in indoor areas with potential contact with water. In this study, the influence of six different surface treatments (traditional and innovative, based on raw materials and on waste) applied on a pre-mixed earth plaster, applied by a roller (r) or as a spray (s), was assessed. The treatments were: limewash (L), beeswax (BW), linseed oil (LO), graphene oxide dispersion (GO), water from paper immersion (WP) and water from gypsum plasterboard paper immersion (WPG). The application of L, BW and LO, despite the color change, improved the water resistance and the surface performance of the earth plaster (less than 80%-86%, 93%-98% and 97%-99% of mass loss from surface cohesion, from water erosion by dripping action and from dry abrasion, respectively, compared to the reference untreated plaster). However, the application of BW and LO had a negative effect on the hygroscopic capacity of the plaster (less than 28%–38% of water vapor adsorbed after 24 h and the MBV decreased 29%–50% compared to the reference plaster). Finally, the application of the remaining surface treatments did not significantly improve the characteristics of the plaster, having even worsened it in certain cases (more than 42%–149% of mass loss from water erosion, compared to the reference plaster). These results demonstrated that, among the treatments analyzed, the L, BW and LO treatments are the best options to apply on an earth plaster. In particular, the application of BW and LO are recommended in situations where it is necessary to improve water resistance and surface performance, and the hygroscopic capacity is not a conditioning characteristic, such as outdoor applications.

**Keywords:** clay binder; durability; hygroscopicity; paint system; plastering mortars; resistance to water

# 1. Introduction

Earth construction is one of the oldest building technologies in the world. As a result, there is a vast earthen heritage almost everywhere in the world. In 2011, according to the UNESCO World Heritage Earthen Architecture Programme (WHEAP) [1], earth constructions accounted for more than 10% of the World Heritage sites. Earth as a building material presents several advantages: it is a natural material, reusable when not chemically stabilized and easily recyclable, although non-renewable; and is an accessible and economical material, with low carbon emissions [2,3] and low environmental impact [4,5]. Very little energy is required for extraction, processing and production of earth materials [4]. Earth is often obtained as a construction waste [2], and the earth excavated for foundations



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**Copyright:** © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). and underground works can be used in earth construction, provided it has the necessary characteristics, instead of being sent to a landfill. In this way, it reduces the costs and energy for its transport and production [2], namely when used on site.

Earth plastering mortars are produced by mixing sieved earth (composed by different types and fractions of sand, silt and clay) with water; additional sand may also be added to control shrinkage, namely if the sand content of the earth is low. As with other plasters, earth plasters are applied indoors [6], coating walls and ceilings, and have several advantages: due to their high capacity to adsorb and desorb water vapor, they can act as passive moisture buffers, balancing the relative humidity (RH) of the indoor air, contributing positively to the comfort and health of buildings' occupants, including indoor air quality [7-10]. In fact, earth plasters can also act as air purifiers [11], being able to absorb and retain indoor air pollutants dissolved in water [12,13]. In different studies, raw earth presented values of carbon dioxide  $(CO_2)$  retention capacity [14,15] and earth plasters showed the ability to capture ozone [16], evidencing their potential for passive regulation capacity. Darling et al. [17], to assess the possibility of clay acting passively in the removal of pollutants, determined the perceived air quality resulting from the application of earth plaster coating gypsum plasterboard; the researchers concluded that the earth plaster improved this characteristic. Compared to a gypsum-based plaster, an earth-based plaster presented higher adsorption and desorption, and potential for CO<sub>2</sub> capture [11].

Nevertheless, unstabilized earth mortars are vulnerable to liquid water (from rain, floods or capillary absorption) [2,18,19], presenting poor resistance against water infiltration due to the hydrophilic nature of raw earth [20]. This can be seen as an advantage but also a disadvantage of earth mortars. When in contact with water, earth plastering mortars regain their plasticity due to their water solubility, allowing them to be easily repaired [21] or reused. On the other hand, this poor resistance may become a disadvantage when they come into accidental contact with water (water splashes from cleaning actions and the use of water, rainwater from an open window), since rapid erosion occurs and plasters can suffer significant damage [19].

Thereby, the application of earth mortars as renders, applied outdoors without the application of any type of protection or stabilization, is advised against [3]. However, a higher resistance to water could increase the durability and the applicability of earth mortars, namely as rendering mortars.

The addition of low binder contents (gypsum, lime or cement, among others) and/or biomaterials (natural oils, waxes, biopolymers or natural fibers, among others) in the formulation of earth plastering mortars or the application of a surface treatment to the hardened surface of the plaster could improve their poor water resistance [18,19,22–25]. However, the addition of low contents of binders, apart from increasing their embodied energy, makes it impossible to reuse the earth plasters just by mixing with water again; in that case, they need to be recycled [26].

In a more generic study, Vissac et al. [24] presented a compilation of some of the organic materials that can be used as earth mortar stabilizers and/or as surface treatments, such as linseed oil and shea butter, among others. González-Sánchez et al. [27] analyzed the positive influence of a vegetal origin gel, obtained by extracting the starch contained in rice, on the mechanical and waterproofing performance of earth mortars. The use of washi paper—a paper traditionally manufactured in Japan—is also referred by Vissac et al. [24] as being similar to recent techniques using clay and paper. Traditionally, in Japan, wastepaper is used in earth plasters, and different types of paper can be used, such as newsprint, packaging paper, and egg cartons, among others [24]. Finally, Vares et al. [28] analyzed the influence of different types of paper (glossy paper, egg cartons, printer paper and newsprint) on the sorption and the moisture buffering properties of paper plasters (composed by paper, glue and water, and clay added as filler); the researchers concluded that this type of plaster presents higher adsorption capacity than earth plasters.

Considering the requirements necessary for the application of earth plasters, it is necessary to take into consideration the influence of the additions and surface treatments on

the final characteristics of plasters. To extend the service life of buildings, more specifically of the plasters, and reduce their maintenance, different treatments are often applied to consolidate the surface [29]. The treatment of earthen plasters' surface should allow the consolidation and protection of that surface and different systems can be applied, namely by painting [3]. In earth construction, limewash was traditionally used [23]. For a long time, limewash was frequently applied as renders and plasters finishing on earth or stone based buildings walls [3]. Therefore, it is of the utmost importance to use options that, in addition to improving the technical performance (resistance to abrasion, erosion and to liquid water), are compatible with the plasters and the hole wall system in which they will be applied: not generating too high stresses or barriers to water vapor transport [3], without significantly affecting the mechanical and hygrothermal behavior of earth plasters and the comfort of the building occupants. Limewash should be applied in several layers, with three to four layers being recommended [13].

Apart from varnishes and paints, producing a superficial film when applied on a building material, there are other organic and mineral-based products used as finishes for earth plasters, which are mostly absorbed by the plaster, impregnating it to a certain depth [3]. Their viscosity influences the way they are applied. Usually, their application does not form a film on the plaster surface, does not significantly change its color or texture, and has the function of consolidation of its surface.

On the contrary [3], painting systems generally change the color of the plaster surface, completely covering its surface with a layer of very low thickness. Clay paints, lime paints and silicate paints are the most compatible type of paints to be applied. On the other hand, materials with low water vapor permeability, such as synthetic paints and vinyl wallpapers, among others, are not appropriate [12]. Ranesi et al. [30] evaluated the effect of two paint systems (based on vinyl and acrylic paints) on the moisture buffer capacity of an earth plaster, showing that in particular the application of acrylic paints is not recommended.

Few studies have been carried out on the protection of earthen renders against atmospheric agents [22,29]. Thus, it is also necessary to study the effect of surface treatments that can protect the earthen plasters but do not significantly impair their hygrothermal behavior.

Table 1 summarizes some of the surface treatments applied by different methods (by painting and spraying, among others) to earthen plasters and other earthen surfaces, and the characteristics analyzed in different studies. Both traditional and innovative treatments are included. The different studies are presented from the most natural to the most industrial surface treatments, and from those applied to earth plasters to those applied to other earthen surfaces.

The influence of the different surface protections by different application methods on earth plasters was analyzed by Faria and Lima [3]. These researchers concluded that the application of casein primer slightly darkened the plaster surface. However, when applied in excess, this change was more significant, giving a yellowish hue and leading to the formation of a film. This application allowed the consolidation of the plaster and did not block the water vapor adsorption and desorption capacity, but it did present limitations in terms of exposure to ultraviolet (UV) radiation and to water. Regarding the application of linseed oil, Faria and Lima [3] reported that there was a change in the color of the plasters' surface and a long drying time, with the evaporation of part of the applied oil, which generated an intense odor. This may limit its application on indoor plasters, due to the excessive release of volatile organic compounds (COVs). This application increased the surface cohesion of the plaster, increasing its resistance to shocks, but it was sensitive to UV radiation. As for water resistance, it mitigated the degradation of the plaster surface in the case of water runoff. Finally, Faria and Lima [3] noted that, when applied on earth plasters, limewash allowed the protection of its surface against abrasion wear and improved its water resistance without affecting its water vapor adsorption capacity.

Authors and Substrate Treated	Surface Treatments	Application Type	Characterization		
Faria and Lima [3] Earth plaster	<ul> <li>Casein primer</li> <li>Linseed oil</li> <li>Potassium silicate primer</li> <li>Clay paint</li> <li>Limewash</li> <li>Potassium silicate paint</li> <li>Casein paint</li> <li>Acrylic paint</li> </ul>	Different applications - Brush - Roller - Spray	Laboratory - Visual observation, color and odor - Adsorption and desorption capacity - Water vapor permeability - Ultraviolet (UV) radiation exposure - Water erosion resistance by dripping - Surface consolidation - Impact resistance - Dry abrasion resistance		
Ferreira [31] Earth plaster	<ul> <li>Linseed oil</li> <li>Casein primer</li> <li>Silicate primer</li> <li>Limewash</li> <li>Casein paint</li> <li>Silicate paint</li> <li>Acrylic paint</li> </ul>	Different applications - Brush - Roller - Spray	Laboratory - Water vapor permeability - Water absorption by pipe method (Karsten tube) - Water erosion resistance by dripping		
Santos et al. [23] Earth plaster applied on different masonry walls (hollow brick, concrete and adobe masonry walls)	<ul> <li>Linseed oil</li> <li>Aloe vera mucilage</li> <li>Pasta and rice cooking water</li> </ul>	Brush	<ul> <li>Laboratory and natural weathering <ul> <li>Visual observation and color</li> <li>Surface cohesion</li> <li>Surface hardness (by durometer and by sclerometer)</li> <li>Sphere impact test</li> <li>Ultrasonic pulse velocity</li> </ul> </li> </ul>		
Vares et al. [32] Earth plaster	<ul> <li>Fine finishing mortar with and without cellulose</li> <li>Casein paint</li> <li>Cellulose base coat</li> <li>Lime paint</li> <li>Casein base coat</li> </ul>	Not specified	Laboratory - Hygroscopic sorption - Water vapor permeability		
García-Vera et al. [33] Earth plaster	- Ethyl silicate	Nebulizer	<ul> <li>Laboratory</li> <li>Peeling test</li> <li>Water absorption (capillary and pipe method (Karsten tube))</li> <li>Durability tests (rainwater, HCl rain and H<sub>2</sub>SO<sub>4</sub> rain simulation) by dripping</li> <li>Mercury Intrusion Porosimetry (MIP)</li> <li>Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy (SEM/EDS) analyses</li> </ul>		
Parracha et al. [19] Earth plaster	<ul> <li>Water supplemented with iron</li> <li>Lysogeny broth medium</li> <li>Lysogeny broth medium supplemented with iron</li> <li><i>Escherichia coli (E. coli)</i> culture supplemented with iron</li> <li><i>E. coli</i> culture expressing protein from the ferritin family (Dps), supplemented with iron</li> </ul>	Micropipette	Laboratory - Visual and microscopic observation - Surface hardness by durometer - Ultrasound transmission test - Surface cohesion - Water drop test		
Stazi et al. [22] Earth plaster	<ul> <li>Nanostructured emulsion of silicon based molecules</li> <li>Nanostructured suspension of titania and silica</li> <li>Aqueous silane-siloxane emulsion</li> <li>Aqueous beeswax emulsion</li> </ul>	Brush	<ul> <li>Laboratory</li> <li>Surface color test</li> <li>Water vapor permeability</li> <li>Wettability (contact angle)</li> <li>Water absorption by pipe method (Karsten tube)</li> <li>Water erosion resistance by dripping and by pressure spray</li> </ul>		

**Table 1.** Substrates, surface treatments and characterization analyzed in different studies on earthen plasters and surfaces.

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Authors and Substrate Treated	Surface Treatments	Application Type	Characterization
Ribeiro et al. [34] Adobe and rammed earth	<ul> <li>Linseed oil</li> <li>Beeswax prepared in a solution of 3% turpentine</li> <li>Commercial water repellent (siloxane)</li> </ul>	Brush	Laboratory - Water absorption by contact sponge test
Aguilar et al. [35] Earthen surface	- Chitosan biopolymer	Briefly dipping	Laboratory <ul> <li>Water contact angle</li> <li>Water erosion resistance by dripping</li> <li>Compression, split and three point bending test</li> </ul>
Elert et al. [29] Rammed earth	<ul> <li>Ethyl silicate</li> <li>Nanolime</li> <li>Nanosilica</li> <li>Alkaline activation</li> <li>Bacterial biomineralization</li> </ul>	Different applications - Brush - Spray	<ul> <li>In situ and Laboratory</li> <li>X-ray diffraction analysis</li> <li>Thermogravimetric differential scanning calorimetry (TGA-DSC)</li> <li>Scanning Electron Microscopy: textural and compositional characteristics</li> <li>Total number of culturable bacteria- Colorimetric characterization</li> <li>Water resistance: water absorption and weight loss</li> <li>Static contact angle- Water vapor transmission rate</li> <li>Total pore volume and pore size distribution</li> <li>Compressive strength and drilling resistance</li> <li>Peeling test</li> </ul>
Elert et al. <mark>[36]</mark> Earthen surfaces	<ul> <li>Ethyl silicate</li> <li>Nanolime</li> <li>Nanosilica</li> <li>Alkaline activation</li> <li>Bacterial biomineralization</li> </ul>	Different applications - Brush - Spray	Laboratory         -       Scanning Electron Microscopy         -       Surface observations (portable digital microscope and 3D microscopy)         -       Colorimetric characterization         -       Surface roughness         -       Air permeability         -       Total pore volume and pore size distribution         -       Surface hardness (HLC) and cohesion         -       Surface wettability properties (water contact angle and micro drop absorption times)

Table 1. Cont.

Cactus and vegetable mucilage can also be used as a surface treatment for earth plastering [37]. Ben Guida [38] referred to the application of a decoction of néré pods/bark and shear water as a surface treatment of earth plasters in Tata Somba homes, in Benin and Togo.

Santos et al. [23] compared the effect of different natural surface treatments and concluded that there was a loss of the plaster surface material during application and that the application of linseed oil, compared to the other treatments applied, improved impact resistance and surface hardness and cohesion. However, its application promoted a change in the color of the plaster's surface.

García-Vera et al. [33] concluded that the application of ethyl silicate as a surface treatment consolidated the earth plaster surface and promoted an improvement in water absorption properties, namely improving the resistance to acid rain.

Parracha et al. [19], analyzing the effect of biotreatments on an earth mortar, concluded that all biotreated specimens improved the water resistance, creating a slight water-proofing effect, except all of the water supplemented with iron treatments. The biotreatments did not

improve the surface hardness of the earth mortar and only the *E. coli* culture supplemented with iron treatment improved the surface cohesion.

Stazi et al. [22] verified that the application of the different surface treatments promoted a slight change in color on the plaster's surface compared to the untreated plaster. In general, surface treatments promoted an improvement in the contact angle between the water droplet and the plaster surface. The silane–siloxane and silicon nano-particles treatments were the only ones to make the plaster water repellent, with a contact angle of about 110°, and remained the same over time. These treated plasters also showed no erosion after water dripping action, whereas silane–siloxane application was the only one not showing erosion after the action of an intensive spray.

Ribeiro et al. [34] studied adobe and rammed earth specimens surface treated with linseed oil, beeswax and a commercial water repellent, concluding that these treatments formed a hydrophobic barrier, significantly reducing the water absorption of the specimens.

Aguilar et al. [35] concluded that the application of chitosan biopolymer improved the water resistance of earth samples, making the surface moderately water repellent, with a contact angle close to 90° and virtually no drip erosion.

Elert et al. [36] in their study about the surface effect of several consolidation treatments on earthen surfaces concluded that none of the consolidants presented optimal performance on the earthen surface, having yet concluded that, for successful consolidation treatments, the penetration of the consolidants is a key parameter. In another study, Elert et al. [29] proved that due to the extreme heterogeneity of earth-based materials, in situ evaluation of the surface treatments' efficacy is very challenging. For this reason, laboratory studies are essential, complementing in situ studies.

Analyzing the results obtained by these different studies, the application of casein primer, linseed oil, limewash, biotreatments and other types of treatments, such as ethyl silicate, silane–siloxane, silicon nano-particles or chitosan biopolymer, presented good performance as surface treatments for earth plasters and other earthen surfaces.

Graphene-based materials are being studied as an additive in several areas, namely for protection of stone-based cultural heritage [39]. However, the graphene would be responsible for increasing the environmental impact, which is why, due to the increasing importance of using sustainable materials, the development of environmentally friendly graphene sources and synthesis methods is currently being investigated [40]. Gallo Stampino et al. [40] analyzed the influence of adding aqueous dispersion of graphene (0.01 wt%, 0.05 wt% and 0.1 wt% of the total of the solid mass) on earth plasters and concluded it increased the cohesion of the mortars, improving their adhesive performance. No studies on surface treatments of earthen plasters with graphene oxide were found.

The aim of the present study was to evaluate the influence of different surface treatments, and the type of application, on the characteristics of an earth plaster. Therefore, limewash (L), beeswax (BW), linseed oil (LO), graphene oxide dispersion (GO), water from paper immersion (WP) and water from gypsum plasterboard paper immersion (WPG) were applied on the surface of a pre-mixed earth plastering mortar, by a roller (for all cases) or by spraying (only for some treatments), and the effect was compared with the same but untreated plaster.

#### 2. Materials and Methods

#### 2.1. Earth Plaster and Specimens

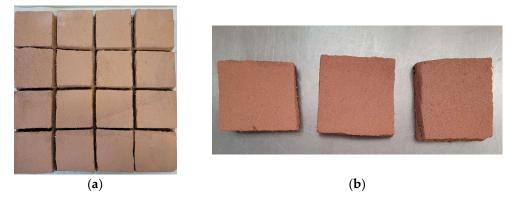
A pre-mixed earth mortar was used, composed of clayish earth, additional sand of 0–2 mm and straw fibers (<10 mm), produced by Embarro Portugal. The exact proportions of each constituent are unknown. However, the same mortar but from a different batch was analyzed by Faria et al. [41] and Santos et al. [42,43], showing that the clayish earth was illitic [41–43]. The mortar was mixed mechanically in a mixer, trying to reproduce the method used on a construction site: the dry pre-mixed earth mortar was placed in a bucket; mixing water was added slowly, and the mortar was mixed for approximately 8 min; a

paused period was used to remove the mortar adhering to the sides of the bucket and to add the remaining mixing water; finally, the mortar was mixed for another 3 min.

The mixing water added was defined as the minimal amount necessary to ensure good workability of the mortar, as evaluated by an experienced technician. The water content, determined by the difference in weight between the dry and wet mass of mortar samples, was 14%. The mortar presented a flow of 116 mm (based on EN 1015-3 [44]) and a wet bulk density of 1.9 kg/dm<sup>3</sup> (based on EN 1015-6 [45]). In another studies [41–43], the same earth mortar but from a different batch presented a flow of 153.5  $\pm$  28.5 mm with a water content of 17.5  $\pm$  2.5%.

The plastering mortar from the same producer but from a different batch was characterized elsewhere [43], being mixed with a similar procedure and water content (15%) and presented the following characteristics: flow of  $125 \pm 8$  mm; wet bulk density of  $2.03 \pm 0.02$  kg/dm<sup>3</sup>; linear shrinkage of  $0.2 \pm 0.1\%$ ; dry bulk density of 1.8 kg/dm<sup>3</sup>; dynamic modulus of elasticity of 4267 N/mm<sup>2</sup>; flexural and compressive strength of 0.25 N/mm<sup>2</sup> and 0.96 N/mm<sup>2</sup>, respectively.

To simulate a plaster, a mortar layer of 20 mm thickness was applied on the back of ceramic tiles with dimensions 450 mm  $\times$  450 mm. Before the application of the earth mortar layer, the back of the ceramic tiles was sprayed with water. The specimens were left for 6 years in environmental laboratory conditions. These plastered tiles were dry cut with an angle grinder into smaller specimens with dimensions of 110 mm  $\times$  110 mm, approximately 106.5  $\pm$  10.5 mm (Figure 1a). The applied plaster layer detached intact from the tiles during the cutting of the specimens (Figure 1b), facilitating the use of these specimens in the present study. Additional specimens of approximately 20 mm  $\times$  110 mm were also prepared for the contact angle test.



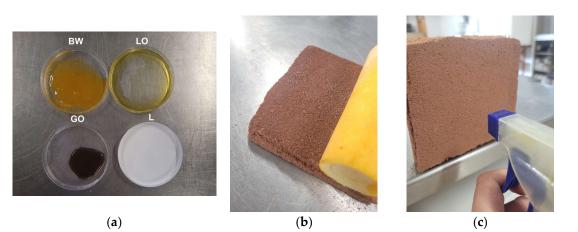
**Figure 1.** Earth plaster specimens: (**a**) cut from the plaster applied on a large ceramic tile; (**b**) final specimens, without the ceramic tile substrate.

#### 2.2. Surface Treatments and Applications

Six different surface treatments were applied on the earth plaster: limewash (L), beeswax (BW), linseed oil (LO), graphene oxide dispersion (GO), water from paper immersion (WP) and water from gypsum plasterboard paper immersion (WPG). L presented a white color, BW and LO had a yellowish color and GO had a black color (Figure 2a). WP and WPG, being water-based products, had no color.

The limewash (L) was obtained by diluting 250 mL of lime putty from Maxical, Portugal, in 375 mL of water (in volume). The beeswax (BW) used is from Margem, Portugal, and the linseed oil (LO) is from Tintinhas, Portugal. These surface treatments had a higher viscosity, especially the BW, compared to the other treatments.

The graphene oxide treatment (GO) was obtained through ultrasonic exfoliation of graphite oxide in water, and a dispersion with a concentration of 0.5 mg/mL in water was applied [39]. The physical-chemical characterization of the material can be accessed in another study [39].



**Figure 2.** Treatments and application methods: (**a**) beeswax (BW), linseed oil (LO), graphene oxide (GO) and limewash (L) treatments; (**b**) application by roller; (**c**) application by spray.

The use of water from the immersion of discarded writing paper (WP) and paper from discarded gypsum plasterboard (WPG), after it has been separated from the gypsum-based waste, aimed to evaluate the possible release of cellulose present in the paper into the water. The WP was obtained by placing 26.2 g of paper cut into small dimensions in 300 mL of water for 8 days. For the WPG, the procedure was similar, with 25.6 g of paper being placed in 200 mL for 5 days. However, the day the treatment was applied it was necessary to add an additional 100 mL of water, as all of the water had already been absorbed by the paper. In order not to use water in excess for the paper's dilution, for each different type of paper, only the amount of water necessary to cover the entire volume of cut paper placed in an equal container was added. After that, the paper (from discarded writing paper and gypsum plasterboard) was drained and the water collected for use.

The treatments were applied through two application methods: by sponge roller (r) and by spraying (s) (Figure 2b,c). The roller applications were carried out with the specimens placed horizontally, with only one coat but passing the roller 10 times in each orthogonal direction. The exception was the limewash (L) application: 3 coats were applied, each coat passing 20 times with the roller in each orthogonal direction. This traditional surface treatment, made with a dilution of lime putty in water, requires the application of more layers to adequately coat the plaster's surface. For application by spray, the specimens were placed vertically during spraying to better simulate a real application. The spray was 2–4 cm from the surface and each coat took about 1 min, for the spray and roller. However, there was a loss of material from the plaster surface due to material flow, which could result in the accumulation of material at the base of the specimen. During the surface treatment application by roller, a loss of material from the plaster surface was verified, being deposited on the roller. This loss of material was most evident in the water-based treatment application.

It was not possible to apply the BW and L by spraying due to the high viscosity of the BW, which made application difficult, and the presence of lime grains on L, which clogged the sprinkler and did not allow for a uniform application of the product, respectively. In another studies, Stazi et al. [22] and Ribeiro et al. [34] applied an aqueous beeswax emulsion and beeswax prepared in a solution of a 3% of turpentine, respectively (Table 1). In the present study, beeswax (BW) was used directly without any type of dissolution since it was possible to apply the product by roller. However, a dissolution of the product would possibly allow its application by spray.

Table 2 summarizes the treatments and application methods, as well as the total mass of products applied. As can be seen, different amounts of material were applied. The application by roller promoted the application of less material, except for the application of WP. In addition, the characteristics of the surface treatment itself led to the application of different amounts of the different treatments applied. The curing conditions and period were 20  $\pm$  6 °C and 54  $\pm$  2% RH and 2 days, respectively, for all specimens.

Specimen	Surface Treatment	Application Method	Mass Applied Per Surface [g/dm <sup>2</sup> ]
Lr	Limewash	Roller	$5.0 \pm 1.0$
BWr	Beeswax	Roller	$2.2\pm0.4$
LOr LOs	Linseed oil	Roller Spray	$\begin{array}{c} 1.1 \pm 0.2 \\ 2.7 \pm 0.1 \end{array}$
GOr GOs	Graphene oxide	Roller Spray	$\begin{array}{c} 1.1 \pm 0.2 \\ 2.7 \pm 0.3 \end{array}$
WPr WPs	Water paper	Roller Spray	$egin{array}{c} 1.8 \pm 0.3 \ 1.9 \pm 0.1 \end{array}$
WPGr WPGs	Water paper from gypsum plasterboards	Roller Spray	$egin{array}{c} 1.1 \pm 0.1 \\ 2.0 \pm 0.5 \end{array}$

Table 2. Surface treatments, application method and mass of treatment product applied.

## 2.3. Methods

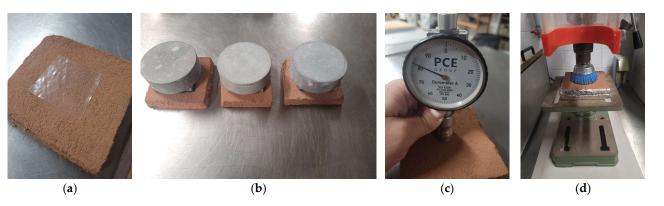
All tests were performed on the untreated (reference) plaster specimens and the ones treated with each product and application. The result of each test on the plaster (reference or with surface treatment) is the average, and the respective standard deviation, of the results of three specimens. The exception is in the determination of the contact angle, for which only one specimen of the selected treatments was used.

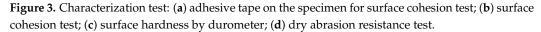
#### 2.3.1. Effect of Surface Treatments on the Plaster's Aesthetics and Odor

The color change of the plaster caused by the application of different surface treatments, as well as any other changes to the specimens caused by the application of the treatments, were assessed through visual observation of the specimens. The possible intensification of the odor promoted by the application of the different surface treatments was also evaluated.

### 2.3.2. Surface Cohesion and Hardness

Surface cohesion was determined based on the procedure defined by Drdácký et al. [46] and the adaptations defined by Faria et al. [41], Santos et al. [43] and Parracha et al. [19]. After weighing, a 50 mm  $\times$  50 mm adhesive tape was placed on the specimens (Figure 3a). A resilient material (neoprene tape) was placed on the adhesive tape and pressed with constant intensity imposed by a weight of 2 kg placed on top, without touching the plaster surface, for 2 min (Figure 3b). Finally, the weight and the resilient material were removed, followed by the adhesive tape, which was observed to assess the particles attached and it was weighed on a balance with 0.001 g of accuracy.





The loss of surface cohesion of the plaster is defined by the increase in mass of the adhesive tape. Therefore, the greater the loss of mass, the lower the surface cohesion of the plaster.

Surface hardness was evaluated on the surface of the specimens based on ASTM D2240 [47] with a Shore A durometer (Figure 3c), with 12 readings being taken on each specimen.

#### 2.3.3. Dry Abrasion Resistance

Dry abrasion resistance was determined based on the DIN 18947 [48] by the visual observation of the plaster's surface and the loss of mass (before and after the test) of specimens, assessed with a precision of 0.001 g, after 20 rotations of a circular nylon brush (from Wolfcraft GmbH, Kempenich, Germany, the medium brush used by Faria et al. [41]), with a diameter of 65 mm, pressed on top of the surface of the plaster with a constant pressure applied by a weight of 2 kg [41,49] (Figure 3d).

## 2.3.4. Water Vapor Adsorption and Desorption

Water vapor adsorption and desorption of the plaster, in a 24 h cycle for each phase, was determined based on DIN 18947 [48] with adaptations defined by Lima et al. [50]. The lateral and base surfaces of the specimens were sealed with aluminum tape, leaving only the upper surface exposed, being the exchange surface. The specimens were pre-conditioned at  $23 \pm 0.5$  °C and  $50 \pm 5\%$  RH in a climatic chamber (Aralab Fitoclima 300EDTU, Rio de Mouro, Portugal). Then, the specimens were weighed on a balance with 0.001 g of accuracy to determine the initial mass. The adsorption phase began when the RH in the chamber was changed from 50% to 80%, maintaining the temperature. The specimens were weighed after 1 h, 3 h, 6 h, 12 h and 24 h. Then, the desorption phase began, changing the RH from 80% to 50%, maintaining the temperature, and weighing the specimens at the same intervals. The adsorption curves were plotted with time on the abscissae and the mass variation per exposed surface on the Y axis. The results of the first 12 h of adsorption allowed for classifying the plasters according to the DIN 18947 [48] classes.

#### 2.3.5. Moisture Buffering Value

The influence of different surface treatments on the hygroscopic dynamic response of the earth plaster was evaluated based on ISO 24353 [51] for the "middle humidity level" conditions. The specimens were pre-conditioned at  $23 \pm 0.5$  °C and  $63 \pm 5\%$  RH in the climatic chamber (mass difference after 24 h less than 0.1 g). The specimens were exposed to  $75 \pm 5\%$  RH for 12 h (adsorption phase), followed by exposure to  $50 \pm 5\%$  RH for 12 h (desorption phase), maintaining the temperature. In the adsorption phase, the specimens were weighed after 3 h, 6 h, 9 h and 12 h, while in the desorption phase, the specimens were weighed at 0 h and 12 h. This daily cycle was repeated four times.

The Moisture Buffering Value (MBV) of the plasters (reference and with surface treatment) was determined by the NORDTEST [52], considering only the last three cycles.

#### 2.3.6. Water Drop Absorption and Contact Angle

To evaluate the water drop absorption by the plaster (both in its untreated form as the reference and with surface treatment), nine water drops were applied to various areas of the specimen. The time it took for each water droplet to be absorbed was recorded, with slower absorption rates by the plaster indicating better performance.

The contact angle was measured using an optical tensiometer Theta Flex Plus (TF300-Plus) from Biolin Scientific, utilizing the accompanying OneAttension software. An 8  $\mu$ L droplet of demineralized water at 25 °C was placed perpendicularly on the horizontal surface of a treated plaster specimen. Based on the preliminary water drop absorption results, the contact angles were measured only for treated plasters that exhibited good water drop resistance. These measurements were taken between 15 to 60 s, depending on the behavior of the surface treatments. More details are provided with the results. The plaster surface was classified as lowly wettable, i.e., hydrophobic or water repellent, when the contact angle exceeded 90°. Conversely, if the contact angle was below 90°, the surface was deemed highly wettable, i.e., hydrophilic [22].

# 2.3.7. Water Erosion by Dripping

Water erosion by dripping was assessed using the Geelong method, defined by the New Zealand standard NZS 4298 [53], with the necessary adaptations to the earth plasters according to Lima et al. [50]. The specimens were tested with a slope of 2/1 ( $\approx$ 63°) and approximately 16 mL of water (considering the necessary adjustment for specimens with 20 mm of thickness) dripped onto them from a height of 400 mm. The specimens were visually observed and weighed before and after the test (when dry) to assess the material loss due to the "slip" effect of water erosion. The maximum depth on the impact zone caused by the "splash" effect of the water was also evaluated [50].

#### 3. Results and Discussion

## 3.1. Effect of Surface Treatments on the Plaster's Aesthetics and Odor

The earth plaster specimens with the application of different surface treatments applied by the roller (r) or by spraying (s) are shown in Figure 4. The color of the plaster's surface is an important characteristic for the aesthetics of the building. In general, changes in color in earth plasters are seen as negative, but that cannot always be the case. As expected, in the present study, the limewash (Lr) promoted the higher color change on the surface of the earth plaster, making it white, followed by the beeswax treatment (BWr), lightening the surface of the plaster, making it whitish. The application of linseed oil (LOr and LOs) caused a slight darkening of the surface despite maintaining the color of the earthen plaster. The plaster surface treated with graphene oxide by spray (GOs) presented some gray nuances on the plaster surface. Finally, there was no significant change in color with the application of graphene oxide by roller (GOr), water from paper immersion (WPr and WPs) or water from gypsum plasterboard paper immersion (WPGr and WPGs).



**Figure 4.** One specimen of the untreated (REF) and each of the differently treated plasters: linseed oil (LO), beeswax (BW), graphene oxide (GO), water from paper immersion (WP) and from gypsum plasterboard paper immersion (WPG), linewash (L), applied by roller (r) and by spraying (s).

In the plaster treated with BW, a film formed on the surface of the plaster, just as L presented a thin layer on the plaster. The remaining treatments seemed to impregnate the plaster surface. The application of surface treatments (by spray or by roller) gave the earth plaster a matte aesthetic, except for the application of beeswax, which gave it a semi-glossy aesthetic.

The application of linseed oil caused an intensification of odor, while in the application of beeswax, this intensification was slight. The remaining treatments did not present an odor.

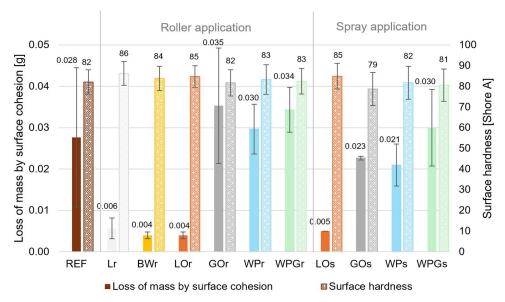
Comparing the application by roller and by spray, it appeared that by roller was more uniform throughout the specimen. In the case of spray application, once the specimens were placed vertically, there was an accumulation of the surface treatment on the lower face, due to the runoff of the products (LOs, Figure 4). As also mentioned by Faria and Lima [3], due to the viscosity of the LO (lower than that of BW), the application by spray is more difficult, which is why the application of the product was not uniform across the surface, with areas where more product accumulated, in the present study. These researchers [3] also explained that applying an excess of linseed oil on an earth plaster did not promote the formation of a film on the surface; it only prolonged the drying time of the product and intensified the odor, darkening and shining the surface of the earth plaster. In the present study, an intensification of the odor and a darkening of the surface was also observed, especially in areas where there was an accumulation of product on the plaster surface (Figure 4).

Stazi et al. [22], when evaluating the influence of an aqueous beeswax emulsion as a surface treatment of an earth plaster (Table 1), found a slight change in the color of the plaster's surface. In the present study, the color change of the earth plaster surface with the application of beeswax was more significant. The greatest influence on the color of the plaster surface seen in the present study can be justified by the higher concentration of treatment applied and by the thickest viscosity of the treatment used, compared with the aqueous beeswax emulsion applied by Stazi et al. [22].

When Santos et al. [23] analyzed the influence of different surface treatments on an earth plaster similar to the one analyzed in the present study, it was also concluded that the application of linseed oil promoted a darkening of the plaster's surface. Other waterbased surface treatments analyzed by Santos et al. [23]—aloe vera mucilage and paste and rice cooking water—presented a more liquid viscosity and did not show significant color changes. The present study followed the same trend: the application of linseed oil darkened the earth plaster's surface, while the water-based treatments did not cause a color change.

#### 3.2. Surface Cohesion and Hardness

Figure 5 presents the loss of mass by surface cohesion and the surface hardness of the earth plaster with and without the application of surface treatments. Two different behaviors promoted by surface treatments were observed: one that improved the surface cohesion of the plaster, while the other maintained or slightly decreased the surface cohesion. These groups are not seen for the surface hardness of the plaster.



**Figure 5.** Loss of mass by surface cohesion (full) and surface hardness (with dots) of plaster with and without different surface treatments: average and standard deviation. Notation: reference (REF, in brown), limewash (L, in light grey), beeswax (BW, in yellow), linseed oil (LO, in orange), graphene oxide (GO, in grey), water from paper (WP, in blue) and from gypsum plasterboard paper immersion (WPG, in green); applied by roller (r) and spraying (s).

It is possible to observe that the application of limewash (Lr), beeswax (BWr) and linseed oil (LOr and LOs) promoted a decrease in the loss of mass by surface cohesion (0.022–0.024 g), compared to the reference plaster without surface treatment (less 80%–86%, compared to REF). This shows an improvement of the plaster surface cohesion with these treatments and can be related to the good impregnation of these surface treatments and the formation of a layer/film on the specimen surface (Lr and BWr), compared to the others. The presence of air lime (L) also promoted better surface consolidation.

The application of the other treatments by roller (GOr, WPr and WPGr) promoted a slight increase in the loss of mass by surface cohesion (around 0.002–0.007 g) and, consequently, a loss of surface cohesion, compared to the reference plaster (more 7%–28%). In the case of spraying, the application of these treatments decreased (around 0.005–0.007 g for GOs and WPs, less 18%–24%) or slightly increased (around 0.002 g for WPGs, more 8%) the loss of mass by surface cohesion. In general, it can be considered that the surface cohesion of these treatments, which, when applied, may have promoted the detachment or loss of some plaster particles from the surface, as already mentioned in Section 2.2.

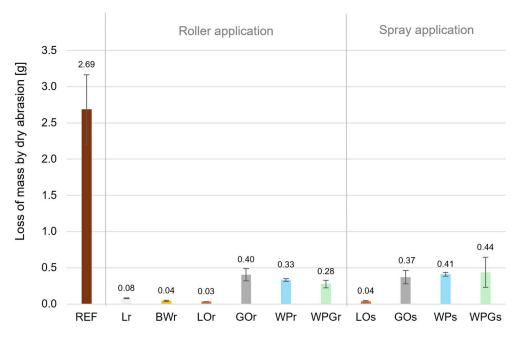
The surface hardness of the plaster remained similar after the application of different surface treatments, compared with the reference plaster, with all of the surface treatments within a range of 7 Shore A, presenting the results of 79–86 Shore A. As shown in Figure 5, a slight increase in the surface hardness of the plaster can be observed with the application of limewash (Lr), beeswax (BWr) and linseed oil (LOr and LOs). This may be related to the good impregnation of these surface treatments and to the formation of a layer or film on the plaster surface (Lr and BWr), compared to the other water-based treatments, which can justify the improvement of the surface consolidation of the plaster, as already mentioned, and, consequently, the improvement of its surface hardness.

Santos et al. [23], when evaluating the effect of the application of linseed oil and pasta and rice cooking waters on a similar earth plaster under laboratory conditions, concluded that the application of linseed oil and rice cooking water maintained the surface cohesion of the plaster, while the application of pasta cooking water promoted a slight decrease in surface cohesion. As for the surface hardness, Santos et al. [23] concluded that there was a slight increase in this characteristic with the application of the treatments, which was a little more evident in the case of linseed oil. In the present study, the surface hardness followed the same trend: the application of LO, WPr and WPGr treatments promoted a slight increase in this characteristic, being more evident for LO (applied by roller and by spray). For surface cohesion, in the present study, the application of linseed oil presented a significant improvement in this characteristic, which may be related to the good impregnation and a possible greater amount of treatment product applied (compared to that carried out by Santos et al. [23]).

#### 3.3. Dry Abrasion Resistance

Figure 6 presents the loss of mass by dry abrasion, showing that the application of the different surface treatments promoted a decrease of loss of mass by dry abrasion. This is a good indicator, since the decrease in loss of mass indicates an increase in the dry abrasion resistance of the plaster.

Two groups of treatments are highlighted: one promoting a very good resistance to abrasion of the treated plasters and another doubling the resistance in comparison to the reference plaster. The limewash (Lr), the beeswax (BWr) and the linseed oil (LOr and LOs) treatments show the lowest loss of mass by dry abrasion (less 97%–99%, compared to reference plaster), which means a significant improvement in the resistance to dry abrasion. The other treatments promoted a decrease of 84%–90% of loss of mass by dry abrasion, compared to REF. This approximately doubled the resistance provided by the plaster.



**Figure 6.** Loss of mass by dry abrasion of plasters with and without surface treatments: average and standard deviation. Notation: reference (REF, in brown), limewash (L, in light grey), beeswax (BW, in yellow), linseed oil (LO, in orange), graphene oxide (GO, in grey), water from paper (WP, in blue) and from gypsum plasterboard paper immersion (WPG, in green); applied by roller (r) and spraying (s).

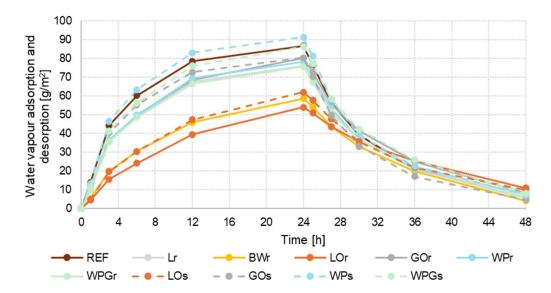
The dry abrasion resistance results followed the trend observed for the surface cohesion of plasters (Section 3.2): the treatments with Lr, BWr, LOr and LOs promoted better surface cohesion, as well as the dry abrasion resistance of the plaster, compared to the reference plaster (without treatments) and to the other surface treatments analyzed.

The conclusions of Faria and Lima [3], referring to the improvement of the abrasion resistance of earth plasters with application of limewash, corroborate the results with limewash in the present study.

The DIN 18947 [48] defines two different classes for classifying earth plasters according to their dry abrasion resistance: S I  $\leq$  1.5 g and S II  $\leq$  0.7 g. All treated plasters analyzed in the present study can be classified as S I and S II class, as they presented a loss of mass by dry abrasion lower than 1.5 g and 0.7 g, respectively. The exception was the REF plaster, which cannot be classified according to these classes because it presented a loss of mass by dry abrasion higher than the defined limits.

# 3.4. Water Vapor Adsorption and Desorption

Figure 7 presents the water vapor adsorption and desorption of the plastering mortar with and without surface treatments. Mainly, two groups of plaster specimens can be observed concerning adsorption, while all of the specimens presented a good desorption capacity, releasing almost all of the previously adsorbed moisture after 48 h. The group with better adsorption capacity had a similar to even slightly higher adsorption than the reference (untreated) plaster, which is very positive. The treated plasters of this group will provide a strong contribution for acting as passive moisture buffers when applied as a coating on indoor walls and ceilings. In this group, some treated plasters presented a slightly lower adsorption, but similar (less  $11 \text{ g/m}^2$ ) after 24 h, compared to the REF plaster; therefore, these treatments did not significantly influence the adsorption capacity of the earth plaster.



**Figure 7.** Water vapor adsorption and desorption of plaster with and without different surface treatments. Notation: reference (REF, in brown), limewash (L, in light grey), beeswax (BW, in yellow), linseed oil (LO, in orange), graphene oxide (GO, in grey), water from paper (WP, in blue) and from gypsum plasterboard paper immersion (WPG, in green); applied by roller (r, full line) and spraying (s, dashed line).

The reference earth plaster without surface treatment presented water vapor adsorption of 87 g/m<sup>2</sup> after 24 h. The application of linseed oil (LOr and LOs) and beeswax (BWr) decreased the water vapor adsorption capacity of the plaster: after 24 h, the plaster with BWr, LOr and LOs adsorbed only 59 g/m<sup>2</sup>, 54 g/m<sup>2</sup> and 62 g/m<sup>2</sup>, respectively (less 28%–38% compared to the REF). This decrease in adsorption capacity was already expected due to the characteristics of these products (the high viscosity) and the formation of a film on the plaster surface (in the case of the BWr) and the previous results (surface cohesion, hardness and dry abrasion resistance presented in Sections 3.2 and 3.3, respectively) that demonstrated the higher consolidation of the surface with these surface treatments. The possible higher impregnation of surface treatments, applied by roller, can probably contribute to blocking the passage of water vapor.

The limewash (Lr) and the graphene oxide (applied by roller and spray, GOr and GOs) and water from paper immersion (WPr) and water from gypsum plasterboard paper immersion (WPGr) applied by roller slightly decreased the water vapor adsorption capacity: after 24 h, the Lr, GOr, GOs, WPr and WPGr adsorbed 76 g/m<sup>2</sup>, 80 g/m<sup>2</sup>, 80 g/m<sup>2</sup>, 78 g/m<sup>2</sup> and 76 g/m<sup>2</sup>, respectively (less 7%–13% compared to the REF). The application by roller or by spray of GO did not influence the water vapor adsorption capacity of the earth plaster after 24 h. Finally, the application of water from paper immersion (WPs) and from gypsum plasterboard paper immersion (WPGs) applied by spray maintained or slightly increased the water vapor adsorption capacity: after 24 h, the WPs and WPGs adsorbed 91 g/m<sup>2</sup> and 86 g/m<sup>2</sup>, respectively (more 0%–6% compared to the REF).

Comparing the application of the surface treatments by roller or by spray, it appears that, despite a higher mass of product being applied by spraying (compared to by roller), there is a greater reduction of water vapor adsorption capacity by the plaster when the surface treatment is applied by roller. This may be related to a higher amount of product impregnated in the plaster surface when applied by roller, compared to by spray, due to the possibility of having exerted some force during the application of the surface treatments and the gravity action (as these specimens were treated in the horizontal position) which is not observed in application by spray.

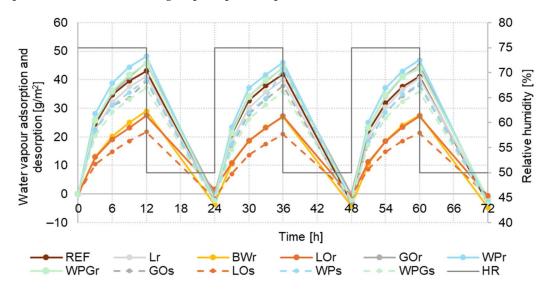
The DIN 18947 [48] defines three different classes for earth plasters according to its adsorption. After 12 h, the classes are: WS I  $\geq$  35.0 g/m<sup>2</sup>; WS II  $\geq$  47.5 g/m<sup>2</sup>; WS III  $\geq$  60.0 g/m<sup>2</sup>. All untreated and treated plasters analyzed in the present study can be classified as WS III, as

they present water vapor adsorption higher than 60 g/m<sup>2</sup>, except the plasters treated with BW and LO that are classified as WS I, as they present water vapor adsorption between  $35 \text{ g/m}^2$  and  $47.5 \text{ g/m}^2$ .

Faria and Lima [3] showed that a limewash did not decrease the water vapor adsorption and desorption capacity of an earth plaster in which it is applied. In the present study, the limewash slightly decreased the adsorption capacity. This can be justified by the different viscosities of the limewashes used and the different amount of surface treatment applied on the plaster surface.

#### 3.5. Moisture Buffering Value

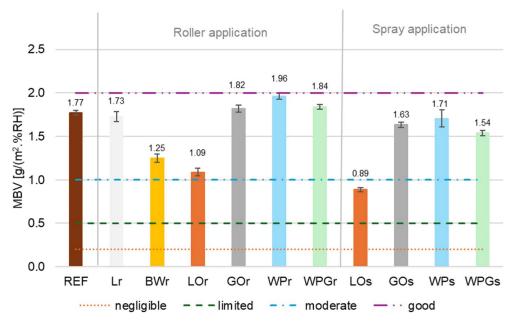
The behavior of the earth plaster with and without surface treatments when exposed to adsorption and desorption consecutive cycles can be analyzed through their adsorption-desorption curves which are presented in Figure 8. All plasters (with and without surface treatments) maintained an approximately constant behavior. This is a good indicator, demonstrating that throughout the adsorption-desorption cycles (which occur daily inside buildings), there is no loss of hygroscopic capacity of the plasters. However, similarly to what was already observed in the water vapor adsorption and desorption capacity of the plasters (Section 3.4), two groups of plasters performance are shown.



**Figure 8.** Water vapor adsorption and desorption of plaster with and without different surface treatments of the last three cycles and programmed RH variations of the climatic chamber. Notation: reference (REF, in brown), limewash (L, in light grey), beeswax (BW, in yellow), linseed oil (LO, in orange), graphene oxide (GO, in grey), water from paper (WP, in blue) and from gypsum plasterboard paper immersion (WPG, in green); applied by roller (r, full line) and spraying (s, dashed line).

The MBV, determined based on these water vapor adsorption–desorption cycles, are presented in Figure 9, as well as the limits defined by NORDTEST [52] for building materials and products. The performance of the treated plasters of the group achieving a higher MBV confirms they can act as passive moisture buffers when applied on indoor walls and ceilings. On the contrary, treatments with linseed oil and beeswax will decrease that capacity. Therefore, these two treatment materials should only be used when other requirements are requested from the plasters.

Compared to the reference plaster (without surface treatment, REF), it appears that all surface treatments promote a decrease in MBV of the plasters, being more significant for BWr, LOr and LOs (less 29%, 39% and 50%, respectively). The exception are plasters with applications of GOs, WPs and WPGs, which have a higher MBV than the reference plaster (by 3%, 11% and 4%, respectively).



**Figure 9.** MBV of the reference plaster and plasters with different surface treatments (average and standard deviation) and limits of classes defined by the NORDTEST [52]. Notation: reference (REF, in brown), limewash (L, in light grey), beeswax (BW, in yellow), linseed oil (LO, in orange), graphene oxide (GO, in grey), water from paper (WP, in blue) and from gypsum plasterboard paper immersion (WPG, in green); applied by roller (r) and spraying (s).

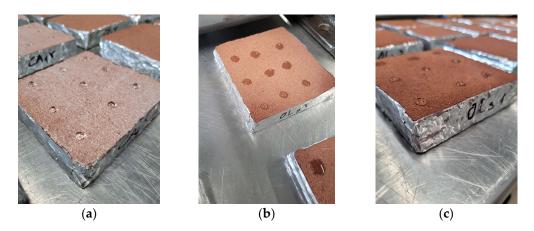
All plasters can be classified by the NORDTEST [52] as having a "moderate" MBV, except the plaster treated with LOs, which is classified as having a "limited" MBV. Although plasters treated with BWr and LOr are classified as having "moderate" MBV, they present MBV close to the limit between "moderate" and "limited" classes. This demonstrates the significant impact of applying beeswax and linseed oil to earth plasters, leading to a loss of their hygroscopic capacity.

## 3.6. Water Drop Absorption and Contact Angle

After applying water droplets to the earth plaster surfaces—both treated and untreated—most droplets were quickly absorbed. The exceptions were the plasters treated with linseed oil (LOr and LOs) and beeswax (BWr).

Beeswax (BWr) significantly enhances water absorption, with water droplets remaining on the surface of the plaster specimens for over 40 min before absorption (see Figure 10a). For linseed oil, the method of application influences the absorption rates. When applied with a roller (LOr), a water droplet is formed but is absorbed quickly within less than a minute (Figure 10b). In contrast, when applied by spray (LOs), the absorption takes longer, exceeding 3 min. Notably, areas with a higher concentration of linseed oil exhibit slower absorption rates (Figure 10c).

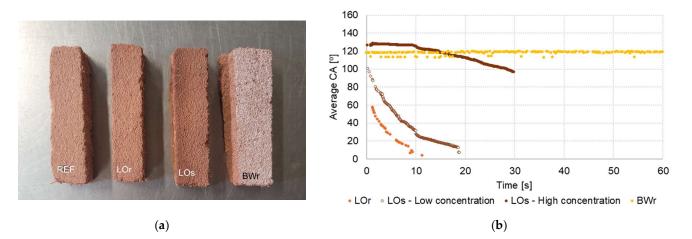
The water droplet formation on the surface of plaster treated with BW and LO, and the time required for its absorption, demonstrates that these treatments effectively create water repellent surfaces, with beeswax showing the most pronounced effect. This hydrophobic behavior of the plaster surface with these surface treatments justifies the reduced hygroscopic capacity, observed in the adsorption and desorption and moisture buffering value tests (Sections 3.4 and 3.5). The high viscosity of beeswax contributes to its effectiveness by forming a film on the plaster surface. Linseed oil, while also viscous, is more fluid than beeswax, which accounts for the longer water droplet absorption times compared to other treatments, though still shorter than with beeswax.



**Figure 10.** Water droplets on the surface of earth plaster with application of: (**a**) beeswax (BWr); (**b**) linseed oil by roller (LOr); (**c**) linseed oil by spraying (LOs).

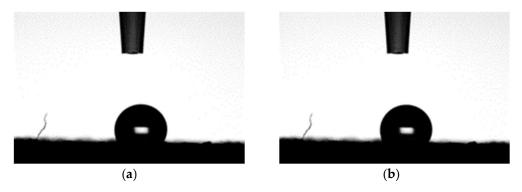
As can be observed in Table 2 and Figure 4, a higher mass of treatment products was applied by spray compared to roller application. Notably, this excess accumulated at the base of the plaster surface, leading to increased absorption times in that area (Figure 10c), which can also justify the slight increase in the time required for water droplet absorption obtained by spray application compared to roller application. The higher amount of linseed oil on the plaster surface may inhibit water droplet penetration in this area, which was not observed in areas with a lower amount of LO.

The presence of water droplets on the plaster surfaces treated with beeswax (BWr) and linseed oil (LOr and LOs) promoted an investigation into the contact angles formed between the water droplet and the plaster's surface. Specimens measuring approximately 20 mm  $\times$  110 mm with these surface treatments (BWr, LOr and LOs) applied by roller or spray were tested (Figure 11a). Depending on the treatment and its properties, the duration for measuring the contact angle varied: 15 s at 17 frames per second (FPS) for LOr, 30 s at 17 FPS for LOs (at both higher and lower amount of treatment area) and 60 s at 3.3 FPS for BWr. Attempts to measure the contact angle of the reference plaster (REF) against BWr, LOr, and LOs were unsuccessful due to the high water absorption of the plaster, which precluded accurate measurements.



**Figure 11.** Specimens and contact angle: (**a**) untreated and treated specimens with linseed oil (LOr and LOs) and beeswax (BWr); (**b**) average contact angle progress over time of selected treated plaster.

Figure 11b illustrates the progression of the average contact angle (average CA) over time for each treated plaster analyzed (LOr, LOs and BWr), and Figure 12 shows the water droplet formed on the surface of the plaster treated with BWr at 0 s and after 60 s.



**Figure 12.** Contact angle between the water droplet and the plaster surface treated with BWr: (**a**) at 0 s; (**b**) at 60 s.

The average contact angle for BWr remained approximately  $120^{\circ}$  at both 0 and 60 s, indicating a stable hydrophobic effect from the beeswax treatment (Figures 11b and 12). In contrast, the contact angle for linseed oil-treated plasters decreased over time, being more evident for the LOr plaster (less than 90°). The higher concentration of linseed oil applied by spray (LOs) initially resulted in a contact angle greater than 90° and it decreased more slowly compared to the lower concentration, which started above 90° but rapidly declined to near 0°, similar to the behavior observed with LOr. These findings align with the results from the preliminary water drop absorption test.

The plaster treated with aqueous beeswax emulsion, as analyzed by Stazi et al. [22], exhibited an initial contact angle of approximately  $90^{\circ}$ , which gradually decreased over a few seconds; after 15 s and 2 min, the angles were  $80^{\circ}$  and  $60^{\circ}$ , respectively. In contrast, the earth plaster treated with beeswax in the present study showed a higher initial contact angle of about  $120^{\circ}$ , remaining stable over 60 s. This difference may be attributed to a higher concentration of beeswax in the treatment used in this study, which was not an emulsion.

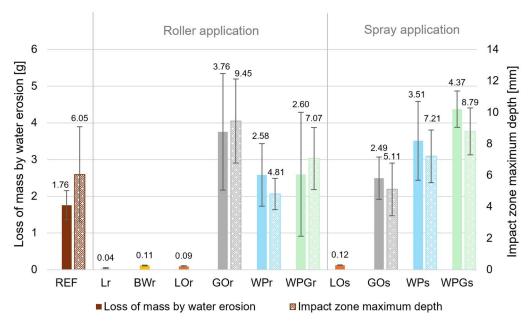
The applications of linseed oil, and especially beeswax, appear to be effective treatments for earth plasters in areas likely to encounter liquid water, and namely requiring frequent cleaning with water, such as close to windows, in bathrooms, toilets and kitchens.

#### 3.7. Water Erosion by Dripping Action

The loss of mass and the impact zone maximum depth by water erosion by dripping action on the earth plaster with and without surface treatment is presented in Figure 13. Two groups of treated plasters are placed in evidence: treatments that significantly increase the resistance of the plaster to dripping and other treatments that do not improve its resistance or even make it worse. The high standard deviation obtained in this test for plasters of the second group should be highlighted, both for the impact depth and the loss of mass.

The application of Lr, BWr, LOr and LOs significantly improved the resistance to water erosion by dripping, since they presented a very low loss of mass (less 93%–98% compared to REF). These specimens did not even show degradation on the surface (impact zone maximum depth of 0 mm). However, the other applications (GO, WP and WPG) promoted an increase in the mass loss by water erosion (by 42%–149%), compared to REF, indicating a loss of resistance by water erosion by dripping. The same applies to the impact zone maximum depth with the specimens with these surface treatments showing an increase of 17%–56%. The exception is the treatments with WPr and GOs, showing a decrease in depth of 16%–20%, compared to REF.

The low water erosion by dripping observed by the plaster treated with beeswax and linseed oil was already expected due to the water repellency conferred by the application of these treatments, as verified in the water drop and contact angle tests (Section 3.6).



**Figure 13.** Loss of mass (full) and impact zone maximum depth by water erosion by dripping action (with dots) of plasters with and without surface treatments: average and standard deviation. Notation: reference (REF, in brown), limewash (L, in light grey), beeswax (BW, in yellow), linseed oil (LO, in orange), graphene oxide (GO, in grey), water from paper (WP, in blue) and from gypsum plasterboard paper immersion (WPG, in green); applied by roller (r) and spraying (s).

Complementing the results obtained for water drop absorption and contact angle, for applications requiring durability to dripping water, surface treating the earth plaster with linseed oil and beeswax seem viable possibilities. Applying a limewash is also viable but when a drastic change in the color surface is not acceptable, clayish pigments used in the limewash can probably be considered.

The plaster treated with aqueous beeswax emulsion evaluated by Stazi et al. [22] showed an erosion depth by dripping action of 3 mm. In the present study, the earth plaster treated with beeswax did not present erosion by dripping action, demonstrating a better behavior of this treatment. Again, this can be due to a higher concentration of the surface treatment (not an emulsion), promoting better protection of the plaster. The water resistance improvement of earth plasters with limewash referred to by Faria and Lima [3] was verified in the present study, validating the results obtained.

#### 3.8. Synthesis of Results

Plasters, particularly ones applied on the walls, are subjected to impacts and abrasion of people and even furniture and equipment. Some increase in hardness and resistance to dry abrasion may therefore be required. The vulnerability of clayish earth in contact with liquid water does not allow their application as earth renders (on the outside of buildings) and sometimes limits their application as plasters in areas exposed to liquid water without protection (namely near a sink or under a window). However, in the case of earth plasters, greater durability should not compromise their hygroscopic capacity, which, along with low environmental impact, is one of the main advantages of this type of plaster, particularly in terms of the comfort and health of building occupants and energy efficiency.

In the present study, the influence of different surface treatments (limewash, beeswax, linseed oil, graphene oxide dispersion and water from paper immersion and from gypsum plasterboard paper immersion) applied by roller or by spraying on the characteristics of a pre-mixed earth plaster was evaluated.

Table 3 presents a summary of the impact that each surface treatment had on the different characteristics of the earth plaster analyzed.

Surface Treatment	Color	Surface Cohesion	Surface Hardness	Dry Abrasion Resistance	Adsorption and Desorption (24 h)	MBV	Water Drop Absorp- tion Time	Resistance to Water Erosion by Dripping
Lr	Change	$\uparrow\uparrow\uparrow$	1	$\uparrow \uparrow \uparrow$	$\downarrow$	=	=	$\uparrow \uparrow \uparrow$
BWr	Change	$\uparrow\uparrow\uparrow$	$\uparrow$	$\uparrow\uparrow\uparrow$	$\downarrow\downarrow\downarrow\downarrow$	$\downarrow\downarrow$	$\uparrow\uparrow\uparrow$	$\uparrow\uparrow\uparrow$
LOr	Change	$\uparrow\uparrow\uparrow$	$\uparrow$	$\uparrow\uparrow\uparrow$	$\downarrow\downarrow\downarrow\downarrow$	$\downarrow\downarrow$	$\uparrow$	$\uparrow\uparrow\uparrow$
GOr	=	$\downarrow$	=	$\uparrow\uparrow$	$\downarrow$	$\uparrow$	=	$\downarrow\downarrow\downarrow\downarrow$
WPr	=	=	$\uparrow$	$\uparrow\uparrow$	$\downarrow$	$\uparrow\uparrow$	=	$\downarrow\downarrow$
WPGr	=	=	$\uparrow$	$\uparrow\uparrow$	$\downarrow$	$\uparrow$	=	$\downarrow\downarrow$
LOs	Change	<b>↑</b> ↑↑	↑	$\uparrow \uparrow \uparrow$	$\downarrow\downarrow\downarrow\downarrow$	$\downarrow\downarrow\downarrow\downarrow$	$\uparrow \uparrow$	$\uparrow\uparrow\uparrow$
GOs	=	$\uparrow$	$\downarrow$	$\uparrow\uparrow$	$\downarrow$	$\downarrow\downarrow$	=	$\downarrow\downarrow$
WPs	=	$\uparrow$	=	$\uparrow\uparrow$	$\uparrow$	=	=	$\downarrow\downarrow\downarrow\downarrow$
WPGs	=	=	$\downarrow$	$\uparrow \uparrow$	$\downarrow$	$\downarrow\downarrow$	=	$\downarrow\downarrow\downarrow\downarrow$

<b>Table 5.</b> Influence of surface frequencing on the characteristics of plaster.	Table 3	. Influence	of surface	treatments	on the	characteristics of 1	olaster.
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Notation: =—maintains the characteristic or is not significantly influenced;  $\uparrow$ —increases the property;  $\downarrow$ —decreases the property;  $\uparrow\uparrow\uparrow$  or  $\downarrow\downarrow\downarrow$ —significant impact on the property;  $\uparrow\uparrow$  or  $\downarrow\downarrow\downarrow$ —impact on the property;  $\uparrow$  or  $\downarrow\downarrow$ —impact on the property;  $\uparrow$  or  $\downarrow\downarrow$ —lower impact on the property; red—negative effect; green—positive effect.

It is possible to conclude that:

- Limewash (Lr) and the application of beeswax (BWr) and linseed oil (LOr and LOs) caused a change in the color of the plaster surface: the Lr formed a white layer; the BWr whitened the surface; LOr and LOs darkened the surface of the plaster. The aesthetic requirements of the plaster may condition the use of some of those treatments. Some, as the limewash, although it can be pigmented, formed a layer, changing the plaster's texture.
- The application of beeswax (BW) and linseed oil (LO), as well as the limewash (L), improved the surface cohesion, dry abrasion resistance and water erosion by dripping resistance. BW made the plaster surface water repellent. The higher amount of LOs applied promoted a higher difficulty in penetration by a water droplet, conferring water repellent characteristics that were lost over time. However, particularly the treatments with BW and LO significantly worsened the hygroscopic capacity of the plaster. Therefore, these treatments should not be applied on common indoor walls and ceilings, so that the moisture buffer effect of the plaster can be optimized, and only in specific walls where resistance to water is required.
- In general, the other surface treatments maintained the characteristics of the plaster without surface treatment, even improving its dry abrasion resistance. The exception was the application of graphene oxide dispersion (GOr) and water from paper immersion and from gypsum plasterboard paper immersion (WPs, WPGr and WPGs), which significantly reduced the water erosion by dripping resistance of the plaster (higher loss of mass by water erosion and impact zone maximum depth, compared to REF). Being more expensive and with higher environmental impact, the GO treatment did not prove necessary.
- The application of the treatments using a roller, especially with the more fluid materials (GO, WP and WPG) promoted detachment and displacement of plaster surface particles, which was not so evident with spraying. On the other hand, spraying promoted an accumulation of surface treatment at the base of the specimens. Considering the bottom of the walls are more prone to dripping, this type of application may even have a positive effect.

#### 4. Conclusions

In the present study, the influence of different surface treatments applied by a roller or as a spray on the performance of an earth plaster were analyzed. The results demonstrate that it is possible to improve water resistance and surface performance, especially surface cohesion and dry abrasion resistance, by treating the earth plaster with a limewash, beeswax or linseed oil (Lr, BWr, or LOr/LOs). Nonetheless, there is a negative impact on the ability to adsorb and desorb water vapor, especially with the application of beeswax and linseed oil. The application of graphene oxide did not demonstrate significant improvements in the characteristics of the plaster, sometimes worsening its performance. However, its dry abrasion resistance was improved with the application of all surface treatments analyzed.

The decision on the need to apply a surface treatment and, if so, the choice of the treatment to apply, needs a definition of the requirements, depending on which type of areas the plaster will be on a building. Thus, the application of BW, LO and even L seems a good option when an earth mortar is intended be applied in an indoor area with some contact with liquid water. However, these treatments should be limited to surfaces that require water resistance, because there is a decrease in the contribution of the plaster to act as a passive moisture buffer. In fact, when considering the application of an earth plaster, for example in a living room or bedroom, these treatments should no longer be recommended, except eventually in areas near windows, due to its negative impact on the plasters' hygroscopic ability. In those dry rooms, a surface treatment with water from waste paper applied by a roller contributed to increased hardness, resistance to dry abrasion and MBV, which seems to be a good choice.

In future works, it is recommended to evaluate the influence of RH on the surface cohesion and hardness, dry abrasion resistance and water erosion dripping action of earth plasters with the different surface treatments analyzed. The influence of these surface treatments on the characteristics of other earth plasters, with other types of clays and mortars formulation, should also be analyzed.

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