TRADITIONAL GYPSUM PAVEMENTS WITH NATURAL ADITIVES

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Abstract

In traditional architecture, the construction systems used have always been linked to the material resources of the environment. In the areas of extraction of gypsum in the Iberian Peninsula, the use of this material as a conglomerant was not limited to the interior lining of vertical and/or horizontal walls or to interior decorations, as is the case today, but proved to be a very versatile material, which could be used both in structural elements and in finishing elements and decorations. This study shows the preliminary results of an ongoing research on the recovery of traditional gypsum pavements, where three types of traditional gypsum plasters with natural products such as collagen, coconut soap, potassium soap, vegetable oil and marshmallow root were tested. The hygroscopic behaviour of the treated samples has been evaluated from the capillarity water absorption and the contact angle, due to their high hygroscopicity. On the other hand, the density and porosity, surface hardness and thermal conductivity of the samples have also been determined.

Keywords:

Traditional gypsum, natural additives, soaps, capillary water absorption, thermal conductivity.

1 INTRODUCTION

In traditional architecture, the construction systems used have always been associated with the material resources of the environment. The basic materials that characterise this architecture are stone, earth, ceramics, wood, lime and plaster (Garate Rojas, 1999). Today, many of these materials continue to form part of contemporary construction systems, although the use we make of them is different to that which they originally had in traditional architecture.

Within this group of materials, stone, wood and ceramics have always been the most appreciated and valued ones, in all periods and architectural trends; although in recent years, and thanks to the search for more sustainable materials with low environmental impact, much research has been carried out on the recovery and use of lime; as well as the study and enhancement of earth as a building material (Veiga, R., 2017) (Foster et al., 2020) (Pacheco-Torgal and Jalali, 2012). However, of all the traditional materials mentioned above, gypsum has always been one of the most modest and little known materials (Garate Rojas, 1999) (González-Sánchez et al., 2020).

In the gypsiferous areas of the Iberian Peninsula, the use of gypsum as a binder was not only limited to the interior coating of walls or interior decorations, as is the case today, but was a very versatile material that could be used in both structural and finishing elements (La Spina and Grau, 2020). Some of the most common examples of the use of this material in gypsiferous areas are earth walls with punctual reinforcements of gypsum mortar, gypsum stone walls covered with gypsum mortar, vaulted ceilings of poured plaster, interior staircases, pavements or exterior cladding (La Spina, 2016) (La Spina, 2018) (Navarro and Julià, 2019).

Traditionally, the improvement of the properties of mortars has always been sought through the incorporation of additives, both in the plaster mass and on the surface of the installed product. In the case of traditional plasters, these recipes have usually been transmitted orally by craftsmen, applicators and/or self-builders over time, although some authors have been able to collect these teachings in treatises, informative texts, user manuals and compilation texts (Espinosa, 1859) (Castinyeria, 1898) (Hero, 1954) (Garate Rojas, 1999). Some of the natural biopolymers, applied in plaster mass, most commonly used in this sense are soaps, oils, waxes and fats (lipids); solutions with a high percentage of collagen (proteins), derived from the use of water obtained by boiling animal bones; the use of starch, alginate, natural glues or marshmallow root (polysaccharide), resins and varnishes, etc. (Vissac et al. 2017).

This paper shows the preliminary results of a larger research project, which aims to recover the traditional continuous gypsum pavements and to provide constructive solutions adapted to the needs of the 21st century. The aim of this study is to analyse the hygroscopic behaviour of three different types of traditional plaster, suitable or

specifically developed for use as pavements, with additives, in plaster mass, with natural products traditionally used in construction, such as collagen, coconut soap, potassium soap, olive oil and marshmallow root.

2 MATERIALS AND METHODOLOGY

2.1 Materials

Three types of commercial gypsum were used: *Yeso Artesano de Albarracín*, manufactured and distributed by the craftsman Antonio Meda (Albarracín, Teruel, Spain); *Argiofloor*, distributed by the company Argio/DécoSystem (Soisy sous Montmorency, Val-d'Oise, France); and *Plâtre à Plancher Finition*, distributed by the company Plâtres Vieujot (Soisy sous Montmorency, Val-d'Oise, France).

According to the commercial information available: *Yeso Artesano de Albarracín* (ALB) is a product that continues to be produced in a traditional way, maintaining the tradition and technique that has been used in the area since the 10th century, achieving a material similar to that produced in the past. The use of this product is mainly intended for the rehabilitation of traditional plaster mortars, although its use has also been extended to the reproduction of traditional construction techniques in new buildings. *Argiofloor* (ARG) is a 100% natural and breathable product made of gypsum, clays, lime and marble chips. It is produced in industrial ovens. It is intended for interior use only, and has been designed for decorative coatings on horizontal and vertical surfaces, and can be given a smooth, sanded and/or oiled finish. *Plâtre à Plancher Finition* (PàP) is a product made from traditional highly calcined gypsum, pigments and possibly coloured marble aggregates. It is produced in industrial kilns. Its use is intended for the plastering of decorative interior pavements, with a minimum thickness of 15 mm, with the possibility of giving a smooth finish, with or without patina, or polished.

As natural additives, collagen powder from the WoldoHearth brand was used; ecological and easily biodegradable natural coconut soap flakes, made from coconut oil (80%) and pomace oil (20%) and sodium hydroxide, from the commercial company LA INDUSTRIA JABONERA; ecological and easily biodegradable natural soft potassium soap, made from combinations of different oils and potassium hydroxide, from the commercial company LA INDUSTRIA JABONERA; refined olive oil from seeds, with 0.2° of maximum acidity, from the commercial company SOVENA ESPAÑA S.A.U.; and dehydrated marshmallow root, crushed and powdered.

2.2 Gypsum chemical characterization

The first step was to carry out the mineralogical characterization, by means of thermogravimetric analysis (TGA), of the three types of gypsum used in the study. This analysis was carried out using NETZSCH thermogravimetric equipment STA449 F5 JUPITER. The NETZSCH-PROTEUS-80 software was used to interpret the data obtained.

2.3 Manufacture of Samples

Previously to the preparation of the samples, the dosage was defined for each of the plasters studied, by means of the test to determine the water/gypsum ratio, by weight, using the shaking table method, in accordance with the UNE-EN 13279-2:2014 standard. The mixing time was also defined, 1 minute for all the samples, and the dosage of each of the additives, as a percentage in relation to the weight of the gypsum. A total of 45 samples of dimensions 50 x 50 x 25 mm were made, grouped in 3 groups of 15 samples each by the manufacturer, classified according to the additive used in each case, in different proportions, for subsequent analysis (Tab.1.).

Tab. 1: Classification samples according to their dosage

ALB W/G	ARG W/G	PàP W/G	NATURAL ADDITIVES					
			C	C _s	K _s	O	M _F	M _G
0.35	0.25	0.35	-	-	-	-	-	-
0.35	0.25	0.35	0.1%	-	-	-	-	-
0.35	0.25	0.35	0.5%	-	-	-	-	-
0.35	0.25	0.35	1%	-	-	-	-	-
0.35	0.25	0.35	-	0.5%	-	-	-	-
0.35	0.25	0.35	-	1%	-	-	-	-
0.35	0.25	0.35	-	5%	-	-	-	-
0.35	0.25	0.35	-	-	0.5%	-	-	-
0.35	0.25	0.35	-	-	1%	-	-	-
0.35	0.25	0.35	-	-	5%	-	-	-
0.35	0.25	0.35	-	-	-	0.5%	-	-
0.35	0.25	0.35	-	-	-	1%	-	-
0.35	0.25	0.35	-	-	-	5%	-	-
0.35	0.25	0.35	-	-	-	-	2.5%	-
0.35	0.25	0.35	-	-	-	-	-	2.5%

ALB = Albarracín gypsum; ARG = Argiofloor gypsum; PàP = Plâtre à Plancher finition gypsum; W = water; G = gypsum; C = collagen; C_s = coconut soap; K_s = potassium soap; O = vegetable oil; M_F = fibre marshmallow root; M_G = ground marshmallow root.

The manufacture of the samples is carried out based on the methodology explained in the UNE-EN 13279-2:2014 standard, although with some nuances. In the case of the incorporation, in plaster mass, of the different natural additives, the additive was first dissolved in the mixing water and then the estimated amount of gypsum was added. The samples were kept in the mould for a minimum of 24 hours, until they were correctly demoulded, and were kept at laboratory temperature and humidity conditions (23°C and 50% Hr.) until they were stable in weight.

2.4 Capillary water absorption

The durability, dimensional stability and potential comfort of gypsum pavements is directly related to their behaviour in relation to water. The capillary water absorption test provides information on the estimated effect that the material may suffer when exposed to liquid water, especially in the non-visible area where it meets the slab. The test is carried out in accordance with the UNE-EN 15801:2009 standard.

2.5 Contact angle

The determination of the surface behaviour of the hardened gypsum samples against the action of water was carried out by means of the static contact angle determination method according to the UNE-EN 15802:2010 standard. For the test, readings were taken in 4 different areas of each hardened gypsum sample.

2.6 Thermal conductivity

The comfort of future users of gypsum screeds is directly related to their thermal transmittance capacity. The thermal conductivity is determined by the QuicklineTM -30 thermal properties analyser, using a surface probe on the hardened gypsum samples. The method is based on ASTM D5930.

2.7 Surface hardness

To analyse the surface static penetration hardness of the hardened gypsum samples, readings were taken at 9 different points, distributed homogeneously over the surface by drawing an imaginary grid over the tested area, using a portable digital hardness tester TYP-100C, for shore hardness C, with a range of up to 100HC.

2.8 Bulk density and porosity

The method for the determination of the bulk density and the percentage of porosity of hardened gypsum samples is carried out according to UNE-EN 1015-10:1999.

3 RESULTS

3.1 Gypsum characterization

The results are shown in Fig. 1, where we can see 4 significant weight loss zones can be distinguished. The first interval is located between 100°C and 200°C, and corresponds to the loss of the water of dehydration of the gypsum. The second interval is located between 200°C and 600°C, and corresponds, on the one hand, to the loss of the remaining water from the dehydration of the gypsum; and on the other hand, to the loss of water due to the decomposition of the clays. It should be noted that the most important mass loss occurs between 350°C and 600°C, which we identify as the decomposition of the clays. The third interval is located between 600°C and 800°C, and corresponds to the loss of CO₂ due to the decomposition of carbonates. The last interval is located between 800°C and 1000°C, and corresponds to the loss of SO₃ by the decomposition of the anhydrite molecule, obtaining calcium oxide.

Fig. 1: Thermogravimetric analysis comparative for different types of gypsum

Normally, traditional gypsum is not characterised by its high percentage of purity, so it is not surprising to find other elements such as clays, limestone, quartz, etc. The percentage of weight loss, for each interval, and the residual mass of the 3 types of gypsum studied is shown in Tab. 2; where we can see that *Argiofloor* is the gypsum that experiences the greatest weight loss, mainly linked to the decomposition of the carbonates it contains. *Albarracín* gypsum is the one with the highest amount of clays, and *Plâtre à Plancher* is the purest gypsum.

Tab. 2: Percentage of weight loss and residual mass for different types of gypsum

Type gypsum	% Weight loss				Residual mass
	100-200°C	200-600°C	600-800°C	800-1000°C	
Albarracín	-1,20%	-1,08%	-0,79%	-1,00%	95,60%
Argiofloor	-1,58%	-0,40%		-10,86%	86,91%
Plâtre à Plancher	-3,08%	-0,75%	-2,04%	-0,13%	93,52%

3.2 Capillary water absorption

The results are shown in Fig. 2, where we can see the gypsum samples with collagen additive, for the three types of gypsum in the dosages 0.5% and 1%, and with marshmallow root, for the *Plâtre à Plancher* gypsum, are the only ones that have a higher water absorption compared to the reference sample without additive.

Fig. 2: Capillary water absorption results for Albarracín samples - ALB (a), Argiofloor samples - ARG (b) and Plâtre à Plancher samples - PàP (c).

The additive with the lowest capillary water absorption is coconut soap, in all its dosages, followed by potassium soap and vegetable oil treatments. Finally, the behaviour of the samples treated with marshmallow root was uneven. In fibre format, the samples end up crumbling, and in crushed format, they behave very similarly to gypsum treated with vegetable oil. The capillary suction coefficients are given in Tab. 3.

Tab. 3: Capillary water absorption coefficient result ($\text{Kg/m}^2 \cdot \text{s}^{1/2}$)

Type	N		C			Cs			Ks			O			M _F	M _G
	-	0.1%	0.5%	1%	0.5%	1%	5%	0.5%	1%	5%	0.5%	1%	5%	2.5%	2.5%	
ALB	26.027	22.963	31.490	27.596	4.468	2.800	1.300	20.867	18.369	12.831	19.044	16.685	21.757	20.764	21.196	
ARG	11.155	6.700	11.132	14.332	1.478	0.981	0.485	5.857	4.088	5.440	6.047	4.500	7.934	11.132	7.887	
PàP	11.839	9.305	20.626	33.681	1.680	1.255	0.494	5.043	5.051	1.804	4.060	5.839	8.356	21.953	18.713	

ALB = Albarracín gypsum; ARG = Argiofloor gypsum; PàP = Plâtre à Plancher finition gypsum; N = Non additive; C = collagen; Cs = coconut soap; Ks = potassium soup; O = vegetable oil; M_F = fibre marshmallow root; M_G = ground marshmallow root.

3.3 Contact angle

The determination of the contact angle between a drop of distilled water and the surface of a material allows the degree of water repellency of the material to be determined (Tab. 4). If the contact angle is less than 90°, the surface absorbs water easily; if the contact angle is greater than 90°, the surface is considered water repellent. Untreated gypsum, in all cases, has a contact angle value of 0° and is therefore permeable to water. The coconut soap treatment shows a slight water repellency. On the other hand, the treatment that has not modified the water-repellent properties of the samples is vegetable oil. *Plâtre à Plancher* is the gypsum that generated the most water repellency, with any additive. It should be noted that, although almost all the additives have developed some kind of water repellency, after a period of time not exceeding 1 minute, all the samples have absorbed the test water.

Tab. 4: Contact angle results

Type gypsum	% dosage	Average static contact angle values per additive						
		N	C	C _s	K _s	O	M _F	M _G
ALB	0.0%	0°						
	0.1%		0°					
	0.5%		0°	68°	0°	0°		
	1.0%		0°	83°	0°	0°		
	2.5%						0°	0°
	5.0%			79°	77°	0°		
ARG	0.0%	0°						
	0.1%		0°					
	0.5%		0°	80°	0°	0°		
	1.0%		29°	69°	0°	0°		
	2.5%						0°	46°
	5.0%			0°	0°	40°		
PàP	0.0%	0°						
	0.1%		0°					
	0.5%		49°	68°	45°	0°		
	1.0%		57°	85°	72°	0°		
	2.5%						40°	55°
	5.0%			64°	72°	76°		

ALB = Albarracín gypsum; ARG = Argiofloor gypsum; PàP = Plâtre à Plancher finition gypsum; N = Non additive; C = collagen; C_s = coconut soap; K_s = potassium soap; O = vegetable oil; M_F = fibre marshmallow root; M_G = ground marshmallow root.

3.4 Thermal conductivity and bulk density

The results are shown in Fig. 3, where we can see only the additive samples with collagen increase their bulk density compared to the reference sample, and they are also the samples with the highest thermal conductivity, compared to the rest of the samples of the group of gypsum to which they belong. Coconut and potassium soap are the additives that modify the bulk density of the samples the most, reducing it by 7 - 11 %, making them lighter; however, their thermal conductivity does not decrease compared to the reference sample, with values ranging between 0.09 and 0.28 W/mK, with a concentration of values around 0.20 W/mK. Samples additised with marshmallow root fibre provide scattered data, but in crushed form they are close to 0.25 W/mK, although their densities are quite different. The samples additised with vegetable oil increase their thermal conductivity with respect to the reference sample, with values ranging between 0.21 and 0.33 W/mK.

Fig. 3: Thermal conductivity vs bulk density

3.5 Surface hardness

The results are shown in Fig. 4, where we can see that the treatments tested have modified, in different ways, the surface hardness of the gypsum tested. If we compare the samples without additives with those with additives, we can see that all the samples made with *Albarracín* have a more elastic surface than the reference sample, reducing their stiffness by between 0.29% and 18.12%. On the other hand, both the samples made with *Argiofloor* and *Plâtre à Plancher* increase their stiffness compared to the sample without additive, except for the sample "ARG_Mg.2.5%" which reduces its stiffness by 8.21%. For *Argiofloor*, the increase in surface stiffness increases between 2.53% and 7.23%; and for *Plâtre à Plancher*, the increase is between 4.49% and 10.89%.

Fig. 4: Average values surface hardness Shore C

3.6 Porosity and capillary water absorption coefficient

The average results are shown in Fig. 5. As we can see, only the treatments with the capillary absorption coefficient lower than that of the reference sample have been taken into consideration. It is significant that, when the values of capillary water absorption are lower (samples with soap and oil additives), their porosity percentage increases, compared to non-additive samples. Moreover, this phenomenon is more evident in the soap-based additives (coconut and potassium), followed by the vegetable oil additive.

Fig. 5: Capillary water absorption coefficient vs Porosity

4 CONCLUSIONS

The results obtained lead us to believe that the application of natural additives in plaster mass, such as soaps or oils, in gypsum intended for the rehabilitation or new construction of pavement can be a good complement and/or alternative to the application of traditional surface treatments (resins, waxes, linoleums, oils, varnishes, etc.); in no case have the additives used produced a filmogenic alteration on the surface of the samples, but they have achieved a water-repellent effect, providing a natural water entry barrier, regardless of their porosity. In addition, it has been observed that the samples additivated with soaps, and to a lesser extent with oil, increase their porosity, decreasing their apparent density. This phenomenon is related to the manufacturing process of the samples, since during the dissolution of the soaps and oil in the pasting water, air is incorporated into the mixture. This particularity is important because, although it does not significantly modify the transmittance of the unadditivated samples, it does make it possible to lighten the dead weight of the pavement on the slab.

The gypsum samples with additives have not managed to reduce the transmittance of the samples without additives, so the resulting product is slightly more heat-conductive. Future studies should be carried out to determine the optimum thickness of gypsum screeds and their use in underfloor heating systems.

Of all the additives used, the one that overall provides the most attractive results for future use in continuous gypsum pavement systems is coconut soap, due to its ease of use, low capillary water absorption, density, porosity and surface hardness.

With regard to gypsum plasters with collagen and marshmallow root additives, it is important to point out that it would be necessary to continue research with new dosages (with a higher percentage of additive) to determine the benefits that these two natural biopolymers can provide us with.

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