Physical- Mechanical Comparison of the Traditional Gypsums from Albarracín and Pallars

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Abstract

The traditional gypsum of Albarracín is probably the only one currently marketed in Spain. In the Pyrenees of Lleida, specifically in the regions of Pallars Jussá and Pallars Sobirá, the traditional constructions are carried out, mainly with traditional plaster.
In order to obtain a traditional gypsum that presents properties similar to those of Albarracín, a traditional oven was built in the Pyrenees of Lleida in 2016, in Senterada (Pallars), obtaining a product of 500 Kg of plaster after cooking.
The present work shows a comparative through an experimental study, at a physical-mechanical level, of the two traditional gypsums, with different water/plaster dosages and with different mixing times.
The results show many similarities between both materials and demonstrated, once again, how the manufacturing factors can affect them.

Introduction

In 2016, the CEG Association (Center d'estudis del Guix) visited the region of Pallars with the intention of making a traditional kiln with the autochthonous stone found in the area and then to obtain a traditional plaster product from it. Once the traditional kiln was built and the processes of calcination and crushing of the stone were finished, approximately half a ton of traditional plaster was obtained, which was stored in bags of 25 kilograms each.

In the year 2017, it was decided to carry out the physical-mechanical characterization from the traditional gypsum product obtained in the region of Pallars. Furthermore, looking for a point of comparison, it was decided to carry out also the physical-mechanical characterization of the traditional gypsum of Albarracín. The last one, probably is the only traditional gypsum which is commercialized in Spain.
As a result of this, the question arises as to whether the traditional plaster of Pallars has sufficient physical and mechanical properties to be able to compete with the traditional commercial plaster of Albarracín.

Previous Knowledge

Gypsum is a natural mineral that is chemically composed from calcium sulphate and water ($CaSO_4 \cdot 2H_2O$). This stone is light in weight but tough in hardness, does not make effervescence with acids and generally presents colors such as: white, ashen, yellow, brown, gray and if it contains ferric oxide, it is presented in reddish white. Likewise, it is known by the name of plaster to the product that results from cooking and crushing the mineral gypsum, while the homogeneous mixture of this product with water is called plaster paste. This paste has the property of hardening through the setting process, thus becoming calcium sulphate dihydrate once again [1].

Traditional gypsum is defined as the product of calcination of calcium sulphate dihydrate (CaSO4 · 2H2O) in traditional kilns, a process which has the following characteristics [2][3]:

- During cooking, there is no temperature control inside the oven.
- Cooking is not homogeneous, but multiphase. Depending on the size of the stone and the exposure to heat, all the phases of calcium sulphate can be found in the product.
- There is no selection or purification process in the raw material.

Generally, traditional gypsum kilns have square floor plan and assembled with three walls attached by an embankment. Among them, the gypsum stone is placed in the form of a vault in a descending manner (meaning that the larger stones should be placed at the bottom and the little ones are above), with the objective that the dehydration or firing of the stone is as equitable as possible [4]. Under the vault formed by the gypsum stone itself is located the area where the fuel that feeds the kiln is located. This combustible is constituted by wood, branches or charcoal.

Materials and Test Methods

Materials

In order to evaluate the human and material resources, like time, labor and cost that were necessary for the elaboration of traditional gypsum, the CEG Association carried out the elaboration of a traditional oven in Pallars Sobirá located in Lerida in the year 2016 (Fig. 1)

The raw material was extracted with the help of a backhoe excavator and the brigade of the town hall of Senterada. All the rocks from the extraction were placed in the kiln and formed part of the firing, as all the extracted material should form a homogenous mixture.

The kiln was assembled in the same place where the extraction was made, taking advantage of the hole left by the cut with the backhoe. For the creation of this traditional kiln, two side walls were built and one in the center, above them, the gypsum stone was placed in the form of a vault, leaving two holes in the lower part for the placement of

the fuel. A plant called "boj" was used as fuel, in addition to wood, material, which was extracted from the same mountain where the gypsum stone was.
When the firing of the material was completed, a plastic layer was placed on top of the extinguished furnace and allowed it to cool. Months later, the brigade returned to crush the product obtained from the firing and store it in bags of 25kg, obtaining 20 bags of traditional plaster in total.

Figure 1. Pictures of the elaboration of the traditional oven in Pallars by the CEG brigade

At present, in Albarracín there is the only one kind of kiln able to create a satisfactory traditional plaster. This kiln is created in form of a vault with gypsum stone from two nearby quarries. This gypsum stones contains silica and clay impurities [5].
The plaster made in Albarracín, is a handmade product that has two different presentations depending on its color: a red color or a grayish color. It is a product backed up for years by its precedence in the coatings and waterproofing of the facades in Albarracín. The extraction of the stone in Albarracín or its mountain it is made with machinery from the existing quarries of gypsum and the selection procedure is manual. Once the product is powdered, it is packed in paper bags. The color of the material is indicated on the bag and a stamp is added as guaranty that the product was created by a trusted artisan [6] (Fig. 2).

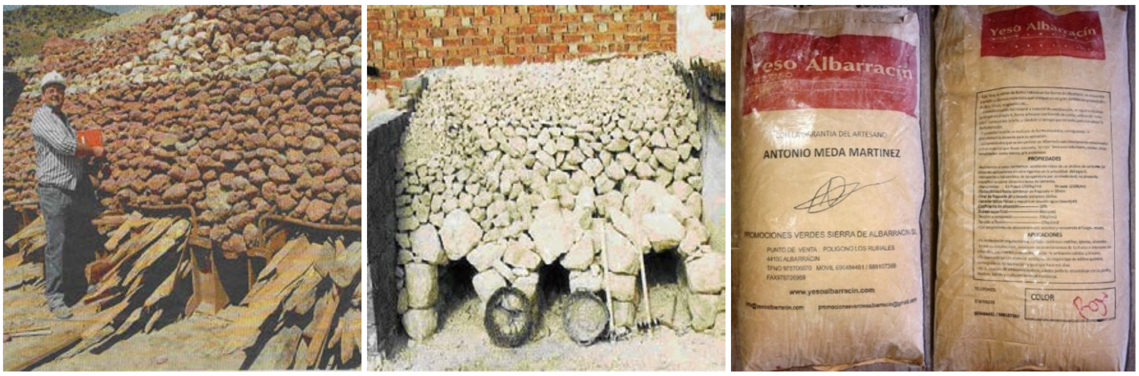


Figure 2. Pictures of ovens and plaster made in Albarracín

Dosages

Considering that the experimental plaster of the Pallars will be compared with the plaster of Albarracín, the technical file of the plaster of Albarracín was taken into account to find the correct dosages of the plaster of the Pallars.

This technical sheet marks the values of water/gypsum ratio (W/G) between 0.34- 0.40. It was also decided to choose relatively close W/G values from the technical sheet, from 0.30 to 0.45, to experiment with the two types of gypsum and to make some mixtures and so to be able to observe the behavior of the material with these amounts of water (Table 1).

Table 1. Water/gypsum test with Pallars's and Albarracín gypsum.

W/G Ratio	Gypsum (gr)	Water (ml)	Pallars Gypsum	Albarracín Gypsum
0,30	50,00	15,00		
0.34	**50,00**	**17,00**		
0.37	**50,00**	**18.50**		
0.40	**50,00**	**20,00**		
0.45	50,00	22.50		

Once the tests had been carried out, it was concluded that the W/G ratios suitable for working with both materials are as follows: W/G = 0.34, W/G= 0.37 and W/G= 0.40.

Sample Preparations

There were made rectangular prism- shaped specimens, both with traditional gypsums (Pallars and Albarracín) for the tests. All of them were design following the Spanish standard UNE-EN 13279-2 [7] using metal molds with measurements of 4x4x16cm and following the dosages of water and plaster previously provided. Additionally, it was taken into account, two different mixing times: one and three minutes.
A total of 18 test pieces were made, three pieces for each W/G ratio, as well as for each mixing time. (Fig. 3).

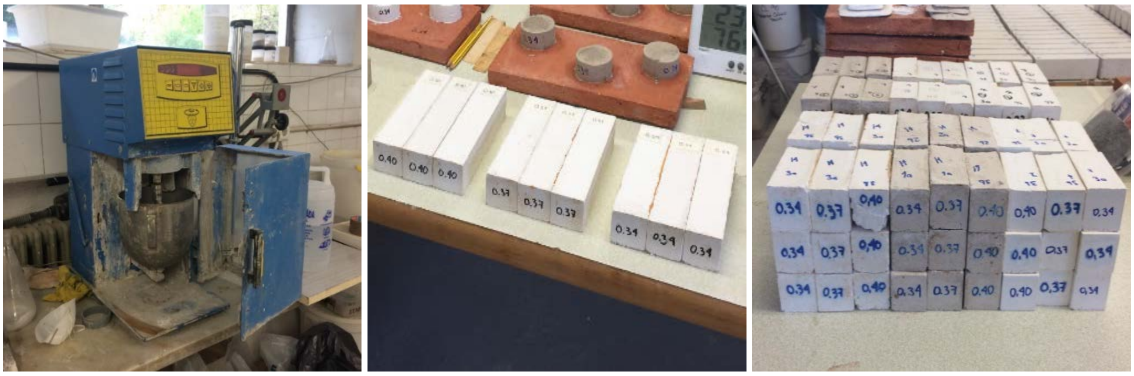


Figure 3. Preparation of the test pieces for the mechanical tests according to the Standard Norm UNE-EN 13279-2.

Hardened Mortar

All the test specimens were allowed to set for seven days, before tensile load and compression load tests were carried out, furthermore the specifications of the UNE-EN 13279-2 standard were followed all the time [7]. The mechanical press used in mechanical tests is Wykeham Farrance brand with a maximum load of 5000 kg. The porosity test procedure followed the Spanish standard norm UNE-EN 1015-10: 2000 [8], while the coefficient of capillary absorption and desorption of the hardened gypsum followed the standard UNE-EN 1925: 1999 [9] which refers to the natural stone.

Table 2. Pallars's gypsum results at a mixing time of one minute.

	W/G	D. Y POROSITIES			MECHANICAL RESISTANCES		WATER MOBILITY		
	Water/ Gypsum Ratio	Apparent Density (g/cm3)	Relative Density (g/cm3)	Porosity (%)	T Flexión (MPa)	T comp (MPa)	c.A.C ($\Delta Kg/m^2/\sqrt{h}$)	Se (%)	Coef. evaporación (%)
PALLARS 1 MIN	0,34	1,54	2,52	39,00	1,39	3,70	20,06	8,79	2,08
PALLARS 1 MIN	0,37	1,48	2,53	41,50	1,08	2,44	27,11	8,23	1,59
PALLARS 1 MIN	0,40	1,42	2,51	43,27	0,81	1,91	25,17	5,66	1,62

Table 3. Albarracín gypsum results at a mixing time of one minute.

Albarracín 1 MIN	W/G	D. Y POROSITIES			MECHANICAL RESISTANCES		WATER MOBILITY		
	Water/ Gypsum Ratio	Apparent Density	Relative Density	Porosity	T Flexión	T comp	c.A.C	Se	Coef. evaporación
		g/cm3	g/cm3	%	MPa	MPa	$\Delta Kg/m^2/\sqrt{h}$	%	%
	0,34	1,46	2,38	38,77	0,91	1,89	19,31	5,68	1,52
	0,37	1,41	2,33	39,37	1,03	1,98	20,58	7,25	1,38
	0,40	1,37	2,33	41,20	1,01	1,95	21,55	12,39	1,55

Table 4. Pallars's gypsum results at a mixing time of three minutes.

PALLARS 3 MIN	W/G	D. Y POROSITIES			MECHANICAL RESISTANCES		WATER MOBILITY		
	Water/ Gypsum Ratio	Apparent Density	Relative Density	Porosity	T Flexión	T comp	c.A.C	Se	Coef. evaporación
		g/cm3	g/cm3	%	MPa	MPa	$\Delta Kg/m^2/\sqrt{h}$	%	%
	0,34	1,39	2,54	45,13	1,88	6,51	19,87	14,53	1,54
	0,37	1,44	2,56	43,67	2,56	6,64	19,30	15,17	1,40
	0,40	1,44	2,55	43,37	2,29	7,08	18,59	14,21	1,56

Table 5. Albarracín gypsum results at a mixing time of three minutes.

Albarracín 3 MIN	W/G	D. Y POROSITIES			MECHANICAL RESISTANCES		WATER MOBILITY		
	Water/ Gypsum Ratio	Apparent Density	Relative Density	Porosity	T Flexión	T comp	c.A.C	Se	Coef. evaporación
		g/cm3	g/cm3	%	MPa	MPa	$\Delta Kg/m^2/\sqrt{h}$	%	%
	0,34	1,46	2,47	40,63	1,43	3,04	20,79	10,65	2,16
	0,37	1,46	2,46	40,53	1,21	2,53	21,58	9,22	1,81
	0,40	1,40	2,49	43,93	1,06	2,04	28,09	6,64	1,80

All the test pieces showed many similarities between Albarracín and Pallars plasters. However, different variations could be observed due to the different water / gypsum ratios and the established mixing times.

Results and Discussion

Densities and Porosities

The summary of the necessary parameters (apparent density and relative density) to calculate the percentage of porosity for Albarracín gypsum, as for Pallars at its different water/ gypsum ratios are noted in Tables 2-5.
It is observed, in all cases, that the relative density is much higher than the apparent density, being logical since the relative density refers to the compact mass without pores.

Nevertheless, it is evident the relation that exist between the quantity of water in the piece and the time of kneading to form the porous network of the piece. In such way that, in a short mixing time (1 min), the greater the amount of water added to the piece, the greater the porosity created in it is. This happens, since the mixing time to which it is exposed is sufficient for the gypsum paste to absorb the particles of water needed to work, but insufficient to be able to evaporate the excess of water. On the other hand, in a longer mixing time (3 minutes), the greater the amount of water in the piece, the smaller the network of pores that is formed, as there is enough time to be able to evaporate the excess water found in the piece.

This means that, having two mixtures with the same amount of water but mixed at different times (one and three minutes), as result, it will end up with a different porosity.

Mechanical Properties

The factors that can affect the mechanical resistance of the gypsum can be many and it is difficult to establish a relation between the properties of this, because they do not influence equally in all cases.
The values of the tension and compression strength of our samples are established in Tables 2-5. In these tables, it can be observed that in average, the highest results for the tensile load are found in the Pallars gypsum at a mixing time of three minutes; while the lowest results are found in Albarracín gypsum at a mixing time of one minute. Furthermore, comparing all the results from both gypsums, the highest result is found in the ratio water/ gypsum= 0.37 at a mixing time of three minutes of the Pallars gypsum, while the lowest result is found in the ratio water/ gypsum= 0.40 of the Pallars gypsum as well, but at a mixing time of one minute, with a difference of 68.35%.
On the other hand, the values obtained from the compression load are found linked to those of the tensile load. In average, the samples with the highest resistance to compression load correspond to those made with the Pallars gypsum at a mixing time of three minutes, with the load values from 6.51 MPa to 7.08 MPa. Meanwhile, the

samples with the lowest compression resistance correspond to those made up with the Albarracín gypsum at a mixing time of one minute, with load values from 1.89 MPa to 1.95 MPa.

Comparing the highest results (Pallars gypsum), against the lowest ones (Albarracín gypsum), we found a difference of up to 73.30%.

Water Transport

The purpose of the water characterization of our gypsum is to evaluate the ability to capture or lose water, in liquid or vapor form. This is of great interest in order to be able to assess the behavior of the material in front of the alteration.
The values obtained from the coefficients of capillary water transport and water desorption for both gypsums are summarized in Tables 2-5. In the absorption by capillarity, the results that we found are very similar to each other, the difference is that for Pallars gypsum the highest values were recorded in the mixing time of one minute, while, for Albarracín gypsum, the highest values are found in the mixing time of three minutes.
On the other hand, it is clearly demonstrated that porosity influences the water absorption. In the graphs (Fig. 4-5) we observed that the porosity increases according to the amount of water added and in turn the capillary coefficient increases according to the percentage of porosity.

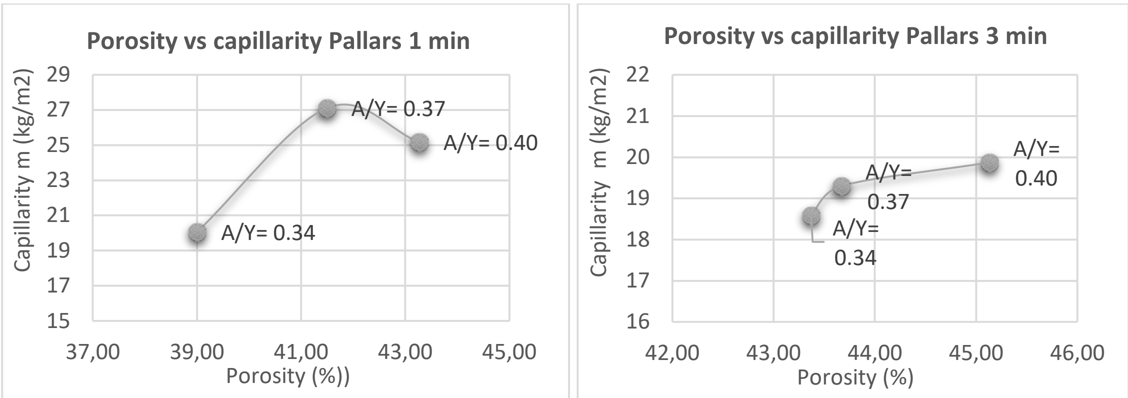


Figure 4. Pallars's gypsum graphics "Porosity vs capillary water transport" in a mixing time 1 and 3 minutes.

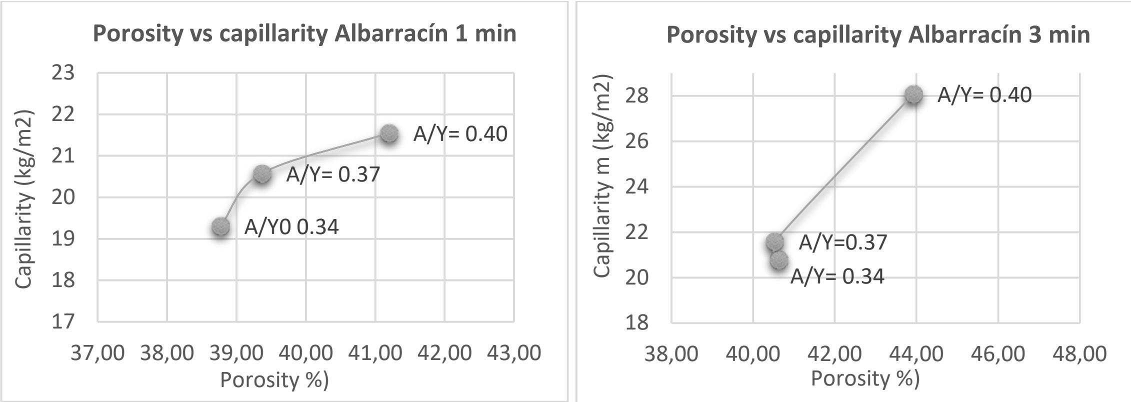


Figure 5. Albarracín gypsum graphics "Porosity vs capillary water transport" in a mixing time 1 and 3 minutes.

Likewise, in the desorption of water, the variations that happen in the loss of liquid of a material depend on the porous network from the same one. The values obtained from the test (Tables 2-5) refer to the percentages of the total water that remained in the piece at the end of a certain time.

In the graphs (Fig. 6-7), we observe that the porosity that exists in the Pallars pieces at a mixing times of one and three minutes, as well as the Albarracín samples at a mixing time of 3 minutes, grow as the W/ G ratio used increases, and at the same time, the percentage of water found in the piece at the end of the desorption test decreases. This is easy to deduce with our porosity values, as them indicates the percentage of existing pores that go from the outside to the inside of the piece, and these in turn, allow a better ventilation in the sample and simpler evaporation of water.

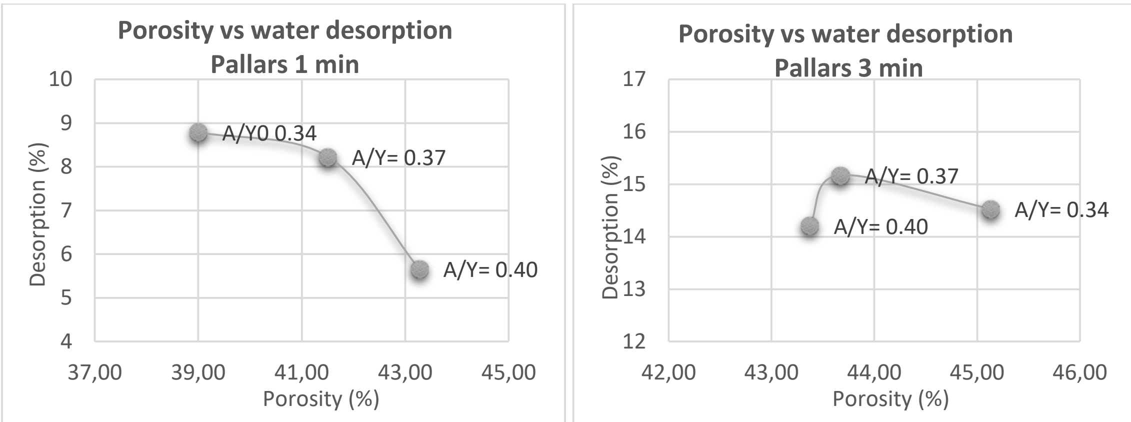
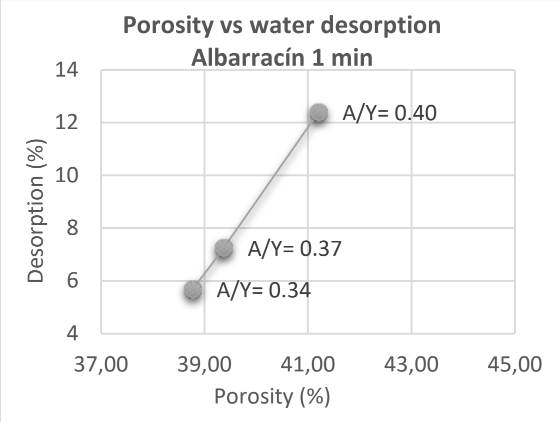


Figure 6. Pallars's gypsum graphics "Porosity vs water desorption" in a mixing time 1 and 3 minutes.



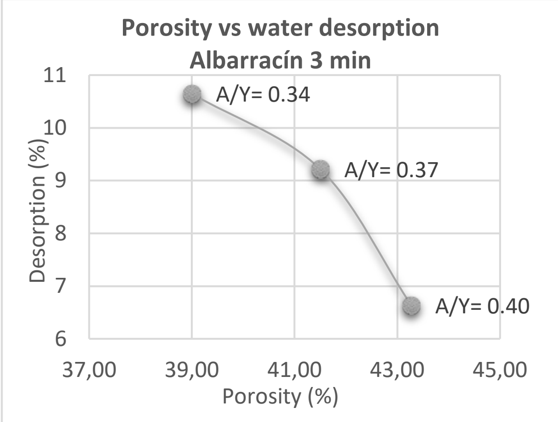


Figure 7. Albarracín gypsum graphics "Porosity vs water desorption" in a mixing time 1 and 3 minutes.

In the graph (Fig. 7) referring to Albarracín plaster at a mixing time of one minute, it is observed that the porosity vs desorption curve behaves completely different from the others, being the opposite of them. On the one hand, like all other samples (test pieces of Pallars at a mixing times of 1 and 3 minutes and test pieces of Albarracín at a mixing time of 3 minutes), the greater the amount of water used in the W/G ratio, the greater the percentage of porosity found; but, the greater the percentage of existing pores, the greater the percentage of water retained in the sample, concluding that the evaporation rate was slower.

Conclusions

The aim of the study was to compare some of the physic-mechanical characteristics of the Albarracín gypsum and Pallars gypsum. Thus, based on the results obtained in the tests that were carried out, the following conclusions are presented.

There are many similarities in the results obtained in the two types of plaster, presenting some notable differences in the kneading times.
Despite the similarities in the results obtained, in the mechanical tests of tensile load and compression load, the Pallars gypsum proved to be more resistant than Albarracín gypsum. In addition, the highest values could be found at higher mixing time.

The porosity test confirmed the hypotheses about the relation between the number and size of pores with the water/gypsum ratio, in conjunction with the mixing time. On one side, the percentage of porosity will be higher, the greater the excess of liquid is in the plaster during the drying and setting process, since this excess at the moment of evaporation will form air bubbles. On the other hand, the network of pores will also be higher if there is an insufficiency of liquid, since the plaster will absorb all the water leaving empty spaces.
Therefore, big percentages of porosity can be found in low water/gypsum ratios at high kneading time, as well as high water/gypsum ratios at short kneading times.

Finally, from the results and graphs of the water transport tests, it is concluded that the two types of gypsum absorb water through their capillaries at a high speed. In addition, as expected, the absorption and desorption properties are related to the percentage of porosity that exists in the samples.

Given that no excessive differences were found between the qualities of the Pallars gypsum and Albarracín gypsum, it is deduced that the mineral found in Pallars could be suitable for use in construction; however, this would require a broader study that includes the composition of the material.

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