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Water Resistance Analysis of New Lightweight Gypsum-Based Composites Incorporating Municipal Solid Waste

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Abstract: Incorporating waste to produce new environmentally friendly construction products has become one of the great challenges of the industry nowadays. The aim of this research is to analyse the behaviour of novel gypsum composites against water action, incorporating recycled rubber aggregates (up to 8.5% vol.) and dissolved expanded polystyrene (up to 10.0% vol.). To this end, a total of 10 dosages have been proposed with the progressive substitution of natural resources by these secondary raw materials. The results show how it is possible to reduce the total water absorption of the gypsum composites by up to 8.3% compared to traditional gypsum material. In addition, it is also possible to reduce water absorption by capillary by up to 52.7%, resulting in lighter composites with good performance against water action. In all composites analysed, the mechanical strengths exceeded the minimum values of 1 MPa in bending and 2 MPa in compression, making them an optimal solution for the development of lightweight prefabricated products for damp rooms.

Keywords: gypsum; end-of-life tyre; expanded polystyrene; water resistance; circular economy

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1. Introduction

Construction gypsum is nowadays considered one of the most environmentally friendly binders due to three key factors: (1) its low production temperature compared to cementitious materials [1]; (2) its raw material ($CaSO_4$-$1/2H_2O$) is generated as a by-product in many industrial processes [2]; and (3) it is a product that is 100% recyclable on a regular basis through the following reaction scheme (1) [3]:

$$CaSO_4{\cdot}2H_2O \xrightarrow{105\ to\ 200^\circ C} CaSO_4{\cdot}\frac{1}{2}H_2O + \frac{3}{2}H_2O \rightarrow CaSO_4{\cdot}2H_2O + \Delta Heat \quad (1)$$

For all these reasons, it is not surprising that this material is currently being extensively studied with the aim of promoting its application in the construction sector. Gypsum-based materials have excellent qualities for use as interior cladding in dwellings due to their high capacity for hygrothermal regulation [4]. In addition, they are widely used for the development of prefabricated housing panels and boards, where their application extends to the execution of false ceilings, interior partitions or façade cladding [5,6]. It is within these prefabricated systems that gypsum composites have the greatest potential for the integration of recycled raw materials. This is because natural resources (gypsum and water) can be largely replaced by secondary raw materials from solid urban waste (SUW) [7]. By relying on the non-structural characteristics of these prefabricated gypsum products, manufacturers can increase the recycled material content without reducing the functionality of these building systems [8].

However, one of the great technical difficulties arising from the use of gypsum is its low resistance to water, which has limited its application to building interiors [9]. In environments where there is high humidity values or where gypsum is in direct contact with water, this material shows a high degree of solubility and mechanical strength loss [10].

For this reason, researchers have shown increasing interest in improving the strength of gypsum-based materials in humid environments [11]. In general terms, as highlighted by Doleželová et al. in their research, there are two methods to improve the water resistance properties of gypsum composites [12]: by surface coating or by modifying the matrix with hydrophobic additions [11,13].

This work combines this double objective, since on the one hand, recycled rubber aggregates with hydrophobic properties are added, and on the other hand, dissolved expanded polystyrene (EPS) is incorporated to waterproof the matrix of the gypsum composite. In any case, a preliminary review of the existing literature should be carried out in order to contextualise this research and briefly describe the recent studies conducted to improve the waterproofing properties of gypsum-based materials.

Regarding waterproofing additions, Li et al. have recently conducted research on the addition of an organosilicon waterproofing agent, determining that the appropriate proportion to be added to obtain good water resistance performance without impairing the mechanical strength is 1.0% [13]. In line with this research and trying to enhance the use of gypsum in humid environments, Wang et al. used a self-prepared organic waterproofing agent in combination with hypromellose to improve the water resistance of these composite materials [14]. The prepared organic compound consisted of a mixture of Polyvinyl alcohol, para-toluene sulfonic acid, $CaCl_2$ and Tween 80, which reduced the total water absorption rate of the gypsum from 20.4% to 10.5%, reducing porosity and capillary absorption. The use of polymethylhydrosiloxane (PMHS) as an additive has also improved the water performance of gypsum composites, as its incorporation increases the surface roughness and decreases the surface energy, making it more difficult for the liquid to wet the composite surface [15].

The modification of gypsum surface by adding potassium methylsilicate to improve its hydrophobicity, which creates a light surface film that hinders the contact between water and the composite, has also been recently addressed [16]. Similarly, Xu et al. modified the microstructure of gypsum composites by using pregelatinised starch and hydrogen–silicon oil, prepared by pressing. They successfully reduced the water absorption rate of the material to less than 1.2% [17]. Finally, it is worth mentioning the treatment of gypsum composites by surface painting [18]. Depending on the nature of paint used (plastic, tempera, etc.), a greater or lesser degree of water penetration through the surface of the material can be achieved. This treatment modifies the hygroscopic properties of the composites, influencing their potential application.

Regarding hydrophobic additions, the incorporation of plastic waste into gypsum composites' matrix to make them more waterproof has been highlighted in recent years. Romero-Gómez et al. used polypropylene waste from crushed coffee capsules as an addition to improve the water resistance of gypsum composites [19]. In this way, additions ranging from 2.5% to 10% by the weight of these residues were used, and the height reached by the water in the capillary test was reduced by up to 35.5% for the composite with the highest residue content. Recently, in line with the previous research, it has been observed that the addition of recycled rubber from end-of-life tyres (ELT) and mineral wool fibre improves the resistance to water action, reducing the capillary height by 31.9% and the water vapour permeability by 28.4% [20]. A similar effect was observed by Ferrández et al. when adding shredded waste from single-use bags to the matrix of gypsum composites, where the effect of this recycled LDPE produced a decrease in capillary water absorption of up to 28.1% [21]. In line with this work, Vidales-Barriguete et al. carried out an investigation by adding up to 70% by volume of plastic waste from the coating of electrical cables to improve the water performance of gypsum composites [22]. In this research, a reduction of up to 50.0% in total water absorption and 17.5% in water vapour permeability was achieved with respect to the traditional gypsum material. In other research, Aybar et al. achieved a reduction of 4% in the total water absorption of gypsum composites by adding crushed tennis ball waste to gypsum composites [23]. The replacement of the original gypsum material by these secondary raw materials was 20%, highlighting the relevance of the rubber nature of these wastes in improving the water-absorption properties of gypsum-based materials.

Finally, it is worth highlighting the relevance of the use of the two wastes used in this research. On the one hand, end-of-life tyres (ELT) represent a major sustainability problem worldwide, especially in developing and transition economies where their inefficient management persists despite legislative efforts [24]. Every year, more than two billion tyres are produced worldwide, generating about 20 million tonnes of poorly managed ELT [25]. In the European Union alone, approximately 3.5 million tonnes of ELT are produced annually and only 52% are recycled [26]. The following three by-products can be obtained from these ELTs that are suitable for construction: steel fibre for the reinforcement of mortars and concretes [27], textile fibre with excellent properties as a thermal insulation material [28], and rubber aggregates commonly used in the manufacture of roads and bituminous mixtures [29]. This research focuses on the use of the latter for the development of gypsum precast composites, which is successfully supported by previous work [30]. In general terms, the addition of these wastes results in gypsum composites with improved thermo-acoustic behaviour and acceptable mechanical properties, which can be improved by the incorporation of reinforcement fibres in the matrix [31,32].

On the other hand, EPS waste has been commonly used to lighten gypsum composites and improve their thermal and acoustic performance [33]. It is a very voluminous waste, with a slow degradation process, and is frequently generated in façade rehabilitation works [34]. Among its disadvantages is the decrease in mechanical properties caused by its integration into the matrix of gypsum-based materials [35]. For this reason, some researchers have tried to improve their properties by pre-heating these residues to reduce their size [36], or by incorporating reinforcement fibres to improve bending strength in prefabricated products [37]. However, recent research has shown that integrating these materials in their liquid state results in a more homogeneous and resistant composite. This advancement has led to the patenting of a new prototype of lightweight precast with improved physical-mechanical properties [38].

The novelty of this research is highlighted by the combined use of two water-repellent additions, namely recycled rubber aggregates and EPS dissolution. These materials are added as a partial replacement of the original gypsum composite, with the consequent substitution of natural resources by secondary raw materials. In this way, a novel construction material is developed under circular economy criteria and its possibilities are explored for its application in prefabricated wet rooms of dwellings. The aim of this work is to study the water resistance properties of these novel gypsum composite materials. For this purpose, a complete experimental campaign is carried out, where several tests have been performed according to current standards and the obtained results are discussed in comparison with previous research.

2. Materials and Methods

This section describes both the raw materials used to make the different gypsum composites and the sample preparation process. It also includes the experimental programme designed to analyse the behaviour of these new materials under the action of water.

2.1. Employed Materials

The following raw materials were used to produce the composites developed in this research: gypsum, water, recycled rubber from end-of-life tyres (ELT), expanded polystyrene (EPS) and universal solvent.

2.1.1. Natural Raw Materials

Firstly, E35 plaster of the IBERYOLA® brand (Saint-Gobain Placo Ibérica, S.A., Madrid, Spain) was used as binder. It is a type A gypsum according to the classification of the UNE-EN 13279-1 standard [39]. The most relevant properties of this building material are listed in Table 1 and have been provided by the manufacturer.

Table 1. Properties of the E35 plaster used in the production of the composites.

λ (W/m·K)	pH	Purity (%)	Granulometry (mm)	Setting Time (min)	Fire Reaction (Euroclass)
0.30	6	90–92	0.0–0.2	15–18	A1

On the other hand, drinking water from the Canal de Isabel II (Comunidad de Madrid, Spain) was used for the mixing of the compounds. This is tap water suitable for human consumption that has been successfully used in previous research [40,41]. Its main characteristics are [42] a hardness of 58.5 mg/l $CaCO_3$, a total chlorine amount of 1.29 mg/l, a pH of 8.15 and an electrical conductivity of 188.04 µS/cm.

2.1.2. Secondary Raw Materials

One of the objectives of this work is to address the management of solid urban waste and provide it with a second useful life through the redesign of conventional gypsum-based materials. Thus, the following recycled raw materials have been used for the elaboration of gypsum composite materials: ELT rubber aggregates and slurry.

The recycled rubber aggregates from ELT used have a particle size between 0–0.8 mm, known as the rubber powder fraction. Figure 1 shows the process of obtaining these products [43].

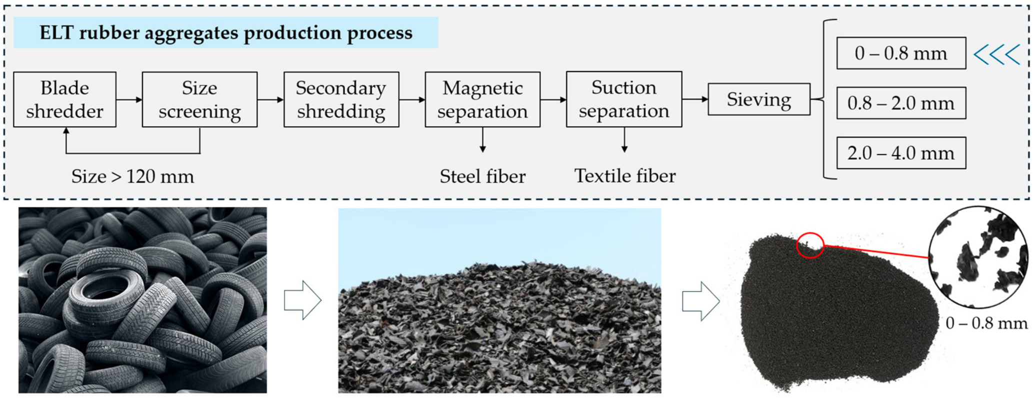


Figure 1. Production process of ELT rubber aggregates applied by SIGNUS Ecovalor S.L.

Therefore, as can be seen in Figure 1, rubber aggregates of angular morphology are obtained, which have a mean density of 1240 kg/m^3 and a moisture content of less than 0.75%. On the other hand, steel impurities do not exceed 0.1% of the total weight and the textile fraction is approximately 0.5% by mass. The manufacturing process ensures a continuous particle size distribution. Regarding the chemical composition of these ELT compounds, it is distributed as follows [44]: ketone extract (10–20%), polymers (40–55%), natural rubber (21–42%), carbon black (30–38%), ashes (3–7%) and sulphurs (less than 5%).

On the other hand, the slurry designed in this research is composed of a solution of EPS and universal solvent with a 1:2 ratio by mass. The EPS used in this work comes from the thermal insulation material discarded during the process of energy rehabilitation of dwellings. About 18.7 Mt of this waste is generated annually, which is even more alarming considering the low density of these materials (28–30 kg/m^3) [45]. Among its main physical properties are its low thermal conductivity 0.031 W/m-K and water vapour diffusion resistance factor $20 < \mu < 100$. On the other hand, the universal solvent used was supplied by Nazza (Madrid, Spain) and has the following properties [46]: density at 20 °C of 812 kg/m^3, a vapour pressure of 85.5 mmHg and a flash point of −8 °C. Furthermore, this product is composed of a mixture of toluene, xylene, n-butyl acetate, ethyl acetate, ethylbenzene, acetone, propan-2-one and propanone. Figure 2 shows the preparation process and a final image of the slurry, which has a liquid density of 660 kg/m^3.

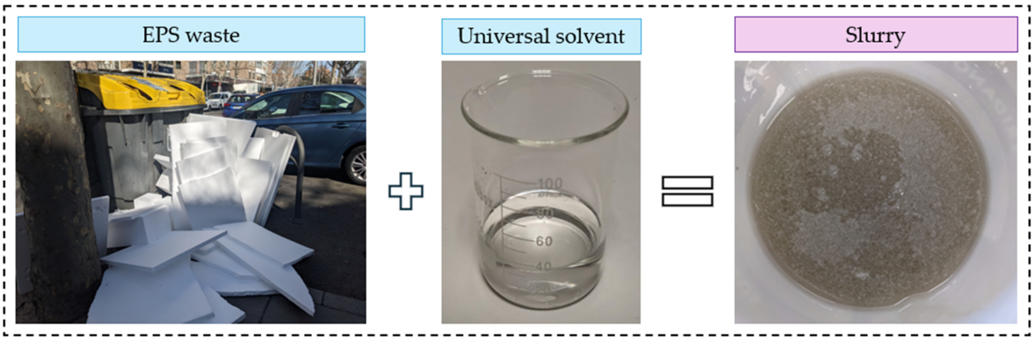
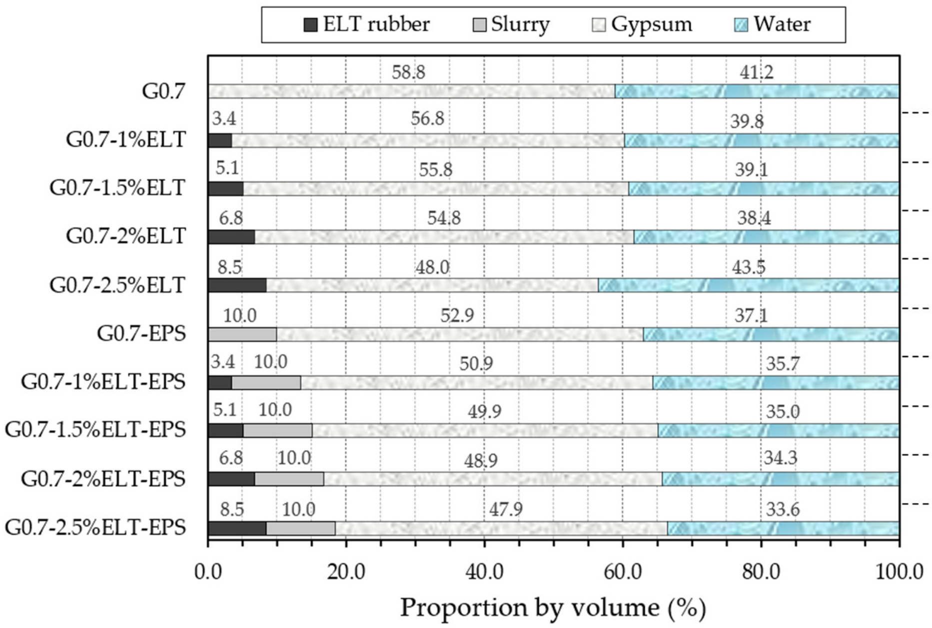


Figure 2. Preparation process of the slurry composed of EPS and universal solvent.

2.2. Sample Preparation Process

The proportions used by weight and volume for the different produced compounds in this research are shown in Figure 3.



Proportions by weight (g)

Gypsum	Water	ELT	Slurry
1000	700.0	—	—
990	693.0	17.0	—
985	689.5	25.5	—
980	686.0	34.0	—
975	882.5	42.5	—
900	630.0	—	170
890	623.0	17.0	170
885	619.5	25.5	170
880	616.0	34.0	170
875	612.5	42.5	170

Figure 3. Dosages used for the elaboration of the different compounds analysed in this research.

As can be seen in Figure 3, a progressive substitution of the original raw material with the recycled material was carried out in all samples. The series first produced contains only ELT rubber aggregates in its matrix, in percentages ranging from 1% to 2.5% by weight, progressively increasing the replacement in amounts of 0.5% by mass with respect to the total composite. On the other hand, in the second series, a replacement of 10% by volume of the composite with the EPS solution (called slurry in this study) is also achieved. In this way, up to 18.5% of the natural resources have been replaced by recycled material for the G0.7-2.5%ELT-EPS composite.

The same techniques and methods have been used for the manufacturing of all the produced composites, as described in the UNE-EN 13279-2 standard [47]. This process is schematically described in Figure 4.

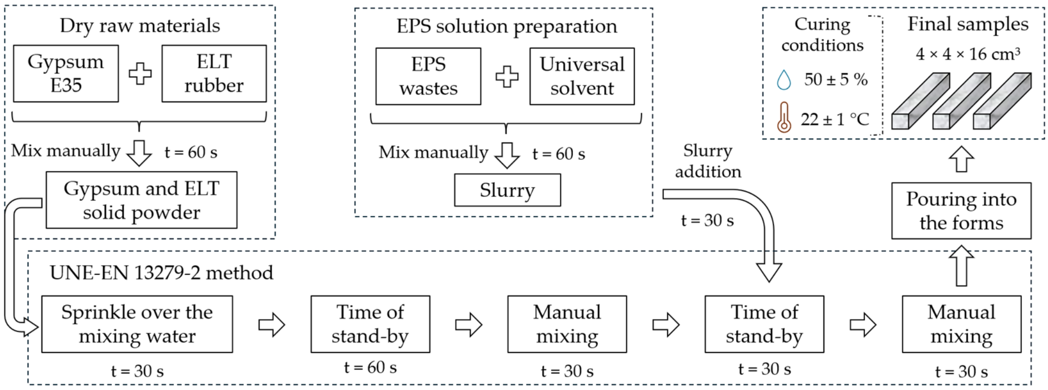


Figure 4. Sample preparation process according to the reference standard.

It is important to emphasise the importance of pre-curing the samples prior to testing. Thanks to the nature of the gypsum compounds that generate an exothermic reaction during the setting process, as well as the 24 h of drying in an oven to which the samples are subjected after seven days of age, the volatilisation of the possible residual solvent contained in the compounds takes place. In this way, the EPS solidifies inside the matrix and the residue is homogeneously integrated into the matrix together with the gypsum material. This drying from 24 h to seven days is carried out under the conditions of a relative humidity of 55 ± 5% and a temperature of 42 ± 1 °C.

2.3. Experimental Programme

For the development of this research, the experimental programme outlined in Figure 5 has been carried out.

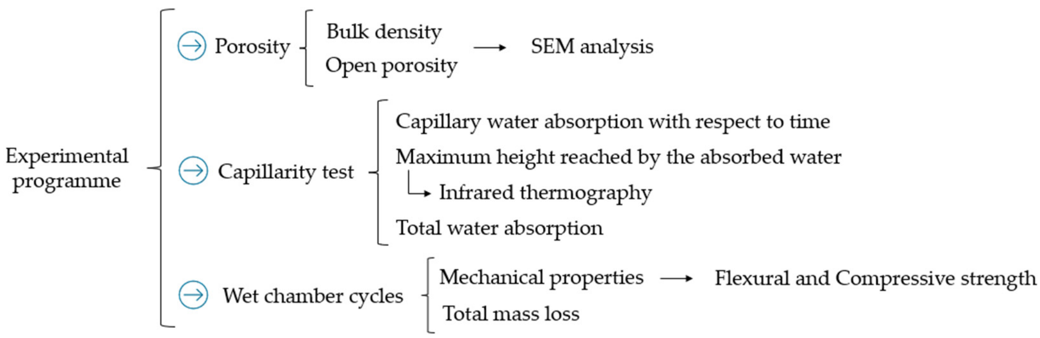


Figure 5. Simplified outline of the experimental programme developed in this research.

Firstly, the porosity of the gypsum composites developed in this research is determined. This property is related to bulk density and is used to analyse the effect of the incorporated residues on the lightening of traditional gypsum-based materials. These tests are described below:

Bulk density: Determined according to the method described in the UNE 102042:2023 standard [48]. This property is defined as the quotient between the apparent mass and the material volume, using three 40 × 40 × 160 mm^3 test samples of each dosage prepared. The mass is determined with the aid of an electronic balance of 0.01 g accuracy and the volume is measured with the aid of a digital caliper of 0.01 mm accuracy.

Open porosity: Obtained using the method described in the UNE-EN 1936:2007 standard [49]. This property is defined as the ratio between the accessible pore volume and the apparent volume of the material. It is determined by using three 40 × 40 × 160 mm^3 test samples of each dosage elaborated and using Equation (2):

$$OP = \left(\frac{W_{sat} - W_{dry}}{W_{sat} - W_{imm}} \right) \times 100\% \quad (2)$$

where W_{dry} is sample mass previously dried before testing, W_{sat} is sample mass saturated with water, and W_{imm} is sample mass saturated with water and weighed with the aid of a hydrostatic balance by immersing the sample into water.

Scanning electron microscopy (SEM analysis): This test is carried out to provide an overview of the microstructure of the produced composites. The G0.7-2.5%ELT and G0.7-2.5%ELT-EPS composites were analysed, as they are considered to be the most representative and have the highest content of recycled material. The images were obtained trying to collect as much information as possible and offering a generic view of the matrix. A TESCAN VEGA Generation 4 microscope was used to carry out these tests and the samples were previously coated with a gold film using a Cressington 108 metalliser.

The behaviour of the designed gypsum composites against the action of water by capillary action is then studied. This phenomenon is the main factor responsible for the transport of liquid water in porous materials and is closely related to the durability of the material [50]. For this reason, it is important to analyse this property in construction materials, for which the following tests have been conducted:

Capillary test: Determines the capacity of the material to absorb water per unit of time and surface area. This property has been determined in specimens of 40 × 40 × 160 mm^3, following an adaptation of the method contained in the UNE EN 1925:1999 standard [51]. The capillary absorption coefficient was determined using the following equation, Equation (3):

$$C_{abs} = \frac{W_t - W_{dry}}{A \cdot \sqrt{t}} \quad (3)$$

where W_t is the sample mass at time instant t, A is the surface area of the 40 × 40 mm^2 sample, which is positioned vertically in a container with water immersed one centimetre, and t is the test duration time.

Determination of the maximum height reached by water: This value in millimetres is obtained after carrying out the previous test. The height reached by water is measured using a 0.01 mm precision caliper, and then thermographic images are obtained to determine the advance of the capillary meniscus inside the composite.

Total water absorption: Determined according to the method described in UNE-EN 520 standard [52]. For this purpose, three samples of each dosage of dimensions 40 × 40 × 160 mm^3 are used and are completely immersed in water in a horizontal position for a period of 120 ± 2 min. After this period, they are removed from the water and weighed by drying them superficially. The total absorption coefficient is obtained using Equation (4):

$$Total_{Abs} = \left(\frac{W_{sat} - W_{dry}}{W_{dry}} \right) \times 100\% \quad (4)$$

where W_{sat} is the mass of the water-saturated sample and is obtained when the difference between two consecutive weighings is less than 0.1% and W_{dry} is the mass of the dried sample.

In a final phase, the produced gypsum composites were subjected to periodic wet chamber cycles. This is therefore a durability test to evaluate the behaviour of these new materials when used in damp rooms of dwellings. A control group has been used as a reference and another group subjected to cycles, with each group consisting of three samples of each dosage of 40 × 40 × 160 mm^3 size. This test has been adapted from the method initially proposed by Del Rio in her doctoral thesis [53], by performing five humidity chamber cycles following the same methodology: 24 h in a humid chamber (95 ± 1% relative humidity and 19 ± 2 °C) and 24 h in a stove (40 ± 1% relative humidity and 40 ± 2 °C). Thus, the following properties concerning these composite materials have been studied:

Flexural strength: Determined according to the indications of UNE-EN 13279-2 standard [47]. For this purpose, an IBERTEST hydraulic press model AUTOTEST 200-10SW is used. This is a three-point bending test where a load speed of 10 N/s is applied until the sample breaks.

Compressive strength: This is also determined using the UNE-EN 13279-2 standard [48] and the same equipment as above. The test is performed on the halves of the obtained samples from the bending test, with a constant load of 20 N/s applied to a 40 × 40 mm^2 surface.

Total mass loss: This is the average percentage difference between the sample weights before and after being subjected to wet chamber cycles. These values were determined with the aid of an electronic balance of 0.01 g accuracy.

Finally, it should be noted that, following these tests and their discussion, a section on critical analysis and future applications of these compounds has been included to analyse their potential use in buildings.

3. Results and Discussion

This section presents the results after the development of the experimental programme, as well as their interpretation and critical discussion.

3.1. Bulk Density and Open Porosity

Firstly, Figure 6 shows the obtained results for the open porosity and its relationship with the bulk density in the developed composites. These results are of particular interest to understand the accessible pore volume and to study the waste effect on the lightweighting of these construction materials.

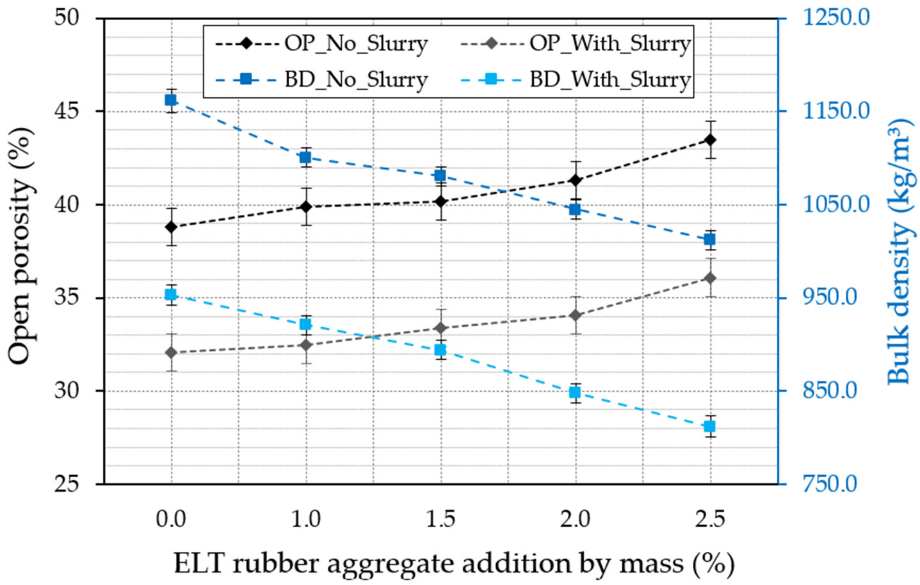


Figure 6. Open porosity and bulk density of the analysed gypsum composite materials.

It can be observed in Figure 6 that there is an inverse relationship between the open porosity and bulk density of the analysed composite materials. As the content of recycled rubber aggregates increases, the open porosity tends to increase, while the bulk density decreases, as observed in the research by López-Záldivar et al. [54]. On the other hand, while the incorporation of ELT rubber waste produces an increase in the open porosity of the composites, the addition of slurry causes the opposite effect. Thus, the series containing the addition of dissolved EPS in its matrix presents lower porosity values, while generating lighter composites as a consequence of the higher replacement of the original gypsum composite by recycled material [33]. Therefore, the beneficial effect of combining both wastes can be observed to generate lighter gypsum materials, which are optimal for prefabricated products. The composite with the higher content of recycled material, G0.7-2.5%ETL-EPS, has a bulk density 30.1% lower than the traditional material without

G0.7 additions. This reduction in weight may provide a competitive advantage in product differentiation, particularly in the development of lightweight prefabricated products that reduce on-site installation times [55].

To examine the microstructure of the produced gypsum-based materials in detail, SEM images for the two composites with the highest recycled material content in each series (G0.7-2.5%ELT and G0.7-2.5%ELT-EPS) are presented in Figure 7. The samples for this test were prepared in a specialised laboratory and extracted from the inner matrix of 40 × 40 × 160 mm^3 samples. The samples' surfaces were not treated in any way that modified the original morphology of the material.

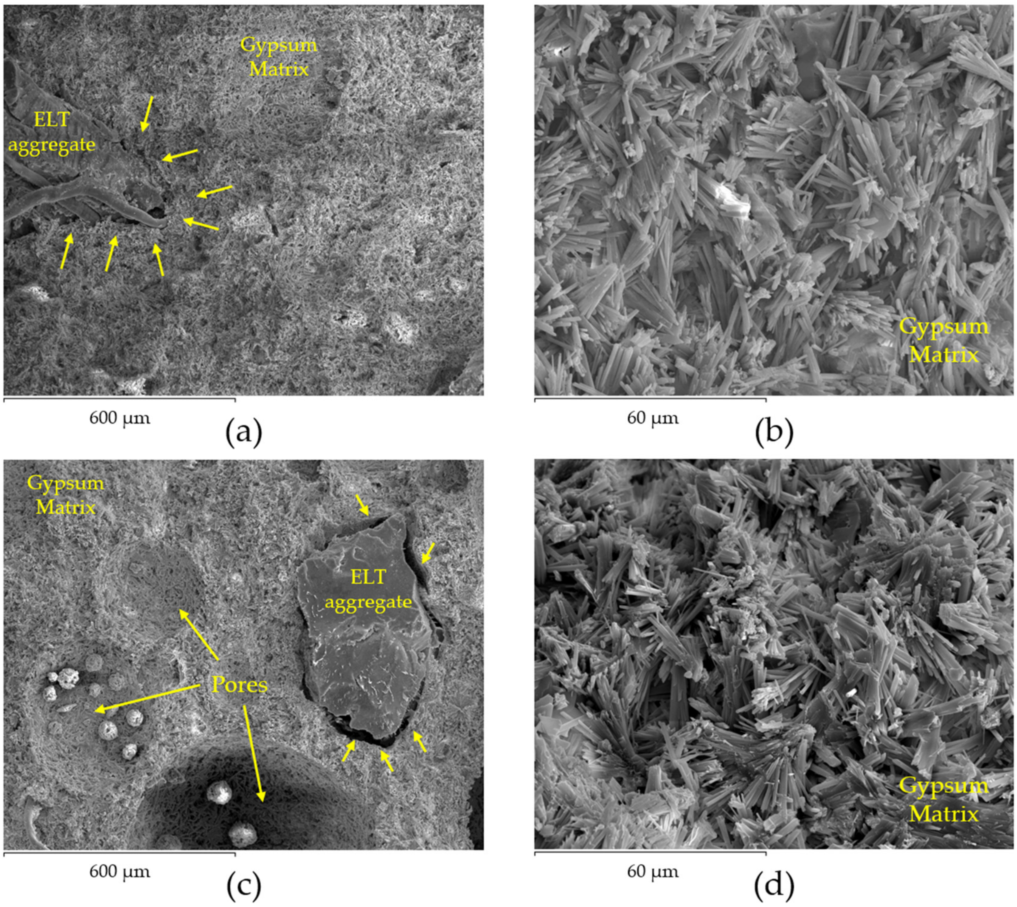


Figure 7. SEM analysis. G0.7-2.5%ELT sample: (**a**) 500× and (**b**) 1000×; G07-2.5%ELT–EPS sample: (**c**) 500× and (**d**) 1000×.

Firstly, Figure 7a shows how dihydrate crystals ($CaSO_4$-2H2O) have formed at the interface between the recycled rubber aggregate and the base material, favouring the integration of the residue and showing good adhesion [56]. On the other hand, Figure 7b provides a detail view of the gypsum matrix in the G0.7-2.5%ELT composite. The correct setting process of these composite materials is evidenced by their characteristic acicular crystallisation morphology [57]. On the other hand, Figure 7c shows an image of the G0.7-2.5%ELT-EPS composite, where a weaker integration of the rubber residue in the composite matrix can be appreciated. In this series with slurry incorporation, voids are observed between the rubber aggregate and the gypsum matrix. These voids could potentially facilitate the detachment of the ELT particles during sample fracturing. In addition, a more porous matrix is observed, which supports the higher lightening and lower density of these composites [58]. However, many of these pores are not accessible from the inside as shown in the obtained results in Figure 6. Finally, Figure 7d shows an image of this composite matrix at the same magnification as that obtained for Figure 7b. The main difference between these two is that Figure 7d shows shorter dihydrate crystals, as well as a dissolved EPS film that is homogeneously distributed within the composite matrix.

To complete this subsection, it is useful to know the total water absorption capacity of the composites developed for this research. These results can be seen in Figure 8 in detail.

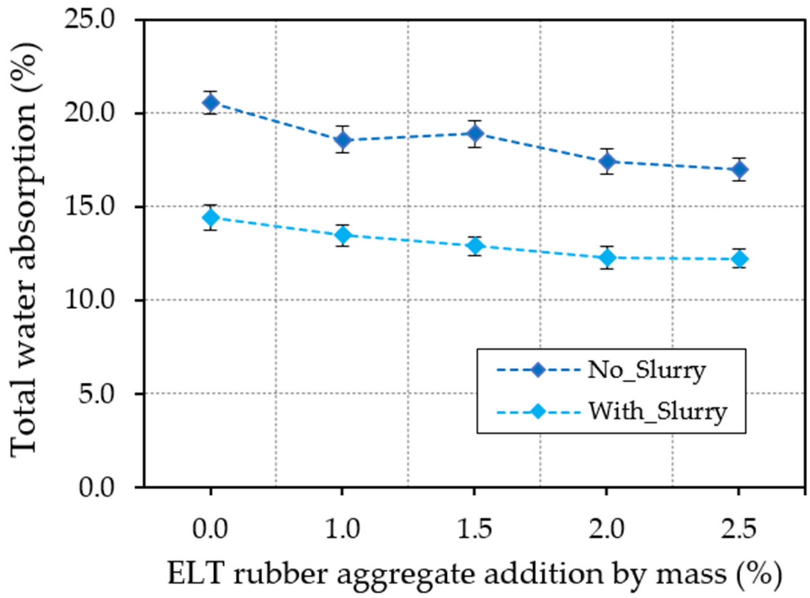


Figure 8. Total water absorption coefficient for the different tested compounds.

In line with the obtained results by Vidales-Barriguete et al. in their study on gypsum composites with the addition of recycled cable plastic aggregates, the total water absorption is reduced by increasing the ELT rubber content in the developed composites [22]. This effect is due to the impermeable nature of these added recycled materials, which has been corroborated in previous studies [59]. Thus, there is a difference of 3.5% between the reference composite (G0.7) and the G0.7-2.5%ELT sample. On the other hand, the composites with a 10% slurry replacement of the original gypsum exhibit even higher resistance to total water absorption. Thus, there is an average reduction of around 5.5% compared to the composites without added dissolved EPS. In any case, it has been possible to corroborate how the joint action of both secondary raw materials used allows for a considerable reduction of this total water absorption. In addition, future work should include conducting a contact angle test to confirm whether the added material has hydrophobic properties.

3.2. Capillary Tests

This section presents the obtained results after the capillary water absorption test. This phenomenon has been approached by first analysing the rate of water absorption per unit area, and then visualising the height reached by the water inside these composites. The results for water absorption per unit time and surface area are shown in Figure 9, and a detailed analysis of these graphs is presented in Table 2.

From the analysis of Figure 9, it can be seen that the incorporation of recycled rubber aggregates progressively reduces the rate of capillary water absorption in the gypsum composites. Furthermore, it can be observed that the series with slurry (Figure 9b) presents lower values than those obtained in the series without EPS dissolution (Figure 9a). Observing the results shown in Table 2, it is evident that the final absorption of the G0.7-2.5%ELT-EPS composite was 17.1 $kg/m^2min^{1/2}$, representing a 52.7% reduction compared to the traditional gypsum material without additions (G0.7). In this sense, it is corroborated that the joint action of these two recycled raw materials makes it possible to reduce the mass of water absorbed by capillary effect in gypsum-based composites. It should be noted that capillary water absorption is a phenomenon related to the distribution and interconnection of the capillary networks, so that when the slurry solidifies inside the composites, this connection can be interrupted, making it difficult for water to rise through the material [60].

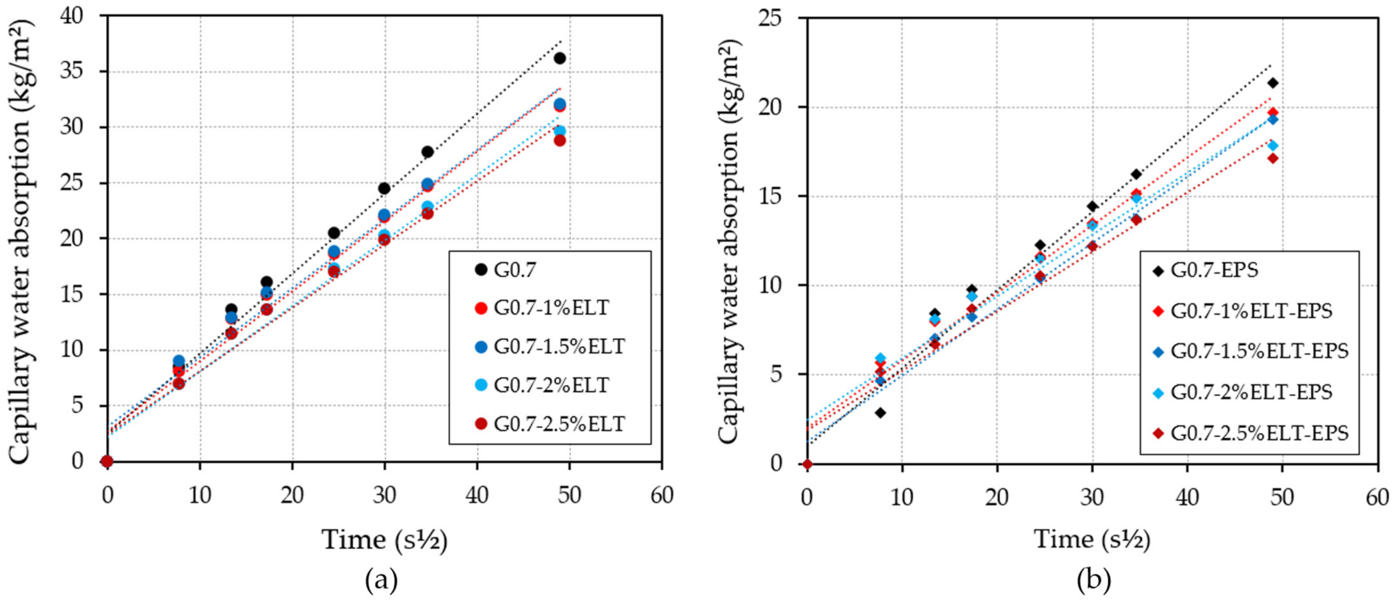


Figure 9. Capillary water absorption: mass absorbed per unit time and surface area. (**a**) Series without slurry, and (**b**) Series with slurry.

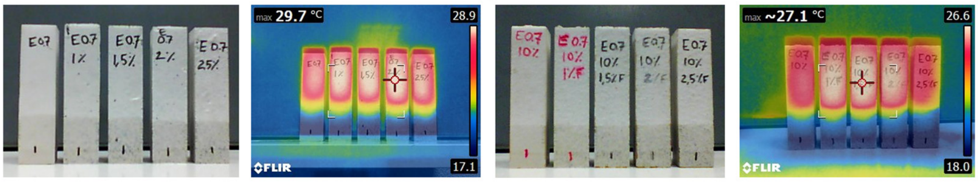
Table 2. Capillary water absorption test. Analysis of the obtained results in Figure 9.

Series	Equation	R^2	Maximum Value ($kg/m^2min½$)	Δ (%)
G0.7	$y = 0.7178x + 2.5003$	0.9864	36.2	-
G0.7-1%ELT	$y = 0.6220x + 3.1358$	0.9750	32.1	11.4
G0.7-1.5%ELT	$y = 0.6255x + 2.7920$	0.9784	31.9	11.9
G0.7-2%ELT	$y = 0.5872x + 2.2879$	0.9821	29.6	18.1
G0.7-2.5%ELT	$y = 0.5672x + 2.4620$	0.9778	28.8	20.6
G0.7-EPS	$y = 0.4381x + 1.0000$	0.9748	21.4	40.9
G0.7-1%ELT-EPS	$y = 0.3793x + 2.0094$	0.9729	19.7	45.6
G0.7-1.5%ELT-EPS	$y = 0.3723x + 1.2266$	0.9880	19.3	46.6
G0.7-2%ELT-EPS	$y = 0.3456x + 2.5032$	0.9466	17.9	50.6
G0.7-2.5%ELT-EPS	$y = 0.3339x + 1.8923$	0.9659	17.1	52.7

Figure 10 below shows the results for the height reached by the water after the capillary tests conducted on the different series of prepared samples for this study.

As can be seen in Figure 10, the height reached by water decreases as the recycled rubber aggregates content increases. Likewise, as in the total water absorption test, the gypsum composites with slurry addition showed greater opposition to water rising by capillary action. This is because the plastic residue of EPS in solution solidifies within the matrix of the gypsum composites, obstructing the capillary networks, as previously mentioned. In summary, it can be affirmed that the combined addition of both recycled raw materials in the gypsum matrix could increase its durability. This is because a reduction in the water absorption by capillary action reduces the probability of pathologies related to the degradation of the dihydrate [61].

Serie: Not slurry | **Serie**: With slurry



Sample	Maximum height (mm)	
G0.7	64.1 ± 0.2	
G0.7-1%ELT	63.8 ± 0.2	↓ 0.47%
G0.7-1.5%ELT	62.1 ± 0.3	↓ 3.12%
G0.7-2%ELT	58.3 ± 0.1	↓ 9.05%
G0.7-2.5%ELT	56.4 ± 0.2	↓ 12.0%

Sample	Maximum height (mm)	
G0.7-EPS	49.2 ± 0.1	
G0.7-1%ELT-EPS	48.9 ± 0.2	↓ 0.61%
G0.7-1.5%ELT-EPS	48.8 ± 0.1	↓ 0.81%
G0.7-2%ELT-EPS	48.1 ± 0.0	↓ 2.23%
G0.7-2.5%ELT-EPS	47.0 ± 0.2	↓ 4.47%

Figure 10. Maximum height reached by the water in the capillary test and thermographic images.

3.3. Wet Chamber Cycles

This section presents the results after subjecting the different gypsum composites to wet chamber cycles, as shown in Figure 11. It should be noted that the samples were placed at least 4 cm apart, and that the climatic chamber has a ventilation system to prevent the surface condensation of the water inside the chamber.

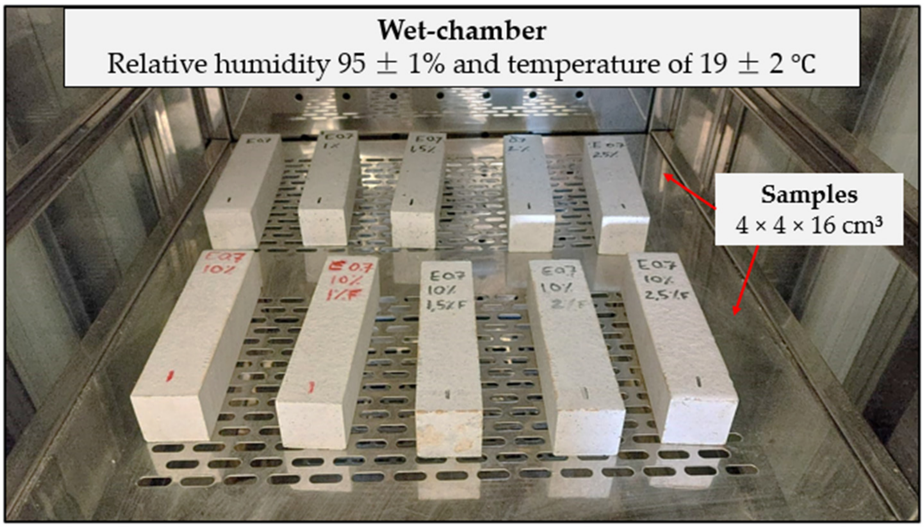
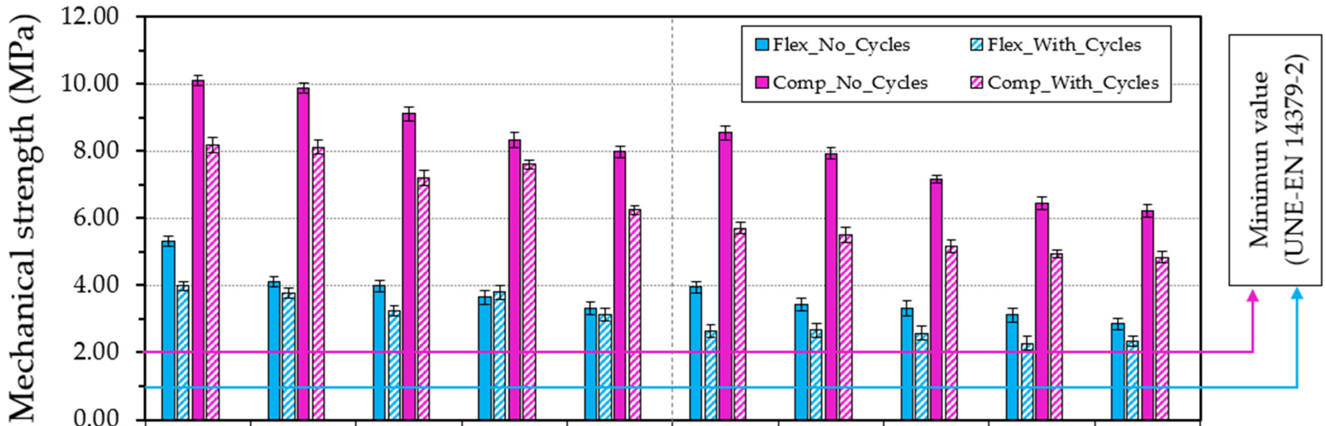


Figure 11. Sample series subjected to wet chamber cycles.

A total of five moisture-drying cycles were carried out under the conditions indicated in the methodology using a total of three samples from each series. The percentage mass loss experienced by the developed gypsum composites, along with their mechanical strength in bending and compression, was determined by comparing them with measurements from a set of three reference samples that underwent no cycles. These results are shown in Figure 12.

Firstly, Figure 12 shows that all the samples subjected to wet-chamber cycling experienced a slight loss of mass at the end of the test. Although there is no clear trend regarding this variation, it is possible to appreciate that the samples with a higher content of recycled rubber aggregates presented a lower mass stability after being subjected to this accelerated ageing test. Thus, the mass loss of the G0.7-2.5%ELT composite was 2.4% higher than that obtained for the base material without additions (G0.7). This effect has been previously observed by other researchers, who found that the mass loss in composites incorporating recycled raw materials was higher than that in traditional composites [62].



	G0.7	G0.7-1%ELT	G0.7-1.5%ELT	G0.7-2%ELT	G0.7-2.5%ELT	G0.7-EPS	G0.7-1%ELT-EPS	G0.7-1.5%ELT-EPS	G0.7-2%ELT-EPS	G0.7-2.5%ELT-EPS
Flex_No_Cycles	5.33	4.12	3.98	3.65	3.32	3.95	3.45	3.32	3.12	2.85
Flex_With_Cycles	3.98	3.78	3.25	3.79	3.14	2.64	2.66	2.58	2.28	2.34
Comp_No_Cycles	10.10	9.88	9.11	8.34	7.98	8.54	7.93	7.16	6.45	6.23
Comp_With_Cycles	8.20	8.11	7.21	7.60	6.24	5.71	5.51	5.17	4.96	4.84
Δ mass (%)	3.2	4.3	4.4	5.1	5.6	3.2	3.6	3.7	3.1	4.2

Figure 12. Results of mechanical tests on gypsum samples with and without wet-chamber cycles, including mass loss.

Regarding mechanical properties, it is important to highlight that all analysed composites exceeded the minimum values of flexural strength (1 MPa) and compressive strength (2 MPa), both in the reference series and in the series subjected to wet chamber cycles. Furthermore, in all cases, a decrease in mechanical strength is observed compared to the reference samples without accelerated ageing cycles.

Analysing the series without slurry addition, included in Figure 12, a progressive decrease in flexural and compressive strength is observed as the ELT recycled rubber content increases. This effect was observed by Serna et al. in their study on gypsum composites with added recycled rubber [30]. Thus, there is a decrease of 37.7% and 21.0% for flexural and compressive strengths, respectively, in the G0.7-2.5%ELT composite with respect to the traditional G0.7 material. However, despite these reductions, the G0.7-2.5%ELT composite exceeded the required minimum flexural strength by 2.32 MPa and the required minimum compressive strength by 4.24 MPa, even after wet chamber cycling. Therefore, after performing this test, it is understood that these gypsum composite materials are optimal for use in wet rooms. This increased wear under extreme humidity–dryness conditions can be attributed to the weakening of the gypsum-residue interphase, which can affect its microstructure and facilitate the detachment of the aggregate when subjected to breaking loads [22].

On the other hand, the series with a 10% replacement of the original gypsum composite by slurry showed a similar behaviour to that obtained in the counterpart series without the addition of EPS in solution. However, the mechanical strength was further reduced, for example, the G0.7-EPS composite shows flexural and compressive strengths that are 25.9% and 15.4% lower, respectively, compared to those of the traditional composite without G0.7 additions. Furthermore, a slight decrease in the mechanical strength of these composites is also observed when subjected to periodic humidity–dryness cycles. In this sense, although the addition of the EPS solution resulted in composites with a lower water absorption capacity, this effect does not significantly improve mechanical properties after accelerated ageing cycles as a consequence of the produced wear in the material [63].

3.4. Critical Discussion and Feature Applications

Once the results in the experimental campaign have been presented, it is convenient to carry out a critical review of them, and to explore the potential applications of these developed composites. In this way, the graph shown in Figure 13 has been elaborated, which visually shows a quantitative and qualitative classification of the different gypsum-based materials according to the obtained results for each property. This classification has been achieved by dividing the average values reached by each dosage in each physical-mechanical test by the maximum value for the analysed property. In this way, the dosage that presents a value of 1.00 in each property corresponds to the series with the highest value among all the analysed gypsum composites.

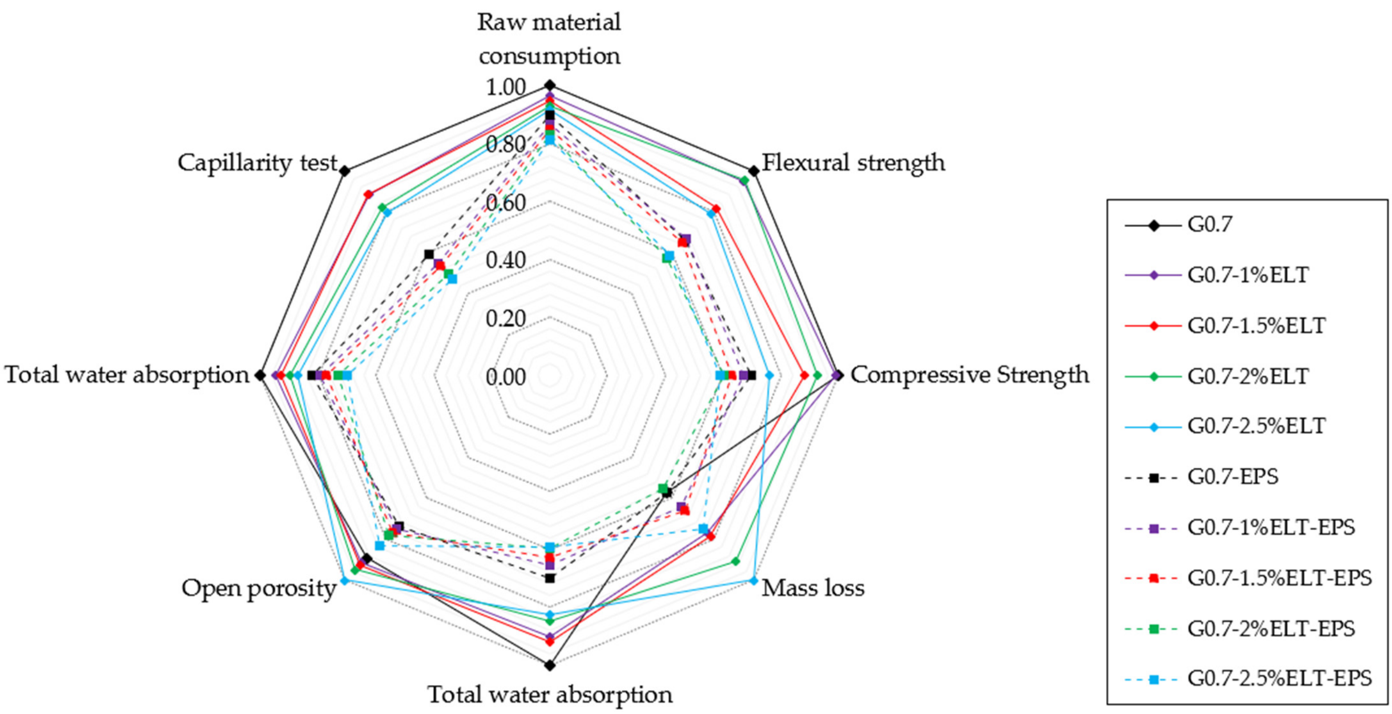


Figure 13. Comparative analysis for the different elaborated dosages in each test performed in this research.

After analysing Figure 13, a progressive decrease in the consumption of natural resources with respect to the base material (G0.7) can be observed. Circular economy strategies, such as the one presented in this research, usually require planning that enables the reincorporation of secondary raw resources in the manufacturing process of new sustainable building materials [64]. In this regard, it is important to have on-demand systems or storage spaces that facilitate the stockpiling of prefabricated products made from recycled raw materials to avoid possible mismatches in the supply chain [65]. Thus, while it is true that the developed composites in this research incorporate two potentially available urban wastes (ELT and EPS), the effective management of these second-life raw materials is crucial to promote their use in the construction industry [66].

Regarding the analysis of the mechanical properties of these gypsum composites after being subjected to humidity and dryness cycles, a decrease in strength has been observed in those dosages that incorporate a greater amount of recycled material. This effect is frequent in gypsum composites that incorporate recycled raw materials. However, due to the non-structural nature of the prefabricated products made with these construction materials (i.e., ceiling panels, gypsum plasterboards, etc.), it is not a property that limits their application [7]. In this sense, for these construction systems, it is more relevant to achieve a reduction in the density of the prefabricated products. This reduction allows us to produce lighter modular parts that are easier to install on-site and that help reduce transport costs [67].

Finally, in line with the objective of this research, the analysis of the properties related to water exposure has demonstrated the positive effect of incorporating these plastic wastes in the manufacture of gypsum composites. Thus, a decrease in water absorption has been observed in all the composites compared to the reference material (G0.7), although it has also been experimentally proven that the incorporation of recycled rubber aggregates increases the open porosity of these composites. This effect has been previously reported by other researchers [22,54], who coincide in highlighting the benefit of adding plastic waste to improve the water resistance properties of gypsum-based composites. In this sense, the use of these secondary raw materials is postulated as an alternative for the development of prefabricated elements for wet rooms, which, in turn, would present a competitive advantage in product differentiation, as they are more environmentally friendly. Additionally, it would be interesting to carry out a life-cycle analysis (LCA) of these products in order to provide, through a reliable tool, a complete overview of the produced environmental impact, which is one of the main limitations of this research paper. On the other hand, it would also reduce the final price of these modular pieces, which would represent a competitive advantage in costs, reducing the demand for natural resources, which currently oscillates around 1.8 million tons annually for the construction industry in Europe [68]. Analysing the feasibility of generating economies of scale from a macroeconomic approach would be valuable, considering the supply of secondary raw materials, the increased production costs and the analysis of the product supply chain.

4. Conclusions

In this work, a novel gypsum composite has been developed and patented with registration number ES 2 933 873 B2 [69]. This construction material has been elaborated under circular economy criteria, replacing part of the original raw material by ELT and EPS waste up to 18.5% in volume. This has enabled the development of gypsum-based material suitable for use in the production of plates and prefabricated panels with good performance against water exposure. The main conclusions that can be drawn from the developed experimental programme are as follows:

- A progressive decrease in bulk density is observed with the addition of a higher content of secondary raw materials in the gypsum composites, reaching a reduction with respect to the traditional gypsum material of up to 30.1% for the G0.7-2.5%-EPS composite.
- The open porosity of the composites increases progressively with the addition of recycled rubber aggregates in the gypsum composites. However, all series decreased by about 6.0% on average when incorporating the EPS residue in the solution for the same amount of ELT aggregates.
- Microscopy has shown how the incorporation of the EPS solution slightly weakens the adhesion between the rubber aggregate and the gypsum matrix, although a homogeneous distribution of the residue and a correct setting of the binder are observed.
- The total water absorption has been reduced by 8.3% compared to the reference material G0.7, which shows the beneficial effect of the addition of these residues to generate more impermeable gypsum materials.
- A decrease in capillary water absorption has been achieved in all the produced gypsum composites compared to the reference material without additions. The maximum reduction was observed in composite G0.7-2.5%ELT-EPS, with a mass absorbed per unit time and surface area at the end of the test of 17.12 $kg/m^2 min^{1/2}$, a decrease of 52.7% compared to sample G0.7.
- Similarly, the maximum height reached by water after this test was reduced by up to 26.6%, representing a significant advancement in promoting the use of these materials for developing prefabricated elements for wet rooms.

- Finally, a slight mass loss has been observed in all the composites when subjected to five moisture–dryness cycles, ranging from 3.1% to 5.6%. It was also observed that the mechanical strength decreased progressively as the recycled material content increased. However, in all the cases analyzed, the produced samples obtained flexural and compressive strengths higher than the minimums set by current standards by 1 MPa and 2 MPa, respectively (even when subjected to durability cycles).

The limitations of this research and future lines of work include the possibility of completing the study by performing mercury porosimetry tests to gain in-depth knowledge of the real pore size and distribution, the skeleton density, and the tortuosity of these composites. Likewise, in order to enhance their application in wet rooms, it would be relevant to know the water vapor permeability of these novel construction materials and to analyze the possible generation of fungi in high humidity conditions, which is a frequent pathology for the prefabricated gypsum used in wet rooms of dwellings. In addition, in order to obtain a more complete mechanical characterization and to corroborate the suitability of these materials in wet rooms, it would be recommended to carry out mechanical tests with the wetted samples.

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