Article

Analysis of the Resistance to Bending of Gypsum with Added Graphene

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Abstract: Gypsum-based interior coatings are used due to their excellent fire resistance and thermal and acoustic properties. In recent decades, the development of these calcium sulfate-based coatings has been linked with the use of new additives that allow the setting times and the water content of the mix to be controlled and offer the possibility of adding different aggregates to the mortar, which further improve its resistance properties. In this article, significant results are presented that compare the flexural strength of mortars formed using added construction plaster with different percentages of graphene powder.

Keywords: gypsum; graphene; resistance; bending; additives

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1. Introduction

Gypsum is a natural stone, called aljez or gypsum stone, composed mainly of calcium sulfate crystallized with two water molecules ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), called calcium sulfate dihydrate or dihydrate. In the dehydration of the stone, the rock should not be subjected to temperatures exceeding 180 °C. The powdered product obtained from the calcination and grinding of the stone described above is composed of several anhydrous or semi-hydrates of the compound, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, which, when mixed with water, has the ability to harden through a physical-chemical process called setting.

In this developed and globalized world, which has witnessed the development of and advances in many new technological, the research and development of new materials are continuous, mainly existing materials are evolving with the aim of improving their mechanical properties, optimizing their production, or achieving an economic improvement.

The case presented in this work is an analysis of the mechanical properties of calcium sulfate added to a material, such as graphene.

Since graphene was discovered by physicists. Geim and Novoselov, researchers at the University of Manchester, who won the Nobel Prize for their pioneering experiments on the two-dimensional material graphene, have had careers in different institutions, research centers, and universities in relation to the research of this material and its applications. Therefore, we aimed to study whether improvements in the mechanical properties of calcium sulfate are produced using graphene as an additive.

The aim of these additive mortars is to improve various properties, such as workability, increased hardness, and flexural and compression resistance, to achieve an innovative material with more-suitable characteristics for applications in industry, in general, and, specifically, in construction as a material providing continuous interior cladding in buildings for industrial and residential use, as a prefabricated material, or as a substitute for ceramic brick because significantly less CO_2 is emitted in its production.

Examining our current knowledge on topics related to plasters, we found many scientific studies in which different materials were incorporated into plasters to modify and improve some of their physical or mechanical properties. Some of the most outstanding are:

- Plaster with cellulose fiber. The intergypsum fiber-gypsum panel [1] is a homogeneous mixture of gypsum plaster and cellulose fiber from recycled paper.
- Applications of cork in the field of construction, with the study of new composite materials based on cork waste and the conglomerates used most frequently in the construction of buildings such as concrete, mortar, and gypsum/plaster [2].
- Combinations of expanded polyethylene (EPS) waste conglomerates with gypsum or plaster for use in construction [3].
- Incorporation of mussel shells in plaster and mortar. The properties of the mussel shell make it a convenient material to be incorporated as an aggregate. Mussel shells, as aggregates, are a feasible option for the valuation of the material, which, in addition to eliminating the dumping of this material, helps reduce the consumption of a natural resource that is becoming increasingly scarce (Figueroa and Altagracia, 2011) [4].

This manuscript presents the results of the bending resistance of plaster mortars through the use of an additive such as graphene.

2. Materials and Methods

2.1. Materials

The materials used to conduct the test, as well as the equipment and instruments used, are described in the sections below.

2.1.1. Water

The mortar for this test was obtained by mixing brown plaster and graphene with water obtained directly from the drinking water supply system. Water was used in the correct amount for the purpose of the tests being undertaken. Water, in the correct amount, facilitates the setting and distribution of the mortar within the molds when preparing test specimens.

2.1.2. Plaster

Brown plaster (also called black plaster) was used for the tests. Its nominal properties are defined according to UNE-EN 13279-1 [5], and we used Gypsum B1 construction, as follows: Thick gypsum was formed of semihydrate ($\text{SO}_4\text{Ca} \cdot 1/2\text{H}_2\text{O}$) with more than 50% hemihydrate with the possible incorporation of setting-regulating additives. The minimum bending and compression resistances were 1 and 2 N/mm², according to European regulations.

Brown plaster is used as a binder material for partitions, in interior coatings, and as secondary construction mortar. This plaster is primarily used for economic reasons as it is a less expensive plaster compared with others of higher purity that provide better results but increase the final cost, which is important in civil and industrial applications.

The plaster that was used has the following technical specifications, as shown in Table 1:

Table 1. Technical specifications of the plaster used (Source: PLACO S.A.) [6].

Specifications of the Plaster Used	
Material	Plaster
Commercial Name	Iberplast
Classification (UNE-EN-13.279-1:2009)	Building plasters and gypsum-based conglomerates for the building industry
Type	B1 (Building plaster) in accordance with the European denomination.
Reaction to fire	Euroclass A1 (does not contribute to fire) has less than 1% in weight or volumen of organic material, according to Directive 89/106/European Economic Community, CEE relative to building materials

Table 1. Cont.

Specifications of the Plaster Used	
Purity Index (%)	>75
Granulometry (mm)	0–2
Water/gypsum content (L/kg)	1, 0–1, 2
Shore hardness (Shore C units)	≥45 (test mean value according to Annex K of the RP 35.01 (Standard for B1 construction plasters))
Compression resistance	>2 N/mm ²
Bending resistance	>2 N/mm ²

2.1.3. Graphene

At present, graphene is marketed under the following types [7]: (1) Graphene in sheets [8] is high-quality graphene. The production of graphene in sheets is expensive, and it is used in fields such as computers, electronics, and aeronautics where a very strong material is required. (2) Graphene powder [9] is of lower quality than graphene in sheets. Its production volume is higher and lower cost.

The technical specifications of graphene are shown in Table 2.

Table 2. Specifications of powder graphene (Source: LucasLed Smart Saving) [10].

Material	Graphene
Composition	Solid powder
Colour	Black
Smell	Odorless
Solid particles	98%
Mean size of particles	10 microns
Density	2.2 g/cm ³
Mechanical properties	1.060 GPa
Chemical composition	C (%): 77–87%, H (%): 0–1%, N (%): 0–1% S(%): 0%, O (%): 13–22%

The tests on gypsum specimens were conducted using a universal testing machine (Lucasled, Madrid, Spain) from the construction laboratory of the Miguel Hernández University to obtain the bending strength, modulus of elasticity, and density of the specimens.

The items used in the construction laboratory of the Miguel Hernández University included: plastic buckets for weighing the materials and mixing the plaster; an electronic scale (Flintec, Barcelona, Spain): with a tolerance of 2×10^{-6} of the maximum weight, equivalent to a precision of 0.01 g; steel molds for the gypsum specimens (160 mm × 40 mm × 40 mm) as per the relevant standard; and auxiliary laboratory material including pipette, spatula, grids for drying, etc.

2.2. Methods

As few studies have been conducted involving the addition of graphene to plaster mortar, the following laboratory methodology was used, which was developed based on the UNE-EN 13279-2 Standard [11]. A total of 27 samples in three batches of nine specimens each were prepared (40 mm × 40 mm × 160 mm) to obtain results to use as a reference when comparing the results obtained with the graphene-additive gypsum mortars. Subsequently, a total of 27 samples in three batches of nine specimens each, with the same dimensions, were produced to obtain results of the gypsum mortar with the graphene additive. For the sake of brevity, only the results corresponding to the mean values of each of the batches are presented.

The experimental methodology for the preparation of the test specimens is described below.

Determination of the Proposed Compound

We selected the minimum water/gypsum (W/G) ratio to allow for the mixing of the product.

We fabricated the gypsum at different doses both with and without additives.

2.3. Characterization of the Prepared Composite Materials

For the determination of the physical and mechanical properties, a series of 40 mm × 40 mm × 160 mm specimens was prepared based on the UNE-EN 13279-2 Standard [11]. Nine specimens per batch were produced in the laboratory with a controlled laboratory temperature of 23 ± 2 °C and a relative humidity (RH) of $50\% \pm 5\%$ over 7 days.

2.3.1. Mixing and Dosing of the Reference Plaster Mortar

These graphene percentages were selected for economic reasons, since graphene is currently an expensive material, so it must be used in small quantities to control the price of the final gypsum product. As fabricated, it can be used in the coverings of buildings, gauze for traumatology, etc. For the preparation of these tests, reference samples of gypsum mortar were fabricated to serve as a basis for referencing the different samples of the graphene-additive gypsum samples produced with different proportions of constituents, as indicated in Table 3.

Table 3. Reference data on gypsum mortar samples.

Reference Plaster Mortar					
Sample Plaster Mortar (Reference)	Weight of the Sample (g) 3000	Batch No. 1	Plaster (%) 65	Water (%) 35	P/W Ratio 1.86

To prepare the samples, a base sample of gypsum mortar was used for each mixing or batch to serve as a reference, and we prepared three batches with nine specimens each, as shown in Table 4.

Table 4. Dosages and water/gypsum ratios of the reference sample.

No. of Batches	Weight of Plaster (g)/Batch	Weight of Water (g)/Batch	Weight of Mix (g)/Batch	% Plaster	% Water	No. of Specimens/Batch
3	1950	1050	3000	65	35	9

2.3.2. Mixing and Dosages of Gypsum Mortar with Added Graphene

Three batches of nine specimens per batch were prepared to assess the impact of the addition of 0.05%, 0.08%, and 0.10% graphene on the mechanical properties of the mixture obtained with the nomenclature indicated in Table 5.

Table 5. Dosages, quantity, and nomenclature of specimens with added graphene.

No. of Batches	Identification of Batch	Additive	Additive %	Additive Weight (g)	No. of Specimens/Batch
3	Batch G1	Graphene in powder	0.05	1.5	9
	Batch G2	Graphene in powder	0.08	2.4	9
	Batch G3	Graphene in powder	0.10	3.0	9

For each formulation, a batch of nine standardized 40 mm × 40 mm × 160 mm prismatic specimens was produced. The test was performed according to UNE-EN 13279-2 [11] using a universal testing machine that was fitted with a 3-point bending device to obtain the bending resistance and modulus of elasticity.

The device consists of a fixed lower part with two rigid bars 100 mm apart on which the specimen to be tested is placed. In the upper part, an arm with free vertical movement

ending in a tilting circular piece with a rigid bar transmits the load to the specimen. In the lower part, two metal pivots help to align the specimen and prevent it from becoming displaced during the test.

Data are collected by software that measures the applied force and the deflection every second during the test, as well as the final strength. The specimens are subject to bending stresses by applying a load of 50 ± 10 N/s until the specimen breaks into two pieces.

The analytical procedure for the determination of the characteristic values that are needed to classify and identify the mechanical properties is described below.

According to the above-mentioned Standard, the bending resistance is calculated once the ultimate breaking load has been recorded using:

$$R_f = \frac{(P \times l)/4}{(b \times h^2)/6} = \frac{3}{2} \times \frac{P \times l}{b \times h^2} = \frac{3}{2} \times \frac{P \times 100}{40 \times 40^2} \quad (1)$$

where R_f is bending strength (N/mm²) and P is the measured breaking load of the specimen (N).

This is simply the general basic mechanics formula used to calculate the bending strength in N/mm² but using the specific dimensions of our specimens. Hence:

$$R_f = \frac{M_p}{W_s} \quad (2)$$

where:

$$M_p = \frac{P \times l}{4} \quad (3)$$

where M_p is the moment generated by P at the centerline of the specimen, in N·mm.

$$W_s = \frac{b \times h^2}{6} \quad (4)$$

where W_s is the section modulus of the specimen, in mm³; l is the distance between test supports (span) = 100 mm; b is the width of the specimen = 40 mm; h is the depth of the specimen = 40 mm.

The results of the bending resistance in N/mm² can be refined using the testing machine by accurately measuring the exact dimensions of each of the specimens to calculate their exact section modulus W_s in mm³, hence avoiding deviations in the results due to dimensional errors during the fabrication of the specimens.

Flexion resistance according to the UNE-EN 13279-2: 2014 Standard [11], simplifying Equation (1), and leaving the equation as a function of a single load variable, we have:

$$R_f = 0.00234 \times P \quad (5)$$

Figure 1 shows the bending test of one of the specimens of a batch.

Figure 1. Flexible tested test tube, (a) is test tube to be tested, (b) is bending specimen rupture.

3. Results

The statistical analysis of the data obtained in the laboratory tests are structured here into two parts: the results of the laboratory tests, and the descriptive statistics of the data obtained.

To compare different mean values on a two-by-two basis (as there were more than two series per group), Tukey's multiple comparison test was used with a 95% significance. To identify similarities between the different results obtained, the Tukey's and Duncan's analyses were applied, from which different conclusions may be drawn depending on their adaptation to the factors studied.

We used computer software (2016) Statistical Package for the Social Sciences SPSS to calculate the statistics [12].

The results of the flexural strength test for the reference gypsum mortar specimens (G/W ratio = 1.86) are detailed in Table 6.

Table 6. Results of the flexural strength test for the reference gypsum mortar (G/W ratio = 1.86).

Flexural Strength (N/mm ²)				
Batch No. Strength (N/mm ²)	1 4.37	2 4.14	3 4.61	Mean value 4.37
	Mean Value			4.37
	Standard Deviation +/−			0.2350
	Coefficient of Variation			0.0538

The results of the flexural strength test for the reference gypsum mortar specimens for the batches with graphene proportions of 0.05%, 0.08%, and 0.10% are shown in Table 7.

Table 7. Test results for the flexural strength of gypsum mortar with add graphene powder.

Batche	Flexural Strength (N/mm ²)			
	Plaster without Graphene	Plaster and Graphene (0.05%)	Plaster and Graphene (0.08%)	Plaster and Graphene (0.10%)
1	4.37	4.98	4.71	5.22
2	4.14	4.34	4.51	5.52
3	4.61	4.76	5.10	5.32
Mean value	4.37	4.69	4.77	5.35
Standard deviation +/−	0.1305	0.3252	0.3001	0.1528
Coefficient of variation	0.0299	0.0693	0.0629	0.0286
Maximum	4.61	4.9800	5.1000	5.5200
Minimum	4.14	4.3400	4.5100	5.2200
Error	−	0.2564	0.0567	0.2539
Covariance	−	0.0486	0.0695	−0.0232

Figure 2 shows the results obtained from the bending test for the mixture of gypsum mortar with graphene added in the proportions of 0.05%, 0.08%, and 0.10%.

The evolution of the results was observed through their third-degree polynomial curve, as well as their regression, with results above 0.85. This means that the results obtained were representative.

When comparing the results obtained in the flexural strength test of graphene-additive gypsum mortar with the results obtained in other similar tests with different additives, we observed a considerable increase in flexural strength when graphene was added.

The comparison of our bending strength results with those of Bernal [13] on gypsum mortars with an organic-type additive with a retardant effect on the setting process and vermiculite are shown in Table 8.

Table 9 compares the above outcome with the results of the flexural strength tests reported in [14] for gypsum mortars with a polymer fiber additive.

Table 8. Comparison of bending strength test results of gypsum mortar with added graphene powder with respect to the research results obtained by Bernal [13].

Bernal [13] Flexural Strength (N/mm ²)			Plaster + Graphene (0.05%)		Plaster + Graphene (0.08%)		Plaster + Graphene (0.10%)	
Batch	Mean Value (N/mm ²)	Mean Value (kg/cm ²)	Mean Value (N/mm ²)	% Increased Flexural Strength	Mean Value (N/mm ²)	% Increased Flexural Strength	Mean Value (N/mm ²)	% Increased Flexural Strength
Gypsum Mortar + Adifoc YM3% + Vermiculite 10%	2.840	28.950	4.69	65.14	4.77	67.96	5.35	88.38
Gypsum Mortar + Adifoc YM3% + Vermiculite 20%	2.520	25.690	4.69	86.11	4.77	89.29	5.35	112.30
Gypsum Mortar + Adifoc YM3% + Vermiculite 30%	1.980	20.180	4.69	136.87	4.77	140.91	5.35	170.20
Gypsum Mortar + Adifoc YM3% + Vermiculite 50%	1.560	15.900	4.69	200.64	4.77	205.77	5.35	242.95
Gypsum Mortar + Adifoc YM3% + Vermiculite 70%	0.360	3.670	4.69	1202.78	4.77	1225.00	5.35	1386.11

Table 9. Comparison of bending strength test results of gypsum mortar with added graphene powder with those in [14].

Garcia Santos [14]			Plaster + Graphene (0.05%)		Plaster + Graphene (0.08%)		Plaster + Graphene (0.10%)	
Batch	Mean Value (N/mm ²)	Mean Value (kg/cm ²)	Mean Value (N/mm ²)	% Increased Flexural strength	Mean Value (N/mm ²)	% Increased Flexural strength	Mean Value (N/mm ²)	% Increased Flexural Strength
Plaster + Acrylic Fiber (Dolanit) 0.3%	3.000	30.580	4.69	56.34	4.77	59.01	5.35	78.34
Plaster + Acrylic Fiber (Nurel) 0.3%	3.032	30.910	4.69	54.67	4.77	57.31	5.35	76.44
Plaster + Polypropylene fiber (Propilan) 0.3%	2.779	28.330	4.69	68.76	4.77	71.63	5.35	92.50
Plaster + Polypropylene in fibers (Halesa) 0.3%	2.975	30.330	4.69	57.63	4.77	60.32	5.35	79.81
Plaster + Monofilament Polypropylene fiber (Concre Fib Halesa) 0.3%	3.130	31.910	4.69	49.82	4.77	52.38	5.35	70.91
Plaster + Polycondensed Melamine and Formaldehyde (MELMENT F.10) 1%	4.700	47.910	4.69	−0.21	4.77	1.49	5.35	13.83
Plaster + Polyvinyl Acetate Dispersion (Mowilith Pulver DS) 1%	4.120	42.000	4.69	13.83	4.77	15.77	5.35	29.85
Plaster + Copolymer Dispersion with Acrylic Acid Esters (Mowiton M-300) 1%	5.224	53.250	4.69	−10.22	4.77	−8.69	5.35	2.42

Figure 2. Flexural test results of gypsum mortar with graphene added in different proportions.

As shown in Table 10, better results were obtained by adding graphene to gypsum mortar than by adding organic- and vermiculite-type additives to gypsum mortar.

As per Table 9, the flexural strength of graphene-added mortar is higher than mortars with the addition of polyester, polypropylene, melanin, and copolymer dispersions.

Table 10 compares the results of the flexural strength to those obtained by del Río Merino [15] for plasters and plaster mortar with the addition of foaming agents, cork, glass fibers, and vermiculite.

Table 10 shows that the flexural strength of the mortar with graphene additive is higher than those of mortars with added cork and lightened plasters.

Table 10. Comparison of flexural strength test results of gypsum mortar with graphene powder with those reported by del Río Merino [15].

Comparison of Results of Flexion Tests								
Del Rio Merino [15] Flexural Strength (N/mm ²)			Plaster + Graphene (0.05%)		Plaster + Graphene (0.08%)		Plaster + Graphene (0.10%)	
Batch	Mean Value (N/mm ²)	Mean Value (kg/cm ²)	Mean Value (N/mm ²)	% Increased Flexural Strength	Mean Value (N/mm ²)	% Increased Flexural Strength	Mean Value (N/mm ²)	% Increased Flexural Strength
Plaster fabricated using carbonated water	3.120	31.800	4.69	50.34	4.77	52.91	5.35	71.50
Plaster lightened by the addition of SKW2 SERIES foaming agent								
8E	3.237	33.000	4.69	44.87	4.77	47.35	5.35	65.26
8ESKW0.5	4.218	43.000	4.69	11.18	4.77	13.08	5.35	26.83
8ESKW1	3.139	32.000	4.69	49.40	4.77	51.95	5.35	70.43
8ESKW2	2.943	30.000	4.69	59.36	4.77	62.08	5.35	81.79
8ESKW3	2.649	27.000	4.69	77.07	4.77	80.09	5.35	101.99
Plaster lightened with expanded clay SERIES								
8EAr20R	2.894	29.500	4.69	62.06	4.77	64.83	5.35	84.87
8EAr10F10A	3.139	32.000	4.69	49.40	4.77	51.95	5.35	70.43
8EAr20A	2.649	27.000	4.69	77.07	4.77	80.09	5.35	101.99
8EAr40A	2.771	28.250	4.69	69.23	4.77	72.12	5.35	93.05
8EAr50A	2.877	29.330	4.69	63.00	4.77	65.78	5.35	85.94
8EAr60A	2.223	22.660	4.69	110.98	4.77	114.58	5.35	140.67
Plaster lightened with perlite SERIES								
8EP20B10	2.485	25.330	4.69	88.74	4.77	91.96	5.35	115.30
8EP10B16	2.191	22.330	4.69	114.10	4.77	117.75	5.35	144.23
8EP20B12	1.994	20.330	4.69	135.16	4.77	139.17	5.35	168.25
Plaster lightened with vermiculite SERIES								
8EV20EI	2.403	24.500	4.69	95.14	4.77	98.46	5.35	122.60
Plaster lightened with expanded polystyrene SERIES								
8EP03	0.751	7.660	4.69	524.13	4.77	534.78	5.35	611.96
8EPO3V	0.981	10.000	4.69	378.08	4.77	386.24	5.35	445.36
8EP03F	1.040	10.600	4.69	351.02	4.77	358.72	5.35	414.49

4. Discussion

Based on the results obtained, we found that adding graphene powder in any proportion to the reference gypsum mortar increases its flexural strength; the best result was obtained for a proportion of 0.10%, which had with a maximum mean flexural strength value of 5.35 N/mm², which means that its flexural strength was increased by 0.98 N/mm², i.e., by 22.43%, with respect to the reference gypsum mortar.

This increase is shown in Figure 3.

Figure 3. Bar graph of flexural strength test results of gypsum mortar with graphene added in the proportions of 0.05%, 0.08%, and 0.10%.

The results obtained when adding graphene to gypsum mortar are improved in comparison with other additives tested as shown in the values in Tables 8–10.

5. Conclusions

The main, general conclusions drawn from these tests are that adding graphene powder to gypsum mortar increases the mechanical properties of the gypsum mortar, leading to an increase in its bending strength. An increase in the bending strength was also obtained in comparison with plaster without any additive when graphene powder was used, with values of up to 5.35 N/mm², whereas mean values of 4.37 N/mm² were obtained in plaster without any additive, which is an increase of 22.42% in the bending strength of the gypsum mortar when 0.10% of graphene was added to the mix.

After comparing the results of the bending tests conducted with the specimens with graphene additive with those with the gypsum mortar without additives, i.e., those used as reference values, we found that when graphene powder is added to the gypsum mortar, the flexural strength increases irrespective of the proportions of graphene added to the reference gypsum plaster.

We specifically showed that the results obtained are improved compared with other tests performed with other additives used.

These results are complementary to future Shore C hardness tests; the results of the compressive strength tests of the gypsum and graphene mixture that were previously published [16] in September 2020.

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