IBRACON Structures and Materials Journal
Revista IBRACON de Estruturas e Materiais
ISSN 1983-4195 ismj.org

ORIGINAL ARTICLE

Performance evaluation of self-leveling mortars using phosphogypsum and white ceramic waste

Avaliação do desempenho de argamassas autonivelantes utilizando resíduos de fosfogesso e cerâmica branca

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Received 26 August 2022
Revised 16 February 2023
Accepted 07 March 2023
Corrected 27 March 2024

Abstract: This study investigates the properties of self-leveling subfloor mortar mixtures with natural and calcined phosphogypsum waste in the form of calcium sulfate called anhydrite and white ceramic, by replacing 50% of the mass of Portland cement CP II Z-32. The effects of using waste were evaluated in the fresh (initial fluidity, flow retention, regeneration time, setting time, and heat of hydration) and hardened (compressive and flexural tensile strength, density, water absorption, void ratio, linear shrinkage, and tensile adhesion strength) state. The optimized mixture at a 1:1.5 cement/sand ratio (by mass) with 50% white ceramic had a 32.66 ± 4.26 MPa compressive strength at 28 days, low shrinkage and heat of hydration, showing the best performance among the wastes studied. In the calcium sulfate mortars, the mixtures with anhydrite showed better results for compressive and flexural tensile strength and tensile adhesion strength, with the waste calcination being beneficial.

Keywords: self-leveling mortar, subfloor, calcined phosphogypsum, white ceramics, waste recovery.

Resumo: Este estudo investiga as propriedades de misturas de argamassas autonivelantes para contrapiso com os resíduos de fosfogesso natural e calcinado, na forma de sulfato de cálcio denominada anidrita, e cerâmica branca, através da substituição de 50% da massa de cimento Portland CP II Z-32. Os efeitos da utilização dos resíduos foram avaliados nos estados fresco (fluidez inicial, retenção de fluxo, tempo de regeneração, tempo de pega e calor de hidratação) e endurecido (resistência à compressão e tração na flexão, massa específica, absorção de água, índice de vazios, retração linear e resistência de aderência à tração). A mistura otimizada na proporção cimento/areia de 1:1,5 (em massa) com 50% de cerâmica branca obteve resistência à compressão de 32,66 ± 4,26 MPa aos 28 dias, baixa retração e calor de hidratação, apresentando melhor desempenho entre os resíduos estudados. Nas argamassas a base de sulfato de cálcio, as misturas com anidrita apresentaram melhores resultados para resistência à compressão e tração na flexão e resistência de aderência à tração, sendo benéfica a calcinação do resíduo.

Palavras-chave: argamassa autonivelante, contrapiso, fosfogesso calcinado, cerâmica branca, valorização de resíduos.

How to cite: E. T. Gruber, C. Effting, A. Schackow, and G. A. Mendes. "Performance evaluation of self-leveling mortars using phosphogypsum and white ceramic waste" *Rev. IBRACON Estrut. Mater.*, vol. 17, no. 1, e17103, 2024, https://doi.org/10.1590/S1983-41952024000100003

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Financial support: None.
Conflict of interest: Nothing to declare.
Data Availability: Data-sharing is not applicable to this article as all data were already showed in this study.
This document has an erratum: https://doi.org/10.1590/S1983-41952024000100011

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1 INTRODUCTION

In the context of technological development, need to modernize civil construction, and optimization of the building execution processes, the use of self-leveling mortars for subfloors stands out. NBR 15575-3 [1] performance standard defines subfloor as the layer responsible for regularizing the substrate, providing a homogeneous support surface, which can serve as an embedding or sloping layer.

Cement-based self-leveling mortars are materials obtained by mixing cementitious materials, aggregates, mineral additions, and chemical additives. The percentage of Portland cement present in these mortars varies between 25 and 40% of the total mass; fine quartz sand varies between 40 and 60% of the total mass; and additives represent about 10 to 15% of the total mass [2].

However, like other cementitious materials, they require large amounts of cement, as they have an inverse relationship between workability and mechanical strength [3], making it difficult to obtain fluid mixtures with absence of exudation and/or segregation [4]. Aiming to modify the rheological properties of mixtures, chemical additives are used, raising the cost of these materials [5].

Associated with this context, one of the sustainability challenges in the civil construction industry is to reduce the use of cement due to the high amount of carbon dioxide (CO_2) released into the atmosphere during the manufacturing process. For this, alternative materials that can reduce this environmental impact are sought [4], also aiming at a better performance and cost reduction [5], [6].

As the increasing waste generation has been leading society to look for solutions that consider waste recycling, reusing and treatment, the use of replacements for cement is an alternative for conservation and rationalization of mineral and energy resources [7]. Industrial by-products have been receiving increasing interest due to their high reactivity, low cost, and greater availability in some regions [8].

Among the by-products that come from the industries are phosphogypsum and white ceramic. Phosphogypsum is a by-product of the phosphate fertilizer industry [9]. The accumulation of waste from these industries occupies considerable portions of land and results in serious environmental problems [5].

In Brazil, 5.6 million tons of phosphogypsum are generated per year, generated during the process of obtaining phosphoric acid, where approximately 5 tons of waste are produced for each ton of phosphoric acid produced [10]. Annual world production is approximately 150 million tons, which causes severe environmental impacts [11].

With the continuous population growth and consequent increase in food and fertilizer production, a considerable increase in phosphogypsum deposits is noted [11]. To minimize the environmental impacts of the incorrect deposition of this material, ways to reuse it are sought [12]. Different destinations were studied in recent years, such as in agriculture, civil construction, environment, and energy [13].

In addition to phosphogypsum, white ceramic waste also comes from the industry. Brazil is responsible for the production of more than 200 million ceramic tiles per year, which is equivalent to 2% of the world production. On average, these companies have a 20% loss rate of raw materials that are discarded [14]. The management of this industrial waste is difficult, as it is considered a non-biodegradable material, with a biodegradation period of up to four thousand years [15].

White ceramic waste can contribute to the performance of self-leveling mortars, as they are predominantly composed of silicon and aluminum, giving improvements in the pozzolanic activity index [4], [16] resulting in better mechanical properties, reduced heat of hydration, and shorter initial and final setting times [17].

The use of by-products and wastes in self-leveling mortars has been evaluated by other authors. Barluenga and Hernández-Olivares [18] used waste from slate mining; Wang and Jia [19] studied self-leveling mortars made with phosphogypsum, sulfoaluminate cement, and granulated blast furnace slag; Yang et al. [5] evaluated self-leveling mixtures composed of phosphogypsum, Portland cement, sulfoaluminate cement, and different granulometries of fine aggregates; Pereira and Camarini [4] worked with porcelain and red ceramic waste from demolitions, replacing Portland cement in mass by 15%, 25% and 50%; and Mendes [20] studied self-leveling mortars with additions of 40% and 50% of marble and granite waste.

In order to continue the studies on the application of wastes and by-products in self-leveling mortars, this study evaluated the use of natural phosphogypsum and calcined phosphogypsum at a 450°C temperature for 4 hours, resulting in the form of calcium sulfate called anhydrite, and white ceramic in the composition of self-leveling mortars dosages for subfloors. The effects of replacing 50% of the mass of Portland cement CP II Z-32 on physical and mechanical properties were investigated.

2 MATERIALS AND EXPERIMENTAL PROGRAM

2.1 Materials

Mortar mixtures were prepared using different proportions of Portland cement CP II Z-32, natural sand, water, superplasticizer and viscosity modifier additives, and phosphogypsum and white ceramic waste.

The cement used is Portland CP II Z-32, as it has a wide application in mortars and concretes and is easily acquired at the site of the study. This type of cement has a composition of 0-10% limestone filler, 76-94% clinker and gypsum, and between 6-14% pozzolan [7].

The sand used in the mixtures has a 1.24 fineness modulus and a 1.18 mm maximum grain diameter, being classified as fine small aggregate by NBR 17054 [21]. According to NBR 16916, the specific mass is 2.60 g/cm^3 and the apparent unit mass is 1.43 g/cm^3 [22].

The waste called natural phosphogypsum (FN) was dried in an oven for 24 hours at a 100 ± 2 °C temperature and sieved until the amount of material retained on the 45 µm mesh sieve was less than 20% of the total sample mass. The grains have an irregular and angular shape, sharp edges, and corners, as illustrated in the microstructural analysis in Figure 1. The granulometry was performed by a laser equipment and showed a 60 µm maximum grain diameter, 50% with dimension less than 5.97 µm, and 90% of the particles with dimension less than 30.80 µm, as illustrated in Figure 2. The specific mass of the waste is 2.69 g/cm^3, as per NBR 16605 [23]. The chemical analysis assay by XRF and the comparison with results obtained by other authors are listed in Table 1. X-ray fluorescence (XRF) chemical analysis indicated that the phosphogypsum waste consists mainly of CaO (calcium oxide) and SO_3 (sulfur).

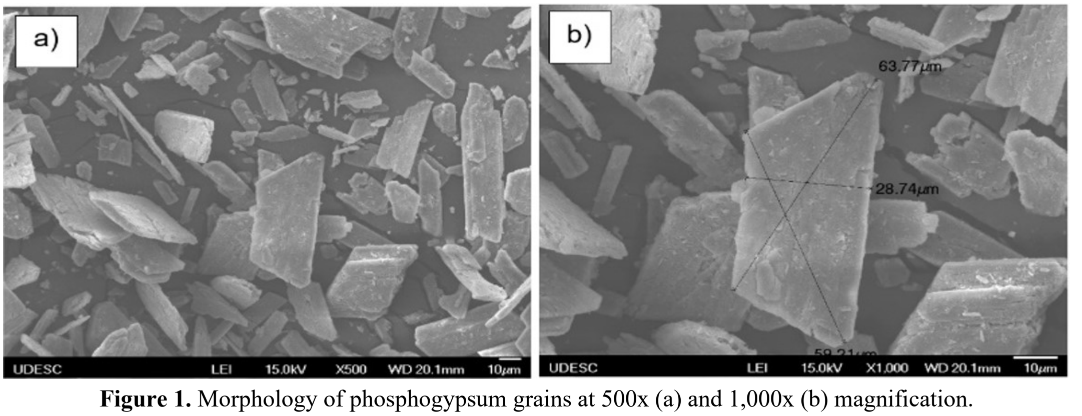


Figure 1. Morphology of phosphogypsum grains at 500x (a) and 1,000x (b) magnification.

10 %D(µm)	20 %D(µm)	30 %D(µm)	40 %D(µm)	50 %D(µm)
1,416	2,060	2,929	4,135	5,973

60 %D(µm)	70 %D(µm)	80 %D(µm)	90 %D(µm)
9,000	14,741	22,307	30,804

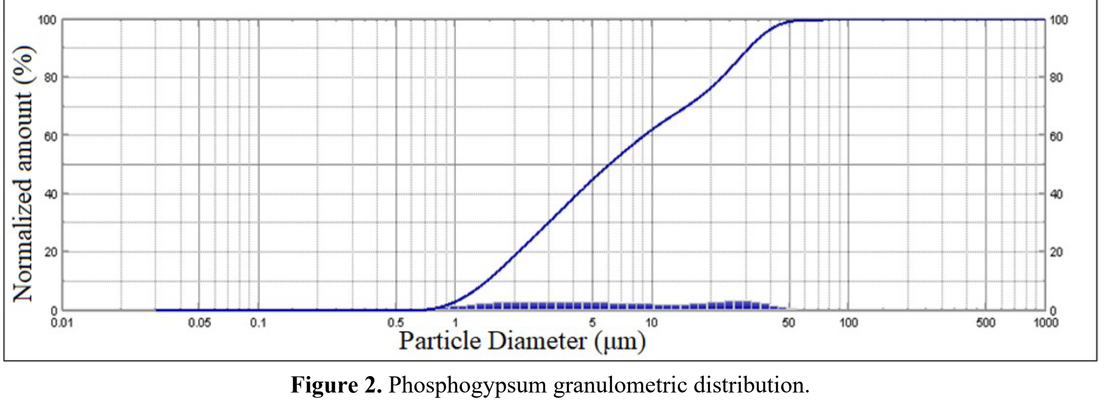


Figure 2. Phosphogypsum granulometric distribution.

In the assays of physical and mechanical properties, calcined phosphogypsum (called FC) at a 450°C temperature, for a 4-hour period, was also used, which results in the form of anhydrite calcium sulfate II ($CaSO_4$), known as phosphoanhydrite, in order to improve the properties of mortars, as observed by Schaefer et al. [27]. The heating rate adopted was 10°C/min. After heating, FC was cooled naturally until it reached room temperature."

The white ceramic was obtained from a tableware manufacturer located in Santa Catarina state. These are materials broken during the tableware manufacturing process, without the transparent and colorless glassy layer, obtained after the firing step. To use the residue in the mixtures, it was necessary to grind it until the amount of material retained on the 45 µm mesh sieve was less than 20% of the total sample mass. The material was oven dried for 24 hours at a 100 ± 2 °C temperature. The grains have a rounded shape, with larger and smaller particles distributed, indicated in the microstructural analysis in Figure 3. Particle sizing by laser equipment indicated a 36 µm maximum grain diameter, with 10% of the particles smaller than 1.17 µm, 50% smaller than 3.53 µm, and 90% smaller than 14.26 µm, as shown in Figure 4. The average grain size is about 41% smaller than phosphogypsum. The specific mass of the residue is 2.73 g/cm^3. The chemical analysis assay by XRF and comparison with results obtained by other authors are listed in Table 2. White ceramic presents in its chemical composition mainly SiO_2 (silicon dioxide) and Al_2O_3 (aluminum oxide).

Table 1. Chemical analysis by XRF of natural and calcined phosphogypsum and results obtained by other authors.

Material (%)	Natural phospho-gypsum	Natural phospho-gypsum	Natural phospho-gypsum	Natural phospho-gypsum	Natural phospho-gypsum	Phosphogypsum calcined at 450°C (Anhydrite II)	Phosphogypsum calcined at 450°C (Anhydrite II)
Author	**[24]**	**[25]**	**[26]**	**[27]**	**The author (2022)**	**[27]**	**The author (2022)**
CaO	35.62	31.51	32.28	38.56	56.21	53.53	71.75
SiO_2	2.39	0.5	1.08	0.82	1.67	0.92	1.05
Al_2O_3	0.03	0.11	0.07	0.41	0	0.10	0
Fe_2O_3	0.83	0.05	0.21	0.89	1.07	1.03	2.06
SO_3	47.10	46.9	43.29	24.59	39.76	34.10	22.18
MgO	0.62	1.59	0.05	0	0	0.00	0
TiO_2	0	0	0.04	0	1.29	0.00	1.92
Na_2O	0	0.03	0	3.99	0	3.85	0
P_2O_5	1.14	0.35	0.58	1.13	0	1.01	0.98
Other	12.27	18.96	22.4	29.61	0	5.46	0.05

The waste performance index regarding pozzolanic activity was determined in accordance with the standards NBR 12653 [33], and NBR 5752 [34], which establish as a minimum physical requirement for pozzolanic materials a performance index with Portland cement at 28 days of at least 90%, and as a chemical requirement, total silica, alumina, and iron oxide concentration greater than 70%, and SO_3 concentration lower than 4%.

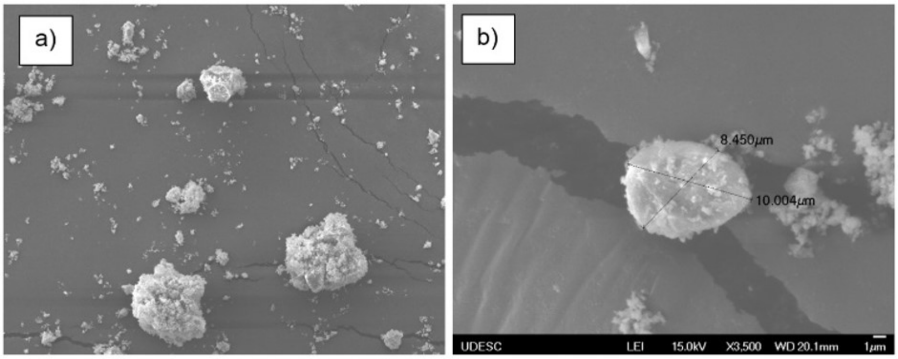


Figure 3. Morphology of white ceramic grains at 500x (a) and 3,500x (b) magnification.

10 %D(µm)	20 %D(µm)	30 %D(µm)	40 %D(µm)	50 %D(µm)
1.174	1.586	2.050	2.662	3.533

60 %D(µm)	70 %D(µm)	80 %D(µm)	90 %D(µm)
4.756	6.446	9.118	14.260

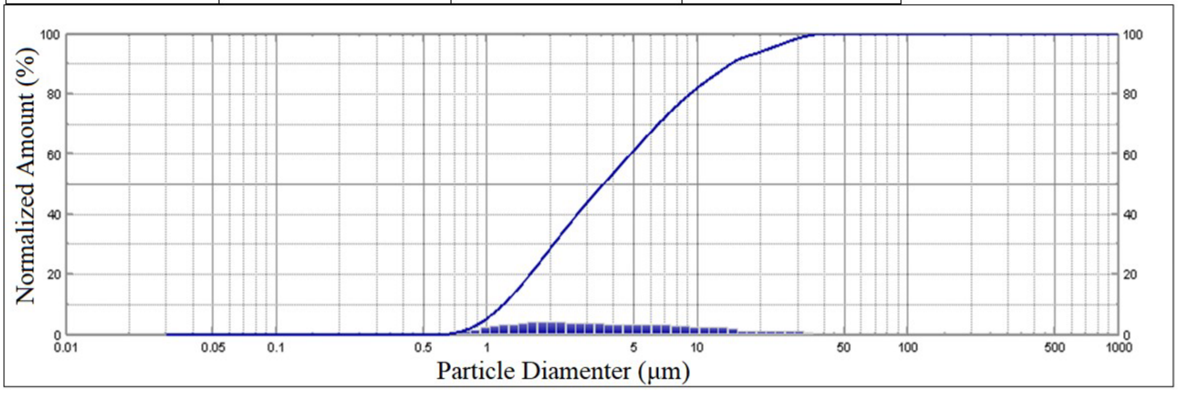


Figure 4. White ceramic granulometric distribution.

In the physical requirement, the performance index of the mortar produced with the replacement of cement paste by white ceramic waste was 118.68%, while the mortar produced with natural phosphogypsum obtained a 31.09% index. In the chemical requirement, natural and calcined phosphogypsum did not meet the minimum requirements to be classified as pozzolanic materials because they do not have the minimum concentrations of silica, alumina, and iron oxide, besides presenting higher amounts of SO_3. The white ceramic has a concentration of $SiO_2 + Al_2O_3 + Fe_2O_3 = 94.17\%$, with no SO_3 concentration in its chemical composition. Thus, only the white ceramic waste met the physical and chemical requirements and can be classified as a pozzolanic material.

Table 2. XRF chemical analysis of white ceramic and results obtained by other authors

Material (%)	White Ceramic	White Ceramic	White Ceramic	White Ceramic	White Ceramic	White Ceramic
Author	**[28]**	**[29]**	**[30]**	**[31]**	**[32]**	**The author (2022)**
CaO	1.42	7.00	2.41	0.20	0.76	2.47
SiO_2	66.62	68.9	67.91	65.00	70.90	62.63
Al_2O_3	15.58	19.8	22.01	21.30	21.10	29.83
Fe_2O_3	8.18	0.90	1.41	1.30	0.81	1.71
SO_3	0	0	0.07	0	0	0
MgO	1.79	0.30	0.29	0.30	0.24	0
TiO_2	0.83	0.70	0.45	0.20	0.33	0.65
Na_2O	1.18	0.30	1.91	2.50	1.47	0
K_2O	3.84	1.70	2.79	3.70	3.57	2.69
Other	0.56	0.40	0.75	5.50	0.82	0.02

The liquid viscosity modifier additive used has a density of 1.00 g/cm^3, transparent coloring, and the manufacturer's recommended dosage is between 0.1 to 1.5% of the cement mass. The liquid superplasticizer adopted is a synthetic superplasticizer based on polycarboxylate polymer technology, brown in color, with a 0.2 to 5% of cement mass dosage and a 1.12 g/cm^3 density.

2.2 Experimental methods

The self-leveling mortar mixtures were prepared in a mechanical mixer with a 5 dm^3 capacity and two speed options: low and high, as provided by NBR 7215 [35].

Since there is no Brazilian standard determining the flow properties of these mixtures, the C1708 standard [36] was used; it evaluates flowability through three tests: initial flow, flow retention, and regeneration time.

The initial flow and flow retention assays were performed with a cylinder with 30 mm in diameter and 50 mm in height, under a rectangular glass plate with 400 mm in dimension and a 6 mm thickness. The initial flow was determined immediately upon mixture preparation, as shown in Figure 5.

The mortar was inserted into the cylinder and 2 seconds later the ring was lifted to a height between 50 and 100 mm from the glass base for a 240 ± 10s time period. The initial scattering corresponds to the average of the scattering measured in the two directions. The C1708 [36] standard determines the spread to be a value between 125 and 150 mm.

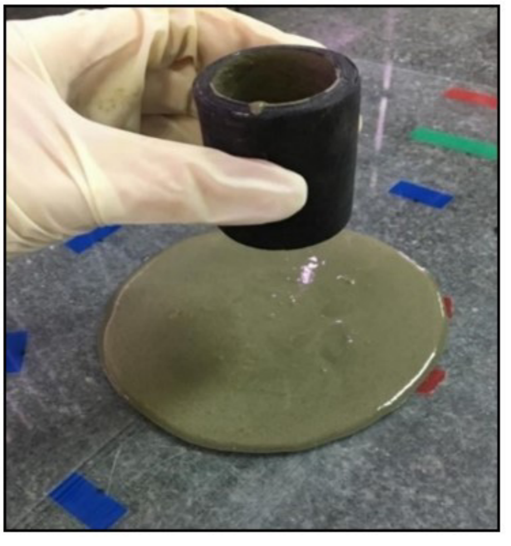


Figure 5. Determination of the spreading diameter.

The evaluation of flow retention is done by repeating the initial flow test after 20 and 30 minutes, counted from the addition of water to dry materials in the mixture, indicating how long these mortars maintain their self-leveling property.

The regeneration time determination procedure consists of making specific cuts in the mortar surface during regular intervals to determine for how long a self-leveling mortar has the ability to return to its original state after deformation, without showing unevenness or marks. The mortar was placed in a rectangular formwork with 210 x 210 mm minimum dimension and 9 mm minimum depth immediately upon the mixture was prepared until it reached a 6 +/- 1 mm thickness. After 10 minutes from the start of the mortar preparation, cuts were made to the full depth of the mortar using a metal bar, with a 45° slope. The first cut started at a 25mm distance from the formwork. Cuts spaced 25 mm apart, every 5 minutes, were made until the material no longer regenerated.

The determination of the initial and final setting time of the mortars was performed in accordance with the NBR 12128 [37] and C191 [38] standards, using the Vicat apparatus, shown in Figure 6; and the evaluation of the heat of hydration of the self-leveling mixtures was performed with the ImpacLog IP7018 brand calorimeter, by following the temperature *versus* time curves for 24 hours.

The compressive strength test was performed according to the NBR 7215 [35] and C109 [39] standards. To evaluate the compressive strength of the self-leveling mortars, 9 cylindrical test specimens with a 50 mm diameter and 100 mm height were molded; 3 specimens were broken on the first day, 3 at 7 days, and the remaining 3 at 28 days. The test specimens were cured at room temperature.

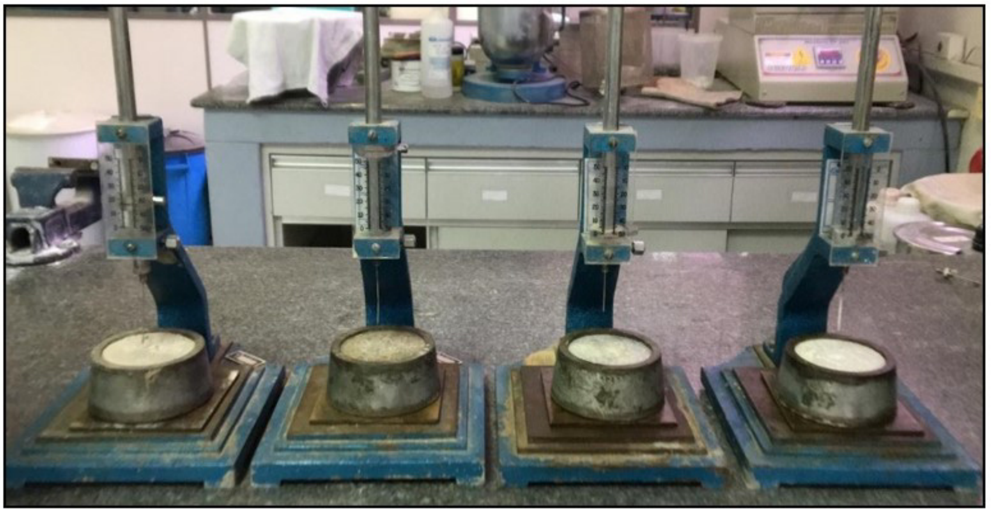


Figure 6. Setting time determination test using the Vicat apparatus.

The standards used to perform the flexural tensile strength test were: NBR 13279 [40] and C348 [41]. For each mixture, 6 prismatic test specimens measuring 40 x 40 x 160 mm were molded. Of these, the C1708 standard [36] indicates that 3 should be broken after 24 hours and the rest at 28 days. However, the hydraulic press available for the tests did not accurately record the flexural tensile strength results after 24 hours, and the first strength verification was adopted at 3 days of age. For the rupture of the specimens a hydraulic press EMIC PC200 was used, applying a 500 +/- 50 N/s load until the rupture of the test specimens.

The linear shrinkage monitoring was done for 28 days and aimed to analyze the possible variations in length due to expansion or shrinkage in mortars; it was performed as provided by the NBR 15261 [42] and C157 [43] standards. To perform the test, three test specimens were molded for each mortar mixture analyzed. Metal prismatic molds with 25 mm x 25 mm x 285 mm in dimension and 250 mm effective length between pins were used, as shown in Figure 7. To measure the variation in length of the test specimens, a dial indicator with a 0.001 mm resolution was used.

The test to determine the specific mass, void ratio, and water absorption in mortars in the hardened state was performed as provided by NBR 9778 [44]. To perform the test, two samples of each mortar mixture were molded according to NBR 7215 [35], with a 40 mm x 40 mm x 160 mm dimension.

Figure 7. Test specimens from the linear shrinkage test

The tensile adhesion strength test of the self-leveling mortars was performed as provided by NBR 13528 [45]. The tensile equipment used in this test was a Solotest manual dynamometer equipped with a reading device, which allows continuous application of centered and orthogonal load to the coating plan. The substrate used for the test was a weathered reinforced concrete slab built to simulate a real situation on the construction site. The test was performed on the 28-day old coating, and for each mixture 12 test specimens were taken with pull-off points spaced at least 50 mm apart from corners and edges.

The results obtained for compressive strength and flexural tensile were evaluated through statistical analysis of variance (ANOVA). The confidence level adopted was 5% ($\alpha = 0,05$), by this way, only the variables that presented p-value less than 0,05 were considered significant in the results.

3 RESULTS AND DISCUSSIONS

3.1 Mortar dosage

Self-leveling mortar mixtures were made with 50% of the cement mass replaced by natural and calcined phosphogypsum and white ceramic waste. The acceptance of the developed mixtures took into consideration the production of mortars with a 125 mm minimum and 150 mm maximum spreading diameter, and the absence of segregation and exudation. The proportion of the materials elaborated by Mendes [20] for the reference mortar was adopted and the mixtures with the waste were optimized by varying the water/cement ratio and superplasticizer additive. Table 3 presents a summary of the selected mortars and the material proportions. Table 4 presents the composition of the mortars.

The fluidity of mortars containing natural phosphogypsum is affected because the specific area of phosphogypsum is larger and its morphology is irregular, requiring more water to maintain the workability of the mixture. The rounded grain shape of white ceramic and the smooth interface contributed to the increased fluidity of these mixtures, compared to the grain morphology of natural phosphogypsum. It is also observed that the calcination of phosphogypsum waste is beneficial for the fluidity of mortars.

Table 3. Summary of material proportions of the mortars studied (in mass)

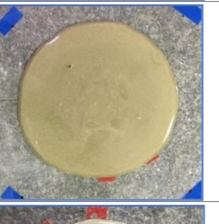
Mortar	Proportion of materials	Flow test images
AR	**1 : 1 : 0.33 : 0.0033 : 0.0067** (cement; sand; water; superplasticizer; viscosity modifying additive)	
FN	**1 : 1.5 : 0.5 : 0.65 : 0.0090 : 0.010** (cement; sand; waste; water; superplasticizer; viscosity modifying additive) (50% of the cement mass replaced by natural phosphogypsum)	
FC	**1 : 1.5 : 0.5 : 0.65 : 0.0050 : 0.010** (cement; sand; waste; water; superplasticizer; viscosity modifying additive) (50% of the cement mass replaced by calcined phosphogypsum)	
CB	**1 : 1.5 : 0.5 : 0.65 : 0.0050 : 0.010** (cement; sand; waste; water; superplasticizer; viscosity modifying additive) (50% of the cement mass replaced by ceramic waste)	

Table 4. Composition of the self-leveling mortars

Mixtures	C (kg/m^3)	FN (kg/m^3)	FC (kg/m^3)	CB (kg/m^3)	S (kg/m^3)	W (kg/m^3)	SP (kg/m^3)	VA (kg/m^3)	W/C
AR	948.18	-	-	-	948.18	312.89	3.12	6.35	0.33
FN	474.09	474.09	-	-	1422.27	616.31	8.53	9.48	0.65
FC	474.09	-	474.09	-	1422.27	616.31	4.74	9.48	0.65
CB	474.09	-	-	474.09	1422.27	616.31	4.74	9.48	0.65

Legend: C – Cement; FN – Natural Phosphogypsum; FC – Calcined Phosphogypsum; CB – Ceramic Waste; S – Sand; W – Water; SP – Superplasticizer; VA – viscosity modifying additive.

The water/cement ratio varied from the reference for the other mixes because the added wastes are very fine materials and absorbed water from the mix. It was necessary to add water to keep the self-leveling mortars.

When increasing the water/cement factor in the mixtures with residues to compensate the absorption of fine residues, it was noticed that there was segregation, so it was necessary to add sand. The mixtures were optimized so that they were suitable according to the images in Table 3.

3.2 Initial flow, flow retention and regeneration time

For initial flow, standard C1708 [36] recommends that the spreading diameter should be between 125 and 150 mm, but it does not set minimum and maximum limits for flow retention. However, the analysis of this property is of fundamental importance for self-leveling mortars, since this material must present adequate workability during the time required for application, which is often carried out by pumping. The results are shown in Table 5.

Table 5. Results of the flow tests

Evaluated mortar	Initial spreading diameter (mm)	Spreading diameter 20 min. (mm)	Spreading diameter 30 min. (mm)	Regeneration time (minutes)
AR	125	125	125	15-20
FN	135	125	125	10-15
FC	125	125	125	15-20
CB	125	125	125	15-20

The AR, FC, and CB mixtures showed no flow retention, maintaining the spreading diameter after 20 and 30 minutes after preparation, while the FN mortar, produced with natural phosphogypsum, showed a 10 mm (from 135 to 125 mm) decrease in the spreading diameter after 20 and 30 minutes, but maintaining the 125 mm minimum spreading.

In addition, the regeneration time of the AR, FC, and CB self-leveling mixtures was ranged between 15 and 20 minutes, while the FN mixture range was between 10 and 15 minutes, indicating that it is unable to return to its original state after undergoing some deformation after a shorter period of time. This result may be related to the initial setting time and heat of hydration tests, which showed that the FN mixture had the shortest initial setting time among all the mortars evaluated, and the highest heat release in the initial hours.

3.3 Setting time

The tests were performed simultaneously for the mixtures, maintaining room temperature and humidity. Table 6 presents the results obtained.

Table 6. Setting time of the evaluated mortars

Evaluated mortar	Initial setting time (h:min)	Final setting time (h:min)	Difference (h:min)
AR	08:40	09:15	00:35
FN	05:25	14:35	09:10
FC	08:20	08:50	00:30
CB	06:10	06:30	00:20

The shortest recorded initial setting time is from the FN mortar, which is 37.5% shorter than the AR initial setting time, followed by the CB mixture, which is 28.85% shorter than the AR's.

The AR and FC mortars presented close values for the initial and final setting times, as well as the difference between these values. Although the difference between the initial and final setting times resulted in analogous values also for the CB mixture, the initial setting time happened 2h 30min earlier compared to the AR.

When analyzing the final setting times, it can be seen that the FN mortar presented a considerably longer time than the others, resulting in a final setting time 57% longer than the AR's. The FC mixture, on the other hand, showed a shorter final setting time compared to the FN mixture. Schaefer [46] explains that this happens because the higher the calcination temperature, the greater the specific area of the phosphogypsum, resulting in faster hardening.

3.4 Heat of hydration

Temperature variation is one way to evaluate the effects of waste on the cement hydration reactions in the mixtures. The curves represent the heat release from the pastes during the hardening process, since the heat released is an exothermic reaction. Figure 8 shows the graph of the temperature variation as a function of time for the four mixtures over the 24-hour period.

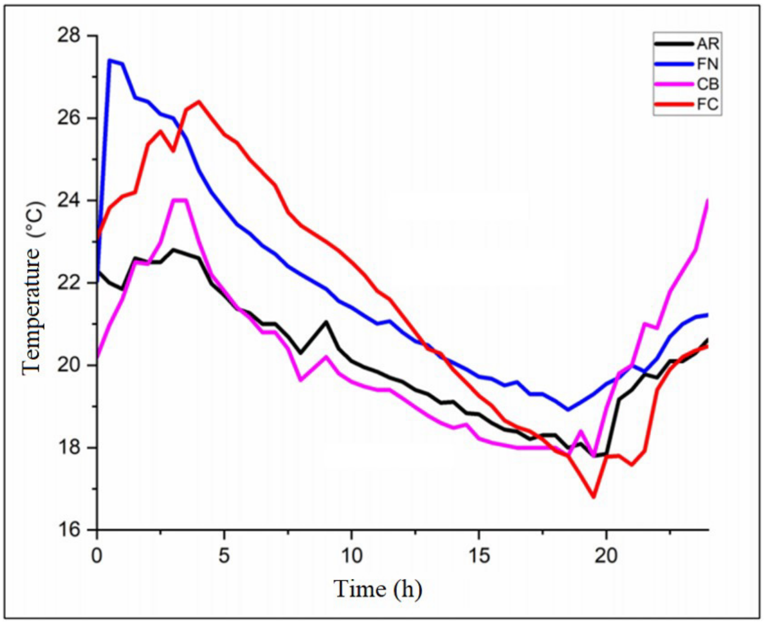


Figure 8. Heat of hydration generated by mortars

Among all the mortars evaluated, FN released the highest amount of energy. The maximum temperature reached by AR was 23.10°C, by CB it was 23.89°C, by the FN mixture, 27.65°C, and by FC, 26.30°C. Regarding the initial temperature of the mixtures, the AR showed a 0.8°C increase, while the CB increased 2.19°C, the FN, 5.61°C, and the FC, 3.76°C.

Furthermore, the graph shows that the mortar containing natural phosphogypsum is the first to reach the peak temperature, which is in accordance with the setting time test using the Vicat apparatus, where the shortest time for the onset of setting was verified.

For ceramics, similar results were found in the studies by Pereira and Camarini [4], who indicated that the 50% replacement of Portland cement with red ceramic did not significantly influence the cement hydration process, therefore, using the heat of hydration as an evaluation parameter can be a viable alternative.

The result of the heat of hydration curve of the FN mixture compared to FC's is consistent with what Schaefer [46] observed, who reports that the anhydrite II (FC), obtained with a calcination temperature of 450 °C, hydrates more slowly compared to other calcium sulfates, such as natural phosphogypsum, resulting in a rightward shift of the curve.

3.5 Compressive strength

High strength values are key technological parameters in self-leveling mortars [5]. To compare the results obtained for the reference mortar, C1708 standard [36] presents inter-laboratory test results. The study named ILS n. 349 was conducted with 5 replicates of commercial self-leveling mortars at 1, 7, and 28 days of age. In ILS n. 349 seven laboratories tested the material for flexural strength, compressive strength (over a 28 days period), and also recorded the initial flow, initial set time, and final set time. The average compressive strength recorded in these studies was 21.76 MPa at 1 day, 32.01 MPa at 7 days, and 39.15 MPa at 28 days. The AR used in the study showed a compressive strength 33% higher than the ILS at 01 day and approximately 37% higher at 07 days and 28 days. The values obtained are recorded in the graph in Figure 9.

The analysis of the results obtained allows us to conclude that the addition of waste influenced the results of compressive strength at all ages, as expected, due to the decrease in cement consumption. In addition, it was necessary to increase the amounts of water and superplasticizer additive to achieve the minimum spread. Silva [47] explains that increasing the water/cement ratio considerably reduces mortar strength because the space occupied by the water, which is consumed in the hydration process, becomes a weak point.

Among the wastes studied, the CB mixture was the one that obtained the best performance in this property, presenting 65.25% lower compressive strength in 1 day, 40.62% lower in 7 days, and 34.46% lower after 28 days, when compared to the RA.

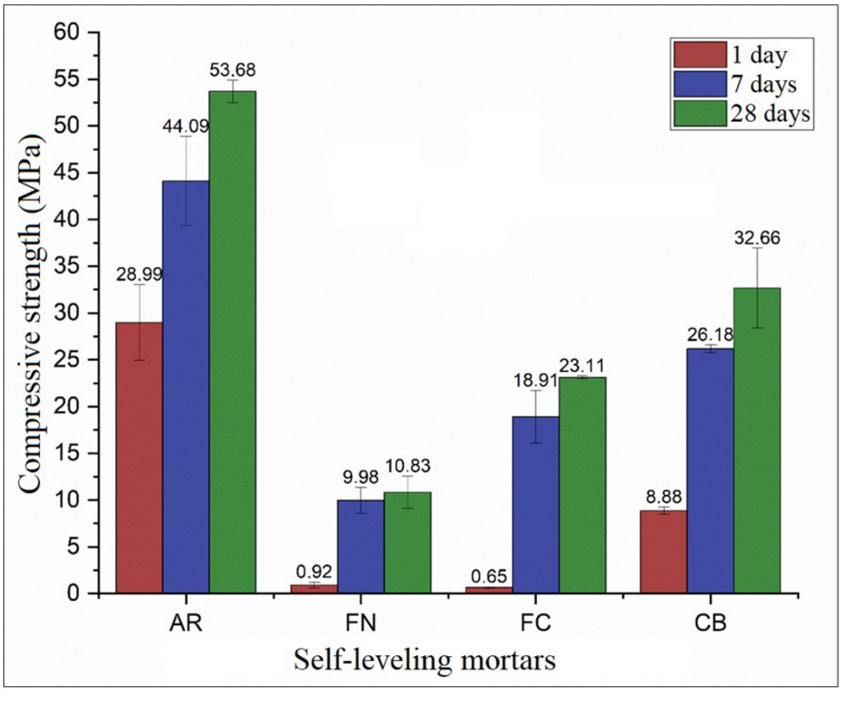


Figure 9. Compressive strength results for mortars at 1, 7 and 28 days.

It is also observed that the calcination of phosphogypsum (FC) had little influence on the compressive strength at 01 day of age, but showed superior results in the analyses with other ages. When compared to the mixture with untreated phosphogypsum (FN), the FC mortar showed approximately twice the compressive strength at 7 and 28 days.

These results indicate that calcination of phosphogypsum is beneficial, favoring better results at 7 and 28 days, and are in agreement with the lower values for void ratio and water absorption.

This can also be explained by the fact that the natural and calcined phosphogypsum waste has no pozzolanic activity, so the strength gains occur later, due to the filler effect. In addition, Gong et al. [24] explain that the use of calcined phosphogypsum is beneficial because the dissolution of the anhydrite produces fewer sulfate ions than natural phosphogypsum, improving mortar performance. However, as observed by Schaefer [46], anhydrite is a calcium sulfate that has a slow dissolution rate, which explains the achievement of better results only after 7 days.

Yang et al. [5] used the Chinese standard for self-leveling mortars in their study, establishing that the compressive strength at 01 day should not be less than 6 MPa. According to this parameter, mortars produced with natural and calcined phosphogypsum do not meet the minimum values required for early ages because calcium sulfate and the presence of residual acids in phosphogypsum delay the onset of hydration [5].

Silva et al. [48] report that in the draft standard prepared in 2019 addressing the execution procedure for self-leveling gypsum subfloor there is a requirement for a minimum compressive strength of 8 MPa. For this criterion, the FC and FN mortars would not meet the 1-day age, but would be above the limit at 7 days of age.

For strength at 28 days, Barluenga and Hernández-Olivares [18] recommend a 10 MPa minimum value for self-leveling subfloors. Thus, all the mixtures produced can be accepted, and the FC and CB mixtures reach this value already at 7 days.

In calcium sulfate-based mortars, Schaefer [46] recommends that the compressive strength should be 20 MPa at 28 days. Among the mortars produced with phosphogypsum, only FC meets the minimum strength value at this age.

In order to evaluate how much the results of compressive strength of studied mortars differ from each other, a statistical analysis of variance (ANOVA) was performed, with a significance level (α) of 5%, as shown in Table 7.

Table 7. Analysis of variance of compressive strength

Variable	Sum of Squares			Degrees of Freedom			F			p-value		
	1 day	7 days	28 days	1 d	7 d	28 d	1 d	7 d	28 d	1 d	7 d	28 d
Between Groups	1595	1885.12	2949.36	3	3	3	127.43	49.77	192.00	$4.31 .10^{-7}$	$1.61 .10^{-5}$	$8.62 .10^{-8}$
Within Groups	33.38	101,00	40.96	8	8	8						
Total	1628.37	1986.12	2990.33	11	11	11						

As p-value < α (p-valor < 0.05), the means between the compressive strength at all ages is proven. To analyze the magnitude of the difference between the means, the Tukey test was used, to complement the analysis of variance, as shown in Table 8.

At the Tukey test, considering a significant level of 5%, there is no difference between the means of FC-FN mortars at ages of 1 and 7 days, as well as in CB-FC mortars with 7 days, since p-value > 0.05. In the other mixtures, the means are considered to be significantly different, as the p-value is < 0.05.

Table 8. Tukey test for compressive strength

Mortars compared	Age	Difference	Inferior Limit	Superior Limit	p-value
AR-FN	01 day	-28.07	-33.41	-22.72	0.0000007
AR-FC		-28.34	-33.68	-22.99	0.0000007
AR-CB		-20.11	-25.45	-14.76	0.0000097
FC-FN		-0.27	-5.61	5.07	0.9983603
CB-FN		7.96	2.61	13.30	0.0061194
CB-FC		8.23	2.88	13.57	0.0050084
AR-FN	07 days	-34.11	-43.40	-24.81	0.0000118
AR-FC		-25.18	-34.47	-15.89	0.0001118
AR-CB		-17.91	-27.20	-8.61	0.0012047
FC-FN		8.92	-0.36	18.21	0.0596687
CB-FN		16.20	6.90	25.49	0.0023185
CB-FC		7.27	-2.01	16.56	0.1331075
AR-FN	28 days	-42.85	-48.77	-36.94	0.0000000
AR-FC		-30.58	-36.49	-24.66	0.0000009
AR-CB		-21.02	-26.94	-15.11	0.0000150
FC-FN		12.27	6.36	18.19	0.0007346
CB-FN		21.83	15.91	27.74	0.0000113
CB-FC		9.55	3.63	15.46	0.0037627

3.6 Tensile flexural strength

As with the compressive strength, the mixture produced with the white ceramic (CB) waste was the one with the best results among the analyzed wastes, as shown in the graph in Figure 10. The value achieved for the tensile strength of the CB mortar at 3 days is only 13.00% lower than the result for the AR mortar, and 3.8% lower at 28 days.

The study called ILS n. 349 of C1708 standard [36] conducted with 5 replicates of commercial self-leveling mortars indicates average resistance to 116 flexural tensile strength of 4.45 MPa at 01 day, and 7.45 MPa at 7 days. Mortars were not evaluated at these ages, but the values are quite consistent.

For this property, more satisfactory performance of natural phosphogypsum (FN) was observed compared to calcined phosphogypsum (FC) after 28 days. In any case, mortars can be considered high strength by presenting tensile strength values greater than 4 MPa, which is the minimum limit prescribed in NBR 11801 [49].

This standard presents the requirements for high floor strength mortars. In addition, Schaefer [46] also recommends that in calcium sulfate-based mortars (phosphogypsum) the flexural tensile strength should be 4 MPa at 28 days.

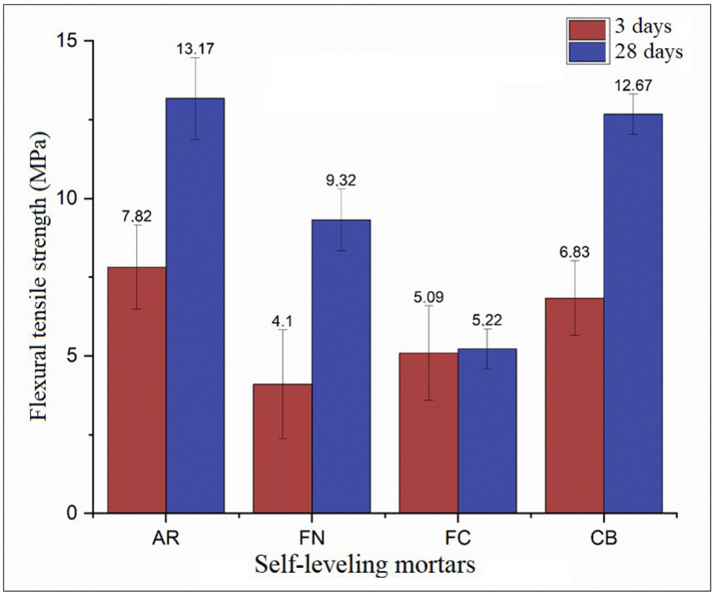


Figure 10. Results of flexural tensile strength of mortars at 3 and 28 days

In the draft standard reported by Silva et al. [48], concerning the execution procedure for self-leveling gypsum subfloor, a 3 MPa minimum flexural tensile strength is required. Therefore, all mortars evaluated also meet this criterion at all ages.

A statistical analysis of variance (ANOVA) was performed for the tensile flexural strength results, to discuss how much the results differ from each other. The results found are shown in Table 9.

Table 9. Analysis of variance of tensile flexural strength

Variable	Sum of Squares		Degrees of Freedom		F		p-value	
	3 days	28 days	3 days	28 days	3 days	28 days	3 days	28 days
Between Groups	25.35	121.29	3	3	6.12	45.94	0.018117	$2.18.10^{-5}$
Within Groups	11.03	7.04	8	8				
Total	36.39	128.33	11	11				

As with the compressive strength, the p-value results were also below 0,05 (p-value = 0.018 at 3 days and 2.18.10-5 at 28 days), adopting a confidence level of 95%. Therefore, the statistical analysis was also complemented with Tukey test, to show the comparison between all pairs of means, as shown in Table 10.

Table 10. Tukey test for tensile flexural strength

Mortars compared	Age	Difference	Inferior Limit	Superior Limit	p-value
AR-FN	03 days	-3.72	-6.79	-0.65	0.0194316
AR-FC		-2.73	-5.80	0.33	0.0822377
AR-CB		-0.99	-4.06	2.07	0.7328111
FC-FN		0.99	-2.07	4.06	0.7346940
CB-FN		2.73	-0.34	5.80	0.0826423
CB-FC		1.73	-1.33	4.80	0.3352774
AR-FN	28 days	-3.84	-6.29	-1.39	0.0045020
AR-FC		-7.94	-10.39	-5.49	0.0000301
AR-CB		-0.49	-2.94	1.95	0.9131918
FC-FN		-4.10	-6.55	-1.64	0.0030318
CB-FN		3.35	0.89	5.80	0.0101720
CB-FC		7.45	4.99	9.90	0.0000486

The analysis of Tukey test allows to conclude that at 3 days there is a significant difference between the tensile flexural strength results only in relation to the AR-FN, considering a significance level (α) of 95%, since p-value is < 0.05. At 28 days, there is no significant difference only in the comparison between AR-CB.

3.7 Linear shrinkage

The volumetric changes that cause shrinkage or expansion are the result of cement hydration reactions and the movement of water in the mixture. The loss of water can occur to the external environment or due to consumption in reactions. Linear shrinkage above established values causes cracks, which can compromise the durability of these materials.

Among the mortars evaluated, higher linear shrinkage values were identified in the mixtures containing calcined phosphogypsum and untreated phosphogypsum, FC, and FN, respectively, throughout the analyzed period. For this parameter, it can be seen that the calcination of phosphogypsum had a negative impact, increasing the shrinkage of the mixtures, as illustrated in the graph in Figure 11.

The shrinkage values measured for the AR and CB mixtures at all ages are below the limit recommended by Barluenga and Hernández-Olivares [18], which is 1.2 mm/m. The graph allows the comparison between the shrinkage values found. The only the lines defined for AR and CB are above the limit established in the literature.

The shrinkage of mortars with natural and calcined phosphogypsum at early ages can be justified by the greater heat release caused by these residues in the hydration of the paste in the first hours. The AR and CB mortars have less heat release, resulting in lower shrinkage values.

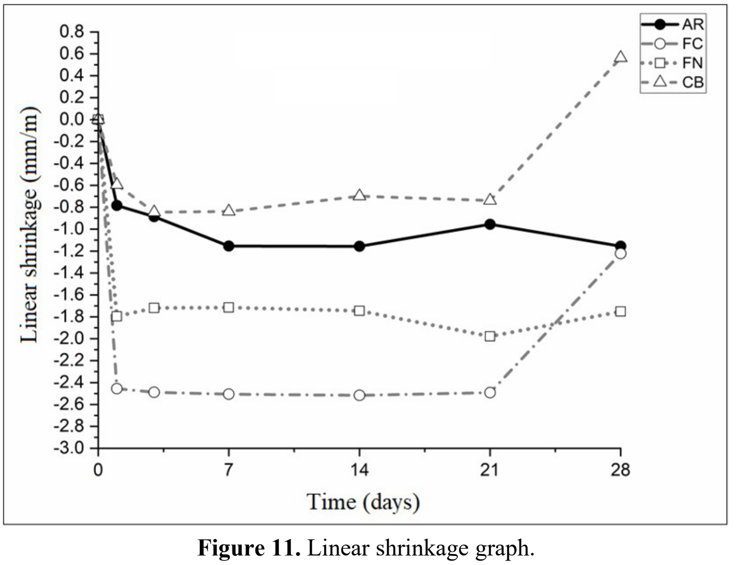


Figure 11. Linear shrinkage graph.

Schaefer [46] also observed that pastes with higher heat release were those that showed higher cracking. According to the author, heat development can be related to ettringite formation. Onishi and Bier [50] explain that the shrinkage that occurs in mixtures with anhydrite-type calcium sulfate, which is obtained by calcining phosphogypsum (FC), occurs due to the formation of monosulfate, in addition to ettringite, in the first hours of hydration. Schaefer [46] also explains that anhydrite is a calcium sulfate with slow dissolution rate, leading to the formation of crystals with long and fine needles, and that the type of calcium sulfate used in mortars and the morphology of the ettringite crystals are related to the dimensional stability in the hardened state.

In the CB mixture, the objective of reducing the dimensional variation in relation to the AR, which has a high cement consumption, was achieved. In contrast, for mixtures FN and FC no reduction in shrinkage values was observed, which may also be related to the waste granulometry.

The granulometry did not act as expected in filling the voids, facilitating the movement of water to the external environment and causing drying shrinkage, as confirmed in the void ratio and SEM tests, which indicate a porous surface in these mixtures.

In the CB and FN mixtures it can be seen that there is a greater shrinkage until 21 days, accompanied by an expansion after this period. Nawa and Horita [51] explain that this effect can be caused by chemical and autogenous shrinkage. According to the authors, when the mixture is in its fresh state there is an apparent variation in volume due to autogenous shrinkage, which occurs at the same time as chemical shrinkage. With this, after the development of the cement grains hydration, there is an increase in the volume of air in the capillary pores.

3.8 Specific mass, void ratio and water absorption

The highest water absorption measured was in the mortar containing natural phosphogypsum (FN), as well as the highest void ratio. For all mortars with wastes studied, the water absorption and the void ratio were higher than those from the reference mortar, as observed in the results in Table 11.

Table 11. Values of water absorption, void ratio, and specific mass of the mortars studied

Evaluated mortar	Water absorption (%)	Void ratio (%)	Specific mass (g/cm^3)
AR	9.79 ± 0.13	20.32 ± 0.21	2.08 ± 0.01
FN	18.65 ± 0.34	32.33 ± 0.17	1.74 ± 0.02
FC	15.59 ± 0.29	28.65 ± 0.24	1.85 ± 0.01
CB	15.03 ± 0.15	27.34 ± 0.32	1.82 ± 0.03

The FC and CB mixtures showed similar values for water absorption, void ratio, and specific mass. The highest value of void ratio observed was in the FN mortar, as this may be associated with its particles morphology, decreasing the void filling effect. However, the low density verified is beneficial in the application of this product, considering it will be lighter [52], but the fact that the porous surfaces are more susceptible to the attack of aggressive agents should also be taken into consideration.

3.9 Tensile adhesion strength

Determination of the tensile adhesion strength was performed 28 days after application of the self-leveling mortars on a cement-based substrate, in order to simulate on-site application. The average resistance results found are listed in Table 12.

Table 12. Results of adhesion strength tests

Evaluated mortar	Average rupture strength (N)	Average tensile adhesion strength (MPa)
AR	3122	1.59
FN	810	0.41
FC	1519	0.77
CB	1295	0.66

During the test, the rupture strengths and failure modes of the extracted test specimens were noted, 12 for each mixture. The predominant failure mode was at the substrate / mortar interface, with the exception of one FN mortar test specimen, which broke in the mortar.

In addition, discrepant mean values and test specimens that broke on the glue were discarded from the data set, as they may be related to problems in the test execution process.

The average tensile adhesion strength value obtained by Mendes [20] is in the range of the values measured in this paper. The author obtained a 0.51 MPa average strength at 30 days for the mixture using RCMG.

3.10 Microstructural analysis

Fragments of the mortars studied were collected after 28 days for the microstructure analysis. The images obtained are illustrated in Figures 12 and 13.

In Figure 10, with images magnified 200x the presence of pores can be identified by the darker areas. The FN and FC mixtures in Figure 12 c) and d), respectively, present a more porous and less homogeneous aspect than the others, which is confirmed in the water absorption and void ratio results. In addition, the porosity and lower homogeneity of the mortars can be related to the lower compressive strengths found for these mixtures.

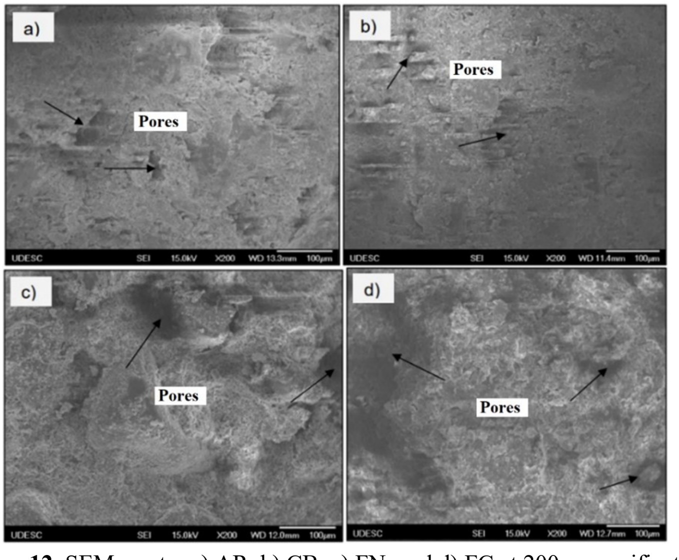


Figure 12. SEM mortar a) AR, b) CB, c) FN, and d) FC at 200x magnification.

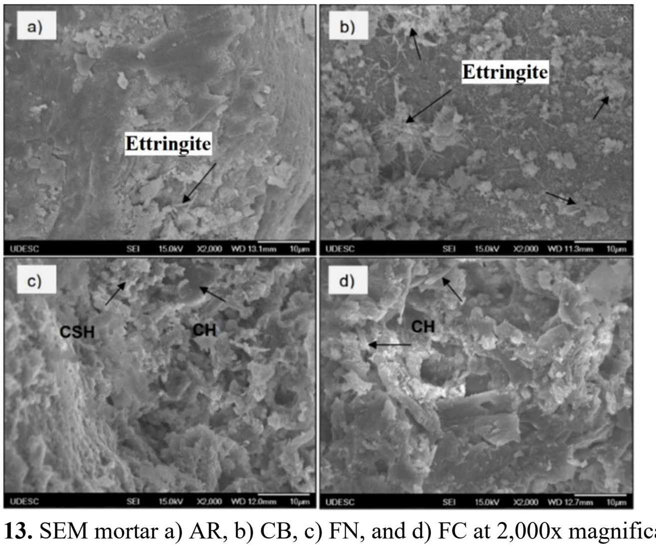


Figure 13. SEM mortar a) AR, b) CB, c) FN, and d) FC at 2,000x magnification.

In this study, the development of the crystalline structure of the mixtures is not studied, however, in Figure 13 it is possible to visually observe some products of the cement hydration, which were formed in the hardening process of the paste. Visual analysis of the AR and CB mixtures allows the ettringite to be identified by the needle-like, acicular morphology.

4 CONCLUSIONS

Considering the results found, it is observed that they were within the expected range for mortars containing white ceramic and phosphogypsum waste calcined at 450°Cfor 4 hours, originating anhydrite, indicating a potential use of these materials in self-leveling mortars for subfloors, reducing cement consumption and promoting a sustainable destination for this waste.

Considering that the use of self-leveling mortars is a relatively new practice in Brazil, self-leveling mixtures were developed for subfloors by replacing 50% (by mass) of Portland cement with waste. Only the white ceramic showed pozzolanic activity.

In the mortar dosages, phosphogypsum in its natural state compromised workability and decreased the regeneration time of the mixtures, requiring larger amounts of superplasticizer additive to achieve the minimum spread of 125 mm, as well as the absence of segregation and exudation. However, calcination of phosphogypsum proved beneficial to the fluidity and regenerability of the mixtures.

In the analysis of the initial and final setting times, natural phosphogypsum showed a large discrepancy between the initial and final setting times. However, the behavior of the mortar produced with anhydrite was similar to the mortar produced with cement only (reference), as calcination increases the specific area of phosphogypsum and provides faster hardening. Unlike all of them, the mixture with white ceramic resulted in an initial setting and hardening time 28.85% shorter than the AR.

Through the proposed methodology, properties in the fresh and hardened state were evaluated. The mechanical behavior of the mortars was impaired by the addition of waste since they demanded higher water/cement (w/c) ratios and quantities of superplasticizer additives to meet flow requirements. However, the CB mixture showed values higher than those established in the literature at all ages studied (1, 7, and 28 days).

The calcination of phosphogypsum had little influence on the compressive strength at 1 day, when compared to the mixture with natural phosphogypsum. However, when compared to the mixture with untreated phosphogypsum (FN), the mortar with anhydrite (FC) showed approximately twice the compressive strength at 7 and 28 days. This result can be explained by the fact that these materials did not show pozzolanic activity, resulting in later strength gains due to the filler effect because the dissolution of anhydrite produces less phosphate ions than natural phosphogypsum and because anhydrite is a type of calcium sulfate that dissolves slowly.

In the evaluation of flexural tensile strength, all mixtures showed a performance higher than 4 MPa at 28 days, being classified as high strength flooring mortars.

As for shrinkage, only the mixture produced with white ceramic waste performed well, resulting in values within the limits established in the literature. The high shrinkage in the mixtures containing phosphogypsum is related to the higher heat release in the initial hours, observed in the calorimeter.

Furthermore, all mixtures containing waste showed higher values of water absorption and void ratio than the AR, which can also be observed in the SEM analysis. Especially for the mixtures with phosphogypsum, a greater porosity is observed, which is consistent with the compressive strength values found.

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Editors: Vladimir Haach, Guilherme Aris Parsekian.