### Influence of chemical pretreatment on the pozzolanicity of recycled glass microparticles used as a substitute for Portland cement

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**ABSTRACT:** This research investigated the influence of using chemically treated glass microparticles as a partial replacement for cement in Portland cement pastes and mortars. The microparticles were obtained by grinding glass waste into three different particle size fractions ( $<75 \mu$ m,  $<45 \mu$ m, and  $<25 \mu$ m), treated with calcium hydroxide (CH), and characterized using SEM/ EDS and a laser particle size analyzer. Samples prepared with the incorporation of glass were characterized using XRD, TGA/ DTG, and SEM/EDS. The pretreatment with calcium hydroxide induced the formation of C-S-H with different morphologies on the surface of the particles, in addition to causing changes in particle size distribution due to the formation of agglomerates. The pastes prepared with treated particles had lower amounts of CH and higher levels of hydrated silicates. However, when indirectly measuring the pozzolanicity of treated particles through the compressive strength of mortars, no significant differences were observed in the strengths of mortars made with treated and untreated particles.

KEY WORDS: Portland cement; Mortar; Calcium silicate hydrate (C-S-H); Portlandite; Pozzolan; Glass particles.

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**RESUMEN:** *Influencia del pretratamiento químico en la puzolanicidad de micropartículas de vidrio reciclado utilizadas en sustitución del cemento Portland.* Este estudio investigó la influencia del uso de micropartículas de vidrio tratadas químicamente como reemplazo parcial del cemento en pastas y morteros de cemento Portland. Las micropartículas se obtuvieron mediante la molienda de residuos de vidrio en tres fracciones de tamaño de partícula diferentes (<75 µm, <45 µm y <25 µm), tratadas con hidróxido de calcio (CH) y caracterizadas mediante SEM/EDS y un analizador de tamaño de partícula láser. Las muestras preparadas con la incorporación de vidrio se caracterizaron mediante XRD, TGA/DTG y SEM/EDS. El tratamiento previo con hidróxido de calcio indujo la formación de C-S-H con diferentes morfologías en la superficie de las partículas, además de provocar cambios en la distribución del tamaño de partícula debido a la formación de aglomerados. Las pastas preparadas con partículas tratadas tenían menores cantidades de CH y niveles más altos de silicatos hidratados. Sin embargo, al medir indirectamente la puzolanicidad de las partículas tratadas a través de la resistencia a la compresión de los morteros, no se observaron diferencias significativas en las resistencias de los morteros hechos con partículas tratadas y no tratadas.

PALABRAS CLAVE: Cemento Portland; Mortero; Silicato de calcio hidratado (C-S-H); Portlandita; Puzolana; Partículas de vidrio.

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#### **1. INTRODUCTION**

Climate change is occurring as a result of human activities, the most significant contribution being due to industrial expansion (1). These changes are caused by CO<sub>2</sub> emissions that enhance the greenhouse effect (2), making climate phenomena increasingly extreme (3). Because of this, the European Union aims to be climate neutral by 2050 - an economy with zero net greenhouse gas emissions (4).

Portland cement concrete is one of the most widely used construction materials. The production of the main constituent of concrete, cement, is energy intensive and highly polluting. Due to this, the cement industry ends up contributing with about 5% to 8% increase in global carbon dioxide (CO<sub>2</sub>) emissions, emitting about 2 Gt of CO, per year (5). And it represents between 12% and 15% of the total industrial energy consumption (6). Clinker, the base material of Portland cement, is produced by the decomposition of calcium carbonate into calcium oxide and high amounts of CO<sub>2</sub>, followed by the sintering of calcium oxide with aluminosilicates and the rest of the raw materials. Calcination of limestone is responsible for about 95% of greenhouse gas emissions, while consuming 80% of total energy demands (7).

More than half of the emissions from the cement production process come from the decomposition of the CaCO<sub>3</sub> present in the limestone, which follows Equation [1] which occurs from 700 °C, the other part of the emission from the processes comes from the energy used in the process (8). The calcination process represents more than 50% of total CO<sub>2</sub> emissions from cement manufacturing, and the reduction of the clinker/cement ratio is intended to reduce energy consumption and improve some characteristics of the final product (9). The high carbon emissions of cementitious materials are increasingly raising concerns about the grand goal of global carbon neutrality (10).

$$CaCO_{3(s)} \rightarrow CaO_{(s)} + CO_{2(g)}$$
 [1]

The great mineral extraction generated by the cement industry is also a cause for concern due to the reduction of natural reserves of limestone and clay. In addition to emissions, cement production consumes large amounts of natural resources and devastates large areas due to the need for clay and limestone mining. 1.5 ton of raw material is used to produce one ton of clinker, while each ton of Portland cement generates almost one ton of CO<sub>2</sub> (7, 11). Faced with these environmental impacts, a portion of cement in concrete is often replaced by a supplementary cementitious material (SCM) that has a lower environmental impact and often lower cost. Despite the favorable influence of SCMs on fresh, hardened and durability properties of concrete, SCM resources are finite and current supply can only meet around 15% of concrete industry demand worldwide (12).

Waste glass (WG) causes serious environmental problems because it is not biodegradable, reducing the useful life of landfills and, increasing the severity of its pollution in the ecosystem. WG is very common in our daily lives, it can be found in different types of products, such as bottles, windows and lamps, which have a limited useful life, with glass recycling being the most promising way to mitigate its environmental (13). The use of WG in concrete and mortar is a promising strategy to mitigate the environmental problems of WG and cement production, but research is needed to comprehensively explore the effects of this use (14).

When glass is pulverized to the size of microparticles, due to its chemical composition rich in silica and the degree of disorder in its atomic structure, it can be used as a cement substitute SCM in cement systems. The most important phenomenon in this substitution is the pozzolanic reaction of the glass, which leads to the formation of large amounts of extra Hydrated Calcium Silicates (C-S-H) in cement mixtures, which are the binding phases of hardened cement (15–18). The increase in the C-S-H gel is found due to the pozzolanic reaction of the glass powder, with portlandite (calcium hydroxide - CH) and water, as shown in Equation [2] (18). The reaction affects the contraction of voids and pore densification of mortar prepared with waste glass (19). The amorphous structure of glass allows it to be easily dissolved in a high alkalinity environment and function as a pozzolanic material in cement systems.

$$\begin{array}{l} \text{Ca(OH)}_2 (\text{Portlandite}) + \text{SiO}_2 (\text{Pozzolan}) + \\ \text{H}_2\text{O} \rightarrow \text{C-S-H} (\text{Calcium Silicate Hydrate}) \end{array}$$
[2]

The characterization of pozzolans through the Pozzolanic Activity Index (PAI) with Portland cement or portlandite, as recommended by NBR 5752 and NBR 5751 (20, 21), respectively, is primarily based on indirect methods for assessing their reactivity. These methods aim to estimate the amount of C-S-H formed considering the mechanical performance of mortars (22). Highly reactive pozzolans, characterized by their elevated amorphous content and, notably, their high specific surface area, tend to consume a significant amount of portlandite. In Portland cement mortars or with hydrated lime, this can deplete the alkaline reserve of the medium. Moreover, these methods do not account for the specific surface area of the additive or the constant water-to-binder ratio, both of which are crucial factors. This oversight can lead to reduced performance when using highly reactive additives, as they require more mixing water to meet the regulatory consistency requirements (22).

These factors can lead methods to underestimate the pozzolanicity of materials and make their hierarchization difficult. The combined analysis of PAI with DRX and SEM/EDS aims to examine the consumption/depletion of portlandite in the systems. This analysis considers that some pozzolans may exhibit low

Influence of chemical pretreatment on the pozzolanicity of recycled glass microparticles used as a substitute for Portland cement • 3

performance when evaluated based on the compressive strength of mortars with CH, despite their ability to bind a high content of calcium hydroxide and form hydrated compounds (C-S-H) (22–24). Many authors have investigated and proposed physical methods to enhance the pozzolanic properties of glass by reducing the size of glass particles and consequently increasing their surface area. It is widely agreed that reducing particle size and increasing surface area of glass effectively alters the kinetics of the chemical reaction toward the pozzolanic reaction (5, 25–30). However, some authors discuss chemical pretreatments/methods aiming to increase the pozzolanicity of SCMs (31–33).

Pretreatments are used as methods to enhance the performance of SCMs in concretes and mortars. These pretreatment techniques include physical methods, such as mechanical grinding, and chemical methods. Chemical pretreatment involves immersing materials in different acidic or basic solutions before use. The objective of a pretreatment is to promote the pozzolanic reaction and allow higher levels of residue incorporation. The effectiveness of a pretreatment is evaluated by testing the concrete or mortar in which a part of the cement has been replaced by the pretreated SCMs, and examining how the pretreatment influenced the durability and mechanical properties of the cement system, as demonstrated in (34).

In the literature, there are still uncertainties regarding a complete understanding of the impact of glass on the durability of cement systems. Pretreatment in alkaline solution on fine glass aggregate causes the formation of a dense layer of Calcium Silicate Hydrate (C-S-H) on the surface of the glass when Ca(OH), is used in the solution, which slows down the dissolution rate of the glass. The reaction product is dense and firmly attached to the glass particle substrate. The product on the surface of the glass particle aggregates will act as a protective layer, which may consequently reduce the risk of alkali-silica reaction. The use of pretreatment can ensure the durability of cement systems that use glass particles, and therefore the incorporation of more glass in cement systems (35). However, Sun et al. (35) explored the effects of such treatment on fine glass aggregate rather than on microparticles, in addition to the effects of pretreatment on pozzolanic activity and microstructure of cement systems containing pretreated particles.

Research into the use of SCMs is advancing, but there is still room to achieve improvements in their performance and greater knowledge about their effects. There are still many gaps in knowledge, and more research is needed on the use of pretreatment in SCM in the concrete industry to produce more ecologically sustainable and efficient materials (34). Considering the potential to expand glass waste recycling methods and aiming to simultaneously reduce cement consumption and associated environmental problems, this article investigates the influence of using chemically treated glass microparticles as a partial substitute for cement in pastes and Portland cement mortars. The mortars were evaluated using the methodology described in the Brazilian standard NBR ABNT 5752 (21). To achieve this, three different sizes of glass microparticles were evaluated, both with and without chemical treatment in an alkaline solution.

#### 2. EXPERIMENTAL PROCEDURE

#### 2.1. Characterization methods

#### 2.1.1. X-ray fluorescence spectroscopy (XRF)

Chemical composition of cement, glass and calcium hydroxide was determined using XRF on a Shimadzu XRF-1800 equipment, with Rh Tube, 4.0 kW, voltage of 30 kV, and current of 60 mA.

#### 2.1.2. X-ray powder diffraction (XRD)

Diffraction patterns were obtained using a Shimadzu LabX XRD-6000 instrument, with a nominal  $2\theta$ scan range between 5° and 60°, a step size of 0.02°, a scan speed of 2°/min, and an operating voltage of 40 kV with a current of 30 mA, using a copper anode.

#### 2.1.3. Scanning electron microscopy (SEM/EDS)

Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) analyses were conducted using a JEOL JSM-5700 instrument at 15 kV. Prior to analysis, the samples were coated with silver for 200 seconds.

#### 2.1.4. Laser diffraction granulometry

The particle size distribution of the glass microparticles was obtained using a Mastersizer MS2000-E equipment with a Helium/Neon laser and a detection range between 0.1 and 1000  $\mu$ m.

#### 2.1.5. Thermogravimetric analysis

Thermogravimetric analysis (TG/DTG) of the pastes was carried out using NETZSCH equipment, model STA 449 F1 JUPITER. Approximately 10 mg of powder was heated from 25 to 1000 °C under a nitrogen gas atmosphere at a rate of 10 °C/min.

#### 2.2. Materials

The Portland cement type II (here denoted as PC, determined by NBR 5752 (21)) and the calcium hy-

droxide  $(Ca(OH)_2$  - here denoted as CH, provided by ACS Científica were used as received. Glass microparticles (GMP) were obtained by grinding waste from soda-lime glass packaging. The glass density of 2.54 g/cm<sup>3</sup> was determined by the pycnometer method. The particle size fractions used were obtained from glass containers that were washed with detergent, dried, ground in a ball mill, and sieved through mesh sieves #200 (<75 µm), #325 (<45 µm) and #500 (<25 µm) (Figure 1).

The chemical composition of the raw materials is indicated in Table 1. Figure 2 shows the X-ray diffraction patterns of the cement (Figure 2.A) and the characteristic amorphous halo of glass (Figure 2.B). Cement is formed by its characteristic phases C<sub>3</sub>S (ICSD 81100), C<sub>2</sub>S (ICSD 39006), C<sub>3</sub>A (ICSD 1841), C<sub>4</sub>AF (ICSD 2841) and gypsum (ICSD 168091). It presents traces of quartz (ICSD 16331) and a high content of calcite (ICSD 423568), as it is a cement with the addition of limestone filler.

TABLE 1. Chemical composition of raw materials.

Oxides	PC (%) *	GMP (%)	CH (%)
SiO <sub>2</sub>	19.45	73.11	0.90
$Al_2O_3$	4.79	2.16	-
CaO	64.87	11.95	94.95
Fe <sub>2</sub> O <sub>3</sub>	2.94	-	-
MgO	3.27	0.79	4.12
Na <sub>2</sub> O	-	11.96	-
$SO_3$	4.66	-	-
Others	0.02	0.03	0.03
		-	

\*Portland cement with addition of limestone filler



FIGURE 1. Summary of the steps involved in preparing the particle size fractions used: (A) packaging used; (B) fragmented glass inserted into the mill; (C) ball mill used; (D) result of the grinding process; (E) set of sieves used; (F) particle size fractions obtained.



FIGURE 2. X-ray diffraction patterns of (A) Portland cement and (B) Glass microparticles.

# 2.3. Chemical treatment of glass microparticles (GMP)

The three different particle size fractions of GMP ( $<75\mu$ m,  $<45\mu$ m,  $<25\mu$ m) underwent a chemical pretreatment in which 250 g of glass (of each fraction) were soaked in a 1-liter saturated water solution with 5 g of calcium hydroxide for 7 days at a temperature of 80°C (35). At the end of the treatment, the material was dried in an oven at the same temperature as the treatment until all the water was removed.

## 2.4. Preparation of mortars and evaluation of the Pozzolanic Activity Index (PAI)

The determination of the Pozzolanic Activity Index (PAI) using Portland cement was conducted in accordance with the standard NBR 5752 (21). This analysis involved mortars produced from three different particle size fractions of glass powder ( $<75 \mu m$ ,  $<45 \mu m$ , <25 µm), both with and without pre-treatment. A reference mortar, without the addition of GMP, was produced, along with six other mortars in which 25% of the cement was replaced by GMP. Mortars containing 25% pre-treated GMP were identified with the letter 'T' in their nomenclature. For each composition, four cylindrical specimens were cast, with dimensions of 50 mm x 100 mm (diameter x height). In accordance with NBR 5752/2017, the amount of water was kept constant at 200 mL for all samples, and those with the addition of GMP presented a consistency similar to that of the reference sample, within a tolerance of ±10 mm. The compositions and quantities of materials required for production are detailed in Table 2.

The specimens were cured in their molds, kept closed at room temperature for the first 24 h, and then placed to cure submerged in water at 23 °C. At the end of the curing process, the specimens were submitted to the compressive strength test.

### **2.5.** Preparation of pastes with compositions similar to the mortars

The preparation of these pastes maintained the water-to-binder (w/b) ratio constant at 0.70. The w/b ratio for pastes needed to be higher compared to mortars due to the fineness of the materials. It was determined based on the minimum amount of water required for cement pastes containing finer particle size fractions ( $25\mu$ m and  $25\mu$ mT) to be adequately mixed and fill the molds without leaving empty spaces. After mixing, the pastes underwent submerged curing in water, carried out at  $23 \pm 2$  °C for 28 days. The pastes from mixtures identical to those of the mortars had the hydration reaction interrupted at the end of the curing process, being submerged for 1 hour in isopropyl alcohol, dried for 1 hour in an oven at 70°C, and stored under vacuum.

#### **3. RESULTS AND DISCUSSION**

#### 3.1. Characterization of GMP

The graphs in Figure 3 show the cumulative grain size distribution curves in % of the particle size fractions of untreated (3.A) and treated (3.B)

Mortar	CP II 32-F <sup>1</sup> (g)	Glass (g)	standard sand <sup>2</sup> (g)	Water <sup>3</sup> (mL)	Consistency <sup>4</sup> (mm)
PC	416	-	1248	200	205
75µm	312	104	1248	200	202
45µm	312	104	1248	200	201
25µm	312	104	1248	200	198
75µmT	312	104	1248	200	199
45µmT	312	104	1248	200	197
25µmT	312	104	1248	200	196

TABLE 2. Proportions of components in mortar mixtures used for PAI with cement.

<sup>1</sup> CP II 32-F - Composite Portland cement with addition of limestone filler.

<sup>2</sup> Standard sand according to NBR 7214 (36): 234 g of each of the four fractions (coarse, #16; medium coarse, #30; medium fine, #50; and fine, #100).

<sup>3</sup> Amount of water of 200 mL fixed by NBR 5752 (21).

<sup>4</sup> Workability obtained in the slump test, using the spreading table, as prescribed by NBR 7215 (37), the consistency of the samples with addition could vary  $\pm 10$ mm from the consistency of the standard.

glass particles. It can be observed that the treatment changed the initial conditions of particle distribution and their maximum size. The treatment of glass particles increased the values of  $D_{10}$ ,  $D_{50}$  and  $D_{90}$ , and reduced their specific surface area, with the exception of the specific surface area of the fraction separated by the #500 mesh sieve (<25µm) as shown in Table 3.

Figure 4A contains scanning electron microscopy images of the untreated glass particles, where the characteristic smooth surface of the glass can be seen. In Figures 4B and 4C, the surface of the particles appears completely covered by pozzolanic C-S-H, a product strongly adhered to the surface of the glass particle (35), originating from the pozzolanic reaction that occurred during the treatment. It can also be seen that the particles were held together by this C-S-H layer.

The pretreatment changed the smooth surface of the glass particles to a rough surface coated with different C-S-H morphologies (38–40). Despite the small amount of calcium hydroxide used for the treatment, 2% in relation to the mass of glass, compared to the large surface area of all the granulometric fractions of glass particles, no particles without C-S-H coating

 TABLE 3. Values of the accumulated frequency of particle size D10, D50, D90, Specific Surface Area (BET) m²/g and Average Diameter (AD) of the respective granulometric fractions of glass used, obtained by laser granulometry.

GMP	D <sub>10</sub> (µm)	D <sub>50</sub> (µm)	D <sub>90</sub> (µm)	Area BET (m <sup>2</sup> /g)	AD (µm)
75µm	4.969	20.636	65.524	0.796	28.814
45µm	4.890	16.116	33.368	0.861	17.851
25µm	3.131	11.988	34.791	1.070	13.254
75µmT	5.122	24.529	71.513	0.719	32.968
45µmT	5.420	20.409	47.047	0.782	27.289
25µmT	3.131	11.988	24.791	1.070	23.459



FIGURE 3. Accumulated particle size distribution curves and SEM images of (A) 3 particle size fractions of untreated glass and (B) 3 particle size fractions of treated glass.

were observed in the SEM images. The C-S-H that covers the particles has an acicular morphology, with small layers in the 'cloisonné' style (bee panel) and with flattened elongated fibers (41).

Still in Figure 4 (B and C) it is possible to observe that the smaller glass particles were encompassed by the C-S-H formed around the larger glass particles, behavior different from that observed in the work by Sun et al. (35). This is due to the ratio between the size of the particles used and the amount of C-S-H generated around the particles. In the work by Sun et al. (35) the particles used were much larger, ranging from 4.76mm - 2.38mm (#4 and #8) and the amount of C-S-H generated was much smaller, which made it impossible for the agglutination of particles to be generated by the C-S-H.

Figure 5 shows an SEM image and its respective EDS analysis of the treated particle surface, highlighting another characteristic morphology of calcium silicate hydrate (42–44). The composition found indicates the presence of calcium, silicon and sodium. The Ca/Si ratio obtained was 1.3, which is within the range to be considered referring to pozzolanic C-S-H (0.7<Ca/Si<2.1). Although the C-S-H phase is traditionally described as predominantly amorphous, Figure 5 shows similarities with natural minerals such as tobermorite and jennite (45, 46).

#### 3.2. Pozzolanic Activity Index (PAI) - mortars

The graph in Figure 6 shows the compressive strength results of the molded specimens according to the NBR 5752 standard (21), which evaluates the pozzolanic activity index using cement after 28 days. In order to verify the pozzolanicity of the different glass parameters used indirectly (through mechanical resistance), the standard establishes that the mortar containing 25% of cement replacement by the supposedly pozzolanic material



FIGURE 4. Scanning electron microscopy (SEM) of (A): untreated GMP; (B) and (C): treated GMP coated with tobermorite-type C-S-H.



FIGURE 5. EDS of the C-S-H formed on the surface of a treated GMP.



**FIGURE 6.** Graph with the compressive strength results of the PAI tests with Portland cement according to NBR 5752 for the studied grain sizes of the glass powder, with and without treatment.

must reach a minimum limit of 90% of the compressive strength of the reference mortar (100% cement), for the material to be considered pozzolanic. By the criterion of NBR 5752 (21), all particle sizes used were considered pozzolanic, it can be seen that both for treated and untreated particles, the smaller the particle size used, the better the mechanical result, as in recent works that investigated the influence of granulometry on the pozzolanic properties of glass (25–30).

Comparing the mortars between the respective particle size fractions with and without treatment, we can observe that for particle sizes  $<45\mu m$  and  $<75\mu m$  the mechanical results did not indicate significant differences. However, for sizes <25µm, the mortar with treated particles showed lower mechanical performance, resulting in reduced compressive strength and the formation of particle agglomerates, as represented in the SEM images of the treated particles. This, in turn, caused mortars containing treated particles of all sizes to have lower workability when the same amount of water was added in the fresh state, as illustrated in Table 2. It is worth noting that all compositions, both with and without treatment, resulted in compressive strengths equivalent to or greater than that of the reference mortar. Notably, the samples with a cement replacement of 45µm and 25µm without treatment exhibited gains of 25% and 33%, respectively, while the treated samples of 45µm and 25µm showed gains of 17% and 19%, respectively.

The methodologies proposed in the NBR 5752 (21) standard constitute an indirect method for classifying mineral additions, based on the compressive strength of pozzolanic systems, which are compared to arbitrary conditions recommended in NBR 12653 (47). The challenge with measuring pozzolanicity through indirect means is that other factors can influence the mechanical properties of the material, such as the water-cement ratio, since pozzolans with a high specific

surface area require more water to maintain the same pre-plasticity, which reduces mechanical resistance (22). It was possible to observe that, with the same amount of water, systems containing treated particles reached lower workability than their equivalent particles without treatment, which influenced the mechanical results.

Considering the standard deviation, the mechanical performance can be considered equal for the mortars with particles of 45 and 75  $\mu$ m, with and without treatment. However, the treatment reduced the mechanical strength of the mortars containing treated 25  $\mu$ m particles compared to the same fraction without treatment. This effect can be attributed to the formation of particle clusters generated by their agglomeration by the formed C-S-H, impairing the filler effect and reducing packing. This observation is corroborated by the particle agglomeration observed in Figure 3.

# 3.3. Characterization of cement pastes with 25% of GMP

#### 3.3.1. X-ray diffraction analysis

XRD analysis was used to estimate the pozzolanicity of GMPs in cement pastes at 28 days of age. The assessment of GMPs' pozzolanicity, with and without treatment, was conducted by examining the remaining portlandite content in various cementitious systems with the addition of glass. The diffraction patterns for standard paste samples (100% OPC) and those with 25% cement replacement by GMPs (<75µm, <45µm,  $<25\mu$ m), both treated and untreated, are displayed in Figure 7. In all formulations, characteristic peaks of portlandite  $(2\theta = 18.08^{\circ}, 28.66^{\circ}, 34.10^{\circ}, \text{ and } 47.12^{\circ})$ , calcite  $(2\theta = 29.40^\circ)$ , and ettringite  $(2\theta = 9.09^\circ)$  are present. Despite the 25% replacement of cement with GMPs, the curve profiles showed no significant variations related to the presence of an amorphous halo. This lack of variation can be attributed to the fact that cementitious products' diffraction patterns naturally include an amorphous halo associated with amorphous C-S-H.

To emphasize the impact of GMPs on portlandite consumption, Figure 8 presents the diffraction patterns of all evaluated compositions, with a focus on the 20 region between 17.0 and 19.0°. Based on the intensity of the peaks, it can be observed that the amount of portlandite decreases with the replacement of cement by glass powder under all conditions, indicating that the glass residues reacted with calcium hydroxide during the hydration process. As the glass particle size decreases, the peaks related to portlandite also decrease; this observation applies to both treated and untreated particles. It is also worth noting that in the pastes produced with treated glass particles, the portlandite peaks have lower intensities than their untreated counterparts. This indicates a greater



FIGURE 7. Diffraction patterns of pastes after 28 days of hydration.

consumption of portlandite due to the pozzolanic reaction (22, 48). This can be attributed to the surface changes that the treatment caused in the glass particles, altering their dissolution mechanisms (35).

The portlandite (CH) content can also decrease due to carbonation caused by exposure to CO<sub>2</sub> and hu-

midity in the aging environment of cement products. Equation 3 indicates, in a summarized and non-stoichiometric way, how the portlandite carbonation process occurs (49, 50).

$$Ca(OH)_2 + CO_2 + H_2O \rightarrow CaCO_3$$
 [3]

The carbonation of the samples led to a decrease in the amount of portlandite and an increase in the amount of CaCO<sub>3</sub>. In this case, the decline in CH content could be misinterpreted and attributed to the pozzolanic reaction. To resolve this doubt, Figure 9 presents a comparison of the intensities of the main calcite peak for all compositions investigated using untreated particles (Figure 9.A) and treated particles (Figure 9.B). It is notable that the reference sample exhibits a slightly more intense calcite peak than the compositions with GMPs. This is primarily due to the high level of substitution carried out (25 %wt of GMP), as the cement used to prepare the samples contains a significant amount of limestone filler. When observing the other samples, they all have similar peak intensities, with no changes caused by particle



FIGURE 8. XRD patterns showing the remaining CH content in the pastes at 28 days. (A) using untreated GMPs and (B) using treated GMPs.



FIGURE 9. Diffraction patterns highlighting the calcite peaks in the pastes at 28 days. (A) using untreated GMPs and (B) using treated GMPs.

size or treatment. This indicates that the reduction in portlandite peaks in the pastes shown in Figure 8 was solely due to its consumption through the pozzolanic reaction.

#### 3.3.2. Thermal behavior of the pastes

In Figure 10, the TGA/DTG curves of pastes with untreated and treated particles are shown, Figures 10.A and 10.B, respectively. Three decomposition regions can be highlighted. The first region occurs in the range of 80 to 400 °C and can be attributed to water loss, dehydration of C-S-H, decomposition of ettringite (AFt), and dehydration of hydrated aluminates (HA) (51, 52). The second region (400 - 460 °C) corresponds to the dehydration of portlandite. The third region (550 - 830°C) results from the decomposition of calcium carbonate due to the release of CO<sub>2</sub> (16, 53, 54). Naturally, pastes produced by replacing glass with GMPs exhibit lower amounts of portlandite due to its consumption by the pozzolanic reaction and a lower cement content. Compared to the reference paste, which experiences a 4.5% mass loss due to portlandite decomposition, pastes containing 25% GMPs exhibit reduced mass loss, ranging between 2.48% and 3.13%. All pastes containing GMPs exhibit lower mass loss related to the C-S-H compared to the reference paste (21.87%). Pastes with treated particles show higher thermal decompositions in the temperature range corresponding to the C-S-H than pastes with untreated particles (75µm  $\rightarrow$  10.52%, 75µmT  $\rightarrow$  14.36%, 45µm  $\rightarrow$  11.26%, 45µmT  $\rightarrow$ 13.43%, 25µm  $\rightarrow$  14.08%, and 25µmT  $\rightarrow$  17.8%), indicating greater formation of this product through pozzolanic reaction.

#### 3.3.3. Morphology of hydrated products (SEM/EDS)

As shown in Figure 11.A, CH is predominant in the reference paste after a curing time of 28 days. When glass waste was incorporated into the paste, a layer of Si-rich gel was formed on the surface of the glass particle due to its incongruous dissolution. The depolymerized silicates in this layer react with  $Ca^{2+}$  to form a C-S-H reaction ring, as shown in Figure 11 (B, C and, D). At the same time, the dissolved silicate can react with Ca(OH)<sub>2</sub> to form the external product (C-S-H), which resulted in a lower Ca/Si ratio (14). Thus, the pozzolanic reactivity of the glass waste was also confirmed by microscopy images.

The mechanism of the pozzolanic reaction is initially due to a large amount of  $Ca^{2+}$  and  $SiO_4^{-2-}$  released into



FIGURE 10. Thermal analysis of the pastes at 28 days. (A.1 and A.2) using untreated GMPs and, (B1 and B.2) using treated GMPs.

the pore solution. In this alkaline environment, the amorphous silica from the waste glass that is dissolved forms a Si layer on the surface of the waste glass particle. The released Ca<sup>2+</sup> ions react with the Si layer and produce C-S-H. As the addition of waste glass increased, more cations such as Na<sup>+</sup> contributed to forming more C-S-H with a higher Si/Ca ratio. Therefore, fewer free Ca<sup>2+</sup> ions and a lower pH of the pore solution can attenuate the hydration process of glass particles due to the dilution effect (14). The formation of extra C-S-H by the pozzolanic reaction explains the superior compressive strength results observed with the use of particles <25µm and <45µm, with and without treatment.

It is evident in Figures 11.B, C, and D the effect of heteronucleation of the C-S-H phase on the surface of the glass particle (55). The dissolved SiO<sub>2</sub> produces denser C-S-H than the CH consumed by the pozzolanic reaction, which develops a rim around the glass powder particles (56). Due to the products formed on the surface of the treated particles with a morphology similar to tobermorite, the C-S-H nucleation power of the glass particles is increased, and they begin to act in a manner analogous to seeds of C-S-H (38). Smaller glass particles were not found adhered to larger particles in cement pastes. The alkaline environment, coupled with the very high surface area of these particles (diameter  $<1\mu m$ ), may have caused them to dissolve completely in the paste, increasing the silicon available to form C-S-H.

The interfacial transition zone (ITZ) between the GMP and the cementitious matrix can be visualized in the SEM images of the untreated pastes (Figure 11.B), but are not seen in the treated pastes (11.C and 11.D). In pastes with treated particles, it was possible to observe that the C-S-H formed was linked directly to the glass particle, without a defined transition between what was C-S-H and what was glass. To evaluate this transition region, an EDS analysis was performed at several points, connecting one end to the other of a particle with surface C-S-H (Figure 12). The composition of C-S-H can vary within the same paste, especially in the case of blended cement pastes where different Ca/Si and Al/Si ratios can be observed depending on the measurement area. In Figure 12, it can be noticed that the Ca/Si ratio tends to decrease in the region on the smooth surface of the residual glass particles, while this ratio increases as the measurement point moves away from the center of the glass particles. Points 1 and 2 have Ca/ Si ratios of 2.00 and 1.57, respectively, within the range to be considered C-S-H. Points 3 to 6 have Ca/ Si ratios below 0.7, representing the regions where the glass did not react. Points 7 and 8 have Ca/Si ratios of 2.82 and 2.42, respectively, which are values above the commonly accepted theoretical values for C-S-H. However, the morphology indicates that it is C-S-H, possibly in a region with a high calcium content due to the presence of limestone filler in the



FIGURE 11. Scanning electron microscopy images of the pastes at 28 days (A) Reference paste; (B) Paste with 25% untreated glass (<45µm); (C) Paste with 25% treated glass (<25µm); (D) Paste with 25% treated glass (<45µm).



FIGURE 12. Point analysis by EDS showing the quantification of the elements Ca, Si, and Na in a paste sample containing treated GMPs.

cement (46). The dissolution of glass residues slowly introduces additional silica and sodium into the system over time. Silica reacts with portlandite to form more C-S-H, while sodium is partially incorporated into the C-S-H (57).

#### 4. CONCLUSIONS

- Although mortars made with treated particles have lower workability compared to untreated ones, which impacts mechanical resistance qualities. All glass particle sizes used provided the mechanical performance necessary to be considered pozzolanic materials.
- The use of pre-treatment with a calcium hydroxide solution at 80°C for 7 days enhances the pozzolanic activity of glass particles. Treated glass particles are coated with C-S-H and act as nucleation seeds for C-S-H, enhancing their pozzolanic activity. However, the pre-treatment induced the formation of clusters of glass particles, which negatively affected the particle filling effect.
- The smaller the size of the glass particles, the higher the consumption of calcium hydroxide. Additionally, the incorporation of treated particles consumed more calcium hydroxide than untreated particles of equivalent size. The increased consumption of calcium hydroxide indicates a greater pozzolanic reaction.
- Glass particles that are very small (<1µm) are not observed by SEM in hydrated pastes, likely because they are completely dissolved in the cementitious matrix. In pastes with treated particles, it was possible to observe changes in the interfacial transition zone (ITZ) between the cementitious matrix and the glass particle, as the matrix-glass ITZ was not as well-defined as in pastes with untreated particles.
- The studied composites can be applied in cement systems aiming to reduce the amount of portlandite, preventing issues such as efflorescence. Additionally, they can be useful for applications where there is a need to decrease the content of aluminates, which are reduced by substituting 25% of the cement with glass particles.

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#### Authorship contribution statement

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#### **Declaration of competing interest**

The authors of this article declare that they have no financial, professional or personal conflicts of in-terest that could have inappropriately influenced this work. Influence of chemical pretreatment on the pozzolanicity of recycled glass microparticles used as a substitute for Portland cement • 13

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