

Glass powder from non-returnable bottles: Pozzolanic additive to mortar

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ABSTRACT

The possibility of reusing solid waste in construction materials, seeking out to improve their properties, has been studied in order to minimize the environmental impacts caused by their incorrect disposal. In this sense, it is proposed to evaluate the addition of finely ground glass from non-returnable long neck bottles, in the mortar production. After waste collection and processing, chemical and physical characterizations were performed by using X-ray diffraction (XRD) and X-ray fluorescence (FRX) methods. Mechanical tests were performed at different rupture ages, after molding specimens with 3, 5, 10 and 15 wt% of glass powder addition in relation to cement mass, resulting in expressive gains of up to 97% of resistance to axial compression, in relation to the reference. The increase in mechanical strength is directly related to the increase in pozzolanic activity index, caused by the glass powder addition in the cementitious matrix. The pozzolanic activity index was determined by standardized test and thermal analysis (TG/DSC), after reactivity between the glass residue and calcium hydroxide (CH) produced in cement hydration. Micrographs performed by scanning electron microscopy (SEM), at advanced ages, indicate the increase of hydrated calcium silicates (C-S-H), a product responsible for mechanical resistance after CH consumption, characteristic of pozzolanic materials. As an additional study, it was evaluated the incorporation of smaller particle size glass grains in the mortars, verifying an increase of resistance of up to 33.7%, when compared to the same concentrations of larger particle size, at the same ages. The results show that it is technically and environmentally viable to use glass waste from long neck bottles as a pozzolanic additive in cementitious matrices.

Keywords Glass residue, Additive, Mortar, Pozzolan.

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1. INTRODUCTION

Glass, in its various forms and uses, is one of the most consumed materials by communities worldwide. In Brazil, for example, only in 2018 the glass production reached 1.3 million tons. Only 6.7 thousand tons (~0.5%) of this portion were collected for recycling, resulting in the generation of more than 1.2 million tons of waste [1]. Between 2018 and 2020, more than 52 thousand tons of glass were recovered, corresponding to 15% of the total amount generated, staying behind paper (54%) and plastic (21%) [2]. This fact is due to the low sale cost (per kg) in the market when compared to other materials that can be recycled. However, glass is the material that emits less CO₂ in the recovery process in comparison with other reused/recycled materials, and even with the virgin glass production [2]. Among the glass waste, there is some that cannot be recycled, as example the long neck - type “non-returnable” bottles.

In order to meet industrial interests in the competitiveness of packaging between glass and aluminum, the long neck bottle was created with the intention of removing some chemical components that added weight to the packaging. This resulted in a decrease in its strength, which implies the non-reuse of these packages by companies, that is, the material is treated as a solid waste after product consumption [3].

In another perspective, although responsible for a large national and global socioeconomic market share, the civil construction sector is, also, a major generator of solid waste [4,5]. This sector has been the subject of many research in the materials area, as it has great capacity to absorb the most varied types of waste, either those generated by the sector itself or those from other activities, such as, for example, non-returnable bottle glasses [6,7]. In general, many solid wastes can be incorporated into construction materials, such as concrete, mortar and cement production, as well as used for replacement or addition of aggregates [8].

The materials from civil construction originated from cementitious matrix are based on Portland cement as the main constituent, which is the most chemically reactive element in the matrix [9]. Portland cement, composed of clinker (a mixture of limestone rocks and clays) and additions, has binder properties in its composition [10]. When in contact with water, the cement powder hardens and remains in this state, unless it undergoes a mechanical interference [11].

In Brazil, the production of different types of cement is carried out by the addition of different materials, still in the grinding phase. In addition to clinker, such materials consist of other raw materials, such as blast-furnace slag and pozzolanic and carbonate materials, giving rise to CP II-E, CP II-Z and CP II-F, respectively [12]. Besides these raw materials, plaster is added to clinker in all cement classes in order to delay the hardening time. Without it, the cement would harden almost instantly when in contact with water [13].

After the contact between cement and water, an exchange of ionic species between the solid and the liquid phases immediately begins. As some clinker components have high solubility content, during hydration they rapidly increase

the aluminate, sulfate, and alkali (sodium, potassium, and calcium) concentration in the liquid phase [14]. With the dissolution of clinker anhydrous phases there is the formation of compounds with lower solubility, precipitating hydrates in the hardened cement phase [15].

After the dissolution of these phases, namely, tricalcium silicates (C_3S), dicalcium silicate (C_2S), known as alite and belite, respectively, tricalcium aluminate (C_3A) and tetracalcium aluminate iron (C_4AF), the hydration products that characterize the cement are generated [16].

Hydrated calcium silicate (C-S-H) is the hydration product responsible for the mechanical strength of cement and calcium hydroxide (CH), better known as portlandite, that is, its chemical durability [9]. Both hydration products can be generated by the alite and belite phases [17]. However, the alite phase hydration is faster than that of belite, which at early ages makes this phase the major responsible to produce C-S-H and CH [15,16].

Siliceous or silico-aluminous materials can be added to Portland cement, providing greater mechanical strength to the matrix, being then called pozzolanic materials [18]. The pozzolanic reaction occurs when these materials, in the presence of water, react with the CH produced by the cement hydration, forming additional C-S-H to the system [19]. Unlike the cement hydration reaction (fast), the pozzolanic activity has a slow reaction, and its effects are perceived in advanced ages of the cement or cement matrix [20,21]. The pozzolanic reaction progress is commonly measured by decreasing the concentration of free CH in the system [22,23].

In the last decades, the literature has shown many works reporting the incorporation of several residues into the cement, or the cement/residue substitution, which contributes to the pozzolanic activity in the cement matrix. In the case of glass powder (GP), it is important to note that the particle size has a direct influence on the possible alkali-silica reactions. If these particles are relatively larger than those of the cement, the result can be harmful to the mechanical performance and durability of the cement matrix [24]. Many works in the literature have shown this influence of GP particle size on the mechanical properties of the cementitious matrix and it is well registered that particle with sizes of the same order as those of cement promote pozzolanic activity in the matrix [25–30].

Taking this into consideration, this work makes a systematic evaluation of the GP pozzolanic activity of long neck-type bottles. The results show the feasibility of reusing this glass-type, a relatively common waste, as a pozzolanic additive to Portland cement matrix. In addition to the scientific contribution to the state of the art, the context of this work allows evaluating the use of glass waste, generated by the disposal of long neck bottles, directly in products applied to the civil construction sector.

2. EXPERIMENTAL

2.1. Glass processing and characterization

Long neck-type glass bottles underwent a grinding process divided into two stages. Initially, the bottles were crushed in a jaw mill (Restch). Then, the previously crushed glass was sprayed using a ball mill (SOLAB, model SL 34T) for 8 hours, with speed of 70 rpm. The container, with 10 liters capacity, was filled with, approximately, 70 volume %, comprising 1.5 kg by previously crushed glass waste, 1.2 kg by water, 1.5 kg by alumina with a 6 mm diameter and the same mass of spheres with 20 mm diameter. After this grinding process, the sprayed glass residue was dried in an oven at 100 °C for 8 hours.

The GP was passed through a set of sieves (Numbers 170 (90 μm), 270 (53 μm), 400 (38 μm) and bottom) using a vibrating sieve machine (Lucadema) for 1 hour. The particle size range of the crushed glass used for the mortar production was from 90 to 53 μm (retained on the Number 270 sieve, corresponding to 73% of the sieved powder). A set of samples was also prepared with the particle size between 53 and 38 μm (retained on the Number 400 sieve, corresponding to 13% of the sieved powder) in order to compare the mechanical strength. The rest of the sieved powder was not used in this work (10% retained on the Number 170 sieve and 4% retained in the bottom).

The GP was characterized by X-ray diffractometry (Shimadzu, XRD - 6000) with Cu-K α 1 ($\lambda = 1.5406 \text{ \AA}$) and Cu-K α 2 ($\lambda = 1.5444 \text{ \AA}$) radiations, voltage of 40 kV and current of 30 mA. The scanning was performed in a 2 θ range from 10° to 80°, using divergence and reception slits with 1° opening, in continuous mode, with step of 0.02 and scanning speed of 2°·min⁻¹. To obtain the composition and the chemical quantification, X-ray fluorescence analysis (Shimadzu, XRF-7000) was performed. This characterization was carried out in a qualitative - quantitative way, on samples also in powder form, using biaxially-oriented polyester substrates of poly(ethylene terephthalate) (boPET, Mylar®), with an analyzed area of approximately 80 mm².

2.2. Preparation and characterization of mortars

For the preparation of mortars, Portland cement was used (Votoran - CP II F - 32) (ABNT - NBR 16697, 2018 [31]; ASTM - C150/C150M, 2018 [32]). Cylindrical specimens measuring 10 cm in height and 5 cm in diameter were produced by adding GP concentrations (90 to 53 μm) in proportions of 0, 3, 5, 10 and 15 wt%, denominated V0, V3, V5, V10 and V15, respectively. A 0.48 water/cement factor was used. The tests were carried out in 7, 14, 28 and 91 curing days using 5 samples for each concentration.

For pozzolanic tests, two different methods were used. One of them complies with Brazilian standards (ABNT - NBR 12653, 2014 [33]; ABNT - NBR 5752, 2014 [34]) and states that the pozzolanic activity evaluation comes from the comparison of axial compression strength values between standard specimens and others containing pozzolanic material. Initially, the pozzolanic activity was verified

in 28-day curing age specimens, whose test evaluate a pilot sample compared to another containing a 25 wt% replacement of cement by the material to be evaluated for pozzolanic activity.

The materials for each mortar preparation to determinate strength activity index are shown in Table 1. Mortar A is the reference (0 wt% GP), while mortar B is the one with 25 wt% GP intended to replace cement.

Table 1. Materials quantity and consistency index obtained in mortars A and B.

Material	Weight (g)	
	Mortar A (0%)	Mortar B (25%)
Cement Portland	624	468
Pozzolanic Material*	-	156
Sand	1872	1872
Water	300	300
Consistency Index (mm)	194	191

*GP (90 to 53 μm)

It can be seen that the water amount used was the same in both mortars (A and B), maintaining the same factor (water/powder = 0.48). Additionally, there was no need to use a superplasticizer additive. The use of this type of additive is only necessary when the consistency index, calculated using standard (ABNT – NBR 12653, 2014 [33]), is greater than 10%. Therefore, it can be noted that the consistency index of mortar B did not differ by more than 10 mm from mortar A, being less than 10%. It is worth remembering that the consistency index was obtained according to Annex A of NBR 7215 (ABNT - NBR 7215, 2019 [35]). According to this standard, the index is established by the (Eq. 1), where I is the performance index (%) of cement at 28 days; f_{cB} is the average strength (MPa) at 28 days of the specimen containing cement add 25% of pozzolanic material (mortar B) and f_{cA} is the average strength (MPa) at 28 days of the specimen containing only cement (mortar B).

$$I_{cement} = \frac{f_{cB}}{f_{cA}} * 100 \quad (\text{Eq. 1})$$

The other method, proposed in the literature [28,36], uses thermal analysis (Differential Scanning Calorimetry-DSC and Thermo-gravimetry-TG, TA Instruments, SDT-Q600), instead of Brazilian standards. In this method, when the sample is subjected to heat treatment it is possible to evaluate the mass loss associated with the endothermic reaction (at a characteristic temperature) between water and calcium hydroxide (CH) in the cement matrix. During this case, CH is consumed by pozzolanic reaction to form hydration products [36]. The samples used for the thermal analysis were prepared separately from the cylindrical

specimens. Initially, a 20 g cement paste was prepared, maintaining the same water/cement ratio and the GP incorporation content. For the thermal analysis test, a 20 mg aliquot was removed from this paste, at the same curing ages as the specimens (7, 14 and 28 days). The scan was performed from 30 to 700 °C at a heating rate of 10 °C.min⁻¹ under a 100 mL.min⁻¹ flow of N₂ inert atmosphere. To evaluate the pozzolanic material reaction (GP) in relation to that of the lime fixation, from (Eq. 2) was used. In this equation, CH₀ is the calcium hydroxide (CH) initial amount before the endothermic peak, and CH_p is the calcium hydroxide (CH) final amount after the endothermic peak obtained in thermograms, i.e., the remaining CH [36]. It is worth remembering that similar methods using DSC/TG have been explored in the literature, although all of them evaluate the residual CH, after its consumption during the pozzolanic reaction in the system [37].

$$\text{Fixed CH (\%)} = \frac{CH_0 - CH_p}{CH_0} * 100 \quad (\text{Eq. 2})$$

The mortar composition used for the cylindrical specimens and the cement pastes used for thermal analysis can be seen in Table 2.

Table 2. Mortar and cement paste compositions.

Mortar composition (g)					
Material	V0	V3	V5	V10	V15
Cement	624	624	624	624	624
Water	300	309	315	330	345
GP	-	18.72	31.2	62.4	93.6
Coarse sand (2.4 to 1.2 mm)	468	468	468	468	468
Medium coarse sand (1.2 to 0.6 mm)	468	468	468	468	468
Medium thin sand (0.6 to 0.3 mm)	468	468	468	468	468
Thin sand (0.3 to 0.15 mm)	468	468	468	468	468
Cement paste composition for DSC (g)					
Material	V0	V3	V5	V10	V15
Cement	20	20	20	20	20
Water	9.6	9.88	10.08	10.56	11.04
GP	-	0.6	1	2	3

Note that the cement amount does not vary, evidencing that the GP acted as an additive to the matrix. The water percentage varies according to the increase in the GP percentage in order to maintain the 0.48 water/cement factor, as recommended by NBR 7215 (ABNT – NBR 7215, 2019 [35]).

After reaching the ages of 7, 14, 28 and 91 days in underwater cure, all specimens (V0, V3, V5, V10 and V15) were submitted to axial compressive and diametral tensile strength tests, according to Brazilian standards NBR 7215 [35]

and NBR 7222 [38], respectively (ABNT - NBR 7215, 2019 [35]; ABNT - NBR 7222, 2011 [38]). For both tests, a universal mechanical press (PAVITEST, model Contenco UMC) was used, with machine loading speed equivalent to $0.25 \text{ MPa}\cdot\text{s}^{-1}$, and with maximum load of 20 tons. Physical property tests, such as water absorption by immersion, void index (porosity) and specific mass, were performed only at the 28-day curing age for all probes with GP contents incorporated, according to the established Brazilian standards (ABNT - NBR 9778, 2009 [39]).

Morphological information and hydration products generated by the pozzolanic activity in the material at different wet curing ages were acquired by scanning electron microscopy (SEM). The images were obtained using ZEISS equipment model EVO LS15. The samples were metallized with gold by sputtering (Quorum).

3. RESULTS

3.1. Glass chemical composition

The glass chemical analysis from ground long neck bottles showed the predominance ($> 73\%$) of silicon oxide (SiO_2), followed by calcium oxide (CaO), with 13.84% , and sodium oxide (Na_2O), with just over 8% . The presence of aluminum oxide (Al_2O_3) in a concentration of 1.53% was also observed, besides other compounds that add to the sample weight with concentrations below 1% (Table 3). The composition of this glass is common to those called soda-lime, whose major components are silicon, calcium, and sodium oxides, yet possibly in different compositions depending on their production [24,40,41].

Table 3. Glass chemical composition.

Oxides	SiO_2	CaO	Na_2O	Al_2O_3	SO_3	MgO	K_2O	Fe_2O_3	TiO_2	SrO	BaO	Cr_2O_3
wt%	73.235	13.837	8.012	1.529	0.896	0.879	0.863	0.609	0.077	0.071	0.061	0.041

3.2. Glass structure

The GP XRD from long neck bottles did not indicate the presence of crystalline phases, showing only the characteristic band of the amorphous material around the Bragg position of $2\theta = 30^\circ$ (Figure 1). These results reveal that the high silica content present in the glass has an amorphous structure. According to the literature, materials with such characteristics, preferably associated with a large surface area, have the potential for pozzolanic activity [16,42,43].

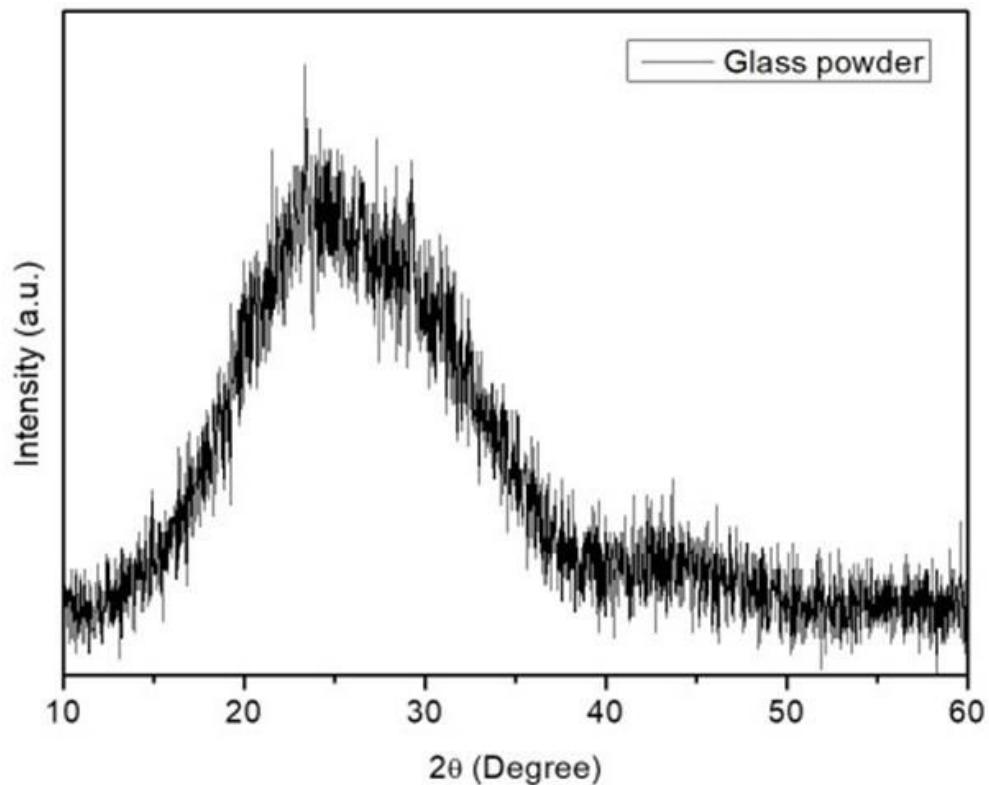


Figure 1. Diffractogram of the GP from long neck bottle.

3.3. GP pozzolanic activity

Figure 2 shows that the performance index of the Portland cement with 25 wt% GP (90 to 53 μm) at 28-day curing age resulted in 4.27 MPa higher than the standard sample (Mortar A). In other words, the replacement of the cement mass by 25% of GP provided an increase of 31.40% in the strength, reaching a value of 17.87 MPa, when compared to the strength value of the reference sample (13.60 MPa). Based on these results, the performance index with cement was obtained at 28 days, as established by NBR 5752 [34]. The performance index (I) (Eq. 1) obtained was 131.4%, showing a GP (90 to 53 μm) pozzolanic activity in compliance with NBR 12653 [33], which determines a performance index $\geq 90\%$ as physical requirements for pozzolanic activity.

In general, these values confirm the role of the GP (90 to 53 μm) reaction with the calcium hydroxide produced by the cement hydration. In cement, alite (C_3S) is a constituent phase that most contributes to this reaction at early ages [44]. As pozzolanic activity has a slow reaction, an even greater increase in the pozzolanic activity index of GP can be expected at older ages. In this case, these late reactions are resulting from the hydration of another cement constituent phase, called belite (C_2S) [36]. However, the NBR 12653 [33] already predicts pozzolanic activity at 28-day curing ages.

Some studies have shown that the use of distinct types of glass waste in different particle sizes (always inferior to 75 μm) results in strength activity index of cement ranging from 85 to 104% [25,29,45]. Therefore, the results presented herein, in addition to complementing the works in the literature, show that GP can

come from long neck bottles waste and, using particle size between 53 and 90 μm , it becomes a promising material to be used as a pozzolanic additive to the cement matrix.

The TG and DSC thermograms for all samples at curing ages of 7, 14 and 28 days (Figure 1 to 5 in Supplementary Material) showed mass losses associated with their respective endothermic peaks around 430 °C due to the consumption of free calcium hydroxide (CH) during the pozzolanic reaction, thus, reducing the free mass of portlandite (CH) [22,46]. In other words, they were caused by the reaction of water chemically combined with calcium hydroxide (CH), i.e., portlandite (Hua *et al.*, 2022). It is worth remembering that the free CH comes from alite (C_3S) and belite (C_2S) hydration products that constitute cement, which also produce hydrated calcium silicate (C-S-H) responsible for its hardening [22]. An increase in the DSC endothermic peak intensity in the reference paste (V0) (observed in Figure 6 in the Supplementary Material) with increasing the curing age is characteristic of common cements (without the addition of pozzolan) [16].

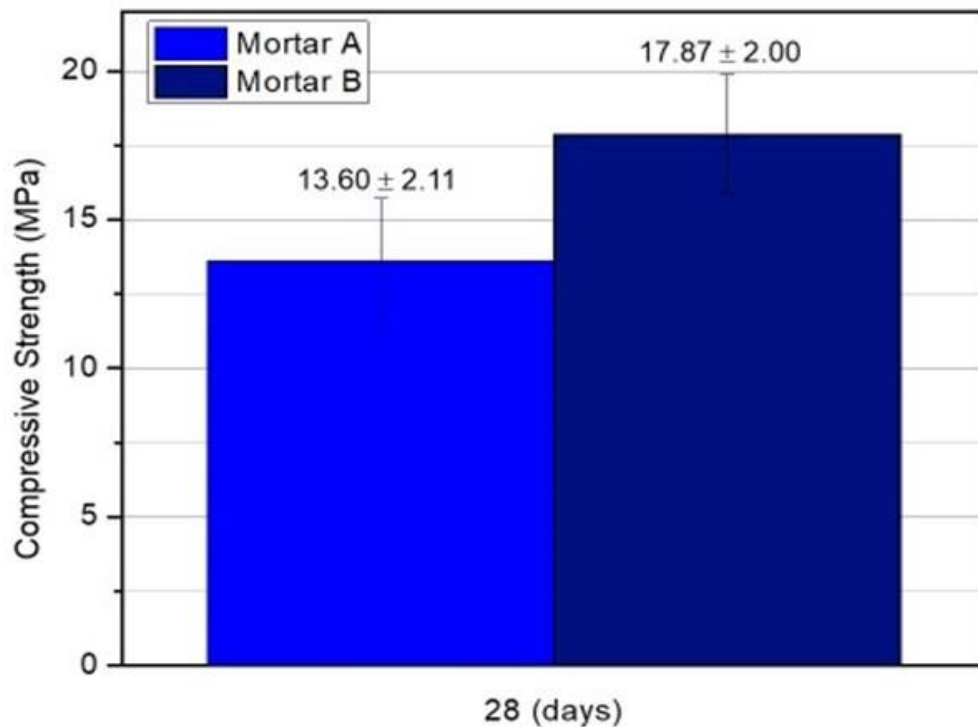


Figure 2. Average axial compression strength of mortars A (0%) and B (25%).

When GP residue is added to the cement paste, a decrease in the DSC peak intensity can be observed around 430 °C at advanced ages, as shown in the results summarized in Table 4 (graphs available in the Supplementary Material, Figure 8 to 10). This behavior is typical of the pozzolanic activity that occurs due to the reaction between amorphous silica (from the incorporated waste glass) and CH (from the alkaline medium), resulting mainly in additional C-S-H to the system [36,47]. Furthermore, the dissolution of finely ground GP in the alkaline medium is accompanied by the release of sodium ions, also present in the glass used [28]. As

the pozzolanic activity is a slow reaction, the peak decrease can be better observed at more advanced ages, such as at 28 days, although at 14 days the GP pozzolanic activity can already be identified in the samples. This behavior refers to the end of the hydration reaction and the beginning of the pozzolanic activity.

Table 4. Quantification of heat flow at the DSC peak of the samples (J/g) *.

Curing age (days)	Samples				
	V0	V3	V5	V10	V15
7	52.34	64.62	107.4	95.26	90.34
14	90.27	99.51	97.41	102.0	100.0
28	97.27	85.31	91.66	74.79	81.84

* Graphs available in the Supplementary Material (Figure 6 to 10).

To identify the CH percentage change in mass and mass loss due to hydrolysis at 430 °C, Table 5 presents the results, according to method analogous to the literature [28,36].

Table 5. Pozzolanic activity by thermogravimetry (TG): reaction between GP and calcium hydroxide.

Sample	Age (day)	CH ₀ (%)	CH _p (%)	CH percentage change in mass (%) Equation 1	Mass loss (TG) associated with the endothermic peak -430 °C (%)
V0	7	94.74	93.35	1.48	1.39
	14	87.06	84.85	2.54	2.22
	28	87.02	84.59	2.79	2.43
V3	7	94.21	92.5	1.81	1.71
	14	86.74	84.20	2.93	2.54
	28	74.12	71.79	3.14	2.33
V5	7	90.26	87.72	2.81	2.54
	14	87.46	84.94	2.88	2.52
	28	94.11	91.46	2.82	2.64
V10	7	^{V5} 90.79	88.38	2.65	2.41
	14	89.18	86.53	2.97	2.65
	28	87.76	85.42	2.67	2.34
V15	7	91.31	88.90	2.64	2.42
	14	89.54	87.04	2.79	2.51
	28	88.66	86.36	2.59	2.31

It is important to note that the water/cement factor was constant, and that no additional CH was introduced into the system; therefore, the initial (CH₀) and final (CH_p) quantities were the result of the cement hydration reaction in the analyzed samples. The calculations were then obtained directly from the mass loss events

observed in the TG curves of the samples associated with the DSC endothermic peak discussed above. To this end, the beginning (CH_0) and end (CH_p) of the mass loss event was identified with the aid of the derivative curve, thus marking each percentage. We can treat the mass percentage variation as inversely proportional to the lime fixation in the system, that is, the smaller the variation, the greater the lime fixation and the higher the pozzolanic activity index.

It was observed that the V3 sample presented a similar behavior to that of the reference sample (V0), showing a small increase in the percentage change in mass with increasing the curing age. Pastes that contained a greater amount of GP (V5, V10 and V15) presented greater lime fixation at older ages due to the fact that the pozzolanic activity reaction is slower than the cement hydration reaction. It can be concluded that glass does cause a pozzolanic reaction when incorporated in a cementitious system, due to amorphous silica present in the GP, which provides more sites for reactivity and increases the rate of hydration [43]. Results at short ages, e.g. 7 days, did not show the same trend as at advanced ages (14 and 28 days). This randomness is due to the parallel performance of the so-called pozzolanic effect and particle effect (filler). The filler effect produces fine particles that fill the empty space between the cement grains, modifying their granular packaging, which implies a change in the initial porosity of the paste. This effect positively or negatively modifies the water demand needed to maintain workability, given the particle size and the proportion of additions [48]. Both effects (pozzolanic and filler) accelerate the cement hydration, resulting in a higher number of hydration products [28,29,36].

3.4. Microstructure and morphology of mortars

The results observed for the pozzolanic activity of samples containing GP waste are directly related to their microstructures. Through SEM images, it was possible to observe the microstructures of the hydration products, as well as their changes with GP incorporation and curing age of the specimens.

The morphology and microstructure of the mortar samples with GP incorporation (90 to 53 μm) are observed in the SEM images displayed in [Figure 3](#). The images show the mortar hydration products observed at the 7, 14 and 28 day curing ages.

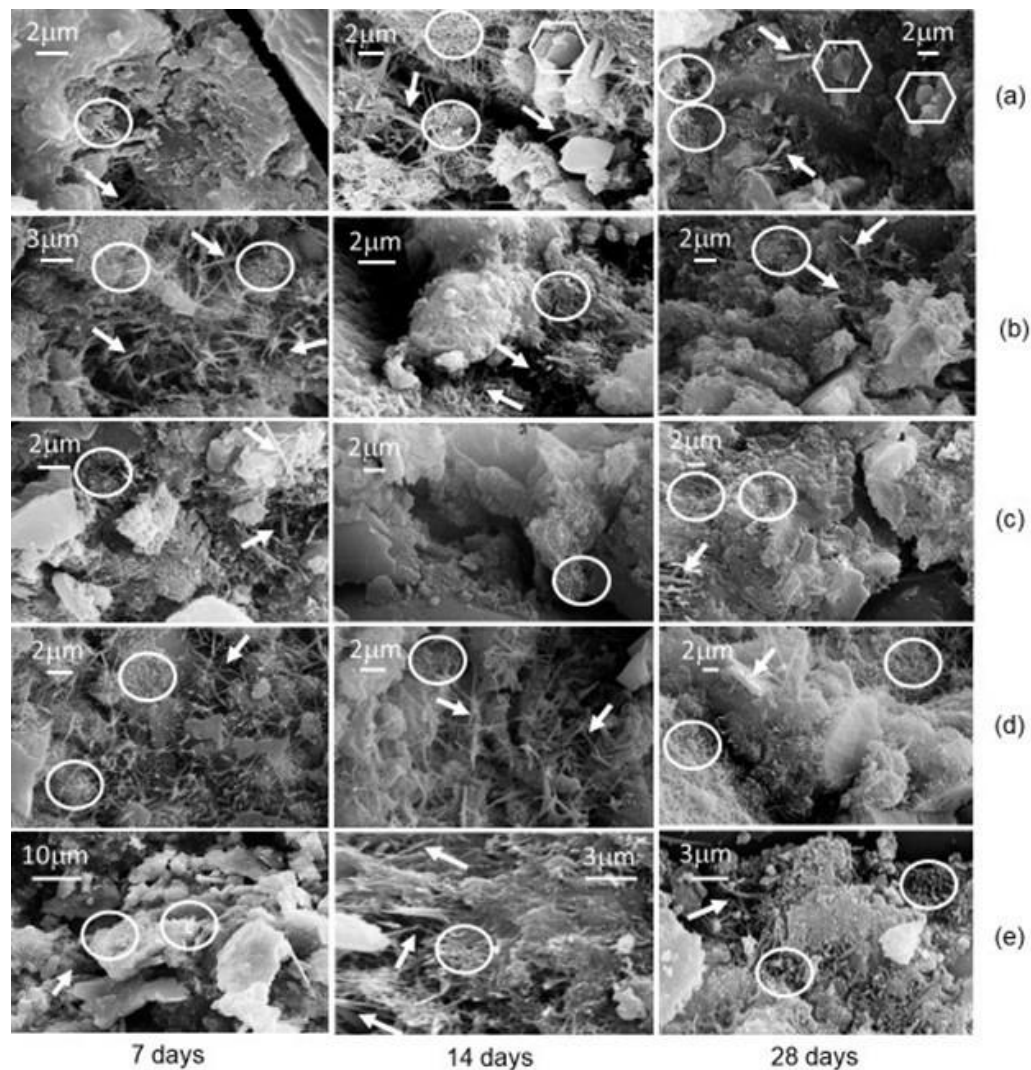


Figure 3. Mortar hydration products at curing ages of 7, 14 and 28 days: (a) V0; (b) V3; (c) V5; (d) V10; and (e) V15.

It can be seen for all samples and at all curing ages that morphologies related to C-S-H, indicated by fibrous regions (circles in Figure 3), as well as to Ettringite (i.e. hydrated calcium sulfoaluminate (C-A-S-H)) were verified [47,49]. It is demonstrated by rod-like particles (arrows in Figure 3), whose function is to bond and harden the cement [50,51]. Note that at all age, the standard sample (V0) revealed particles with hexagonal morphology (hexagons in Figure 3 (a)), indicating the presence of CH, which in turn decrease to the samples containing incorporated GP (90 to 53 μm) [47]. These morphological observations are in agreement with the pozzolanic activity results, that is, the C-S-H that appears predominantly in samples containing GP is resulting from the reaction between the amorphous silica of the incorporated glass and the CH. As previously mentioned, at older ages the hydration reactions decrease, giving rise to the pozzolanic activity. It is important to highlight that samples containing incorporated GP at 28 days showed a predominance of C-S-H (fibrous particles, Figure 3 (b) to (e)). This effect was noticed in all GP concentrations inserted into the cement matrix, whose

presence was greater with the increase of the residue concentration.

If we observe the morphology at 91 days, for example, a considerable increase in these regions can be seen (Figure 4). A significant decrease in the microstructure pores for all samples can also be noted. For the samples with incorporated GP (90 to 53 μm) it was possible to observe a predominance of denser regions with fibrous morphology (circles in Figure 4), indicating the presence of C-S-H and few particles corresponding to Ettringite (C-A-S-H), whose morphology is similar to small rods (arrows in Figure 4) [47]. This indicates that both the increase in the percentage of GP incorporated into the mortar and the curing age of 91 days promoted the pozzolanic activity. There was, therefore, a consumption of CH, resulting in additional C-S-H, which in turn filled the capillary voids [16], corroborating the results previously discussed.

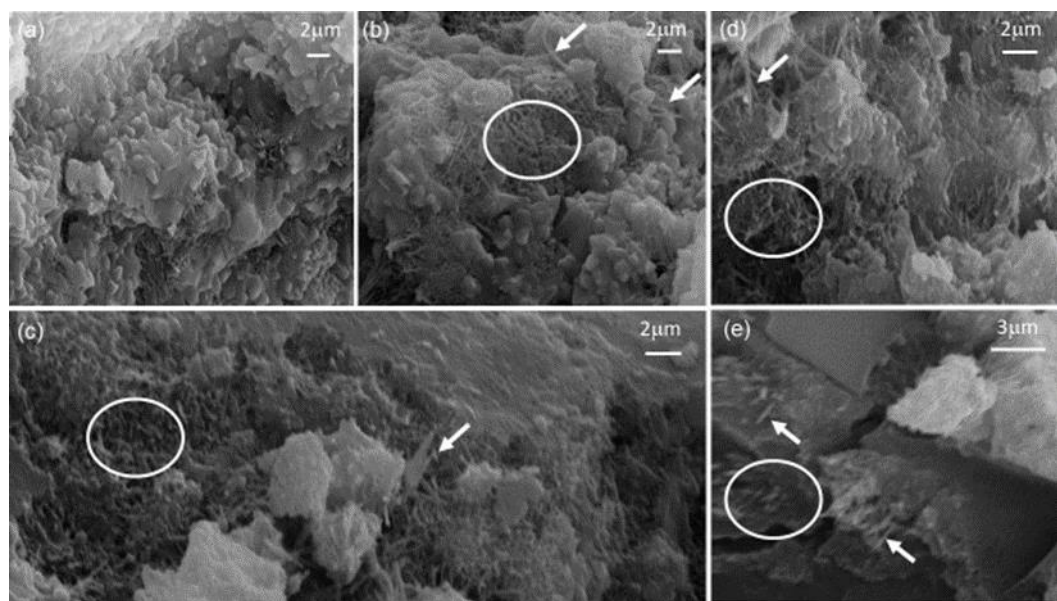


Figure 4. Mortar hydration products at a curing age of 91 days: (a) V0; (b) V3; (c) V5; (d) V10; and (e) V15.

3.5. Axial compression and diametral tensile strength

Figure 5 shows the results obtained for the axial compression strength of the specimens. All values were obtained by an arithmetic mean of the compression strength results carried out on the mortar specimens V0, V3, V5, V10 and V15 at curing ages of 7, 14, 28 and 91 days.

These results showed a strength increase of the mortars with GP incorporation (90 to 53 μm) in all concentrations for curing ages of 28 and 91 days in relation to the standard mortar (V0). At 7 and 14 days, it was possible to observe some fluctuations in the results according to the incorporated waste percentage; however, they were generally higher values than those of the standard mortar.

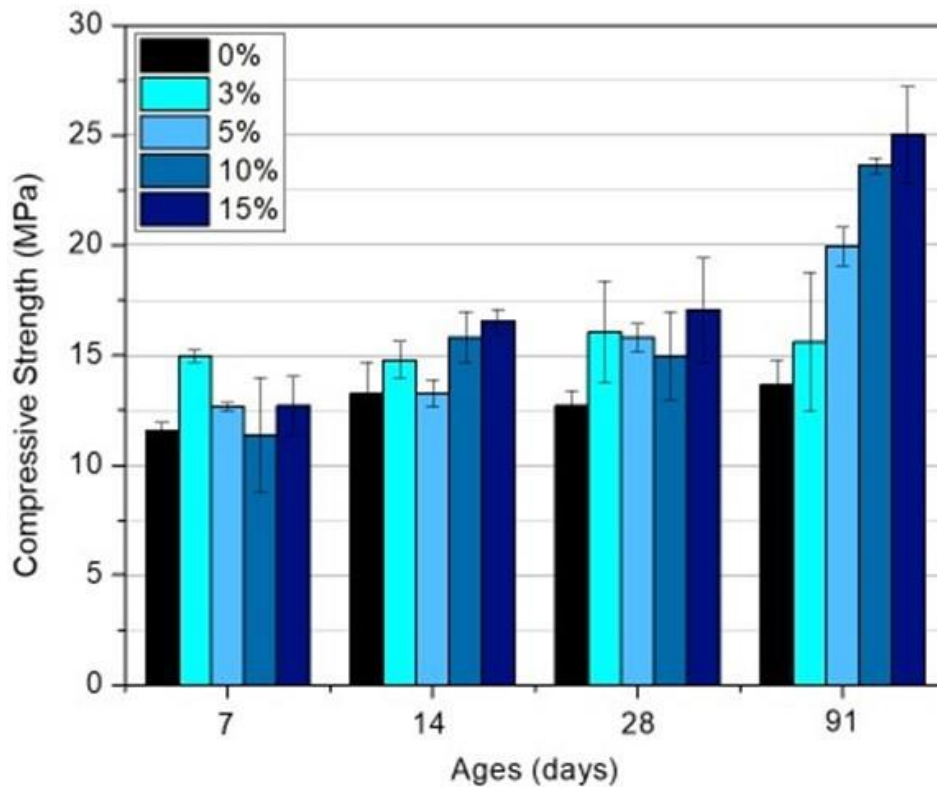


Figure 5. Axial compression strength of mortar specimens at different ages.

The greatest increase in compressive strength occurred in the specimen with the highest GP content. After 28 curing days, mortar V15 already reached strength superior to 34% in relation to the reference (V0). At 91 days, V15 showed an expressive increase of approximately 97%, that is, almost twice as much as the mortar V0 at the same age. In the work by [52], the glass powder was incorporated as a substitute for cement in proportions of 25, 50 and 75 wt%, showing that the compressive strength results (at 7 and 28 days) were always lower than the standard values. Patel and co-authors [29] also replaced cement with up to 20 wt% GP in two different sizes (75 and 63 mm).

Their best results revealed that for the 20 w% replacement, the compression strength rate decreased by only 6% compared to the standard trace for samples with 75 mm GP, whereas for samples with 63 mm GP the results were equivalent to the standard sample. Shi and co-authors [25] used GP with particle size distribution of the same order as the cement particles (< 30 μm) and achieved an increase of 30% in relation to the reference sample, but only at the curing temperature of 65 °C at 28 days. At lower curing temperatures (23 and 35 °C), the values had a minor percentage increase (11 and 15%, respectively) at the same age.

This shows that in contrast to replacing cement with GP waste, such incorporation results in higher strength values, as shown in this work. For comparison purposes, the same axial compressive strength test was performed on five sets of specimens with 53 to 38 μm GP (retained on a Number 400 mesh sieve) at a curing age of 28 days, as shown in Figure 6.

These results clearly showed the increase in the compressive strength of

mortars V3, V5, V10 and V15 in relation to the reference, V0. As the concentration of GP (53 to 38 μm) in the specimens increased, the strength also increased, except for V10. However, it could be observed that the largest standard deviation also occurred in mortar V10, implying that this result is likely to fit the linearity pattern of increased strength. It was also noted that all samples with incorporated GP sized $< 53 \mu\text{m}$ presented higher values than those with GP sized $> 53 \mu\text{m}$, as predicted in the literature [24].

The compressive strength results of sample V15 (GP 53 to 38 μm) showed a significant increase of 79.39% in relation to the reference mortar, V0. The mortars V3, V5 and V10 from the same set reached a compressive strength increase of 32.89%, 69.39% and 65.85%, respectively, in relation to V0. By comparing the best results (between V15 at 28 days with GP $< 53 \mu\text{m}$ and V15 at 28 days with GP $> 53 \mu\text{m}$), it was possible to observe an increase of 45%.

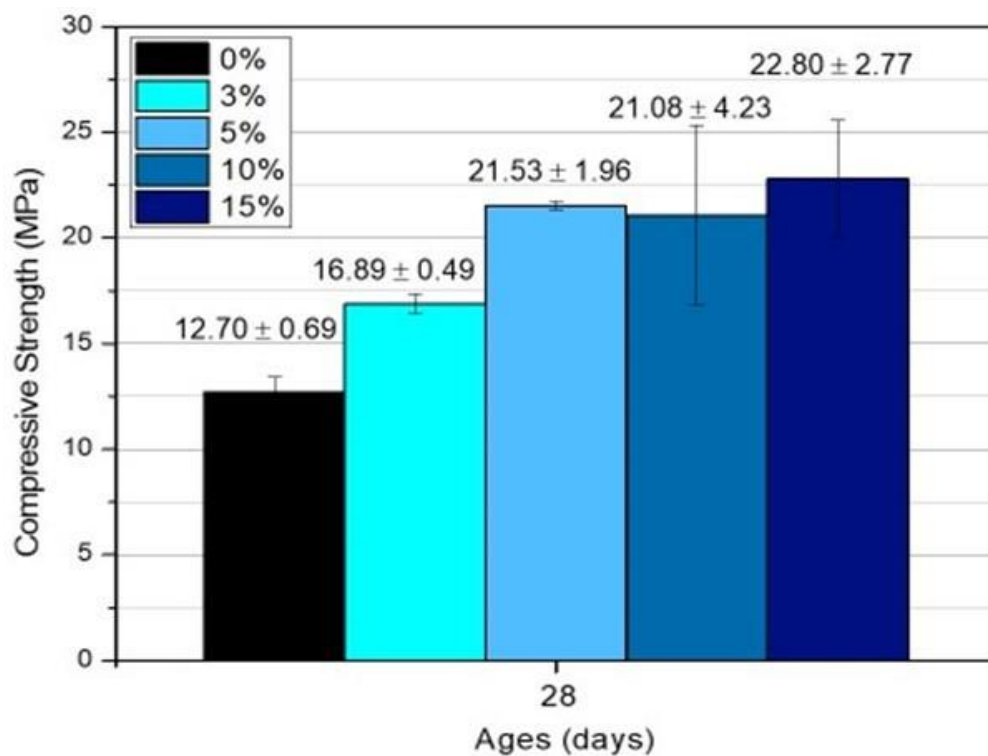


Figure 6. Axial compression strength of mortar specimens at 28 days (53 to 38 μm GP).

These results are in agreement with some works in the literature. Cordeiro et al [53] evaluated the relationship between particle size, specific surface area and pozzolanic activity, and concluded that the smaller the particle size and the larger the surface area, the greater the reactivity of this material. The relationship between pozzolanic activity and particle size had already been confirmed by Massazza [22] and reviewed by Jiang et al [24].

The results presented together with those shown in the literature indicate the high pozzolanicity index of the GP residue when added to the cement matrix,

as well as the positive influence of the particle size decrease in the pozzolanic activity, verified by the axial compressive strength tests shown here [54–56].

Although the cement matrix constituents are designed to resist compression strength, traction tension cannot be discarded, since concrete and mortar cracking is usually the result of a tensile strength caused by restricted retraction [16].

In this context, the diametral tensile strength values of the specimens V0, V3, V5, V10 and V15 at curing ages of 7, 14, 28 and 91 days were also determined. The values, obtained by an arithmetic mean, are shown in Figure 7.

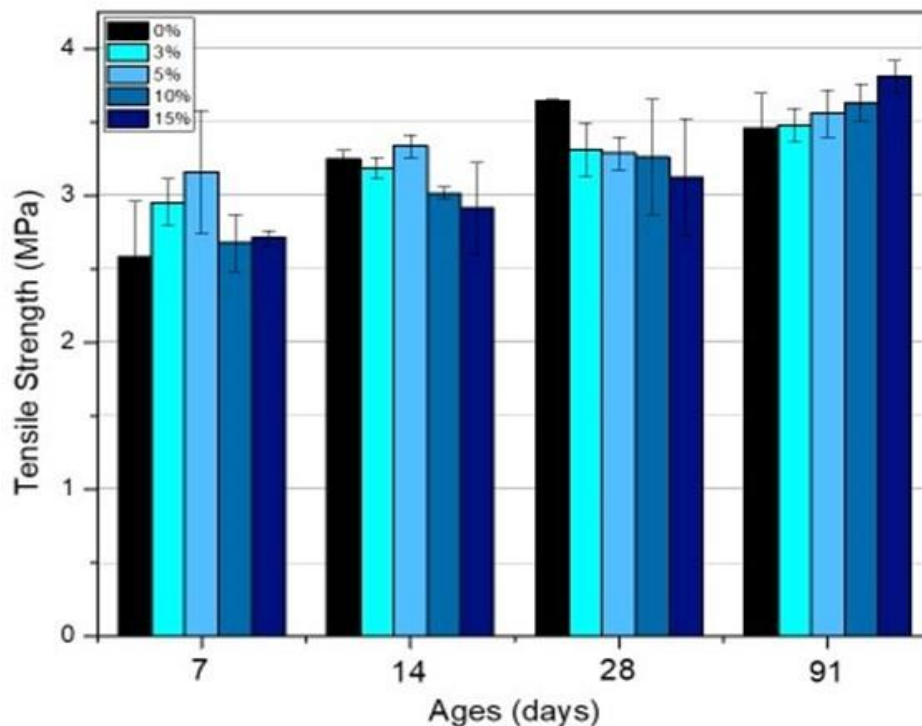


Figure 7. Tensile strength by diametral compression of mortar specimens at different ages.

A random increase in the initial strength values could be noted at a curing age of 7 days for all concentrations of GP in relation to the reference mortar (V0). At 14 and 28 days, a slight decrease in the strength values was observed as the GP concentration increased (90 to 53 μm).

However, at 91 days the specimens with GP incorporation achieved better results of tensile strength by compression than mortar V0, presenting increasing values as a function of GP concentration. The samples V3, V5, V10 and V15 had an increase of 5.06%, 8.37%, 11.25% and 21.87%, respectively, in relation to V0.

The obtained values regarding tensile strength by compression and the increase in the tensile strength at older ages with GP incorporation into the mortar are confirmed by other works in the literature [57,58].

3.6. Water absorption, void index, and specific mass

The results concerning the physical properties of water absorption by immersion,

void index (porosity) and specific mass after 28 days of underwater cure are given in Table 6.

Table 6. Test results regarding physical properties of samples at 28 days of underwater.

Sample	Absorption (%)	Standard deviation	Void index (%)	Standard deviation
V0	13.38	±0.025	26.21	±0.088
V3	13.96	±0.116	27.31	±0.179
V5	14.15	±0.117	27.86	±0.063
V10	13.88	±0.097	27.10	±0.074
V15	14.21	±0.111	27.55	±0.273

Specific mass (g.cm ⁻³)						
Sample	Dry	Standard deviation	Saturated	Standard deviation	Real	Standard deviation
V0	1.96	±0.005	2.22	±0.002	2.65	±0.0006
V3	1.96	±0.003	2.23	±0.002	2.69	±0.002
V5	1.96	±0.002	2.27	±0.0002	2.71	±0.004
V10	1.95	±0.002	2.22	±0.002	2.67	±0.005
V15	1.94	±0.002	2.22	±0.0004	2.68	±0.006

It can be observed that the water absorption, void index (porosity) and specific mass results presented by mortars with GP (90 to 53 μm) were similar to those of the reference mortar (V0). The absorption by immersion was, around, 13 - 14%, the void index was, approximately, 26 - 28%, the dry specific mass was 1.94 - 1.96 g.cm^{-3} , the saturated specific mass was 2.21 - 2.27 g.cm^{-3} and the real specific mass was, about, 2.65 - 2.71 g.cm^{-3} .

Despite the reduction in pores being characteristic of pozzolanic activity, this effect was not noticed at 28 days. However, as already mentioned and observed in other tests, the pozzolanic reaction has a slow action and can then cause a pore refinement of the structure, increasing the impermeability of the system at older ages [16], as observed in the SEM images of samples at 91 days.

The values regarding water absorption by immersion, void index and specific mass of mortars at a curing age of 28 days obtained here corroborate the results found in the Brazilian literature [59]. In summary, the physical properties of mortars after GP incorporation (90 to 53 μm) at concentrations of 3, 5, 10 and 15 wt% did not undergo significant changes when compared to the reference mortar.

4. CONCLUSIONS

The incorporation of GP from disposable long neck bottles into the cement matrix showed satisfactory and relevant results. For the particle size conditions used, it was shown that the pozzolanic activity increases according to the GP waste percentage incorporated into the cement matrix, which consequently reflects on the

material properties. Our results showed that, at advanced ages (> 28 days), the mechanical strength of the material increases considerably (97% increase in strength at 91 days for samples containing 15% glass). In addition, we could note an important influence of particle size on these properties: the smaller the GP particle size, the greater the reactive surface area, and consequently the greater and better its pozzolanic response. In this case, an increase of 79% in mortar strength was observed at 28 days of curing.

It can also be concluded that glass, under the conditions presented here, has technical feasibility to be used as a pozzolanic additive by the cement industry. In general, the literature shows works that report the replacement of cement by a material with pozzolanic activity, which results in a significant savings in cement. On the other hand, the results presented in this work show that the glass addition to the matrix provides a considerable improvement in the mechanical property of the mortar, reaching double when compared to a standard sample at advanced curing ages. Thus, based on these studies, precedents are opened for the expansion of application and use of mortar, since its resistance can be predicted according to the content and particles size of glass powder incorporated. These results also suggest a viable alternative for reducing the environmental impacts caused by thousands of tons of non-returnable bottles discarded in the environment.

5. AUTHOR INFORMATION

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6. SUPPLEMENTARY MATERIAL

TG and DSC thermograms for V0, V3, V5, V10 and V15 samples at different curing age.

The [Figure S1 to S5](#) show TG and DSC thermograms for all samples at curing ages of 7, 14 and 28 days. These thermograms show the mass losses region associated with their respective endothermic peaks around 430 °C.

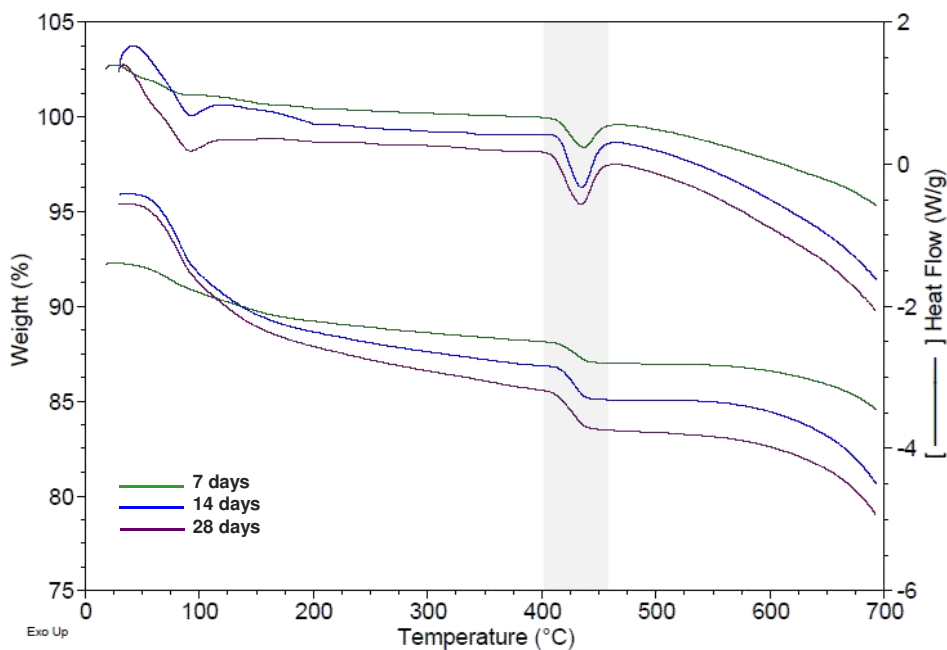


Figure S1. TG and DSC thermograms for V0 sample.

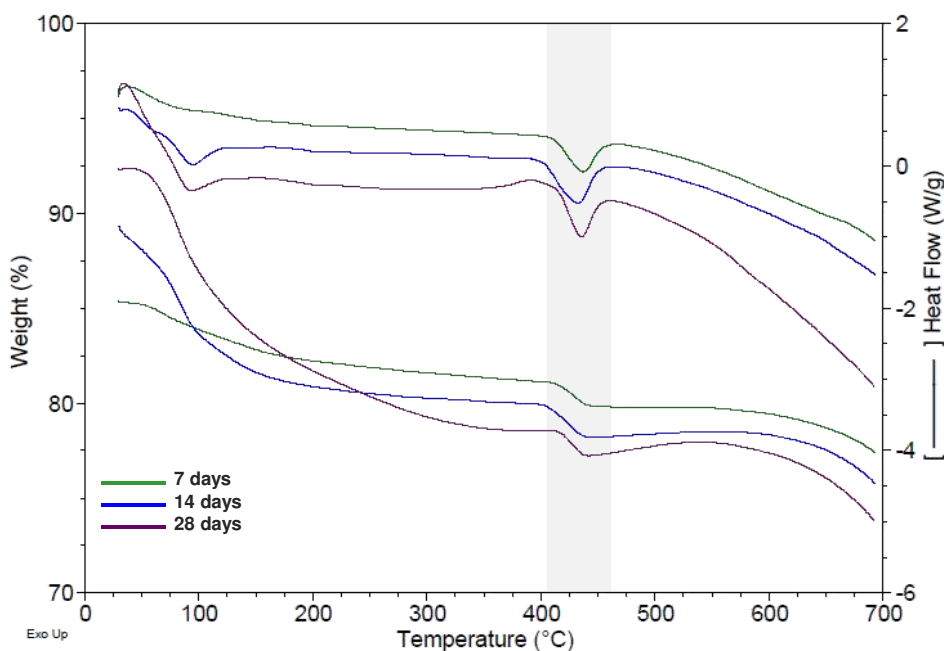


Figure S2. TG and DSC thermograms for V3 sample.

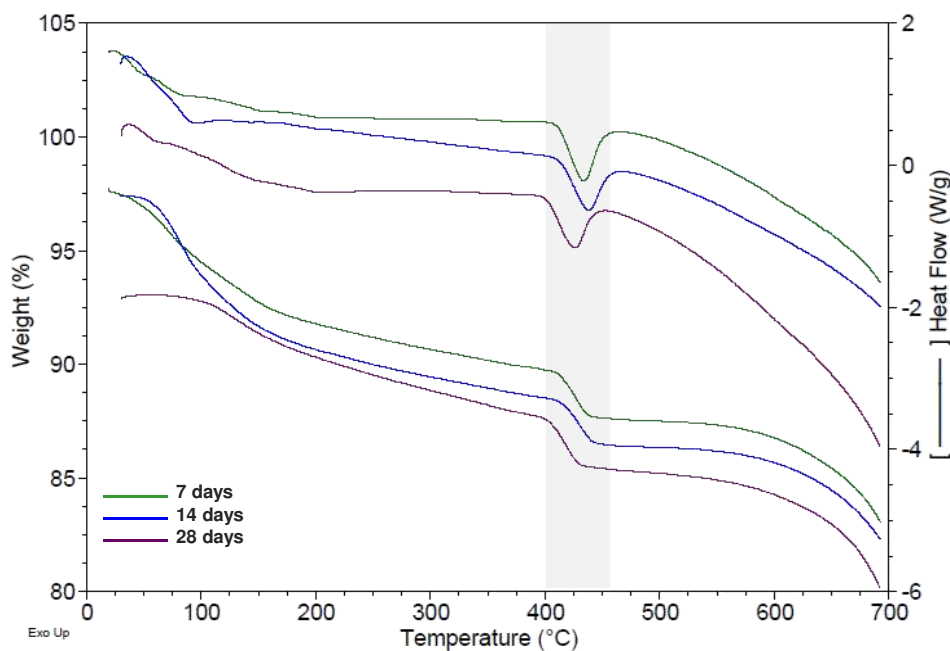


Figure S3. TG and DSC thermograms for V5 sample.

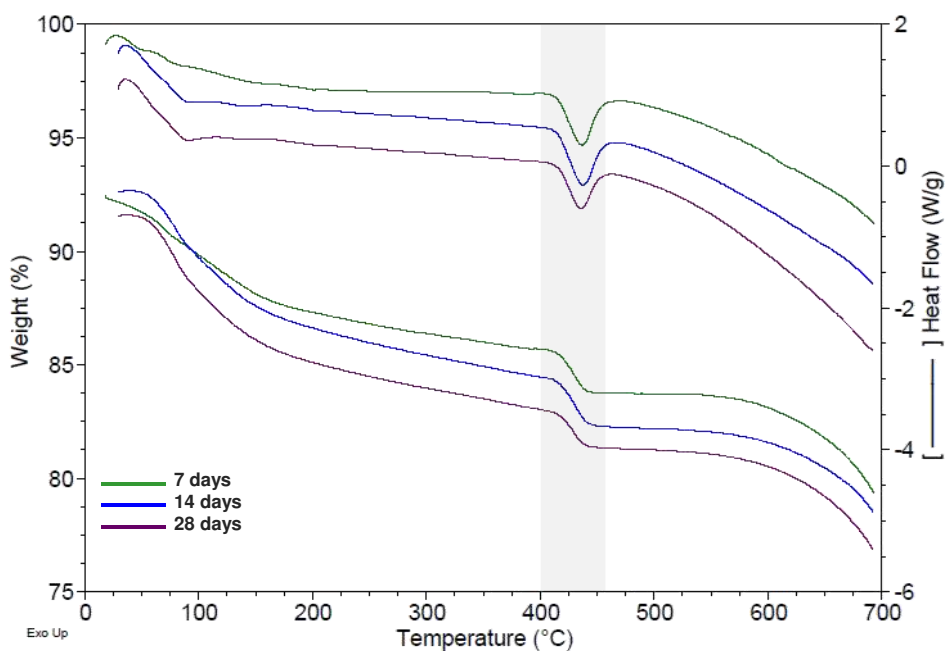


Figure S4. TG and DSC thermograms for V10 sample.

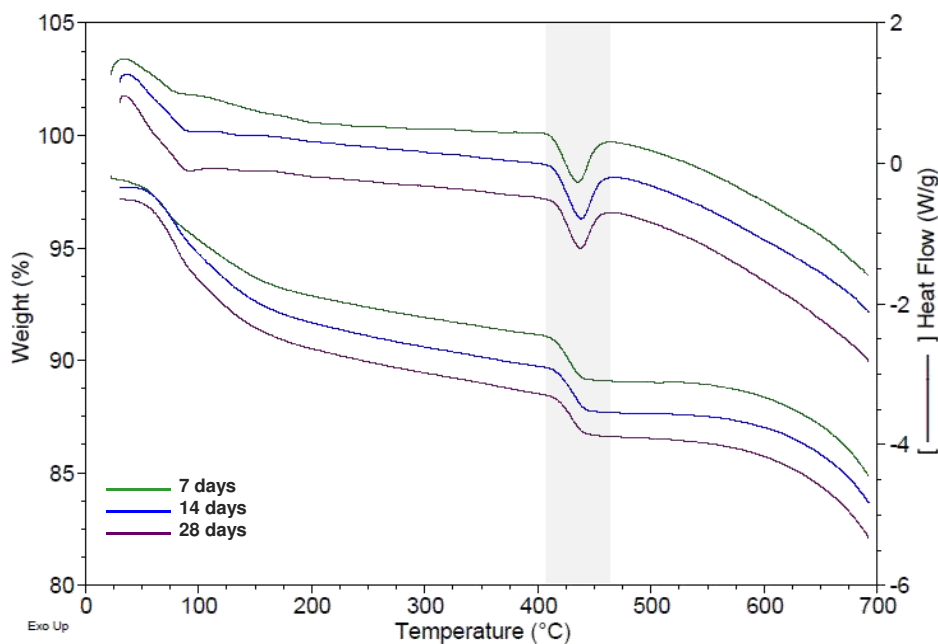


Figure S5. TG and DSC thermograms for V15 sample.

Figure S6 to S10 show the change of the DSC endothermic peak for all samples at curing ages of 7, 14 and 28 days. Note that the peak variation was determined in joule/g, that is, the amount of energy per second, per gram of the compound consumed during the reaction.

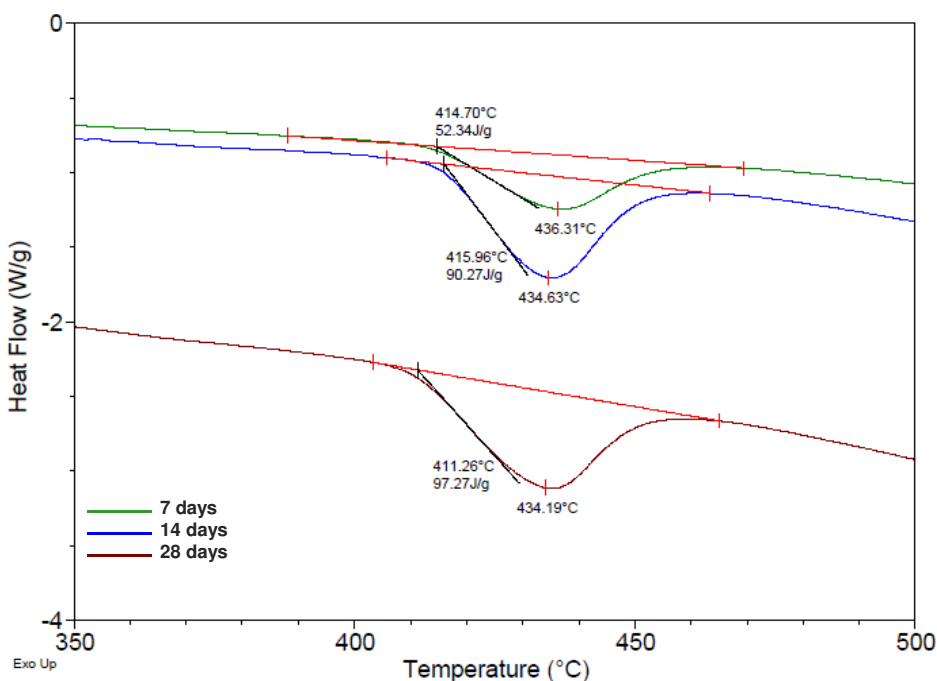


Figure S6. DSC endothermic peak intensity for V0 sample.

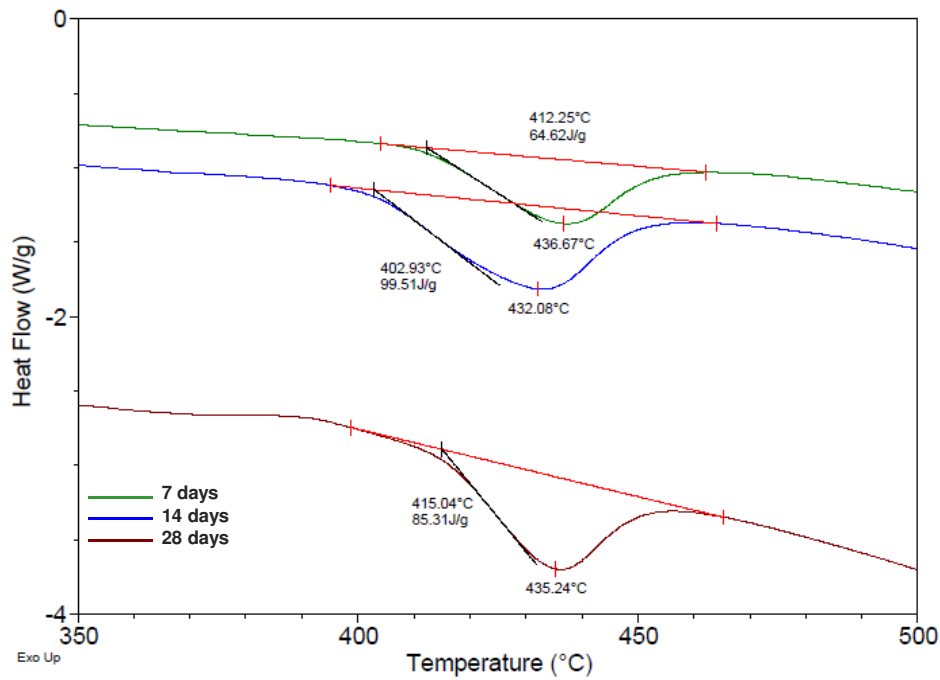


Figure S7. DSC endothermic peak intensity for V3 sample.

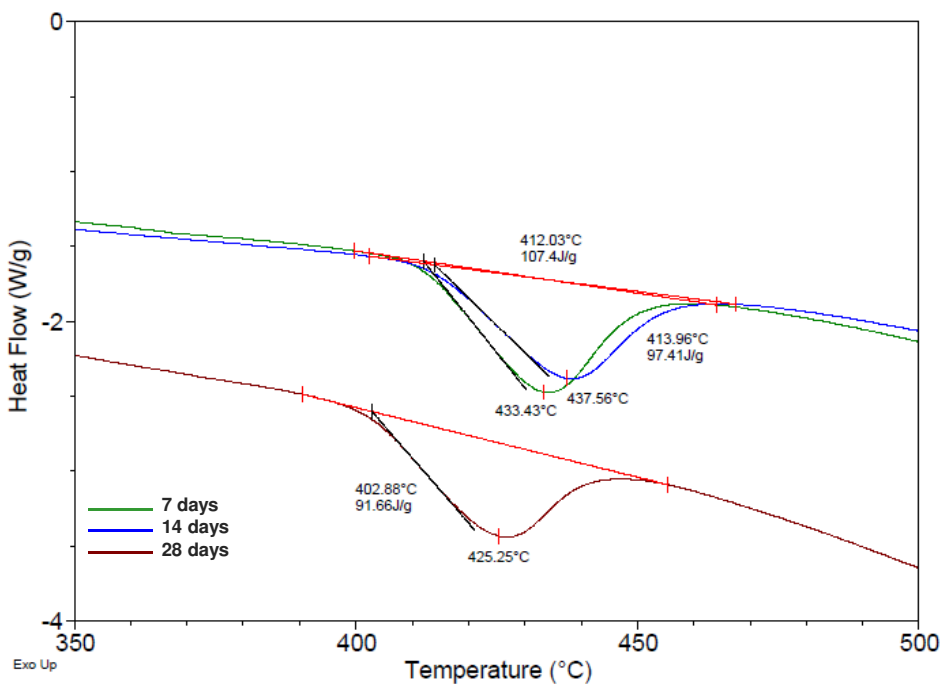


Figure S8. DSC endothermic peak intensity for V5 sample.

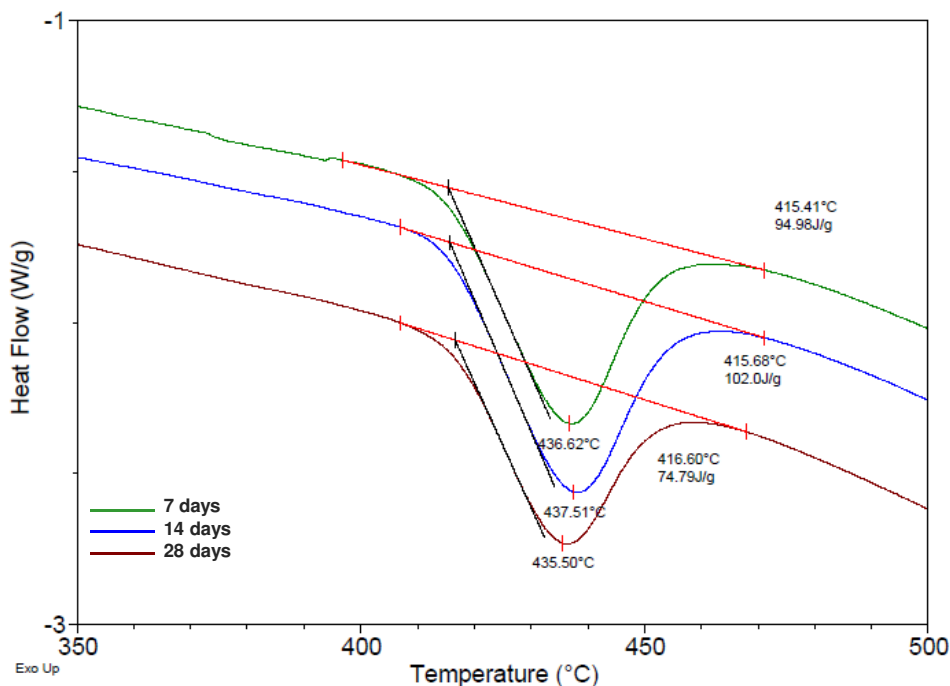


Figure S9. DSC endothermic peak intensity for V10 sample.

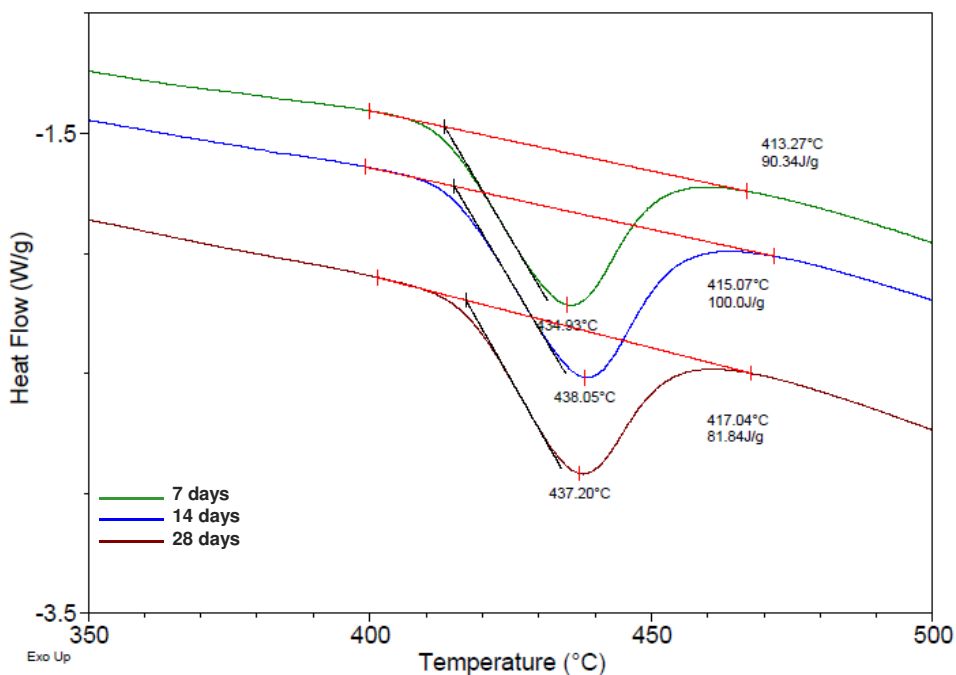


Figure S10. DSC endothermic peak intensity for V15 sample.

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