Performance of concrete with the incorporation of waste from the process of stoning and polishing of glass as partial replacement of cement

Desempenho de concretos com a incorporação de resíduo do processo de lapidação e polimento do vidro como substituto parcial ao cimento

Abstract

The incorporation of waste glass as a partial replacement for cement in concrete can provide an alternative destination for the waste, reduce the consumption of cement (minimizing CO₂ emissions and consumption of natural resources), and improve the concrete performance. Thus, this research evaluated the performance of concrete incorporating waste glass sludge (GS), resulting from the process of stoning and polishing of soda-lime flat glass, as a supplementary cementing material. Mechanical strength and durability properties were assessed through compressive strength, alkali-silica reactivity, electrical resistivity and chloride permeability, diffusivity and migration tests. Mixtures containing metakaolin (ME) were also evaluated. The results indicated that the use of the waste ground to an adequate size can replace up to 20% of cement. At this content, it caused a reduction of chloride penetration of over 80%, reduced ASR and conserved compressive strength. The combination of waste with metakaolin replacing 20% of cement also improved all the concrete properties, increasing the compressive strength up to 12% at 28 days.

Keywords: waste glass. pozzolan. metakaolin. chloride. alkali-aggregate reaction.

Resumo

A incorporação de resíduos de vidro em concretos como substitutos parciais ao cimento pode proporcionar um destino alternativo aos resíduos, reduzir o consumo de cimento (minimizando as emissões de CO₂ e o consumo de recursos naturais) e melhorar o desempenho do concreto. Assim, esta pesquisa avaliou o desempenho de concretos com a incorporação de lama de resíduo de vidro (GS), resultante do processo de lapidação e polimento de vidros planos sodo-cálcicos, como material cimentício suplementar. As propriedades de resistência mecânica e de durabilidade foram avaliadas por meio de testes de resistência à compressão, reatividade álcali-silicato, resistividade elétrica e permeabilidade aos íons cloreto por meio de processos de difusividade e migração de cloreto. Misturas contendo metakaolin (ME) também foram avaliadas. Os resultados indicaram que o uso do resíduo de vidro moído, adotando-se dimensões de partículas adequadas, pode substituir até 20% do cimento. Proporcionando assim, uma redução na penetração de cloreto acima de 80%, reduzindo a ASR e conservando a resistência à compressão. A combinação de resíduos de vidro com metakaolin, substituindo 20% do cimento, também melhorou todas as propriedades do concreto, aumentando a resistência à compressão em até 12% em 28 dias.

Palavras-chave: resíduo de vidro, pozolana, metakaolin, cloreto, reação álcali-agregado.
1. Introduction

The increase of natural resources and energy consumption correlates with the increase of population. This has generated concerns regarding the finitude of resources, waste generation and gaseous emissions. Alternative technologies and waste reutilization emerge as possible solutions to these issues [1-4], the construction sector being of major importance due to its high consumption of energy and resources and waste generation. Portland cement used in concrete production has clinker as its main component, which is produced by burning limestone with other ingredients at high temperatures, consuming natural resources and emitting CO₂. In the chemical reaction to dissociate 1 ton of limestone, 440 kg of CO₂ is emitted, and only 560 kg goes into the clinker composition [5].

According to Shi and Zheng [6], among the urban solid wastes, glass can be considered the most suitable as a cement replacement due to its physical properties and chemical composition. Waste glass generation worldwide has not been precisely quantified, mainly due to the lack of data in several countries, such as those situated in the Middle East. Jani and Hogland [7] state that the world production of glass in 2007 was about 89.4 million tons, and it is expected to rise due to industrialization and improvement of the quality of life. Saito and Shukuya [8] estimate that for each kg of float glass produced, 1.73 kg of raw materials and 0.15 m³ of water are used. Additionally, due to the need for elevated temperatures (up to 1600 ºC) to produce the glass, they estimate 16.9 MJ of waste heat for each kg of glass sheets.

Several studies indicate that the use of glass powder (or waste glass) in concrete production can improve the mechanical and durability properties. Regarding the mechanical properties, an increase in concrete compressive strength after 56 days is noted when replacing 20% of cement by glass powder with particle sizes smaller than 20 µm [9-11]. Using particle sizes between 75 and 100 µm, a strength loss is verified at the same replacement level [12-13]. For durability, an improvement in chloride penetration resistance is reported, by means of chloride migration tests, for the same replacement level [11-16]. It noted that the pozzolanic activity of the glass powder increases with grinding and that the ideal particle size should be smaller than 40 µm for use as a partial replacement for cement [17]. Nevertheless, there is discussion regarding the use of materials with alkalis content above the normative limits for concrete production, as the alkalis can react with the siliceous compounds present in the reactive aggregates, causing deleterious expansive mechanisms in the hardened concrete [18]. Studies point out that the use of supplementary cementitious materials (SCM) can help reduce this mechanism called the alkali-silica reaction (ASR). The use of SCM can help to reduce the calcium hydroxide content in the pores solution by causing pozzolanic activity [11-12].

Research has also revealed that the particles size influences the occurrence of these reactions [12, 19]. There is little literature regarding the analysis of chloride penetration by different transport mechanisms and the ASR assessment of concrete mixtures with waste glass combined with metakaolin. Authors have mainly assessed the chloride penetration resistance by migration tests (rapid migration test - RMT) [11-20] and no diffusion by immersion tests have been performed. Thus, an evaluation of chloride resistance by different tests is regarded as relevant, due to the discussion related to the tests procedures and the diffusion coefficients obtained from chloride diffusion tests (bulk diffusion test - BDT) and migration tests (RMT) [21-23]. Additionally, there is a lack of studies concerning the durability of concrete incorporating wastes from the process of stoning and polishing soda-lime flat glass (waste glass sludge). A study by Kim et al. [24] evaluated the use of glass stoning waste in concrete, aiming at improving the durability properties in freezing and thawing cycles. Improvements were noted in the compressive strength and chloride penetration resistance of mixtures combining 10% glass powder and 10% fly ash compared to those with only fly ash as SCM.

Regarding the use of glass sludge waste in concrete production, the literature is even scarcer. Pignaton [25] assessed the properties of concrete containing waste from the process of stoning of glass (without subsequent grinding) and noticed a reduction in compressive strength with the increase of partial cement replacement by waste. Furthermore, Lee et al. [26] replaced 20% of cement with the stoning glass waste and verified a decrease in the compressive strength of concrete at 28 days, while noticing an increase at 91 days. Thus, due to its fine particle size, waste glass is considered as a viable option for incorporation into concrete, with little or no further processing (grinding). Therefore, this study aims to contribute to the studies related to the performance of concrete incorporating the waste generated by the stoning and polishing of soda-lime flat glass as a cement replacement, for the mitigation of environmental impact. The influence of further grinding of the waste prior to cement replacement was assessed. Mechanical and durability properties (chloride penetration by different transport mechanisms – migration and diffusion; ASR) were evaluated. Furthermore, metakaolin in isolation and combined with waste glass sludge was used for comparative purposes.

2. Materials and experimental program

2.1 Materials

High early-strength Portland cement, CPV-ARI (equivalent to CEM I 52.5 R) was used in this study. As high mineral additions content can influence test results, this cement was used as it is the commercially available cement in Brazil with the lowest percentage of additions (up to 5% limestone powder). Natural quartz sand and granitic coarse aggregate were used as aggregates in the mixtures. The coarse aggregate had maximum grain size of 19 mm, true density of 2.77 g/cm³, bulk density of 1.45 g/cm³ and water absorption of 0.73%. The fine aggregate had maximum grain size of 2.4 mm, fineness modulus of 1.92 and true density of 2.57 g/cm³. The physical and chemical characterization of cement, metakaolin (ME), glass sludge without grinding (GS1) and ground glass sludge (GS2) are presented in Table 1. Grinding of the waste glass was used to intensify the material reactivity.

Figure 1a shows the particle size gradation curve of GS1, GS2, cement and metakaolin used in the experimental program. Additionally, Figure 1b presents the X-ray diffraction (XRD) of the ground waste, where an amorphous state can be verified, due to the absence of visible peaks in its spectrum. Studies have pointed out that the pozzolanic reactivity increases if more amorphous phases are present in the supplementary cementing materials [27,28].
Table 1
Physical and chemical properties of GS1, GS2, metakaolin and cement

| Composition (%) by mass | Glass sludge (GS1) | Ground glass sludge (GS2) | Metakaolin (ME) | Cement |
|------------------------|--------------------|---------------------------|-----------------|--------|
| Silica (SiO₂)          | 64.77              | 64.77                     | 58.7            | 19.42  |
| Alumina (Al₂O₃)        | 2.81               | 2.81                      | 33.1            | 4.87   |
| Iron oxide (Fe₂O₃)     | 0.44               | 0.44                      | 1.8             | 2.93   |
| Calcium oxide (CaO)    | 6.92               | 6.92                      | 0.2             | 63.69  |
| Magnesium oxide (MgO)  | 4.49               | 4.49                      | 0.2             | 0.86   |
| Sodium oxide (Na₂O)    | 19.36              | 19.36                     | 0.2             | —      |
| Potassium oxide (K₂O)  | 0.11               | 0.11                      | 1.8             | 0.8    |
| Sulphur trioxide (SO₃)| 0.21               | 0.21                      | 0.2             | 3.02   |
| % retained # 400 mesh   | —                  | —                         | —               | 2.2    |
| % retained # 325 mesh   | 19.65              | 7.64                      | 8.14            | —      |
| % retained # 200 mesh   | 10.63              | 4.08                      | 0.92            | —      |
| Average particle size (μm) | 42.17        | 35.02                     | 23.8            | 16.06  |
| Median particle size – d50 (μm) | 16.06          | 10.28                     | 16.65           | 13.75  |
| Specific gravity (g/cm³) | 2.51             | 2.51                      | 2.55            | 3.07   |
| Blaine specific surface area (cm²/g) | 6121         | 8015                      | 18549           | 4751   |

2.2 Waste from the process of stoning and polishing of glass

The glass powder used in this study is a waste originated in the process of stoning and polishing flat glass. It is obtained through the water recycling process, according to Figure 2. In summary, ducts crossing the glass manufacturing area collect the slurry that contains the water used in the stoning process along with the glass powder. This waste output is then conducted by pipes to reservoirs, where submerged pumps raise it to vertical silos for decantation. Subsequently, the sludge (water and glass powder) is pumped to vertical silos, where chemical products (compounds of ethanol, oxides and polar solvents) are added for decantation. The excess water is then transported to another reservoir where it is pumped back to the manufacturing process. Afterwards, the waste proceeds to the sludge mixer, and it is pumped to the filter press for dehydrating. The sludge is pressed, the excess water returns to the manufacturing process, and the final waste is obtained (Figure 2c). With this system, approximately 95 to 97% of the water is reused in the process [25]. The waste used in this study was obtained from a factory which generates around 290 kg of the final waste daily, totaling approximately 84 tons yearly. After collection, the waste was partially air dried, and then oven dried at 100 °C until mass constancy.

Figure 1
(a) Size gradation curve of cement, GS1, GS2 and metakaolin. (b) X-ray diffraction (XRF) of GS2
Performance of concrete with the incorporation of waste from the process of stoning and polishing of glass as partial replacement of cement

(Figure 2d). Subsequently, the waste was crushed to break the lumps using a ceramic mortar and pestle (Figure 2e). Additionally, aiming to intensify its reactivity, the waste was also ground. For the grinding process, a ball mill was used, composed of steel balls of 40 mm diameter and 0.278 kg. The internal diameter of the mill was 380 mm with a volume of 47 litres with the capacity of 14 kg per batch (Figure 2f). Thus, two types of glass powder were produced: GS1, without grinding, and GS2, ground.

2.3 Pozzolanic activity

The premises from the ABNT NBR 12653 [29] standard were used to assess the pozzolanic activity of the materials, where limits regarding the physical and chemical properties are established, as per Table 2. The standard classifies the materials in three classes (N, C and E) according to their nature. The glass waste fits in class E, comprising percentage amounts of SiO₂ + Al₂O₃ + Fe₂O₃ greater than 50%, whereas metakaolin fits into class N, comprising percentage amounts greater than 70% for the same compounds. The method according to Luxán et al. [30] was also used for complementing the information regarding the materials’ pozzolanic activity. This method, which can be rapidly executed, is based on measuring the conductivity of a saturated solution with calcium hydroxide, before and after adding the potential pozzolanic material. According to Rodrigues [31], variation of the conductivity occurs due to the pozzolanic reaction with the ions Ca²⁺ and (OH)⁻; that is,

| Properties                          | Limits ABNT NBR 12653 [29] | Limits ABNT NBR 12653 [29] | Glass sludge (GS1) (class E) | Ground glass sludge (GS2) (class E) | Metakaolin (ME) (class N) |
|-------------------------------------|-----------------------------|-----------------------------|-------------------------------|-------------------------------------|---------------------------|
| Fineness by sieve #325 (45µm) (% retained) | < 20 | < 20 | 19.65 | 7.64 | 8.14 |
| Pozzolanic activity with lime (MPa) [32] | ≥ 6 | ≥ 6 | 4.14 | 6.28 | 11.53 |
| Pozzolanic activity with cement (%) [33] | ≥ 90 | ≥ 90 | 83 | 90 | 137 |
| SiO₂ + Al₂O₃ + Fe₂O₃ (%) | ≥ 50 | ≥ 70 | 68.02 | 68.02 | 93.6 |
| Loss on ignition (%) | ≤ 6 | ≤ 10 | 0.21 | 0.21 | 0.2 |
| Available alkalis in Na₂Oₑₑ (%) | ≤ 1.5 | ≤ 1.5 | 19.43 | 19.43 | 1.38 |

* Na₂Oₑₑ = Na₂O + 0.658 K₂O
the pozzolan reduces the quantity of free ions in the solution and, consequently, diminishes its conductivity. From the difference of conductivity, it was possible to classify the material according to its pozzolanic activity, where materials which presented a variation of conductivity above 0.4 mS/cm can be classified as pozzolans. Figure 3 shows that the ground glass sludge (GS2), as well as metakaolin, can be classified as materials containing pozzolanicity materials according to the method of Luxán et al. [30]. It is also possible to compare the materials used in this research with other materials commonly used in concrete structures, such as: ornamental rock residues and active silica.

It can be observed that the glass powder did not comply with the standard ABNT NBR 12653 [29] regarding the available alkalis content, presenting significantly higher values (Table 2). This observation motivated the execution of tests to assess the ASR, which can cause expansive mechanisms and concrete cracking. The ASR occurs during the hydration process, due to the reaction between reactive aggregates and the alkalis present in the cement and glass powder [14]. Furthermore, it was noted that the GS1 did not fulfill the pozzolanic activity tests with lime [32] and cement [33], fact that was corroborated by the Luxán et al. [30] method. Therefore, a preliminary study was performed to assess the necessary grinding time in a ball mill. The required time reached was 2 h, reducing the retained percentage in sieve #325 (45 µm) from 19.65 to 7.64% (maximum reduction obtained) (Figure 4a). The GS2 (Figure 4b) and GS1 aspect (Figure 4c) are also presented. Figures 4d and 4e show the microscopic analysis of the ground residue where its angular geometry and particle sizes ranging from 2 µm to 10 µm can be identified.

After this process, the resulting product was a ground waste, called GS2, finer than metakaolin, which complied with the required limits to be classified as a pozzolanic material according to all tests performed [30,32,33] (Table 2).
2.4 Specimens preparation

Mixtures containing 10 and 20% of glass sludge waste as partial substitute for cement (by volume) were used, adopting the waste without grinding (GS1) and after grinding (GS2) (Table 3). The cement replacement by metakaolin (ME) was also evaluated at the same contents, for comparative reasons, and the metakaolin combined with both glass powders (GS1ME and GS2ME) (Table 3) with aim of intensifying the mechanical and durability properties. The water/cementitious materials ratio (w/cm) used was 0.60, this being the highest accepted in the standard ABNT NBR 6118 [34] in urban applications. Furthermore, at this w/cm ratio, a concrete of high porosity could be obtained, making it easier assess the possible pore-filling effects of the cement replacement by waste glass. Since the density of the waste glass powder and metakaolin is less than that of the cement, a volumetric compensation was used. It is important to note that the amount of water was maintained from the control mixture, considering the water/cement ratio of 0.60, changing the water/binder ratio for the other mixtures thereafter. The dosage method adopted was the IPT-EPUSP Method. Cylindrical specimens of 100 × 200 mm in diameter and height were prepared, according to the standard ASMT C192 [35], and cured in a moist chamber with relative humidity higher than 95% and temperature of 23 ± 2 °C, until the testing day. The concrete fresh properties were assessed using slump test, according to ASMT C143 [36], and density measurement, according to ASMT C138 [37]. The slump value used for the control mixture was 200 ± 10 mm. This value, from the batching and consistency study, was adopted to compensate the reduction of the slump caused by the incorporation of fine and not immediately reactive materials (metakaolin and glass powder). Thereby, an appropriate workability was obtained for the preparation of mixtures, since the use of superplasticizer was not considered for the study in order to not add new variables that might hamper the results identification.

Table 3
Mixture proportions for 1 m³ of concrete

| Mix design | Cement replacement (%) (by volume) | Cement (kg/m³) | Glass sludge (GS1) (kg/m³) | Ground glass sludge (GS2) (kg/m³) | Metakaolin (ME) (kg/m³) | Fine aggregates (kg/m³) | Coarse aggregates (kg/m³) | Mixture density (kg/m³) | Water content (kg/m³) |
|------------|-----------------------------------|----------------|--------------------------|-----------------------------------|------------------------|----------------------|------------------------|----------------------|----------------------|
| Control    | 0                                 | 302.85         | —                       | —                                 | —                      | 875.24               | 1133                   | 2420                 | 181.71               |
| GS1-10     | 10                                | 272.57         | 24.77                   | —                                 | —                      | 875.24               | 1133                   | 2402                 | 181.71               |
| GS1-20     | 20                                | 242.28         | 49.57                   | —                                 | —                      | 875.24               | 1133                   | 2371                 | 181.71               |
| GS2-10     | 10                                | 272.57         | —                       | 24.77                             | —                      | 875.24               | 1133                   | 2391                 | 181.71               |
| GS2-20     | 20                                | 242.28         | —                       | 49.57                             | —                      | 875.24               | 1133                   | 2430                 | 181.71               |
| ME-10      | 10                                | 272.57         | —                       | —                                 | 25.17                  | 875.24               | 1133                   | 2396                 | 181.71               |
| ME-20      | 20                                | 242.28         | —                       | —                                 | 50.30                  | 875.24               | 1133                   | 2396                 | 181.71               |
| GS1ME-10   | 10                                | 272.57         | 12.42                   | —                                 | 12.72                  | 875.24               | 1133                   | 2423                 | 181.71               |
| GS1ME-20   | 20                                | 242.28         | 24.77                   | —                                 | 25.17                  | 875.24               | 1133                   | 2409                 | 181.71               |
| GS2ME-10   | 10                                | 272.57         | —                       | 12.42                             | 12.72                  | 875.24               | 1133                   | 2409                 | 181.71               |
| GS2ME-20   | 20                                | 242.28         | —                       | 24.77                             | 25.17                  | 875.24               | 1133                   | 2412                 | 181.71               |

2.5 Methods

2.5.1 Mechanical strength tests

The specimens were cured in a moist chamber until the testing ages of 28, 56 and 91 days. The upper and lower parts were polished for levelling, and then the specimens were submitted to the compressive test, according to standard ABNT NBR 5739 [38].

2.5.2 Rapid chloride permeability test (RCPT)

The rapid chloride permeability test (RCPT) was idealized by Whiting [39], and it is recommended by standard ASTM C1202 [40]. For each mixture, three specimens – 50 mm thick and 100 mm in diameter, extracted from the central part of the single original cylinder specimen – were tested at 28 and 91 days. The test consisted in exposing one side of the specimen to a sodium chloride solution (3% NaCl by mass) and the other to a sodium hydroxide solution (0.3 N NaOH). In each solution, a conducting copper electrode was introduced, and connected to a 60 V ± 0.1 V source, creating an electrical current that induced the chlorides to migrate through the concrete specimen. Amperage readings were taken every 30 min, totaling 6 h of testing. The sum of the electrical current by time, expressed in coulombs, specified the total charge passed through the specimen, indicating the chloride penetration resistance of the concrete.

2.5.3 Rapid migration test (RMT)

Proposed by Luping and Nilsson [41] and consolidated in the standard NT BUILD 492 [42], this method uses as procedures the potential difference and a colorimetric indicator (spray of AgNO₃ – 0.1 M). It provides quantitative data on the final chloride penetration depth and chloride diffusion coefficient. Specimens 50 mm thick and 100 mm in diameter, extracted from the central part of the single original cylinder specimen, were tested at 56 and 91 days. The specimens were exposed to sodium chloride solution (2N NaCl) on one side
and on the other to sodium hydroxide solution (0.3 N NaOH). The test duration and the voltage adopted depended on the initial passing current reading in the concrete specimen when applying a different potential of 30 V. Usually, the test can last from 24 h, for ordinary concrete, to 96 h, for high performance concrete. To calculate the apparent chloride ion migration coefficient, Equation 1 was used.

\[ D_{\text{app}} = \frac{0.0239 \times (273 + T) \times \left( x_1 - 0.0238 \sqrt{\frac{273 + T}{\Delta x}} \right)}{(U - 2)t} \]  

(1)

Where: \( D_{\text{app}} \) is the non-steady-state migration coefficient multiplied by 10-12 (m²/s); U is the absolute value of the applied potential (V); \( T \) is the average value of the initial and final temperatures in the anolyte solution (°C); \( L \) is the thickness of the specimen (mm); \( x_1 \) is the average value of the chloride penetration depth (mm), and \( t \) is the test duration (h).

### 2.5.4 Bulk diffusion test (BDT)

The method for determining the apparent chloride diffusion coefficient on cementitious mixtures by bulk diffusion is based on the NT BUILD 443 [43] and consists in keeping the concrete specimens immersed in a chloride solution (165g of NaCl by litre) to induce accelerated diffusion mechanisms. The immersed test specimens (100 mm thick and 100 mm in diameter) were placed in a hermetically sealed plastic container, the solution being agitated weekly. The specimens were cured for 28 days in a moist chamber and subsequently immersed in the solution for 60, 120 and 180 days. The chlorides profile was obtained by milling the material in layers parallel to the exposed surface. The thickness of the layers was adjusted according to the expected chloride profile, such that at least six places encompassed the profile between the exposed surface and the depth reached by the chlorides. The content of chloride soluble in acid in the specimens was determined according to NT BUILD 208 [44]. The immersed test specimens were demolded 24 h after their casting, and submerged in water at 80 ºC for 24 h. Subsequently, they were submerged in a solution of NaOH (1N) at 80 ºC and their initial lengths were registered. During 30 days, length readings were performed with the frequency of 3 days. The mentioned standard considers that if the expansion is higher or equal to 0.19% at 30 days, the aggregate is considered potentially reactive.

### 3. Results and discussions

#### 3.1 Slump tests

The Figure 5 presents the slump values of the concrete mixtures. By using the glass sludge without grinding (GS1) and after grinding (GS2) there is a reduction in the slump value in comparison with the control mixture, as also found in other studies [46-48]. However, it was still an adequate workability (100 ± 20 mm) to produce the concrete specimens and for the conventional applications of reinforced concrete, placed without pumping (ABNT NBR 8953 [49]). The use of materials with elevated superficial area and which do not react immediately in the mixture increases the demand for water in the fresh concrete. Thus, the use of very fine materials, as it is the case of the glass powder (average particle size 35-45 µm) used in this study, confirms this mentioned effect, which can also be related to the poor geometry of the waste glass. According to Ismail and Al-Hashmi [46], the reduction in the slump value can also be attributed to the geometry of the glass powder, resulting in a lower fluidity of the mixtures. Furthermore, mixtures with glass powder (GS1 and GS2) presented higher slump value than those mixtures that used metakaolin, indicating the lower demand for water of the glass sludge compared to metakaolin.

### 3.2 Mechanical strength tests

A compressive strength loss was identified at 28 days by replacing the cement by GS1 (Figure 6), as also verified in previous studies [10-13, 25]. Nevertheless, the concrete using the ground waste at 10 and 20% cement replacement (GS2-10 and GS2-20), presented similar values to the control mixture, without significant statistical variation. It is also observed that the combination of the glass sludge waste with metakaolin, at 10% cement replacement...
Performance of concrete with the incorporation of waste from the process of stoning and polishing of glass as partial replacement of cement

Figure 6
Concrete compressive strength results

Each (GS2ME-20), presented an increase in compressive strength of 12% at 28 days, compared to the control mixture. Moreover, the positive effect of using glass powder can be seen at more advanced ages. At 56 and 91 days, the mixture with 20% ground glass powder (GS2-20) had results slightly higher than those of the control mixture. These results can be explained by the physical effect of pore-filling, where the empty pores left by the cement paste are filled by the glass particles, reducing the porosity, and contributing to the retention of the mixture water, enhancing the cement hydration process. Another process that may have contributed to the compressive strength gain is the possible pozzolanic effect of the glass waste. This possible chemical reaction between the glass powder and the cement (due to the formation of more stable compounds, such as the production of C-S-H originated from the reaction of calcium hydroxide and water) diminishes the empty spaces in the transition zone between the paste and the aggregates. Moreover, the GS2ME-20 presented superior values to that of the control mixture at all ages, and at 56 and 91 days, its values were similar to the mixture containing only metakaolin as a cement replacement, at 20% (ME-20). Thus, replacing the use of 10% metakaolin by glass waste is justified, promoting also possible economic and environmental aspects.

The ANOVA showed that there is a statistically significant (p-value < 0.05) reduction in the compressive strength as the glass sludge without grinding (GS1) content increases (Fig. 7a). Thus, initially indicating that the cement replacement by glass powder

Figure 7
Statistical analysis of concrete with glass wastes. (a) mixtures with GS1 (b) mixtures with GS2
without grinding did not cause a positive effect on the compressive strength. This is possibly due to the non-pozzolanic effect of the material (as observed in the pozzolanic activity tests – Table 2), combined with the fact the pore-filling effect was not sufficient to compensate the strength loss caused by the cement content reduction. It became necessary the identification of which treatments presented the variation; thus, the Tukey’s test verified that the GS1 provided similar results to that of the control mixture. Therefore, indicating that it is possible to replace the cement by the GS1 without reducing the concrete strength, at up to 10%, whereas at 20% replacement, a loss in compressive strength is verified. Additionally, using this statistical analysis to the GS2, it is noted that there is no significant difference between the concrete with and without the waste, since the GS2 content in the compressive strength presented p-value of 0.642 (> 0.05) (Fig. 7b and Table 4). Hence, it is possible to replace the cement by up to 20% of ground glass powder without negative effects in the compressive strength. This performance can be justified due to the pozzolanic reaction with the cement hydrated compounds, as identified in the pozzolanic activity tests performed in the ground waste (Table 2).

3.3 Rapid chloride permeability test (RCPT)

The use of glass powder in the contents of 10 and 20% increased the chloride penetration resistance of the mixture, by reducing the total charge passed (Figure 8), as noted in previous studies [11-16]. It is noted that both GS1, by the pore-filling effect, and GS2, by the pore-filling and the possible pozzolanic activity effect, produced a reduction in the total charge passed in the mixtures in comparison with the control, being the GS2 the most effective. The mixture with 10% glass sludge without grinding (GS1-10) did not reach a moderate level of chloride penetrability; nevertheless, it reduced the charge passed by 13 and 30% at 28 and 91 days, respectively, in comparison with the control. For 20% content (GS1-20), at 91 days it is verified a reduction of 83% compared to the control, being equivalent to the reduction obtained by the mixtures with ground waste (GS2-20, with 80% reduction) and metakaolin (ME-10, with 84% reduction, and ME-20, with 79%). Figure 8 presents the qualitative classification used by the test standard and classifies GS1ME-20 and ME-10 as of very low chloride penetrability level, a significant reduction from the high level observed for the control mixture. This may be due to the combined effect of the metakaolin pozzolanic activity and the pore-filling effect caused by the waste. Furthermore, at 10% glass powder, the chloride resistance is improved with the grinding of the glass waste (GS2-10), and the increase of glass powder content also improves the chloride resistance.

According to the qualitative assessment proposed by Gjørv [50], the GS1-10 did not reach a moderate resistance to chloride penetration. However, the GS1ME-20 and ME-10 mixtures presented a significant reduction in the chloride penetrability level, as indicated by the test standard classification (Figure 8). This performance is attributed to the combined effect of the metakaolin pozzolanic activity and the pore-filling effect caused by the waste. Furthermore, the GS2-10 mixture with 10% glass powder, which was not ground, also showed improved chloride resistance compared to the control mixture.

### Table 4

| Factors          | SS    | DF | MS    | F      | p-value   | Results   |
|------------------|-------|----|-------|--------|-----------|-----------|
| GS2 content      | 10.30 | 2  | 5.15  | 0.447  | 0.641849  | Non-Significant |
| Age              | 199.92| 2  | 99.96 | 8.676  | 0.000550  | Significant |
| Error            | 610.66| 53 | 11.52 | —      | —         | —         |

**Figure 8**

Total charge passed results and chloride resistance classification.
penetration (Figure 9), corroborating the results from total charge passed (ASTM C 1202 [40]). Thus, to improve the resistance to chloride penetration with 10% glass sludge waste addition, it was needed to grind the material (GS2-10), or to use it in combination with 10% metakaolin (GS1ME-20), replacing 20% of cement. The increase of glass sludge content to 20% (GS1-20) provided improvements in chloride resistance, advancing the classification to high chloride resistance at 56 days and very high at 91 days. At 56 days, the reductions in the chloride migration coefficient in comparison to the control mixture were 18% for GS1-10 and 78% for GS1-20; whereas at 91 days, these reductions were 22 and 85% for GS1-10 and GS1-20, respectively. This evolution can be attributed to the mechanism of pore-filling. Furthermore, the use of ground glass sludge waste in the contents of 10 and 20% (GS2-10 and GS2-20) provided improvements in chloride resistance comparable to those caused using metakaolin.

Thus, it can be confirmed, just as previous studies [11,12,15] that have used the RMT test (NT BUILD 492 [42]), that the glass powder use as a cement replacement at 20% content provides an improvement in chloride penetration resistance, mainly at 56 days. This delayed influence in the improvement of concrete microstructure can be attributed to the pozzolanic effect or to the pore-filling effect. For the control mixture, no variation in chloride resistance from 56 to 91 days is noted, whereas for the glass sludge and metakaolin mixtures, increases in chloride resistance are observed.

Pignaton [25] performed a microstructural analysis in concrete containing a glass sludge waste equivalent to the one used in this study at similar replacement contents; but without grinding the waste. The analysis focused on assessing the interfacial transition zones between the coarse aggregate and the cement paste after 28 days age. It was noted that the partial replacement of cement by GS1 at 20% content provided a reduction in the cement matrix porosity and the interfacial transition zones were reinforced by crystalline formations due to the chemical reaction with the glass powder in the hydration process. Furthermore, the concrete have not presented any cracking that could indicate the production of expansive gel or ASR.

This study assessed the microstructure of concrete with the ground waste (GS2) and metakaolin (ME) focusing on the improvement of the interfacial transition zone between cement paste and aggregate (Figure 10). 500x, 1000x and 3000x enlargements were used. The scanning electron microscope (SEM) images (Figure 10) shows that the mixtures with GS2 and ME presents a more compact structure with fewer pores in relation to the REF. The formation of fibrous C-S-H crystals filling the pores among the hydrated cement particles is verified, explaining the improvement in the durability properties of the mixtures. In the GS2-2- and ME-20 mixtures, the GS2 and ME particles were encapsulated and dispersed in the gel hydrated compounds (Figure 10f and 10i). Furthermore, no cracking due to the possible expansive reaction of the waste glass was found.

### 3.5 Bulk diffusion test (BDT)

Figure 11 presents the linear regression analysis of the chloride content through the concrete depths, from the diffusion by immersion test according to NT BUILD 443 [43]. The use of GS1 and GS2 provided a reduction in the chloride content through the concrete, and these reductions are greater for increased contents of glass powder (Figure 11a and 11b). In Figure 11c, by analyzing the mixtures with glass powder at 20% content (GS1-20 and GS2-20), the grinding presented itself as a determinant factor, the ground waste being

![Figure 9](image-url)  
**Figure 9**  
Non-steady-state chloride migration coefficient and chloride resistance classification
more efficient for chloride resistance. From the liner regression analysis, it was possible to calculate the diffusion coefficients identified in Figure 12, as defined in NT BUILD 443 [43]. For comparative purposes, Figure 12 also presents the results of chloride diffusion coefficients obtained from NT BUILD 492 [42] test for the age of 56 days. Additionally, the limits for the qualitative assessment proposed by Gjørv [50] are also presented. From the test results of chloride diffusion (NT BUILD 443 [43]) and migration (NT BUILD 492 [42]), it is verified that the use of glass powder and metakaolin provided an increase in chloride resistance, especially in the mixtures using the ground glass powder and 10 and 20% (GS2-10 and GS2-20). Additionally, the GS2 provided similar values to those of metakaolin. The GS2ME-20 mixture (GS2 and ME at 10% each) presented the lowest diffusion coefficients, surpassing even the mixtures with only metakaolin. This mixture presented as very effective due to the combination of pozzolanic effect of both materials along with the pore-filling effect. According to Zibara et al. [51], the chloride diffusion through the concrete depends mainly on its microstructure and ions fixation capability. For mixtures with ordinary Portland cement, the key fixation mechanism is the formation of Friedel’s salt and aluminates related complexes [52]. The anhydrous compounds of cement which react quickly with the chloride ions producing the Friedel’s salt is the tricalcium aluminate (C3A). In concrete with SCM, it is

Figure 10
Microstructure analysis of concrete mixtures with GS2 and ME. (a) REF (500x magnification); (b) REF (1000x magnification); (c) REF (5000x magnification); (d) GS2-20 (500x magnification); (e) GS2-20 (1000x magnification); (f) GS2-20 (5000x magnification); (g) ME-20 (500x magnification); (h) ME-20 (1000x magnification); (i) ME-20 (5000x magnification)
indicated that the fixation capability is a function of the aluminates content [53]. Thus, the metakaolin, by having in its chemical composition 33.10% alumina (Al₂O₃) (Table 1), fosters the formation of C₃A compounds and helps to increase the chloride ions fixation. It was also observed that the chloride diffusion coefficients obtained from NT BUILD 443 [43] were higher than the chloride migration coefficients obtained from NT BUILD 492 [42] (Figure 14). This can be explained by the time difference of testing, where for the migration test the mixtures were cured for 56 days (concrete age with a more developed microstructure), and for the diffusion test, the specimens were cured for 28 days and then exposed 180 days to the chloride solution, providing more time for the chloride to propagate; a different adopted time frame from the used in studies that aimed at assessing the correlation between both tests [21, 23]. The BDT (NT BUILD 443) is relevant for the comparison among different concrete of the same study due to be an alternative accelerated method which do not require the use of electrical currents and the adoption of colorimetric indicators – procedures used by RMT (NT BUILD 492 [42]) which are criticized by some researchers [21-23]. From this comparison of results, it can be noted that by adopting a content of cement replacement of 20%, the glass powder can be used without grinding (GS1), presenting increases in chloride penetration resistance, comparable to the values obtained in the mixtures with metakaolin use. Moreover, this chloride resistance can be further increased if the waste is ground (GS2).

3.6 Alkali-silica-reaction (ASR) –

**Figure 11**
Linear regression analysis of chloride content through concrete depths. (a) Glass sludge waste without grinding (GS1); (b) Ground glass sludge waste (GS2); (c) GS1 and GS2 at 20% content

**Figure 12**
Chloride diffusion coefficient results as according to NT BUILD 443 and NT BUILD 492
1. The mixtures containing glass sludge waste presented a reduction in workability in comparison with the control mixture, further noticed with increase of the content. Nevertheless, the slump values were higher than the mixtures containing only metakaolin.

2. The use of glass waste, originated from stoning and polishing of glass, generally did not cause a reduction in compressive strength with statistical significance (with exception to the glass without grinding at 20%, GS1-20), and provided durability improvements. The resistance to chloride penetrability was improved, evidenced by chloride ions diffusion and migration tests. Additionally, the ASR potential was reduced by using the glass powder, and further diminished by grinding the glass waste.

3. Many advantages in incorporating the ground glass sludge waste as a cement replacement at 20% (GS2-20) were verified. Its use did not cause significant influence on compressive strength and provided significant improvements in chloride penetration resistance. The reductions obtained in comparison with the control mixture were 80% in total charge passed, 89% in chloride migration coefficient, 73% in chloride diffusion coefficient and 58% in ASR related expansion.

4. The combined use of metakaolin and ground glass sludge waste at 10% each (GS2ME-20) presented the best performance among the mixtures which contained glass sludge waste, providing more than 12% increase in compressive strength and more than 74% reduction in chloride penetrability. It can also be an alternative to the isolated use of metakaolin (ME-20), since equivalent values of compressive strength and chloride resistance were observed. Thus, financial and environmental benefits may be attained.

5. This study concludes that the incorporation of ground glass sludge waste, a waste from the process of stoning and polishing of glass, can be adopted in reinforced concrete structures without detriment in the mechanical properties and providing an increase in durability in structures in an urban maritime environment. The material can replace the cement up to 20%, without affecting the compressive strength at 28 days, and reducing the chloride penetration by up to 90%, besides providing a reduction in ASR-related expansion.

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