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# INFLUENCE OF GLASS PROCESSING WASTE QUANTITIES ON THE FREEZE-THAW RESISTANCE OF SUSTAINABLE CONCRETE

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*Two difficult to utilise wastes - glass sludge, obtained from processing glass, and concrete sludge, obtained by washing concrete trucks, were used. In the sustainable concrete composition, glass sludge was used as a replacement for cement, varying from 5 % to 30 % and concrete sludge was used as a filler. Additionally, sand, dolomite, a superplasticiser, a crystallising additive, and an air-entraining additive were used and a consistent water-to-binder ratio was maintained. The content of the entrained air, porosity parameters, compressive strength, water absorption, density and sorptivity index for the hardened samples were measured. The resistance to freezing was assessed by the volumetric method and scaling tests in a de-icing salt solution to determine the mass losses, while the damage degree of the internal structure was evaluated. The results suggest that the synergistic interaction between the glass sludge and the crystallising additives promotes pore healing, enhancing the closed porosity and reducing the open porosity at glass sludge levels of 20% or less. The findings indicate that glass sludge has no detrimental effects on the frost resistance qualities and may even enhance them if up to 20 % of cement is substituted with glass sludge.*

## INTRODUCTION

As the world's population increases and various industrial sectors grow, the demand for different types of produced goods also rises. With the increasing volume of production, the amount of waste generated also grows and accumulates in various landfills [1]. The accumulating waste from industry and the need to find ways to recycle this waste present a challenge [2-5]. One area where various types of waste can be successfully utilised is in the manufacturing of building materials.

Cement, as the highest energy-intensive component of concrete, is responsible for the significant usage of natural resources, the alteration of the natural landscape, enormous energy consumption during its production and combustion processes, and negative impacts on the environment [6-8]. For these reasons, cement production is not considered sustainable [9,10]. According to various calculations, cement production is one of the most contaminating industrial sectors in the world in terms of greenhouse gas emissions into the environment [11-13]. To reduce the environmental impact, blended cements are becoming more popular, which incorporate additional materials or replace cement with other pozzolanic materials. Some industrial waste, such as fly ash or slag, are being used in the manufacturing of building materials to replace traditional components. However, concrete made with waste often encounters technological problems related to the complex structures, leading to issues with the durability of the concrete [14]. Therefore, it is vital to study the effects of these waste materials on the characteristics and durability of the mixtures in concrete production.

Currently, various glass wastes are receiving wider attention. The quantities of these wastes are steadily increasing due to the rising demand for glass products. Glass is broadly used in daily items like windows and packaging, and in various other industries due to its recyclability, durability, and transparency [17]. Non-recycled glass processing wastes present a significant threat for our environment, because glass is an inert material that is biologically non-degradable and does not burn during incineration, leading it to accumulate in landfills and create large amounts of waste [18,19]. According to studies, in the European Union in the year of 2018, 16.5 million tonnes of glass waste was produced, and 12 million tonnes were reclaimed [20].

Glass waste is beneficial in building materials due to its high silica content [16], and these wastes are often pozzolanic. These properties suggest that using glass waste in concrete could lessen the volume of waste in landfills and lower $CO_2$ emissions, as replacing a portion of the cement with waste would significantly decrease the usage of natural raw materials in concrete. The proper use of glass wastes in construction products can decrease the water and soil pollution, reduce the landfill area usage, and lessen the negative impact on human health [15]. Efforts to use glass waste in concrete production date back to 1960 [21]. Various researchers [22-25] have tried to utilise glass waste like aggregates, while powdered glass waste has been tested as a cement substitute. Some researchers have concluded that glass waste enhances the workability of concrete mixtures due to the very smooth surface and lower water absorption of the glass compared to both natural aggregates and cement [26, 27]. Some opposing results have indicated that glass waste deteriorates the properties of fresh concrete mixtures, as reported by other researchers [28, 29]. Due to the observed poorer workability characteristics, hypotheses have been raised that this might be due to the different shapes of the particles, irregular geometry, and rough glass texture. It was also found [30] that due to the lower water absorption and smooth particle surfaces, glass particles may reduce the friction among the glass and cement paste particles, which can increase the risk of segregation and water separation.

Tests conducted with self-compacting concrete mixtures concluded that adding glass waste as an additive can yield similar flow properties compared to conventional mixtures and reduce the amount of superplastisers used in the mixtures, as the adhesion and friction between the glass particles and cement paste are reduced [31, 32].

When investigating the concrete density, researchers have often observed that the glass additive in the concrete reduces both the density of the fresh mix and the density of the hardened concrete [33, 34]. Such results are typically obtained because natural aggregates have a higher specific gravity than glass particles [19]. Some researchers [35] have noted an increase in the density when replacing fine aggregate with glass waste by 15 %, but as the replacement amount of the fine aggregate increases, the density begins to decrease.

When studying powdered glass waste in concrete, it has been observed that the pore framework in the concrete can be enriched by very fine and spherical-shaped particles which may reduce the water absorption and enhance the resistance to corrosion, like sulfate corrosion or alkali-silica reactions [36]. Glass powders can also improve the interconnection of pores and enhance water movement in the internal composition of the concrete [37]. Initially, using glass waste instead of cement in the concrete may diminish the porosity, but the improved density of hardened concrete shows a decrease in the formation of large pores [38]. Due to the better pore structure and their interconnection, the water absorption of the concrete can decrease as more glass powders are used [39]. The mixes water-to-binder ratio also affects the pore structure [40], where the amount of water can significantly impact the workability and density, which, in turn, influences the pore structure.

Researchers have obtained varying results when investigating the flexural strength. Some researchers [41, 42] have discovered that mixing concrete with glass waste has a positive influence on the flexural strength. These results were obtained by substituting fine aggregates with glass waste. Other researchers [43, 44] determined the opposite, that the flexural strength decreased in concrete compositions using glass waste, who also replaced fine aggregates with glass waste. It is worth mentioning that the same researchers [43] observed a higher tensile strength when glass waste was used instead of fine aggregates.

Typically, when aggregates are replaced with glass waste, the compressive strength obtained is lower, and glass particles negatively impact this because their particle strength is lower, they have a smooth surface, and the angularity of the particles combined with reduced adhesive forces within the structure [19, 43, 45]. Researchers [46] established that if the glass waste particles are smaller than 38 mm, they can participate in pozzolanic reactions. Due to the pozzolanic properties of glass waste and its additional participation in chemical reactions, it can lead to enhanced compressive strength [47, 48].

Fine particles of glass waste, when replacing fine aggregate up to 21 % in concrete mixes, improved concrete microstructure [52]. Replacing cement by glass waste powder also reduces the chloride ions penetration of into the inner structure, as the glass powder participating in hydration reactions increases quantity of C–S–H in concrete mixtures [53, 54]. Another reason for reducing the penetration of chloride ions to the concrete is the non-porous nature of the glass particles.

Another property being investigated for durability, aside from material water absorption and resistance to aggressive ions, is the reactivity of particles in an alkaline environment. Since glass particles are amorphous silicon dioxide, they tend to react with alkaline substances, leading to an increase in the particle volume. This reaction is known as the alkali-silica reaction (ASR). Therefore, when using these wastes in concrete, the ASR must be monitored [55]. It has been observed that various factors can influence these reactions, such as the size, quantity, colour of the glass particles, and supplementary cementitious materials (SCMs) [56, 57]. ASR reactions are typically promoted by a higher quantity of glass particles in concrete mixtures [58], but these risks can also be managed by including additional pozzolanic materials, like metakaolin or micro silica [59].

When discussing the durability of concrete, one property that affects the longevity of concrete products is the water absorption. The lower the absorption, the more likely it can be assumed that the concrete will be more durable. If the water absorption of the samples significantly increases, it can make a tremendous result on the concrete functionality and durability, especially its frost resistance [60]. Research indicates that tests conducted on concrete containing glass waste resulted in lower water absorption [49, 50, 51]. Such results are obtained because normally used natural aggregates have higher water absorption compared to glass particles.

Another important property for concrete durability is shrinkage. To reduce the shrinkage, special concrete additives can be utilised. Concrete additives have proven to have a positive effect in reducing the self-shrinkage in the conducted studies [17, 61, 62]. In a previous study, the shrinkage, porosity, and mechanical-physical characteristics of concrete with different quantities of a crystallising additive (CA) were tested. Samples containing 0.9-1.0 % CA shrank the least over 190 days. It was concluded that the CA protects against cracking within the sample structure [63].

In addition to the glass waste already reviewed, another type of waste worth mentioning is washed concrete waste, the quantities of which are constantly growing. Washed concrete waste in concrete could be used as a natural fine aggregate [64].

In the opinion of the reviewed literature, it could be deduced that glass waste has a workable option for concrete mixtures and can be experimented with as a supplementary cementitious material by altering the amount of cement in the samples. There are a lack of durability tests in the literature associated with frost resistance with de-icing salts. Understanding the frost resistance could significantly affect future research concerning the use of glass waste in concrete mixtures.

The study looked into the effect of changing the amounts of glass processing waste in concrete in the place of cement and effect on the characteristics of concrete with a crystallising additive. Additionally, part of the natural fine aggregate in the compositions was replaced with washed concrete waste. Six different concrete mixture compositions with varying amounts of glass sludge were tested to determine the impact of three components – glass sludge, crystallising additives, and washed concrete waste – on the physical-mechanical properties and frost resistance. The results obtained can enrich the application of the developed concrete in the production of building materials. This study may provide new insights into the durability of concrete and solutions for the industry regarding the resulting waste.

## EXPERIMENTAL

In these experiments, materials adhering to the current European standards for concrete production were used. Cement CEM I 42.5 R, meeting the specifications of EN 197-1 [65], served as the primary binding agent. The aggregates for the concrete tests conformed to standard EN 12620 [66]. The fine aggregate used was sand with a 0/4 fraction, while the coarse aggregates comprised two fractions of crushed dolomite: 5/8 and 4/16. To ensure the characteristics of both the concrete mix and hardened concrete, technological additives such as a superplasticiser, air-entraining agents, and a crystallising additive were employed. The crystallising additive was used to reduce the water absorption, extend the service life, and seal cracks up to 0.4 mm. All the additives adhered to EN 934-2 [67] standards.

Concrete sludge, obtained by washing concrete mix-ures and separating wash water from coarse aggregates, was also utilised in the concrete mixtures. The dried concrete slurry functioned as a substitute for the fine aggregate, while the wash water partially replaced the tap water in the mixtures. The chemical oxide composition of the dry concrete slurry is shown in Table 1, with additional properties detailed in Table 2.

The crystallising additive came from the German company Ha-Be Betonchemie GmbH, known globally. DURAHIT® Crystal Ad 2000 (DM) was the specific crystallising additive used, in a powder form, also adhered to EN 934-2 [67].

To minimise the cement usage, glass sludge, which emerges during glass product processing, was incorporated. This waste is collected using special equipment that

Table 1. Chemical compositions of the additives.

| Oxide composition of concrete sludge (%) | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| CaO | $SiO_2$ | $Al_2O_3$ | $SO_3$ | $Fe_2O_3$ | MgO | $K_2O$ | $P_2O_5$ | $TiO_2$ | SrO | MnO | Cl |
| 40.9 | 14.9 | 3.14 | 2.40 | 2.37 | 2.26 | 0.89 | 0.38 | 0.21 | 0.06 | 0.04 | 0.03 |

| Oxide composition of the glass sludge (%) | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| $SiO_2$ | $Na_2O$ | CaO | MgO | $Al_2O_3$ | $SO_3$ | $K_2O$ | $CeO_2$ | $Fe_2O_3$ | $La_2O_3$ | Cl |
| 69.0 | 10.4 | 8.68 | 3.55 | 0.93 | 0.24 | 0.15 | 0.148 | 0.11 | 0.074 | 0.027 |

| Oxide composition of the crystallising admixture (%) | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| CaO | $Na_2O$ | $SiO_2$ | $SO_3$ | ZnO | $Al_2O_3$ | $K_2O$ | MgO | $Fe_2O_3$ | $P_2O_5$ | Cl |
| 27.0 | 21.0 | 17.0 | 1.72 | 1.43 | 1.21 | 0.76 | 0.75 | 0.49 | 0.34 | 0.11 |

Table 2. Characteristics of the materials.

| Properties | Glass sludge | Concrete sludge | Cement | Sand | Dolomite |
|---|---|---|---|---|---|
| Specific surface ($cm^2 \cdot g^{-1}$) | 6670 | 316 | 4600 | – | – |
| Particle density ($kg \cdot m^{-3}$) | 2500 | 2774 | 2700 | 2660 | 2800 |
| Bulk density ($kg \cdot m^{-3}$) | 826 | 826 | 1475 | 1610 | 1480 |
| Pozzolanic activity ($mg \cdot g^{-1}$) | 927 | – | – | – | – |

separates the fine glass particles from the technological water through flocculants and cyclones, followed by sedimentation, resulting in a glass sludge. The chemical composition and characteristics of the glass slurry are detailed in Tables 1 and 2.

The pH level of the concrete sludge was measured at 11.4, in accordance with the LST ISO 4316:1997 standard [68]. This elevated pH is attributed to the presence of numerous dissociated calcium oxides, as shown in the reaction outlined in Equation 1 [69], which increases the concentration of electrolytes and the pH value. The concrete sludge was evaluated following the LST EN 1008:2005 standard [70] to ascertain the quantities of the dry materials and the material density. It was determined that the density of the concrete sludge is 1220 kg·m$^{-3}$, and the calculations indicated that the solid particle content within the concrete sludge is 500 kg·m$^{-3}$

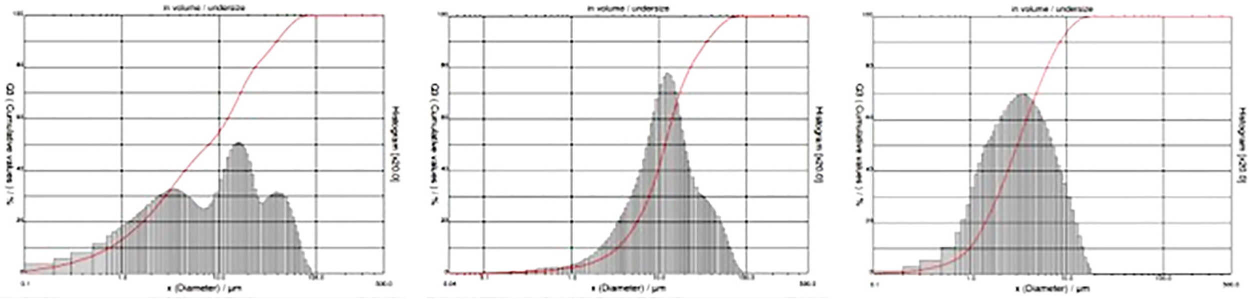
$$CaO + H_2O \rightarrow Ca(OH)_2 + \Delta H \tag{1}$$

The particle size distribution for cement, glass, and concrete sludge was analysed, with the findings illustrated in Figure 1. Cement particle sizes vary between 0.1 and 70 µm with three main maxima of the particle size distribution (5-8 µm, 15-18 µm and 45-60 µm). The average particle size for the dried concrete sludge is measured at 15.85 µm, whereas the average particle size for the glass sludge is 3.98 µm.

The formulations utilised in the experiments are detailed in Table 3. Seven distinct sample batches were prepared, with the first batch functioning as the control. In all the formulations, 5 % of the sand was replaced with dried concrete sludge, and 10 % of the tap water was substituted with the wet concrete sludge. Each batch included a consistent amount of crystallising additive, set at 1 % of the cement weight. The proportion of glass sludge in the various batches was increased, while the replaced cement content varied at 5 %, 10 %, 15 %, 20 %, 25 %, and 30 %. A uniform water-to-binder ratio (w/b) was maintained across all the test mixtures.

Concrete cubes were produced from the mixtures in both plastic and metal moulds measuring 100 × 100 × × 100 mm and 150 × 150 × 150 mm. The concrete mixture was compacted on a vibrating table for a duration of 20 seconds for each sample. After 12 hours of hardening in a humid environment at a temperature of 20 ± 2 °C, the samples were removed from the moulds and submerged in water at the same temperature, where they were cured for 28 days. Different sizes of the concrete samples were employed to meet the requirements of various standard methodologies.

To determine the specific surface area and particle size distribution, a dry method was utilised with a Cilas 1090 LD analyser, covering a range of 0.01 to 500 µm and using air as the carrier gas. The particles were ultra-



a) cement b) concrete sludge c) glass sludge

Figure 1. Particle size distribution: a) cement; b) concrete sludge; c) glass sludge

Table 3. Concrete recipes for 1 m$^3$.

| Mixes | 1 | 2 | 3 | 4 | 5 | 6 | 7 |
|---|---|---|---|---|---|---|---|
| Cement (kg) | 400 | 380 | 360 | 340 | 320 | 300 | 280 |
| Sand 0/4 (kg) | 845.5 | 845.5 | 845.5 | 845.5 | 845.5 | 845.5 | 845.5 |
| Crushed dolomite, 4/16 (kg) | 1040 | 1040 | 1040 | 1040 | 1040 | 1040 | 1040 |
| Superplasticizer (kg) | 2.8 | 2.8 | 2.8 | 2.8 | 2.8 | 2.8 | 2.8 |
| Air entraining agent (kg) | 0.4 | 0.4 | 0.4 | 0.4 | 0.4 | 0.4 | 0.4 |
| Glass sludge (kg) | 0 | 20 | 40 | 60 | 80 | 100 | 120 |
| Dry concrete sludge (kg) | 44.5 | 44.5 | 44.5 | 44.5 | 44.5 | 44.5 | 44.5 |
| Wet concrete sludge (kg) | 16 | 16 | 16 | 16 | 16 | 16 | 16 |
| Crystallising admixture (kg) | 4 | 4 | 4 | 4 | 4 | 4 | 4 |
| Water (kg) | 144 | 144 | 144 | 144 | 144 | 144 | 144 |
| w/b | 0.4 | 0.4 | 0.4 | 0.4 | 0.4 | 0.4 | 0.4 |

sonically dispersed until a 12 % particle size distribution was achieved. The measurement duration was 60 seconds, and data processing along with equipment control was handled using a Fraunhofer operating system.

An X-ray structural analysis was performed with a Bruker AXS D8 ADVANCE X-ray diffractometer (XRD), employing CuKα radiation, a Ni filter, a detector step of 0.02°, and an intensity measurement span of 0.5 seconds at an anode voltage (Ua) of 40 kV and current (I) of 40 mA. The accuracy of the XRD measurements was set to $2\theta = 0.01^{\circ}$.

The X-ray fluorescence spectroscopy was carried out using a Bruker X-ray S8 Tiger WD spectrometer, which utilised an Rh target X-ray tube with an anode voltage of up to 60 kV and a current of up to 130 mA. The samples were analysed in a helium atmosphere, and measurements were conducted using the SPECTRA Plus QUANT EXPRESS method. The microstructures of the materials were examined with a scanning electron microscope (SEM) (SEM JEOL JSM-7600F).

To assess the porosity of the samples, the water absorption was measured. Initially, the concrete samples were placed in an oven at a temperature of 100 ± 5 °C to ensure complete drying, then naturally cooled to room temperature for weighing ($w_1$). Subsequently, the cubes were submerged in water at 20 ± 2 °C for 48 hours. After soaking, the samples were taken out, the surface water was wiped off, and then they were weighed again ($w_2$). The averaging of the weighing results was based on three samples, and the final water absorption was calculated using Equation (2) provided below [71].

$$K = \frac{w_2 - w_1}{w_1} \tag{2}$$

To assess the water absorption kinetics, a sorptivity index was calculated. The concrete samples were dried in a drying oven at 100 ± 5 °C. The dried samples were removed and cooled to room temperature, and the cooled samples were coated with paraffin on all sides. The prepared samples were placed on two crossbars and a basin was filled with water to submerge the concrete samples by 5 mm. All the samples were weighed after 5, 10, 20, 30, 60, 180, 360, and 1440 minutes of immersion. Subsequently, the sorptivity index was calculated using Equation (3) [72].

$$\frac{Q}{A} = k\sqrt{t} \tag{3}$$

where $Q$ – the volume of absorbed water (mm³), $A$ – the surface area interacting with water (mm²), $k$ – the capillary absorption coefficient (mm/s$^{0.5}$), $t$ – the time (s).

Other concrete properties were evaluated in accordance with the relevant standards: LST EN 12350-7 for measuring entrained air in fresh concrete [73], LST EN 12390-7:2019 for determining the density of hardened concrete [74], LST EN 12390-3:2019 for assessing the compressive strength [75], LST 1428-17:2016 for analysing concrete frost resistance using the volume freezing and thawing method [76], and CEN/TS 12390-9:2016 for evaluating the resistance to cyclic freezing and thawing in the presence of de-icing salts [77].

Following the volume freezing method, the internal structure of the samples was assessed after varying numbers of freeze-thaw cycles. The degree of internal structural deterioration was calculated using Equation 4 [78]. To analyse and quantify the internal damage, the ultrasonic wave velocity was measured. This ultrasonic wave velocity was recorded for all the samples, and the average value from three samples was computed.

$$D_n = 1 - \frac{V_n^2}{V_0^2} \tag{4}$$

where $D_n$ – the degree of internal damage of the samples after a certain number of freeze-thaw cycles (%), $V_n$ – the ultrasonic wave velocity of the samples after a certain number of freeze-thaw cycles (km·s$^{-1}$), and $V_0$ – the ultrasonic wave velocity of the samples before frost resistance testing (km·s$^{-1}$).

## TEST RESULTS AND ANALYSIS

The microstructure of the glass sludge waste was analysed using an X-ray structural analysis, with the findings illustrated in Figure 2. The X-ray diffraction pattern obtained reveals only peaks corresponding to calcite. These results suggest that the waste predominantly consists of amorphous and fine crystalline materials that cannot be detected by the X-ray analysis. The findings closely resemble the composition of glass, which is primarily made up of approximately 70 % amorphous silicon dioxide, along with smaller proportions of sodium and calcium oxides [79]. The presence of calcite crystallisation observed in the XRD results indicates the carbonation of calcium.

Microstructural studies of the glass sludge were carried out using the SEM method, with the results displayed in Figure 3. The images reveal that the material comprises irregularly shaped smaller and larger crystals, with the smaller, rounder crystals identified as calcite minerals. These calcite minerals encase the larger glass

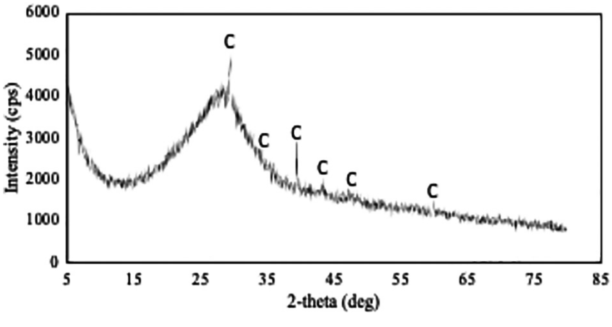
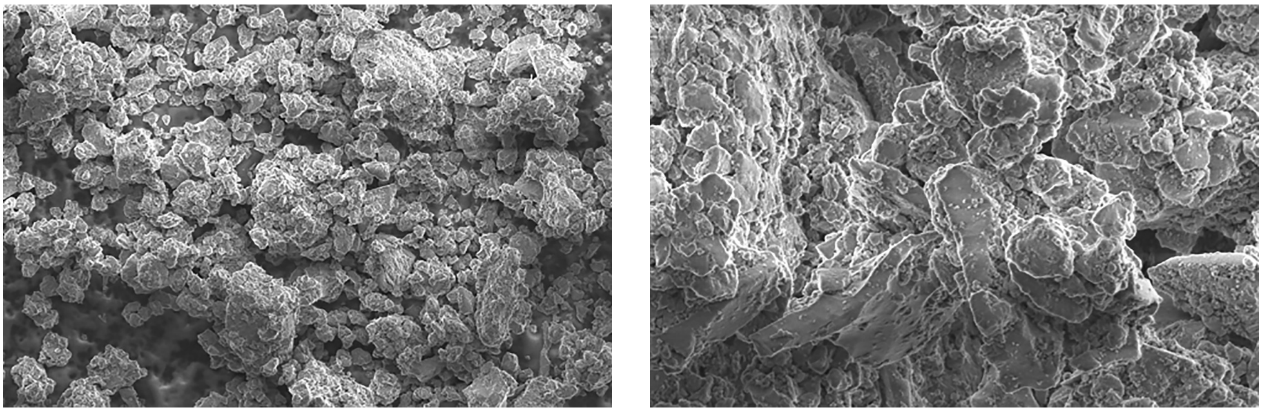


Figure 2. X-ray of glass sludge. C – calcite ($CaCO_3$).



a) ×2000 magnification

b) ×10000 magnification

Figure 3. Microstructure of the glass sludge.

particles. This structural arrangement may enhance the adhesion between the cement and glass slurry particles. The findings support the conclusions drawn from the X-ray structural analysis conducted earlier.

To analyse the mineral structure of the crystallising additive, an X-ray structural analysis was conducted, with the results shown in Figure 4. The analysis revealed that the crystallising additive contains 32.7% sodium carbonate, which can promote the hydration process of cement, especially the hydration of $C_3S$ minerals. According to the literature, an additional amount of $C_3S$ is incorporated into the crystallising additive to enhance the hydration and improve the early strength of the concrete. It is also noted that sodium carbonate ($Na_2CO_3$) accelerates the development of the early strength in the kinetics of tricalcium silicate hydration reactions [80].

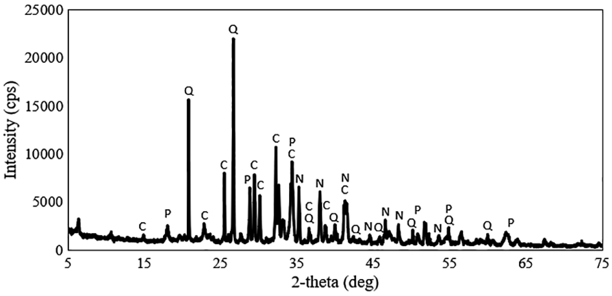
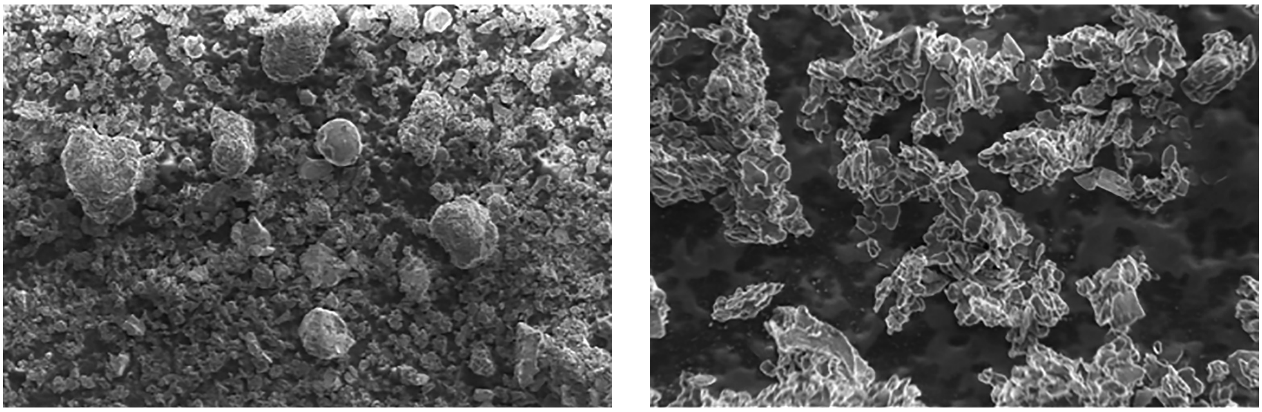


Figure 4. X-ray of the crystallising additive; N – sodium carbonate ($Na_2CO_3$), C – tricalcium silicate ($Ca_3SiO_4O$), Q – quartz ($SiO_2$), P – portlandite ($Ca(OH)_2$).

The second most prevalent compound identified was quartz, comprising 31.4 % of the mixture. The third most abundant mineral was tricalcium silicate, which accounted for 29.4 %, while the smallest proportion was portlandite, making up 6.2 % of the additive.

The crystallising admixture was analysed using a scanning electron microscope, with the resulting images shown in Figure 5. In image (a), taken at a magnification of 1000 times, the crystallising admixture is composed of irregularly shaped particles, interspersed with round-shaped crystals. These round crystals may potentially be quartz particles, which were also identified in the X-ray structural analysis. Image (b) features a magnification of 6500 times, revealing irregularly shaped crystals along with small plate-like structures, which could likely be portlandite crystals.



a) ×1000 magnification

b) ×6500 magnification

Figure 5. Microstructure of the crystallising additive.

To assess the impact of the glass sludge on the amount of entrained air in the concrete mixture, measurements of the entrained air content were taken. The results are illustrated in Figure 6. The findings indicate a slight increase in the amount of entrained air with higher glass sludge content in the mixtures, up to 15 %. Specifically, by replacing 15 % of the cement with the glass sludge, the entrained air content rose by 10.2 % compared to the control sample. As the glass sludge content increased further, the air content began to increase more sharply; in the mixture containing 30 % glass sludge, the entrained air increased by nearly 47 % compared to the control sample. This increase in the air content is attributed to the retention of air on the surfaces of the glass particles, which prevents it from escaping from the concrete mixture [81]. Additionally, the presence of a layer of calcite particles on the glass surfaces may enhance the trapping of air bubbles.

The density of the hardened concrete samples was also measured, and these density results are displayed in Figure 6. There is a correlation between the density and the amount of observed entrained air. As the glass sludge content increases – given that it has a lower density than cement – the overall density of the samples decreases. The density shows a slight reduction with the substitution of cement for glass sludge, specifically experiencing a decrease of 56 kg·m$^{-3}$ in the sample where 15 % of the cement was replaced. In the sample with 30 % glass sludge, the density dropped by 141 kg·m$^{-3}$.

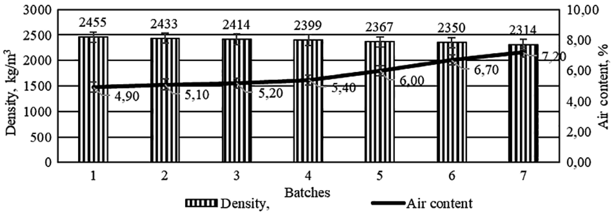


Figure 6. The relationship between the density and air entrainment of the concrete specimens and the quantity of glass sludge used.

Water absorption is a critical property of concrete that influences its durability; thus, this study assessed the water absorption levels. The results are illustrated in Figure 7, showing absorption values ranging from 2.42 % to 2.63 %. The lowest water absorption was observed in the sample where 20 % of the cement was replaced with glass sludge, with this sample showing a 7.3 % reduction in absorption compared to the control sample.

A notable trend emerged: as the percentage of glass replacing cement increased to 20 %, the water absorption decreased; however, further increases in the replacement began to increase the absorption levels. This phenomenon is hypothesised to occur because the sample with 20 % glass generates fewer open pores, attributed to the synergistic effect of the crystallising admixture and glass. It is thought that the crystallising admixture interacts with the carbonates on the surface of the glass, leading to the formation of hydration products that promote pore healing, resulting in a higher number of closed pores. When more than 20 % of the cement is replaced, water absorption starts to gradually rise, likely due to an increased amount of entrained air, which limits the crystallising admixture's ability to sufficiently facilitate the formation of closed pores. An additional reason is the high pozzolanic activity of waste glass. During the hydration process, new formations are created, which filled the pores. This may be the reason for the decreasing water absorption. The same tendencies were observed by researchers [82].

In comparison, the sample containing 30 % glass sludge exhibited slightly higher water absorption than the control sample that did not contain glass sludge.

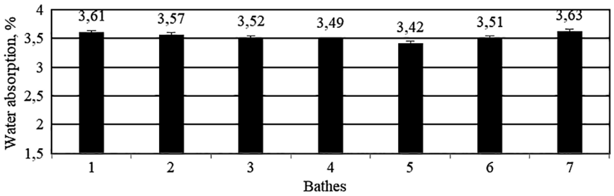


Figure 7. Water absorption of the different concrete mixes.

Like other typical porous materials, concrete has an internal structure characterised by a complex network of pores. These pores play a crucial role in withstanding freeze-thaw cycles, as they facilitate water transport during freezing while also maintaining thermodynamic and pressure equilibrium within the concrete. Although high water absorption can diminish the frost resistance, lowering the permeability of the internal pore network – along with reducing its connectivity and tortuosity – can help decrease the water absorption [83]. The results for the sorptivity index are shown in Figure 8. The findings indicate that the sorptivity index decreases when up to 20 % of the cement is replaced with glass sludge. However, when more than 20 % of the cement is substituted, the sorptivity index begins to rise, approaching levels like those of the control sample. This decrease in the sorptivity index can be attributed to the increased difficulty of water penetrating the internal structure through closed and gel pores, especially after fine glass particles occupy the open pores in the concrete [84], potentially leading to the formation of additional open pores. For the sample with 20 % of the cement replaced by glass sludge, the sorptivity index after 1440 minutes (or 24 hours) was measured at $0.72 \times 10^{-2}$ cm/min$^{0.5}$, which is 13 % lower than that of the control sample. This improvement is likely due to the formation of a denser concrete structure, as the glass particles effectively fill various voids and react with the crystallising admixture. This synergistic effect increases the proportion of closed pores in the internal pore structure, resulting in a more refined composition and better resistance to water permeability [58].

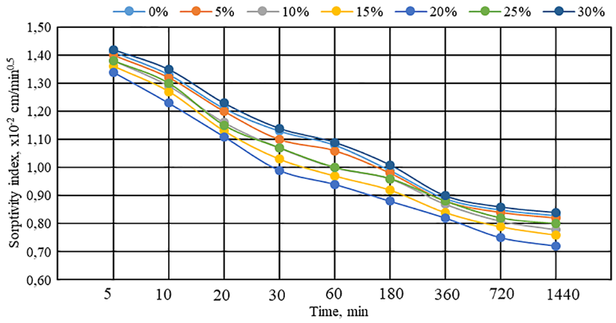


Figure 8. Association between the sorptivity index and the amount of glass sludge.

To confirm the above assumption, the porosity parameters of the hardened samples were assessed, with the results shown in Figure 9.

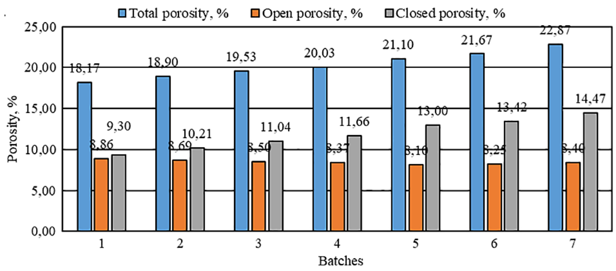


Figure 9. Relationship between the porosity parameters and the quantity of replaced cement to glass sludge in the concrete.

The results indicate that as the proportion of glass sludge replacing cement increases, both the total and closed porosity rise, while the open porosity only decreases up to the 20 % replacement of cement with glass sludge; beyond this threshold, the open porosity starts to increase. When 20 % glass sludge was incorporated into the mixture, the closed porosity rose by approximately 55 %, the total porosity increased by nearly 26 %, and the open porosity decreased by 8.5 % compared to the control sample. The rise in total porosity and open porosity can be attributed to the higher amounts of entrained air. However, the reduction in the open porosity with up to 20 % glass sludge replacement suggests that the crystallising admixture effectively minimises the open porosity while enhancing the quantity of the closed pores, despite the overall increase in the total porosity. Figures 10 and 11 further illustrate these findings, with Figure 10 depicting the relationship between the open and closed porosity, and Figure 11 showing the correlation between the water absorption and closed porosity.

The results indicate that when the cement is replaced with up to 20 % glass sludge, the ratio between the closed and open pores decreases proportionally. This confirms the hypothesis about the synergistic interaction between the carbonates present on the surface of the glass particles and the crystallising admixture, whose minerals encourage the formation of new compounds between the glass and cement particles and the growth of pores. The trend presented in Figure 11 reflects the effect of the glass content on the concrete structure; the greater number of open pores is not compensated by the new compounds formed due to the interaction of the carbonates present on the glass particles and the crystallising admixture. The number of open pores begins to dominate, and the total porosity increases.

The samples were evaluated for the compressive strength prior to conducting the frost resistance tests using the volumetric method, as well as after 200 and 300 freeze-thaw cycles. A 3 % NaCl solution was used as the immersion medium for the volumetric freezing method. The compressive strength results before and after freezing are illustrated in Figure 12. The data indicate that, as the percentage of glass sludge in the mixture increases to 20 %, the compressive strength improves; however, beyond this threshold, any further increases in the glass sludge led to a decline in the strength. Specifically, the compressive strength of the sample with 15 % of the cement replaced by glass sludge showed a 14.6 % increase compared to the control sample. This enhancement can be attributed to several factors: first, the fine glass particles serve as micro-fillers, occupying voids in the concrete structure and contributing to a denser microstructure [85]. Second, there is a synergistic interaction between the glass particles and the crystallising admixture, which leads to the formation of larger open pores through hydration products within the denser microstructure; a similar effect may also occur inside the closed pores. Third, the pozzolanic nature of the glass

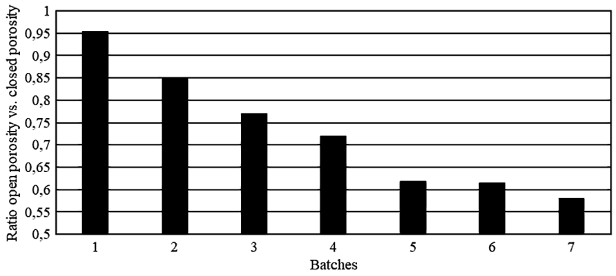


Figure 10. Ratio between the open and closed porosity in the concrete with different replacement amounts of cement to glass sludge.

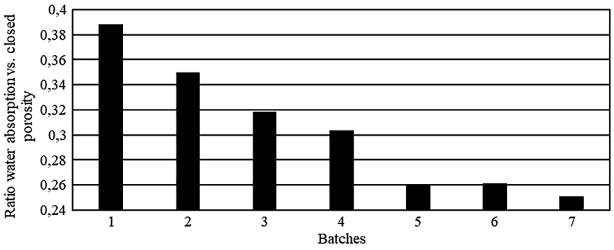


Figure 11. Ratio between the water absorption and closed porosity in the concrete with different replacement amounts of cement to glass sludge.

sludge can lead to the formation of CSH, which increases the compressive strength of the concrete [86].

When the cement replacement exceeds 20 %, the compressive strength begins to wane. The sample containing 30 % glass sludge exhibited a compressive strength slightly lower than that of the control sample. This reduction is likely due to an increase in the number of open pores and overall porosity, as well as the excessive presence of fine glass particles, which weaken the bonding forces among the particles within the structure due to the smoother surface of the glass particles [87].

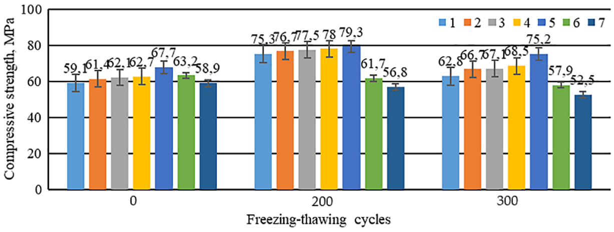


Figure 12. Relationship between the compressive strength and the quantity of glass sludge following volumetric freeze-thaw cycles.

After evaluating the compressive strength following 200 and 300 freeze-thaw cycles, a similar pattern was observed in comparison to the control samples. In the samples where up to 20 % of the cement was substituted with glass powder, no reduction in strength was detected after either 200 or 300 cycles. Conversely, in the samples containing more than 20 % glass, a significant decrease in strength was noted after the freeze-thaw cycles. This decline may be attributed to the high porosity, which can result in increased damage and cracking. Such porosity levels considerably diminish the samples' resistance to the effects of these cycles. The changes in strength following 200 and 300 freeze-thaw cycles are detailed in Table 4.

As indicated by the changes in the compressive strength following the previous freeze-thaw cycles, the samples that had up to 20 % of the cement replaced with glass sludge only demonstrated a strength increase after 200 freeze-thaw cycles, averaging around a 23 % gain across all the samples. The ratio of glass sludge to the crystallising additives likely supports the ongoing formation of new products that repair the structure. After 300 freeze-thaw cycles, the strength of these same samples with up to 20 % glass sludge also did not decrease compared to the unfrozen samples, achieving an average strength increase of approximately 8.7 %. This enhancement is attributed to the glass sludge contributing to a denser concrete structure [85], facilitated by the formation of new products between the glass and crystallising additives. The larger specific surface area of the glass powder enhances the concrete matrix's strength due to improved bonding between the cement paste and the glass powder [58]. When evaluating the degradation of the internal structure using ultrasonic pulse velocity, the changes in the internal structure after 200 and 300 freeze-thaw cycles were analysed. The results are shown in Figure 13. It was noted that compositions with up to 20 % glass powder exhibited less internal damage during the freeze-thaw cycles compared to the control sample. After 300 cycles, the sample containing 20 % glass powder experienced 82 % less internal damage compared to the control sample. This improvement could be attributed to a more refined concrete structure with smaller and closed pores, enhanced internal porosity, interconnected pores, and a reduced number of capillary pores, which contribute to the better compaction of the concrete mixture and increased frost resistance. Additionally, according to the osmotic pressure theory, smaller pores freeze at lower temperatures [88]. In contrast, compositions with more than 20 % glass sludge exhibited greater internal structural damage after 200 freeze-thaw cycles than the control sample, and, after 300 cycles, the internal structure deterioration in the concrete without the glass powder was among the most significant, only surpassed by the mixture containing 30 % glass powder. This situation likely arises from the excessive number of glass particles, which increased the open porosity, enhanced the water absorption, and resulted in a higher occurrence of pores and micro-cracks within the internal structure. This can be explained by the limited energy

Table 4. Variation in the compressive strength following volumetric freeze-thaw cycles.

| Mixes | Alteration in the compressive strength (%) | |
|---|---|---|
| | following 200 cycles | following 300 cycles |
| 1 | 27.4 | 6.3 |
| 2 | 24.9 | 8.6 |
| 3 | 24.8 | 8.1 |
| 4 | 21.1 | 9.3 |
| 5 | 17.1 | 11.1 |
| 6 | –2.8 | –8.4 |
| 7 | –3.6 | –10.8 |

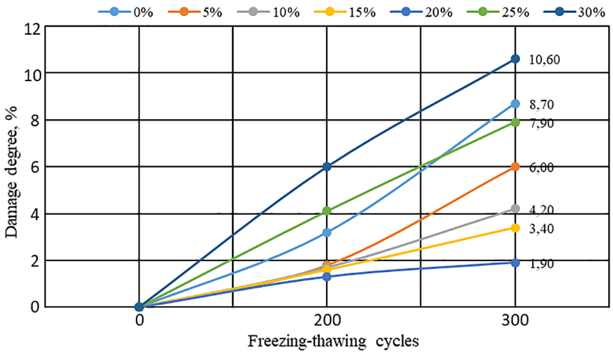


Figure 13. Relationship between the degree of damage and the quantity of replaced cement to glass sludge in the concrete following 200 and 300 volumetric freeze-thaw cycles.

absorption and deformation resistance of the glass particles, leading to the continuous development and exacerbation of internal pores and cracks under the constant stresses induced by freezing and thawing, severely compromising the frost resistance.

Mass losses are a critical factor in evaluating frost resistance. The results obtained are depicted in Figure 14. During freezing, concrete generally tends to absorb more water, which results in an increase in the sample's mass. This increase in mass due to water absorption can somewhat offset the losses incurred from spalling or flaking in concrete, a trend observed in the control concrete sample. However, it can be noted that the concrete containing glass sludge exhibited greater mass losses. With up to 30 % of the cement replaced by glass sludge, a consistent increase in the mass loss was recorded. Specifically, the mass loss of the sample with 20 % glass sludge was six times greater than that of the control sample after 56 freeze-thaw cycles. This observation may be attributed to the fact that the samples with glass sludge, given the dense structure of the glass, absorb less water, resulting in a lower compensatory increase in mass to offset the losses from spalling.

A significant increase in the mass losses was noted in the mixtures containing 25 % and 30 % glass sludge, with the mass losses after 56 cycles being 16 and 21 times greater than that of the control sample, respectively. The mass losses were manifested as fine spalling, where small particles broke off around the cavities on the sample's surface. Additionally, there was evidence of delamination in the thin layer of cement paste, exposing the internal pores. The substantial deterioration was attributed to the excessive damage to the open porosity caused by the glass particles, leading to deeper water absorption and further damage.

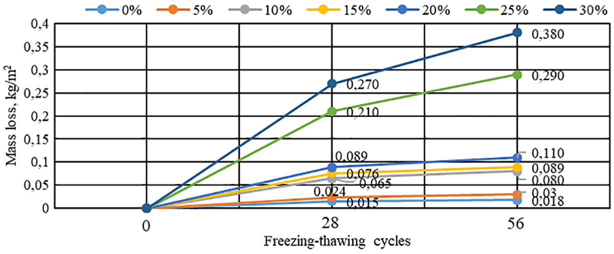


Figure 14. Relationship between the mass loss and the quantity of replaced cement to glass sludge in the concrete following the scaling tests.

## CONCLUSIONS

These studies highlight several aspects of the properties and durability of sustainable concrete mixtures, particularly their frost resistance. Tests were conducted on various parameters, including the entrained air content, density, porosity, frost resistance through volume methods, scaling methods, mass loss assessment, sorptivity index, and changes in the internal structure as influenced by the amount of utilised glass sludge.

The analysis of the impact of glass sludge on the entrained air content in the concrete mixture revealed that with 15 % or less glass sludge, there was no significant effect on the amount of entrained air. However, when higher amounts of glass sludge were used, the entrained air content began to rise significantly. This trend was mirrored in the concrete density studies, showing that when more cement is replaced with glass sludge, the lower the density of the hardened samples.

The results regarding water absorption and the sorptivity index demonstrated that replacing up to 20 % of the cement with glass sludge led to a decrease in the water absorption, resulting in a slower water uptake by the concrete. Conversely, when more than 20 % of the cement was substituted with glass sludge, both the water absorption and the rate of absorption increased.

Increasing the glass sludge content in concrete mixtures results in a higher total porosity due to the inclusion of entrained air. However, the synergistic interaction between glass sludge and the crystallising additives fosters pore healing, which enhances the closed porosity and reduces the open porosity when the glass sludge content remains at or below 20 %. Beyond this point, when the glass sludge content rises to 30 %, the open porosity begins to increase.

The measurements of the compressive strength prior to freeze testing revealed that the hardened concrete samples with up to 20 % glass sludge exhibited increased compressive strength, while higher glass sludge additions negatively impacted the strength, leading to a decline.

After conducting 200 and 300 volume freeze-thaw tests, it was found that samples with up to 20 % glass sludge showed an increase in the compressive strength. Notably, after 300 freeze-thaw cycles, these samples demonstrated a superior strength gain. In contrast, the samples containing more than 20 % glass sludge showed no strength gain after the freeze-thaw cycles and displayed significantly reduced strength.

An assessment of the internal structural deterioration indicated that mixtures with up to 20 % glass sludge experienced less internal damage during freeze-thaw cycles, attributed to the synergistic effects between the glass and crystallising additives. However, in mixtures with more than 20 % glass sludge, the internal structural damage due to increased porosity after 200 freeze-thaw cycles exceeded that of the control sample. After 300 cycles, the most significant internal structural damage was seen in the concrete sample containing 30 % glass sludge.

Following the freeze-thaw tests and mass loss evaluations, it was evident that concrete with glass sludge recorded greater mass losses. A direct correlation was noted for the samples with up to 20 % glass sludge, which exhibited a proportional increase in the mass losses, while mixtures containing 25 % and 30 % glass sludge demonstrated a drastic rise in mass losses.

In summary, the results indicate that glass sludge does not impair the frost resistance properties and may even enhance them slightly due to the synergistic effects of the glass and crystallising additives on the closed porosity when up to 20 % of the cement is replaced with glass sludge. However, using larger amounts leads to decreased frost resistance, making the concrete more susceptible to the effects of freeze-thaw cycles and de-icing salts.

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