



ORIGINAL ARTICLE

Assessment of properties of ultra-high performance cementitious composites with glass powder waste

Avaliação das propriedades de compósitos cimentícios de ultra-alto desempenho com resíduos de pó de vidro

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Received 14 March 2022

Accepted 25 October 2022

Abstract: Novel developments on concrete technology such as high and ultra-high-performance concrete (HPC and UHPC) are notorious by its high consumption of Portland cement. Supplementary cementitious materials have been used as partial replacement of Portland cement aiming to minimizing clinker content, optimizing the use of binders, reducing CO₂ emissions, and increasing durability. Waste glass powder could be an alternative due to its silica-rich nature and wide availability. This work aims to assess the influence Portland cement substitution by finely ground waste glass powder in contents of 10%, 20%, 30% and 50% on physical and mechanical behavior of ultra-high-performance cementitious composites (UHPC). Results indicates the use of glass powder as substitution up to 50% of Portland cement does not significantly affect the analyzed properties at 28 days.

Keywords: cementitious composite, ultra-high performance, glass powder.

Resumo: Novos desenvolvimentos na tecnologia do concreto, como concretos de alto e ultra-alto desempenho (CAD e UHPC) são notórios não apenas pela elevada resistência mecânica e durabilidade, como também pelo alto consumo de cimento Portland. Materiais cimentícios suplementares têm sido utilizados como substitutos parciais do cimento Portland visando minimizar o consumo de clínquer, otimizando o uso de aglomerantes, reduzindo as emissões de CO₂ e aumentando a durabilidade. O pó de vidro residual pode ser uma alternativa viável devido à sua natureza rica em sílica, ampla disponibilidade e baixo custo. Este trabalho tem como objetivo avaliar a influência da substituição do cimento Portland por pó de vidro residual finamente moído em teores de 10%, 20%, 30% e 50% no comportamento físico e mecânico de compósitos cimentícios de ultra alto desempenho (UHPC). Os resultados indicam que o uso de pó de vidro como substituição de até 50% do cimento Portland não afeta significativamente as propriedades analisadas aos 28 dias.

Palavras-chave: compósito cimentício, ultra-alto desempenho, pó de vidro.

How to cite: S. M. Soares, T. O. G. Freitas, A. Oliveira Júnior, F. G. S. Ferreira, and J. A. A. Salvador Filho, "Assessment of properties of ultra-high performance cementitious composites with glass powder waste". *Rev. IBRACON Estrut. Mater.*, vol. 15, no. 6, e15612, 2022, <https://doi.org/10.1590/S1983-41952022000600012>

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Financial support: None.

Conflict of interest: Nothing to declare.

Data Availability: The data that support the findings of this study are openly available in <https://repositorio.ufscar.br/handle/ufscar/14126?show=full>.



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1 INTRODUCTION

The use of cement as the main material in civil construction is associated with high environmental pollution and high-energy consumption. Given the limitations of fossil fuel resources and strict environmental regulations, to develop sustainable cement production is an urgent initiative to be followed up [1]. Some of the alternatives for decreasing the use of finite resources in clinker production are energy efficiency improvements, waste heat recovery, reduced clinker/cement ratio, alternative raw materials, and substitution of fossil fuels by alternative energy sources [2].

Although UHPCC production uses high cement consumption, the performance achieved by these composites is proportionally superior to traditional cementitious composites regarding the consumption of materials for its production. One of the most efficient alternatives for reducing Portland cement consumption in the UHPCC is the use of supplementary cementitious materials, consisting mainly of pozzolanic materials. Industrial tailings with pozzolanic properties contributed to the improvement of cementitious composites. minimizing clinker content, optimizing the use of binders, reducing CO₂ emissions, and increasing durability.

According to Jokar and Mokhtar [1], the ground glass powder is potentially used as a pozzolanic material [3]–[12]. About 980 thousand tons of glass are produced per year in Brazil, and 53% of this production is not recycled [13]. This glass waste also represents about 3% of all municipal waste produced in Brazil [14].

Glass is a material composed mainly of amorphous silica and present pozzolanic properties when finely ground [7]–[9]. According to Patel et al. [15], glass particles up to 75 µm can replace Portland cement in amounts from 10% to 25%, showing satisfactory results in the production of cementitious composites, and several authors attributes improvements in mechanical properties to pozzolanic reactions from glass powder [3]–[12].

Pozzolanic materials reduce the strength gain rate of cementitious composites at the early age, so that composites containing pozzolanic material, such as glass, acquire a slow strength and should be analyzed at older ages [16]. According to Li et al. [3], when the waste glass powder (WGP) particle size was 20–44 µm, the compressive strength was lower than that of the control group at 7 days of curing, whereas it was 3.5% and 9.6% higher than that of the control group at 28 d and 90 days curing, respectively, with a WGP content 20%. According to Raydan et al. [4], glass powder with particle size < 75 µm can exhibit pozzolanic characteristics inhibiting the ASR gel formation, improving the durability and strength performance. This behavior was confirmed by Higuchi et al. [8], Fanijo et al. [9] and Jiang et al. [10].

This work aims to investigate the effects of the replacement of Portland cement by glass powder at high levels, up to 50% on the mechanical behaviour and microstructure ofUHPC mixtures. The use of glass powder as Portland cement replacement in ultra-high performance cementitious composites is relatively recent. In addition, the replacement of 50% cement by glass powder and its influence on mechanical properties is not yet established in the scientific community.

2 MATERIALS AND METHODS

2.1 Materials used

In this work were used Brazilian Portland cement type CP V ARI, silica fume, glass powder, quartz sand, superplasticizer admixture, shrinkage reducing admixture and water to produce cementitious composites.

Amber glass bottles were used to minimize uncontrolled variations in chemical composition of the cementitious composites mixtures. The glass powder used is the bypass in the 200-mesh sieve, obtained from grinding in a ball mill for 14 hours and mechanically sieved. The fine natural aggregate used is quartz from a riverbed and manually sifted through a 1.2 mm aperture mesh.

The authors Wille and Boisvert-Cotulio [17] used fine aggregate up to 1.2 mm to obtain high strength composites. According to Azme and Sha [18], the use of smaller aggregates reduces their heterogeneity and promotes the densification of the mixture. In Figure 1 are presented the dry materials used.



Figure 1. From left to right: fine aggregate, glass powder, silica fume, and cement.

Were performed laser granulometry, specific gravity [19] and specific surface [20] trials to analyze the physical characteristics of the Portland cement, silica fume and glass powder. The fine aggregate specific gravity was determined according to NBR 16916 [21]. In Figure 2 are presented particle size distribution of Portland cement, silica fume, and glass powder determined by laser granulometry and the characterization of fine aggregate by NBR 17054 [22].

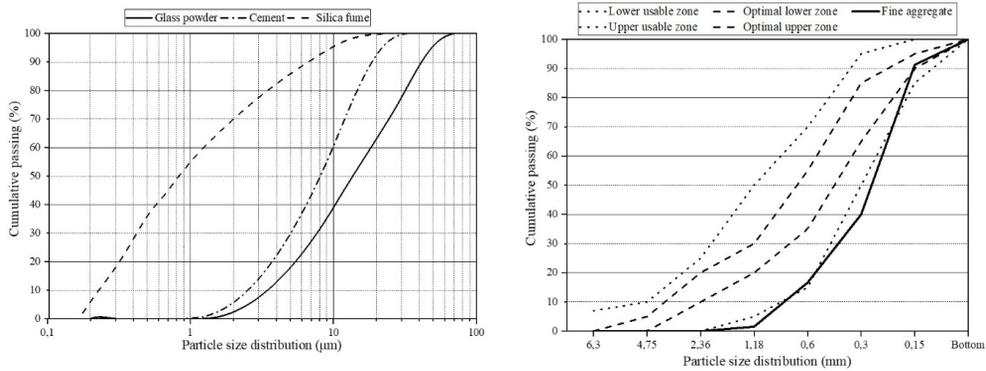


Figure 2. Particle size distribution results of the Portland cement, silica fume and glass powder (left) and fine aggregate (right).

The fine granulometry of the glass powder contributes to its pozzolanic activity. Matos and Sousa-Coutinho [23], Soliman and Tagnit-Hamou [24] and Pan et al. [25] used glass powder with particle size in d_{50} of 9 µm, 12 µm and 20 µm, respectively. Table 1 shows the physical properties values of Portland cement, silica fume, glass powder, and quartz sand.

Table 1. Physical properties of the materials.

Physical properties	Portland cement	Silica fume	Glass powder	Quartz sand
Specific gravity (g/cm ³)	3.16 ^a	2.25 ^a	2.55 ^a	2.56 ^b
Specific surface (m ² /kg) [*]	665	247	393	-
Water absorption (%)	-	-	-	0.64 ^b
Maximum dimension (mm) ^{**}	0.03	0.023	0.075	1.2

^{*} NBR 16372 [20]. ^{**} Maximum size determined by laser granulometry, except in quartz sand which was determined by NBR 17054 [22]. ^a NBR 16605 [19]; ^b NBR 16916 [21]

The chemical characterization of the fine materials was performed by X-ray fluorescence spectrometry assay, which allows the identification of the elements present in each material. Table 2 shows the percentage of chemical composition of cement, silica fume, and glass powder.

Table 2. Chemical composition of cement, silica fume, and glass powder.

Components	Portland cement (%)	Silica fume (%)	Glass powder (%)
Silica oxide (SiO ₂)	23.00	94.10	74.00
Ferric oxide (Fe ₂ O ₃)	2.49	< 0.5	0.42
Aluminum oxide (Al ₂ O ₃)	4.31	< 0.2	3.70
Calcium oxide (CaO)	61.40	< 0.2	9.10
Sulfuric anhydride (SO ₃)	2.97	-	-
Strontium Oxide (SrO)	0.27	< 0.2	0.04
Thorium Oxide (ThO ₂)	< 0.01	< 0.01	< 0.01
Potassium oxide (K ₂ O)	0.96	1.28	0.56
Sodium oxide (Na ₂ O)	-	-	11.00
Uranium Oxide (U ₃ O ₂)	< 0.01	< 0.01	< 0.01
Phosphorus oxide (P ₂ O ₅)	0.52	-	-
Chloride (Cl ⁻)	0.12	-	-
Magnesium oxide (MgO)	-	-	0.74
Loss on ignition	4.05	3.60	0.58

The presence of high levels of SiO_2 (74%) in glass powder is highly desirable for the development of the pozzolanic reaction. The chemical composition of binders is equivalent to those found by researchers such as Matos and Sousa-Coutinho [23], Soliman and Tagnit-Hamou [24], Pan et al. [25], Harbec et al. [26] and Ibrahim and Meadwad [27].

For mineralogical analysis, we used the X-ray diffraction (XRD) to characterize the mineralogical phases present in each material. Figure 3 shows the diffractograms of Portland cement, silica fume, and glass powder.

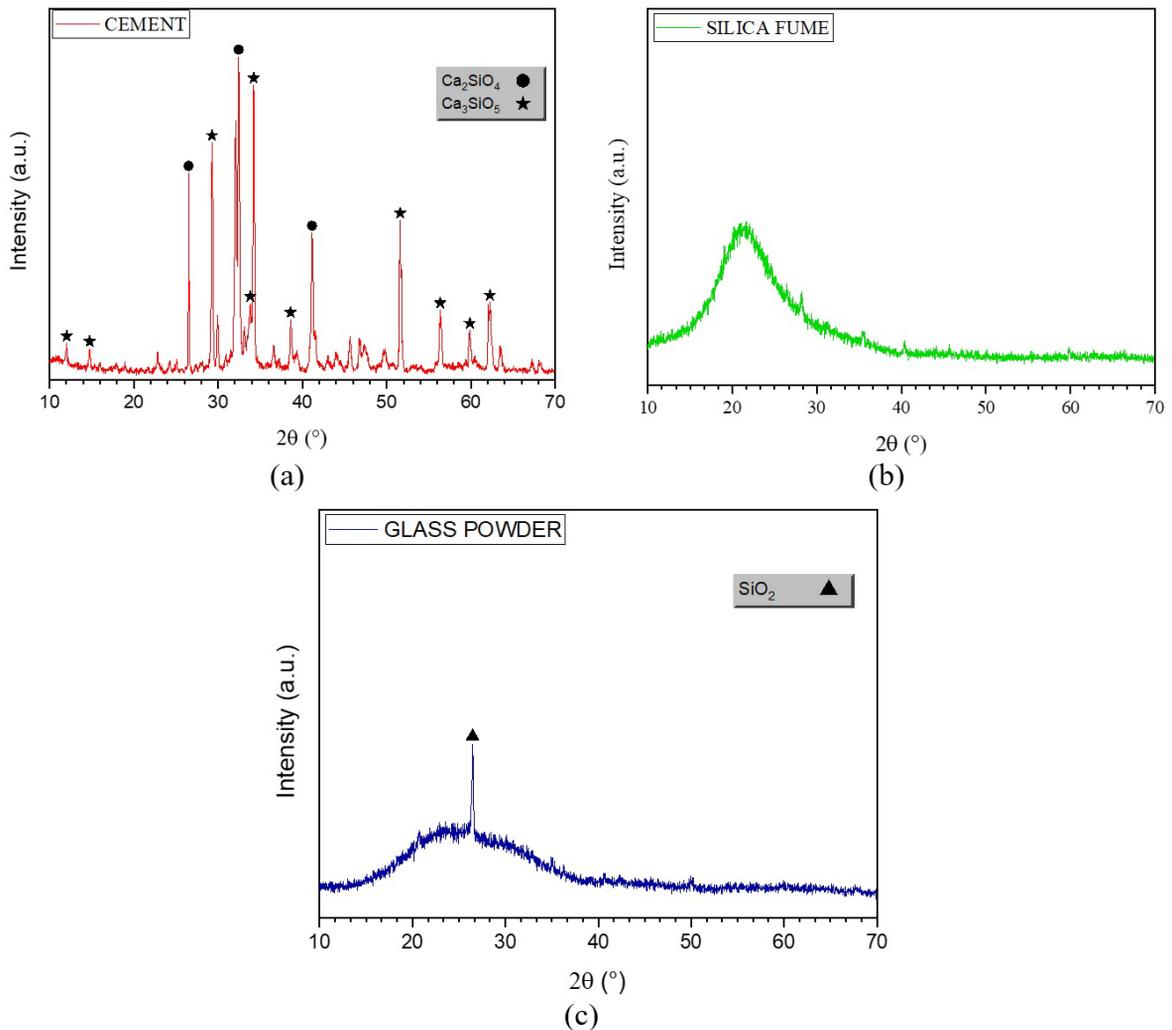


Figure 3. X-ray diffraction patterns for cement (a), silica fume (b) and glass powder (c).

In glass powder diffractometry, the material showed a large amount of amorphous silica, except for a crystalline quartz peak, probably incorporated into the material in one of the grinding steps consisted of using steel ball, concrete mixer, and flint stone ball mill (crystalline silica).

Elaqra and Rustom [28] identified contamination of glass powder by cement that was derived from material residues in the grinding equipment. For other authors such as Soliman and Tagnit-Hamou [24] and Ibrahim and Meadwad [27], the glass powder showed completely amorphous behavior in the DRX analysis.

The pozzolanic activity of the glass powder was evaluated by determining the lime performance at 7 days according to NBR 5751 [29]. It showed compressive strength of 4.97 MPa, below the limit of 6.0 MPa defined by the standard. By the modified Chapelle method, according to NBR 15895 [30] it presented calcium hydroxide fixation of 654 mg $Ca(OH)_2/g$. Raverdy et al. [31] and Hoppe Filho [32] established the limit of 330 mg $Ca(OH)_2/g$ from the modified Chapelle methodology.

By scanning electron microscopy (SEM) analysis performed on the FEI Company Inspect F50 equipment, we observed the morphology of the Portland cement, silica fume, and glass powder (Figure 4).

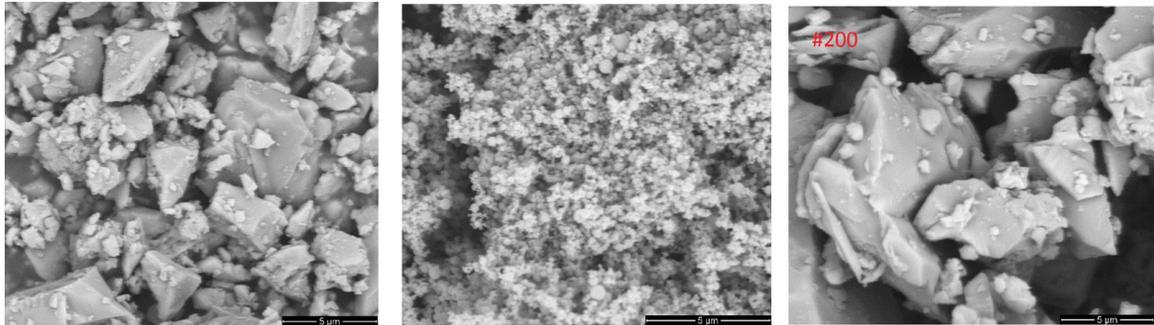


Figure 4. Photomicrographs from SEM of Portland cement (left), silica fume (center) and glass powder (right).

The morphology of Portland cement is similar to the morphology of glass powder, with angular particles of different sizes and shapes with well-defined and smooth surfaces, while the silica fume, highly thin material, presents regular and spherical particles.

A polycarboxylate based superplasticizer admixture with a content of 2.25% relative to binder volume and the shrinkage-reducing admixture was used at a content of 1%, also relative to binder volume.

The saturation point of the superplasticizer admixture is the level from which the effect of the admixture on the mixture is no longer significant and was determined by the Marsh cone test performed according NBR 7681 [33]. The 2.25% superplasticizer content was the optimal content.

The mini-slump test developed by Kantro [34] was performed to determine the compatibility between the groups with glass powder and the superplasticizer admixture used. The mini-slump test with the optimum content of 2.25% was performed to verify the compatibility of the superplasticizer admixture with the glass powder. The mini-slump test results for each analyzed paste of 0%, 10%, 20%, 30% and 50% glass powder content was calculated. The incorporation of glass powder did not interfere with the compatibility of the superplasticizer admixture with the binders.

Although the absolute values of scattering are different, the difference between the largest and smallest is not significant, only 7 mm, or approximately 2%. Thus, the superplasticizer was shown to be compatible with Portland cement and glass powder.

2.2 Cementitious composites mixtures

Five groups of cementitious composites with different glass powder contents were prepared. The water/binder ratio adopted was 0.18, with the addition of 8% silica fume concerning the Portland cement consumption of the reference group. The use of glass powder contents was carried out in the levels of 0%, 10%, 20%, 30%, and 50% in volumetric replacement to cement, represented by the nomenclature REF, VD10, VD20, VD30, and VD50, respectively. Table 3 shows the consumption of materials used in each mixture.

Table 3. Consumption of materials to produce the cementitious composite.

Group	Consumption of materials (kg/m ³)							Consistency (mm)
	Cement	Silica fume	Glass powder	Quartz sand	Water*	SP ⁽¹⁾	SR ⁽²⁾	
REF	1000	80	-	1074	181.28	24.3	10.8	379.5
VD10	900	80	81	1074	181.28	24.3	10.8	377.0
VD20	800	80	161	1074	181.28	24.3	10.8	375.5
VD30	700	80	242	1074	181.28	24.3	10.8	381.5
VD50	500	80	403	1074	181.28	24.3	10.8	382.5

*Corrected water value for 46% solids of superplasticizer. SP⁽¹⁾ = superplasticizer; SR⁽²⁾ = shrinkage reducer.

Replacing a product (cement) with a slightly thicker product (glass powder) ensures greater availability of water for fluidizing the mixture. However, water adsorption on the surface of the glass powder particles decreased the amount of water for fluidization, as commented by Pan et al. [25]. The combination of these two effects may have ensured equivalent dispersion values.

Soliman and Tagnit-Hamou [24] observed an increase in workability, with approximately 13% difference between the mixtures with 0% (reference) and 50% of glass powder substitution. The researchers attributed the higher workability to the greater availability of water for fluidization due to the low water absorption by the glass particles, dilution of the replacement cement, and the lower friction between the mix components and the smooth glass surface.

The consistency of cementitious composites was verified according to NBR 13276 [35], and the determination of specific gravity and air entrained content were evaluated according to NBR 13278 [36].

Cylindrical specimens 5 x 10 cm were molded, manually compacted, and cured in lime-saturated water. The tests to verify the compressive strength of UHPCC were performed according to NBR 5739 [37], at ages 2, 7, 28 and 91 days, and the tensile strength tests by diametral compression, performed according to NBR 7222 [38], at the age of 28 days.

The test for obtaining the static elastic modulus of the UHPCC was performed according to NBR 8522 [39], at the age of 28 days. Besides the determination of the static modulus of elasticity, the dynamic modulus of elasticity was also determined, which is a non-destructive test performed by the propagation of longitudinal waves obtained by ultrasonic pulses, according to ASTM E1876-21 [40] and ASTM C215-08 [41]. Capillary water absorption from the UHPCC was also determined following the recommendations of NBR 9779 [42].

3 RESULTS AND DISCUSSION

3.1 Specific gravity and air entrained content

Figure 5 presents the results of the UHPCC specific gravity and air entrained content test.

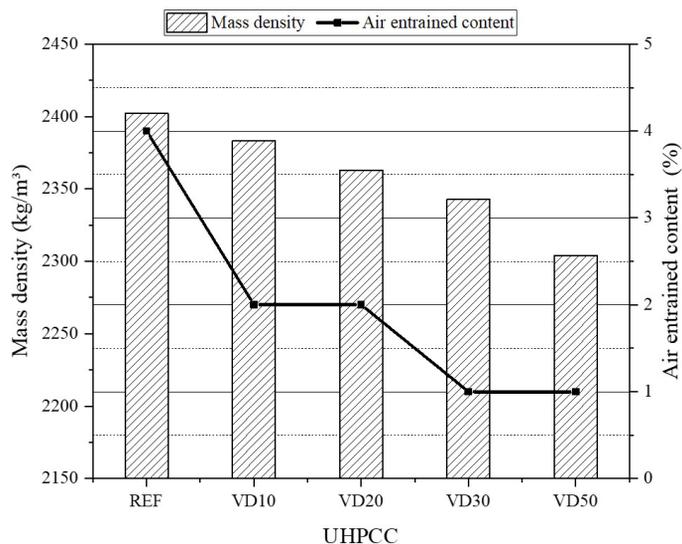


Figure 5. Specific gravity and air entrained content of UHPCC.

The density of the mixture decreased with increasing glass powder incorporation. The reference group presented higher specific gravity, 2338 kg/m³, and the 50% of glass powder group present 2273 kg/m³, approximately 4% difference, with the replacement of a denser product (cement) by a less dense product (glass powder) being the most likely hypothesis.

Although replacing denser products with less dense products reduces the density of the mixture, the lower presence of air entrained content in the 50% of glass powder group ensured that the difference to the reference group was not so pronounced.

A similar behaviour is described by Sharif et al. [43] which considers that blends with glass powder present the mixture densification by filler effect by air content reduction. Although Soliman and Tagnit-Hamou [24] observed a decrease in mass density of the mixture in minor values, with differences of approximately 3% between the reference line and the 50% glass powder line.

3.2 Mechanical and physical properties

Figure 6 shows the average results of the compressive strength test and capillary water absorption values at seven days and at 28 days of UHPCC.

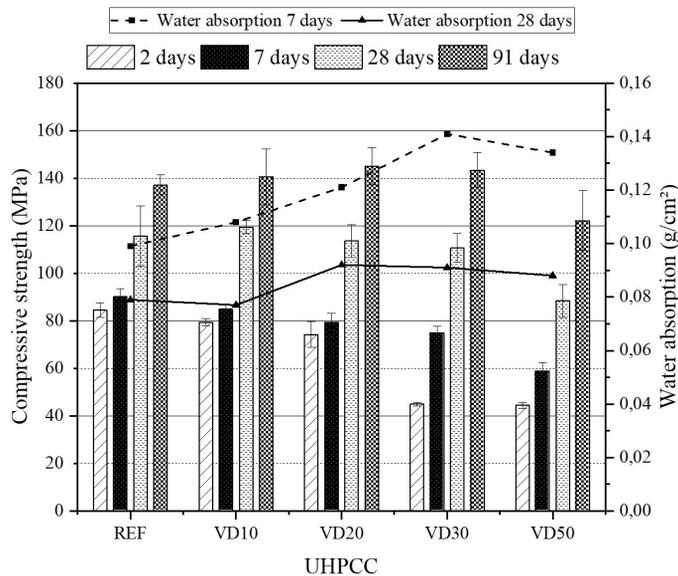


Figure 6. Compressive strength and water absorption of UHPCC.

Changes in the compressive strength development were sensitive to different glass powder incorporation amounts for different ages.

Researchers such as Matos and Sousa-Coutinho [23] analyzed contents of up to 20% of glass powder and did not observe compressive strength higher than the reference group at 28 days.

Soliman and Tagnit-Hamou [24] obtained compressive strength values at approximately 28 days for contents of 0%, 10%, 20%, and 30% of glass powder, being 170 MPa, 165 MPa, 170 MPa and 165 MPa, respectively.

Pan et al. [25], with a 5% glass powder content and 53 MPa compressive strength at 28 days obtained a higher value than the 47 MPa without glass powder group, while the 10% content presented 45 MPa compressive strength.

The better development of compressive strength at older ages can be attributed to the formation of C-S-H by the pozzolanic effect, which promotes the composite densification. The incorporation of a larger amount of glass powder as a substitute for cement directly changes the water/binder ratio, making more water available for a smaller proportion of cement, intensifying the formation of $\text{Ca}(\text{OH})_2$ which naturally reacts with the highest amount of pozzolanic material available by glass powder [24].

The differences observed at the age of 7 days could be explained by the incorporation of larger amounts of glass powder in the VD30 and VD50 groups, which in early ages could not densify the cementitious composite efficiently. At 28 days age, matrices with glass powder presents better developed hydration, with refined pores and lower water absorption through the capillary pores.

Matos and Sousa-Coutinho [23] observed that at up to 4.5 hours of capillary water absorption, samples with 0%, 10% and 20% glass powder at two months age presented no significant absorption differences. The authors attributed the small absorption difference to the Portland cement and glass powder particles size, both around 9 μm at d50, clogging the pores by physical effect.

Figure 7 shows the average results of the UHPCC splitting tensile strength test and the mean values of dynamic and static modulus of elasticity.

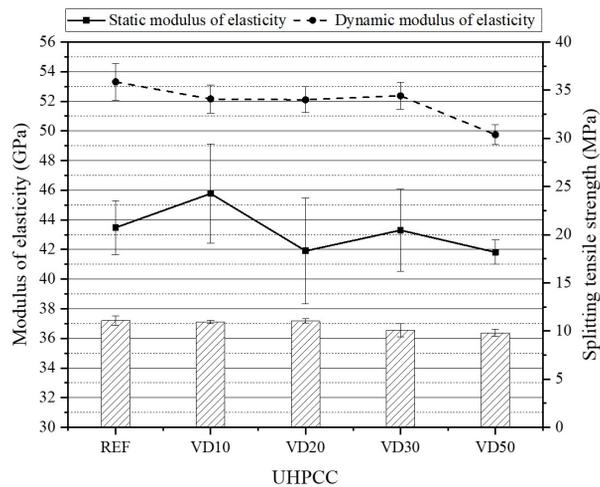


Figure 7. Splitting tensile strength and modulus of elasticity of UHPCC.

Using 0%, 10%, 20%, 30%, and 50% glass powder contents, Soliman and Tagnit-Hamou [24] observed slight differences between the modulus of elasticity values between the different groups, ranging from 50 to 55 GPa. The authors also commented that the glass powder particles naturally present high stiffness (in the order of 70 GPa) and may contribute indirectly to the stiffness of the composite.

In Figure 8 are presented three curves of empirical models by Abdelgader and Ben-Zeitun [44], Rajabi and Moaf [45], and Li et al. [46] and one new model that follows a polynomial equation curve, proposed to describe the correlation between compressive strength and tensile strength of ultra-high performance cementitious composites.

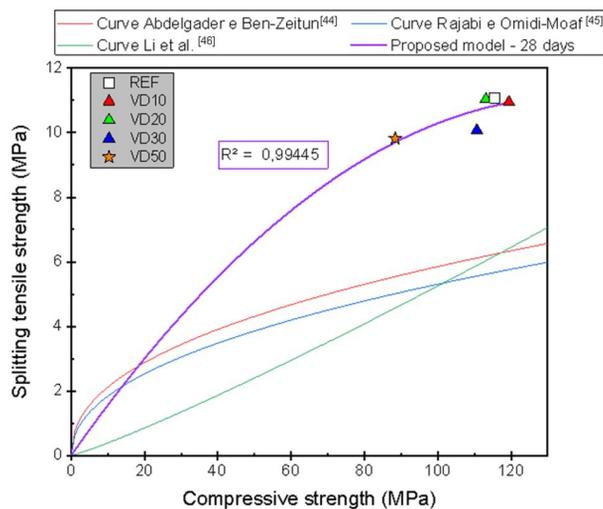


Figure 8. Splitting tensile strength versus compressive strength of UHPCC.

The prediction of polynomial curve presents an R^2 value of 0,99 that indicates the experimental results of the UHPCC are above the empirical curves. UHPCC presents a higher splitting tensile strength / compressive strength ratio compared to other composites, suggesting a greater potential for application in engineering due to the greater tensile strength.

3.3 Microstructural Analysis

In Figure 9 are presented the diffractograms of the REF and VD50 cement pastes mixtures at the ages of 28 days and 91 days. The dilution effect of cement promoted by its replacement with glass powder is mainly responsible for

reducing peaks on older ages. For the portlandite peaks between the angles of 15° to 20° and 45° to 50° at 28 days, mixtures REF and VD50 presented similar intensity since the glass powder participation in pozzolanic hydration still show minor differences. While at 91 days ages, the same portlandite peaks already show visible differences between mixtures REF and VD50, due it consumes by the glass powder pozzolanic reaction.

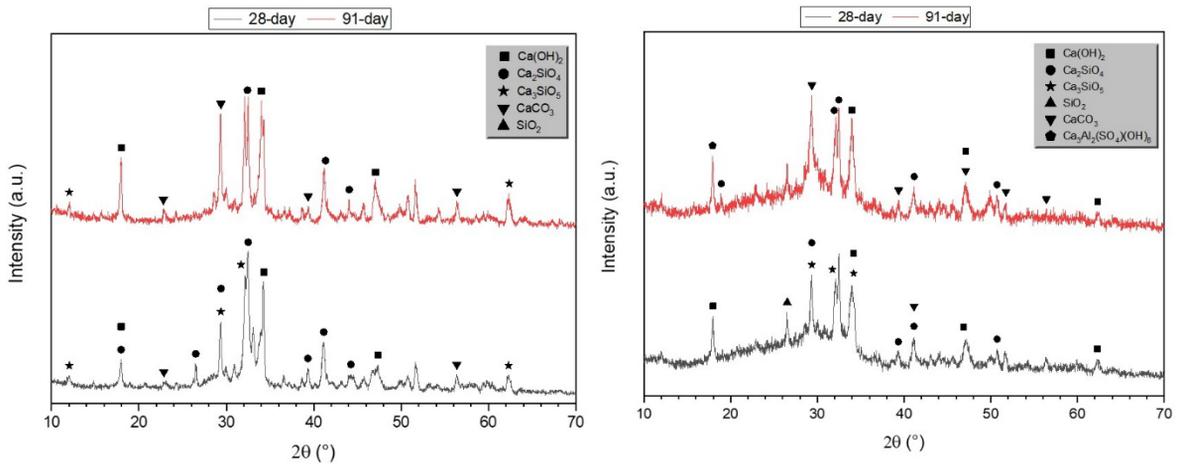


Figure 9. X-ray diffraction patterns at 28-day and 91-day of REF (left) and VD50 (right).

The presence of silica fume, a pozzolanic material, in UHPCC composition may influence the observations of the pozzolanic effect of glass powder. Silica fume presents a greater specific surface and much smaller particles which reacts more quickly and intensely than glass powder. These characteristics allow the consumption of much the previously available portlandite and which could react with glass powder.

In Figure 10 are presented the SEM microstructural analysis on REF and VD50 mixtures at 28 days and 91 days.

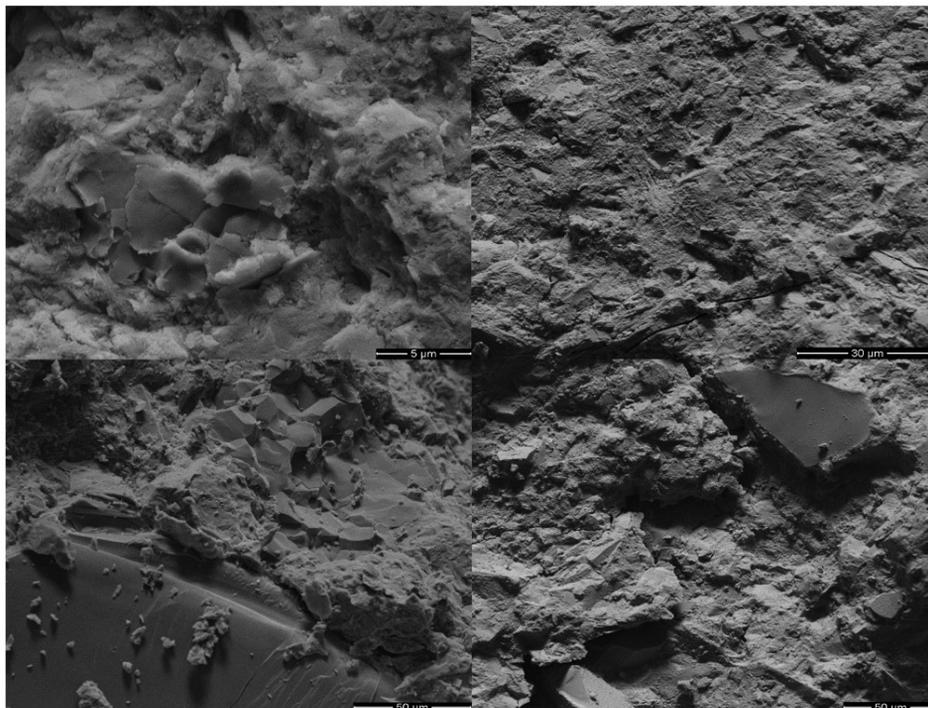


Figure 10. Photomicrographs from SEM of REF 28-day (upper left), REF 91-day (upper right), VD50 28-day (lower left) and VD50 91-day (lower right).

The presence of small cracks in cementitious matrix can be observed in the micrographs of the reference paste at 91 days. And, in addition to the presence of small cracks, the presence of glass dust particles is observed at the ages of 28 days and 91 days in VD50 paste.

3.4 UHPCC statistical analysis and summary of the properties

Table 4 shows the summary of the statistically analyzed properties of UHPCC with glass powder at the age of 28 days concerning the REF group, which is without glass powder.

Table 4. Summary of the properties and statistical analysis of UHPCC.

Properties	UHPCC				
	REF	VD10	VD20	VD30	VD50
Compressive strength (MPa)	116.0	119.0	114.0	111.0	88.0
Statistically different from REF?	-	No	No	No	Yes
Tensile strength (MPa)	11.08	10.94	11.03	10.07	9.82
Statistically different from REF?	-	No	No	No	No
Static modulus of elasticity (GPa)	43.09	45.51	41.67	42.94	41.51
Statistically different from REF?	-	No	No	No	No
Dynamic modulus of elasticity (GPa)	53.31	52.15	52.10	52.37	49.75
Statistically different from REF?	-	Yes	Yes	No	Yes
Capillary water absorption at 72h (g/cm²)	0.079	0.077	0.092	0.091	0.088
Statistically different from REF?	-	No	No	No	No

Although the UHPCC studied have high cement consumption, it is observed high-energy efficiency in its use compared to traditional cementitious composites. They showed the same order of magnitude of composites efficiency with cement consumption around 450 kg/m³, as shown in Table 5.

Table 5. Energy efficiency of UHPCC.

Groups / Researchers	Cement Consumption (kg/m ³)	SCM Consumption (kg/m ³)			Compressive strength at 28 days (MPa)	Energy efficiency of binders (kg/m ³ MPa ⁻¹)	Cement energy efficiency (kg/m ³ MPa ⁻¹)
		Silica Fume	Fly Ash	Glass Powder			
REF	1000	80	-	0	106.81	10.11	10.11
VD10	900	80	-	81	98.58	10.76	9.13
VD20	800	80	-	161	121.69	8.55	6.57
VD30	700	80	-	242	110.70	6.3	9.20
VD50	500	80	-	403	105.86	9.29	4.72
Pelisser et al. [47]	472	-	52	-	67.2	7.8	7.02
Corinaldesi and Moriconi [48]	440	-	100	-	45.0	12.0	9.78
	625.7	41.7	-	-	113.0	5.9	5.5
Li et al. [49]	426.1	28.4	-	-	116.2	3.9	3.7
	341.5	22.8	-	-	121.1	3.0	2.8

Figure 11 shows the results of a binder intensity (BI) benchmark on compressive strength and binder consumption data from concretes produced in Brazil and 28 other countries. The results obtained in this study were included in this chart.

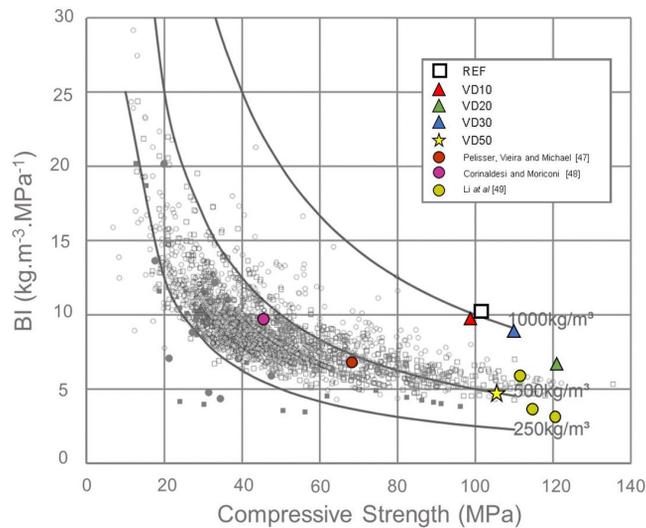


Figure 11. Benchmark of binder intensity adapted from Daminielli, Pileggi and John [50].

It is observed for most concretes that compressive strength above 50 MPa represents binder intensity is above $5 \text{ kg}\cdot\text{m}^{-3}\cdot\text{MPa}^{-1}$. In this study, the cement efficiency of ultra-high performance concretes was from 4.7 to $10.11 \text{ kg}\cdot\text{m}^{-3}\cdot\text{MPa}^{-1}$, for mixtures with the incorporation of 50% glass powder in substitution of Portland Cement and reference mixtures without glass powder, respectively. It is also noteworthy the use of a high content of glass powder, providing an appropriate destination for a waste, since the industry cannot absorb all the recycling demand of the waste glass it produces. Therefore, the incorporation of great amounts of glass waste powder ins substitution of Portland cement is a possible way to produce more sustainable UHPC using lower amounts of clinker.

4 CONCLUSIONS

From the characterizations and results, we obtained the following conclusions:

- The glass powder used in the study was suitable for UHPCC Portland cement replacement as a binder. Although the granulometry is 75% larger than in the cement and the specific surface is approximately 59% smaller, the experimental results analyzed presented values equivalent to the reference UHPCC, despite the high levels of substitution.
- The incorporation of glass powder in contents of 10, 20, 30, and 50% did not affect the workability and density of the cementitious composite in the fresh state, presenting values close to the reference line, with a maximum difference of 1% and 4%, respectively.
- For early ages such as 2 and 7 days, the greater the incorporation of glass powder, the lower the development of compressive strength due to the pozzolanic effect. However, at 28 days, the incorporation of high glass powder content as 50% have decreased the strengths refer to the powder-free group, with strengths of 88,37 MPa and 115,58 MPa, respectively. At 91 days there is no significant difference in compressive strength between the evaluated mixtures.
- In mechanical and physical evaluations such as splitting tensile strength, static modulus of elasticity, and capillary water absorption, at 28 days, there were no statistically different values. Although in the dynamic modulus of elasticity there was not a statistical difference between the REF and VD30.
- The results of compressive strength and tensile strength by diametrical compression obtained at 28 days for the cementitious composite with 50% replacement of the Portland cement by glass powder allow to classify it as a high-performance concrete.
- An amount of 50% of glass powder enable the greatest reduction of cement consumption and better energy efficiency as none of the studied mixtures obtained significantly different values and, mainly, no performance reduction on the evaluated properties.

ACKNOWLEDGMENTS

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

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Author contributions: S M Soares contributed with the activities of conceptualization, development, results and discussion, writing and preparation of the original text; T. O. G. Freitas contributed to the activities of development, results and discussion of results and writing; A. Oliveira Júnior development, results and discussion of results and writing; F. G. S. Ferreira contributed with conceptualization, supervision, discussion of results and review and J. A. A. Salvador Filho contributed with conceptualization, supervision, discussion of results and review.

Editors: Edna Possan, Mark Alexander