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Highlights on the properties of the soda-lime-silicate glass residue that enable its use as filler in ultra-high performance concrete

Destaques sobre as propriedades do resíduo de vidro de sílica-soda-cal que viabilizam seu uso como fíler em concreto de ultra alto desempenho

Destaca las propiedades del residuo de vidrio de sílice-soda-cal que permiten su uso como relleno en hormigones de ultra alto rendimiento

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João Victor da Cunha Oliveira
ORCID: https://orcid.org/0000-0003-1545-0082
Federal University of Campina Grande, Brazil
E-mail: joaovictorwo@gmail.com
Frankslale Fabian Diniz de Andrade Meira
ORCID: https://orcid.org/0000-0002-0306-3221
Federal Institute of Education, Science and Technology of Paraíba, Brazil
E-mail: frankslale.meira@ifpb.edu.br
Kennedy Flávio Meira de Lucena
ORCID: https://orcid.org/0000-0002-5565-9748
Federal Institute of Education, Science and Technology of Paraíba, Brazil
E-mail: kennedy.lucena@ifpb.edu.br

Abstract
The exponential advancement of cutting-edge technologies in the scope of civil construction, seeks to give cement-based materials the eco-efficient potential linked to mechanical performance that enables different applications. This work aims to evaluate the glass residue regarding the pozzolanic potential through ABNT NBR 5752:2014, as well as to verify whether through the characterization tests of x-ray fluorescence, x-ray diffraction and laser diffraction granulometry, if it is viable of application as supplementary cementitious material (filler), in ultra-high performance concrete. The glass residue submitted to the tests proposed in this study, was crushed in a jaw crusher, milled in a bench ball mill at 47 rpm, and was sieved in a 75 µm opening mesh (ABNT nº 200 mesh). For the test of pozzolanic activity, CP II F-40 class cement, normal sand, water from the public supply network, and superplasticizer additive were used for the mix with 25% of the residue replacing cement, while for the other characterization techniques, the glass residue was applied in its processed form (after sieving), dry or wet. The evaluated glass residue did not reach the minimum rate of 75% established by ABNT NBR 5752:2014, achieving only 45.72%, being classified as non-pozzolanic, which indicates its inert behavior in the presence of calcium hydroxide. The characterization tests confirmed, based on the specialized literature on ultra-high performance concrete, its viability as a filler when adopted as an alternative raw material for presenting chemical and mineralogical composition, in addition to granulometric distribution, very close to those used in studies that demonstrated satisfactory results when using the glass residue as an input.
Keywords: Glass residue; Supplementary cement material; UHPC systems.

Resumo
O avanço exponencial das tecnologias de ponta no âmbito da construção civil, busca conferir aos materiais de base cimentícia o potencial ecoeficiente atrelado ao desempenho mecânico que possibilite aplicações diversas. Este trabalho objetiva avaliar o resíduo de vidro quanto ao potencial pozolânico através da ABNT NBR 5752:2014, bem como verificar se através dos ensaios de caracterização de fluorescência de raios x, difração de raios x e granulometria por difração a laser, se o mesmo possui viabilidade de aplicação como material cimentício suplementar (fíler), em concreto de ultra alto desempenho. O resíduo de vidro submetido aos ensaios propostos neste estudo, passou por trituração em britador de mandíbulas, moagem em moinho de bolas de bancada à 47rpm, e foi peneirado em malha de abertura de 75 µm (peneira ABNT n° 200). Para o teste de atividade pozolânica, utilizou-se cimento de classe CP II F-40, areia normal, água proveniente da rede de abastecimento público, e aditivo superplastificante para o traço com 25% do resíduo em substituição ao cimento, enquanto que para as demais técnicas de caracterização, o resíduo de vidro foi aplicado na sua forma beneficiada (após peneiramento), por via seca ou úmida. O resíduo de vidro avaliado não alcançou o índice mínimo de 75% estabelecido pela ABNT NBR 5752:2014, conseguindo apenas 45,72%, sendo classificado como não pozolânico, o que indica seu comportamento inerte na presenta do hidróxido de cálcio. Os

ensaios de caracterização confirmaram, com base na literatura especializada sobre concreto de ultra alto desempenho, sua viabilidade como fíler ao ser adotado como matéria-prima alternativa por apresentar composição química e mineralógica, além de distribuição granulométrica, bastante próximas dos utilizados em estudos que demonstraram resultados satisfatórios ao utilizarem o resíduo de vidro como insumo.
Palavras-chave: Resíduo de vidro; Material cimentício suplementar; Sistemas CUAD.

Resumen
El avance exponencial de las tecnologías de punta en el ámbito de la construcción civil, busca dotar a los materiales cementosos del potencial ecoeficiente ligado al desempeño mecánico que permite diferentes aplicaciones. Este trabajo tiene como objetivo evaluar el residuo de vidrio en cuanto al potencial puzolánico mediante ABNT NBR 5752:2014, así como verificar si a través de las pruebas de caracterización de fluorescencia de rayos X, difracción de rayos X y granulometría de difracción láser, si es viable de aplicación como material cementoso suplementario (masilla), en hormigón de ultra altas prestaciones. El residuo de vidrio sometido a las pruebas propuestas en este estudio, se trituró en trituradora de mandíbulas, se trituró en un molino de bolas de banco a 47 rpm y se tamizó en una malla de apertura de 75 µm (tamiz ABNT nº 200). Para la prueba de actividad puzolánica se utilizó cemento clase CP II F-40, arena normal, agua de la red de abastecimiento público y aditivo superplastificante para la mezcla con 25% del residuo en sustitución del cemento, mientras que, para las demás técnicas de caracterización, el residuo de vidrio se aplicó en su forma procesada (después del tamizado), seco o húmedo. El residuo de vidrio evaluado no alcanzó la tasa mínima del 75% establecida por ABNT NBR 5752:2014, logrando solo el 45,72%, siendo clasificado como no puzolánico, lo que indica su comportamiento inerte en presencia de hidróxido de calcio. Los ensayos de caracterización confirmaron, con base en la literatura especializada sobre hormigones de ultra alto rendimiento, su viabilidad como relleno al ser adoptado como materia prima alternativa para presentar composición química y mineralógica, además de distribución granulométrica, muy cercana a las empleadas en estudios que demostraron Resultados satisfactorios al utilizar el residuo de vidrio como insumo.
Palabras clave: Residuos de vidrio; Material de cemento suplementario; Sistemas HUAR.

1. Introduction

Currently, the design of studies that seek to incorporate the eco-efficient potential into conventional materials is becoming increasingly present due to the considerable increase in the consumption of non-renewable raw materials. Studies with solid residues, for effective use in the civil construction sector, are favorably configured by the industry being one of the most consumed inputs of non-renewable origin, such as natural aggregates for mortar and concrete, as well as with high load environmental, such as Portland cement.

Regarding solid waste, it must be emphasized that the management of industrial by-products needs to consider a work perspective that is supported by a program to mitigate emissions directly at the generating source, as well as through the recycling mechanism itself (Evangelista, Tenório and Oliveira, 2012). Associated with the search for this reduction, the cement industry seeks, through the use of mineral admixtures, to reduce costs with the production of cement, mainly of an energy order, and that, in line with this, it was possible to obtain benefits in the quality of the final product (Garcia, Cabral Junior, Quarcioni and Chotoli, 2015).

Verifying the presence of pozzolanic behavior in industrial by-products makes it possible to classify whether supplementary cementitious materials (SCM) are inert (filler) or reactive (pozzolan). The pozzolanic potential of a material is configured to detonate cementing properties when it reacts in the presence of calcium hydroxide (CH), which originates from the hydrated phases of Portland cement, producing low-density calcium silicate hydrated (C-S-H) (Cunha Oliveira, Chagas, Meira, Carneiro & Melo Neto, 2020; Cunha Oliveira, Meira & Lucena, 2021), similar to that produced by C_3S and C_2S (tricalcium and dicalcium silicates), and which increases the durability of the cement matrix in the hardened state.

According to Ramanathan, Croly and Suraneni (2020), SCM are increasingly being used to partially replace Portland cement, both to improve the properties of concrete in the long term and to reduce carbon emission rates. Jiang, Ling, Mo and Shi (2019) state that SCM as fly ash, silica fume, ground granulated blast furnace slag, and metakaolin are among the alternatives used to reduce the consumption of Portland cement. In addition, the glass powder, being amorphous and containing a relatively high amount of silica, was also recognized as an SCM with the ability to trigger the pozzolanic reaction

(Jiang, Ling, Mo & Shi, 2019).

However, it is noted that in most cases in the literature, for the UHPC mixing project, the quantities of mineral admixtures are provided directly without any detailed explanations or theoretical support, and furthermore, due to the complex cement system of this type of composite, the influence of different mineral admixtures on the hydration kinetics and properties of UHPC still needs further clarification (Yu, Spiesz & Brouwers, 2015). This makes it possible to say, even, that the behavior of the glass powder, and other additions, in presenting reactive potential as a pozzolanic SCM requires complementary evaluations.

Vaitkevičius, Šerelis and Hilbig (2014), observed that, when applying powdered soda-lime-silicate glass residue in an attempt to certify it as pozzolanic, it did not have pozzolanic properties as good as microsilica, which consumes almost 5 times more CH than glass powder during hydration of Portland cement. It is worth mentioning that the addition of the glass powder occurred together with the silica fume, resulting in the greater resistance of the study (221 MPa), being an indication that the authors already expected that the residue would act more as a filler than as a pozzolan Using x-ray diffraction, Vaitkevičius, Šerelis and Hilbig (2014) showed that the peak referring to the mineralogical phase of CH still exists in the composition that there is only glass powder, being the 2nd largest among the mixtures, that is, its consumption is not it was completely accomplished. Thus, it is understood that the powdered glass residue tends to have a greater potential to be a SCM with a character of a filler, and not a pozzolan, although Vaitkevičius, Šerelis and Hilbig (2014) also affirm that, the glass powder finely ground with particles up to 100 µm can eventually act as a pozzolanic material. Other factors such as the surface area and its diameter at 50% (d_{50}), can help in understanding the reactivity of the glass, as well as its effect as a chemical activator.

The production of glass waste has a record of generation of powdered residues on a scale of at least 48 thousand tons of powder each year in Brazil (Rodier & Savastano Jr, 2018), and the adoption of powdered residues to improve the properties of the microstructure of cementitious matrix materials is one of the most developed aspects of study in the world due to the interesting characteristics it has: amorphous structure and a large amount of silicon present (Bouchikhi, Benzerzour, Abriak, Maherzi & Mamindy-Pajany, 2019).

Several studies indicate that the glass residue, when finely ground (in granulometric ranges below 75 µm), can present pozzolanic potential, and when used in cement substitution ranges from 10% to 25%, gives satisfactory results (Patel, Tiwari, Shrivastava and Yadav, 2019). In addition to providing better long-term performance, the addition positively reduces the permeability of concrete, resulting in increased durability due to the densification of the material's microstructure through the micro-filler effect (Lee, Hanif, Usman, Sim & Oh, 2018). The application of this residue occurs in various ways in mixtures of concrete and mortar, involving the sphere of aggregates, composing the class of aggregates and acting as a filling material for the larger size of the grains in the mixture, and the sphere of non-aggregates, which due to their high fineness and reactivity make up the group of mineral admixtures (Lee, Hanif, Usman, Sim & Oh, 2018).

Bearing in mind that UHPC systems use high percentages of mineral admixtures to increase their performance in terms of axial compression and durability (Cunha Oliveira, 2020), due to the pore refinement on a microstructural scale resulting in high compactness (Cunha Oliveira, Meira & Lucena, 2021), the glass residue incorporated into the composition as SCM (filler), due to reduced particle size, recommends its use for particle sizes below the fine aggregates, because the specific mass is similar and has less absorption (Soliman & Tagnit-Hamou, 2017a).

The objective of this work is to evaluate the pozzolanic activity index of the glass waste using ABNT NBR 5752:2014, as well as to characterize the residue after beneficiation by means of x-ray fluorescence, x-ray diffraction, and particle size by laser diffraction, to test whether its properties are equivalent to those of the glass waste used as an alternative raw material in UHPC systems.

2. Methodology

2.1 Processing of Glass Residue

The residues of silica-lime glass, originating from the disposal of bottles, packaging, and household appliances, of different colors, were collected and sanitized to remove most of the impurities, and then the processing took place, which took place at the Laboratory of Comminution of IFPB – Campus Campina Grande. The first step consisted of crushing the waste by means of a jaw crusher (Figure 1), to reduce the vitreous artifacts to particle sizes suitable for grinding.

Figure 1 – Jaw crusher (left), and crushed waste (right).

Source: Authors.

Consequently, the excess impurities (metals, pieces of paper) were removed and the crushed residue was milled in a bench-ball mill at 47 rpm, adopting an interval of 3 hours for each grinding cycle (Figure 2).

Figure 2 – Ball mill (left), and ground residue (right).

Source: Authors.

Finally, the crushed residue was sieved through a 75 µm opening sieve (ABNT n° 200) with the aid of a mechanical stirrer in 30-minute cycles at a frequency of 8 Hz (Figure 3). All the material passed through the mesh was stored in plastic bags, and the retained material was taken to the ball mill for grinding.

Figure 3 – Screening in ABNT n° 200 (left), and final visual aspect of the residue (right).

Source: Authors.

2.2 Evaluation of Glass Residue Properties

2.2.1 Pozzolanic Activity Index

Based on ABNT NBR 5752:2014, which designates the method to assess the pozzolanic potential of materials, in powder form, in the presence of Portland cement, CP II F-40 cement from the manufacturer Elizabeth Cimentos, sand was used normal based on ABNT NBR 7214:1982, water from the public supply network in the city of Campina Grande-PB, and superplasticizer additive of the type MasterGlenium® 51 from the manufacturer BASF, for the mixture with 25% of the residue to replace cement.

In order to measure the quantity of materials for the production of the specimens, the quantitative suggested by ABNT NBR 5752:2014 was taken as a parameter, however with the water/cement factor adaptation suggested by it from 0.48 to 0.60, an since the amount of water to be used must recommend an abatement of 225 mm (Cunha Oliveira, Chagas, Meira, Carneiro & Melo Neto, 2018).

The 5x10 cm cylindrical molds were made, with a 1:3 mix (cement and sand), composing two mortars: the reference mortar (A), and the one with the replacement of cement by 25% of the residue (B), with rupture performed at 28 days of age following ABNT NBR 5752:2014. Table 1 shows the quantities for the two mortars manufactured, and Table 2 shows the granulometric composition of normal sand according to ABNT NBR 7214:1982.

Table 1 – Quantitative inputs for mortars A and B according to ABNT NBR 5752:2014.

Mortar A – Reference (g)	Mortar B – Glass #200 mesh (g)
624 – Cement	468 – Cement
1872 – Sand	156 – Glass
374 – Water	1872 – Sand
–	374 – Water
–	5 – Additive

Source: Authors.

Table 2 – Retained values in each granulometric range corresponding to ABNT NBR 7214:1982.

Retained Values	Sieves
50.00 g	#10 *mesh* (2.0 mm)
200.00 g	#16 *mesh* (1.2 mm)
250.00 g	#30 *mesh* (600 µm)
250.00 g	#50 *mesh* (300 µm)
250.00 g	#100 *mesh* (150 µm)
1.00 Kg	–

Source: Authors.

2.2.2 X-Ray Fluorescence

The X-Ray Fluorescence (XRF) test was performed at the Materials Characterization Laboratory (CCT/UAEMa), at UFCG – Campus Campina Grande. A dispersive energy spectrometer SHIMADZU Model EDX-720 (Energy Dispersive X-Ray Spectrometer) was used, with generation of x-rays through a Rh target tube, in a vacuum atmosphere and collimator with a 10 mm opening.

2.2.3 X-Ray Diffraction

The X-Ray Diffraction (XRD) test was carried out at the Materials Characterization Laboratory (CCT/UAEMa), at UFCG – Campus Campina Grande, using the powder method. The SHIMADZU Model XRD-6000 equipment (X-Ray Diffractometer) was used, which has a fixed x-ray tube in high vacuum with tungsten filament, with the sample rotation in θ and the arm in 2θ. The radiation used was Kα (monochromatic) for the target metal Copper (Cu), with λ = 1.5406 Å, under a voltage of 40 kV and a current of 30 mA, with slits opening at 1.0°, 1.0° and 0.3 mm. The scanning speed was set at $2°.min^{-1}$, at a sampling step of 0.02° and a stop time of 0.60 s, in the range of 5° to 60°. The Jade 5.0 software was used to identify the clay minerals present, using the ICDD (International Center for Diffraction Data) crystallographic charts.

2.2.4 Laser Diffraction Granulometry

The Laser Diffraction Granulometry test was performed at the Materials Technology Laboratory (CCT/UAEMa), at UFCG – Campus Campina Grande. The particle size distribution was studied using the liquid phase particle dispersion method, associated with an optical measurement process through laser diffraction, combining the proportional relationship between the diffracted laser and the concentration and particle size. The sample was dispersed by sodium hexametaphosphate (HMFNa) + sodium carbonate (Na_2CO_3), in 470 ml of distilled water, with the aid of a Hamilton Beach® Model N-5000 shaker, at 17,000 rpm for 20 minutes. The CILAS Model 1064 equipment was used, operating in a wet way (DL), which works under data processing using the Fraunhofer algorithm, with results denoted in granulometric curves (histogram and accumulated) in the range of 0.04 to 500 µm.

3. Results and Discussion

3.1 Pozzolanic Activity Index

Seeking to interpret the result obtained from the pozzolanic potential of the glass residue in producing C-S-H in the presence of Portlandite, with a particle size of less than 75 µm, Table 3 presents the arithmetic mean for the 4 tested specimens of each composition made (mortars A and B).

Table 3 – Pozzolanic activity index of soda-lime-silicate glass residue.

Mortars	Compressive Strength – 28 days (MPa)	Pozzolanicity (%)
Reference	31.58	–
Glass #200 mesh	14.44	45.72

Source: Authors.

It is clearly noted that for these study conditions, from the processing of the residue for use as a binder to the manufacture of mortars, it did not have any potential to act as a supplementary cement material, reaching only 45.72%, while the minimum established by ABNT NBR 5752:2014 is 75%, showing that it does not consume the CH present in the paste after the start of the hydration reactions of Portland cement, in accordance with what was stated by Vaitkevičius, Šerelis and Hilbig (2014).

3.2 X-Ray Fluorescence

Table 4 shows the chemical composition of the glass residue, obtained through the XRF test.

Table 4 – Chemical composition of glass residue.

Elements	Quantitative (%)
SiO_2	67.01
CaO	13.76
Na_2O	9.58
Al_2O_3	4.99
Fe_2O_3	1.55
K_2O	1.25
MgO	0.91
SO_3	0.37
Cr_2O_3	0.16
Others	0.42

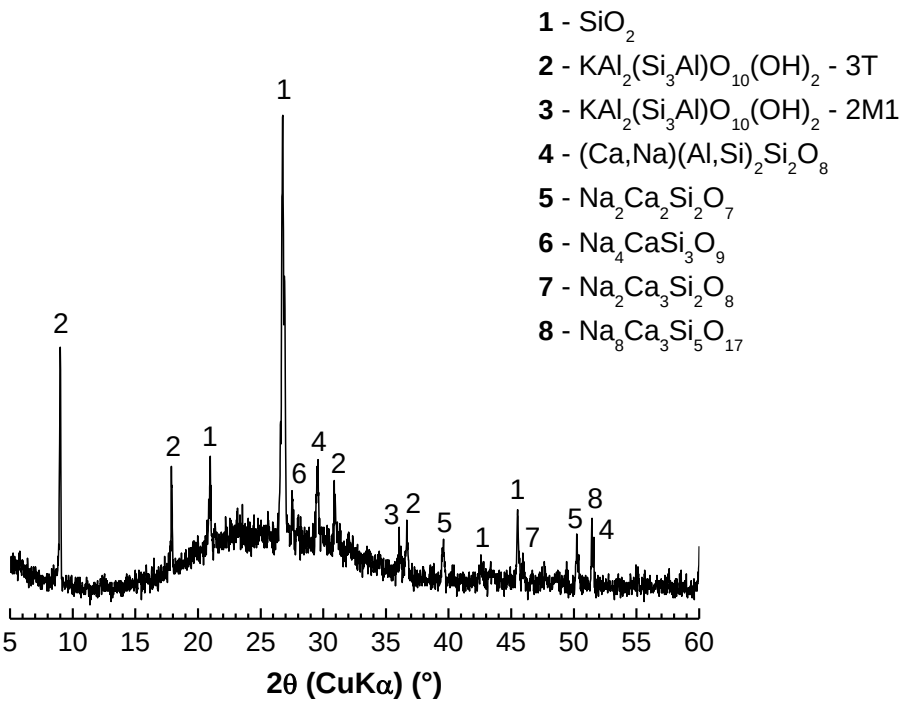
Source: Authors.

As expected, high percentages were observed for SiO_2 (67.01%), CaO (13.76%), and Na_2O (9.58%), confirming that the glass residue used is soda-lime-silicate, in addition to being close to the percentages stipulated by Vogel (1994), Shelby (2005), and Bauer (2019) as a soda-lime-silicate glass system. Comparing the quantities of these main oxides with those presented in the studies on UHPC that used the residue as an alternative raw material, a great similarity was observed with the studies by Vaitkevičius, Šerelis and Hilbig (2014), Soliman and Tagnit-Hamou (2016), Soliman and Tagnit-Hamou (2017a), Soliman and Tagnit-Hamou (2017b), Mosaberpanah, Eren and Tarassoly (2019), Pezeshkian, Delnavaz and Delnavaz (2019), Wilson, Soliman, Sorelli and Tagnit-Hamou (2019) and Jiao et al. (2020), being an indication of the compatibility with the UHPC systems of the studied glass residue.

3.3 X-Ray Diffraction

Figure 4 shows the diffractometric pattern of the glass residue, obtained through the XRD test.

Figure 4 – Mineralogical composition of the glass residue.



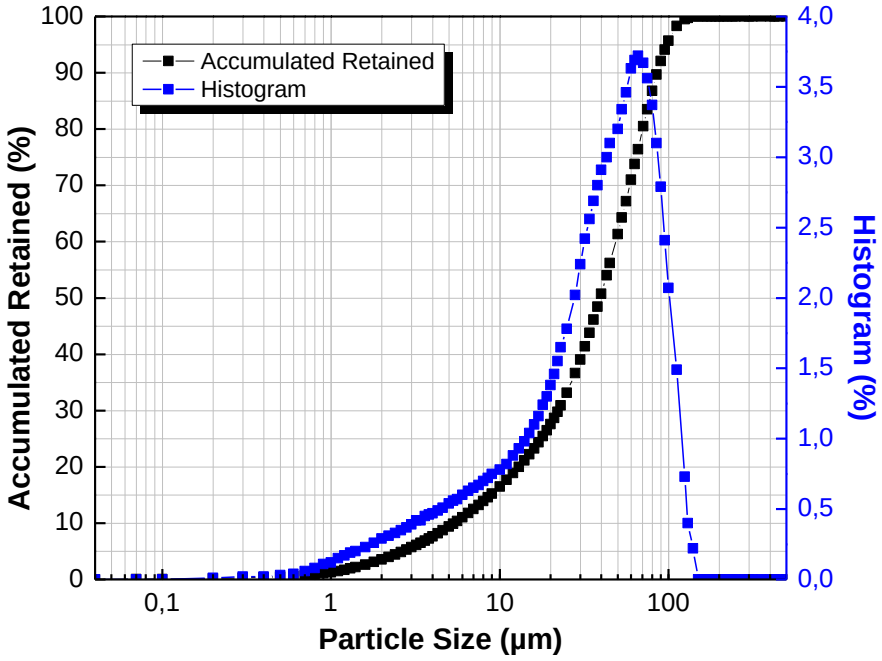
Source: Authors.

It was identified that the glass residue presents quartz (SiO_2, PDF#65-0466) and muscovite - 3T ($KAl_2(Si_3Al)O_{10}(OH)_2$, PDF#07-0042) as the mineralogical phases, most prevalent in the diffractogram , caused by possible contamination during beneficiation during the milling stage. Muscovite - 2M1 ($KAl_2(Si_3Al)O_{10}(OH)_2$, PDF#46-1409), sodium anorthite ($(Ca,Na)(Al,Si)_2Si_2O_8$, PDF#20-0528), was also observed, as well as sodium and calcium silicate in four variations ($Na_2Ca_2Si_2O_7$, PDF#10-0016), ($Na_4CaSi_3O_9$, PDF#37-0282), ($Na_2Ca_3Si_2O_8$, PDF#23-0668) and ($Na_8Ca_3Si_5O_{17}$, PDF#10-0053). The presence of these different variations for sodium and calcium silicate in glasses occurs because, according to Bradtmüller, Villas-Boas, Zanotto and Eckert (2020), these materials represent a small group of glasses that undergo homogeneous nucleation instead of following the thermodynamically favored path of surface crystallization, which according to Yuritsyn (2015), represents convenient models in the study of the mass nucleation of homogeneous crystals. For Cormack and Du (2001), ternary soda-lime-silicate systems are structurally complex, even though they are present in the silicate group, since, according to Greaves, Fontaine, Lagarde, Raoux and Gurman (1981), the modification of cations Na^+ and Ca^{2+} break the three-dimensional network of SiO_2, allowing the formation of stable glasses, and despite the structural change, SiO_2 as a tetrahedral unit persists in silicates, as identified in the diffractogram at $2\theta = 26.76$.

3.4 Laser Diffraction Granulometry

Figure 5 shows the particle size distribution of the glass residue, obtained through laser diffraction.

Figure 5 – Particle size distribution by laser diffraction of the glass residue.



Source: Authors.

It was possible to observe that, the powdered glass residue presented 90% (d_{90}) of the particles with diameter less than or equal to 77.99 µm, 50% (d_{50}) less than or equal to 36.65 µm, and 10% (d_{10}) less than or equal to 5.35 µm, as shown in Table 5 below. In view of the grinding time of 3 hours at 47 rpm used to obtain the powdered glass residue, the d_{50} of 36.65 µm obtained is within the expected range, since the input applied to the UHPC was passed in ABNT n° 200 mesh. However, when comparing with the studies that used the powdered glass residue as SCM in UHPC systems, it was noticed that the d_{50} were lower, to quote: Vaitkevičius, Šerelis and Hilbig (2014) (d_{50} = 16 µm, using a vibrating disc mill with speed between 750 and 940 rpm); Soliman and Tagnit-Hamou (2016) (d_{50} = 12 µm), Soliman and Tagnit-Hamou (2017b) (d_{50} = 12 µm), e Wilson, Soliman, Sorelli and Tagnit-Hamou (2019) (d_{50} = 12 µm), where both three jobs used air classifier and jet mill with speed between 2,000 and 22,000 rpm. Despite the difference of at least 20 µm as compared to the d_{50}, it is possible to highlight the energy savings in the production of SCM, since the focus on the use of glass waste is to combine sustainable practices with Portland cement matrix materials.

Table 5 – Particle sizes of the glass residue.

General Parameters	
d_{10}	5.35 µm
d_{50}	36.65 µm
d_{90}	77.99 µm

Source: Authors.

4. Conclusion

The evaluated glass residue did not reach the minimum rate of 75% established by the standard, achieving only 45.72%, being classified as non-pozzolanic, that is, it remains inert during the hydration process because it does not react with the calcium hydroxide released by the Portland cement. The glass residue characterized by XRF, XRD and laser granulometry

had technical conformity in terms of chemical, mineralogical and granulometric composition, being an indication that enables its application as an alternative raw material in UHPC systems, as it is equivalent to the results observed in the literature on the development of UHPC that adopted glass residue as an ecological input.

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