ORIGINAL ARTICLE



Recycling of waste glass extracted from a WTP into ceramic materials

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Abstract

Glass waste from an industrial wastewater treatment plant (WTP) was studied to recycle the material in ceramic materials. A white kaolinitic clay was used replacing the waste in percentages of 0-30%, producing $115 \times 25 \times 10$ mm specimens pressed with 20 MPa burned in the 850–1050 °C range. Burned specimens were evaluated for plasticity, dry mass density, water absorption, firing shrinkage, flexural strength, optical microscopy and scanning electron microscopy. It was found that the application of the glass waste aided the properties such as water absorption and tensile strength and despite increasing the linear shrinkage, it did not damage this property excessively, except for the application of the waste at a firing temperature of 1050 °C. The properties obtained with the use of glass are attributed to the higher liquid-phase formation promoted by the waste due to the chemical and mineralogical composition of the waste, which presents alkaline compounds. The results proved the viability of recycling the wastewater from WTP glass in ceramic materials, promoting an environmentally correct disposal of the waste. The application of 30% of burnt waste at 850 °C, for example, provides a burn shrinkage of about 2%, 17% water absorption and 7 MPa tensile strength, which enables the values established by technical standards to be met application for both tiles and ceramic bricks.

Keywords Glass waste · WTP · Ceramics · Clay

Introduction

Industrial wastewater treatment plant (WTP) is facilities used to treat urban or industrial waste, before the material is disposed of in the environment [1, 2]. Even if it reduces the levels of contaminants in sewage and effluents, the final disposal of these materials is usually exposing them to the environment, causing numerous contaminations, or having to apply high costs to specialized companies, so that the sludge from the treatment is disposed of in licensed industrial landfills [3, 4].

It is known that the civil construction sector is one of the most consuming natural resources in the world, contributing significantly to the emission of greenhouse gases that are studied during the production of materials such as cement [5, 6]. Thus, the urgency of the application of waste in construction materials is perceived, aiming at achieving the sustainable development of the sector, as reported by several authors [7-10].

In addition, it is known that the use of waste in clay-based materials, such as ceramic blocks and bricks, for example, promotes the confinement or adsorption of problematic materials present in the waste composition [11]. Because of that, several studies have promoted the application of wastes in ceramic materials, which is also the objective of this work.

Areias et al. [12] studied the incorporation of sludge from the sewage treatment plant in a kaolinitic clay. The authors carried out the physical, chemical, and mineralogical characterization of the two materials and incorporated contents of up to 15% of sludge to replace the studied clay, testing the materials produced by compressive strength and water absorption. The authors verified that although there is a tendency toward a reduction in the compressive strength and an increase in the water absorption of the material, the incorporation of contents up to 2.5% by mass is possible, without excessive loss of properties.

Regarding glass waste, it is worth highlighting the works by Azevedo et al. [13] in which the authors incorporated the waste into adhesive mortars, obtaining satisfactory

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results; [14], where the authors applied the glass waste to produce a synthetic clay aggregate; the works by Lu et al. [15], Liu et al. [16], and Khan et al. [17], in which the possibility of using glass waste from different sources (bottle glass, household appliances, for example) in cement-based materials such as concrete, pastes, and mortars is addressed. Other important studies were carried out using glass waste in geopolymeric materials. The works by Azevedo et al. [18, 19] who proved the possibility of alkaline activation of these materials.

The work by Gutiérrez et al. [20], where the authors proposed the use of glass waste as a catalyst for the alkali activation reaction of metakaolin-based mortars and obtained excellent results when thermal treatments were performed, since, as highlighted and proven by the authors, the glass waste provides the formation of a vitreous phase during firing, contributing to the increase in the strength of the materials. Raut and Gomez [21] used glass powder to evaluate the efficiency of blocks used for thermal insulation, highlighting that this type of material contributes to improving the thermal efficiency of sealing devices.

Still on the application of glass waste in construction materials, as a possibility of recycling the material, it is worth highlighting the work by Vieira et al. [22] in which the authors applied fluorescent lamp glass waste to red ceramic materials and found that the incorporation of 30% of the waste increased the flexural strength of the materials from 712 to 1361 N, in addition to providing a reduction in water absorption from 24.1 to 15.7%, which was attributed to the formation of a liquid phase and increased vitrification of the studied ceramic artifacts.

In this context, the objective of this work is to evaluate the possibility of applying glass waste from an effluent treatment plant of an industry that produces flat glass in ceramic materials, using a white kaolinitic clay with chemical and mineralogical composition usually used in these applications. The main innovations of this article are to carry out the application of waste from a source that has never been studied, from the wastewater treatment plant.

The waste studied in this manuscript was chosen due to its disposal, which is problematic. The materials are disposed of

in water resources, using an aqueous solution that contains the glass waste in a concentration of 3–7.5%, according to data from the industry where was extracted. Some data from previous publications with the same waste indicate that the annual generation of the material is approximately 3000 tons per year [18, 19]. This highlights the urgency of recycling the waste, as it contains materials that are harmful to the environment, such as sodium, magnesium, and iron, which will be highlighted in the text below. Therefore, the objective of this manuscript is to recycle the waste extracted from the industry, before it is disposed of in water resources, as this way it is easier to obtain this material.

Methodology

Aiming to study the incorporation of glass waste from WTP in ceramic materials, initially the materials used in the research were collected. The clay used was extracted from Brazil, and is named as white kaolinitic clay. The glass waste was collected from the WTP in Brazil. Figure 1 shows the materials used.

The WTP from which the waste was obtained is an industrial facility and does not serve domestic sewage, which is why its chemical composition is not organic like other sewage obtained from domestic urban waste. WTP only has a primary treatment stage, as the waste generated is predominantly inorganic. This treatment is carried out through grating, desander, and settling tanks. It is at this stage that the residue is extracted, after the decantation process.

The two materials were characterized granulometrically through sieving and sedimentation (ABNT NBR 7181, 2016) [23], mineralogically through X-ray diffraction using a Sheifert brand diffractometer, model URD 65, operating with Cuk α and 2 θ radiation ranging from 5° to 60° and chemically using X-ray fluorescence spectrometry in Philips PW 2400 equipment.

The glass waste from WTP was thermally analyzed by optical dilatometry using an optical dilatometer brand Misura where a maximum temperature of 1600 °C was used, with a heating rate of 40 °C/min. The glass waste was

Fig. 1 Materials used in the research: **a** white kaolinitic clay and **b** glass waste from WTP



(b)

crushed until it was in powder form, being dried in an oven at 110 °C and later ground in an annular mill. The white kaolinitic clay, on the other hand, was thermally characterized by differential thermal analysis in a thermal analyzer of the TA instruments brand, model SDT2960 with a heating rate of 10 °C/min to speed up the test and a maximum temperature of 1150 °C. The glass waste was also morphologically characterized by scanning electron microscopy (SEM) using a Jeol model JSM 6460 LV microscope with an energy-dispersive spectrometer (EDS). After the characterization of the raw materials, clayey masses were formulated containing 0%, 10%, 20%, and 30% of glass waste in mass to replace white kaolinitic clay. The evaluation of the plasticity parameters of all studied masses was carried out through the Atterberg limits (ABNT NBR 6459, 2016; ABNT NBR 7180, 2016) [24, 25] and specimens were produced through unixial pressing using 8% moisture and applying pressure 20 MPa. The test specimens produced, with dimensions of $115 \times 25 \times 10$ mm, were dried in an oven for 24 h to lose shaping water and later burned using a laboratory furnace type Muffle by Maitec model FL 1300. The firing temperatures were chosen as a function of the thermal analysis performed, using 850 °C, 900 °C, 950 °C, 1000 °C, and 1050 °C. The specimens were tested for dry bulk density, linear burning shrinkage, water absorption, and flexural strength (ABNT NBR 15270-1, 2017; ABNT NBR 15270-2, 2017; ABNT NBR 15310, 2009) [26-28]. For further characterization of the material, optical microscopy (OM) was performed using an Olympus model CGA confocal microscope. Finally, the SEM was performed on the fired ceramic bodies to assess the microscopy and rupture surface of the materials.

Results and discussion

Characterization of waste glass and white kaolinitic clay

The chemical analysis of the waste glass from WTP and the white kaolinite clay are shown in Table 1. On the clay used, it can be seen that the material has 49.45% of SiO2 and 31.31% of Al2O3, typical mineral compositions clay. The SiO₂/Al₂O₃ ratio is 1.58, with the theoretical kaolinitic ratio being 1.18 [29]. If the SiO₂/Al₂O₃ ratio was exactly 1.18, this would indicate that the studied clay is entirely kaolinite-based. However, it is observed that the ratio between SiO_2/Al_2O_3 was 1.58, above the theoretical value. Deviation of SiO_2 amounts is related to the presence of quartz sand or muscovite mica [30, 31]. The presence of kaolinite in this clay is confirmed by Fig. 2a and by the high loss to fire found for the material, of 14.4% [10, 32].

The low Fe₂O₃ contents below 3% indicate that the color of the fired ceramic pieces will not be reddish. The levels obtained for TiO_2 above 3%, on the other hand, favor the occurrence of yellow coloration after burning. These two oxides are called dye oxides [2, 33]. The low Na₂O and K₂O contents, on the other hand, indicate the formation of little liquid phase after burning, as alkaline oxides are melting materials. Furthermore, the low percentage of CaO and MgO is indicative of the absence of carbonates [34]. Still on Table 1, it is possible to verify that the glass waste has a typical sodo-calcium composition. As highlighted by Kielf et al. [35] soda-lime glasses typically have a composition of 74% SiO₂, 14% Na₂O and 6% CaO, while the glass waste has 71% SiO₂, 13.5% Na₂O and 10% CaO. Silicon oxide is of great importance in the composition of the glass structure, as they are network-forming oxides. Sodium and calcium oxides are network modifiers, which reduce the viscosity of the glass and allow the formation of a viscous flow that can help to reduce the porosity of the ceramic and thus contribute to improving its mechanical properties. It is worth noting that the high amounts of K_2O , which is a fluxing oxide, contributes to the formation of the liquid phase, changing the fluidity and allowing the removal of bubbles and chemical homogenization after burning [34]. It is observed in Table 1 that the waste glass has in its composition 13.57% of Na₂O, 2.44% of MgO and 1.25% of Fe₂O₃. These compounds when diluted in water can cause environmental problems, such as eutrophication of water resources due to algae proliferation, death of aquatic animals and/or contamination of drinking water [36, 37].

Figure 2 shows the mineralogical analysis of white kaolinitic clay and glass waste. It is verified that the clay is predominantly kaolinitic, and this clay mineral adds plasticity to the clayey mass [38]. There is also the presence of quartz, which helps to control post-burn shrinkage, but causes a drop in strength in the material [39] and the presence of gibbsite, muscovite and/or illite mica and microclimate [40]. It is noteworthy that the presence of these minerals helps in the formation of a liquid phase during the sintering of the material, due to the presence of alkaline materials [41].

Table 1	Chemical	analysis of
studied	materials	

Material	SiO ₂	Al ₂ O ₃	K ₂ O	TiO ₂	Fe ₂ O ₃	MgO	CaO	Na ₂ O	Others	LOI
White kaolinitic clay (%)	49.45	31.31	3.26	1.68	1.44	0.39	0.29	0.34	0.40	14.4
Waste glass (%)	67.88	2.31	0.30	0.17	1.25	2.44	8.44	13.57	0.84	2.80

LOI loss on ignition





On the mineralogical analysis of the glass waste (Fig. 2b), it is verified the absence of crystalline phases, containing only bands related to the vitreous phase, characteristic of amorphous materials [42]. This is explained by the rapid cooling process characteristic of glass production. The peaks detected in the figure between 20 and 30° are the result of the presence of semi-crystalline silica [43]. The presence of amorphous material is desirable for application in ceramic materials, since this crystal structure favors the occurrence of a liquid phase and vitrification of the material, in addition to helping to reduce post-firing porosity [44]. Figure 3 shows the thermal analysis of clay. Some events can be identified and are described below: an exothermic peak occurs between 65 °C and 70 °C attributed to the loss of kneading water that causes a mass loss of approximately 0.96%; another exothermic peak occurs between 265 °C and 270 °C, with a mass loss of 2.15%, related to the transformation of gypsum; another exothermic peak is identified at approximately 490 °C, with a mass loss of almost 9%, attributed to the transformation of kaolinite into metakaolinite, proving that the clay is kaolinitic in nature, and finally an endothermic peak is identified at approximately 980 °C,



Fig. 3 Thermal analysis of white kaolinitic clay

attributed the sintering of the material and transformation of metakaolinite to form resistant phases [29]. This helps to justify the chosen burning temperatures, three below 980 °C and two above this value.

Figure 4 shows the thermal analysis of the glass waste using optical dilatometry. Two important events in the application of waste glass are noticeable in the figure: around 850 °C, the softening of the glass occurs, where the material starts to behave as a superheated and low-viscosity fluid, which can help the vitrification and densification of ceramic materials. Another event that can be described is the melting of glass, which occurs just before 1100 °C. At this stage, the glass turns into a liquid and is easier to flow in the ceramic matrix, causing the occurrence of defects in the material, such as segregation [45]. Therefore, the chosen firing temperatures were limited to the range of 850 °C–1100 °C, since below this range, the glass tends to have no effect on the material, and above this range, the incorporation of glass is not beneficial.

Figure 5 shows the particle size of the materials used in this research. It is verified that the glass waste presents a very fine granulometry, being relatively similar to the clay granulometry used in the research, which is a positive characteristic to substitute the clay for the waste under study. This fact can be proven by analyzing the equivalent diameters D20 and D85, where it is clear that there is an overlapping of the materials curve. The fineness of the waste is proven through scanning electron microscopy, illustrated in Fig. 6, where it is noticed that the waste particles have diameters in micrometric orders, ranging from about 2 to 20 μ m.

Characterization of ceramic masses

Figure 7 is defined through the parameters plasticity limit and plasticity index, which is obtained through the numerical difference between the liquidity limit and the plasticity limit. It is verified that the 0% mass is within the acceptable extrusion range, since the clay used is very plastic. Using 10% waste glass shifts the plasticity of the material to the optimal extrusion zone, while using 20% and 30% excessively reduces the plasticity of the material. This fact makes the masses containing 20% and 30% of the waste present plasticity slightly lower than the recommended range for extrusion of ceramic materials. Thus, following the plasticity criterion, the mass containing 10% of waste is the most suitable. The 20% and 30% masses were not discarded, as the technological properties obtained by these materials will be evaluated.

Figure 8 shows the dry bulk density of the studied masses. It is verified that the use of WTP glass waste made the ceramic mass lighter. This fact is directly related to the amount of clay used in the material. It is observed that the masses containing greater amounts of clay have higher dry density, because, as they are more plastic, they keep more





Fig. 5 Granulometry of the studied materials

Passing percentage (%)

water bound to clay mineral particles, especially kaolinite particles, the main compound responsible for plasticity [38, 46]. Thus, the results obtained by Fig. 8 confirm and prove the results obtained by Fig. 7.

Figure 9 shows the post-firing properties obtained by the ceramic masses. It can be seen from Fig. 9a that there was an increase in post-burning retraction as the burning temperature increased and as the percentage of waste used Fig. 6 SEM of glass waste





Fig. 7 Prognosis of composite extrusion. Plasticity index numerical difference between the liquidity limit and plasticity limit



Fig. 8 Dry bulk density

increased. This fact can be attributed to the greater amount of liquid-phase formation promoted by the waste [47], which causes an effect of reducing the size of the material. Although the Brazilian standard does not present maximum retraction values, [48] recommend that shrinkage be limited to a maximum of 7% for ceramic materials that require high-dimensional accuracy. Thus, of the masses burned at a temperature of 1050 °C, only the percentage with 0% meets the recommended value. At other temperatures, all studied masses present shrinkage values less than 7%. This discards the use of waste at a burning temperature of 1050 °C.

Regarding water absorption, as seen in Fig. 9b, there are two patterns of values obtained: at temperatures of 950 °C, 1000 °C, and 1050 °C, there was a reduction in water absorption as higher waste contents were incorporated, due to liquid-phase formation and vitrification promoted by the waste [49]. At lower temperatures, this reduction did not occur, which shows that the formation of a liquid phase by the waste at lower temperatures was not as intense. If the waste does not act to form a liquid phase, the presence of this material in the ceramic matrix only causes defects, increasing porosity and water absorption. Brazilian standards for the application of ceramic tiles limit the absorption of water to 22% [28], while limiting the absorption to 25%for the application of ceramic blocks [26]. As a result, it is verified that all the studied masses at all fired temperatures can be applied both for tiles and for ceramic blocks. This fact is beneficial with regard to recycling WTP glass waste into ceramic materials.

Regarding the flexural strength, illustrated in Fig. 9c, it appears that there is no defined standard for the values obtained, which is attributed to the large number of variables involved in the analysis, such as: application of the glass waste, which forms different liquid-phase proportions depending on the temperature and proportion of waste applied, chemical and mineralogical composition of the white kaolinitic clay, which provides different amounts of flux minerals, minerals that act as fillers and minerals that act in a plastic way, different calcination temperatures, which provide different effects to the materials studied [50, 51]. In

Fig. 9 Properties: **a** linear shrinkage, **b** water absorption, and **c** flexural strength



general, it appears that the most resistant compound formed was calcined at 1050 °C containing 10% waste, while the least resistant was calcined at 950 °C containing 30% waste. Based on these results, it appears that there is an optimal WTP glass waste content to be used in ceramic materials; since the more glass waste is applied, the more liquid phase is formed after firing. However, the formation in excess of the liquid phase is as harmful to mechanical strength as the non-formation of this phase [47]. On the minimum values stipulated by Brazilian recommendations, above 6.5 MPa is adopted for ceramic tiles [2, 51]. This value is obtained using 10% of waste burned at 1000 °C or 1050 °C, and using 20% burned at 950 °C or 1050 °C. With the use of 30% acceptable values are obtained for all temperatures with the exception of 950 °C. It is interesting to note that for the reference mass, only at 1050 °C, it is possible to obtain flexural strength values greater than 6.5 MPa. This fact proves the beneficial nature of the application of the waste on ceramic materials. For ceramic blocks, the minimum value is 2.5 MPa [2, 51]. It is observed that all evaluated compositions meet this value. Thus, although only some compositions can be used in the manufacture of ceramic tiles, a material whose commercial value is greater, and all compositions can be applied to the manufacture of bricks.

Figure 10 shows optical microscopy (OM) for the studied masses burned at 1050 °C. It is verified that there was an intensification of the vitreous phase formed in the materials [47, 49], which has a whiter appearance in the OM. It is also possible to notice the greater presence of glass crystals in the



Fig. 10 Optical micrographs of samples fired at 1050 °C

masses that have a higher percentage of waste, and a smaller amount of pores in materials containing a higher percentage of waste. The image that has the reference composition, for example, has voids in dark form that are easy to detect in the image. These images help to confirm the results obtained in the parameters evaluated and discussed in the previous paragraphs.

Figure 11 shows the scanning electron microscopy (SEM) for the masses containing 30% of waste burned at the temperatures of 850 °C, 950 °C, and 1000 °C. The images demonstrate how the waste provided pore reduction in the ceramic matrix, especially in the 1000 °C image, where even with a $100 \times$ magnification in the material, the pores are not easily detected. It is verified that the WTP glass waste was satisfactorily homogenized in the ceramic matrix, since even evaluating the mass with higher waste contents, it is not possible to easily detect the presence of glass particles. This proves that the waste acts as a flux and forms a liquid phase, being incorporated into the matrix after burning, not just occupying empty spaces. In other words, the waste actually acts as a liquid-phase former, and not only as a material filling effect, as is the case with materials such as quartz sand [52].

Conclusion

The objective of this work was to evaluate the recycling of waste glass extracted from WTP into ceramic materials. The main conclusions obtained were:

- The characterization of clay and glass residue proved that both materials are suitable for use in ceramic materials. The presence of alkaline materials in the glass residue is a beneficial factor, because it contributes to the formation of a liquid phase during firing, improving ceramic properties.
- The plasticity analysis showed that the residue is not plastic, affecting the prognosis of the extrusion. With 10% waste, however, the ceramic mass was in the ideal extrusion zone.
- An increase in shrinkage was observed with the increase in the burning temperature and the percentage of waste used, which proves the formation of a greater liquid phase promoted by the glass waste. It was found that burning at 1050 °C causes excessive retraction for the masses containing residues, which discards the application of the material at that temperature.
- The values obtained for water absorption are compatible for application on ceramic bricks (maximum 25%) and ceramic coverings (maximum 22%).
- The flexural strength values obtained were greater than 2.5 MPa recommended for ceramic bricks in all composi-

Fig. 11 SEM of 30% composition ceramics



850°C



1000°C

tions. Only the composites with 20% burning at 950 °C or 1050 °C and the compositions with 30% burning at any temperature, except 950 °C, met the minimum value for application on 6.5 MPa tiles.

As a result, the feasibility of recycling glass waste into ceramic materials in different compositions is proven. Some examples of viable compositions are 10% waste burned at 1000 °C and/or 20% waste burned at 950 °C for ceramic tiles; and 30% of waste burned at 850 °C for ceramic bricks. Therefore, the objective of the article was achieved.

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