# Characterization of waste of soda-lime glass generated from lapping process to reuse as filler in composite materials as thermal insulation

## (Caracterização do rejeito de vidro sodo-cálcico da lapidação e sua utilização como carga em isolantes térmicos)

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#### Abstract

The beneficiation plate process by soda-lime glass lapping in the glass industry generates, an untapped residue (waste). The waste of this material is sent to landfills, causing impact on the environment. This work aimed to characterize and evaluate the waste of soda-lime glass (GP) lapping. After its acquisition, the GP was processed by grinding and sieving and further characterized by the chemical/mineralogical analysis (XRF, EDS and XRD), SEM morphology, particle size by laser diffraction, thermogravimetric analyses (TGA and DSC) and thermophysical analyses. It was observed that the GP particles are irregular and micrometric with the predominant presence of Na, Si and Ca elements characteristic of amorphous soda-lime glass. The assessment of the chemical/mineralogical, morphological, thermophysical and thermal gravimetric characteristics of GP suggest its reuse as reinforcing fillers or filler in composite materials to obtain thermal insulation.

**Keywords:** GP, soda-lime glass, glass waste, thermal insulator.

#### Resumo

O processo de beneficiamento de chapas por lapidação de vidro sodo-cálcico na indústria vidreira gera, por si só, um resíduo não aproveitado (rejeito). O rejeito deste material é encaminhado para aterros sanitários, ocasionando impactos ao meio ambiente. O presente trabalho objetivou caracterizar e avaliar o rejeito da lapidação de vidro sodo-cálcico (PV). Após sua aquisição, o PV foi processado por moagem e peneiramento e posteriormente caracterizado através das análises química/mineralógica (FRX, EDS e DRX), morfológica por MEV, de granulometria por difração a laser, análise termogravimétrica (TGA e DSC) e análises termofísicas. Observou-se que as partículas de PV são irregulares e micrométricas com a presença predominante de Na, Si e Ca, elementos característicos de um vidro amorfo sodo-cálcico. A avaliação das características química/mineralógica, morfológica, termofísicas e termogravimétrica do PV permitiu sugerir seu reaproveitamento como cargas de reforço ou de enchimento em materiais compósitos para obtenção de materiais isolantes térmicos.

Palavras-chave: PV, vidro sodo-cálcico, rejeito de vidro, isolante térmico.

#### INTRODUCTION

Glass is a non-crystalline inert material, non-porous and fragile and is considered as a thermal insulator. By presenting these features and being impermeable to the passage of oxygen or carbon dioxide, the use of glass debris as filler in the manufacture of PUR can offer a number of advantages, such as reduction of collection costs, reducing environmental pollution, improving the economy and reducing the consumption of natural resources [1]. On the other hand, there are also many applications for waste glass.

Gomes and Santos [2] fabricated a pre-molded piece, referred to as PAVER, consisting of waste construction material, stone powder, glass powder and cement. According to the results, the recycled material that showed the best efficiency was glass powder which replaced the RCC, and had the best mechanical resistance with 20.19 MPa (1:5

ratio) and 17.77 MPa (1:9 ratio), in 28 days of curing, with consistent values for concrete with waste construction material and no structural function for pre-molded pieces of concrete designed for sidewalks and gardens. It had a smaller void ratio if compared with the waste used in the samples and occurred so that the glass powder had a maximum diameter of 1.2 mm, filling the void space, and thereby improving the packaging.

The introduction of recycled glass, obtained by comminuting discarded bottles, in the manufacture of ceramic pieces in replacement of a part of sand associated with the clay-like material was evaluated [3]. The glass was mixed in proportions of 3, 5 and 10% with a clay-like material. They observed excellent results for introduction of recycled glass in the production of ceramic pieces, mainly by the positive effects from the ceramic properties of water absorption, apparent porosity, loss to fire and apparent

density, particularly when a 10% glass powder was used in the ceramic mass.

The sustainability of concrete and cement industries consists of using pozzolanic additions, especially if obtained from waste such as waste glass was analyzed [4]. Crushed waste glass was ground (WGP) and used in mortar as a partial cement replacement (0%, 10% and 20%) material to ascertain applicability in concrete. An extensive experimental program was carried out including pozzolanic activity, setting time, soundness, specific gravity, chemical analyses, laser particle size distribution, X-ray diffraction and scanning electron microscopy (SEM) on WGP and resistance to alkali silica reaction (ASR), chloride ion penetration resistance, absorption by capillarity, accelerated carbonation and external sulphate resistance on mortar containing WGP. The WGP10 mortar showed a remarkable resistance to sulphate attack, far higher than silica fume (SF). Although soda lime glass presents a high alkali content, use of ground waste glass as cement replacement in mortar, improved resistance to ASR and chloride penetration with replacement dosage and greatly improved sulfate resistance without compromising strength. Therefore, this waste material can successfully enhance durability and further contribute to sustainability in construction.

The behaviour of waste glass powders of different fineness with that of natural pozzolana, coal fly ash and SF was compared [5]. Blended both with Portland cement and lime, ground glass improved strength, resistance to chloride penetration and resistance to sulphate attack of mortars more than natural pozzolana and similarly to fly ash. Mortars with ground glass immersed in water for seven years did not show any sign of degradation, increased their compressive strength and were also not affected by fineness.

The feasibility of using soda-lime waste glass powder for latent heat storage application, where n-Octadecane was loaded into glass powder (GP) was studied [6]. The TGA and thermal cycling results confirmed that the composite PCM is thermally stable and reliable. A thermal performance test showed that the cement paste panel with composite PCM reduced the indoor temperature by 3 °C. The thermal performance test on a small scale showed that the n-octadecane-GP composite PCM is effective in reducing the indoor temperature, as well as the temperature fluctuations. Therefore, it can be used in buildings for thermal energy storage in order to reduce energy cost, the scale of air-conditioning and flatten the fluctuation of indoor temperature.

The effects of soda-lime waste glass, from the recovery of bottle glass cullet, in partial replacement of Na-feldspar for sanitary ware ceramic production were discussed [7]. Measurements have been performed by in situ high temperature X-ray powder diffraction, scanning electron microscopy, thermal dilatometry, water absorption and mechanical testing. The glass substituted feldspar from 30 to 50% wt allows one to accelerate the mullite growth reaction kinetics, and to achieve macroscopic features of the ceramic output that comply with the latest technical requirements.

Then, by accelerating new perspectives on energy saving by reducing the fuels required at the firing stage and as a consequence, the CO<sub>2</sub> emissions. On the other hand, the replacement of a part of feldspar implies savings in terms of natural resources, preservation of the landscape and effective management of glass cullet waste.

Waste glass and fly ash to prepare foam glass were studied [8]. The result from TG–DTG revealed that the appropriate foaming temperature was around 950 °C. The foam glass had a bulk density of 267.2 kg/m³, a compressive strength of 0.9829 MPa, and a porosity of 81.55%. According to these results, the starting materials and SiC were successfully employed as a high temperature foaming agent.

The feasibility of incorporating soda-lime glass originated from the glass lapidary process in the manufacturing of red ceramic products was demonstrated [9]. The characterization by means of fluorescence X-ray testing, X-ray diffraction, particle size distribution and thermal behavior confirmed the mineralogical and chemical similarity between the cutting waste glass and soda-lime glass, especially at the high level of SiO<sub>2</sub> (69%). They also found that in addition to this similarity, the use of residue glass contributed to the accelerated densification process during firing of the ceramic material, facilitating the glassy phase formation and enabling the production of red ceramic with appropriate technological properties.

The foam glass composite toughened by glass fiber prepared by sintering technique, using the waste from sodium-calcium silicon flat glass powder as the main raw materials was improved [10]. In this study, the preparation and properties of the samples were characterized by differential thermal analysis (DTA), field-emission scanning electron microscopy (FESEM) and mechanical property test. The specific strength of the composite was defined for the first time, and applied into the investigation of mechanical property. The results improved the bending strength by 10.45–22.26 MPa/(g/cm<sup>3</sup>), and the specific compressive strength of 30.45-34.34 MPa/(g/cm<sup>3</sup>) can be displayed when sintered at 790–815 °C with the addition of 5–25% wt. glass fiber. Good correlations between the microstructure (in particular the fiber distribution), the high specific strength and the high modulus of elasticity of glass fibers. The mechanically improved foam glass composites toughened by glass fiber were successfully prepared.

The multiple benefits of the new recycling process to RFG (recycled foam glass) were investigated [11]. They mainly used a mix of rejected waste glass from conventional container glass recycling and waste of special glass such as monitor glass, bulbs and glass fibers. According to their research, the green building product is a RFG to be used in high efficiency thermally insulating and lightweight concrete. The environmental gains have been contrasted against induced impacts and improvements have been proposed. In accordance with an eco-efficiency principle, a recourse has been highlighted to high energy intensive recycling which should be limited to waste that cannot be closed-loop recycled.

Building material (foam glass granules) that is much lighter and is registered with the properties of heat insulation and acoustics improved based on cullet in order to recycle it and for improving the present laws about the waste products was obtained [12]. The grinding of waste glass to particle sizes of less than 0.1mm was conducted by adding 1% of CaCO<sub>3</sub> content to provide the production of material with particle density of 0.5 g/cm³, strength of 17.50 MPa, water adsorption of 95% and presented excellent thermal (0,031 W/m-K) and acoustic (15 dB) properties.

The use of glass in the production of new composite materials and through his work on glass powder sintering (GP) was studied [1], concluding that as the average particle size was reduced (98.6 μm, 30.7 μm, 14.9 μm), the greater its chemical reactivity due to increased surface energy. From this analysis, he noted that the initial temperature of shrinkage during the sintering process was directly proportional to the size of these powders. Consequently, there would be a decrease in energy consumption and environmental impact caused by the generation of gas. The size of the glass particles can influence the volume expansion process of rigid PU foam, a fact that has already been noted [13] by obtaining vitreous foams using waste glass. This behavior by adding the glass powder (GP) from waste glass in the rigid foam castor matrix was shown [14, 15]. They observed that with the addition of glass powder in a percentage in the manufacture of composite castor polyurethane foams (PURM), the thermal conductivity showed similarities with the pure PUR, especially the PURM-GP 5 and PURM-GP 10 composites, then this waste glass can be used by being applied as thermal insulation.

The characterization of GP residue coming from the glassware industry aimed to provide technological and environmental alternatives for recycling and application of waste; the properties were investigated by thermogravimetric, morphological, chemical, thermophysical and mineralogical GP analysis.

## MATERIALS AND METHODS

This work dealt with the characterization of GP through particle size analysis by laser diffraction, chemical/mineralogy by XRF and XRD, morphology by SEM and thermogravimetric (TGA/DSC) after recycling process for refinement of its particles to ensure greater applicability of waste arising from the glass cutting and decrease its disposal into the environment.

Acquiring GP from the Lapidary Waste Glass: the waste glass (given by DVN Glasses LTDA., Natal, RN, Brazil) was passed from the refining process through grinding using mortar and pestle (Fig. 1a) to reduce the particle size.

The coarse powder was sieved (ABNT # 20-850  $\mu$ m) in its wet state and grinded using horizontal mill balls (Fig. 1b) to obtain micrometric particles (Fig.1c). Then a mechanical sifter used for selection of glass powder particle sizes GP.







Figure 1: Process of refining glass powder particles: (a) mortar and pestle (b) ball mill, and (c) the aspect of powder after sieving. [Figure 1: Processo de refinamento da partícula de pó de vidro: (a) almofariz e pistilo, (b) moinho de bolas e (c) aspecto do pó após peneiramento.]

The GP was trapped between the sieves ABNT # 270 (53  $\mu$ m) and # 230 (63  $\mu$ m). Finally, GP was dried in an oven for further characterization.

Characterization methods: the determination of average particle size was performed by the technique of

granulometry by laser diffraction particle size analyzer, Cilas 920. Thermogravimetry analysis was performed in Netzsch, STA 449 F3 Jupiter equipment. The GP sample was heated for 30 min to 1500 °C at a heating rate of 5 °C/min. under continuous flow of 50 mL/min Ar, and Al<sub>2</sub>O<sub>3</sub> as an inert reference material.

The chemical and morphological analyzes were performed using the X-ray fluorescence techniques (XRF Shimadzu 1800) in the semi-quantitative mode, X-ray diffraction (Shimadzu XRD-7000 , 30 kV, 30 mA, scan  $5^{\circ}$ /min with Cuk $\alpha$  radiation), energy dispersive spectroscopy (Hitachi SwiftED 3000, with chemical microanalysis with 161 eV resolution) and scanning electron microscopy (Hitachi TM300, BSE, 5 kV).

Thermophysical analysis: the thermal properties (thermal conductivity, thermal diffusivity and specific heat) were evaluated in a KD2 Pro equipment, with a SH-1 (double thermal needles) sensor to collect data after 2 min insertion in the specimen. This sensor operates in the range of 0.02 to 2.00 W/m-K. Measurements were performed at  $26 \pm 1$  °C with relative humidity  $32 \pm 1\%$ .

#### RESULTS AND DISCUSSION

Particle size: GP particles trapped between the sieves with ABNT # 270 and # 230 mesh were classified with average diameters of  $D_{10}$ ,  $D_{50}$  e  $D_{90}$ , with cumulative measures of 10%, 50% and 90% of the total sample analyzed, respectively. Fig. 2 shows a histogram of particle size distribution of the obtained particles generated, in which the median diameter of 32.59  $\mu$ m with mean sizes 10, 50 and 90% (cumulative values) were 2.28  $\mu$ m, 28.00  $\mu$ m and 71.31  $\mu$ m, respectively. This dispersion in particle diameter is inherent to the mechanical milling GP process. According to Fig. 2, it can be seen that during this testing, it was noted that the particles dispersed upon contact with water and thus the obtained particles were smaller in major

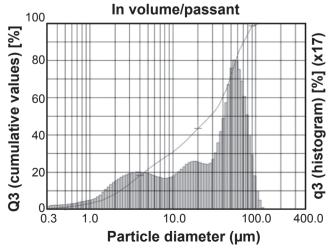


Figure 2: Histogram of particle size distribution of the glass powder. [Figura 2: Histograma da distribuição granulométrica do pó de vidro.]

proportion. This can be justified by the presence of moisture during the screening process of the GP, which favored the agglomeration of the particles. Also, according to this analysis the GP particles can be classified having diameter  $D_{\rm 50}$  of 33  $\mu m$ .

*X-ray diffraction and X-ray fluorescence*: diffractogram of GP presented in Fig. 3 shows the absence of crystalline phases; this spectrum represents a typical amorphous band around 27° derived from the presence of silica in the sample. This features GP as an amorphous solid without symmetry and/or long rang periodicity in the atomic arrangement. These values corroborate with these presented by the waste glass from CRT (Cathode Ray Tubes) computer monitors characterized by [16]. The chemical composition in percent [%] of surrounding oxides is presented in Table I.The predominant presence of SiO<sub>2</sub> as the forming agent of the glass network can be observed, while Na<sub>2</sub>O and CaO are treated as typical sodalime glass. The importance of oxides observed in Table I, highlighting their influence on properties of analyzed GP was commented [15]. A great potential for recycling waste glass is that has a large amount of silicon oxide, which is responsible for forming the glass network [16].

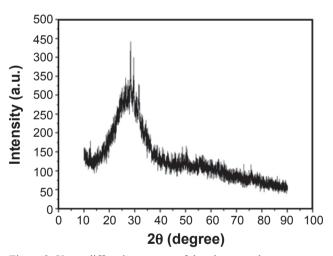


Figure 3: X-ray diffraction pattern of the glass powder. [Figura 3: Difratograma de raios X do pó de vidro.]

Table I - Glass powder chemical composition [oxides, %] obtained by XRF.

[Tabela I - Composição química do pó de vidro (óxidos, %) obtida por FRX.]

Oxides	Percentage	Oxides	Percentage
SiO <sub>2</sub>	59.245	Fe <sub>2</sub> O <sub>3</sub>	0.212
CaO	20.436	$SO_2$	0.203
Na <sub>2</sub> O	10.712	$IO_3$	0.057
MgO	3.969	TiO <sub>2</sub>	0.024
$ZrO_2$	3.411	$SnO_2$	0.019
$Al_2O_3$	1.156	$P_2O_5$	0.006
$K_2O$	0.551		

Thermogravimetric analysis: Fig. 4 shows the graph with curves obtained by means of thermogravimetric (TG) and differential calorimeter (DSC) of GP. They indicate the thermal behavior of the GP, i.e. the weight loss and power variations during the heating of this material, where it is possible to extract information about possible reactions and transformation phases. A loss of the initial mass of about 10.3% to a temperature of 235 °C is observed in the TG curve of Fig. 4, which is related to the loss of water/moisture of the material. In the temperature range of 235.6 °C and 716.4 °C, smaller weight loss (5%) occurred. The transformation of quartz  $\alpha$  to quartz  $\beta$  occurred at about 593 °C, which is located in an endothermic band. The exothermic peak in the DSC curve (-0.65 µV/mg), i.e., greater energy release occurred at 390.9 °C. This temperature is related to the onset of decomposition of the carbonates. From ~716 °C, the material showed thermal stability with no significant weight loss up to 1500 °C.

The formation of sodium silicate is known as vitrification, which contributed to the thermal stability of the material [13]. In the evaluation of the thermograms of four types of sodalime glass starting from ornamental rocks, an exothermic peak at 480 °C was found, the decomposition range belongs to magnesium carbonate, followed by decomposition of calcium carbonate (600 °C) and sodium (850 °C) [17].

Morphology (SEM) and Microanalysis Chemistry (EDS): the SEM micrographs are shown in Fig. 5, at magnifications of 500 and 2000 times. The chemical microanalysis EDS was used to identify the composition of the points A and B selected in (Fig. 5b), which is broken down in Fig. 6. It can be seen in Fig. 5 that the particles have irregular geometry with sizes below and above 33 μm, a fact evidenced from the technical particle size by laser diffraction. Its disintegration is due to this sample having suffered dehumidification in a greenhouse before this analysis. As was shown by XRF analysis, the chemical compositions shown by EDS analysis show large percentages of oxygen, silicon, calcium, sodium and aluminum, which are the predominant elements of typical soda-lime glass.

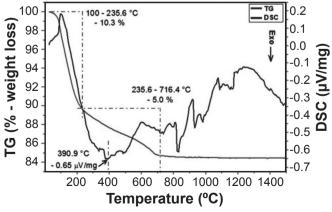
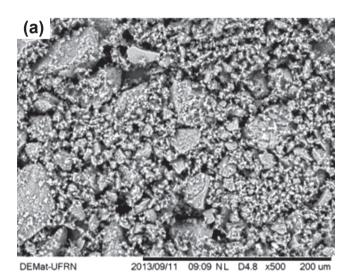


Figure 4: TG and DSC curves of GP. [Figura 4: Curvas TG e DSC do PV.]

Analysis of the Thermal Conductivity, Thermal Diffusivity and Specific Heat: the thermal conductivity values (W/m-K), specific heat (MJ/m<sup>3</sup>-K) and the PV thermal diffusivity presented the following respective results: 0.161  $\pm$  0.014 W/m-K; 1.292  $\pm$  0.063 MJ/m<sup>3</sup>-K and 1.25x10<sup>-7</sup>  $\pm$ 5.86x10<sup>-9</sup> m<sup>2</sup>/s. It was noted that the GP can be used as filler or reinforcement in thermal insulation. According to [18], the glass is characterized as a thermal insulation, whose thermal conductivity is between 0.72 and 0.86 W/m-K and for the typical glass, the specific heat is 669 J/kg-K. Attila, Güden, Taşdemirci [19] investigated thermal conductivity behavior of a powder residue waste of a soda-lime window glass used in foam glass, which showed values between 0.048 and 0.079 W/m-K. These observations showed that the foam density was proportional to thermal conductivity. The thermal conductivity of cement paste disk prepared with and without n-octadecane-GP composite PCM was evaluated [6], obtaining 0.62 W/(m-K) and 0.9 W/(m-K), respectively. It was suggested that the thermal conductivity



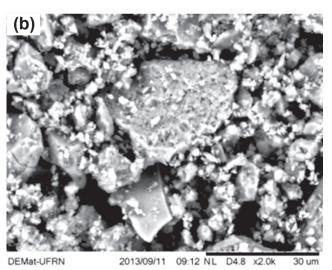
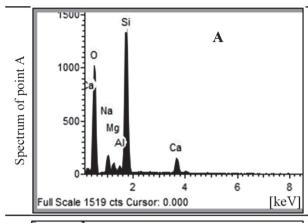
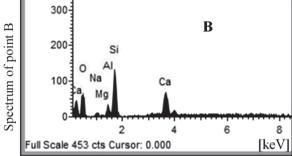


Figure 5: SEM micrographs of glass powder at magnifications: (a) 500x (b) 2000x.

[Figura 5: Micrografias por MEV do pó de vidro com ampliações: (a) 500 x e (b) 2000 x.]



Element	Weight [%]	Atomic [%]
Oxigen	61.566	73.787
Sodium	4.758	3.969
Magnesium	1.769	1.395
Aluminum	0.900	0.639
Silicon	26.309	17.962
Calcium	4.698	2.248



Element	Weight [%]	Atomic [%]
Oxigen	54.337	70.249
Sodium	1.864	1.677
Magnesium	0.368	0.313
Aluminum	3.748	2.873
Silicon	20.007	14.734
Calcium	19.676	10.154

Figure 6: EDS diagram of GP identifying the points A and B of Fig. 5. [Figura 6: Diagrama EDS do PV com identificação dos pontos A e B da Fig. 5]

could be improved by incorporating various additives such as graphite powder. The formation of vitreous foams produced by [20] present adequate thermal insulation (between 0.16 and 0.08 W/m-K) mainly with optimized compositions containing 90% glass and 10% graphite.

## **CONCLUSIONS**

The work dealt with the use of waste derived from the lapping of glass powder production (GP) through its refining and its chemical characterization, thermogravimetric, morphological and thermophysical analyses aiming for noble applications mainly as a thermal insulator material, to the contribute to the environment by reducing pollution and especially decreasing the extraction of raw materials. The main conclusions are: the refinement of the GP generated micrometric particles (D<sub>50</sub>) of 33 µm; Afeter XRD and XRF analyses, it can be confirmed that the material was an amorphous soda-lime glass with the predominant presence of Si, Na and Ca; the thermograms showed major spots where thermal degradation of the material occurred, i.e. its weight loss, decomposition of carbonates, followed by transformation of quartz  $\beta$  to  $\alpha$ ; the morphological analysis revealed the presence of GP particles of various and irregular diameters and absence of agglomerates; the thermophysical analysis of the GP showed results in an equivalent range to other developed thermal insulator materials for this application; it is suggested to reuse it as reinforcing fillers or filler in composite materials to obtain thermal insulation.

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