

Recycling of construction and demolition waste generated by building infrastructure for the production of glassy materials

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Abstract

The use of waste materials generated by construction and demolition industry to yield valuable glassy materials, i.e. enamel for glazed ceramic tiles and cellular glasses is presented in this study. Both types of materials are produced by one-step treatment at moderate temperatures after simple waste chemical composition adjust. The enamels are manufactured directly from the initial waste powder by melting, while the expanded materials result from mixing of the vitreous material obtained after waste vitrification with an adequate foaming agent and posterior thermal treatment. Through the manuscript the feasibility of one step production of second generation profit materials is discussed in order to help achieving sustainable development and environmental protection.

Keywords: construction and demolition waste, expanded materials, enamel, glazed tiles

Introduction

In the last century the construction industry, either building or assembling, reached very high activity indexes. Although a sign of wellness, and considered as one of the key industries for economical worldwide growth, this industry is accompanied with an extraordinary waste generation. Thereby, construction and demolition waste (CDW) is considered as the heaviest and bulkiest waste in the European Union (EU), representing about 25% - 30% of all generated waste. The CDW issued principally from activities such as construction or demolition of buildings and civil infrastructure, road planning and maintenance varies in composition and contains different materials, like concrete, bricks, gypsum, wood, glass, metals, plastics, solvents, asbestos or excavated soil, many of which can be recycled [1].

Referring to the Age of sustainable development, the waste generation problems must be addressed, especially in the construction industry, where valuable raw materials can result. In this sense, CDWs are considered as priority treatment for European Union. In addition, CDWs present high potential for recycling and reuse, since some of its components possess high resource value. For example, aggregates derived from CDW are frequently reused in roads, drainage or other construction projects [2,3]. Nowadays the technology for separation and recovery of CDW is well established, being readily accessible and in general inexpensive. However and despite its potential, the percentage of recycling and material recovery of CDW varies greatly (from 10% to 90%) across the EU [1].

In this context, it is worth to mention the European zero waste program, which estimates that the waste management could reduce the material's input needs by 17–24% in 2030, representing an overall saving potential of €630 billion/year for the

European industry. The later will lead to the satisfaction of 10% to 40% of the raw materials demand, while contributing to achieve also the EU target to reduce greenhouse gas emissions by 40% [4].

Waste reduction, reuse and recycling are very important elements in the waste management resulting in natural resources conservation, valuable landfill space reduction, raw materials and energy needs diminishing, air and water pollution control and, not at last place, new jobs creation potential [5]. There are, however, some constraints on reusing waste materials; they must fulfill some engineering requirements in terms of physical and compositional properties and they should not contain excessive amounts of harmful components which might cause problems in use [6].

The properties of CDWs vary considerably depending on its origin and composition. It is convenient to distinguish between materials originated from construction and demolition of buildings and those from pavement. The former offers composition in which a wide variety of wastes are included, presenting sometimes even dangerous components, which can contaminate other recyclable items and are time and money spending. The vitrification is reported as the safest approach for non flammable hazardous waste treatment [7]. It is a process able to convert directly the waste materials without separation into homogeneous stable glasses for subsequent application [8-10]. However, vitrification is reported as high-energy consuming treatment, producing glasses with low transparency. The use of this treatment appears more expensive than waste deposition in landfills or waste immobilization into cementitious matrix. Thus, a possible cost reduction of this technology involves conversion of the produced glassy materials into directly marketable products such as cellular glasses and enamels for glazed ceramic tiles production [11].

The cellular glasses are light rigid materials, with low thermal conductivity, good mechanical properties and acoustic insulation capacity [12]. Being fire and waterproof materials, the cellular glasses are increasingly considered in civil engineering as insulating or lightweight filling materials [13-15].

Cellular glasses are formed in two steps: i) viscous flow sintering of fine glass powders and ii) subsequent foaming of the pyroplastic mass with specific additives under heating at 850–1000 °C [14]. The foaming process occurs upon releasing different gases (CO, CO₂, SO_x) generated from the thermal decomposition of the additives (typically carbonates [16] or sulfates). However, the gas release may represent an environmental problem. An alternative to the above-mentioned foaming process is cellular glass production by means of redox reactions between additives and glass components. However if carbon-containing species (C, SiC) are used as additives greenhouse effect gases will be produced, meanwhile the use of iron(III) oxide or aluminium nitride [14, 17], producing oxygen or nitrogen during redox reaction, are examples for environmentally friendly process.

The enamel is a glassy substance chemically formulated to adhere on the surface of preformed tile and subsequently fused into the body when fired. It is essentially composed by silica (glass forming element), alumina (for stability) and additives (to help melting). Various minerals, oxides and chemical compounds could be also employed for color.

The CDWs due to their composition are good candidates for being recycled as raw materials for the production of the above-mentioned materials. In this context, the present work is devoted to the feasibility of glassy materials directly obtained from construction waste as marketable products with a special emphasis made on material's final state and their possible future quick utilization without any further treatments.

1. Materials and methods

Three groups of CDWs are used as raw materials, i.e. bricks, glazed tiles and concrete wastes. Additionally, some commercial products are employed either to adjust chemical compositions (MgO, SiO₂ and NaOH) or as foaming agents (AlN and CaCN₂).

The chemical composition of the samples is determined by X-Ray microfluorescence spectrometry (XRMF) in an EDAX Eagle III spectrophotometer using Rh source of radiation.

X-ray diffraction (XRD) analysis is performed on X'Pert Pro PANalytical diffractometer using Cu-K α radiation ($\lambda = 1,5404 \text{ \AA}$), working at 40 mA and 45 kV and equipped with position sensitive detector. The diffractograms are recorded over 2θ -range $5-80^\circ$ using $0,05^\circ$ step size and 80 s step time.

Thermogravimetric analysis is carried out on Seiko Exstar 6000 thermobalance, up to 1200°C in air, using a heating rate of $10^\circ\text{C}\cdot\text{min}^{-1}$.

The material's density is measured by the He pycnometry method on Pentapycnometer 5200e Quantachrome Instrument.

The tribological properties are tested on Microtest® pin-on-disk apparatus, providing continuous measurement of the material's friction coefficient.

The mechanical strength analysis is carried out on Microstest EM1/FR under compression strength.

2. Results and discussion

All waste materials are milled prior any characterization or use. The chemical compositions obtained by XRMF are shown in table 1. For bricks and glazed tiles silicon is the main element according to the preponderance of silica and silicate phases, observed in the diffractograms (figures 1, brick, and 2, glazed tile). Similarly, the concrete sample is rich in silicon and calcium, in agreement with the presence of silica, silicates and especially calcium carbonates as main crystalline phases (figure 3).

Table 1. Chemical composition of the used CDW (oxides wt%)

Waste	% weight							
	SiO ₂	CaO	Al ₂ O ₃	Fe ₂ O ₃	K ₂ O	Na ₂ O	MgO	Others oxides
Brick	69,7	5,9	14,2	4,7	3,7	1,6	--	0,13 (V, Mn)
Glazed tile	68,7	7,9	15,4	4,9	3,0	--	--	0,01 (Mn)
Concrete	20,4	67,0	1,5	2,7	0,7	--	7,4	0,4 (Ti)

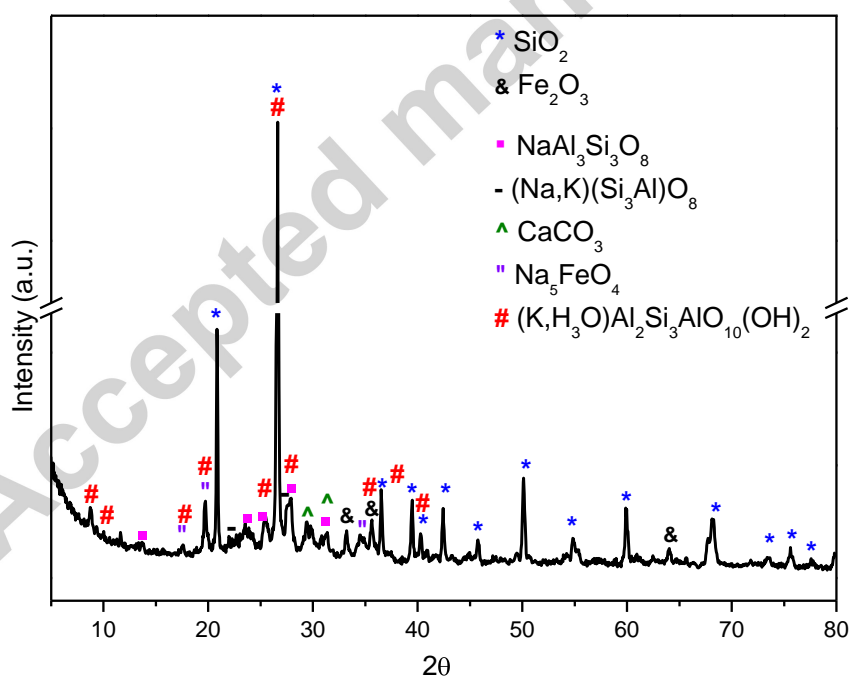


Figure 1. Diffractogram of brick sample

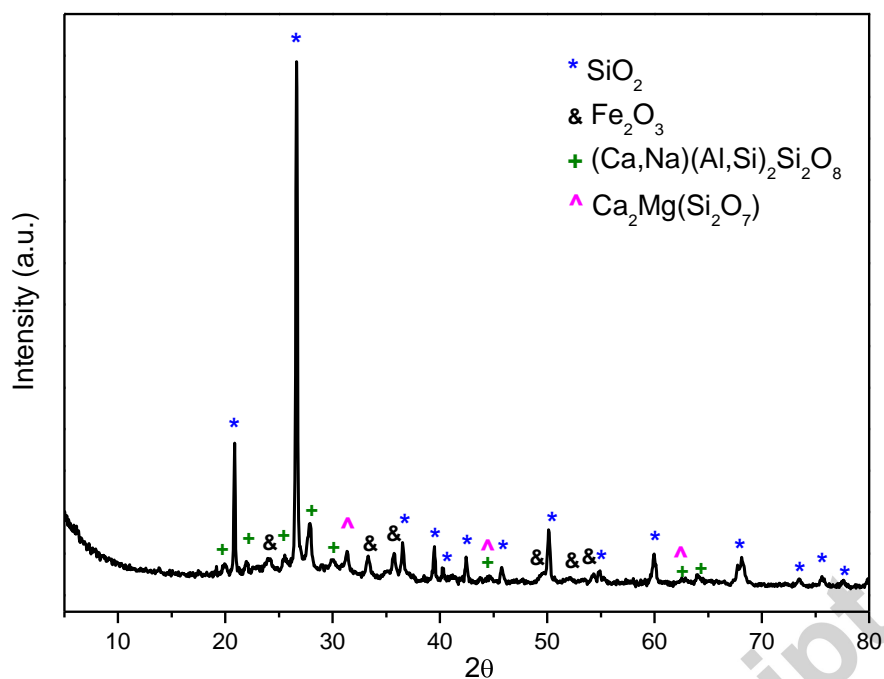


Figure 2. Diffractogram of glazed tile sample

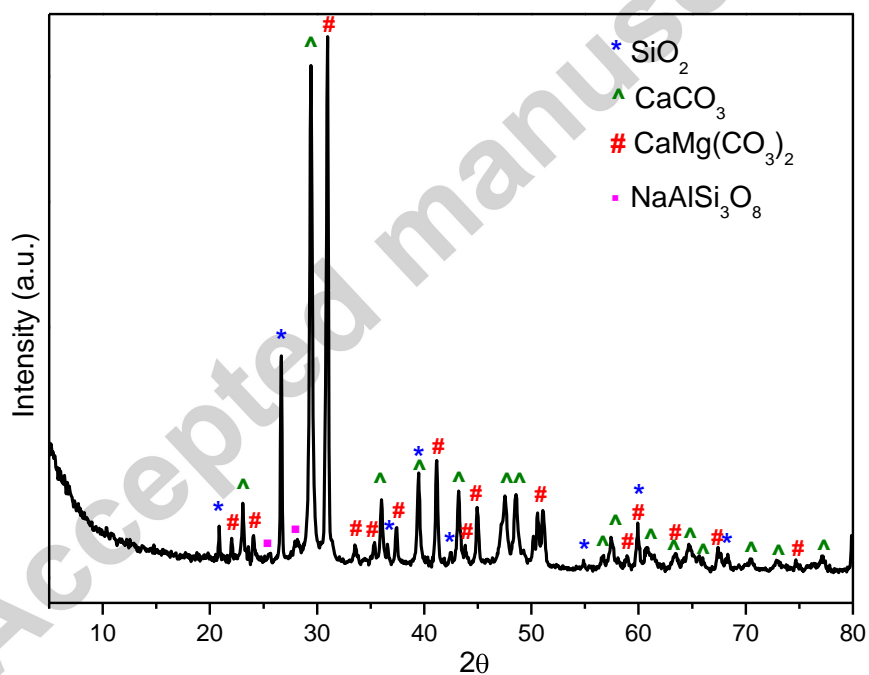


Figure 3. Diffractogram of concrete sample

In the case of the brick sample, seven characteristic phases are identified by XRD (figure 1): iron (III) oxide, Fe_2O_3 (JCPDS#01-073-0603); silica, SiO_2 (JCPDS#01-078-1253), two feldspars $\text{NaAlSi}_3\text{O}_8$ (JCPDS#00-009-0466) and $(\text{Na,K})(\text{Si}_3\text{Al})\text{O}_8$

(JCPDS#00-009-0478), illite (JCPDS#00-026-0911), calcium carbonate, CaCO_3 (JCPDS#01-086-2334) and sodium iron oxide, Na_5FeO_4 (JCPDS#00-036-0874).

For the glazed tile sample, the aluminosilicate occurrence is confirmed by the presence of $(\text{Ca},\text{Na})(\text{AlSi})_2\text{Si}_2\text{O}_8$ (JCPDS#00-020-0528); together with crystalline calcium magnesium silicate, $\text{Ca}_2\text{MgSi}_2\text{O}_7$ (JCPDS#01-076-0841); iron (III) oxide Fe_2O_3 (JCPDS#01-073-0603) and silica, SiO_2 (JCPDS#01-078-1253) (figure 2). Manganese traces determined by elemental analysis were not identified as crystalline phase.

The components of the concrete sample are identified by XRD as calcium carbonate, CaCO_3 (JCPDS#01-086-2334) and calcium-magnesium carbonate, $\text{CaMg}(\text{CO}_3)_2$ (JCPDS#00-036-0426), silica, SiO_2 (JCPDS#01-078-1253) and sodium aluminosilicate, $\text{NaAlSi}_3\text{O}_8$ (JCPDS#00-009-0466) (figure 3).

Usually the main problem of reusing CDW wastes is their high aluminum content, which indicates necessity of high temperatures for vitrification. Jordan et al. [18] reported the elaboration of soda-lime type glass at temperatures around 1100°C from natural zeolite with comparable aluminum content to that observed for our brick and glazed tile samples. Based on this study, and on the fact that the compositions of the waste materials, either bricks and or glazed tiles, are very similar between them, the later is selected for the preparation of two samples (Z1 and Z2) with compositions reported by Jordan et al. [18]. To re-adjust the glassy precursor composition various additives are employed: MgO , SiO_2 and NaOH . The relative proportions of waste material and additives and the corresponding chemical compositions of the glassy precursor are listed in table 2 and table 3 respectively.

Table 2. Mixtures compositions, wt.% of wastes and commercially available additives

Component (wt%)	Brick	Concrete	Glazed tile	MgO	SiO ₂	NaOH
Z1			75,59	0,75	22,68	0,98
Z2			89,10	0,89	8,91	1,15
Z3	50,33	11,18			30,01	8,48

Table 3. Chemical composition of the prepared powder mixture (oxides wt%)

Sample	Na ₂ O	Al ₂ O ₃	SiO ₂	K ₂ O	CaO	V ₂ O ₅	MnO	Fe ₂ O ₃	TiO ₂	MgO
Natural zeolite [18]	2,00	11,70	69,00	1,31	4,20	0	0	2,10	0,14	0,41
Z1	0,76	11,67	74,79	2,3	5,99	0	0,01	3,73	0	0,75
Z2	0,89	13,75	70,29	2,72	7,07	0	0,01	4,39	0	0,88
Z3	7,5	7,48	68,72	1,97	10,70	0,05	0,01	2,7	0,03	0,83

The vitrification temperature is fixed to 1200 °C, with a heating rate of with 10°C.min⁻¹ and 4h duration. The sample is then cooled down to room temperature under the inertia of the oven without special precaution considered.

Figure 4 presents the diffractograms corresponding to the samples Z1 and Z2 before and after thermal treatment. Signals due to the presence of crystalline phases are detected together with the characteristic halo of all amorphous materials.

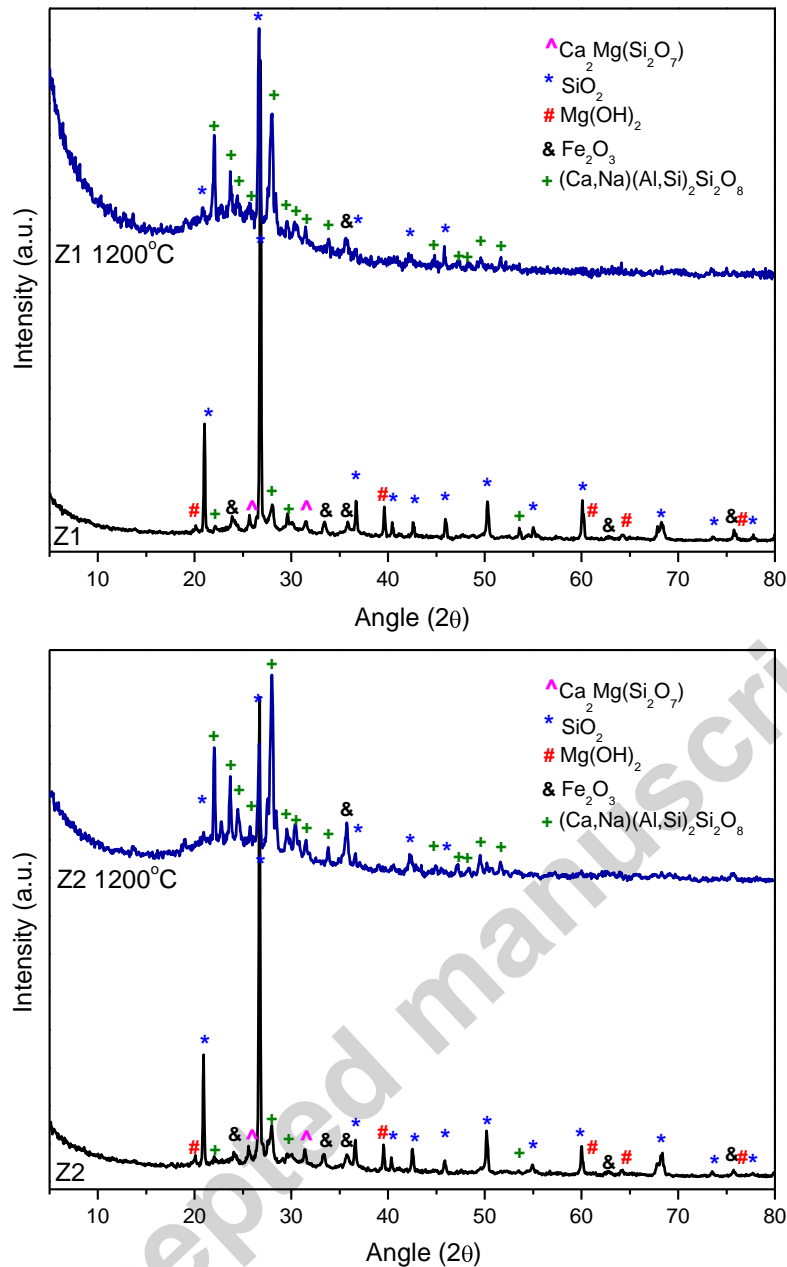


Figure 4. Diffractograms of samples Z1 and Z2 before and after thermal treatment

It is clear from the analysis that the sample's higher alumina content does not allow complete vitrification, that is why, a new waste mixture with lower Al_2O_3 and higher Na_2O proportion is prepared where the later, acts as flux. Instead of glazed ceramic tile, used for Z1 and Z2, an additional sample is prepared from brick and concrete, being both the main components of CDW [19]. Some additives such as SiO_2 and NaOH are added to the milling process of the CDW and the final mixture results in the Z3 sample.

Their relative proportions and corresponding chemical compositions are presented in table 2 and table 3.

For Z3 sample, the thermal treatment at 1200 °C leads to completely vitreous material with diffraction pattern typical for an amorphous material (Figure 5).

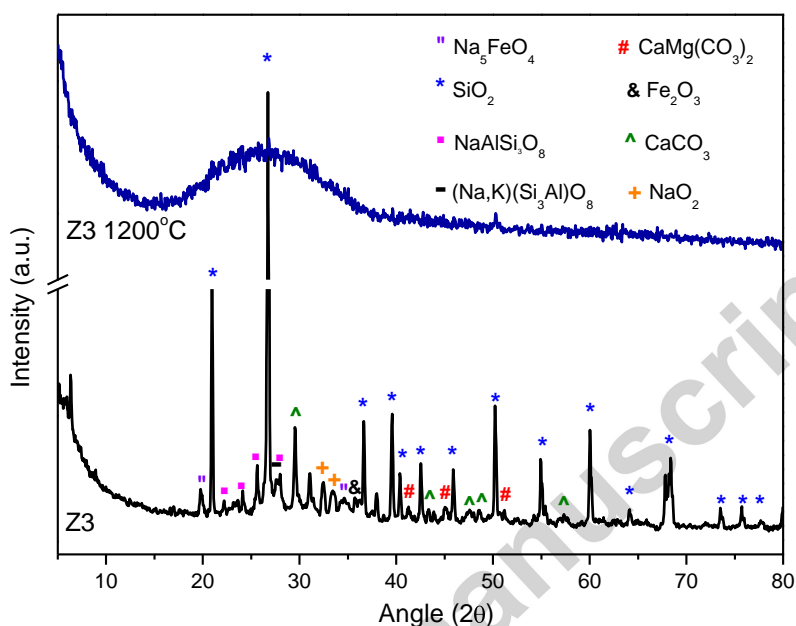


Figure 5. Diffractogram of sample Z3 before and after thermal treatment

The validation of the vitrification temperature obtained by thermogravimetric analysis is shown in figure 6. A slow mass loss process (approx.1%), is observed in the room temperature - 500 °C range, and should be associated to processes like dehydration and evaporation normally accompanied by an endothermal uptake, however, in simultaneous equipment (TG + DSC as in our case) the heat flow measurements lacks precision. In the 600-1000°C range few different process are possible. Taking into account that the raw materials, bricks and concrete in particular, are already thermally treated the presence of clay materials, although not discarded, is considered as minor component. Only a few traces of illite are detected by XRD, suggesting that the brick used in this study is based on this clay. This phase could be responsible for the

endothermic/exothermic reactions occurring between 500 and 800 °C caused by the dehydroxilation of the octahedral sheet and subsequent hydroxyl groups removal from the tetrahedral sheet [20], processes related to small mass lost (<0.25%). The high temperature heat release (around 980°C) could be also related to this phase and assigned to its transformation to the corresponding spinel. López-Arce et al. [21] also reported similar processes; they linked the small endothermic (700 °C) and exothermic peaks above 850°C to the destruction of illite and formation of new phases in a complex fluid–gas process. However, the presence of delaminated clays like kaolinite could not be excluded, and the high temperature exothermic process assigned to its transformation to pseudomullite [22].

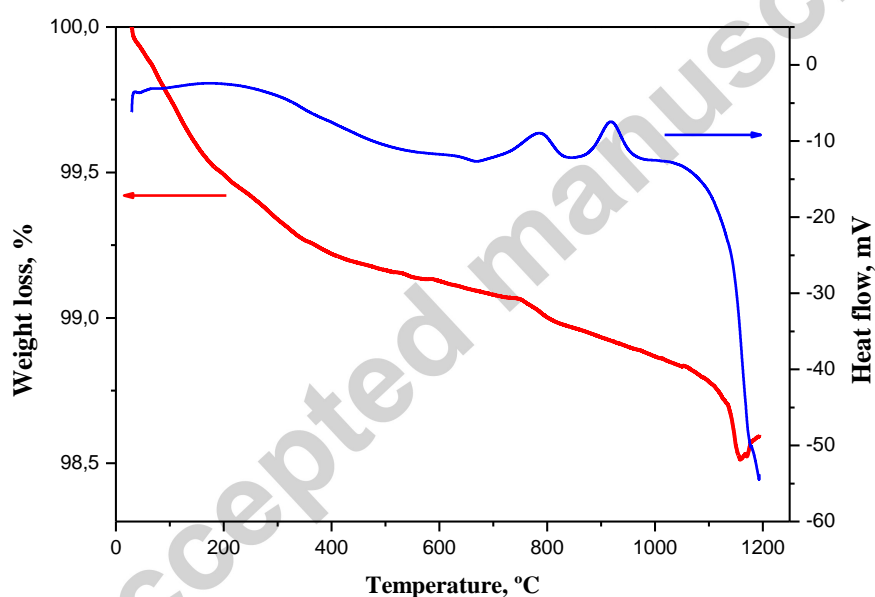


Figure 6. Thermogravimetric analysis of the Z3 sample

2.1. Glazed ceramic tiles

According to the above-mentioned results, the only composition that completely vitrifies (Z3) is selected to produce glazed ceramic tiles. Two different strategies are used; i) the mixture Z3 directly applied on the ceramic piece and subsequently fused and

ii) firstly the glass is obtained, milled and then deposited on the ceramic piece and secondly fused.

The first method could be considered as the most direct and simple one, saving costs of one fusion processes. This procedure repeated by using different weights of sample (2, 4 and 20 g) leads to similar results, pointing to easy scale-up of the method. The raw materials are mixed in adequate proportions (mixture Z3, table 3) and milled at 300 rpm in a planetary mill during 30 min. The resulted powder is wetted with water (powder/water ratio of 1.33) in order to obtain slurry with adequate viscosity to spread on ceramic tile. The piece is then dried at 120 °C for one hour and 12 hours at room temperature. Finally, the heat treatment is performed to form a glass on the ceramic base (1200 °C for 4 hours with a ramp of 10°C.min⁻¹ and then cooling).

In the second method, the Z3 mixture (table 3) is melted at 1200 °C, quenched in water to obtain glass, then milled and sieved with the fraction under 50µm retained. The XRD pattern at this stage, as expected, corresponds to an amorphous solid (not shown). The vitreous material is then used to prepare slurry by mixing with water and spread on the ceramic pieces. The temperature of the thermal treatment is 1100 °C for 4 hours with a ramp of 10°C.min⁻¹ and cooling the reason being that starting from already prepared vitreous material, the second melting process needs lower temperature (1100° vs 1200 °C).

Figure 7 shows the glazed ceramic tiles prepared using both methods. When the first is employed, bicolor glass coating is achieved. On the contrary, the second method results in coating with uniform color, suggesting compositionally more homogeneous coverage of that obtained by the first method. However, this coating appears to be more porous, suggesting worse mechanical properties.

The easier preparation of the first method sample prior vitrification, compositional adjust of the CDW and milling, directly balances the choice toward the use of this method for glazed tile production.

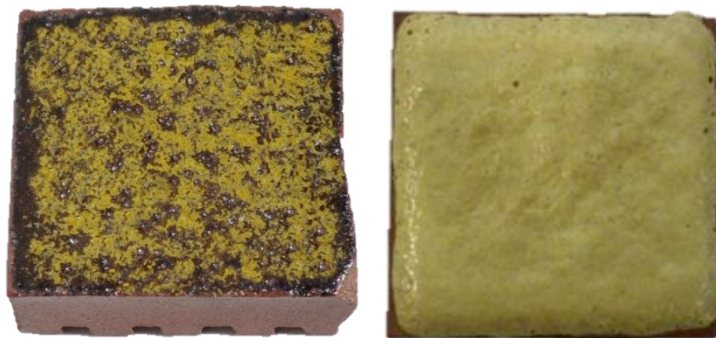


Figure 7. Glazed pieces obtained by the first (left) and the second (right) method

In order to compare the quality of the resulted glazed tiles obtained by the first method with commercially available tiles two standard tests for chemical resistance and friction are carried out.

Chemical resistance is tested according to the UNE EN ISO 10545-13 norm. Referring to that norm, one of the most unfavorable tests for chemical resistance is to use concentrated solution of potassium hydroxide (100 g/L). After the results, the glazed pieces prepared by the first method could be classified as GHA(V) class, without visible effects, the highest category considered by the norm.

The tribological properties of the coating are evaluated by ball-on-disk friction tests in unlubricated sliding against 6-mm carbide balls. The test parameters are set to 5 N of applied load, $40 \text{ m}\cdot\text{s}^{-1}$ of linear speed, radius track between 6-10 mm and 2000 m of sliding distance. The average width of the striation produced by the friction test is similar for both tiles, prepared and commercial (583 vs 570 μm , respectively). Both materials present the same friction coefficient ($f \sim 0,7$), Figure 8, which, according to

the ISO 13006:2012 norm allows classifying the glazed ceramics as Class 2, as a material to be used when a high slip resistance is needed.

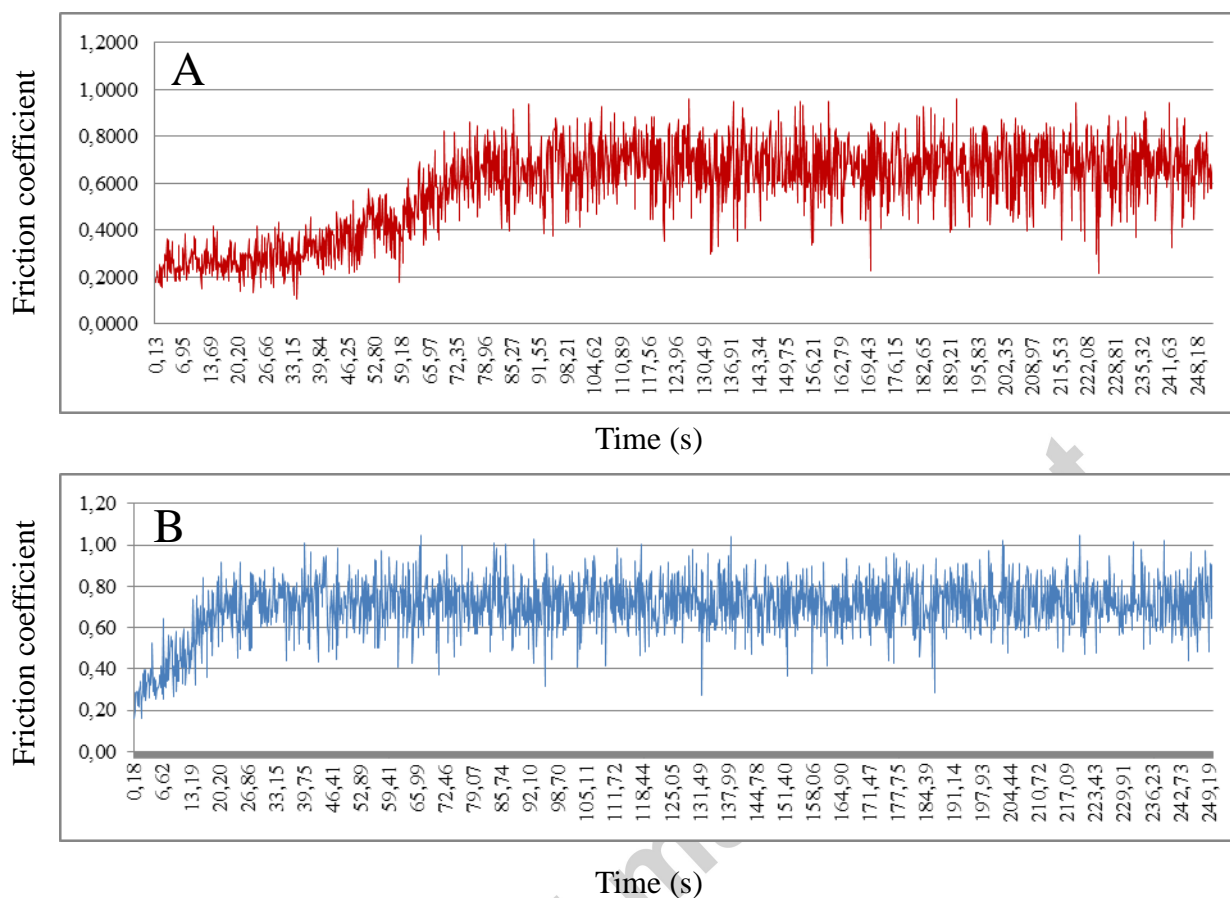
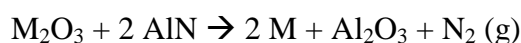


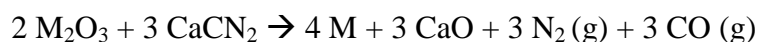
Figure 8. Friction coefficient versus time. (A) Commercial glazed tile. (B) Prepared glazed tile.

2.2. Cellular glasses

The second part of this work is devoted to the preparation of cellular glasses directly from the CDW with the aim to re-use in building insulation. Aluminum nitride and calcium cyanamide are used as foaming additives. Both compounds could react with metallic oxides present in the starting material (Z3 glass) through redox reaction. The redox reaction that takes place between aluminum nitride, AlN, and a metallic oxide M_2O_3 is:



On the other hand, calcium cyanamide (CaCN_2) is a product commonly used in agriculture, with the advantage of lower cost compared to aluminum nitride, but with disadvantage of generating CO during the reaction:



The presence of iron oxide in the CDW (table 3) used in the preparation of the glass enables such redox reactions during the melting process. Therefore, expansion of the molten material is possible. Both, aluminum nitride and calcium cyanamide yield to positive results as foaming agents.

The porous materials are prepared by joint milling of the Z3 glass powder and foaming agent. Then the obtained mixture is thermally treated at 1100°C during 85 min, with a heating rate of 5°Cmin^{-1} , being those conditions a result of optimization after various tests.

When samples equal in initial weight and percentages of foaming agent are compared, the use of AlN results in greater expansions and more porous materials than the use of CaCN_2 (figure 9).

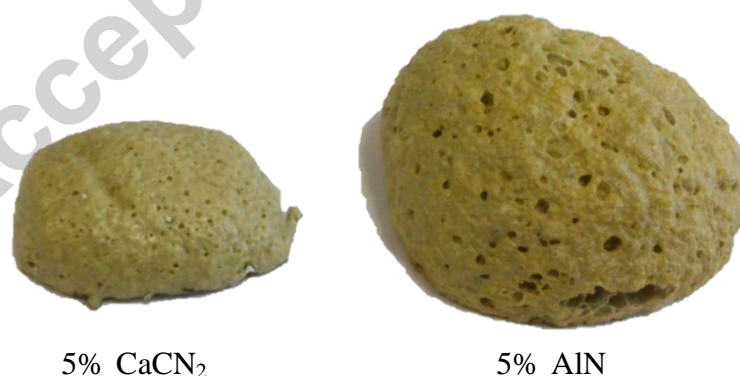


Figure 9. Comparison between 5% CaCN_2 and 5% AlN expanded materials.

With the aim of more easily comparison of the obtained foams, its apparent density is estimated. With this purpose, the foams are cut in parallelepipeds (figure 10) and by

measuring their dimensions and weight the corresponding volume and apparent density could be calculated (table 4).

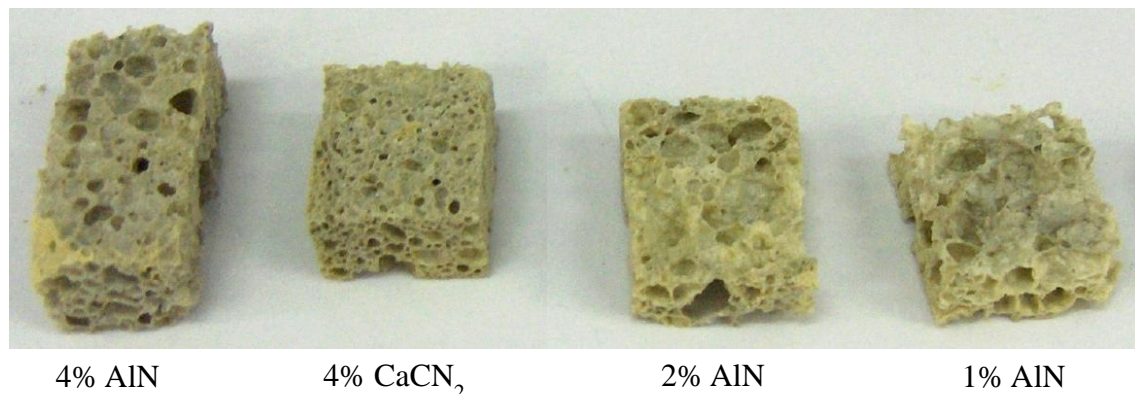


Figure 10. Examples of obtained foams

Table 4. Dimensions, volume, weight and apparent density of the selected foams

Blowing agent (weight %)	Weight (g)	Length (cm)	Width (cm)	Height (cm)	Volume (cm ³)	Apparent density (g.cm ⁻³)	Porosity, ϵ %
4% AlN	1,167	3,42	1,67	1,65	9,49	0,12	94.3
4% CaCN ₂	1,233	2,24	1,87	1,28	5,39	0,23	89.3
2% AlN	0,807	2,67	2,00	0,90	4,82	0,17	92.0
1% AlN	0,805	2,33	2,19	0,98	5,04	0,16	92.8

Taking into account the real (ρ_r) and the apparent density (ρ_a) (Table 5) the porosity (ϵ %) could be estimated according to the following relationship: $\epsilon \% = (1 - \rho_a / \rho_r) * 100$. Porosity at around 90 % is obtained (Table 4). Within the series prepared with the same foaming agent (AlN), similar porosity is detected when lower charges of AlN are used (1 and 2%). However, when the AlN percentage is increased up to 4%, more expansion of the glass is reached, resulting in 95% of porosity.

On the other hand, the same percentage of different expansion agents produces very dissimilar apparent densities. Foam prepared with 4% CaCN₂ presents an apparent density of 0,23 g.cm⁻³, nearly the double of that obtained with 4% of AlN (0,12 g.cm⁻³). Moreover, the average pore size obtained with calcium cyanamide is smaller than the formed with aluminum nitride (figure 10).

In addition to the bulk density, the real density of the materials is determined in helium pycnometer (Table 5). As the starting glass is the same in all cases, the same density is expected for all foams. Excepting the samples 5% AlN and 5% CaCN₂, all the others present very similar values of density with an average of 2.16 g.cm⁻³. Although, the final material composition could vary with the concentration and nature of the foaming agents and could influence the density, the more plausible explication of the values below 2 g.cm⁻³ are the presence of very small pores fraction whose volume was not occupied by helium and, therefore, the total volume of the sample is overestimated and its density underestimated.

Table 5. Real density obtained for some of the prepared foams.

Blowing agent (weight %)	Density (g.cm ⁻³)
5% AlN	1,83
4% AlN	2,12
2% AlN	2,13
1% AlN	2,22
5% CaCN ₂	1,76
4% CaCN ₂	2,15

X-ray diffraction analysis of the products shows the formation of two silicates: (Mg_{0.992}Fe_{0.008})(Ca_{0.999}Fe_{0.029})(Si₂O₆), JCPDS#01-083-008 and Ca_{2.87}Fe_{0.13}(SiO₃)₃, JCPDS#01-083-2198 for all samples (Figures 11 and 12) which implies partial crystallization of the starting glass during the thermal treatment to achieve the expansion of the material. With the aim to check if the crystallization is related to the presence of foaming agent, the same heat treatment of the bare glass sample without expansion agents is carried out and also results in crystallization of some silicates. This indicates that the crystallization is only a thermal effect of the initial glass material.

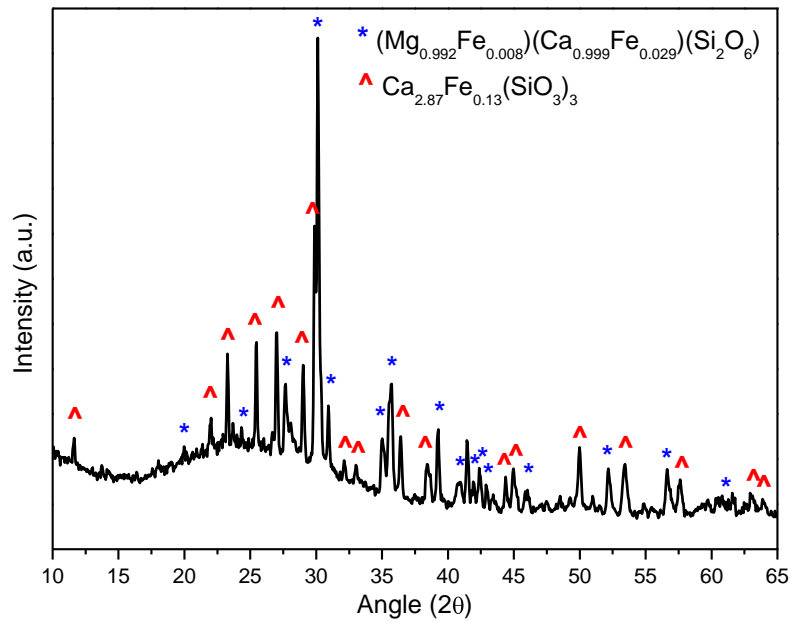


Figure 11. Diffractogram of sample 4% AlN

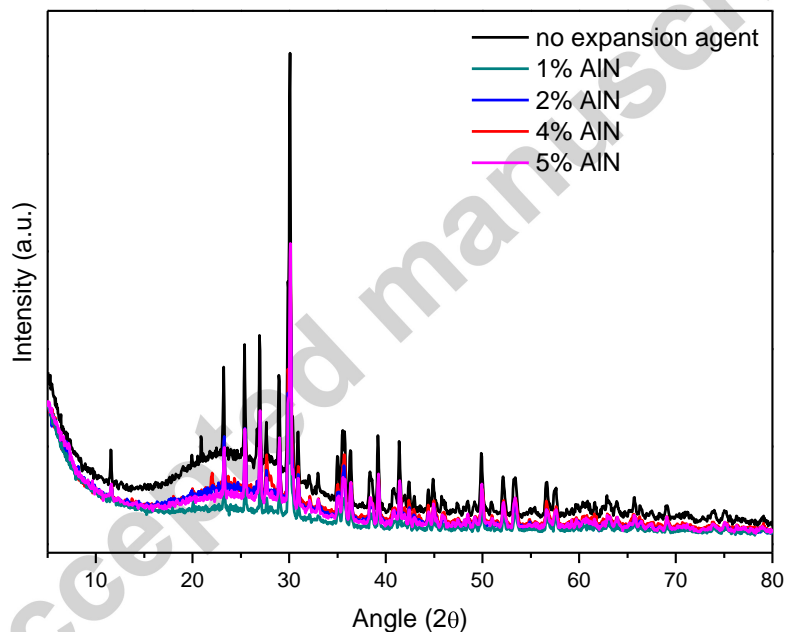


Figure 12. Comparison of the diffractograms of the samples with and without foaming agents

The resistance of the cellular glasses under compression strength is also studied. A sample obtained without foaming agent is compared to samples foamed with 4% AlN and 4% CaCN₂ respectively. As expected, the resistance of the materials is a function of their porosity. The sample prepared without foaming agent presents initial resistance to crack formation of 500 N/m² then quick propagation of the later leading to material breaking at 4750 N/m². Within the series with foaming agent, the smaller the pores size

and distribution the higher the mechanical resistance. The high apparent density measured for the sample prepared with cyanamide, and smaller porosity (89.3%), corresponds to higher mechanical strength, being the later slightly inferior to that of the material prepared without foaming agent (3600 vs. 4750 N/m²). On the other hand the increased porosity (94%) observed for the sample obtained with 4 wt.% AlN shows an instantaneous crack formation and maximum resistance to applied load of 450 N/m².

4. Conclusions

This study shows that the generated CDW could be recycled to generate products able to be applied in the same industry, thus reducing the waste generation and the problems related to the its treatment. A sustainable recycling could be applied in this industry. Simple procedures, such as milling, composition adjusting and vitrification could be applied to convert the waste into primary materials. The glazed tile materials resulting are produced from CDW with the same qualities as the commercial units. The direct fusion process is chosen as the more appropriate and cost saving method for such production. In a similar manner, a method for production of cellular glasses with possible application as thermal and acoustic insulators is proposed. The production path consists in glass formation from CDW and its expansion with the use of foaming agent, being the properties of the final material easily controlled by agent's quantity and composition.

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