LIFECERAM. ZERO WASTE IN CERAMIC TILE MANUFACTURE

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1. ABSTRACT

Ceramic tile manufacture in the EU generates waste in different production process stages. The estimated total amount of waste is of almost 1.5 million tonnes per annum. A significant percentage of this waste cannot be recycled in current ceramic manufacturing processes or products owing to the resulting changes in the behaviour of the ceramic compositions during the manufacturing process and final tile properties. As a result, a significant amount of waste is either landfilled or used as filler materials that provide very low added value. The main purpose of the LIFECERAM project is to achieve zero waste in ceramic tile manufacture. To do so, the project has two principal objectives. The first is to develop a new type of ceramic tile for outdoor use (urban flooring), in which a high ceramic waste content can be incorporated. The second is to design a highly sustainable body preparation process, based on dry milling and granulation technologies, capable of recycling all types of ceramic wastes.

This paper sets out the results of the ceramic waste characterisation, from a physico-chemical viewpoint and with regard to waste behaviour in the ceramic process. The results are then presented of the formulation study, based on the waste characterisation results, of compositions for the urban flooring body. The compositions, made up in their entirety of wastes, were characterised from both a technical and environmental viewpoint. Different dry milling and granulation systems were concurrently evaluated to select the most appropriate systems for the preparation of the urban flooring body press powder.

2. INTRODUCTION

Ceramic tile manufacture in the EU generates waste in different production process stages [1]. Figure 1 shows the arising wastes and a scheme of the process showing where each waste is generated (frit waste and polishing sludge are generated in frit production and polishing plants, respectively).

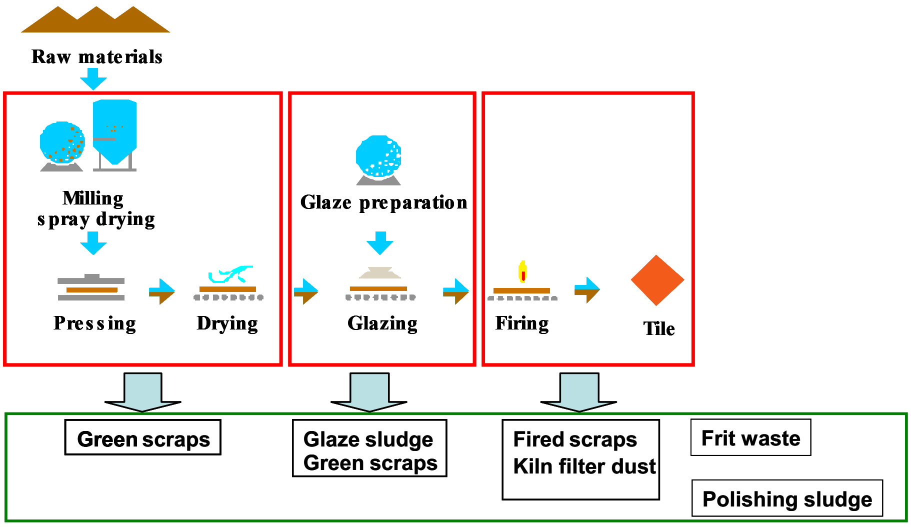

Figure 1. *Scheme of the ceramic tile manufacturing process and points where waste is generated.*

Ceramic tile production in the EU + Turkey (1.8 billion m^2 in 2014 [2]) is estimated to generate about 1.4 million tonnes of the wastes mentioned above (expressed on a dry basis) per annum, accounting for about 5% of raw materials consumption. Figure 2 illustrates the estimated percentage of each type of waste relative to total waste [3].

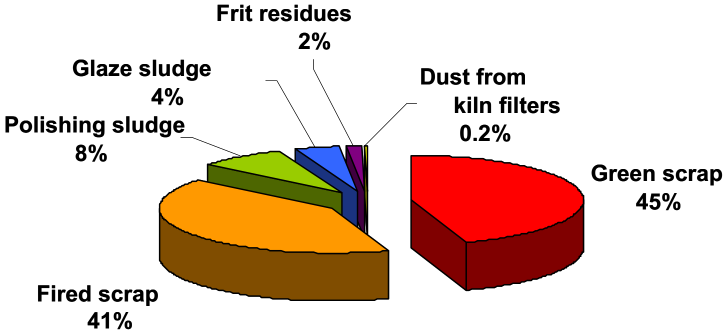

Figure 2. *Waste generated in ceramic tile and glaze manufacturing processes.*

About 65% of these wastes is recycled in the same process as raw material: the wastes are separated by type, homogenised, and introduced in small quantities (<5%) as raw materials in body and glaze compositions. The remaining 35% (500 000 tonnes/annum) is either landfilled or used as product filler materials with low added value.

As may be observed in Figure 3, the recycling of green tile scrap, frit waste, and glaze sludge is widespread practice in the EU. On the other hand, fired tile scrap from red-firing tiles is not usually recycled owing to the high cost involved in milling such scrap and the low cost of red-firing clays [4]. In white-firing tiles, mainly involving porcelain tiles, the main disadvantages are the high hardness of the product and the existence of pigments in the tile scrap, which can lead to incidental colour changes in the body, which may be visible at the glazed surface. Even so, some companies recycle up to 10% of these wastes (in some of the compositions), owing to the high cost of white-firing raw materials. Higher recycling percentages are not commonly found because of the considerable increase in milling time. The remaining wastes, polishing sludge and kiln filter waste, are hardly recycled in the tile manufacturing process because they contain soluble salts that cause the suspension to flocculate before it is spray dried. In addition, polishing sludge contains organic compounds that are difficult to remove during tile firing, and abrasive particles (SiC) that give rise to defects in the tile surface [5].

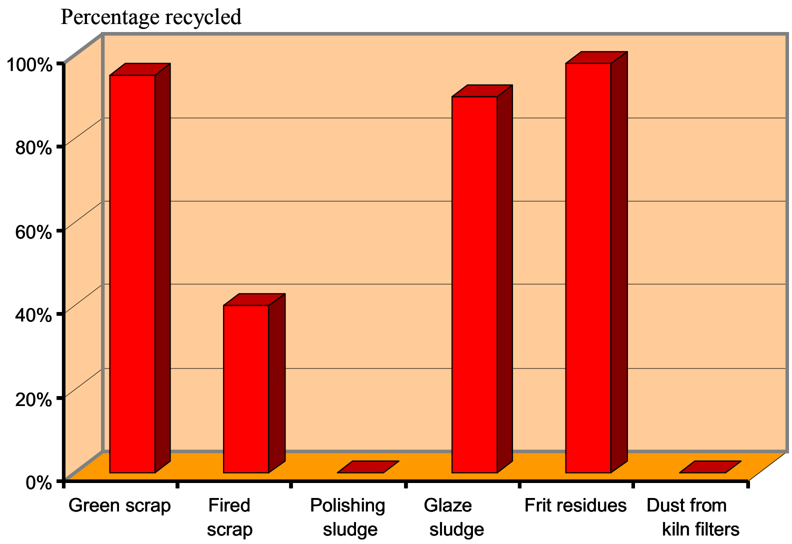

Figure 3. *Recycling percentage of each type of waste.*

The great quantity of ceramic waste being landfilled lay at the origin of the LIFECERAM project, which seeks to attain zero waste in the ceramic tile manufacturing process [6, 7] by developing a ceramic tile for outdoor use (urban flooring) with a very high waste content (>80%). This application (still in its early stages), in which textured matt finishes are required, will provide the ceramic sector with the opportunity to enter this new market with a tile consisting mainly of waste.

At present, porcelain tiles are being used as urban flooring [8]. These have a high manufacturing cost owing to the raw materials used (largely imported) and of the high embedded energy in the production process (vigorous wet milling, spray drying, and firing at high temperature, about 1200ºC), constraining the introduction of ceramics into the urban flooring market. However, the warm reception by citizens of the use of ceramics in some European and non-European cities [9] signals the great possibilities for the use of ceramic tiles in this field.

3. OBJETIVES

The present paper sets out some key results of this project to date, in particular those aimed at designing the composition of the urban flooring tile body, based on prior characterisation of the different wastes, and the preparation process of the body composition. With a view to reducing manufacturing costs, this process will be based on dry milling and granulation technologies and must be sufficiently robust to enable all ceramic wastes to be recycled, including fired tile scrap and wastes containing soluble salts (polishing sludge and kiln filter waste).

4. EXPERIMENTAL

4.1. MATERIALS

Table 1 details the chemical composition of the wastes used in this study:

- Green tile scrap. This displays the typical elements of the body compositions and, to a lesser extent, the elements commonly found in glazes and engobes [4].
- Glaze sludge. The major elements are those typically found in frits, glazes, and engobes [10].
- Fired tile scrap. This exhibits a chemical composition similar to that of green tile scrap except the loss on ignition [4].
- Dust from kiln cleaning filters. This contains calcium as a result of the use of basic additives ($CaCO_3$, $Ca(OH)_2$, etc.) in kiln bag filters, in addition to fluorine, sulphur, and chlorine initially present in the acid gases produced during tile firing [11].
- Polishing sludge. Its chemical composition stems from the presence of fired material as well as from ingredients from cutting, edge-grinding, and polishing tool wear [5]. These tools consist of a polymer or cement matrix (typically containing chlorine and magnesium), which hold the (silicon carbide and diamond) abrasive particles [12].
- Frit waste. This is generated in the frit manufacturing process, during the changeover in production from one frit to another. The major elements are those typically found in frits [10].

Type of waste	Green scrap	Fired scrap	Glaze sludge	Kiln filter dust	Polishing sludge	Frit waste
SiO_2	63.3	67.2	58.0	1.00	60.9	60.6
Al_2O_3	16.9	17.9	12.3	0.31	15.2	4.7
B_2O_3	0.1	0.1	2.6	0.22	0.93	8.0
Fe_2O_3	3.8	4.1	0.40	<0.15	0.84	0.1
CaO	3.0	3.3	9.7	62	3.81	12.0
MgO	1.2	1.2	1.66	0.44	3.57	2.9
Na_2O	1.8	1.9	1.98	0.25	3.33	2.0
K_2O	2.8	3.0	2.69	0.46	2.48	3.4
TiO_2	0.7	0.7	0.16	0.01	0.46	0.1
ZrO_2	0.2	0.2	2.47	0.01	0.46	2.9
ZnO	0.2	0.3	4.09	0.12	2.51	6.6
BaO	0.1	0.1	1.09	0.02	0.65	2.4
S	-	-	0.02	2.53	0.03	-
Cl	-	-	0.05	1.41	0.79	-
F	-	-	-	32.0	-	-
L.O.I.	6.0	0.2	2.72	7.63	3.90	0.2

Table 1. *Chemical composition of the wastes [wt%].*

4.2. TESTS CONDUCTED

The following tasks were performed:

- Characterisation of the ceramic behaviour of the waste. This consisted of determining waste behaviour (milled in a hammer mill with output sieve of 1 mm) in the ceramic tile manufacturing process stages (pressing and firing) and its final properties (shrinkage, porosity, colour, etc.). The procedures used to conduct these tests are described elsewhere [13].
- Selection of the most appropriate mill and granulator variables for body composition preparation. Different dry milling and granulation systems were used for this purpose, determining the characteristics of the wastes processed with these systems.
- Design of the urban flooring body composition. Different mixtures of wastes, prepared with the optimum process variables, were characterised until the most appropriate composition was obtained, from a technical and environmental viewpoint, which would allow zero waste in the ceramic tile manufacturing process to be attained. The environmental characterisation consisted of determining the acid gas emissions [14] and performing leaching tests (according to standard UNE-EN 12457-2:2003).

5. RESULTS

5.1. CHARACTERISATION OF THE WASTES

The following table details the green properties and fusibility of the different wastes characterised in order to establish their feasibility for use in the urban flooring body. It clearly shows that, except the green tile scrap, the other wastes exhibited inappropriate behaviour in the forming stage, giving rise to low bulk density and low dry mechanical strength values. Consequently, a large quantity of green tile scrap needs to be included in the formulated compositions to ensure suitable processability in the green state.

Waste		Dry bulk density [g/cm^3][1]	Dry mechanical strength [kg/cm^2] [1]	Water absorption (W.A.) at 1150ºC [%][1]
Green scrap	Earthenware wall tile	1,95	24	14
	Glazed stoneware tile	2,00	26	2,0
	Porcelain tile	1,89	22	4,0
Fired scrap	Earthenware wall tile	1,59	1,0	14
	Glazed stoneware tile	1,67	1,0	7
	Porcelain tile	1,71	1,0	10
Glaze sludge and frit waste		1.70	5	<0,1
Kiln filter waste		1.36	1,0	<0,1
Polishing waste		1.40	7	<0,1

(1) Test pieces formed by pressing (moisture content: 5.5%; pressing pressure: 250 kg/cm^2).

Table 2. *Waste characterisation results.*

In regard to firing behaviour, the most refractory material was the fired tile scrap, while the polishing sludge, kiln filter waste, and glaze sludge provided high fusibility. However, in the case of polishing sludge, a bloating phenomenon was observed owing to the presence of silicon carbide particles that decomposed in firing releasing CO_2 and CO [5]. Figure 4 shows some fired test pieces, which displayed a different colour and texture depending on the waste involved. Owing to the high particle size of the fired tile scrap and to the presence of glaze, the test pieces made from these wastes exhibited non-uniform colours, which could be useful in obtaining the typical finishes of porcelain tiles used in urban flooring [8, 9]. The other wastes, owing to their smaller particle size, displayed homogeneous colours.

Figure 4. *Appearance of the fired test pieces.*

5.2. SELECTION OF DRY MILLING AND GRANULATION TECHNOLOGIES

For the study of the dry milling and granulation technologies, green tile scrap and fired tile scrap were used, as wastes that typically exhibit low and high hardness, respectively. Laboratory scale evaluation was performed of dry milling technologies of the fired tile scrap using a hammer mill (Figure 5) and a disc mill (Figure 6) because of scrap hardness, without excessively small particle sizes being required. The green tile scrap was tested in the hammer mill and in a pendulum mill (Figure 7), as smaller particle sizes were required to favour green tile scrap reactivity and mixing with the fired tile scrap.

Figure 5. *Hammer mill.*

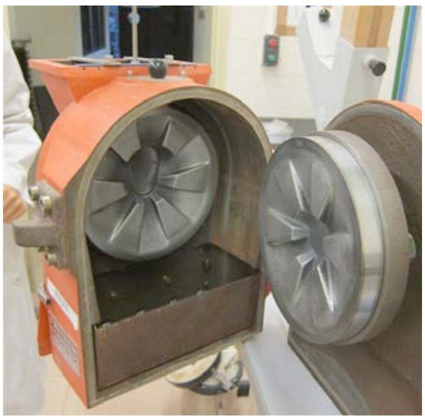

Figure 6. *Disc mill.*

Figure 7. *Pendulum mill.*

Table 3 details the particle size distributions of the two types of tile scrap processed in the different mills under different conditions.

Mill	Hammer	Hammer	Pendulum	Pendulum	Hammer	Hammer	Disc	Disc
Mill conditions	200 µm	500 µm	more vigorous	less vigorous	500 µm	1000 µm	500 µm	1000 µm
Product	Green scrap				Fired scrap			
Reject at 100 µm (%)	40.1	63.1	3.7	7.7	35.7	53.0	64.9	79.0
Reject at 150 µm (%)	35.7		2.5	3.4				
Reject at 180 µm (%)	8.0		1.1	1.3				
Reject at 200 µm (%)		13.1			19.4		51.2	
Reject at 400 µm (%)		0.3			2.4		29.7	
Reject at 500 µm (%)						12.6		54.0
Reject at 710 µm (%)						3.3		43.7

Table 3. *Particle sizes of the wastes processed in the different mills.*

The following conclusions may be drawn:

Milling of soft materials

- The milling of the green tile scrap, and by analogy, of other soft materials, in a pendulum mill enabled small particle sizes, with a particle size cut-off of about 100 µm, to be obtained. When a hammer mill was used, practically 50% of the material was obtained at 100 µm, even when using output sieves with a small aperture.
- To mill soft materials, it is recommended to use a hammer mill for moderate particle sizes (particle size cut-off at 200–300 µm) and to use a pendulum mill when the particle size cut-off is to be 100 µm or lower.

Milling of hard materials

- The use of a hammer mill led to smaller particle sizes, with a more spherical shape. The disc mill produced particles with a more laminar shape.

In view of these results, the hammer mill with an output sieve of 300 µm was chosen for the soft materials. The hammer mill was also chosen for the hard materials, though a higher output sieve, namely 1 mm, was used. Milling of hard materials was thus facilitated, while the possible metallic contamination by the mill decreased. If smaller green tile scrap particle sizes are required, a pendulum mill should be used.

With regard to granule preparation, the efficiency of two types of mixing and granulation systems was studied: a high-speed and a low-speed system (Figures 8 and 9). The composition consisted of 50% green tile scrap and 50% fired tile scrap.

Figure 8. *Low-speed granulator (mixer useful rotation speed 380 rpm).*

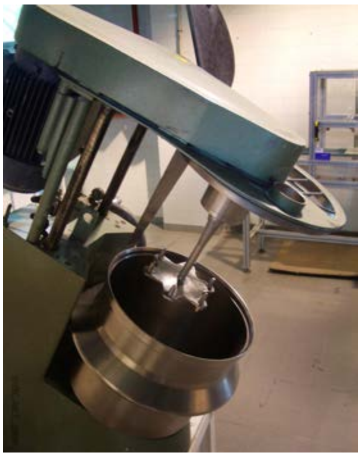

Figure 9. *High-speed granulator (mixer useful rotation speed 4800 rpm).*

During the granulation process it was verified that the water needed for granulation in both types granulator was very similar, as was the moisture content of the resulting granulates (12%).

Figure 10 shows the granule size distributions, while Figures 11 and 12 show their appearance.

Table 4 details the main results obtained in the characterisation of granule behaviour in the process.

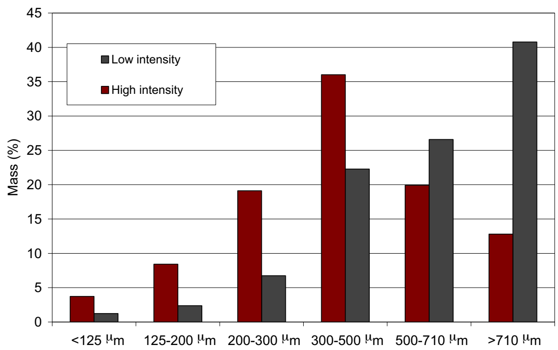

Figure 10. *Granule size distributions.*

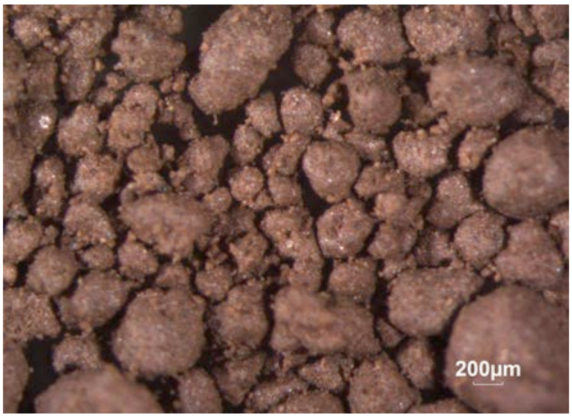

Figure 11. *Granulate obtained in the low-speed system.*

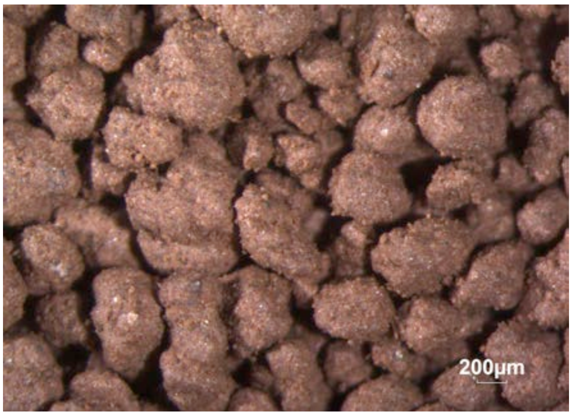

Figure 12. *Granulate obtained in the high-speed system.*

Granulator	High speed	Low speed
Hausner ratio	1.25	1.22
Filling bulk density (g/cm^3)	1.111	1.180
Dry bulk density (g/cm^3)	2.035	2.055
Dry mechanical strength (kg/cm^2)	18	21
Temperature at W.A. =3% (ºC)	1159	1148
Bulk density (W.A. =3%) (g/cm^3)	2.345	2.385

Table 4. *Granulate characterisation results.*

The results indicate the following:

- The high-speed granulator yielded narrower particle sizes, more centred in the 300–500 μm fraction, which is the fraction that it is sought to maximise by analogy with the usual spray-dried powder particle sizes used in pressing ceramic tiles.
- The flowability values (determined as the Hausner ratio) were appropriate in both cases.
- With regard to ceramic behaviour, the behaviour obtained with the low-speed system provided pieces of greater dry bulk density and dry mechanical strength, indicating lower granule hardness, making the granules more deformable and increasing inter-particle bonds. This also explains the lower temperature required to reach a water absorption (W.A.) of 3%, selected at the outset of the project as the target value based on a comparative study of products customarily used in urban flooring [15, 16].
- It may be concluded, nevertheless, that the two granulates provided good dry bulk density and dry mechanical strength, and appropriate fusibility.

In view of the above, the high-speed granulation system was selected for the development of the body composition, mainly because of the granule size distribution that it provided. This distribution resembled more closely that of the spray-dried powders used in the sector, while the proportion of large granules. liable to generate defects in the tiles, was lower.

5.3. DESIGN OF THE URBAN FLOORING BODY COMPOSITION

To formulate the urban flooring body composition a mixture referenced PU-1, entirely consisting of wastes (Table 5), was used. The percentage of each waste was defined such that it was similar to the volumes generated in industrial practice of these wastes, with the added requirement that the green tile scrap content was above 40% to assure appropriate pressing behaviour. The green and the fired tile scrap was a mixture consisting of equal parts of the different types of ceramic bodies (earthenware wall tile, stoneware tile, and porcelain tile).

The procedure used to prepare the compositions was the method selected above (section 4.2):

- Milling of the softest wastes in a hammer mill to a particle size <300 μm
- Milling of the fired tile scrap in a hammer mill to a particle size <1.0 mm
- Granulation in the high-speed granulator.

Waste	PU-1	PU-2	PU-3
Green scrap	44.75	44.75	44.88
Fired scrap	44.75	44.75	44.88
Glaze sludge	5.00	7.50	7.50
Polishing sludge	5.00	2.50	2.50
Kiln filter waste	0.50	0.50	0.25

Table 5. *Tested composition formulations.*

The vitrification diagrams of the formulated compositions are shown in Figure 13. The horizontal black line indicates 3% water absorption. The vertical lines show the temperatures at which this water absorption was reached. The first formulated composition (PU-1) displayed a high tendency to bloat at temperatures near firing temperature. A new composition was therefore formulated, reducing the polishing sludge content (PU-2 composition, Table 5), as this waste exhibited the greatest tendency to bloat (section 4.1). There was further a third composition in which the kiln filter dust content was halved (PU-3) in order to determine its effect on the acid compound emissions, the results of which are shown in Figures 14 and 15. These results indicate that in order to assure no high environmental impact of the urban flooring (with regard to emissions), the PU-3 composition was more appropriate than the PU-2 composition.

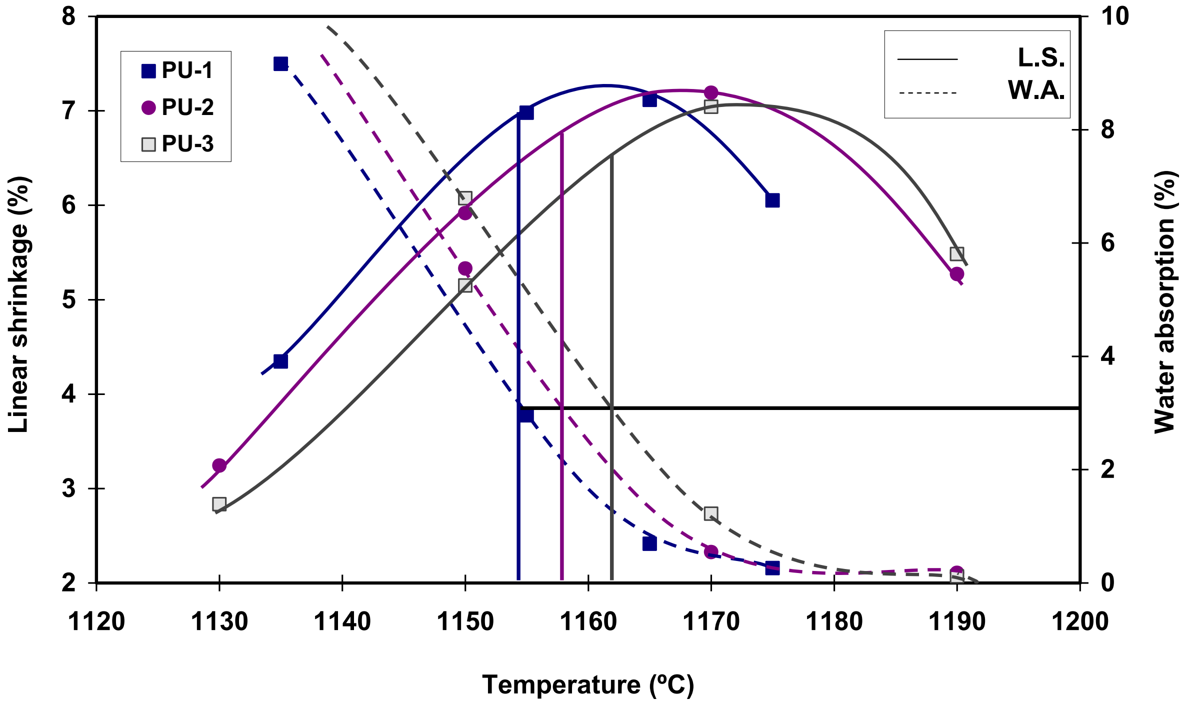

Figure 13. *Vitrification diagrams of the compositions formulated for the urban flooring body.*

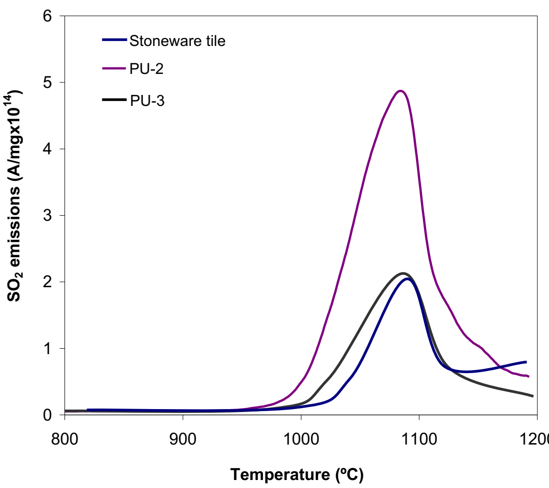

Figure 14. *SO_2 emissions.*

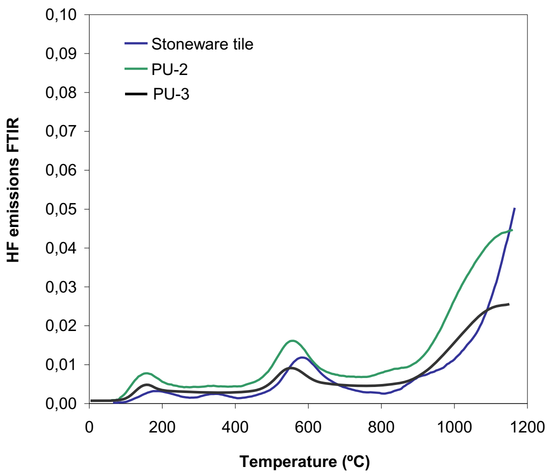

Figure 15. *HF emissions.*

Composition PU-3 was fully characterised and its behaviour compared with that of two standard industrial body compositions (stoneware tile and porcelain tile). Table 6 details the pressing conditions and the dry properties. The comparative analysis with the industrial body compositions indicates that the pressing behaviour of the formulated composition was appropriate, though dry mechanical strength was lower. As urban flooring would be thicker (15 mm) than the tiles used indoors (10 mm), this mechanical strength would suffice to obtain a satisfactory breaking strength, as shown in the table.

Composition	PU-3	Stoneware tile body	Porcelain tile body
Pressing moisture content (%)	5.5	5.5	5.5
Pressing pressure (kg/cm^2)	250	250	350
Dry bulk density (g/cm^3)	2.005	2.021	1.953
Dry mechanical strength (kg/cm^2)	20	31	35
Breaking strength (N)[1]	294	203	229

(1) Calculated for bodies of 40x40 cm (thickness of 10 mm in the case of the stoneware tile and the porcelain tile and thickness of 15 mm for the urban flooring body, composition PU-3).

Table 6. *Forming conditions of the PU-3 body composition and of standard stoneware tile and porcelain tile compositions.*

The vitrification diagram of the developed composition is plotted again in Figure 16, together with those of the two industrial bodies. Table 7 lists the firing temperatures of each material and the properties of the pieces at these temperatures. Figure 17 shows the microstructure of fired pieces made from the developed composition and from a stoneware tile composition prepared by the usual wet process and by a dry process similar to the one used in this study. Finally, Table 8 details the results of the leaching tests, together with the limits established for classifying a waste as inert according to Directive 1999/31/EC. The results obtained allow the following conclusions regarding the urban flooring body composition to be drawn:

- Firing behaviour was similar to that of the industrial body compositions, though the firing range was smaller (greater slope of the shrinkage–temperature curve). The tendency to deform by pyroplasticity was similar to that of stoneware tile bodies.
- As the tiles are intended for outdoor use, the dimensional stability requirements are lower than when tiles are intended for indoor use, so that the firing behaviour of this composition was deemed appropriate.
- Despite the lower water absorption and greater density of composition PU-3 compared to that of the stoneware tile, its mechanical strength was lower. This was because of cracks in the microstructure of the fired pieces (Figure 17c), resulting from the lower deformability of the dry granules, similarly to what was also observed in the stoneware tile composition processed by the dry method (Figure 17b) compared to that processed by the wet method (Figure 17a). To increase this mechanical strength, granule deformability would need to be increased either by acting on the granulation process or on the pressing variables (moisture content and pressing pressure).
- The urban tile can be classified as non-hazardous based on the results of the environmental characterisation tests (leaching tests and determination of gas emissions).

Composition	PU-3	Stoneware tile body	Porcelain tile body
Temperature (°C)	1163	1133	1185
Water absorption (%)	3.0	4.0	0.5
Bulk density (g/cm^3)	2.355	2.322	2.382
Linear shrinkage (%)	6.8	6.5	7.6
Pyroplasticity index (cm^{-1}x10^5)	2.3	2.5	3.5
Mechanical strength (kg/cm^2)	380	430	580

Table 7. *Optimum firing temperature and properties at this temperature.*

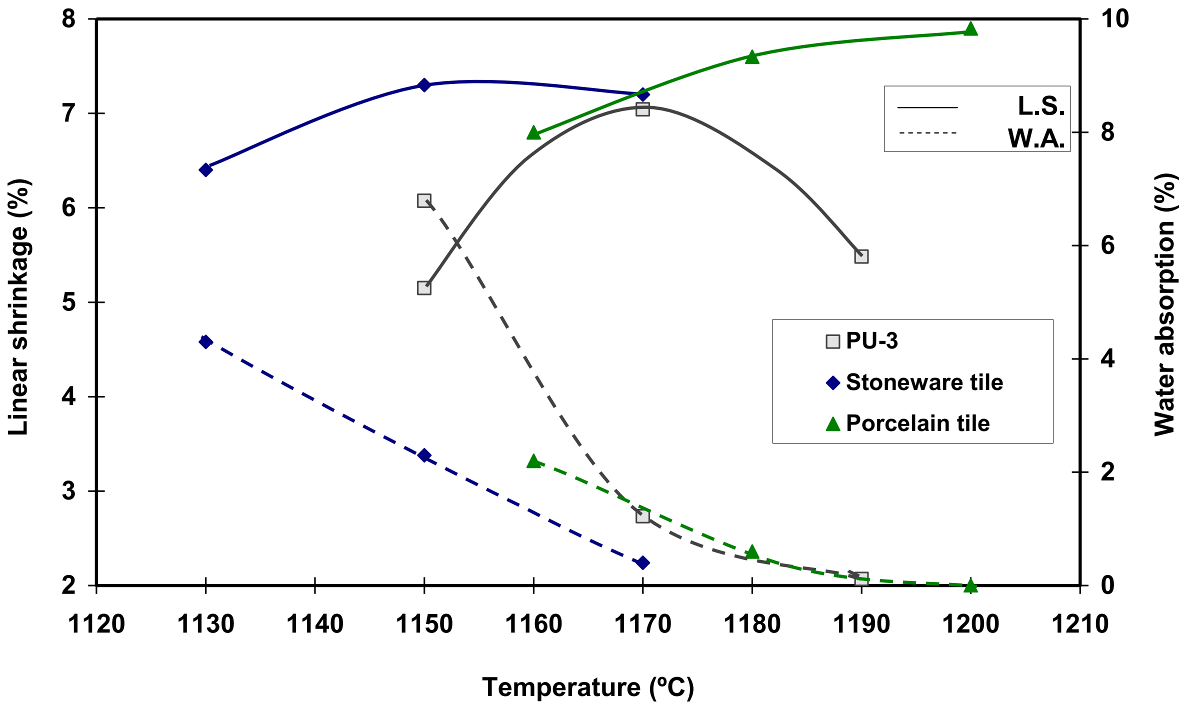

Figure 16. *Vitrification diagram of the optimum body composition and of commercial products.*

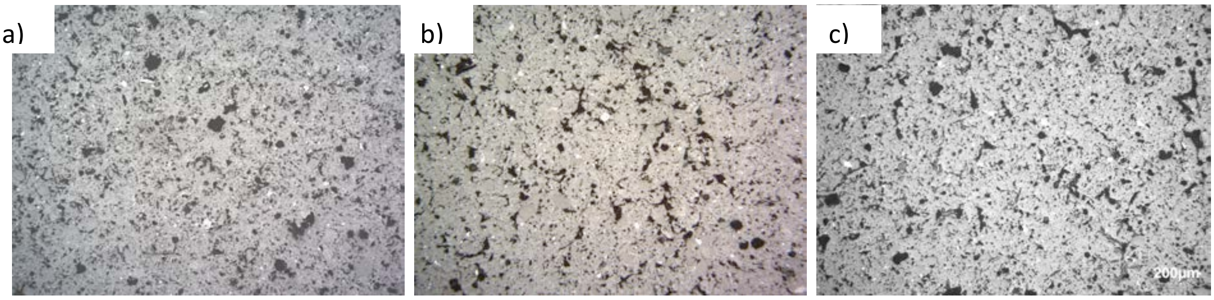

***Figure 17.** Microstructure of the fired samples: a) stoneware tile composition prepared by wet milling and spray drying; b) the same body composition prepared by dry milling in a pendulum mill and granulated; and c) urban flooring body composition PU-3.*

Element ($mg \cdot kg^{-1}$)	Inert wastes	PU-3
Pb	0.5	<0.5
Chlorine	800	29
Sulphur	1000	28

***Table 8.** Allowable limits for inert wastes according to Directive 1999/31/EC and leaching test results.*

6. CONCLUSIONS

In the LIFE CERAM project, the different wastes generated in the ceramic tile manufacturing process were characterised, and an urban floor tile was developed whose body is entirely made up of these wastes (green and fired tile scrap, glaze and polishing sludge, and kiln filter dust, in relative proportions similar to the waste volumes generated in industrial practice). For the fabrication of this tile, a highly sustainable body preparation process was designed that allows all ceramic wastes to be recycled. The characterisation of the composition indicates that it displays appropriate behaviour in the different production process stages and exhibits the required properties for use as urban flooring. In addition, the product was verified to be respectful with the environment, despite being made up of wastes, as the acid compound emissions were similar to those of current body compositions and the ion concentrations in the leaching tests were lower than those required to classify a waste as inert.

Pilot scale trials have been conducted to validate the results obtained in the laboratory and their results are been satisfactory.

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