

BIO-CALCIUM CARBONATE FROM EGGSHELLS: A NEW RAW MATERIAL FOR THE CERAMIC INDUSTRY

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1. ABSTRACT

It is estimated that around 150,000 tonnes of eggshells are generated in Europe and destined for landfill each year. They often cause problems, such as foul odour or growth of bio-organisms, which lead to complaints and reports to the authorities. In the case of Spain, the costs of transporting eggshell waste to disposal sites vary between €25 and €200 per tonne, which for medium-sized egg production companies entails a cost disbursement of between €50,000 and €100,000 per year.

In alignment with the principles of the Circular Economy, the aim of the LIFE EGGSHELLENCE project is to demonstrate the technical feasibility of using bio-calcium carbonate from eggshells as a secondary raw material in earthenware tile manufacture. To that end, earthenware tile compositions were formulated and characterised, in which bio-calcium carbonate was included in partial replacement of mineral calcium carbonate. These compositions were initially prepared at laboratory scale, then at pilot scale, and finally industrial tests were carried out. The results obtained allow the conclusion that it is technically feasible to introduce up to 5% of bio-calcium carbonate as partial replacement of mineral calcium carbonate in this type of compositions.

Furthermore, the feasibility of using bio-calcium carbonate in the formulation of ceramic frits as a substitute for mineral calcium carbonate is being studied within the framework of the OVOVAL project. The results obtained so far indicate that the compositions formulated with bio-carbonate can be suitably processed and used to produce glazes with similar properties to those obtained when mineral calcium carbonate is employed to contribute CaO to the composition.

2. INTRODUCTION

In December 2015, the Action Plan for a circular economy in Europe was published with a series of measures which the European Commission considered it necessary to enact in the 5 years thereafter in order to advance transition to a circular economy in the EU. Despite the EU's proclamation, only 39.2% of waste was recycled in 2020 and 32.2% was landfilled.

Continuing the efforts initiated in 2015, the European Commission has recently adopted a new Action Plan on the Circular Economy, which is one of the main items in the European Green Deal, Europe's new agenda for sustainable growth [1].

In Spain, the Spanish Circular Economy Strategy (known by its Spanish acronym EEEEC) [2] is aligned with the objectives of the two previously mentioned EU circular economy action plans and the 2030 Agenda for Sustainable Development [3]. The EEEEC identifies six priority business sectors as targets for the challenge to achieve a circular Spain: construction, agri-food, fisheries and forestry, industry, consumer goods, tourism, and textiles and clothing.

Within that context, this study aims to explore strategies for valorising a waste product from the food industry, namely eggshells from both egg-producing companies and poultry farms, in the manufacture of ceramic tiles, thus amalgamating two priority sectors: food and construction.

It is estimated that around 150,000 tonnes of eggshells are generated in Europe and destined for landfill each year [4][5]. They are often the cause of problems such as foul odour or growth of bio-organisms, leading to complaints and reports to the authorities. In Spain, the corresponding amount is approximately 16,000 tonnes and the costs of transporting and landfilling that waste vary between €25 and €200 per tonne.

Eggshells are mostly composed of calcium carbonate (95% of shell weight), magnesium carbonate (1%), calcium phosphate (1%) and water [5]. Therefore, once the membrane is separated, they can become an alternative source of calcium carbonate.

In line with the principles of the Circular Economy, the objective of the LIFE EGGSHELLENCE project [4] is to demonstrate the technical feasibility of using bio-calcium carbonate obtained from eggshells as a secondary raw material in earthenware tile manufacture. The feasibility of the solution stems from the fact that companies manufacturing spray-dried powders and ceramic tiles consume significant amounts of calcium carbonate because white-firing earthenware tile bodies contain between 10% and 15% by weight of calcium carbonate, as well as clays and sands [6].

Consumption of calcium carbonate is also very high in the manufacture of ceramic frits. Given its properties as a flux and, in high proportion, as a matting agent, CaO is a common oxide in ceramic frit compositions, present in variable proportions (usually between 5% and 25%), depending on the properties required for the glaze and the type of tile for which it is intended.

In this context, the OVOVAL project [7] addresses the feasibility of using bio-calcium carbonate in the formulation of ceramic frits as a secondary raw material to contribute CaO to frit compositions, in replacement of mineral calcium carbonate.

3. EXPERIMENTAL DEVELOPMENT

Firstly, the bio-calcium carbonate obtained from eggshells was characterised using the separation process designed in the LIFE EGGSHELLENCE project [8]. The chemical composition was determined by wavelength dispersive X-ray fluorescence spectrometry using reference materials that guarantee measurement traceability, and the crystalline phases present were determined by X-ray diffraction (XRD). Total carbon was also determined using a LECO CS-200 carbon analyser, while nitrogen content was measured with a LECO TN-436 elemental analyser. Finally, the microstructure of the bio-carbonate was observed both at the surface and in cross-section using the backscattered electron signal from a field emission scanning electron microscope (FE-SEM).

The bio-carbonate was then used as a substitute for mineral calcium carbonate in the manufacture of earthenware tiles and ceramic frits.

With regard to the earthenware tiles, after studies at laboratory and pilot scale, two industrial tests were carried out, in which 20 tonnes of spray-dried powder were obtained, each one having 5% bio-carbonate in partial substitution of mineral calcium carbonate. In one of the trials, the bio-carbonate was included with the resulting particle size (less than 2 mm) after the membrane separation process, while in the other trial, it was micronised (by a pendulum mill to a size of less than 63 μm). Both the spray-dried powders with bio-carbonate and the powder obtained without adding bio-carbonate were characterised by determining their behaviour during pressing (compaction diagram and dry mechanical strength) and firing (variation of linear shrinkage, bulk density and water absorption with firing temperature), as well as their after-firing properties (mechanical bending strength and moisture expansion after being treated in the autoclave for 5 hours with a steam pressure of 10.2 kg/cm²). Moreover, the spray-dried powders were used to produce 2000 m² of tiles, which were characterised, on the one hand, by determining the crystalline phases present and observing their microstructure with a field emission scanning electron microscope (FE-SEM). On the other hand, their modulus of rupture and breaking strength (UNE-EN ISO 10545-4:2019), water absorption (UNE-EN ISO 10545-3:2018), crazing resistance (UNE-EN ISO 10545-11:1997) and moisture expansion (UNE-EN ISO 10545-10: 2022) were determined. In addition, the composition of the gas stream evacuated through the stack of the kiln where the tiles were fired was analysed using FTIR (Fourier-Transform Infra-Red).

The feasibility study on the use of bio-carbonate in the development of ceramic frits was carried out at laboratory scale. To that end, a frit composition with 18% CaO by weight was selected, which would produce matt and opaque glazes. The work undertaken consisted of producing a frit in which CaO was included using micronised bio-carbonate and the proportion of the other raw materials used was adjusted as necessary to match the composition of a reference frit formulated with natural calcium carbonate.

The frits were obtained by melting the mixture of raw materials in an electric furnace up to a maximum temperature of 1500°C and then pouring the melt on cold water. Both frits were characterised by determining their thermal behaviour using a MISURA hot stage microscope and subjecting them to a heating cycle at a rate of 25°C/min. Subsequently, the frits were used in the development of glazes to manufacture porcelain stoneware tiles. With the aim of better understanding the influence of the raw material used to provide the CaO on the processability of the compositions and on the characteristics of the resulting vitreous coatings, glazes were prepared in which 92% of the composition corresponded to the frit under study. Both glazes were applied on an unfired porcelain stoneware tile body, engobed using an applicator with an aperture of approximately 400 µm, before firing the test pieces at different maximum temperatures (1180°C, 1200°C and 1220°C). The colour coordinates of the fired glazed pieces were determined by means of a spectrophotometer using a D65 illuminant and a CIE 10° observer. In addition, gloss was measured with a reflectometer at an angle of 85°. Finally, the dilatometric behaviour of the glazes was assessed using a Netzsch 402-Expedis absolute dilatometer and subjecting them to a thermal cycle with a heating rate of 5°C/min.

4. BIO-CARBONATE CHARACTERISATION

The chemical composition and the C and N contents of the bio-calcium carbonate obtained from the separation process developed in the LIFE EGGSELLENCE project [8] are shown below (Table 1). From that analysis, the percentage was calculated of the major phase, CaCO₃, this being 94%, and those of the two minor phases, MgCO₃ and Ca₃(PO₄)₂, these being 1.3 and 0.7%, respectively, which is consistent with the literature [5]. From these percentages it can be established that the remaining organic fraction is 4%. The same percentage can be obtained if the loss on ignition associated with the calcium and magnesium carbonates is deducted from the total bio-carbonate loss. Finally, from the total C content of the bio-carbonate, when the C associated with the calcium and magnesium carbonates is discounted, an organic C content of 1.1% is obtained. It is this organic C that can cause black core problems in earthenware tiles made with bio-carbonate. It should be noted, however, that, once the bio-carbonate has been included in the ceramic composition at a percentage of 5%, it will only increase the organic C content of the composition by 0.05%. This increase is considered acceptable bearing in mind that earthenware tile compositions have organic carbon contents of between 0.10% and 0.20%. It is important to note that the chemical composition, as well as N and C contents, do not vary from one batch to another, which demonstrates the immutability of both the composition of the eggshells and of the newly developed separation system. Indeed, the initial waste shell, prior to the membrane separation, contains 1.2% N and 14.8% C, percentage levels that also remain constant from one batch to the next. All of the above is of vital importance to implementing an industrial symbiosis process between egg production and ceramic tile companies as a guarantee of the invariability of the characteristics of the by-product to be valorised.

Chemical composition	Bio-carbonate
CaO (%)	53.0
MgO (%)	0.61
Fe ₂ O ₃ (%)	0.01
Na ₂ O (%)	0.13
K ₂ O (%)	0.06
P ₂ O ₅ (%)	0.31
Loss on ignition at 1000°C (%)	45.58
C (%)	12.6
N (%)	0.42

Table 1. Chemical composition of the bio-carbonate.

As a complement to the chemical determinations, Figure 1 shows the results of the identification of the crystalline structures, in which only the major phase, calcite (calcium carbonate), was identified. Magnesium carbonate and calcium phosphate were not identified because their content percentage was below the detection limit of the X-ray diffraction technique (2–3% depending on the phase).

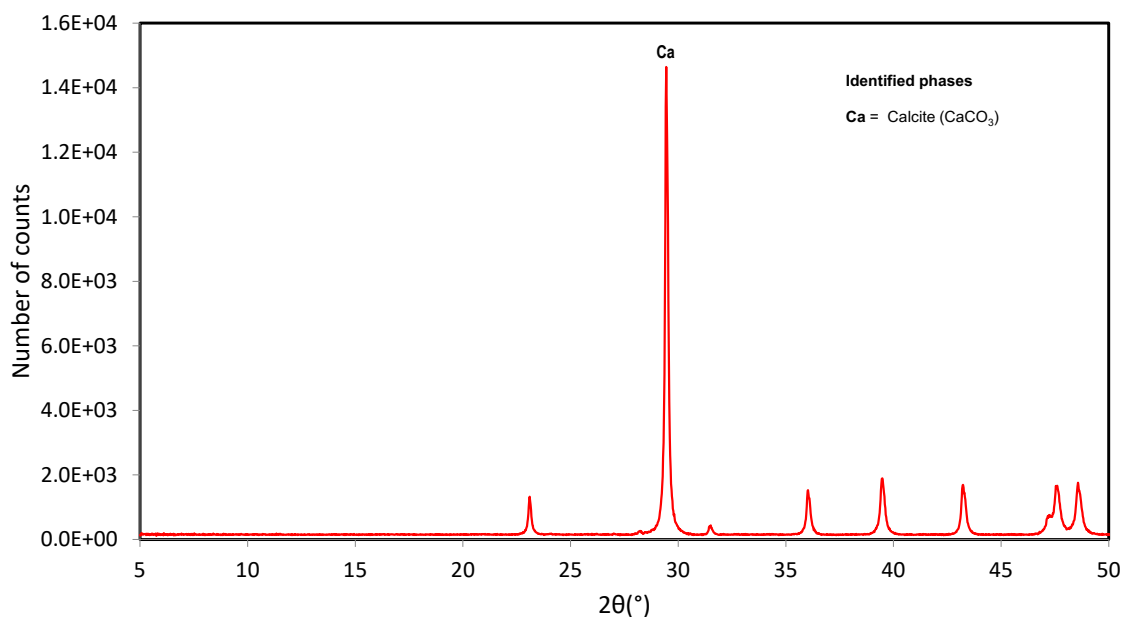


Figure 1. Diffractogram (area enlarged from 2θ= 5° to 2θ= 50°) of bio-calcium carbonate

As far as the microstructure of the bio-carbonate is concerned, the images in Figure 2 show the appearance of the particles obtained in the separation process as well as of particles prior to the separation process. Images a) and b) in Figure 1 clearly show the membrane adhered to the bio-carbonate columns in particles from the original waste, and images c) and d) show how the membrane is almost completely removed in the separation process.

These images suggest that the residual organic fraction is not in the form of a membrane but must be part of the bio-carbonate columns. This bio-carbonate has calcite as its major crystalline phase.

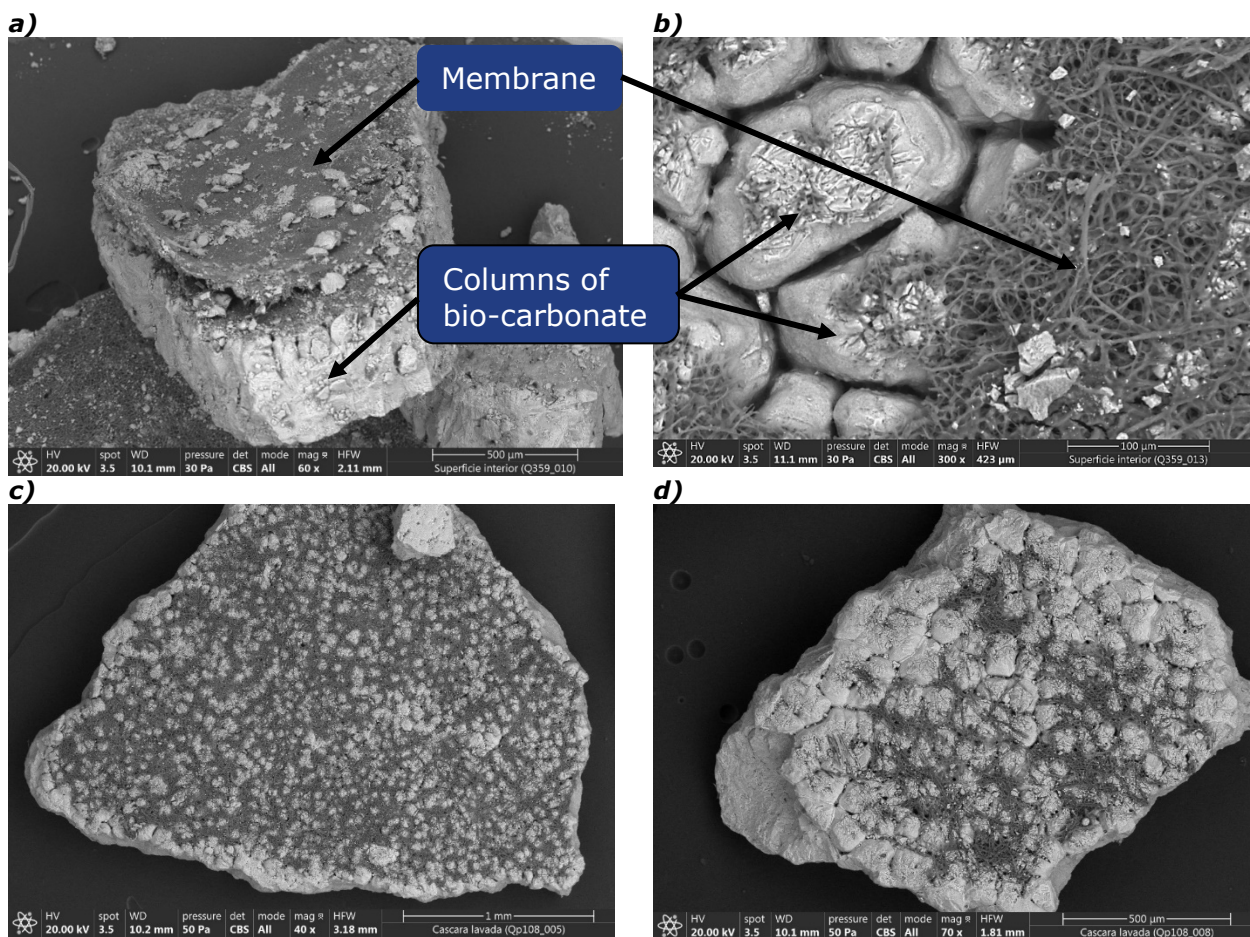


Figure 2. Microstructure of different bio-carbonate particles: a) particle with membrane prior to the separation process, b) detail of the surface of a particle with membrane, c)–d) surface of bio-carbonate particles after the separation process.

5. FEASIBILITY OF USING BIO-CARBONATE IN CERAMIC TILES

Composition development was initially carried out at laboratory scale, then at pilot scale, and finally at industrial scale. In the laboratory trials, bio-carbonate was introduced in full replacement of mineral carbonate (Table 2); the compositions with the bio-carbonate were seen to require a slightly higher deflocculant content and slightly lower solids content (Figure 3 and Table 3).

Raw materials	C-STD	C-CM
Mix of clays	55	55
Mix of non-plastic materials	30	30
Calcium carbonate	14	-
Micronised bio-carbonate	-	14

Table 2. Formulated compositions (% by weight)

Property	C-STD	C-CM	
Solids content (SC) (%)	72.0	71.9	70.8
Deflocculant content (%)	0.42	0.60	0.56
Minimum viscosity (cP)	613	1015	563

Table 3. Deflocculation behaviour

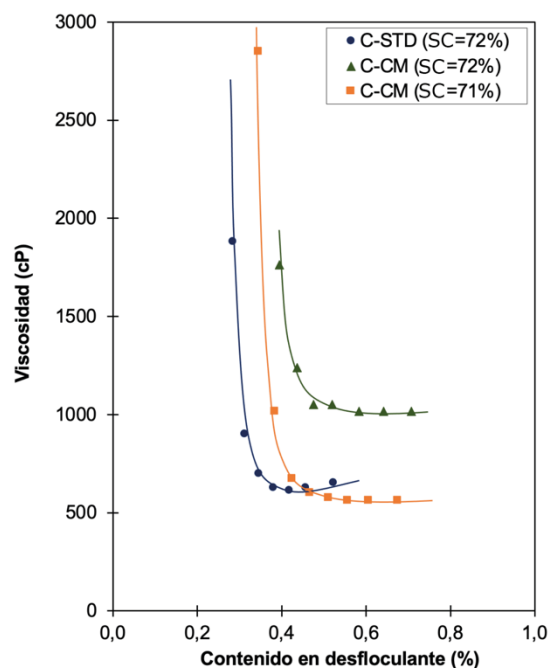


Figure 3. Deflocculation curves

With regard to the industrial trials, the test compositions and the characterisation results of the spray-dried powders are shown below. The results indicate that the partial substitution of calcium carbonate mineral by micronised bio-carbonate does not produce significant changes in spray-dried powder behaviour during the process, except for a slight increase in the compression pressure required to achieve the same compaction. This is logical bearing in mind that mineral carbonate was also micronised and that only one third of it had been replaced. As for the non-micronised bio-carbonate, a further increase, albeit slight, in compression pressure was required, and increased moisture expansion was noted, which could be due to the fact that the formation of calcium crystalline phases from the CaO after decomposition of the carbonates and silico-aluminates following clay dehydroxylation is delayed by the larger size of the bio-carbonate particles. Indeed, the bio-carbonate introduced in the C-2L composition had a size of less than 2 mm, which partly decreased during composition milling but very possibly without reaching the sizes of mineral carbonate and micronised bio-carbonate. Proof of this fact comes from the carbonate content in the fraction with sizes larger than 100 μm, which is practically non-existent in spray-dried powders STD and C-1L and notable in spray-dried powder C-2L.

Raw materials	STD	C-1L	C-2L
Mix of clays	55	55	55
Mix of non-plastic materials	30	30	30
Calcium carbonate	14	9	9
Micronised bio-carbonate	-	5	-
Bio-carbonate < 2 mm	-	-	5

Table 4. Compositions used in the industrial trials (% by weight).

Property	STD	C-1L	C-2L
Carbonate content (%)	13	13	13
Carbonate content in the residue on 100 µm sieve (%)	1.2	1.9	8.2
Moisture content (%)	6.0	6.0	6.0
Pressure (kg/cm ²)	275	305	315
Dry bulk density (g/cm ³)	1.927	1.926	1.927
Dry mechanical strength (kg/cm ²)	34	34	33
Linear shrinkage at 1140°C (%)	0.4	0.3	0.4
Bulk density at 1140°C (g/cm ³)	1.762	1.758	1.768
Water absorption at 1140°C (%)	18.6	17.8	17.5
Moisture expansion at 1140°C (‰)	0.80	0.80	0.90

Table 5. Behaviour during pressing and firing.

With these spray-dried powders, about 1000 m² of 30x90 cm earthenware tiles were produced and no changes in tile size or defects in the fired glaze surface were noted. On the other hand, it was necessary to adjust kiln cooling slightly (especially in the case of the composition with non-micronised bio-carbonate) so that tile curvature at the kiln exit would not vary from that of the STD composition.

From an environmental point of view, some changes were observed in the compounds present in the gases evacuated from the kiln stack, Table 6. This table only shows the gaseous compounds in which changes were noted between the firing of the STD composition and the firing of the composition with micronised bio-carbonate. Due to the small percentage of N present in the bio-carbonate, an increase in the content of all nitrogenous compounds was seen. Even so, in the case of NO_x (which is the only nitrogen compound for which there is an emission limit value in integrated environmental permits for the Valencia Region), the result is well below the limit value (250 mg/Nm³). A small increase in total Volatile Organic Compounds (tVOC) was also seen, although again the increase is practically insignificant compared to the limit value (50 mg/Nm³).

Parameter	Units	STD	C-1L
N₂O	mg/Nm ³	2	5
NO	mg/Nm ³	26	39
NO₂	mg/Nm ³	20	12
NH₃	mg/Nm ³	1	10
NO_x	mg/Nm ³	60	71
tVOC	mg/Nm ³	4	8

Table 6. Composition of the gas stream during firing of the STD and C-1L compositions. Values on a dry basis in relation to 18% O₂.

The microstructure of the fired pieces is as expected for earthenware tile bodies, Figure 4, exhibiting high porosity (dark zones), and abundant acicular crystals of anorthite (phase determined by XRD) are seen to have formed around the pores. This is logical as the porosity stems mainly from the decomposition of calcium carbonate particles (both from the mineral and from the eggshell calcium carbonate), so that the area around the resulting porosity is rich in calcium, which encourages the formation of anorthite from the reaction between CaO and the silico-aluminates from the dehydroxylation of the clay mineral [9].

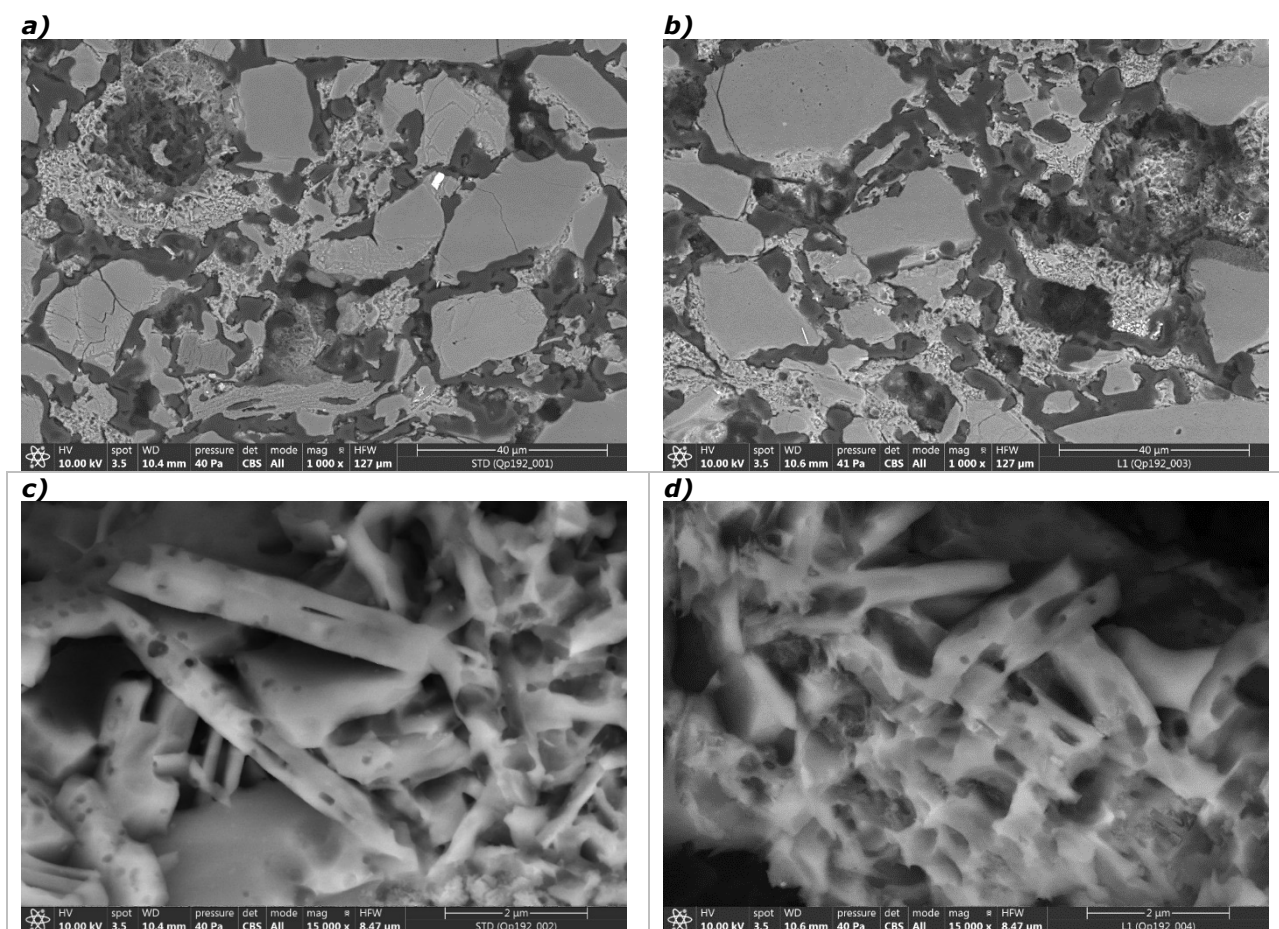


Figure 4. SEM micrographs of samples STD (a & c) and C-1L (b & d).

Figure 5 shows one of the tiles obtained with the spray-dried powder containing 5% bio-carbonate, and Table 7 details the results of the bending strength, water absorption, crazing resistance, and moisture expansion tests. The results obtained indicate that it is technically feasible to use bio-calcium carbonate in earthenware tile compositions as a partial replacement of mineral carbonate.



Figure 5. Earthenware tile produced industrially from a composition with 5% bio-carbonate.

Property	STD	C-1L	C-2L	Requirements
Water absorption	E=17.4% min 17.4%	E=18.6% min 18.5%	E=17.5% min 17.4%	E>10% min 9%
Bending strength	e ≥ 7.5 mm	e ≥ 7.5 mm	e ≥ 7.5 mm	e ≥ 7.5 mm
Breaking strength	1460N	1350N	1340N	≥600N
Bending strength	Mean = 23 N/mm ² Min. = 21 N/mm ²	Mean = 23 N/mm ² Min. = 22 N/mm ²	Mean = 23 N/mm ² Min. = 22 N/mm ²	Mean ≥ 12 N/mm ²
Moisture expansion	0.1 mm/m	0.1 mm/m	0.1 mm/m	Declared value
Crazing resistance	Resistant	Resistant	Resistant	Required
Crazing resistance (2 additional cycles)	Resistant	Resistant	Resistant	-----

Table 7. Tile properties.

6. FEASIBILITY OF USING BIO-CARBONATE IN CERAMIC FRITS

The charge formula for the test frits is detailed in Table 8. As indicated in the table, frit M-BC was formulated using micronised bio-carbonate and the necessary adjustments were made in the amounts of other raw materials used in order to match the reference frit composition (M-C frit) formulated with natural calcium carbonate.

Total substitution of natural calcium carbonate by bio-carbonate does not produce appreciable changes in the firing behaviour of the resulting frit (Figure 6), only a slight increase in its characteristic temperatures (Table 9) being observed.

These differences are possibly due to the difference in composition between bio-carbonate (Table 1) and natural calcium carbonate and their influence on frit thermal behaviour.

Raw material	Frit M-C	Frit M-BC
Quartz	7.7	7.5
Mix of feldspars	54.6	54.5
Dolomite	11.4	10.8
Alumina	5.8	5.7
Calcium carbonate	20.5	-
Bio-carbonate	-	21.7

Table 8. Charge formula of the test frits (% by weight).

Characteristic temperature (°C)	Frit M-C	Frit M-BC
Shrinkage onset (T _{IC})	780	785
Shrinkage end (T _{FC})	890	900
Softening (T _R)	1165	1175
Sphere (T _E)	-(*)	-(*)
Half-ball (T _{1/2})	1195	1205
Melting (T _F)	1215	1230

Table 9. Characteristic frit temperatures (°C).

*) Could not be determined

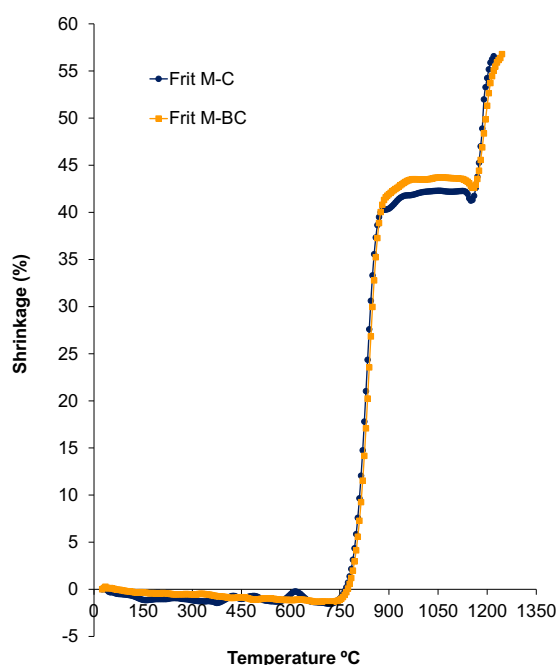


Figure 6. Frit shrinkage-temperature curves.

The developed frits were used to prepare the corresponding glazes. The colour coordinates and gloss of the vitreous coatings obtained with each of them are summarised in Table 10. The table also includes the colour difference (ΔE^*) determined for each temperature between a glaze made with bio-carbonate (with colour coordinates L^* , a^* , b^*) and the one made with natural calcium carbonate (with colour coordinates L_0^* , a_0^* , b_0^*), from the expression:

$$\Delta E^* = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2}$$

The total substitution of calcium carbonate by bio-carbonate produces a slight increase in coordinates L* and b* of the glazes and a decrease in colour coordinate a*. In no case are the differences significant and the colour variations (ΔE^*) compared with the reference composition are less than 0.5 for all test firing temperatures. In terms of gloss, the use of bio-carbonate gives rise to glazes with slightly lower mattness, which may be related to the small differences detected in frit melting behaviour (Table 9 and Figure 6). However, the differences are once again hardly noticeable. Figure 7 shows a comparison of the appearance of the fired glazes obtained with both compositions.

Ref	Temp (°C)	L*	a*	b*	Gloss (85°)	ΔE^*
M-C	1180	90.7	0.04	3.56	19	-
	1200	90.2	-0.27	3.37	25	-
	1220	90.1	-0.48	2.81	31	-
M-BC	1180	90.9	-0.03	3.80	15	0.4
	1200	90.4	-0.33	3.56	21	0.3
	1220	90.2	-0.53	3.11	29	0.3

Table 10. Colour coordinates, colour differences (ΔE^*) and gloss of the glazes

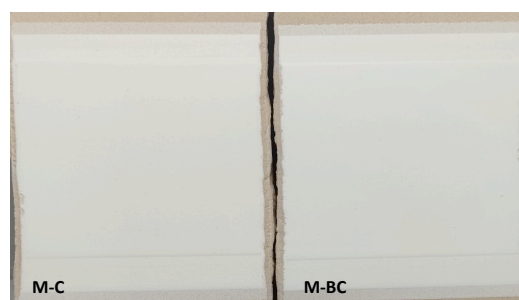


Figure 7. View of glazes obtained at 1200°C. Composition M-C (left) and M-BC (right)

Finally, the influence of using bio-carbonate on the dilatometric behaviour of the glazes was assessed. The results (Figure 8 and Table 11) indicate that the modifications made to the mixture of raw materials used to obtain the frits did not produce significant changes in glaze thermal expansion, which might lead to defects from a mismatch of glaze and body thermal expansion.

Coefficient of expansion	M-C	M-BC
$\alpha_{50-300} (°C^{-1}) \times 10^7$	69	67
$\alpha_{300-500} (°C^{-1}) \times 10^7$	80	78

Table 11. Coefficients of expansion in the test glazes ($°C^{-1}$).

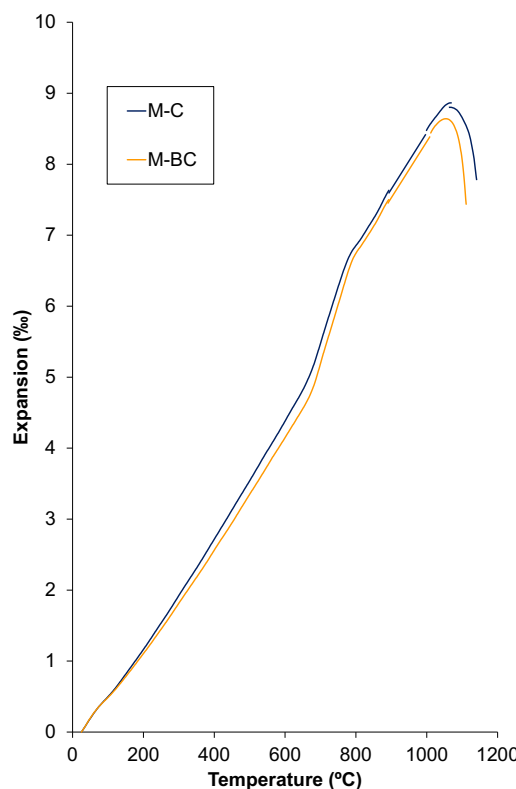


Figure 8. Dilatometric behaviour of the test glazes.

7. CONCLUSIONS

The main conclusions drawn from the study are as follows:

- The use of bio-calcium carbonate in partial replacement of mineral carbonate in earthenware tiles is technically and environmentally feasible in the percentage tested (5%), given that the earthenware tiles obtained present no defects or significant changes in their properties.
- However, small changes in some process variables (pressing pressure and adjustment of kiln cooling) are needed when micronised bio-carbonate is introduced (size below 100 μm). When used with a particle size resulting from the implemented membrane separation process (< 1 mm), the changes are slightly higher.
- The technical feasibility of using micronised bio-carbonate in the development of ceramic frits has been verified without any significant changes taking place to their properties or to those of the resulting vitreous coatings. In light of these results and given the greater porosity of eggshells compared with natural calcium carbonate, the use of bio-carbonate with a larger particle size in the development of frits is being considered, which would thus lower the cost associated with frit production.
- The two valorisation strategies studied are technically feasible and could represent an important step forward in the transition from a linear economic model to a circular economy for both the egg production and ceramic sectors.

8. ACKNOWLEDGEMENTS

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