# DESIGN OF HYPOCARBONIC WALL TILE COMPOSITIONS BASED ON TALC

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#### ABSTRACT

The ceramic tile industry faces a major challenge related to its complete decarbonisation. Although the main contribution to  $CO_2$  generation comes from the burning of natural gas, there is an important contribution from the process itself, since most compositions for the manufacture of wall tiles are formulated with calcium carbonate as a calcium oxide supplier, giving rise to an additional source of  $CO_2$  in the firing process. Consequently, the design of compositions that reduce, or eventually eliminate this contribution is a strategic issue in the future of the industry. Although there have been many attempts to replace calcium carbonate with other raw materials, it is not easy to replace it for technical and economic reasons.

This work proposes the design of porous glazed compositions incorporating talc as magnesium oxide-contributing raw material to replace a large part of the calcium carbonate currently used. At the same time, for the total elimination of calcite raw material, the use of a little amount of secondary calcium oxide-contributing raw material, such as marble dust or tailings from diatomite mining, is also addressed. The selected raw materials were characterised in terms of chemical and mineralogical compositions. Based on these characterisations, a series of compositions were designed, using the aforementioned raw materials, trying to reproduce process behaviour and the final properties of the ceramic pieces obtained with a reference composition containing natural calcium carbonate. To evaluate this behaviour, the grinding, pressing and firing stages were reproduced at laboratory level, following procedures similar to those used in industrial practice. Finally, with a selected composition, a pilot trial was carried out with the aim of shortening the firing cycle, by which glazed pieces were produced.

The results obtained have shown that total substitution of calcium carbonate is possible by combining the magnesium oxide-contributing raw materials (talc) and the secondary raw material (marble dust or diatomite tailings). It has also been shown that the proposed composition allows acceleration of the firing process by considerably reducing the calcium carbonate decomposition stage of the firing cycle, with the consequent decrease in energy costs and  $CO_2$  emissions associated with the firing process.

#### **1. INTRODUCTION**

The European Union seeks to reduce its  $CO_2$  emissions by 40% in 2030 relative to those of 1990 as a temporary goal on the way to full decarbonisation of the European economy in 2050. The emissions reduction target for the industry to which the ceramic sector belongs lies at 34–40% by 2030 and 83–87% by 2050. In the ceramic sector, achieving the almost 90% emissions reduction target in 2050 will require combining endogenous and exogenous technologies, which will demand radical changes in product design, modifications of processes and technologies, and changes in available energy sources.

Endogenous measures include the elimination of calcium carbonate in white body tile compositions, since this raw material, which provides the calcium necessary for the manufacture of the product, is responsible for up to 30% of the total  $CO_2$  emissions of this type of tile. On the other hand, the decomposition of calcium carbonate, a strongly endothermic reaction, requires a significant contribution of thermal energy, which, due to the combustion of natural gas, represents an increase in energy costs and further  $CO_2$  emissions.

The alternative to calcium carbonate as a calcium oxide supplier is not simple, neither technically nor economically, due to its low price. The decomposition of calcium carbonate, at temperatures between 800 °C and 900 °C, provides a source of calcium oxide that reacts with the silica and alumina from the destructuring of clay minerals, giving rise to the desired crystalline phases of calcium silicate and silicoaluminate, wollastonite and anorthite, respectively, which leads to ceramic pieces with low shrinkage during firing, which are stable against the action of moisture once fired [1].

The direct alternative to calcium carbonate is natural wollastonite, which provides the desired crystalline phase without any emissions. Numerous studies have been carried out on the substitution of calcium carbonate by wollastonite, proving, with the necessary adjustments in the composition, that the substitution is technically feasible [2,3]. However, its excessive price makes it an unfeasible proposal today.

Given the very limited availability of alternatives to calcium carbonate, another line of research of interest is related to raw materials that provide another alkaline earth, which is magnesium oxide, with the same objective of developing crystalline phases during firing without  $CO_2$  emissions.

If dolomite is discarded, since it is a double carbonate of calcium and magnesium, a common ceramic raw material that could be used is talc.

Even though talc can provide magnesium oxide after destructuring at around 1000 °C, thermodynamics tells us that the crystalline phases are fully developed at temperatures that can even exceed usual wall tile firing temperatures [4]. For this reason, the most extensive use of talc in the ceramic tile industry is related to its incorporation, in low proportion, in porcelain stoneware compositions to lower the firing temperature due to the fluxing effect of magnesium in the vitreous phase.

Finally, a line of research that has been developing enormously in recent years, in the framework of the circular economy, is related to the use of wastes from other industries, known today as secondary raw materials. In a recent review, Zanelli et al. compile the main wastes that have been investigated for use in ceramic tile compositions [5]. In the case of calcium carbonate substitution, the paper describes some tested wastes, such as those coming from the cement industry or from blast furnace slag.

A waste product used as a substitute for calcium carbonate is marble dust [6]. During the extraction, cutting and treatment of marble, a significant amount of dust is produced which, although it is also used as an aggregate in construction, is of great interest to the ceramic industry because of its composition (practically made up of calcium carbonate). Spain is one of the main European producers of marble, with most of the production concentrated in the southeast of the country (Alicante, Murcia and Almeria), so there is high availability of this waste.

Another waste that could be of interest for use as a replacement for calcium carbonate comes from the mining of diatomaceous earth or diatomite. Diatomite is basically a mixture of amorphous silica and calcium carbonate (chalk) with multiple applications as a filler or filtering material in numerous industries. In areas of Albacete and Murcia there are large deposits that generate important quantities of waste that could be used in the ceramic industry, specifically, for total or partial replacement of calcium carbonate. Some previous research was performed to transform diatomite mineral samples into a wollastonite-rich raw material for wall tile compositions [7], but no study is known where the possible direct use of this diatomite tailing in wall tile compositions has been addressed.

In light of the above, this work is aimed at using talc together with waste from marble and diatomite mining as possible alternatives to calcium carbonate in a standard wall tile composition. The behaviour of the alternatives proposed in the different stages of the manufacturing process will be analysed and the products obtained will be characterised in a comparative way. All this with the ultimate aim of proposing alternatives to reduce  $CO_2$  emissions and energy consumption of current wall tile compositions.

# **2. EXPERIMENTAL**

## **2.1. MATERIALS**

A standard wall tile composition was formulated as reference for comparative purposes. This composition was made up of a Spanish white ball-clay (BC), a feldspathic sand (S) and a commercial calcite (CaC). All these materials are commonly used in Spain for wall tile manufacturing. For the alternative compositions, on the one hand, two types of talc (T1) and (T2) with different purity were selected as sources of magnesium. On the other hand, marble dust (M) and diatomite tailings (D) were used as calcium carbonate waste. Marble dust is composed almost entirely of calcium carbonate with traces of dolomite, while diatomite tailings are made up of a mixture of calcium carbonate and amorphous silica. These materials were supplied by IMERYS, with the exception of the marble dust which was obtained from Centro Tecnológico del Mármol (CTM). Table 1 details their chemical analyses.

Material	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	Other	LOI
T1	46.8	8.0	1.6	31.5	1.6	0.3	0.2	0.5	9.5
T2	39.3	12.5	2.1	32.1	1.7	0.4	0.5	0.4	11.0
М	1.2	0.2	52.2	2.5	0.1	-	0.1	0.3	43.4
D	65.4	1.3	15.6	0.4	0.5	0.1	0.1	0.2	16.4

Table 1. Chemical analyses of the raw materials used for the alternative compositions

# 2.2. DESIGN, PREPARATION AND CHARACTERISATION OF PROPOSED COMPOSITIONS

Table 2 shows the compositions tested together with the standard composition. As can be seen, two series of compositions were designed from the standard composition (STD), with the aim of reducing or even eliminating calcium carbonate in its entirety.

Dow motorial	STD	Mg	compositi	Ca/Mg comp.		
Raw material		T1-15	T1-30	T1T2	МТ	DT
Ball-clay (BC)	50	50	41	41	36	30
Sand (S)	35	35	29	29	29	25
Calcite (CaC)	15	-	-	-	-	-
Talc 1 (T1)	-	15	30	15	30	15
Talc 2 (T2)	-	-	-	15	-	15
Marble dust (M)	-	-	-	-	5	-
Diatomite (D)	-	-	-	-	-	15

Table 2. Formulated compositions series based on talc

In the first series, calcium carbonate was completely replaced by raw materials incorporating only magnesium as an alkaline earth element (T1 and T2). Since the molar contribution in magnesium oxide of talc is lower than that of calcium carbonate in calcium (0.12 moles Mg vs 0.15 moles Ca per 100 g product), two compositions were proposed, one with the same proportion of raw material with talc as the standard in calcium carbonate (15%) and another with twice as much (30%), so as to verify more clearly the effect of this raw material on the composition as a whole. A last composition was also prepared with a mixture of two types of talc (15% T1 and 15% T2). The compositions with talc were referenced T1-15 and T1-30 in relation to the percentage they contained. The composition with the mixture was referenced T1T2.

In the second series, calcium- and magnesium-containing raw materials were used to reduce the amount of natural calcium carbonate. As indicated in the literature related to calcium-rich products [8,9], a 5% contribution of calcium carbonate can be very effective in reducing expansion due to humidity and providing long time stability in use. Talc was again used as magnesium raw material, while marble dust and diatomite tailings were employed as calcium secondary raw materials. In the case of the diatomite tailings (D), 15% was introduced in order to incorporate, approximately, the equivalent of 5% calcium carbonate (see Table 1). The compositions, referenced MT and DT, are detailed in Table 2.

The different compositions were prepared at laboratory scale following the usual processing route for ceramic materials with wet milling, drying, pressing and firing in an electric furnace. Grinding was carried out in planetary mills with ball loading for 25 min to obtain a 63  $\mu$ m sieve residue of less than 1%. The slurry was dried under infrared lamps and dry milled to obtain a fine powder. This powder was wetted to a water content of 5.5 wt% and pressed at a pressure of 250 kg/cm<sup>2</sup>. The cylindrical specimens, with a diameter of 40 mm and a thickness around 5 mm, were fired following a cycle with a fast heating ramp up to 500 °C, a ramp at 25 °C/min from 500 °C to maximum temperature and a dwell time at maximum temperature of 6 min. Firing temperatures were 1100 °C and 1150 °C, seeking to cover the usual working range in industrial practice.

The laboratory fired pieces were characterised by determining linear shrinkage and water absorption. Linear shrinkage was calculated from the difference between the dried and fired diameters of the specimens measured with a calliper, defining this parameter on a dry basis. Water absorption was determined by the vacuum method according to the ISO 10545-3:2018 standard from the weight gained by the specimens after the test.

Regarding moisture expansion, which is a characteristic to be minimised in fired wall tiles [10], it is usually calculated by direct dimension measurement. However, as the absolute expansion value is too low, determination by weight is preferable in the interest of greater accuracy. Thus, moisture adsorption (MAd) was obtained from the weight gain ( $m_{f}$ - $m_{i}$ ) of the specimens after being exposed to an autoclave cycle with a pressure of 10 kg/cm<sup>2</sup> for 5 hours, following Equation (1). The pieces were dried in an oven at 110 °C for 20 minutes before determining the weight at the end of the test ( $m_{f}$ ).

$$MAd(\%_0) = \frac{m_f - m_i}{m_i} \cdot 1000 \qquad Eq.(1)$$



## 2.3. DEVELOPMENT OF A PILOT TRIAL

Once the laboratory phase had been completed, a pilot trial was carried out with the aim of optimising the firing step and verifying the reduction of cycle time that could be achieved from compositions with reduced carbonate content. According to the literature [11], the replacement of carbonates in porous wall tile compositions by other raw materials that neither decompose nor undergo endothermic transformations, as well as the reduction in cycle time that this involves, leads to a significant reduction in specific energy consumption and  $CO_2$  emissions of the firing step. For the pilot test, small-sized glazed ceramic tiles were prepared from one of the analysed compositions. The pieces were prepared by wet-milling, spray-drying, pressing, glazing and firing in a gas kiln.

The device used for wet grinding was a ball mill of 50 cm diameter and 53 cm length. The material load was 40 kg with 122 kg of alumina balls at a rotational speed of 47 rpm. Spray-drying was carried out using a Newtech spray dryer. It has a maximum evaporation power of 30 kg water/hour (by means of a gas burner) and allows obtaining of spray-dried powders with very similar characteristics to those obtained industrially. Table 3 shows the main variables used in the milling and spray-drying steps.

Pressing of the pieces was carried out using a Maer semi-industrial press, obtaining a format of 15x15 cm and a thickness of about 7 mm. The pressing conditions were a moisture content of 5.5 wt% for the powder and pressure of 250 kg/cm<sup>2</sup>. The engobing and glazing operations were performed in an industrial glazing line of dried bodies by means of cascading applications. Industrial slips were used for the applied layers with an engobe weight of 400 g/m<sup>2</sup> and a glaze weight of 1250 g/m<sup>2</sup>. A glaze with a transparent, glossy finish was used. Finally, firings were performed under industrial conditions, following a wall tile cycle, and using a Proying pilot kiln. Different firing cycles were performed based on a standard profile of a wall tile composition containing carbonate in order to shorten this standard cycle.

Condition	Value
Slip solids content (%)	68
Slip density (g/cm <sup>3</sup> )	1.76
Slip viscosity in a Ford cup (s)	44
Residue at 63 µm sieve (%)	2.0
Inlet gas temperature in spray dryer (°C)	300
Outlet gas temperature in spray dryer (°C)	130
Peristaltic pump speed (%)	40
Air pressure in the spray nozzle (bar)	2

Table 3. Summary of the milling and spray-drying conditions used for the pilot trial

# **3. RESULTS AND DISCUSSION**

## 3.1. COMPARATIVE ANALYSIS WITH STANDARD COMPOSITION OF THE PROPOSED SERIES OF COMPOSITIONS

Figure 1 represents, in the form of histograms, the water absorption, firing shrinkage and moisture adsorption at the two tested temperatures for the alternative compositions tested together with the STD composition. As can be seen, all the compositions present, at any temperature, porosity values well above 10%, corresponding to the group of porous wall tiles according to ISO 13006:2018.

Regarding the linear shrinkage and its variation with temperature, it is found that, in all cases, the linear shrinkage values exceed those presented by the standard composition. In general, this stems from the crystalline phases that would be expected to develop in the  $SiO_2$ -Al<sub>2</sub>O<sub>3</sub>-MgO system compared to the  $SiO_2$ -Al<sub>2</sub>O<sub>3</sub>-CaO system [12]. Although these phase diagrams represent thermodynamic equilibrium situations, which are guite far from those taking place in industrial practice, their information can be equally useful. Thus, for a composition with 15% calcium carbonate, a phase equilibrium of 30% mullite and 70% anorthite (calcium silico-aluminate) would be expected to exist at a temperature of approximately 1170 °C. On the other hand, for a composition with 15% talc, the expected equilibrium would give rise to 16% mullite and 84% cordierite (magnesium silico-aluminate), although, in theory, this would occur at temperatures above 1300 °C. Consequently, the formation of crystalline phases that stabilize the pieces with temperature (alkaline-earth silicates and silico-aluminates) is much less effective in the case of compositions incorporating magnesium oxide instead of calcium oxide. However, shrinkages for any of the proposed compositions are acceptable for wall tiles, especially at temperatures close to 1100 °C.

Analysing the moisture adsorption, it is quite evident that compositions with only magnesium as an alkaline earth element show too high values, although there is a small difference depending on the talc used due to the different refractoriness between them. In fact, industrial wall tile compositions show values ranging from 1‰ to 3.5‰. However, moisture adsorptions are reduced by introducing a small amount of calcium. This is associated with the formation of anorthite in these compositions during the firing process, the existence of which has been confirmed by XRD analysis. The presence of anorthite in the body, even in low proportion, stabilises/mineralises the metastable minerals to avoid their rehydration and creating moisture expansion [13]. Furthermore, the results in Figure 1 also confirm the technical feasibility of using waste as calcium carbonate source, marble dust in the case of the MT composition and diatomite tailings in the case of the DT composition. Thus, the proposed compositions promote waste revalorisation, following circular economy principles, in particular the DT composition which incorporates 30% waste.



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*Figure 1. a) Linear shrinkage, b) Water absorption and c) Moisture adsorption at the tested temperatures for the two series of compositions together with the STD composition* 

Figure 2 shows a series of plots to clarify the effect of phase composition and water absorption (porosity) on moisture adsorption. For this purpose, the range of variation for the maximum firing temperature is increased and the composition is modified according to the percentage of MgO.

Figure 2a presents the moisture adsorption as a function of porosity for the STD composition (with CaCO<sub>3</sub>), the T1T2 composition (with Mg) and the MT composition (with Ca and Mg). As can be seen, moisture adsorption decreases as the porosity of the sample is reduced and higher amounts of crystalline phases are formed, enstatite in the case of compositions containing talc (as confirmed by XRD analysis), and anorthite for compositions with calcium carbonate. Moreover, adsorption increases for the Ca-free composition and the effect of calcium carbonate is demonstrated, even when the addition is small (5%).

To understand the influence of the composition, Figure 2b illustrates moisture adsorption as a function of the amount of MgO (talc) when the sample has a porosity typical of wall tiles  $(17\pm2\%)$ . This figure reveals the effect of alkaline earth elements on adsorption. Thus, an increase in the amount of Mg decreases moisture adsorption, although the variation is more pronounced if the sample contains Ca, in the form of calcium carbonate or marble dust. This is a consequence of the interaction between Ca and Mg, which favours lower adsorption. Thus, as recently reported, MgO develops a mineralisation effect on anorthite crystallisation [12]. Nevertheless, this hypothesis should be validated with further investigation.



*Figure 2. a)* Moisture adsorption versus water absorption (porosity) for compositions with different amounts of Ca and Mg and b) Moisture adsorption versus amount of MgO for compositions with similar porosity

#### **3.2. DEVELOPMENT OF A PILOT TRIAL FOR FIRING OPTIMISATION**

Based on the results obtained in the laboratory, the MT composition was selected for the pilot test according to the conditions described in section 2.3. This composition shows suitable firing properties, especially regarding moisture adsorption.

The starting point was cycle 1 in Figure 3, corresponding to a standard firing curve for wall tile, in which, as can be seen, there is an important section, between 800 °C and 900 °C, with a lower heating rate, destined for the decomposition of calcium carbonate (in some industrial kilns this section can reach up to 30% of the total firing cycle).

Another cycle, designated cycle 2, was designed by eliminating the calcium carbonate decomposition section mentioned above. In this curve, in addition, the heating rate was softened in the section between 900 °C and 1000 °C, given the dehydroxylation reaction that talc presents in that temperature range [4]. This profile leads to a shortening in the firing time by more than 10 minutes.

As for the appearance of the tiles, it was even better in the pieces fired according to curve 2 than in the pieces fired according to curve 1. Despite the reduction in firing time, no appreciable defects were found on the glazed surface due to the decomposition of the carbonates, which validates the proposal presented.





**Figure 3.** Firing cycles performed in the pilot test with the MT composition, distinguishing the temperature in the upper and lower zone

Following the solutions proposed, Table 4 summarises the reductions in  $CO_2$  emissions that could be achieved, distinguishing the contribution due to the change in composition with respect to the STD composition from that achieved through the savings in energy consumption by shortening the firing cycle. For this, it must be considered that the reduction in calcium carbonate content means a  $CO_2$  reduction of 0.44 kg  $CO_2$  per kg  $CaCO_3$  and the shortening of the firing cycle leads to a lower gas consumption and a reduction of  $CO_2$  emissions at a rate of 0.2 kg  $CO_2$  per kWh. Furthermore, a specific gas consumption in the firing cycle of whiteware wall tiles equal to 885 kWh per tonne of product and a specific weight of 20 kg/m<sup>2</sup> is considered, according to the literature [14].

The table considers several possibilities depending on the carbonate content of the final composition and the reduction in the firing cycle. The most favourable case corresponds to a composition in which calcium carbonate is completely eliminated and a similar cycle reduction is achieved as in the pilot test (energy savings of around 17%). In the worst case, the composition still contains a small amount of calcium carbonate (5%) and the modification in firing cycle is more limited (energy savings of 8%).

As can be deduced from the values in Table 4, the reduction in  $CO_2$  emissions is achieved by the combination of both measures, the reduction in carbonate content and the shortening of the firing time, neither of which is negligible. This is caused by the high energy consumption during wall tile firing, so that any variation in the cycle produces considerable emissions savings. The total estimated emissions reduction is between 1 and 2 kg  $CO_2$  per m<sup>2</sup> of product, depending on the composition and the firing curve.

Carbonate	Energy	Reduction in CO <sub>2</sub> emissions (kg/m <sup>2</sup> )				
content (%)	savings (%)	Material	Firing cycle	Total		
0	8	1.32	0.28	1.60		
0	17	1.32	0.60	1.92		
5	8	0.88	0.28	1.16		
5	17	0.88	0.60	1.48		

**Table 4.** Estimated CO2 emissions savings depending on the amount of calcium carbonate inthe final composition and the applied firing cycle reduction

#### **4. CONCLUSIONS**

The aim of this work was to eliminate calcium carbonate from a typical wall tile composition. For this purpose, the introduction of magnesium oxide-contributing raw materials, such as talc, was considered. The characterization was based on the key aspects of wall tile compositions: their stability with firing temperature during processing (low firing shrinkage and small variation of this parameter with firing temperature) and their stability against the action of humidity once the pieces are finished (low adsorption by humidity).

It has been found that none of the compositions proposed achieves the high dimensional stability associated with the standard composition with calcium carbonate, as a result of the greater capacity for the formation of crystalline phases of the SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-CaO system compared to the SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-MgO system. Also, the moisture adsorption values obtained are higher than those achieved with the calcium compositions. To reduce moisture adsorption, the introduction of marble dust or diatomite tailings, which are secondary raw materials, has been found to be very efficient and adds additional benefits to the composition.

For this reason, a pilot test has been carried out with one of these compositions on glazed pieces. It has been proven that with small adjustments in the firing cycle, it is possible to fire pieces in considerably shorter cycles than those usually used in the industry with compositions that incorporate calcium carbonate, fundamentally due to the elimination of the firing curve section with the slowest heating rate for the decomposition of calcium carbonate. This important reduction of the firing cycle translates into a significant reduction of natural gas consumption and  $CO_2$  emissions associated with this fuel.



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