DB3.2.1 Report of the industrial trials

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1. Introduction

The actions contemplated in the LIFE EGGSHELLENCE project are structured in five groups of actions, being those of type "B" the Implementation actions of the project. This deliverable corresponds to Action B3 “Industrial trials” and describes the industrial trials performed by EATOMIZADO and ADM and the subcontracted ceramic companies, with the help of ITC-AICE and UA, in the manufacture of tiles with bio-$CaCO_3$ at industrial scale. The bio-$CaCO_3$ used in these trials has been provided by AGOTZAINA by processing eggshell in the prototype already installed by MAINCER in its facilities.

In the following sections, the performance of the industrial trials is described.

2. Spanish trials

The spray-dried powders prepared by EATOMIZADO were taken to a tile manufacturer, where approximately 1000 m^2 of 30x90 cm tiles were manufactured.

It was necessary to modify the pressing pressure in order to achieve similar bulk density values, while the rest of variables weren’t modified.

It was observed that the manufactured tiles with a 5% of bio-$CaCO_3$ didn´t present any changes in tile size or defects in the glazed surface (*Figure 1)*.

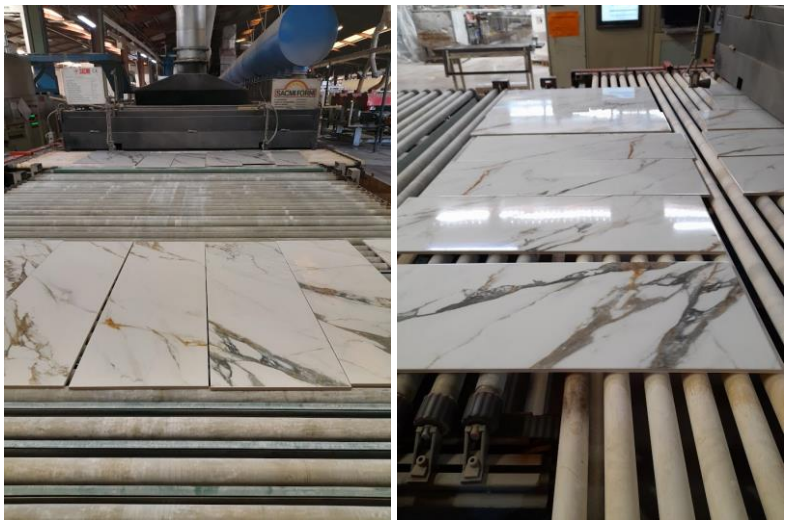

Figure 1. Exit of the kiln with the processed pieces.

In addition, some tiles were decorated with a special design containing the bio-carbonate resulting of the separation process implemented in AGOTZAINA, as well as the logo of the project, the logos of project partners and LIFE logo.

Figure 2. Specially decorated tiles obtained with the spray-dried powder containing 5% of bio-carbonate.

Apart from this industrial trial, smaller trials were performed in other tile manufacturers with the same spray-dried powders. The objective of these small trials was to confirm the absence of defects in the glazed surface

in different tile sizes and kilns associated with the use of bio-calcium carbonate. This absence of defects was checked in all the cases (3 small trials) in which medium sized tiles were manufactured (up to 30x90 cm as in the case of the big trial) but there were two trials in which large sized tiles (40x100 cm and 60x180 cm) were manufactured and pinholes were observed (Figure 3) in the case of the spray-dried powder with bio-carbonate not pre-milled. This is due to the fact that larger tiles are usually manufactured with higher thickness and then, carbonate decomposition takes longer time (and the same thing happens with coarser particles of carbonate). As in Spain the great majority of tile producers consume compositions in which mineral calcium carbonate is micronized, firing programs are adjusted for this type of spray-dried powders. Therefore, the larger the tile size, the more probable the presence of defects in the case of spray-dried powders with bio-calcium carbonate in its original size.

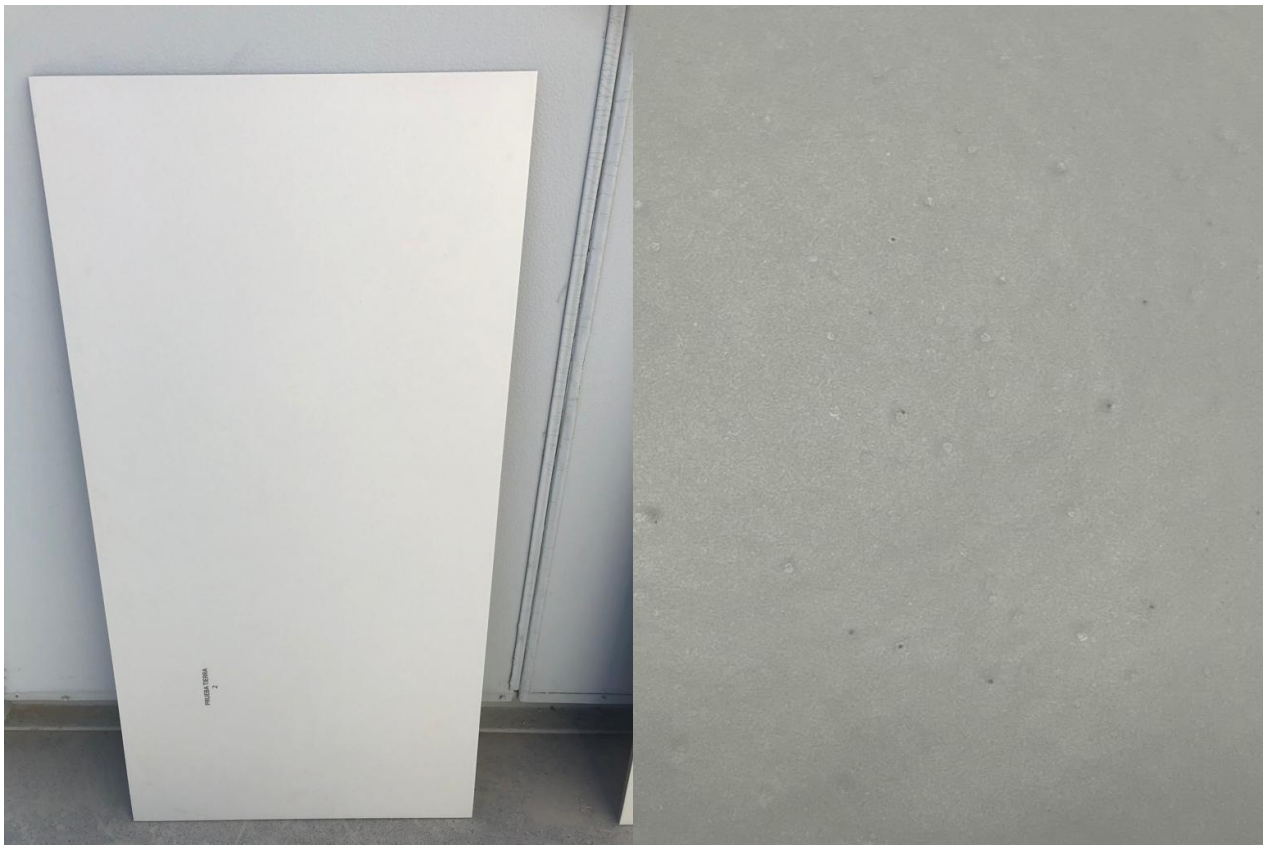

Figure 3. Large sized tiles with pinholes obtained with the spray-dried powder containing 5% of bio-carbonate (< 2 mm).

3. Portuguese trials

3.1. Test with Bio-$CaCO_3$ (50% replacement)

An industrial test was carried out with the composition in which Bio-$CaCO_3$ was introduced in partial replacement (50%) of mineral calcite. Approximately 1000 m^2 of tiles were pressed with a format of 30x60 cm (*Figure 4*).

Figure 4. The pressed tiles dried and entering in the glazing line at about 85ºC.

For the glazing of the manufactured tiles, a white glossy glaze was chosen.

Figure 5. Glazing of the tiles.

The manufactured tiles were fired in a continuous roller kiln with a firing cycle of 55 min.
In order to control the shrinkage and to avoid a problem, burner 26 was switched off.

The main defects observed in the manufactured tiles were pinholes in the glaze, which resulted in second-quality material. In addition, fragmented corners and holes were detected.

A total of 976.6 m^2 were packed in the first selection, the percentage of first quality being 76%.

Figure 6. Wall tile with bio-carbonate exposed in ADM facilities.

The tiles with STD paste had a percentage of 79% of first quality.

The main defects were holes and debris, which resulted in 2nd class material, and for disposal there were tiles with air bubbles and glass drips.

The material with bio-carbonate showed very favourable warping.

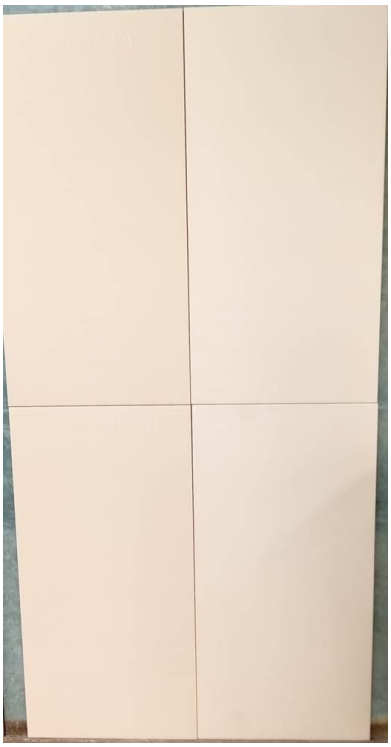

Figure 7. Fired wall tiles without glaze (left STD, right Bio-$CaCO_3$).

3.2. Test with Bio-$CaCO_3$ (60% replacement)

The spray-dried powder was delivered to a customer to produce glossy wall tiles of 15x15 cm. Moisture content was 6.4% and pressing pressure was 280 kg/cm^2. Firing conditions were 1050ºC maximum temperature and 48 minutes of firing program.

Approximately 920 m^2 of wall tiles with the bio-$CaCO_3$ spray-dried powder were manfactured and classified as first quality (around 90% of the manufactured tiles). STD composition provided slightly higher percentage of first quality (+ 2%) and due to the small size of the tiles there were no significant changes in tile size, planarity and mechanical properties.

4. Conclusions

The trials performed allow the following conclusions to be withdrawn:

- **Spanish trials:**
  - ✓ Pressing pressure had to be increased in the compositions with bio-calcium carbonate (specially in the case of bio-carbonate not milled), which is in good agreement with the results of the characterization of the spray-dried powders.
  - ✓ The tiles manufactured showed no defects in the glazed surface in the trial.
  - ✓ Smaller trials were performed in other companies with the aim of confirming the absence of defects in the glazed surface in different tile sizes and kilns associated with the use of bio-calcium carbonate. This absence of defects was checked in all the cases in which medium sized tiles were manufactured but pinholes were observed in two trials in which large sized tiles were manufactured. Therefore, in Spanish ceramic sector some tile manufacturers may require the use of micronized bio-calcium carbonate in order to avoid defects in glazed tiles.
- **Portuguese trials:**
  - ✓ Almost no change has been observed in the behaviour of the compositions with bio-carbonate in the manufacturing process with respect to STD formulation in the two trials (30x60 and 15x15 cm tiles).
  - ✓ The percentage of first quality tiles of the compositions with bio-carbonate was almost the same than with STD tiles (although the value depended strongly on tile size: 30x60 cm tiles had a significantly lower percentage of first quality than 15x15 cm tiles).