Case Study 2: Ceramic Powder Processing

Case study host:

Euroatomizado, Onda, SPAIN

Case study leader:

Newcastle University, Newcastle, UK

Case study team:

Newcastle University, Newcastle, UK

Euroatomizado, Onda, SPAIN

ITC, Castelló, Spain

Analisis DC, Madrid, SPAIN

David Reay & Associates, Newcastle, UK

Brief description of process unit(s) of interest for intensification and motivation:

This case study has been focused on the intensification of the free-flowing powder preparation process involved in the very beginning of the ceramic tile manufacturing process. Nowadays, the most widely used ceramic tile shaping method is uniaxial pressing of spray-dried powder in hydraulic presses .The base case involves a wet route procedure where the initial free-flowing powder is obtained from a high solid content slurry, which itself produced by wet grinding of a mix of different solid raw materials with water and additive in continuous or discontinuous mills.

A free-flowing powder with a certain granule size distribution is required to assure a good distribution of the powder during the filling of press cavities. This is achieved in a spray dryer. Indeed, a heterogeneous distribution of the powder would cause different kinds of defects after firing e.g. differences in final tiles size, lack of orthogonality, or product deformations.

Together with the free-flowing character of the resulting powder, spray drying is also beneficial for the ceramic process because of the highly homogeneous final composition of the powder obtained by wet milling of solid raw materials. This is of major importance to dilute the possible impurities and thus lower their negative influence in the final product.

Spray-drying is proven to be a capable technology allowing one to obtain a granulated powder, the physical properties of which are specially adapted to the shaping of ceramic tile bodies by uniaxial pressing. Together with its homogeneous chemical composition, the spray dried powder shows four main properties which are key during the ultimate shaping stage:

1. Moisture content of 5-7 %: ideal for developing the plasticity of the clay fractions contained in the raw materials mixes.

2. The granulated powder obtained by spray-drying shows an almost spherical shape with very smooth surfaces.
3. The free-flowing spray died powder has a very narrow grain size distribution containing agglomerates from 125 to 1000 microns in size.
4. The spray dried granules are hollow inside because of the physical phenomenon involved in their formation.

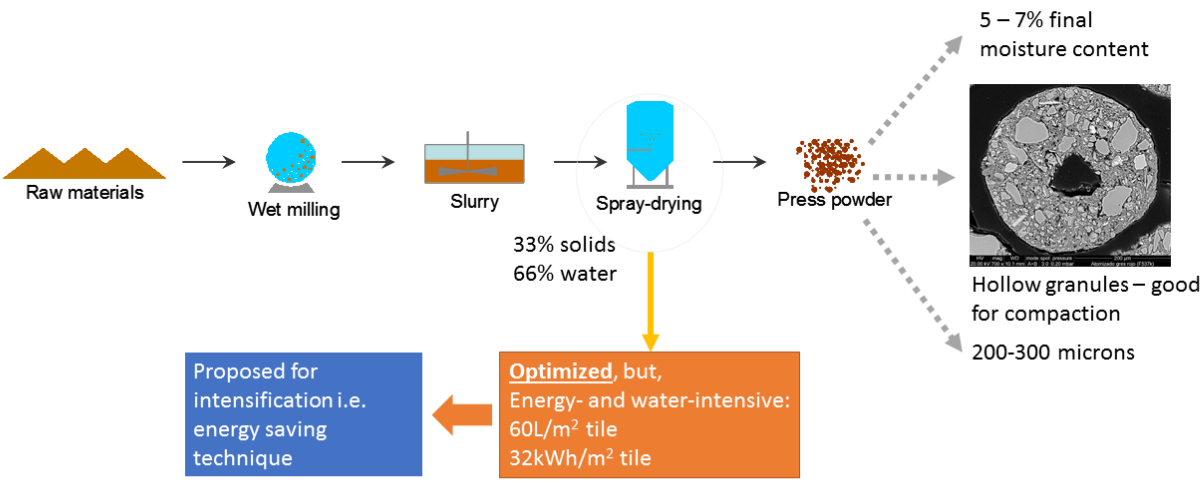


Figure 1. General flowsheet of the spray drying process in ceramic processing, which forms the basis behind case study CS2.

The primary objective of the case study was testing several dry route processing as an energy-efficient alternative to the established wet route processing, as shown in figure 2:

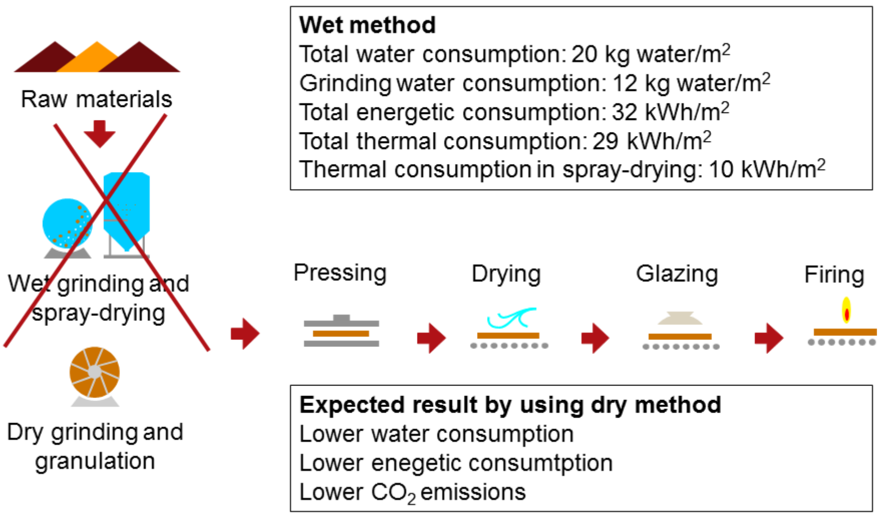


Figure 2. The merits of the dry route process. These are related to its energy and water savings.

Tests have not only included the validation of the intensified routes to produce a free-flowing powder, but also pilot tests to verify its behavior in the whole ceramic tile manufacturing process. And thus, to confirm that the physical and chemical properties of the final product prepared from the new powders are in accordance with the quality standards of the market.

Two technologies have been evaluated. On the one side, a direct high-shear mixing granulation in which a free-flowing powder is directly produced by agglomeration of the dry powder provided by a pendulum mill, using water as agglomeration agent. And on the other side, a rolling compaction methodology in which the dry powder provided by a pendulum mill is compacted by a pelletizer system and then the resulting pellets are grinded to obtain a powder with the desired particle size distribution.

The main difference between the two evaluated technologies is that after granulation, free-flowing powder needs to be dried to achieve the desired working moisture content. This post-process drying is not necessary when using the rolling compaction approach as the resulting powder contains the right amount of water.

Brief description of PI technology chosen:

As explained, two process intensification technologies have been chosen and proved during the development of the project: high-shear granulation and rolling compaction. Both technologies have been used to process dry powders provided by a pendulum mill. In both proposed dry routes, the pendulum mill would define the particle size distribution of the primary powder particles. By processing these primary powders, the process intensification technologies have been tested, adapted and optimised to obtain free-flowing powders with the best possible physical properties. The free-flowing powder physical properties considered during the technologies optimization have been the granule size distribution, the moisture content, the shape and strength of the granules and the flowability of the powder.

The high shear mixer-granulator is an established technology which has been retained from a KBE analyses conducted together with the different partners involved in the case study. Its highlight is that the granules are less spherical and denser, which presented lower deformation capability during tile pressing.

Together with Offenburg University staff, and other IbD partners, different workshops and working sessions have been conducted to define, by means of a TRIZ approach, new possible intensified technologies for the case study. By defining the problems associated with the case study functional analysis, different potential solution concepts were generated at the last workshop. And for this solution concepts the most suited technologies were the following ones:

- High shear granulation process:
- Roller compactor for granulation
- Screw granulation

Two of the proposed technologies suited for the detected solution concepts were already present in the KBE list of promising technologies, concretely, the high shear granulation and the screw granulation. The third one, the roller compacting technology for granulation, showed a good potentiality to be used as intensified technology. After some tests were performed with good results in one small scale facility located in Manfredini & Schianchi Srl site, in Sassuolo (Italy), further investigation was decided to be conducted in a complete pilot facility.

Granulation is a process involving particle size enlargement and is a complex process controlled by various mechanisms such us: wetting, nucleation, agglomeration and consolidation, breakage/attrition and layering. The final characteristics of the obtained granulates depend on the equipment design. The equipment used in this demonstrator is the special EIRICH mixing system comprising 3 main components. The way in which these components are used can be varied in a particularly flexible mode when designing the mixing processes.

- The rotating mixing pan, which delivers the mixture into the area of the mixing tools
- One or more mixing tools arranged eccentrically. The direction of rotation and the speed of the mixing tool(s) can be optimally adapted to the different applications.
- The bottom/wall scraper, providing additional agitation action. It prevents caking on the wall and bottom of the pan and facilitates discharge when the mixing cycle is complete.

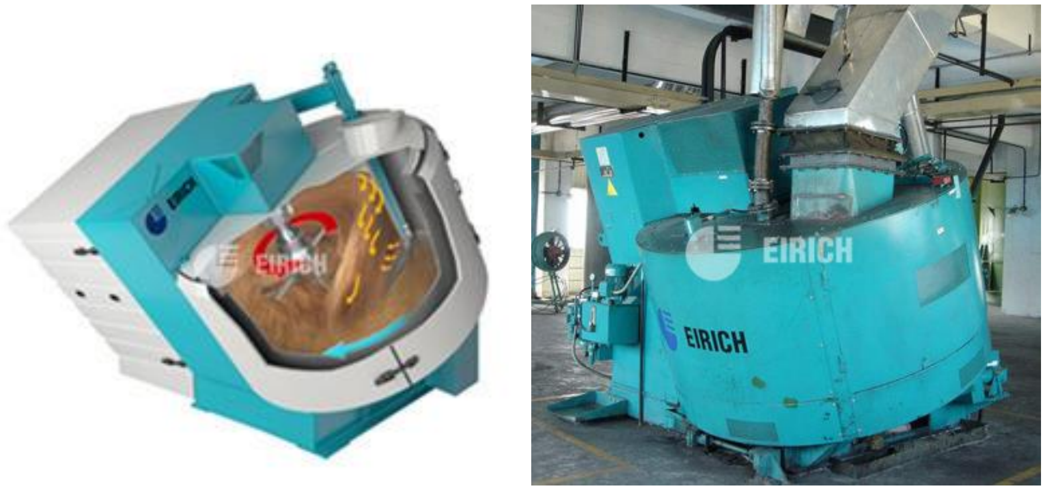


Figure 3 Eirich granulation-mixing technologies for dry granulation of ceramic powders (left: batch mixers, right: continuous mixer).

Concerning the **rolling compaction technology,** a pilot facility provided by the Italian company Manfredini & Schianchi was installed in Euroatomizado’s site. This facility was based in the Fusion technology developed by Manfredini and it has been used to verify the preliminary results obtained in several tests conducted in the supplier facilities. Using the Manfredini roller system based on the principle of press-agglomeration, particulate material is introduced into the nip of the two counter-rotating rollers by means of vertical feeders. As the material was compacted, pressure within the material increases, reaching a peak just above the line of closest approach between the rollers, and subsequently drops rapidly to zero. During this process, the apparent density of the mixture increases by a factor of 1.5 to 3 due the decreasing void volume of the bulk material. The resulting product is typically a pelletized material as the one shown in figure 4.

The pelletized material produced in the roller press should be broken up into granules and classified into the desired particle size range. Often, the first crushing step takes place in a specially designed flake breaker installed directly below the roller press. The pellets were broken into smaller pieces (flakes) which make perfect feed for subsequent crushers.

Using a multi-deck screen, the material was then separated into oversize, product and undersize classifications. The undersize (which passes directly through to the lowest screen deck) was re-circulated for compaction, while the oversize formed a residue on the upper screen deck which was fed back into the size reduction process.

Figure 4. Pelletized material resulting from the rolling compaction stage of the Manfredini Fusion system.

The main advantage of this system is that the quantity of water which is required for powder pelletizing is the same as the one required in the final product. Because of this, it is not required to use a big amount of water to guarantee a good granulation of the powder and no ulterior drying operations would be required. However, during pilot testing, special efforts have been conducted to achieve a homogenous shape in the final granulates as, by defect, they show irregular shapes after the crushing stages conducted after pelletizing.

Different pictures of the pilot rolling-compaction facility deployed at Euroatomizado's site are shown. On the one hand the pelletizer and the crushing equipment, and on the other hand, a detail of the pelletizing molds.

Figure 5. Pictures of the rolling-compaction pilot facility.

Different pictures of the high shear granulation equipment are presented below:

Figure 6. High shear mixing equipment.

Brief summary of results:

Main results obtained for both technologies are summarized below:

- **Granulation:**

    - Good granule size distributions could be obtained with the tested technology. The resulting granules sizes are like the ones obtained by the wet non-intensified technology. Moreover, the working parameters to control these distributions have been identified and studied. The evolution of the granules size during the process have been determined and used to validate simulation models

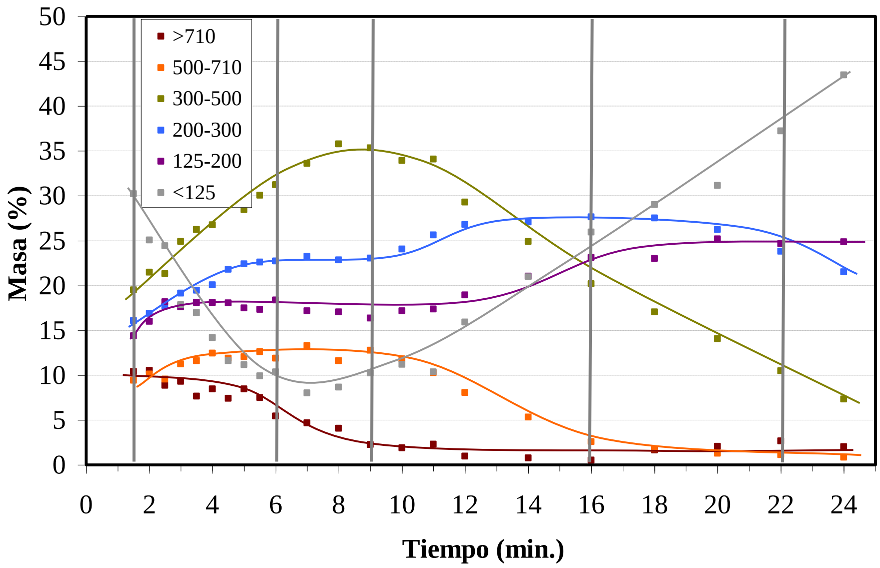


Figure 7. Evolution of granule size fractions with granulation time at variable granule moisture contents

- The evolution of granule bed density and of granule hardness with granulation time was determined. The hardness of the granules obtained with the high-shear granulator was greater than that of material obtained by spray drying, after stirring times above 2 minutes.

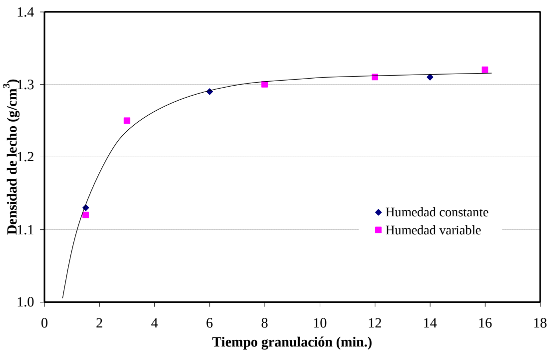


Figure 8. Bed density

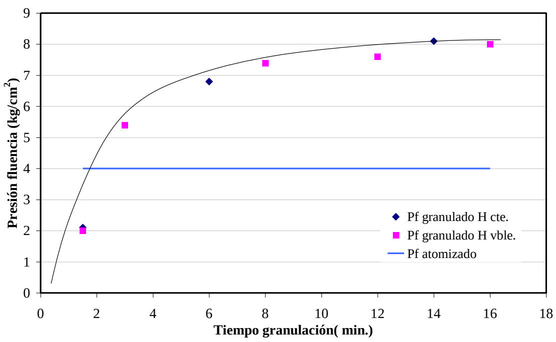


Figure 9. Yield pressure

- Properties of the resulting tiles shaped by using granulated powders are similar to the ones obtained with spray-dried powders. The main disadvantage of the technology is that a post-drying is necessary to condition the moisture content of the powders after granulation.

- **Rolling compaction:**

  - The properties of the resulting tiles obtained with the free-flowing powders provided with this technology are very good and not moisture conditioning is necessary after the process.

	Intensified Technology	Standard spray-drying
Powder moisture content (%)	6,5	6,5
Dry bulk density (kg/m^3)	2079	2160
Dry Mechanical Strength (kg/m^2)	27,5	26,4
Linear Shrinkage (%)	-0,75	-0,56
Fired Bulk Density (kg/m^3)	1761	1831
Water Absorption (%)	18,70	16,44
Fired Mechanical Strength (kg/m^2)	23,8	23,6

  - The technology needs to be improved by using other grinding technologies to provide a better control of the final granule size distribution.

Final conclusions from case-study:

The main conclusions drawn from the validations and works shown in this report are as follows:

- Concerning the **high shear mixing technology**, the water addition method did not influence granule size distribution. When the agglomerate moisture content remained constant, the growth mechanism prevailed throughout the test. The granule size fraction between 300 and 500 µm maximised at about 8 minutes. This maximum practically coincided with the minimum of the fraction below 125 µm under the tested conditions. This granule size curves are like that obtained with spray drying base case.

- Regarding the **rolling compaction technology**, the grain size distribution which could be obtained is not so good as the one obtained with the high shear mixer granulator. The conducted tests shown that with the current range of variation in the working parameters it is difficult to obtain final powders with a grain distribution curve like the one obtained by spray drying. Results evidence that further development in the grinding technology should be done to reduce the thinner fractions of grains. Anyway, the resulting powders have shown a very good ceramic behaviour and research on alternative grinding technology have been started with very good preliminary results.

- TRIZ methodology has been useful to identify alternative PI technologies. The most promising results have been obtained with the rolling compaction technology which has been proposed from a the TRIZ workshop sessions.

- All the evaluated technologies involve a dramatic reduction of energy and water consumption. From this point of view, the rolling compaction technology linked, with an appropriate grinding technology is the most suitable technology, as it allows a powder preparation with a restricted use of water. High shear-mixing technology requires using higher amounts of water which lately need to be removed by drying.

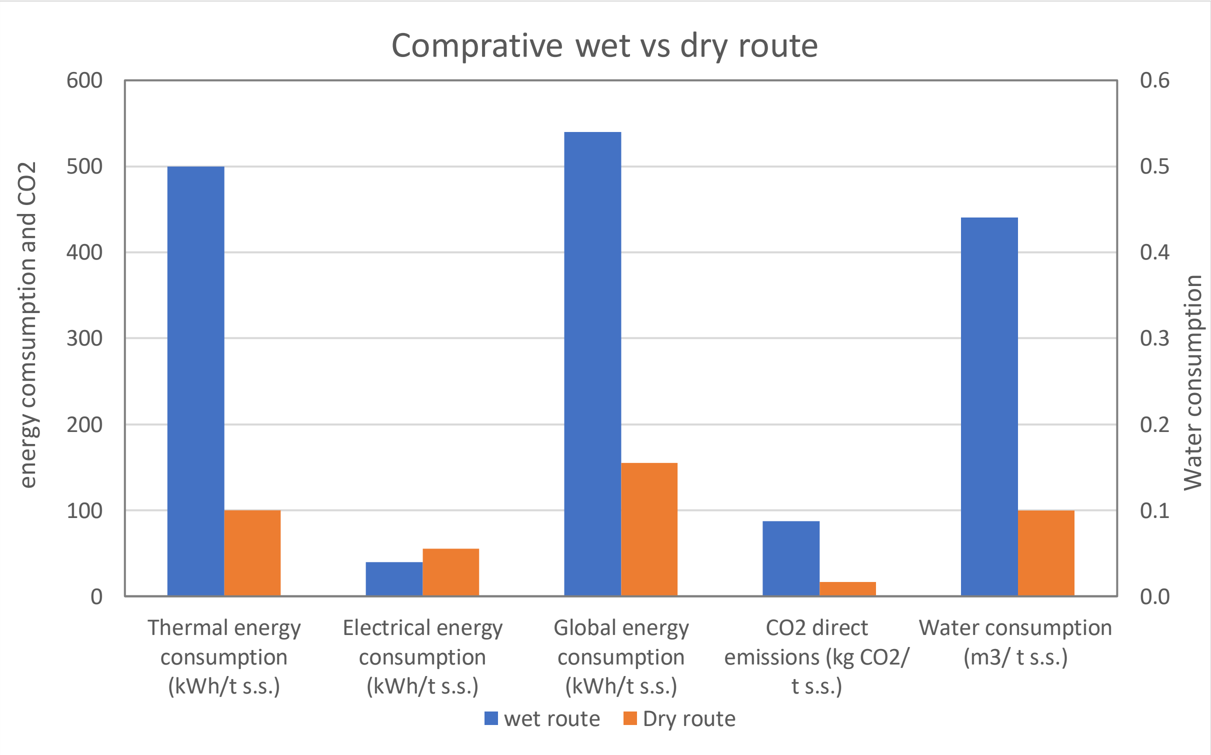
- Additionally, the impact of the case study has been evaluated for impact by predicting energy savings, reduction in water consumption and other environmental benefits as follows.

In the following tables the water and the energy consumptions, as well as the CO_2 emissions, are summarized for both technologies: the standard wet method and the intensified dry method ((a) d.s.: dried solid (b) HHV: Higher Heating Value (c) Emission factor for natural gas: 0.202 kg CO_2/kWh).

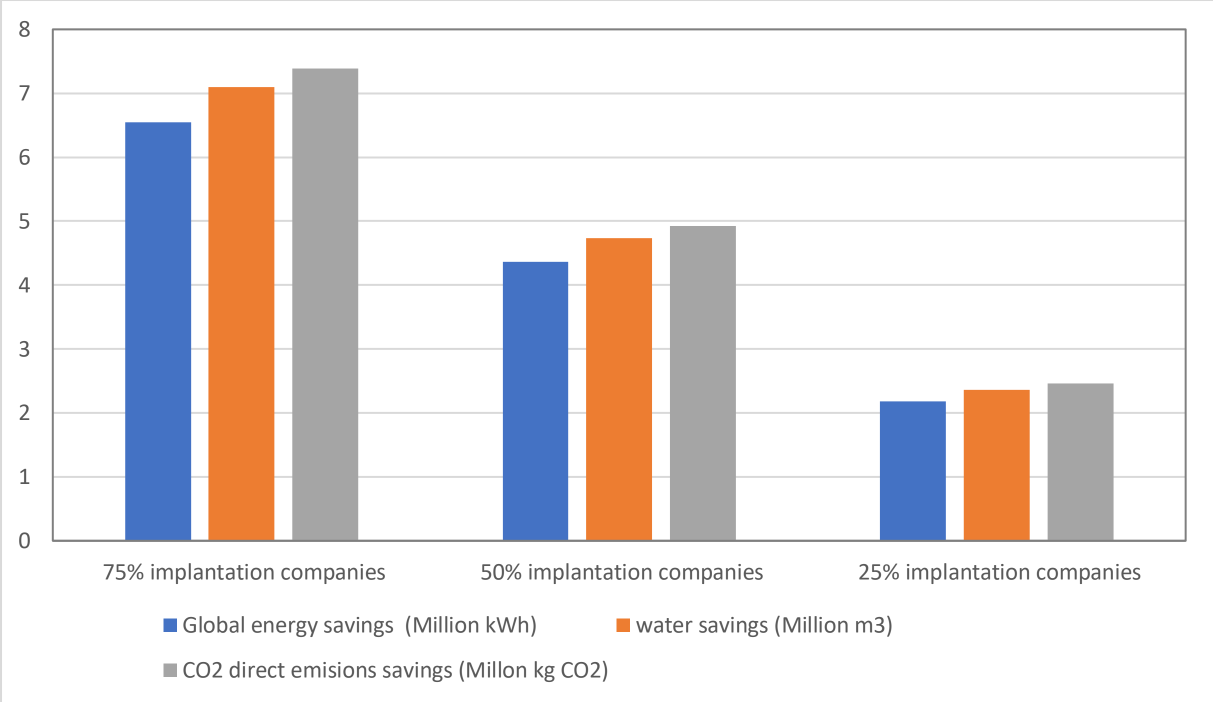
Wet process	Consumption
Water consumption	0.47-0.59 m^3/Mgd.s.[a]
Electrical energy consumption	50-54 KWh/Mg d.s.
Thermal energy consumption (in HHV[b])	442-462 kWh/Mg d.s.
CO_2 direct emisions [c]	80-84 kg CO_2/Mg d.s.

Dry process	Consumption
Water consumption	0.12-0.16 m^3/Mg d.s.[a]
Electrical energy consumption	31-35 KWh/Mgd.s.
Thermal energy consumption (in HHV[b])	88-108 kWh/Mg d.s.
CO_2 direct emisions [c]	16-20 kg CO_2/Mg d.s.

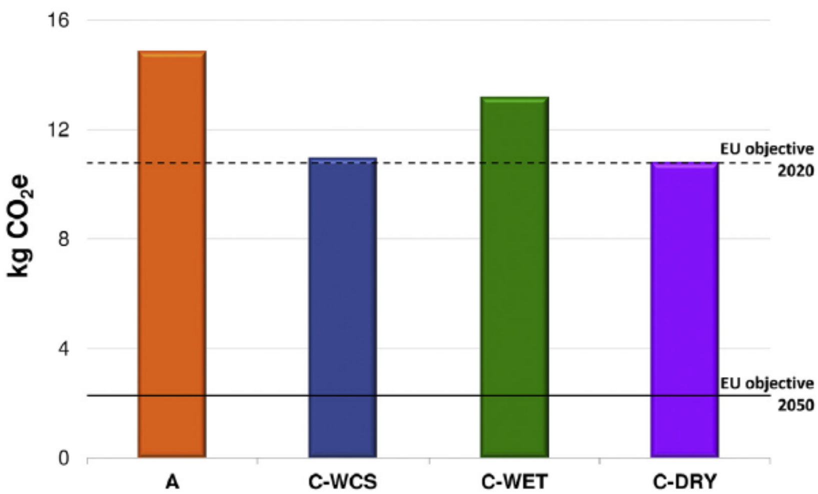
All of these results are represented in the following comparative chart which evidence the impact achieved by the transformation to a dry route process. Especially important is the thermal energy reduction attained by the dry route procedure.



These results have been extrapolated to a global production of 13 million of tons of spray dried product. The impact data referred to this global situation area plotted in the following chart.



Finally, a comparison between the impacts of different technological alternatives has been also conducted. The considered alternatives are: wet route (WET); wet route using combined heat and power cogeneration systems (WCS); and dry route (DRY). The WCS is very popular in the Spanish ceramic tile industry. A combined heat and power cogeneration system (CS) installed in the spray-drier allows for the simultaneous production of electric and thermal energy with high efficiency, but it entails more natural gas consumption than the thermal process itself. As it can be seen, the DRY alternative is the most suitable from the point of view of Carbon emissions.



TRL of PI Technology:

Both proven technologies can now be considered TRL 7-8 for manufacturing free flowing powder for ceramic tile manufacturing.