STUDY OF THE SPRAY-DRYING OPERATION OF CERAMIC POWDERS ON AN INDUSTRIAL SCALE, ITS CONTROL AND AUTOMATION

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ABSTRACT

This study addressed control and automation of the spray-drying operation. This required previously selecting the suitable operating variables, and the appropriate measuring instruments.

On analysing the data obtained on the different variables, an empirical correlation was derived between drying gas temperature, flow rate of the solids suspension, and moisture content of the produced agglomerate. The correlation allowed the drying facility to be automatically controlled.

1. INTRODUCTION

The great offer at present available in the ceramic wall and floor tile market, together with growing quality demands by users, make final tile quality vital to the product's competitiveness.

One of the parameters affecting tile quality is its dimensional stability. Green compaction of ceramic tile decisively impacts dimensional stability of the fired ware [1].

The main variables affecting compaction of the pressed pieces can be split into two groups: the variables associated with the agglomerate powder feed and variables related to the pressing operation itself. To be highlighted in the first group are the raw materials of the composition and the physico-morphological characteristics of the agglomerate (size, shape, moisture content, flowability, etc.) [2][3]. As the raw materials involved are characteristic of each of the many compositions used industrially, the study performed mainly focused on agglomerate powder characteristics.

In the composition preparation process by the wet route, the most widely implemented method in the Spanish ceramic wall and floor tile sector, agglomerate powder is obtained in the drying stage by spray-drying an aqueous suspension of clay raw materials [4-6]. Highly stable agglomerate characteristics entail greater uniformity in green compaction of the moulded pieces, both in one and the same piece (elimination of wedging)) as well as among varying pieces (keeping differences in dimensions, i.e. sizes, from arising).

Quality demands, as far as dimensional stability is concerned, which ceramic wall and floor tile manufacturers require of their products, far exceed the requirements laid down in current regulations on quality (EN Standards) [7].

Thus, for stoneware floor tile, a dimensional difference of ± 0.5 mm in the pieces is usually considered a deviation in size. Joint analysis of the vitrification and compaction diagrams shows that this difference can arise as a result of a variation in green bulk density of about ± 0.015 g/cm³ [8]. However, the value of this interval of bulk densities must be obtained in each specific case, according to the composition used.

This variation in compaction can be due to alterations in some of the above variables related to spray-dried powder.

The situation detected industrially, depicted in Figs. 1 and 2, shows swings in moisture content values of an agglomerate obtained in a spray dryer, which are typically those of a deficiently controlled process.

It was also shown that particle-size distribution of the powder is held within reasonable, suitable bounds in order to subsequently, correctly carry out the pressing step.

If spray-dried powder moisture content is assigned full responsibility for changes in the compaction of green ware, in order to bring about a variation of 0.03 g/cm³ (size), a change of powder moisture content is required of ± 0.004 kg water/kg dry solid, for a standard kind of body in the usual working moisture content ranges.

Therefore, a change in agglomerate moisture content of 0.008 kg water/kg dry solid, can of itself give rise to sizes in tile manufacture.

Given the great variability observed in moisture content of the produced agglomerate, and its marked impact on the possible appearance of sizes among the fired pieces, it was considered advisable to take this variable as the principal variable to be controlled.

The fluctuations in moisture content of agglomerates produced in spray-drying facilities, can be due to the following causes:

- Alterations in the properties of the suspension to be spray dried and/or of the pumping equipment.
- Modifications in the physical properties of the hot air used in the drying operation.
- Variations in the parameters governing the workings of the spray-drying facility.



Figure 1. Daily evolution of agglomerate powder moisture content.



Figure 2. Weekly evolution of agglomerate powder moisture content.

2. OBJECTIVE AND SCOPE

Owing to the deficiencies mentioned above, referring to the variability in agglomerate powder moisture content, it was considered of interest in this study to attempt to establish reliable empirical correlations between different operating variables and the moisture content of the solid discharged from the drying facility. The correlations found would allow implementing more thorough control of the operating conditions of the drying facility, for subsequent automation.

The following courses of action were planned to obtain this objective:

- Selecting the most important process variables to be taken into account in the facility and choosing the most suitable measuring instruments, as well as their location in the industrial plant.

- Running trials to obtain the empirical correlations between the moisture content of the spraydried powder and the following variables:
 - Temperature and humidity of the hot gases entering the drying chamber.
 - Feed flow rate of the aqueous clay suspension.
- Implementing an automatic control system for the spray-drying operation.

3. EXPERIMENTAL FACILITY

An industrial spray-drying facility used in stoneware floor tile manufacture was utilized to carry out the trials. The drying facility has a nominal maximum evaporation capability of 4.70 m³ evap. water/h.

A peculiar characteristic of the facility is the use of hot air from a cogeneration turbine as drying air. The hot air flow rate remained virtually constant during the whole operating time of the drying facility. Before the gases enter the drying chamber, they must cross an "air stream" burner, whose purpose is guaranteeing the targeted gas temperature.

4. MATERIALS AND EXPERIMENTAL PROCEDURE

The study was performed using an aqueous suspension of wet ground, red illitic-kaolinitic clays, which are employed in stoneware floor tile manufacture, as feed slurry.

Raw materials processing to obtain the slurry that was fed to the dryer, took place at the same industrial plant where the study was carried out. The slurry obtained after wet grinding was stored in suitably stirred tanks, where its density and viscosity was controlled, so that these remained practically constant throughout the trials.

Four series of trials were run, corresponding to different slurry feed flow rates. Four different trials were performed for each flow rate tested, modifying drying gas temperature.

It was attempted in developing each trial, prior to effecting the measurements, to have the dryer working in the targeted operating conditions for a sufficiently long initial period to have established stationary conditions when the values of the different process variables were measured.

Data on the different operating variables, measured in the ongoing operation in real time, were acquired and stored in each test, by means of data acquisition equipment.

As an important precautionary measure, it was attempted to keep the distribution of the suspension spraying nozzles and their average outlet openings as constant as possible during testing time, so that particle-size distribution of the agglomerate powder obtained would be affected to the least possible extent.

5. RESULTS AND DISCUSSION

5.1. SELECTION OF THE MOST IMPORTANT VARIABLES TO BE TAKEN INTO ACCOUNT IN THE FACILITY STUDIED. LOCATION AND ACCURACY OF THE MEASURING INSTRUMENTS

The following variables were continuously or periodically measured:

5.1.1. Periodically measured variables

These operating variables were intermittently determined in the course of each trial. The two variables monitored were:

- Viscosity of the slurry (μ). A rotary viscometer was used to measure this variable.
- Density of the slurry (ρ). This was determined by using a previously tared, graduated test tube, weighing a given suspension volume.

5.1.2. Variables that were measured continuously

The following operating variables were continuously measured:

- Volume flow rate of the slurry feed (Q).

An electromagnetic flow-meter was chosen to measure this variable [9-10]. The choice was made by taking into account the field of application of the equipment and its limitations, as it had to measure flow rates of fluids with rather abrasive suspended solids, continuously and in real time.

The equipment was placed in the vertical duct taking the slurry, after it had crossed the filters, into the drying chamber.

The accuracy of the equipment is $\pm 1\%$ of the suspension flow rate.

- Temperatures of the drying gases entering the chamber and of the exiting humid gases.

K-type thermocouples were used to measure both temperatures, located at the respective duct points at which the temperature profiles were most stable and symmetrical.

The accuracy of thermocouples depends on the temperature range to be measured:

- When the temperature interval to be measured ranges from 450 to 700°C, thermocouple accuracy is ± 0.75 °C.
- For temperatures from 90 to 110° C, accuracy is $\pm 3^{\circ}$ C.
- Mass flow rate of drying gases entering the dryer (G_i) .

Mass flow rate measurements were performed with a TORBAR type modified Pitot tube. This instrument allows measuring at high temperatures (500-700°C), continuously provides an average reading from the full profile analysis of dynamic pressures in the piping and is simple to install and maintain.

The equipment was placed in the vertical duct of the gases entering the dryer, at a point where gas flow was shown to be stable. It has an accuracy of $\pm 1\%$ of the gas flow rate.

- Moisture content of the produced spray-dried powder (X_{\downarrow}) .

The following series of characteristics were considered in choosing the equipment to measure the moisture content of the solid obtained: a) measurement of the moisture content of granular solids, b) reliability in measuring moisture contents ranging from 0.03 to 0.07 kg water/kg solid, c) robustness in the face of abundant ambient dust and possible water vapour condensation, d) easy handling, e) no or low dangerousness, and f) yielding a continuous moisture content reading in real time.

Taking these considerations into account, an infrared device was chosen to measure solids moisture content. Before choosing the instrument a series of laboratory verifications was run, in collaboration with the firm manufacturing the instrument, allowing an algorithm and some special filters for measuring moisture content in ceramic materials to be obtained [11].

The equipment was installed on the conveyor belt transferring spray-dried powder from the spaydryer exit to the storage and rest bins.

Its correct functioning also required installing a spray-dried powder sampler that would take representative fractions of the total solids flow. This assembly assures sampling objectivity and continuously supplies the moisture sensor with homogeneous samples.

The equipment has an accuracy of ± 0.001 kg water/kg dry solid.

Figure 3 illustrates the location of the different measuring devices that were installed in the plant.

After having calibrated the measuring equipment, the electric signals from the installed sensors were gathered in a digital control data acquisition system that allowed each of the process variables mentioned to be continuously and independently monitored.

5.2. RELATIONSHIP BETWEEN TEMPERATURE AND HUMIDITY OF THE HOT GASES ENTERING THE DRYING CHAMBER AND MOISTURE CONTENT OF THE PRODUCED SPRAY-DRIED POWDER

In order to determine the relationship between drying gas temperature and moisture content of the produced spray-dried powder, four series of trials were run at four slurry flow rates. The temperature of the gases at the spray-dryer entrance was modified so that the moisture content of the spray-dried powder obtained would vary within preset limits (0.04 to 0.07 kg water/kg dry solid) (Figure 4).

The first trials substantiated that humidity of the gases at drying chamber entrance remained almost unvarying.

Figure 5 plots the results obtained for each of the slurry feed flow rates tested, for each average incoming gas temperature, virtually keeping the remaining operating variables constant.



Figure 3. Placing of the measuring instruments



Figure 4. Evolution of the measured process variables, for a slurry flow rate of 9.5 m^3/h



Figure 5. Relationship between incoming gas temperature and moisture content of the produced spray-dried powder, for varying slurry feed flow rates.

A linear relation can be observed between incoming gas temperature and average moisture content of the discharged spray-dried powder, of the form $X_4 = mT_1 + b$.

As at a practical level, when the moisture content of the agglomerate powder leaves the preset limits, the incoming drying gas temperature will be the actuating variable in controlling the spraydrying process, a multiple regression equation has been fitted of the form:

$$T_1 = 352.3 + 61.14 \cdot Q - 4909 \cdot X_4$$
 $r^2 = 0.993$

This equation allows predicting what the incoming drying gas temperature must be, in order to obtain a targeted spray-dried powder moisture content, from a preset slurry flow rate.

Figure 6 plots the experimental values of the incoming hot gas temperatures versus the temperatures predicted by the fitted empirical equation. Both temperatures match well.

This correlation then represents the first step in establishing a control system for the spray-drying operation, and makes it possible to fully automate this drying stage in the ceramic tile manufacturing process.

There are different alternatives for implementing an automatic control system for the spray-drying process. One involves the control loop that has been used on an industrial scale, shown in Figure 7. The drying facility's peculiarities have been taken into account in working out the control loop.



Figure 6. Relationship between incoming gas temperatures monitored in the different trials and those predicted by the fitted empirical equation.



Figure 7. Schematic of the industrially implemented control loop.

After implementing the control loop of the moisture content in the spray-dried solid, the fluctuations currently arising industrially are depicted in Figs. 8 and 9, showing substantial improvement in the stability of the solid's moisture content.



Figure 8. Daily evolution of spray-dried powder moisture content on implementing the automatic control



Figure 9. Weekly evolution of spray-dried powder moisture content on implementing the automatic control loop.

6. CONCLUSIONS

The following conclusions may be drawn from the study:

- 1. The fluctuations in the values of the operating variables, before performing the study, indicated a great lack of uniformity in moisture content of the produced spray-dried powder, at the facility studied. This fact is, of its own accord, capable of giving rise to problems of dimensional stability in ceramic tiles, impairing their quality.
- 2. The temperature of the drying gases entering the spray dryer and the flow rate of the aqueous suspension of clay raw materials, are the most suitable operating variables for controlling moisture content of the agglomerate powder obtained in the drying facility. This is due to the

ease of measurement and particularly, to the simplicity and accuracy with which these variables can be modified.

- 3. Empirical correlations of a linear kind were found between incoming drying gas temperature and moisture content of the produced spray-dried powder for each of the slurry flow rates tested, while holding the physical properties of the suspension and the other operating variables of the drying facility.
- 4. The empirical correlation obtained, which is valid for the industrial facility studied and the interval of operating conditions tested, allows implementing an automatic control system for the moisture content of the produced powder agglomerate.
- 5. The greater homogeneity achieved in agglomerate powder moisture content assures the disappearance of the problems of dimensional stability associated with this variable, thus enhancing final quality of ceramic tiles.

7. NOMENCLATURE

G₁: Mass flow rate of a gaseous stream (kg/s)

- P : Average dynamic pressure of the gaseous stream (N/m^2)
- Q : Volume flow rate (m^3/h)
- r²: Coefficient of determination
- T₁: Incoming drying gas temperature (°C)
- T₂: Dryer exiting gas temperature (°C)
- X₄: Moisture content of the produced solid (kg water/kg dry solid)

 $-\Delta P$: Decrease in pressure in the drying chamber

- ρ : Density (kg/m³)
- μ : Viscosity (Dp)
- p: Predicted by the fitted equation
- s : Dry solid
- w : Water

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