

ANALYSIS OF THE EFFECT OF PROCESS PARAMETERS ON THE AVERAGE AND VARIABILITY OF CERAMIC TILE DRY BULK DENSITY

**^(1,3) D. Santos Barbosa, ⁽²⁾ B. Vicente, ⁽³⁾ D. Hotza,
^(3,4) R. A. F. Machado**

⁽¹⁾ Universidad Nacional de Colombia, Chemical Engineering Department
Bogotá, Colombia

⁽²⁾ Serviço Nacional de Aprendizagem Industrial de Santa Catarina
SENAI/SC, Tijucas, SC, Brazil

⁽³⁾ Universidade Federal de Santa Catarina, PGMAT/UFSC
Florianópolis, SC, Brazil

⁽⁴⁾ Universidade Federal de Santa Catarina, CPGENQ/UFSC
Florianópolis, SC, Brazil

ABSTRACT

In this study, statistics were applied to analyse the effect of process parameters on the average and variability of dry bulk density of ceramic tile bodies of the porcelain tile type, on an industrial level. Additionally, it was evaluated how the control variables need to be altered in order to establish the ideal combinations between these variables with a view to obtaining better quality products. The experimental design considered three factors: P pressing pressure, dryer temperature, and dryer roller velocity. Each factor was studied at two levels and a complete 2^3 factorial design was selected. In addition to the classic statistics application, an analysis was performed of the parameters on the average and the variability of dry bulk density, to establish the robustness of the drying operation in the ceramic tile production line.

1. INTRODUCTION

The industrial process analysed involves the manufacture of type BI-a porcelain tile by the wet method, with discontinuous milling, single firing, and forming by pressure. Figure 1 presents the sequential stages of this production process. Only the variables related to the pressing and drying stages have been specified, since these are the focus of this study. The complete information on the variables related to the other stages may be consulted in Santos [5].

Bulk density is used to express the degree of compaction [1] and determines some of the most important characteristics of the end product [2], enabling certain defects to occur, such as the following [3]:

- Structural defects, such as black core, cracking, or inappropriate porosity
- Surface defects, such as small depressions, bubbles, and holes
- Dimensional defects, such as lens shapes and wedging.

Another study [4] has addressed the influence of tile bulk density on subsequent process stages, which have a determining effect on finished product quality. These influences are schematically illustrated in figure 2.

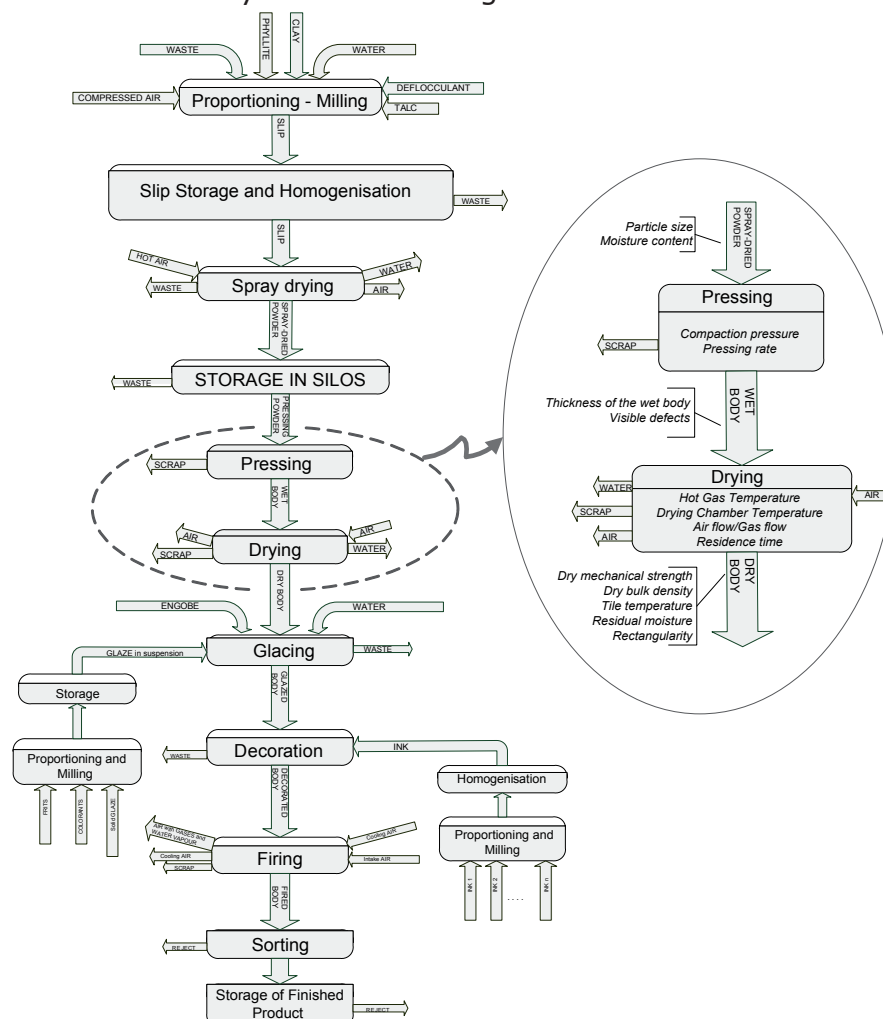


Figure 1. Material flow chart of the porcelain tile production process: Pressing and drying stages.

In the analysis performed of the ceramic tile manufacturing process of an industrial production line, from a control standpoint [5], the controlled variable was identified, how it was measured, and what action was performed when the variable involved did not behave as expected. Dry bulk density is generally not controlled despite being a very important variable, since (as noted above) it affects the aesthetic and functional characteristics of the end product. Additionally it was established that:

- Bulk density uniformity must be assured in the entire piece, in the different bodies that leave the same cavity, and between the pieces located in different cavities.
- It is very important to perform a comprehensive analysis of the process based on a detailed study by stages.
- The variables of interest can be controlled by managing variables in the same stage or by acting upon foregoing stages. The integration of the process cannot be achieved by just performing serial control strategies.

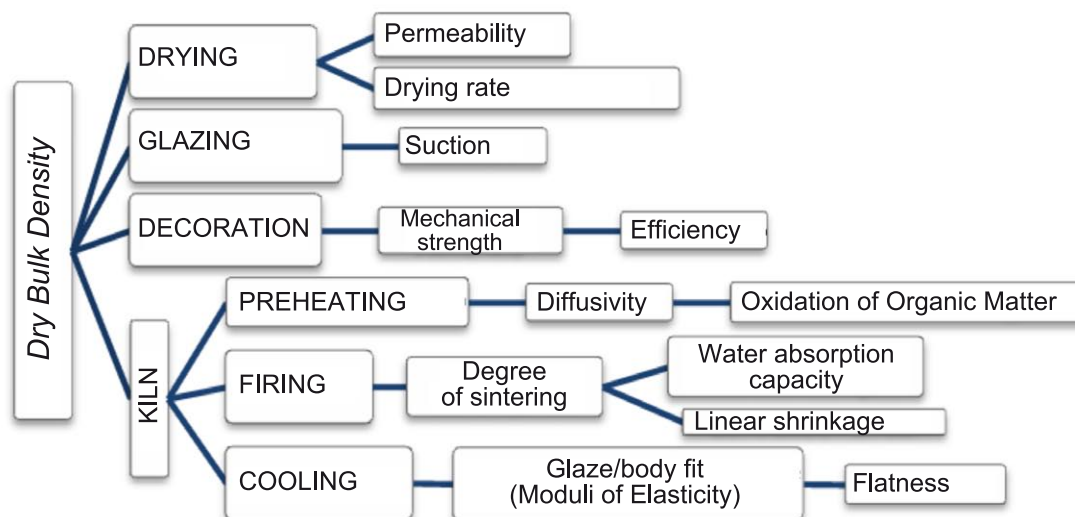


Figure 2. Effect of bulk density in the process stages¹.

The first step in determining a possible course of action for dry bulk density control is to analyse the factors that affect bulk density.

The relationships between the pressing variables and the characteristics of the green and fired tiles obtained have been studied in various papers. For the analysis of dry bulk density, these studies specifically focus on the following:

- They examine the influence of particle size distribution of the spray-dried powder, powder packing density, and the speed of the press feed carriage [6] [7].
- They establish relationships with specific positions, rates, and pressures of the hydraulic press operation [8].

1. Adapted from Blasco [4], p.326.

- They focus on the relationships between spray-dried powder moisture content and pressing pressure in regard to bulk density [1] [7] [9] [10]. In these studies, among other considerations, it is assumed that at a given compaction pressure, there is a linear relationship between dry compaction and powder moisture content, except for high pressures and moisture contents.

Most efforts have focused on the implementation in-line of a dry bulk density sensor, which is continuous, automatic, environmentally safe and has the required precision to be used in an automatic control loop. Studies have been developed to measure bulk density by ultrasounds [11] [12], radiation [13], strain sensors [4], and through the measurement of moisture content. The existing automatic bulk density control strategies are based on final compaction depending almost exclusively on pressing pressure and material moisture content, only addressing variables in the pressing stage [14] [2].

The studies cited on the relationships between pressing variables and tile characteristics have been conducted under controlled conditions with laboratory instruments. The difficulty is evident when it is sought to perform this type of analysis on an industrial level. There are constraints, imposed by the production needs of industrial plants, on acting on certain parameters and on obtaining significant differences in the response variables. Some of these constraints are as follows:

- The levels of variation in the factors cannot always be sufficiently altered to obtain a measurable and significant response, without altering the characteristics of the end product. In order to achieve this, some changes in the pressing and drying stages must be offset by others in later stages in order to hold the final properties.
- The time during which the variables can remain at a different level from that of standard running conditions is sometimes insufficient to obtain good process stabilisation. As a result, the expected changes may be masked.

The studies have focused on the pressing stage and no studies were found that analysed the influence of drying operation variables on dry bulk density. The drying and firing stages are together responsible for 95,5% of total energy consumption [15] [16]; an analysis has therefore initially been made of the drying operation in order subsequently to develop a methodology applicable to the firing stage.

The statistical design of experiments method has been used in the present work to analyse, on an industrial level, the effects of different factors on the key properties of ceramic bodies in the pressing and drying stages. In this first phase, the effect of factors such as dryer temperature, compaction pressure, and residence time in the dryer on dry bulk density is analysed. In addition to contributing important information for a better understanding of process performance, robust control strategies can be suggested based on statistical analysis of the factors on the average and the variations of ceramic tile properties after the drying stage.

An initial analysis was performed with a classic approach and the Dr. Genichi Taguchi concept of robustness was then used to determine the levels of the factors with which the maximum average bulk density could be obtained and the variability could be reduced. The methodology proposal by Taguchi [18] addresses the elimination of the negative effects generated by noise factors, without raising production costs, by conducting experimental studies that allowed appropriate levels to be found of the controllable process factors, which minimise the variance, while keeping the average at its nominal value.

The ultimate objective of the project is to determine the critical parameters for the implementation of a control and automation technology for the ceramic tile manufacturing process, in which the first phase focuses on the drying process.

2. MATERIAL AND EQUIPMENT

In order to perform the analysis under conditions close to those found on an industrial level, the experimental work was conducted at the model ceramic tile production plant of SENAI/SC (*Serviço Nacional de Aprendizagem Industrial*) located in Tijucas, Santa Catarina, Brazil. The approximate daily production in that plant is 1000 m² porcelain tile, size 10x10 cm, and the production line is continuous from the pressing stage.

The material was formed by uniaxial pressing differentiated with a double effect. A 530-ton hydraulic press and pressing line with six cavities were used. The pressing pressure, P , was calculated from the press pressure gauge reading.

The drying operation was conducted in a horizontal roller dryer (1,6 m wide and 8,4 m long) by means of hot air circulation. The natural gas combustion at the burner was used for heat transfer. A schematic illustration of the dryer is shown in figure 3. Dryer temperature, T , was measured with a thermocouple (Thermocouple 1 in figure 3), located in the dryer chamber. Roller velocity, V , is an indirect measurement of tile residence time in the dryer and it was read in Hertz. Bulk density was measured by the Archimedes method.

3. FACTORIAL DESIGN

In order to analyse the effect of certain variables (factors) on the properties of the ceramic tiles in the drying stage, two complete 2³ factorial designs were prepared in the SENAI/SC plant.

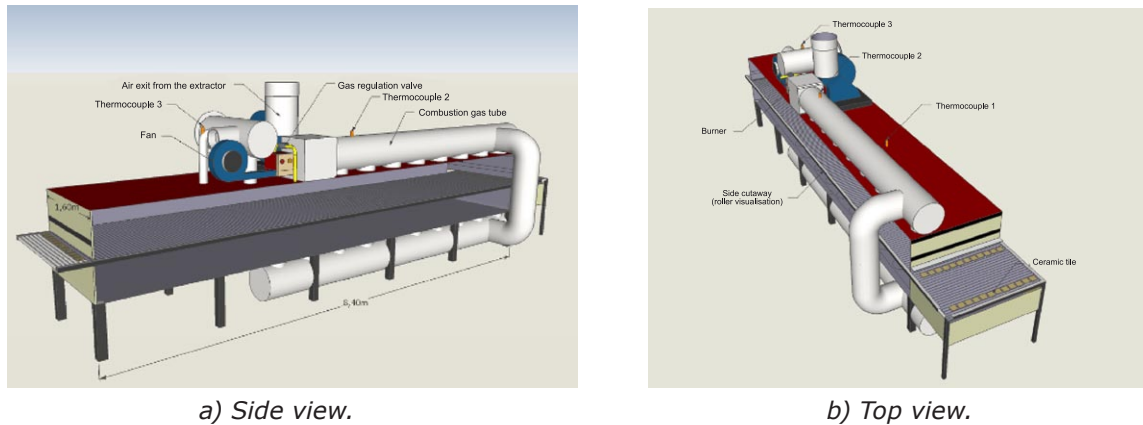


Figure 3. Scheme of the dryer used.

In the present work, the 2^3 experimental design was analysed at constant moisture content to determine the most advisable operating condition, on an industrial level, between the low and high levels of the factors, in order to obtain a bulk density of at least 1.95 g/cm^3 . This value was chosen as an ideal minimum. The operating conditions of this experiment (*) were set in the isocompaction diagram (constant density) shown in figure 4 and it was observed that the bulk density values for the operating conditions were about 1.95 g/cm^3 , the selected ideal minimum value.

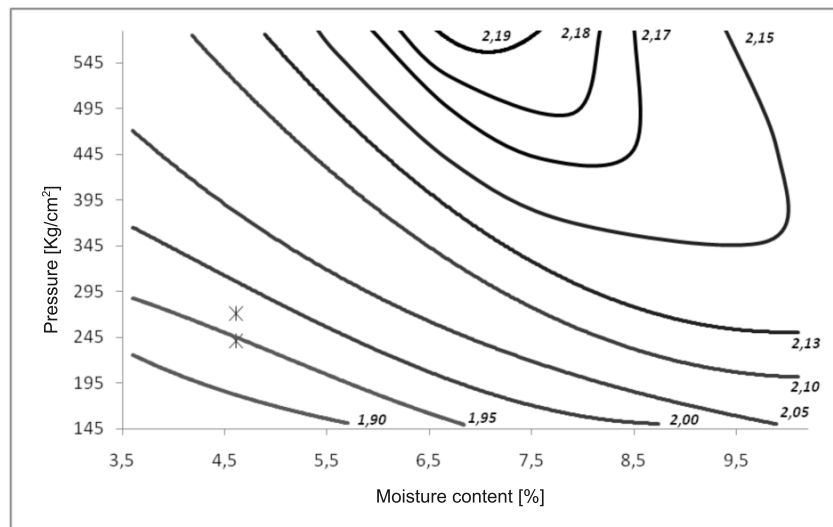


Figure 4. Isocompaction diagram² with operating conditions.

Based on the knowledge of the ceramic tile production process, and the explanation given above, the bulk density at the dryer exit was selected as the response variable, D_{ap} [g/cm^3], and the following were selected as factors:

- **V**: Roller velocity [Hertz]. This represents the residence time in the dryer.
- **T**: Temperature in the chamber [$^{\circ}\text{C}$]. This could be changed by adjusting the corresponding fuel flow valve (see figure 4).

2. This figure was adapted from the isocompaction curves for pressed and dry ceramic tiles of a given composition obtained by Navarro^[1], p.271-274.

- **P**: Pressing pressure [kgf/cm²].

Two levels of variation were chosen for each of the three factors (see table 1), thus yielding a 2^3 experimental design. The combinations of that design are given in table 2 as a design matrix and are presented in figure 5 as a geometric view. In order to take into account the measurement uncertainties and to obtain an estimation of the experimental error, two replicates were defined for each test.

FACTORS	Low level (-)	High level (+)
T [°C]	165	255
P [kgf/cm ²]	241	271
V [Hz]	36	44

Table 1. Values of the factors.

#exp	T [°C]	P [kgf/cm ²]	V [Hz]	label
1	-	-	-	1
2	+	-	-	t
3	-	+	-	p
4	+	+	-	tp
5	-	-	+	v
6	+	-	+	tv
7	-	+	+	pv
8	+	+	+	tpv

Table 2. Experimental design 2^3 . Design Matrix.

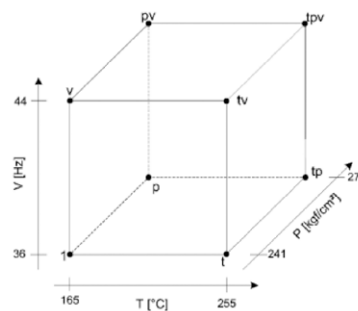


Figure 5. 2^3 experimental design. Geometric view.

Owing to operating restrictions in the plant during the experiments, it was difficult to fit the factors, **P** and **T**, at the values of the desired levels. An average value was used (shown in table 1) and to verify that the scatter towards the average value was not high, the coefficients of variation were calculated from:

$$Cv = \frac{s}{\text{average}} * 100$$

Equation 1.

Table 3 details the minimum and maximum values of each range, the **average**, the standard deviation (**s**), and the coefficient of variation (**Cv**). The results show values smaller than 4% for **P** and **T**.

$$Cv = \frac{s}{average} * 100$$

Equation 2.

		Min	Max	average	s	Cv [%]
T [°C]	—	147	176	165	5.83	3.54
	+	248	266	255	5.83	2.29
P [kgf/cm ²]	—	226	251	241	8.41	3.48
	+	268	276	271	4.33	1.6
H[%]		4.41	4.86	4.61	0.19	4.19
Particle size [%]		61.41	78.98	70.21	4.67	6.66

Table 3. Coefficient of variation of factors and parameters.

The spray-dried powder moisture content, **H**, and particle size are considered to be parameters, since they were factors that remained constant for the experiment. On analysing the coefficients of variation for these parameters, it is concluded that their distributions have a low variability since they have coefficients of variation smaller than 7% (see table 3). Particle size analysis was performed with the oversize on mesh #65 (212 µm opening), which was considered the predominant particle size fraction.

The experiments were randomly conducted, as far as plant working conditions allowed. However, the replicates performed were genuine since they were not repetitions of the test measurements, though they did reproduce the same measurement methods and procedures.

4. ANALYSIS OF VARIANCE

In order to analyse whether the effect of the factors and their interactions on dry bulk density were statistically significant, a variance analysis (ANOVA) was carried out, using the F-test. The MINITAB statistical program was used as calculation tool for the variance analysis. The F-test is based on the comparison of the variances owing to the factors and their interactions with pure error.

Taking into account the constraints of continuous operation at industrial level, to calculate the pure error different numbers of replicates were made at different points in the experimental space, the pure error being the overall average variance of these replicates. The pure error is the variation obtained when genuine replicates are made and it is calculated from:

$$e_{pure} = \frac{\sum S_i^2 GL_i}{\sum GL_i}$$

Equation 3.

where S_i^2 is the variance of the replicates at the experimental point i and GL_i is the number of degrees of freedom of the point for the calculation of the experiment variance i .

In this analysis, the order-three interactions were not taken into account because, in general, the interactions between three or more variables were negligible because the main effects and the interactions of a lower order predominated [17].

5. RESULTS AND DISCUSSION

The result of the F-test for variance analysis is given in table 4.

D_{ap}					F_{TAB} 0.01;1.7 = 12.25	F_{TAB} 0.025;1.7 = 8.07	F_{TAB} 0.05;1.7 = 5.59	F_{TAB} 0.1;1.7 = 3.59	F_{TAB} 0.25;1.7 = 1.57
Source	GL	Sum of Squares	Mean square	F_c					
T	1	0.00032	0.00032	1.91					
P	1	0.00191	0.00191	11.55					
V	1	0.00004	0.00004	0.25					
T*P	1	0.00076	0.00076	4.58					
T*V	1	0.00014	0.00014	0.84					
P*V	1	0.00207	0.00207	12.49					
RESIDUES	8	0.0012	0.0002		FC > FTAB				
Lack of fit	1	0.00004	0.00004						
Pure error	7	0.00116	0.00017						
Total	14	0.00643							

Table 4. Analysis of variance for dry bulk density as a function of dryer temperature (**T**), compaction pressure (**P**), and roller velocity (**V**).

where F_c is the value of F calculated with relation to the variances and F_{TAB} is value of F obtained³ of the F distribution, taking into account the degrees of freedom involved and the type I statistical error.

From the ANOVA of Table 4, the following may be concluded:

- The variables that are statistically significant are pressure, **P**, and temperature, **T**, with a level of significance of 2,5% and 25% error, respectively.
- The interactions **P*V** and **T*V** are significant with 1% and 10% error, respectively, and if we take into account the hierarchy principle⁴, it needs to be considered that there is an effect of the factor **V** on D_{ap} , and the roller velocity, **V**, must be included in the model.

3. The value of F was obtained from Montgomery [17], Table IV of the appendix, p 656-660.

4. The hierarchy principle indicates that if a model has a high order term (such as A^2B) this must also have all its constituent terms of lower order (in this case A^2 and AB) [17].

5.1. Fit of the model.

In order to verify whether there is a fit or not to the linear model, the assumption was made that the sum of the squares of the lack of fit (SS_{lf}) was equal to the sum of the squares owing to the pure error (SS_{pe}).

If $SS_{lf} > SS_{pe}$ then there is lack of fit.

Since $SS_{lf} = 3.71 \times 10^{-5}$ and $SS_{pe} = 1.6552 \times 10^{-4}$ then it is obeyed that $SS_{lf} < SS_{pe}$.

In addition, we calculated the value of F for the pure error: $F_c = 0.22$, and since $F_{TAB} = 1.57$ for 25% error, then $F_c < F_{TAB}$. If $F_c < F_{TAB}$ with 25% error, then $F_c < F_{TAB}$ for any smaller percentage of error.

In view of the above, and since there is no evidence of lack of fit of the model, the model derived from the experimental design may therefore be considered to be linear.

5.2. Graphic analysis of the average and of the scatter.

Applying the Taguchi concept of robustness[18], the levels were found of the factors with which the maximum value of the bulk density average could be obtained and, in turn, a minimum variability could be obtained.

Figures 6, 7, and 8 shows the response graphs (**average of D_{ap}**) versus one of the factors for the two levels of the other factor. In addition, the scatter is included in each of the levels for the two factors.

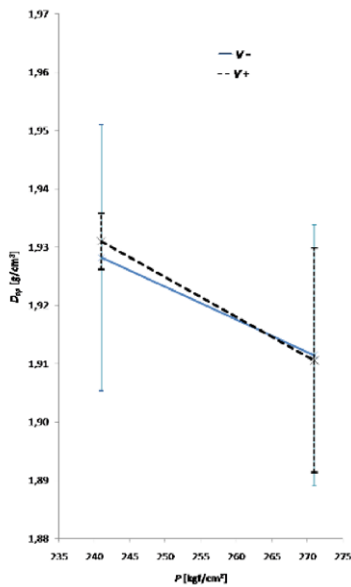


Figure 6. Graph of Interaction $P*V$.

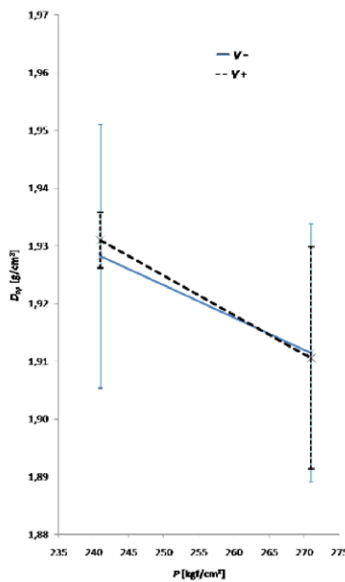


Figure 7. Graph of Interaction $P*T$.

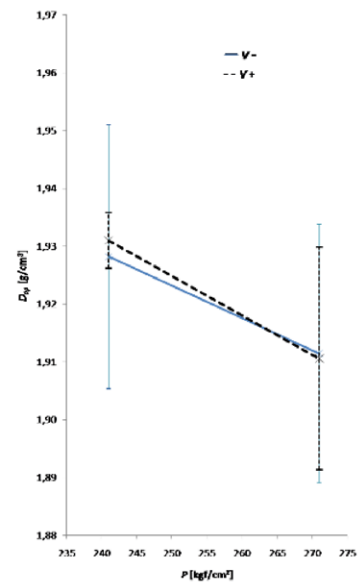


Figure 8. Graph of Interaction $V*P$.

Figure 6 shows that the effect of V is very small for the two levels of P , compared with the effect of P on average bulk density, D_{ap} . Figure 7 displays the effects of P and T , which are in comparison larger than those of V . It may therefore be inferred that the factors that most affect the **average** are compaction pressure and dryer temperature.

On analysing the values of the average D_{ap} , when going from the low level to the high level, it is found that under the conditions of the low levels of T and P , the highest bulk density is obtained with an average value of 1,95 g/cm³. That condition is economically desirable because it is an operation point with less energy consumption in both pressing and drying.

However, when the variability under that condition was analysed (figure 7), an undesirably high scatter was found, with a standard deviation of 0,04 g/cm³.

When a compromise is sought between the greater effect on the average and the smaller data scatter, it may be observed (see figures 7 and 8) that at low levels of pressure and temperature (T - and P -) the scatter decreases notably when working at a high level of velocity, V +

We corroborated the inclusion of V in the model by the high level of statistical significance of the interaction $T*V$ in ANOVA.

6. CONCLUSIONS

Despite the constraints of conducting an experiment on an industrial level, it was possible to use the tools of statistical design of experiments with positive results.

The combination of (classic and Taguchi) statistical tools was critical to reaching a practical conclusion: It may be stated that the maximum average value of bulk density with the smallest variability was obtained when working under operating conditions that corresponded to a high level of roller velocity, and low levels of compaction pressure and dryer temperature. This value corresponded to 1.95 g/cm³. Since it was verified that there was no lack of fit of the linear model deriving from the experimental design, that condition may be considered to be optimum.

With the results obtained it is possible to include other properties of the drying stage in the analysis, and to consider spray-dried powder moisture content as an analysis factor, with a view to proposing robust control strategies applied, in principle, to the drying stage in order subsequently to extend this approach to other ceramic tile manufacturing process stages.

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