# STUDY OF THE FACTORS INFLUENCING THE APPEARANCE OF DIFFERENTIAL DEFORMATIONS IN CERAMIC TILE MANUFACTURE

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## **ABSTRACT:**

This work sets out to analyze the variables of influence in the presence of **differential deformations** *between* tiles subject to the same process and even *within* the same tile.

There have already been studies of certain factors that have an influence on ceramic deformations but these were always done on a laboratory scale with standard individual test specimens. Industrial tile manufacture is a matter of *mass production*, which means a continuous sequence of a great number of tiles (up to 100 per minute) so that individual behaviour, though a determining factor, undergoes the statistical variability corresponding to series on this level. Furthermore, apart from this variability, influential factors cannot be considered statically or unidirectionally, since ceramic tiles display the normal variations involved in any process, and what is more, the variables are not constant for all the tiles or even throughout a single tile.

A result of all this is that mass produced tiles have *differential deformations*, which means these are different to each other and even within the same tile. There are differences

-between tiles fired at the sides and centre of the kiln. -between the front and back of a tile -between parallel tile sides.

These differential deformations cannot be explained only in the light of the aforementioned studies and a more detailed study is required, both from a statistical standpoint and as regards new influential factors.

This work aims to determine the factors influencing differential deformation, and so systematize their control, always from the standpoint of mass production, that is, in ordinary production conditions, which are clearly the real, definitive laboratory.

The factors to be studied were the ones producing such deformation in terms of materials and processes. To be specific, these were:

Materials: -Body -Glaze +Engobes +Bases Processes: -Compaction

-Thickness ratios. Quantities -Drying and glazing -Firing

The materials involved in this study were floor and wall tiles and their corresponding glazes and production processes.

The processes were in all cases the same industrial processes.

On this basis we can affirm that both the materials and the processes are the ones normally used in the industry, since the «laboratory» was an INDUSTRIAL PLANT, and **statistical criteria** were applied in order to analyze the product as a *MASS PRODUCED PRODUCT* and to draw conclusions that are fully applicable to particular cases of individual products.

The study has a further conclusion, bearing in mind the components of the multidisciplinary team, which is to propose specific solutions from diverse angles: Materials and Processes. There is also a proposal for manufacturing control in order to monitor the *differential deformation phenomenon*, so commonly found in present-day production.

The study consists of a first part in which the **variability** of the production parameters taken as statistical series is determined. Thereafter these parameters are placed under **statistical control.** Here we are talking of "**ON-LINE**" control.

We branch off into two aspects from the analysis of the factors found and their variability: one consists of reducing the variability of the ones intervening in the phenomenon being studied and the other is to determine the influence, prioritizing and interrelating these factors in the appearance of the phenomenon (experiment design). This is "OFF-LINE" control.

With an approach like this, one can decide whether the parameters causing deformations can be acted on or not, and also quantify their effect. This is always done from the variability angle and for this reason from the presence of **differential deformations**.



The following figure represents the general work scheme:

## **INTRODUCTION:**

Over the last fifteen years the single-firing process has gradually but totally taken over in the Spanish Floor and Wall Tile Industry (Fig.1), though we could extend this affirmation to world-wide production, which nowadays predominantly uses this process.

FIGURE 1	
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	THOUSANDS OF SQUARE METRES PER DAY						
YEAR	TWICE-FIRED TILE	STONEWARE TILE	MONPOROSA TILE	TOTAL			
1980	250000	15000	0	265000			
1982	330000	60000	0	390000			
1984	300000	110000	3000	413000			
1986	230000	170000	20000	420000			
1988	120000	220000	70000	410000			
1990	110000	300000	110000	520000			
1992	40000	330000	220000	590000			
1994	30000	370000	270000	670000			





During this period of time, another phenomenon found was that of a constant increase in sizes, both in floor and wall tiles (Fig.2), though in this aspect there are differences between producing countries and this increase has been more spectacular in Spain than in other countries. In any event, it is a process that is taking place at different rates in all countries producing this ware. Furthermore, this growth, mainly in the case of wall tiles, has been accompanied by a gradual abandoning of square formats for rectangular ones, with greater or smaller diagonal-side ratios.

#### FIG.2

YEAR	WALL TILES					FLOC	OR TILE	ES			
	15x15	15x20	>20x20	>20x30	>25x40	20x20	20x30	>30x30	>40x40	>45x45	>50x50
1960	80%	15%	5%								
1970	60%	30%	10%			100%					
1975	50%	35%	10%	5%		80%	20%				
1980	40%	40%	10%	10%		50%	30%	20%			
1984	30%	40%	10%	20%		30%	30%	30%	10%		
1988	15%	30%	15%	30%	10%	5%	15%	30%	25%	20%	5%
1990	5%	15%	25%	35%	20%	5%	5%	30%	30%	25%	5%
1992	5%	10%	20%	40%	25%	5%	5%	25%	25%	30%	10%
1994	5%	5%	15%	40%	35%	5%		20%	30%	30%	10%

#### **EVOLUTION IN SIZES**

A third factor which we can observe in this evolution throughout the aforementioned period has to do with the production capacity of the roller kilns used in the process. The industry has gone from using kilns with an approximate production of 1500 square metres per day to one of 2000, 2500, 3000 with a jump to 5000 and even 6000 square metres a day (Fig.3). A process like this has meant the constant redesign of the kilns and their sizes, both in length and in width (Fig.4), as well as the whole conveying system and particularly the combustion, insulation and internal temperature distribution.

If we add to these factors the widespread implementation of single firing for wall tile manufacture (with all the consequences involved in the greater amount of water used with the ware) and a greater number of applications to the unfired body, we come to the referential structure of the issues raised in this work:

-Increase of the surface area to be pressed:

Possibility of greater non-uniformity in compactionPossibility of greater non-uniformity in thickness distribution

-Greater quantity of water used with the tile:

-The tile becomes more brittle

-Greater difficulty in handling these materials

-Larger kilns sizes: -Greater variations in temperature

Furthermore, the manufacture of larger sizes in larger kilns and the increasing demand for quality of the finished product (whether this is as regards its technical characteristics, its aesthetic features or finish) brings a new factor into play, which is "continuity" or "uniformity" in production and for which a determining factor is the uniformity of the raw materials used. Though the process is the same, there can indeed be no doubt that the greater internal demands made (due to the new characteristics of the product during the process) and external demands (greater requirement for uniform product quality) have made it necessary to obtain a consistency in raw materials which formerly was not such a determining factor.

We can conclude from all this that **variability** is one of the main factors in the production process and materials involved and thus something that needs to be studied in order to go on developing tile products of a similar or higher quality to those that were made only ten or fifteen years ago.

**Variability** or the natural scatter in the process is a basic part of the statistics of probabilities and it is necessary to grasp this in order to facilitate the evolution of relationships throughout the whole manufacturing facility.

EVOLUTION OF SINGLE-FIRING KILN CAPACITY				
YEAR	M2 / DAY			
1975	1800			
1980	2000			
1984	2500			
1988	3000			
1990	3500			
1992	4000			
1994	6000			

FIG. 3

FIG. 4

<b>EVOLUTION OF SINGLE-FIRING KILN SIZE</b>						
YEAR	LENGTH	ENTRANCE				
1975	50	0.8				
1980	60	1				
1984	75	1.50				
1988	90	1.80				
1990	90	2.2				
1992	90	2.5				
1994	110	2.5				

When a foreman or operator takes a measurement and finds this to lie far from the desired parameter, though within the standard (as could be the case of size measurement, curvature, water absorption, bulk density, thickness, bending strength, grams of glaze, tile temperature etc.) he should not jump to conclusions there and then, but should take further measurements even though the time available is scarce. It would be the same as drawing conclusions on a batch based on measurements taken of one sole piece and yet this is nevertheless quite often done, to the extent that action is taken on the process, or against the operator who allowed such variation, without knowing if this was due to the normal variability of the process or one of its components (the four Ms: Method, Machinery, Materials and Manpower). One should also ask if this deviation is real, statistically speaking.

When the size and the scatter connected with any yield are principles shared by all those working in a production plant, a number of a priori attitudes can be ruled out. A common language is set up which enables greater cooperation between production, methods, quality, development, as well allowing better communication throughout the whole hierarchical system.

The measurement of Capacity Indices enables an instant evaluation of the process yield and also of the overall performance over a longer period to be obtained. These indices have a bridge function between process control and its capacity for respecting the external or internal customer's demands. They act as «feed-back» indicators on the level of ongoing efficiency improvement. After carrying out measurement of process performance and yield, its degree of conformity with the customer's demands can be assessed. The statistical tool allows modelling performance by means of a law of statistical distribution, since each measurement taken is associated with an obtainment probability. Thanks to this statistical model, the percentage of non-conforming products can be evaluated, but one can also define the central position of production and the higher or lower convergence of tiles produced around this central value.

It should be remembered that the whole control process should be based on **prevention** criteria, since controls run only to separate the good products from the bad ones do not have any effect on their quality. **Prevention** is a set of measures taken to prevent a flaw or defect appearing and making the products worthless. It is clear that the improvement of quality has to entail defect prevention. Setting up such a prevention process involves two stages:

# -Preventing a defect from reappearing. -Preventing the causes that can give rise to a defect from appearing.

In the first of these cases one has to find the cause of the problems and identify the signs indicating the appearance of this cause. In the second case, there has to be evolution from a check made on a product's characteristics to a check on process parameters. We can thus say that prevention is based on:

The establishment of clearly formulated requirements and standards
A well defined process
Validation of the process
Executing and controlling the process
Laying down guidelines, methods and systems for controlling and corrective action.

In the ceramic production process, a number of variables are subject to checking (moisture content of spray-dried powder, bulk density, pressing pressure, green mechanical strength, drying temperature, residual moisture, tile temperature, grams of glaze, firing temperature, sizes, curvature, and so on) which do not give direct information to the operator on the performance of the process. But to be able to prevent faults, which means being able to act before this tile becomes defective, the signs that announce the presence of the defect have to be recognized. Normally the detection of these signs is something left to sheer experience, but this is highly subjective and unquantifiable. The use of statistical tools is the right resource for detecting such signs.

The role of **statistics** cannot be reduced to one of a tool for use by specialists at universities and research centres. The eighties bore witness to a profound change in approaches to quality and productivity in all sectors and perhaps the most striking result of these new approaches is the central role given by them to the systematic use of statistics on all levels in enterprise. Tile manufacturers cannot be the exception and must incorporate and extend statistical concepts as well as implementing the new concept of Quality. It has been many years since Deming himself stated that statistics was a little-used resource in enterprise and that on the other hand there is no field of knowledge that can help so much to improve quality, productivity and competitiveness. But it should furthermore be a field of knowledge spread across all levels, as Ishikawa pointed out over twenty years ago, when he said that basic statistical tools should be known and used by everyone in a company, from top management to line workers.

It is clear that we are only at the outset of the introduction of statistical techniques in the tile branch, and that their importance will rise as their use increases. One should remember that the basic responsibility of a technician is to show the way for the continuous improvement in quality, and productivity in all the processes that depend on him. But to improve processes, these have to be changed and modified, and these changes, if they are to be rational, can only be the fruit of data analysis, and not of impulsive decisions. Two basic needs arise from this fact:

- The need to generate data containing relevant information
- Having a method to draw information from such data and being able to act in consequence.

The use of statistics is the response to both these requirements as it enables us to define the data to be collected as well as to analyze these and draw the necessary information from the outcomes. The time of statistics has arrived, in spite of having been ignored and even disdained and attacked by people and groups of researchers of recognized prestige. But truth is stubborn and even though this is based on tools such as SPC, Experiment Design and Taguchi Method becoming fashionable, it is quite true that statistics is becoming increasingly important in our daily ceramic work.

## **EXPERIMENTAL PROCEDURE:**

The first part of this work addressed the variability of the parameters studied in normal working conditions. For this purpose the relevant *Capacity studies* were carried out, both for material and process variables:

-Spray-dried powder: - Standard spray-dried powder: -Oversize at 40 microns -Carbonate content -Shrinkage -Glazes: -M1, M2, M3 Glazes: -Coefficient of expansion -Quantity applied -Pressing: -Standard presses: -Bulk density -Thickness -Standard glazing facilities: -Firing: -Standard kilns: (2.2x100 metres) - Temperature Distribution

Table-I gives the details of the materials and machinery used.

LIST OF MATERIALS AND FACILITIES USED				
PH 2000 / PH 2500 PRI	ESS			
INDUSTRIAL SPRAY-DRI	ED POWDER			
GLAZES M1	SEMITRANSPARENT GLOSSY			
M2 OPAQUE GLOSSY				
M3 TRANSPARENT GLOSSY				
INDUSTRIAL GLAZING F	ACILITY			
KILNS H3	WELKO 60X2.2			
H2	SACMI 86X2.4			
H1	SACMI 90X2.2			
DEFLECTION MEASURE	MENTS: "MOBILE FLATNESS METER"			

### TABLE 1

Values were taken of all the above factors at different intervals and periods, always within the typical production process, in trouble-free conditions. The values were determined by the standard procedures used in factories and described in the literature.

The deviation from flatness or deflection of the finished product was controlled, since at all times this will be the *response variable*. Controls were carried out at different points in the kiln and in the tiles, going by a regular preset sequence, in order to introduce the **variability** concept:

-Deviation: -Each size -Each plate -Each side -Each kiln position -Each firing condition

For the deflection measurement (Concavity (-) and Convexity (+) ) a «mobile flatness meter (R)» was used with a Mitutoyo micrometer (patented apparatus).

The data obtained were used to make the relevant Capacity Study and calculation of Capacity Indices.

After determining the "standard" variability of the parameters considered, as well as establishing some initial relations between the variables studied and the "response", an experiment was designed in order to determine the relations quantitatively and prioritize corrective action. This experiment design was set up in the light of the variability already found in which the glaze was taken as a fixed variable, this being furthermore logical in view of the working conditions.

Taking into account the number of variables considered and the levels, a *Design of Three Factors with two Levels each* (8 tests) was chosen.

Both in Part A and in Part B a computer program for statistical applications (Estatgraphics) was used.

The trials in the experiment design were carried out under industrial conditions, which means that the variables were located in the production process and production took place to obtain the results (always of statistical series), given that at all times the intention was to obtain «real» results for specific and direct application.

## **RESULTS AND DISCUSSION:**

Table 2 shows the average values of the parameters considered in spray-dried powder for stoneware and monoporosa tile, as well as their standard deviation and range (oversize at 40 microns, carbonate content and shrinkage). For the particular case of the oversize and carbonate content Figures 5, 6 and 7 show the distribution of the actual values versus the preset tolerances (for the porous body). Table 3 gives the Capacity Indices for these parameters. It can be seen that the real values display greater variability than what was initially envisaged (Tolerances), though it is true that the tolerances are mainly set in a "arbitrary" manner without having made a capacity study beforehand. For the case we are now concerned with, given the absence of problems caused by this initial variability, tolerances "should be" the ones specified in Table 4 with the corresponding corrected Capacity Indices. This leads us to concluding that the variability of the Carbonate Content exceeds the normal level and that the oversize is excessive. A different case, as will be seen later on for other variables will be the one requiring further study for reducing the variability and thus improving the Capacity Indices.

SPRAY-DRIED POWDER. DATA OBTAINED AND TOLERANCES					
PARAMETER	MONOP.	TOLERAN.	STONEW.	TOLERAN.	
CARBONATE % AVERAGE STD DEVIATION RANGE	14.72 0.916 2.54	13-15	3.86 0.485 1.77	< 4	
SHRINKAGE % AVERAGE STD DEVIATION RANGE	0.66 0.052 0.18	< 1	5.98 0.263 1.17	5.5-6.0	
OVERSIZE (40 MIC) AVERAGE STD DEVIATION RANGE	7.66 1.491 5.18	< 8	7.04 1.407 5.97	< 8	

SPRAY-DRIED POWDER FOR MONOPOROSA TILE				
CAPACITY INDICES	СР			
CARBONATE CONTENT	0.36			
OVERSIZE AT 40 MICRONS	0.89			
FIRING SHRINKAGE	2.56			

# TABLE 3

REAL AND OBTAINED CAPACITY INDICES SPRAY-DRIED POWDER FOR MONOPOROSA TILE						
	TOLERANCES					
CARBONATES	13 % + / - 2	13 % + / - 2	13 % + / - 3			
СР	0.36	0.73	1.09			
OVERSIZE 40 µ	< 8 %	< 8 %	< 8 %			
СР	0.89	0.92	1.12			



FIG. 5. CARBONATES MONOPOROSA TILE %









Table 5 shows the average values and standard deviation for the parameters considered in pressing (bulk density and thickness of the tile) for 41x41 sizes of stoneware and 25x41 of monoporosa tile. Figures 8 and 9 show the distributions of real values as compared with tolerances for the case of 41x41 stoneware. Table 6 lists their Capacity Indices. Similarly to what was stated for the parameters of the spray-dried powder, it can be observed that the tolerances were set with criteria that were rather "arbitrary" resulting in Capacity Indices under 1. Table 7 shows the Tolerances that "should be applied" as a result of the study of normal variability and the consequent Capacity Indices, which again results in excessive variability.







FIG. 9. THICKNESS 41 X 41 MM

GREEN TILE. AVERAGE VALUES OBTAINED AND TOLERANCES						
	25 X 41	TOLERANCE	41 X 41	TOLERANCE		
THICKNESS						
AVERAGE	8.99	9 + / - 0.1	9.8	9.8 + / - 0.1		
STD DEVIATION	0.129		0.101			
RANGE	0.55		0.4			
BULK DENSITY (Dap)						
AVERAGE	2.03	2.01-2.04	2.15	2.14-2.16		
STD DEVIATION	0.013		0.012			
RANGE	0.09		0.06			

PRESSED TILE. 41 X 41 STONEWARE			
CAPACITY INDICES	СР		
BULK DENSITY	0.29		
THICKNESS	0.66		

## TABLE 6

ASSUMED AND REAL TOLERANCES. 41 X 41 STONEWARE					
	ASSUMED	REAL			
BULK DENSITY	2.13 + / - 0.1	2.13 + / - 0.4			
THICKNESS	9.8 + / - 0.1	9.8 + / - 0.35			

Excessive variability can be observed in the case of the distribution of bulk densities in the 41x41 size for stoneware, with a consequently low Capacity Index, compared with the 25x41 size in monoporosa tile. A more detailed study of each of the plates, Fig. 10, 11, 12 shows the farthest shifted value distribution in the case of plate-1 which leads to the greatest average variability. The control of variability should thus be considered in the light of individual tiles, in this case for individual plates, since taking the 41x41 product as an average will lead to results that are wrong or at least not very accurate. When the deflection data for the 41x41 products are given, it will be possible to verify the above affirmation of the need to "individualize" the products, which is furthermore the aim and conclusion of this study.



FIG. 12. 12 BULK DENSITY 41X41P3

In the case of the 25x41 size, as shown in Figures 13, 14, 15, 16, the values are more concentrated for the plates.

The variability found for bulk density (Dap) as well as for the thickness, is taken as what is standard for the process (process under control) and will thus be adopted in later controls, that is products with this variability will be employed for making subsequent deflection tests.



Table 8 shows the average values and standard deviation for the amount of glaze deposited, for each of the three glazes considered. Figures 17, 18 and 19 show the variability of the amount of glaze deposited versus the tolerances, for each glaze. Table 9 reports the values of the Capacity Indices. It can be observed that the quantity of glaze applied has a very low variability, in comparison with the previously studied parameters, although there is also a high level of "arbitrariness", especially for M1 and M3 in the case of tolerances for the amount of glaze applied (the application file). With a variability of +/-5 grams we would be in the domain where shades appear.

GLAZE LAYER. AVERAGE VALUES OBTAINED AND TOLERANCES				
	25 X 41	TOLERANCE	41 X 41	TOLERANCE
M3				
AVERAGE	100.7	100 + / - 3		
STD DEVIATION	1.25			•
RANGE	6			
M2				
AVERAGE	100.5	100 + / - 3		
STD DEVIATION	0.65			
RANGE	2.2			
M1				· · · · · · · · · · · · · · · · · · ·
AVERAGE			99.2	100 + / - 3
STD DEVIATION			1.7	
RANGE			3.6	







FIG. 18. GRAMS OF GLAZE M2



GLAZE. CAPACITY INDICES AT DIFFERENT TOLERANCES				
СР	100 + / - 3	100 + / - 3		
M1	0.586	0.978		
M2	1.541	2.57		
M3	0.798	1.33		

Table 10 and 10 (b) show the average values for the temperatures taken at different points along the length and breadth of the kiln and inside the roller. Figures 20 and 21 depict plots of these values. It can be observed that the variability is low in continuous working conditions, but still above what has traditionally been considered. Furthermore, Table 11 shows temperature variation in time, at one point in the kiln, with the presence of a gap. The variability in the case of the kiln can thus be considered from two standpoints.

-Point variability in a steady state -Average

For the case we are now concerned with, special attention will be given to variability in continuous working conditions, that is, with a full kiln operating.

TEMPERATURE DISTRIBUTION IN KILN H1				
STONEWARE	TILE TOP S	URFACE	INSIDE R	OLLER
POINT	WALL	CENTRE	WALL	CENTRE
1	617	535	550	515
2	780	765	740	650
3	840	810	770	740
4	880	850	835	795
5	900	880	870	838
6	920	905	900	875
7	977	958	1060	975
8	1045	1040	1110	1055
9	1073	1083	1075	1100
10	1105	1110	1100	1125
11	1123	1133	1117	1150
12	1130	1145	1125	1155
13	1130	1122	1115	1145
14	1093	1098	1085	1125
15	1065	990	1078	1115
16	1015	970	1020	1070
17	880	870	930	1020
18	640	590	705	685
19	510	570	585	595
20	325	405	410	430

TEMPERATURE DISTRIBUTION IN KILN H2				
MONOPOROSA	TILE TOP	SURFACE	INSIDE ROLLER	
POINT	WALL	CENTRE	WALL	CENTRE
1	750	720	713	685
2	865	890	825	840
3	890	875	923	923
4	935	935	940	935
5	940	940	950	950
6	965	960	980	970
7	980	970	990	980
8	1010	1000	1065	1055
9	1120	1110	1135	1120
10	1105	1095	1045	1050
11	1015	990	1020	1040
12	880	870	910	930
13	670	650	690	730
14	530	570	585	595
15	360	405	410	470

TABLE 10 (B)

# TEMPERATURES IN STONEWARE KILN TILE TOP SURFACE



POINTS OF THE KILN

FIG. 20



POINTS OF THE KILN

VARIATION OF TEMPERATURE WITH GAP				
3.5 minute g	3.5 minute gap			
MONOPOR	OSA TILE KILN. P	OINT 15. INSIDE ROLLER		
TIME	TEMP	ERATURE		
MINUTES	WALL	CENTRE		
0	1135	1120		
1	1150	1170		
2	1150	1190		
3	1150	1210		
4	1150	1195		
5	1125	1120		
6	1135	1120		

TABLE 11



Table 12 shows the average values and standard deviation of the deflection for the two sizes in standard production conditions, with the variabilities stated above in each of the parameters considered. Figures 22 and 23 show the variability as compared with tolerances for the deflection in the 41x41 stoneware and the 25x41 monoporosa tile.

DEFLECTION. AVERAGE VALUES OBTAINED AND TOLERANCES				
	25 X 41	TOLERANCE	41 X 41	TOLERANCE
DEFLECTION				
AVERAGE	0.93	-0.15 / + 0.3 %	0.42	-0.15 / + 0.3 %
STD DEVIATION	2.81		0.28	
RANGE	59.8		1.41	

TABLE 12



FIG. 22. DEFLECTION (MM) 41X41



FIG. 23. DEFLECTION (MM) 25X41

Table 13 details the Capacity Index for different tolerances of the 25x41 monoporosa tile.

DEFLECTION. AVERAGE VALUES OBTAINED AND TOLERANCES				
M2 AND M3. 25 X 41 MONOPOROSA TILE				
% IN MILLIMETRES ON MEASURED SIDE				
TOLERANCE				
ТОР	+ 0.3 %	+ 0.2 %	+ 0.15 %	
BOTTOM	- 0.15 %	- 0.1 %	- 0.7 %	
CAPACITY INDEX	1.69	1.13	0.83	

## TABLE 13

Bearing in mind what was stated above about differences in the press plates and temperatures in the kiln, that is by introducing variability as a *differential* element, it becomes necessary to *individualize* deflection measurements considering several criteria:

-Press plate -Side of the press plate -Location in the kiln (in its section) -Type of glaze



Figures 24 and 25 (b) show the array used for individualizing the tiles.

#### FIGURE 25

Table 14 and Figs. 26 and 27 show the distribution of the deflection in the side of the tiles parallel to the wall, in different positions inside the kiln, for plate 1 of 25x41 monporosa tile (the 14 points correspond to the tile sides parallel to the kiln wall: 7 tiles x 2 sides). This is shown for the cases of the M2 y M3 glazes. A clear convex deflection distribution across the width of the kiln can be observed so that there is more deflection by the wall and less in the centre, confirming the temperature distribution in this section. The M2 glaze furthermore shows less deflection than the M3, as can be seen from Fig. 28.

DEFLECTION DISTRIBUTION IN THE MONOPOROSA				
TILE KILN. SIDES PARALLEL TO THE WALL				
PLATE 1				
POSITION + SIDE	M3	M2		
12	0,56	0,3		
14	0,5	0,25		
22	0,49	0,04		
24	0,46	0,04		
32	0,43	0,02		
34	0,44	0,02		
42	0,39	0,11		
44	0,38	0,16		
52	0,41	0,13		
54 0,41 0,17				
62	0,42	0,12		
64	0,47	0,18		
72	0,51	0,18		
74	0,58	0,32		







FIG. 27



Deflection is thus a factor subject to variability as a function of the time when this is measured. Figure 29 shows how it develops in time (the time elapsing from the first measurement taken at the kiln entrance) for the 25x41 monoporosa and the 41x41 stoneware tile.

VARIATION OF DEFLECTION IN TIME			
TILE EXIT FR	OM KILN		
HOURS	MONOPOROSA TILE	STONEWARE TILE	
0	0,64	1,17	
2	0,55	1,11	
4	0,48	0,87	
10	0,34	0,67	
24	0,33	0,6	
48	0,35	0,61	



We also show the influence entailed by the variability relative to the front side of the glazed tile that enters, as a result of being the leading side for glaze application and not having been scraped properly. This influence is shown in Tables 15, 15 (b) and 16, where it can be seen that for the case of the 41x41 stoneware, the fact of the edge coming in glazed or clean is a determining factor for deflection. The difference is a deviation of 0.4-0.5 mm. For monoporosa tiles this difference is much less, to the extent of being almost negligible (0.1 mm). The influence implied by the position of the sides in respect of the kiln feeding movement can also be observed: the same side has a different deflection through the simple fact of being the leading edge or the rear one. In the case of stoneware this difference is additive when the edge is glazed. Tables 17 and 18 show the influence of the position of the face (front, rear, parallel). Table 19 details the influence of the position of the tile in respect of the kiln measured in all working conditions. Table 20 shows the influence of the glaze (Typology).

With the data obtained and taking into account the variability detected in the previous operations, a number of conclusions can be drawn:

- There is a clear relationship between deflection and the position of the tile in the kiln.
- This relationship is influenced by temperature.
- There is a relationship between deflection and the type of glaze used.
- There is a relationship between deflection and tile compaction.
- There is a difference in the behaviour of the leading side and the rear one as the tiles advance through the kiln.

<b>DEFLECTION. INFLUENCE OF GLAZED EDGE</b> CENTRE 41 X 41 STONEWARE TILE M1, H1				
FACE				
EDGE	FRONT	REAR		
GLAZED	0.19 -0.25			
UNGLAZED 0.02 0.13				

TABLE 15

DEFLECTION. INFLUENCE OF GLAZED EDGE			
CENTRE 25 X 41 MONOPOROSA TILE M3, M2			
	FACE		
EDGE	FRONT	REAR	
GLAZED	- 0.5	0.01	
UNGLAZED 0.02 0.04			
$\square A D I \square (= (D))$			

TABLE 15 (B)

DEFLECTION. INFLUENCE OF FACE POSITION				
AVERAGE DATA. AL	L CONDITIONS			
	MONOP.	STONEWARE		
FRONT	0.15	0.94		
REAR	0.09	0.94		
PARALLEL (1)	1.25	0.92		
PARALLEL (2)	1.40	0.95		
(1) SIDE CLOSEST TO WALL				
(2) SIDE FARTHEST FROM WALL				
TABLE 16				

DEFLECTION. INFLUENCE OF FACE POSITION					
CENTRAL TILE. ALL CONDITIONS					
MONOPOR. STONEWARE					
FRONT	0.17	0.95			
REAR	0.08	0.95			

DEFLECTION. INFLUENCE OF TILE POSITION					
AVERAGE DATA. ALL CONDITIONS					
	AVERAGE SIDE CENTRE				
MONOPOR.	0.42	0.45	0.38		
STONEWARE 0.86 0.87 0.86					

## TABLE 18

DEFLECTION IN MONOPOROSA TILE.INFLUENCE OF FIRING						
Bulk density (Dap) 1, thickness 1, glaze M 3						
KILN	AVERAGE	SIDE	CENTRE			
H1	0.99	0.98	1.01			
H2	0.70	0.87	0.62			
H3	0.61	0.76	0.46			

#### TABLE 19

DEFLECTION IN MONOPOROSA TILE. INFLUENCE OF GLAZE						
BULK DENSITY (Dap) 1, THICKNESS 1, KILN 3						
GLAZE	AVERAGE SIDE CENTRE					
M1	0.48	0.56	0.32			
M2	0.82	0.88	0.78			
M3	0.78	0.82	0.71			

#### TABLE 20

All the foregoing means that the deformation of tiles, measured by means of deflection, cannot be approached in a general way. The tile's "history" has to be taken into account, and tiles have to be individualized in so far as a number of parameters are concerned, given that these display a variability which prevents and invalidates talking in general terms. Deformation is caused by a number of parameters with variability and it is precisely this variability which makes the tiles have individual behaviour and thus means that possible corrections or corrective action should be individually approached. General corrections would give rise to general modification and some tiles would be affected in a different way to others - i.e. the results would still be non-uniform and involve variability. A twofold approach is required:

- Correcting until variability is minimized or eradicated and introducing general corrections.
- Introducing individualized corrections

Either of these two forms is valid and one could even describe them as being complementary, but in the best of cases variability can be minimized and never completely suppressed. For this reason it is advisable to make progress in the process of individualizing corrective action. This process would involve:

1st.-Reducing variability 2nd.-Individualizing corrective action

Both of these imply in-depth knowledge of the variables associated with the parameters studied, their quantification and improvement. Capacity Studies and Experiment Design will be suitable tools for this process:

1.a.-Centring the process
1.b.-Reducing variability
1.3.-Reducing tolerances
2.a.-Prioritizing and quantifying the cause-effect relations producing deformation
2.b.-Identifying residuary variability and trends
2.c.-Individualizing corrective action:

2.c.1.-Pressing
2.c.2.-Glaze application
2.c.3.-Firing conditions

Within the work scheme described above and as was stated in the EXPERIMENTAL PROCEDURE section an experiment was designed with 8 tests (three factors on two levels each) used for quantifying the cause-effect relationship of the parameters studied in respect of deflection. These were carried out in factory under steady working conditions, taking the variability criteria of the parameters in all cases and identifying the individuality of others.

Table 21 details the experiment design for 25x41 monoporosa tiles (taking the average deformation values of all the tiles and sides). Table 21 (b) gives the specific working conditions and Table 22 the relevant ANOVA. It can be observed that the factors considered (bulk density (Dap), thickness and firing) are significant, the same as the interactions, given that the test F for significance (for a 95% reliability level) gives values under 0.05. To put this in other words it can be stated that the variation of one of the variables considered will affect the deflection. The main effect of a factor (variable studied) is the increase in value of the variable considered (deflection) as it modifies the other three (bulk density, thickness, firing) from level 1 to level 2. For the case of monoporosa tile, and in Figures 30, 31 and 32 the influence of these three factors on deflection is included and in Figures 33, 34, and 35 the additive effects of parameters taken two by two are shown.

EXPERIMENT DESIGN WITH MONOPOROSA TILE						
BULK DENSITY	THICKNESS	FIRING	DEFLECTION			
1	1	1	0.94			
1	1	2	0.43			
1	2	1	0.61			
1	2	2	0.31			
2	1	1	0.74			
2	1	2	0.24			
2	2	1	0.34			
2	2	2	0.12			

PROGRAMM	]		
	1	2	EXTRA
BULK DENSITY	2.08-2.09	2.14-2.15	2.18-2.19
THICKNESS	-0.5	NOMINAL	+0.5
GLAZE	M2	M2	M1 AND M3
FIRING	H3		
M2 : OPAQUE GL			

### **TABLE 21 (B)**

EFFECT	SIGNIFICANCE LEVEL
SIMPLE	
BULK DENSITY	0.05
THICKNESS	0.04
FIRING	0.03
INTERACTIONS	
BULK DENSITY-THICKNESS	0.5
BULK DENSITY - FIRING	0.4
THICKNESS-FIRING	0.08

#### TABLE 22

From the statistical study it can be said that the determining effect is firing and that both bulk density and thickness have significant effects. It is also important to point out that the factors are additive and that none of them can be considered alone due to the influence among them (additive influence). One of the advantages of the experiment design is displayed here since the influence of each factor is considered for the remaining factors on their different levels.

The optimum value of deflection can also be obtained for the parameters studied and their corresponding levels, that is, conditions in which bulk density is at level 2, thickness at level 2 and firing at level 2 (Table 23). One must remember that these values will be the optimum ones for the case studied but they give little indication of the "ideal" value for each one of the factors for minimizing deflection. For this purpose it would be advisable to undertake an experiment in which the factors are presented at three levels and thus enable maximums and minimums to be determined.

EXPECTED OPTIMUM VALUE				
GENERAL AVERAGE	0.47			
BULK DENSITY FACTOR (-)	- 0.19			
THICKNESS FACTOR (-)	- 0.12			
FIRING FACTOR ( - )	- 0.1			
OPTIMUM DEFLECTION VALUE 0.6				

#### TABLE 23

Table 24 gives the dilatometric values for different samples in which compaction, firing conditions and tile position in the kiln vary. Table 25 shows the water absorption for these samples. Tables 26, 27, 28 and 29 detail the variation of peak intensity 4.26A of quartz for the separate different samples to identify the influence of each factor.

In these tables it can be observed that the dilatometric values are not very sensitive (negligible) to changes in bulk density for the same firing conditions. A greater variation is seen in the dilatometric values for variations in the firing conditions, even within the channel for extreme cases of improper temperature distribution. The same can be said of water absorption, which is much more sensitive to variations in firing conditions than to compaction conditions. If we now associate all this with the presence of free silica in the fired product, we can conclude that the silica peak is indeed sensitive to variations in firing conditions in firing in the coefficient of expansion, except in the case of kiln H3 (both in comparison with other kilns and between side and central tiles). In any event, the technique turns out to be rather insensitive for determining deformation on the scale of industrially made tiles, subject to the variability of the process and the materials.



level of FLEPORO.ES by FLEPORO.CO

FIG. 33

FLEPORO.ES

FIG. 34

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FIG.35

VALUE OF (ALPHA X 10 ^ - 7/ k )												
STONEWARE SAMPLES UNDER DIFFERENT CONDITIONS RELATIVE TO: PRESSING, KILNS AND POSITION IN THE KILN												
TEMPERATURE	1H1C	1H1L	2H1C	2H1L	1H2C	1H2L	2H2C	2H2L	1H3C	1H3L	2H3C	2H3L
50		58.89	62.91	61.54	87.4	61.4	58.03	63.48	59.32	56.71	56.76	67.31
100		64.11	66.62	65.51	68.69	66.13	64.9	66.96	64.66	63.62	64.15	68.17
150		66.94	68.64	67.9	69.84	68.83	67.8	68.76	67.53	66.69	67.49	69.38
200		69.09	70.27	69.94	71.37	70.65	69.94	70.32	69.61	69.18	69.8	71.02
250		70.97	71.74	71.55	72.09	71.78	71.21	71.16	71.07	70.81	71.49	72.16
300		72.55	73.11	72.87	72.63	72.91	71.92	71.69	72.45	72.31	72.86	73.22
350		74.24	74.59	74.54	73.68	74.49	73.2	72.7	74.15	74.04	74.52	74.22
400		76.22	76.39	76.52	75.11	76.48	74.48	74.29	75.96	76.01	76.46	76.34
450		78.44	78.57	78.83	77.12	78.65	77.03	76.44	78.22	78.33	78.84	78.45
500		81.33	81.38	81.78	79.91	81.39	79.8	79.25	81.03	81.27	81.79	81.19
550		85.47	85.47	86	83.96	85.41	83.91	82.33	85.08	85.51	86.05	85.15
600		95.65	95.49	96.19	94.53	96	94.69	94.24	95.39	95.96	96.71	95.23
650		92.65	92.36	93.05	91.42	92.84	91.61	91.12	92.31	92.84	93.59	92.07
20-300		72.6	73.1	72.9	72.6	72.9	71.9	71.7	72.9	72.3	72.9	73.2
300-500		93.04	92.8	94	89.9	93.1	90.7	89.7	92.9	93.6	94.1	92.1
500-650		127.9	127	128.6	127.8	128.9	128.9	128.6	127.9	129.4	130.8	126.4

VARIATION IN WATER ABSORPTION				
41 X 41 STONEWARE TILE				
FACTOR	%			
Bulk density 1 H1	4.4			
Bulk density 2 H1	4.6			
Bulk density 1 H2	7.9			
Bulk density 2 H2	9.3			
Bulk density 1 H3	5.0			
Bulk density 2 H3	6.4			
H1	4.5			
H2	8.6			
H3	5.6			
DIF (CENTRE - SIDE) H1	0.1			
DIF (CENTRE - SIDE) H2	0.1			
DIF (CENTRE - SIDE) H3	0.3			
Bulk density 1 : 2.13 - 2.15				
Bulk density 2 : 2.09 - 2.11				
TABLE 25				

VARIATION IN WATER ABSORPTION						
	<u>41 X</u>	41 STONEW	ARE TILE			
Bulk density	Bulk density H POSITION PEAK					
Bulk density 1	H1	CENTRE	-			
		SIDE	410			
Bulk density 1	H2	CENTRE	398			
		SIDE	440			
Bulk density 1	H3	CENTRE	679			
		SIDE	812			
Bulk density 2	H1	CENTRE	453			
		SIDE	417			
Bulk density 2	H2	CENTRE	454			
		SIDE	350			
Bulk density 2	H3	CENTRE	469			
<b>b</b>		SIDE	388			

VARIATION IN WATER ABSORPTION						
	41 X 41 STONEWARE TILE					
Bulk density H POSITION PEAK						
Bulk density 1	H1	SIDE	410			
Bulk density 2	H1	SIDE	417			
Bulk density 1	H2	SIDE	440			
Bulk density 2	H2	SIDE	350			
Bulk density 1	H3	SIDE	812			
Bulk density 2	H3	SIDE	388			

			·				
VARIATION OF QUARTZ 4.26 A PEAK INTENSITY							
XRD OF 41 X 41 STONEWARE TILE. VARIATION IN POSITION							
Bulk density	Н	POSITION	PEAK				
Bulk density 1	H1	SIDE	410				
Bulk density 1	H1	CENTRE					
Bulk density 2	H1	SIDE	417				
Bulk density 2	H1	CENTRE	453				
Bulk density 1	H2	SIDE	440				
Bulk density 1	H2	CENTRE	398				
Bulk density 2	H2	SIDE	350				
Bulk density 2	H2	CENTRE	454				
Bulk density 1	H3	SIDE	812				
Bulk density 1	H3	CENTRE	672				
Bulk density 2	H3	SIDE	388				
Bulk density 2	H3	CENTRE	469				

VARIATION OF QUARTZ 4.26 A PEAK INTENSITY							
XRD OF 41 X 41 STONEWARE TILE. VARIATION IN THE KILN							
Bulk density	Н	POSITION	PEAK				
Bulk density1	H1	SIDE	410				
Bulk density1	H2	SIDE	440				
Bulk density1	H3	SIDE	812				
Bulk density1	H1	CENTRE					
Bulk density1	H2	CENTRE	398				
Bulk density1	H3	CENTRE	672				
Bulk density2	H1	SIDE	417				
Bulk density2	H2	SIDE	350				
Bulk density2	H3	SIDE	388				
Bulk density2	H1	CENTRE	453				
Bulk density2	H2	CENTRE	454				
Bulk density2	H3	CENTRE	469				

TABLE 29

DILATOMETRIC VALUES OF THE GLAZES							
VARIATION IN THE DIFFERENT BATCHES							
COEFFICIENT		M 1	M 2	M3			
20.100	AVERAGE	5.76	5.73	5.63			
	STD DEV.	0.06	0.06	0.2			
20-300	AVERAGE	58.87	60.13	60.16			
	STD DEV.	0.5	0.9	0.4			
300-500	AVERAGE	62.3	64.2	64.1			
	STD DEV.	0.4	0.5	0.6			
TG (ºC )	AVERAGE	644	618	648			
	TRAVEL	10	9	5			
SP (ºC )	AVERAGE	823	810	824			
	TRAVEL	2	11	12			

Deflection varies with the position in the kiln and is related with the temperature variation arising in that particular part of the kiln (given that the glaze is constant and the variations in thickness are negligible during production), and this affects the modulus of elasticity of the body and its expansion coefficient. This leads to the conclusion that if we have established the relationships between the factors that have an influence on deflection, then we have to design working conditions which will minimize this influence:

-Adjusting the tolerances of the values of the spray-dried powder variables

-Adjusting the tolerances of the values of the pressing variables (especially that of compaction, since thickness varies little)

-Monitoring Capacity Indices by means of Statistical Process Control (including X/R graphs).

-Glazes maintain their form conditions much more closely than other variables in the plant or process.

-Adjusting the thickness of the glaze layer over the whole tile (especially at the edges). -Improved cleaning of the glazed edge.

-FUNDAMENTAL adjustment of temperature and its UNIFORMITY in the kiln, above all as regards temperature distribution in the kiln cross section along the whole channel:

-Top and bottom temperature (including action on rollers)

-Side and central temperature

-Minimum spacing between tiles (both laterally and front to back)

-Systematic checking of deflection at the exit of the kiln for two purposes: -To adjust this

-To monitor the variability of other variables

#### **CONCLUSIONS:**

The conclusions can be quite simply summed up by stating that the variability of the parameters for materials and processes should be considered in any determinations made on floor and wall tiles, and that this is a consequence of the nature of the materials used, as well as the production process involved. Ignoring this could lead to wrong or at least inaccurate results, and thus to making mistaken decisions.

Deformation is one of these factors. The deformation of tiles should be considered from the standpoint of the variability of the factors of influence. In the case studied these factors were the following:

-Green bulk density of the body -The thickness ratios between the body and the glaze -The nature of the glaze coating -Firing conditions

The influence of these factors on the glaze-body fit was studied extensively in previous works (References), which confirmed the validity of the equations proposed for assessing curvature and thus for predicting the curvature of the fired tile due to the glaze-body fit. In this work the previously found results are confirmed for cases involving each one of the factors individually under steady conditions (no variability) of influencing factors. As was already stated in that work, to obtain a correct estimation based on the aforementioned thermal expansion curves, of the elasticity moduli and the thicknesses of both components, the procedure for preparing the test specimens used for determining the thermal expansion curves of the glaze and the body must be very similar to the industrial manufacturing process.

The truth of the matter is nevertheless, that the industrial process exhibits a certain **variability** which, though not invalidating those results, requires taking them in a *unidirectional* sense, that is for tiles with specific conditions or, more correctly expressed, for the parts of the tiles in which such conditions are found.

Setting out from the reality of the industrial ceramic process, with its **variability**, we can say that the temperature, or differences in temperature are the determining factors in the appearance of deformations (deflection) as a result of increasing the body's modulus of elasticity (in the case of constant glaze) according to the proposed equations (References)). The greater the temperature the lower the deflection, and this is even more so for stoneware tile.

The influence of green bulk density has an influence on the deflection of fired tile, especially in the case of monoporosa tile (other factors being equal), as a result of the increase in the modulus of elasticity. The greater the compaction the less deflection.

The glaze coat or thickness ratio is shown to be negligible in the cases studied, and even more so if we take into account that the variations produced are greater than the ones displayed by normal production variability. In any event, the thicker the coat of glaze the more deflection found.

The nature of the glazes used affects deflection but to a lesser extent than temperature and compaction. The glazes used are widely employed for the production of glossy, transparent and opaque products, both in porous single-fired and stoneware tile manufacture.

The previous conclusions notwithstanding, in the line of earlier works, the most important conclusion is that the influence of temperature, compaction and the thickness ratio has *necessarily* to be approached individually, on each part of the tile, between tiles and with the variability of the process and of the materials. Only from this stance can we explain the presence of *differential variations* in deflection:

-Between the tiles at the sides and centre of the kiln

-Between the front and back of the tile

-Between the parallel sides of the tile

Approaching this variability means corrective action can be designed *individually*, since otherwise action which improves one aspect may well have an adverse affect on another. It is not enough to increase the temperature to reduce deflection. A further step has to be taken: the temperature has to be made uniform, but yet still another step must follow, specifying in which zones the temperature should be increased or reduced or made uniform. The same can be said for compaction and for the thickness of the glaze.

The operative recommendation is thus:

- 1.- To make temperature uniform in the channel: wall and centre. front and back.
- 2.- To make temperature uniform in the tile: sides-corners-centre-top-bottom.
- 3.- Temperature increase (in the event of needing to reduce the expansion coefficient of the body and increase the modulus of elasticity).
- 4.- To make green compaction uniform.
- 5.- To make the thickness of the green tile uniform.
- 6.- To make the thickness of the glaze layer uniform.
- 7.- To adjust the expansion curve of the glaze by means of the engobe layer.

Point 1 has already been widely studied and put into practice, though the part dealing with front/back uniformity is not often paid attention to. For this purpose, the maximum

compactness of the "carpet" of tiles inside the kiln is a determining factor.

Point 2 may well be the most controversial and difficult to achieve. To succeed here, one needs to obtain the greatest compactness of the "carpet" of tiles as well as to get rid of the roller effect. The roller does indeed screen off heat from the bottom of the tile and thus causes temperature differences. The study of different roller types or compositions and the way these are handled to make sure heat gets to the product in spite of their presence would be steps furthering temperature uniformity. The ultimate objective would be eliminating the rollers.

Points 3, 4 and 5 tend to be the most commonly found ones amongst measures taken to correct deformations.

Point 6 involves keeping not only a constant coat or quantity of applied glaze, but also uniformity in the way this is spread across the tile, avoiding starved glaze and especially glaze build-ups along the edges, where glaze layers can be doubled, a fact which, along with the temperature differential, makes the corners "stick up".

Point 7 becomes a necessity, bearing in mind the difficulty and sometimes impossibility of correcting glaze expansion and maintaining the sought-after effects. In this respect, engobes with a very high expansion coefficient indeed are being successfully introduced. The limit will be getting the best fit between the three layers, since in the long run, crazing can arise as time goes by, which may be impossible to detect with traditional autoclave tests.

Statistical monitoring of the variables of influence by means of Statistical Control (XR graphs) will be an extremely important tool for preventing deviations.

The flow diagram for operations of detection and correction of differential deformations is schematically displayed in Figure 36.



.



FIGURE 36 (CONT.)



The measurement of deflection at the kiln exit, keeping regular times, location and type of tile will also be a determining factor in the detection and correction of the phenomenon. For this purpose we propose the use of the patented MOBILE FLATNESS METER (R), which can give the deflection measurements for the different parts of the tile in a straightforward fashion.

Lastly it should be said that this work forms part of a more extensive project in which tests of the above point 2 are included.

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