RAPID, HARMLESS, AND NON-DESTRUCTIVE MEASUREMENT OF CERAMIC TILE BULK DENSITY

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ABSTRACT

This study addresses the technical feasibility of using a new method of determining the bulk density distribution in ceramic tiles of all possible sizes. This new technique is based on the measurement of pressure distribution inside the press die cavity, using paper that is sensitive to the applied pressure, in addition to the composition compaction diagram.

The measurement procedure and methodology have been fine-tuned, studying how this measurement, together with its precision and reproducibility, is influenced by certain external factors. After the capability of the method had been confirmed, it was used to obtain the bulk density distributions of industrially processed ceramic tiles.

This new measuring method is **rapid, simple to use, and neither destructive nor toxic.** The developed computer application enables a **bulk density map to be obtained,** defective areas to be readily detected, and allows complete data analysis.

1. INTRODUCTION

The porosity of freshly pressed ceramic bodies conditions green tile behaviour during the process (drying, glazing, and firing) and largely determines the properties of the final product (dimensions, curvature, etc.). This makes it necessary to control tile porosity during shaping. Due to the difficulty of measuring ceramic tile porosity, bulk density is the physical magnitude that is actually measured to control the pressing stage.

The most common method used to measure green tile bulk density has been by mercury displacement [1]. The main advantages of this method are its ease of use and high precision (absolute error of ± 4 kg/m^3). Nevertheless, it has the drawbacks of being destructive, discontinuous, and manual. Furthermore, the high toxicity of mercury implies a grave health risk for workers performing industrial compaction controls. Although there are no specific regulations on mercury exposure yet in Spain, on 31 January 2005 the European Commission approved the so-called 'Community Strategy on Mercury'. This strategy envisages a series of actions designed to reduce mercury use and emissions on both a Community and worldwide level, mainly based on a progressive suppression of mercury exports until 2011. It appears, therefore, that the chosen EU option is to prohibit the use of mercury, rather than actively to legislate concerning its industrial use. This means that companies must, in the near future, look for alternatives to the use of mercury in tile bulk density measurement.

In effect, new methods have been developed in recent years, which, as a whole, conform to the basic principles in the following ways:

1. *Volume measurement of the sample:* from the thrust resulting from submerging the samples in water [2], by the reconstruction of the volume of the samples using laser telemeters and covering them with flexible membranes using air or water [3].
2. *Measurement of a property directly related to the bulk density of the sample:* absorption of X-ray radiation [4] and the speed of ultrasound transmission [5].
3. *By the use of compaction diagrams:* the installation of strain gauge sensors inside the punch used for pressure measurement [6], and moisture measurement through infrared radiation [3].

ITC studies have shown that, if all other physical and chemical properties of the spay-dried powder are held constant, the bulk density of freshly pressed ceramic bodies (D_{ap}) depends entirely on pressing pressure (P) and spay-dried powder moisture content (H). The relation between these parameters constitutes the well-known compaction diagrams, used to control the pressing operation.

$$D_{ap} = (AH + B) \ln (P) + (CH + D)$$

Equation 1.

Where A, B, C, and D are empirical fitting parameters that depend on each composition.

It is currently possible to use these types of equations and sensors to measure pressing powder moisture content [3] and the maximum pressure in the hydraulic circuit of the press, and to maintain automatically the average bulk density of the pieces over time [3]. Nevertheless, the equalization of the bulk density in tiles of the same pressing batch, or even within the individual pieces themselves, which is needed to avoid important defects in the final product, requires measurement of the average pressure and its distribution throughout all press die cavities.

The initial attempt to determine the pressure distribution inside the die cavity consisted of introducing strain gauge sensors inside the cavities [6]. This method, of great interest in the study of industrial pressing operations, turned out to be impractical from the point of view of controlling the process due to its technical complexity and high cost.

Out of the various systems presently available for measuring the pressure distribution between two contacting surfaces [7,8], the present study has examined the use of pressure-sensitive paper. The pressure-sensitive paper, especially designed for this application, contains a series of ink microcapsules that break under pressure and free their ink, thus changing the intensity of the colour of the paper depending on the maximum applied pressure. Though this pressure-sensitive paper does not allow real-time measurement of the pressure, it is adequate for this application, easy to use, and cheap.

2. OBJETIVE

The aim of this study is the evaluation of the technical feasibility of the use of a pressure-sensitive paper to estimate the bulk density of the ceramic tile bodies after compaction.

3. MATERIALS AND METHODOLOGY

The present study was conducted with a typical porcelain tile composition used in porcelain tile manufacture. Using the methodology described in other studies [3], the following compaction diagram was obtained for this powder:

$$D_{ap} = (-2.68*H+161)*\ln(P)+31.2*H+900$$

Equation 2.

Once the powder compaction diagram is known, paper colour intensity needs to be related to the applied pressure. The paper manufacturer recommends obtaining this relation for each material on which the pressure is applied. For this, a metal die, 40 mm in diameter, is used, on the bottom of which a portion of the indicator paper is placed. Among the different types of pressure-sensitive paper, one was chosen that was valid for the usual range of pressures found in the forming of glazed floor and wall tiles on an industrial scale (100-500 kg/cm^2).

The spray-dried powder was conditioned to a moisture content of 0,062 kg water/kg dry solid, weighed and introduced into the die using a small glass funnel, in order always to fill it in the same way. Once the piston was in position, the whole was vibrated to achieve maximum uniformity. The die with the powder and the paper was placed on the crosspiece of a universal testing machine, and the maximum pre-set pressure was applied. Once this pressure was reached, the sample and the paper were extracted.

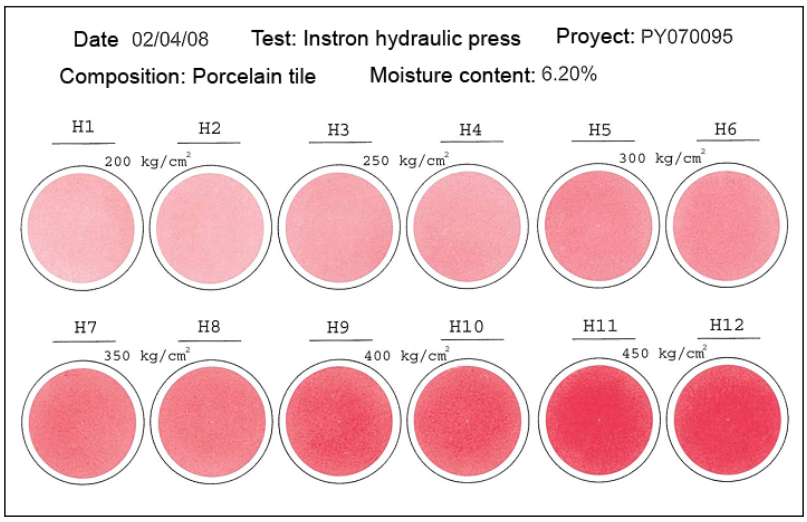

Figure 1. Paper samples obtained under different pressures.

Modifying the maximum pressure in the press yielded a series of samples with different densities, plus fragments of paper with various colour intensities (figure 1). One can observe how an increase in pressure results in a corresponding increase in the intensity of the colour of the paper.

Scanning the pieces of paper and using the appropriate software, it is possible to calculate their average colour intensity (I). The colour intensity is calculated from the measurement of the chromatic coordinates R, G, and B of the colour of each coloured piece of paper, from the following equation [9]:

$$I=255-0.30*R+0.59*G+0.11*B$$

Equation 3.

Figure 2 presents a plot of colour intensity versus the maximum applied pressure on the spray-dried powder. The figure shows that the relation between

both of these variables is semi-logarithmic; fitting the experimental points by lineal regression yields the relation equation between both variables, with a high coefficient of correlation. This expression, combined with equation 2, allows the following relation to be obtained:

$$D_{ap}=(-2.68*H+161)*\ln(381*\ln(I)-1409)+31.2*H+900$$

Equation 4.

This equation permits the calculation of bulk density in any point from the measurement of colour intensity (I) and spray-dried powder moisture content (H).

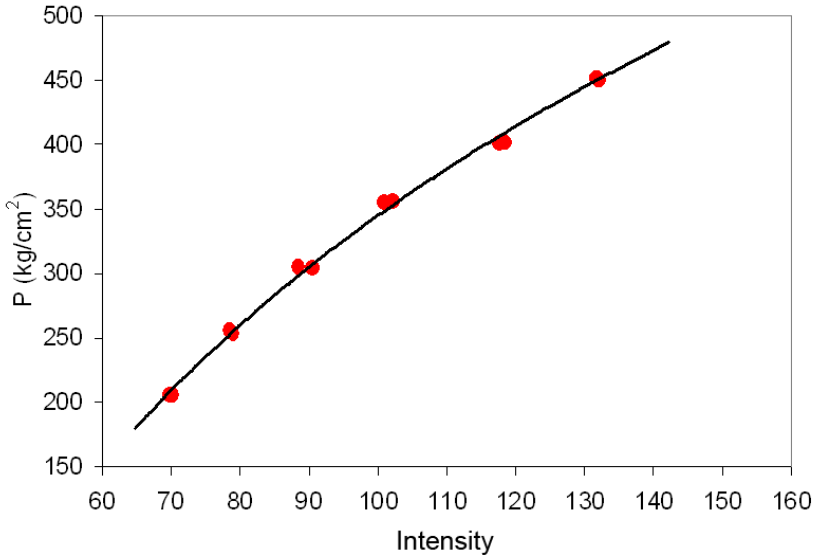

Figura 2. Paper calibration.

The software used generates false-colour images of the pressure distribution recorded by the paper, as well as the estimated bulk density, and facilitates complete processing of these data (frequency distribution histograms, measurement of the variables in the areas chosen by the user, maximum and minimum pressure and density values, the value of the variables in lines drawn by the user, 3D images, iso-pressure and iso-density diagrams, etc.)

4. EXPERIMENTAL RESULTS

4.1. Validation of the method.

In order to validate the measurement method, cylindrical pieces, 40 mm in diameter, were pressed at different pressures using the methodology described in the previous paragraph, with a spray-dried porcelain tile powder conditioned at three different moisture levels (0.04, 0.05, and 0.065 kg water/kg dry solid).

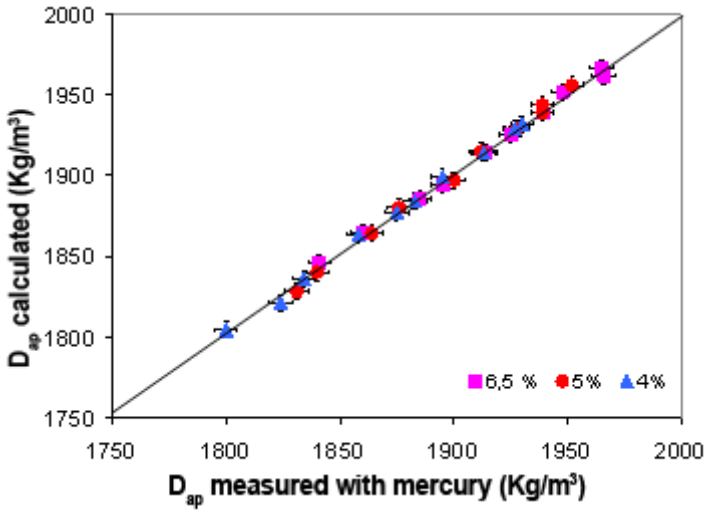

Figure 3. Bulk density calculated with the proposed method, and by mercury displacement.

The bulk densities obtained by the displacement of the pieces in mercury were measured, and then estimated from the intensity measurements of the papers used during compaction of the pieces, from equation 4. Both values are plotted in figure 3, the plot corroborating the validity of the method proposed for estimating bulk density. Indeed, all the values are situated on the diagonal, the differences between them in every case never greater than ± 5 kg/m^3 (the amplitude of the error bars represented in figure 3).

Repetitive testing in the measurement of paper colour intensity, and the use of equation 4 yielded an absolute error in pressure measurement of ± 4 kg/cm^2 and of ± 3 kg/m^3 in the estimation of bulk density. Thus, the method in itself is sufficiently precise. Nevertheless, if one takes into account that a compaction diagram obtained from mercury displacement is used to measure the bulk density of the pieces, the resulting total absolute error is ± 7 kg/m^3.

4.2. Influence of different variables on the measurements made.

This section addresses the influence on paper calibration (figure 2) and, therefore, on the bulk density estimation of the following variables: the evolution over a period of time of the intensity of the paper once it has been used, spray-dried powder moisture content, powder composition, thickness of the piece, and nature of the surface on which the pressure is applied.

4.2.1. Influence of the time between pressure application and paper processing.

The ink fixing process on the paper is not an immediate one. During some time, as in the process of photo development, colour intensity is modified until it reaches a stable value (figure 4). In effect, until about 17 hours have passed, the paper does not reach its maximum intensity or its final stability. During this period, colour intensity can increase by about 5%. On the other hand, during the

first three hours at least, the slope of the curve is very steep, indicating that this parameter should be carefully controlled, especially in this period.

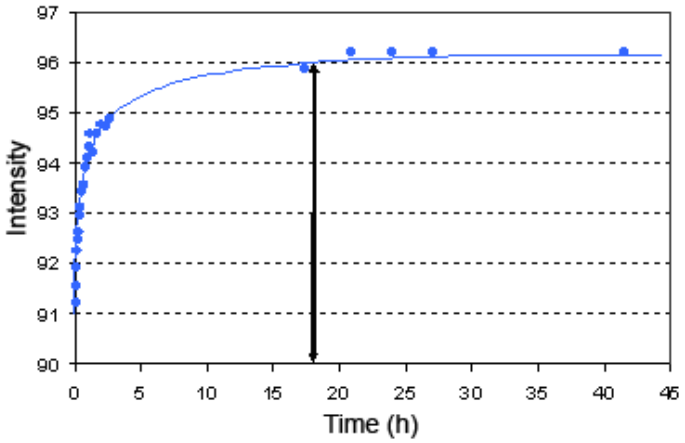

Figure 4. Evolution of paper intensity over time.

In industrial practice, immediate results are usually required. For this reason, it is important to distinguish two situations. On the one hand, if precise quantitative results are desirable, the time between the test and paper processing should be fixed to coincide with the calibration of the paper, for example 15 minutes. On the other hand, if one wishes to obtain qualitative pressure distribution and/or bulk density data in order to know if the die is being properly loaded, image processing can be immediate.

4.2.2. Influence of spray-drid powder moisture content.

In order to study the influence of moisture content on the relation P=f(I), samples of spray-dried porcelain tile powder were pressed at different pressures, using a powder whose moisture content had been previously conditioned at 0,04 kg water/kg dry solid. The methodology described in paragraph 3 was followed.

The results obtained are plotted in figure 5. It may be observed that the points corresponding to a bed of spray-dried powder of 0,04 kg water/kg dry solid (solid squares) lie on the paper calibration line, obtained for a powder of the same composition (porcelain tile) but with a different moisture content (0,06 kg water/kg dry solid). These results show that under the conditions used for the experiments, and at the moisture levels studied, this variable does not affect paper calibration.

4.2.3. Influence of powder composition.

In order to analyse the effect of powder composition on paper calibration, test pieces were pressed following the method described in section 3, using red spray-dried stoneware powder at 200, 250, 300, 350, 400 and 450 kg/cm^2 with a moisture content of 0.06 kg water/kg dry powder. Figure 5 presents the results obtained (solid triangles). One can see that the points corresponding to the red stoneware composition lie near the line representing the porcelain tile, indicating that composition does not greatly influence paper calibration.

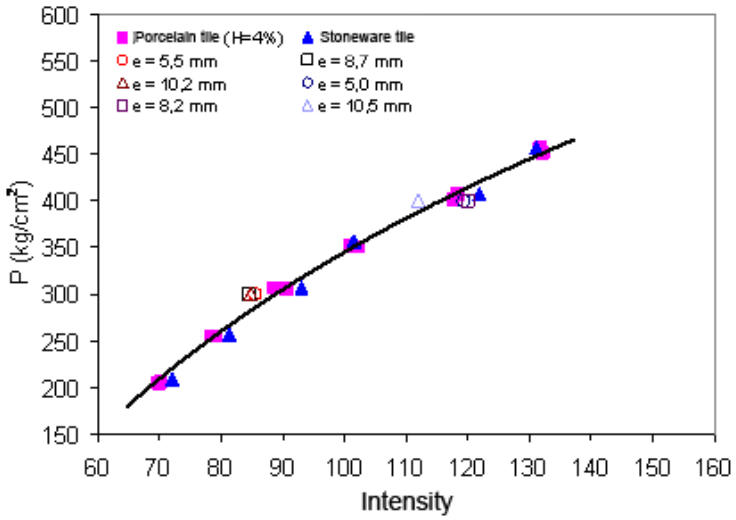

Figure 5. Influence of moisture, composition and end thickness of the piece on the relation P=f(I).

4.2.4. Influence of end thickness of the pieces.

In order to study the influence of the thickness of the pieces, samples of different thicknesses in the industrial working range (5,5 to 10,5 mm) were pressed, applying two different maximum pressing pressures, 300 and 400 kg/cm^2 with a porcelain tile powder. The results are shown in figure 5 (hollow symbols). It may be observed that the points representing the different thicknesses (h) lie very close to the base line, indicating that at the thickness levels tested, this variable does not significantly influence paper calibration.

4.2.5. Influence of the nature of the surface on which the pressure is applied.

The punch surface that enters into direct contact with the spray-dried powder is a rubber that is extremely resistant to abrasion, of differing hardness, directly vulcanised on to the metal part of the punch.

In order to study the influence of the nature of the surface that transmits the pressure to the spray-dried powder, samples were pressed using four different types of punches. Two of these had a metal surface that was applied to the powder; one was cylindrical with a 40 mm diameter (referenced as cylindrical metal) while the other was square with 15 cm sides (referenced as square metal). The other two punches were similar to the latter, but each part that was applied to the powder was covered by a rubber of different hardness (referenced Shore A 90-92 and Shore A 96-98).

Figure 6 presents the results of the experiments (solid symbols). The figure shows that, though the behaviour of all the test surfaces was similar, it differs from that observed up until now. Indeed, the curve showing the initial calibration of the paper (solid line) seems to have shifted slightly higher (dashed line).

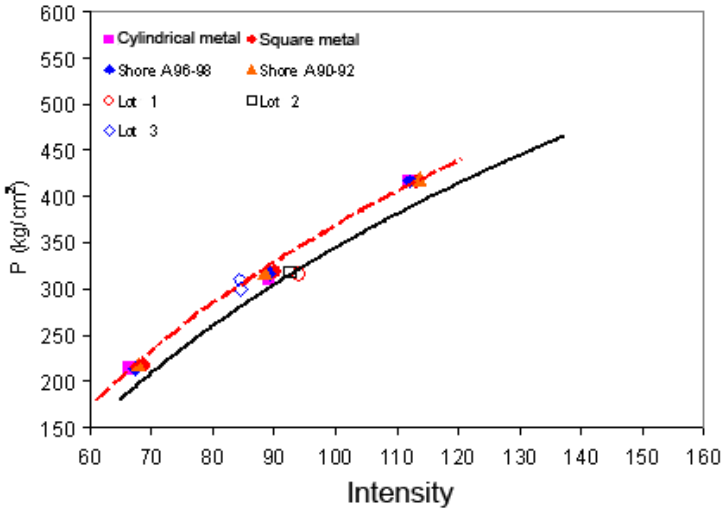

Figure 6. Influence of contact surface and paper lot on the calibration.

Since the paper used in this experiment came from a different lot than that used in the rest of the tests, it was decided to compare the behaviour of the different lots. This was done by applying the same pressure of 300 kg/cm^2 to the three available lots. The papers were then processed and calculated for their intensity. Figure 6 shows the results in the form of hollow symbols. It may be observed that the papers from lots 1 and 2 behaved similarly (circle and square, respectively) and were close to the initial calibration, the points representing lot 3 (rhomboids) exhibited less colour intensity under the same pressure, aligning with the points corresponding to the previous experiment (using surfaces of a different nature), in which this same paper was used. The study of these results led to the drawing of a new calibration curve that coincided with the dashed curve in figure 6. These findings indicate that, for the calculations to be sufficiently precise, the calibration curve of the paper should be checked, at least when a change of lots occurs.

4.3. Tests performed on an industrial scale.

Industrial scale tests were performed in a hydraulic press of the type customarily used for producing ceramic tile bodies, equipped with isostatic top punches. This was being used to make four porcelain tiles with each stroke, with a nominal fired size of 33 cm x 33 cm, whose compaction diagram corresponds to equation 2.

In this case, the pressure distribution measurement inside the press die cavities was made by placing the sensitive paper on the bottom punch (smooth face) of the press. To do this it was necessary to stop the press momentarily, place the papers, and then run the programmed pressing cycle. The press was then stopped; the freshly pressed pieces were withdrawn, as were the papers, which were to be subsequently processed.

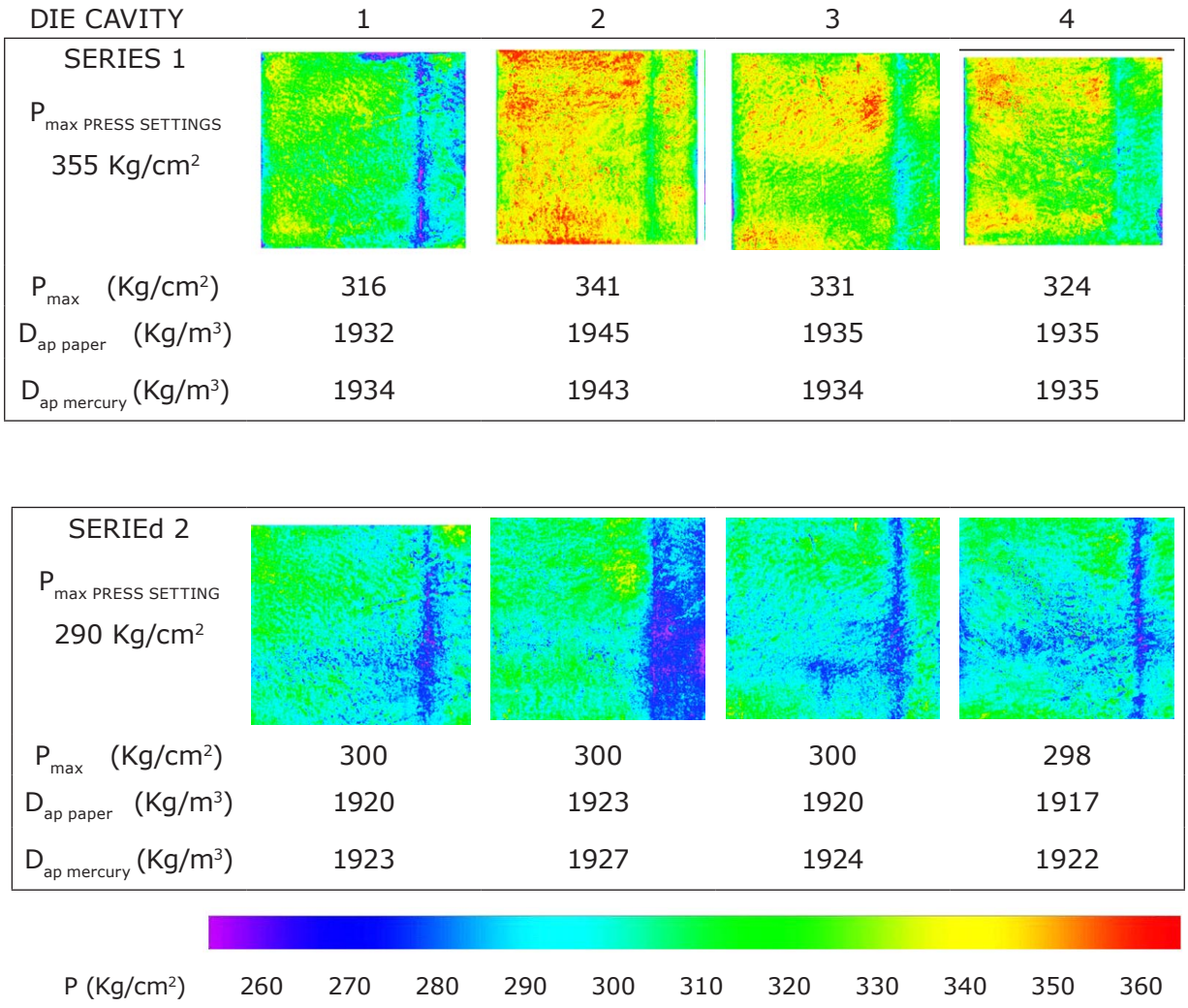

Figure 7. False-colour graphics of pressure distributions, pressure values, and average bulk densities.

4.3.1. Average bulk density and pressure measurement tests.

Following the procedure described previously, 8 test pieces were pressed at a pressure of 355 and 290 kg/cm^2, obtaining two series (series 1 and series 2, respectively) of four pieces each. Figure 7 displays the false-colour maps of the pressure distributions in both series, the estimated maximum pressure values (P_{max}), and average bulk density ($D_{ap\ paper}$) of each piece, in addition to those of the average density of the pieces measured by mercury displacement ($D_{ap\ mercury}$). Note, first, that the estimated density obtained from the sensitive paper and that from mercury displacement are very close, which corroborates the validity of the method on an industrial scale.

The false-colour maps allow one to distinguish at a glance which piece has been compacted at the highest pressure; in this case, it obviously corresponds to a piece from series 1, processed in die cavity 2. This piece was compacted at a maximum pressure of 341 kg/cm^2, and presents the greatest bulk density (1943 kg/m^3). As might be expected, the pieces from series 2 all reached lower pressures, and thus exhibit lower bulk densities.

The system used for measuring pressure allows quantification, for the first time, of the average maximum pressure reached in the different die cavities of the press in the same pressing cycle. While the difference in pressure between the pieces in series 1 varies considerably, those in series 2 are much more uniform, all being compacted at a lower pressure. In effect, the difference in pressure between the pieces in series 1 is as much as 25 kg/cm^2 (between pieces 1 and 2), while in series 2 it hardly reaches 2 kg/cm^2. As a result, the pieces in series 2 exhibit a more uniform average bulk density, with a difference of hardly 5 kg/m^3 between them, while this value rises to 9 kg/m^3 in the pieces from series 1, this being a difference that would require calibration in porcelain tile compositions.

In the pieces from series 1, the pressure applied on the pieces located at the sides of the press (die cavities 1 and 4) is noticeably lower than that applied in the centre. This fact, known empirically from the different bulk density of the tiles traditionally pressed in these positions, cannot be quantified with current methods.

4.3.2. Pressure distribution and bulk density measurement in a ceramic piece.

With the aim of evaluating the validity of the proposed method for estimating the bulk density distribution inside each piece, the industrial press was adjusted to produce greater bulk densities in the front region of the tiles. The bulk density of 16 portions of the test pieces was determined by mercury displacement. In addition, using the pressure-sensitive paper, the pressure in each of these portions was determined and their bulk density was estimated using the respective pressure values. The results obtained for one of the pieces are shown in figure 8; the figure also shows the average values for these variables for the entire piece and in each of the rows and columns.

It may be noted, first, that the bulk density values obtained by both methods (by mercury displacement (second in the values) and estimated from the pressure (third values)) are similar, though the maximum differences are greater than those found between the average values of the entire piece (figure 7 and figure 8). This fact could be due to the difficulty in getting the portions of the pieces exactly to match the surface used to measure the pressure in each area, mainly owing to the irregularity of the cuts. This effect is more noticeable in the measurement of the zonal density values (portions of the piece) than in the average values of the test piece.

As expected, the greatest bulk density was found in the front part of the piece (row 4) In effect, in this row the average pressure (363 kg/cm^2) was much greater than in the entire piece (328 kg/cm^2); the bulk density corresponding to this row (1952 kg/m^3) was also much greater than the average (1933 kg/cm^3). In fact, the area of greatest measured pressure corresponds to portion A4, with 426 kg/cm^2 (red area in the piece); this was also the portion that displayed the greatest bulk density, in both the mercury displacement measurement (1970 kg/m^3) and in the measurement estimated from the pressure determination (1973 kg/m^3).

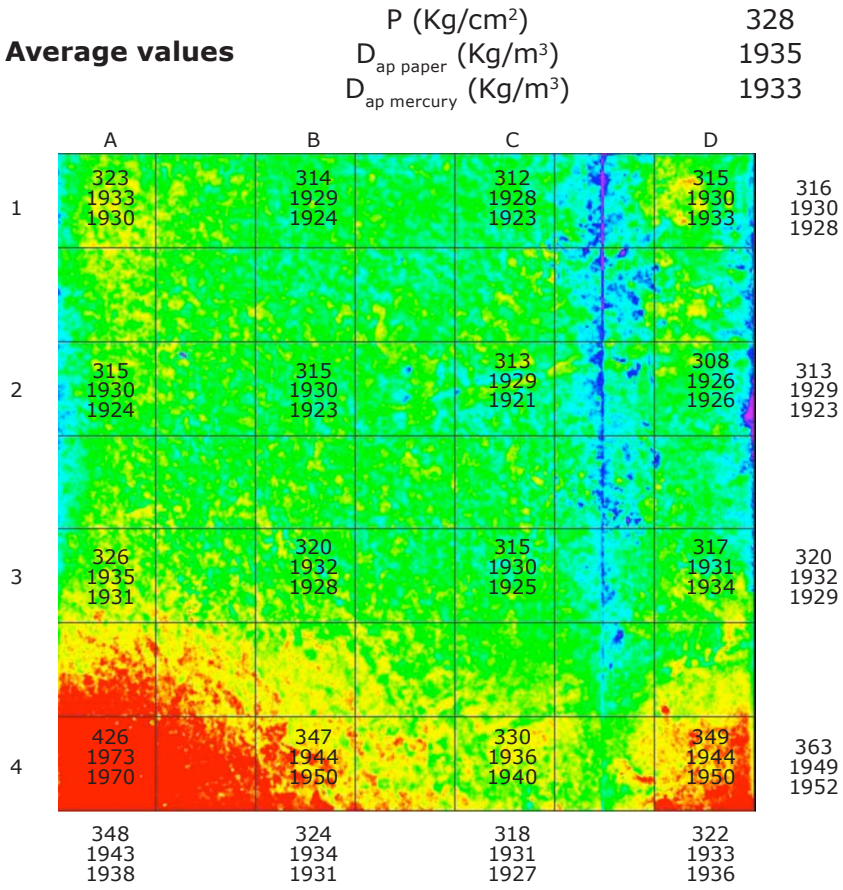

Figura 8. Pressure values, D_{ap} estimated with sensitive paper and D_{ap} measured by mercury displacement ($P/D_{ap\ papel}/D_{ap\ mercurio}$).

In respect to the other rows, the pressure and bulk density distribution was very uniform. In effect, the maximum difference in average bulk density between rows 1, 2 and 3 hardly reached 6 kg/m³, the lowest density values being in row 2. Such homogenous bulk density values in this part of the piece could lead the less dense areas identified by the different methods, though very close together, not to coincide exactly. In effect, while the minimum bulk density measured by mercury displacement corresponds to area C2 (1921 kg/m³), that estimated from pressure is found in area D2 (1926 kg/m³), in the same row and next to it.

Finally, it may be pointed out that the maximum variation in bulk density detected by both methods is very similar: 48 kg/m³ in the case of mercury displacement (areas A4-C2), and 46 kg/m³ when sensitive paper is used (areas A4-D2). The results obtained from the rest of the test pieces are similar, corroborating the validity of the proposed method. Notice also that the information supplied by the sensitive paper is more exhaustive than that supplied by mercury displacement, since it implies the possibility of continuous analysis of pressure distribution, rather than the point and discrete results provided by mercury, and hence of bulk density inside the piece.

5. CONCLUSIONS

The following conclusions may be drawn from the study:

- The measurement of the distribution of maximum pressing pressure inside the die cavity, using sensitive paper, is a precise measurement (± 4 kg/m^2) that allows the bulk density of ceramic tiles to be estimated with a difference of ± 5 kg/m^3 with respect to the mercury displacement method.
- In order to make a precise estimation of bulk density it is very important to control the time between the application of the pressure, and subsequent paper processing, and to control the paper calibration line at least once for every new lot of paper used. If this method is to be used for purely qualitative purposes, these aspects are less important.
- Powder moisture, composition, final thickness of the piece, and nature of the surface used to apply pressure on to the powder have no significant influence on measurement results.
- The application of this measurement method in an industrial press enables the distribution of the maximum applied pressure on a tile in the die cavity, and tile bulk density to be obtained rapidly, harmlessly, non-destructively, and precisely.

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