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ScienceDirect
Journal of the European Ceramic Society 30 (2010) 2927–2936
www.elsevier.com/locate/jeurceramsoc

Non-destructive measurement of bulk density distribution in large-sized ceramic tiles

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Available online 12 March 2010

Abstract

An apparatus has been designed, built, and patented that is able, non-destructively, to determine bulk density distribution in large-sized ceramic tiles. The proposed method, based on the X-ray absorption technique, provides numerous advantages compared with current methods: it enables complete maps of the bulk density distribution, and is neither destructive nor toxic. The measurements are performed with a low-power X-ray emitter tube. The measurement system is housed in a shielded enclosure.

This technique has been used to examine ceramic tiles fabricated under different industrial conditions, modifying press operating parameters. The use of high-precision laser telemeters also enables maps of tile thicknesses to be obtained, which allow tile surface mass to be determined. The non-destructive character of the method enables fired tiles, which had previously been examined in the unfired state, to be inspected, thus facilitating the interpretation of manufacturing defects that could originate in the forming and/or firing stage.

Keywords: Bulk density; Non-destructive evaluation; Pressing; X-ray methods; Traditional ceramics

1. Introduction

1.1. Importance of the bulk density measurement

The porosity of the ceramic tile body decisively affects green tile behaviour during processing (drying, glazing, and firing) and largely determines end-product properties (dimensions, curvature, frost resistance, mechanical strength, presence of black core, surface finish, etc.). This makes thorough control of body porosity essential during forming, with a view to appropriate further processing and to maintaining end-product properties within pre-set variation ranges.[1–3]

At present, the most widely used ceramic tile forming method is uniaxial pressing of spray-dried powder in hydraulic presses. In view of the difficulty of directly measuring ceramic tile porosity in industrial practice, the physical magnitude that is actually controlled is unfired tile bulk density. The importance of bulk density lies in its direct relationship to tile inner microstructure: inappropriate bulk density distribution in a ceramic tile will cause non-uniform fired shrinkage, which can lead to lack of dimensional stability in the end-product, such as irregularities, curvatures, or differences in size, inside a tile and between different tiles.

Historically, unfired tile bulk density has been measured by the mercury displacement method. This destructive technique is based on the measurement, by mercury displacement, of the apparent volume of samples cut from tiles. The individual bulk density values of different fragments provide information on tile bulk density distribution, and the average value of the bulk densities of all the tiles reveals differences in porosity that may exist between the tiles.

The main advantages of the mercury displacement method are its ease of use and high-precision (absolute error of $\pm 4\ kg/m^3$).[4] It is a destructive, discontinuous, and manual method, however, while the high toxicity of mercury also entails important health risks for the operators that perform such industrial compaction controls. These disadvantages have led to the search and development of new methods for the determination of ceramic tile bulk density in recent years.[5] The first improvements consisted of the determination of sample volume from the upthrust undergone by the test pieces when they were submerged in water. Though the precision of the resulting bulk density measurement was acceptable, this new method was not widely accepted on an industrial level because it was more laborious and remained destructive, discontinuous, and manual.

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doi:10.1016/j.jeurceramsoc.2010.01.033

A series of devices then appeared that allowed bulk density to be measured of samples cut from a tile, based on the determination of their apparent volume by a method other than that of immersion in a liquid. These methods notably included, on the one hand, the reconstruction of sample volume by using laser telemeters and, on the other, a method based on the measurement of the volume of water displaced by the sample, protected by a plastic membrane, when it was introduced into a mass of water. Despite the good measurement precision provided by some of these devices, the methods used remained destructive and discrete.

The Instituto de Tecnología Cerámica (ITC) has recently published a series of research papers[6,7] on two prototypes for the determination of ceramic tile bulk density, using methods based on the measurement of a property directly related to bulk density. These non-destructive methods are the non-contact ultrasound method and the X-ray absorption method. Unlike all previous methods, these methods are non-destructive and allow maps to be obtained of bulk density distribution in entire tiles. The good results obtained in these studies have led to the development and construction of an apparatus for the non-destructive measurement, by X-ray absorption, of bulk density distribution in industrial ceramic tiles.[8] This paper describes the most important technical characteristics of the apparatus, together with a series of experiments that demonstrate the system's capabilities in studying ceramic tile forming conditions.

1.2. Measurement of bulk density by X-ray absorption

X-ray inspection is a non-destructive test technique that is able to detect variations in density inside materials, which is why it is widely used in numerous industrial sectors to inspect materials for macroscopic discontinuities and flaws in their inner structure. Many commercial X-ray inspection instruments are currently available that enable density variations inside pieces with a complicated geometry (Computer Axial Tomography) to be analysed. However, in view of the high cost of these instruments and their technical complexity in handling, ITC has addressed the design and construction of a specifically adapted X-ray inspection system for the measurement of bulk density distribution in ceramic tiles.

The measurement of bulk density by X-ray absorption is based on the Lambert–Beer law of absorption, according to which, when electromagnetic radiation crosses a material, the fraction of incident radiation that is absorbed by the material depends exclusively on the material's chemical nature, thickness, and bulk density.[9] Eq. (1) represents the Lambert–Beer law applied to monochromatic radiation and to a perfectly homogeneous material in all its thickness:

$$\frac{I}{I_0} = e^{-\mu h \rho} \tag{1}$$

where I_0 is incident radiation intensity, I is transmitted radiation intensity, h is the thickness of the material, ρ is bulk density, and μ is the mass absorption coefficient for the radiation wavelength.

If the nature of the material is not modified and the thickness is known, it is thus possible to determine the material's bulk density from the quantity of absorbed energy. In practice, however, Eq. (1) is only approximate because the X-ray tubes customarily used for this type of test emit polychromatic radiation and the analysed materials are usually not homogeneous. This significantly complicates the relationships between transmitted radiation intensity and the X-ray absorption coefficient, which depends on incident radiation wavelength. If it were assumed that the analysis region is sufficiently small to be considered homogeneous, the bulk density measurement would not be limited by the heterogeneities of the material. In contrast, the dependence of the absorption coefficient on incident radiation energy makes the analytical resolution of Eq. (1) quite difficult in order to calculate the density of the material.[10] For this reason, in practice, the simplest approach is to obtain the absorption coefficient of the material to be analysed by prior calibration.

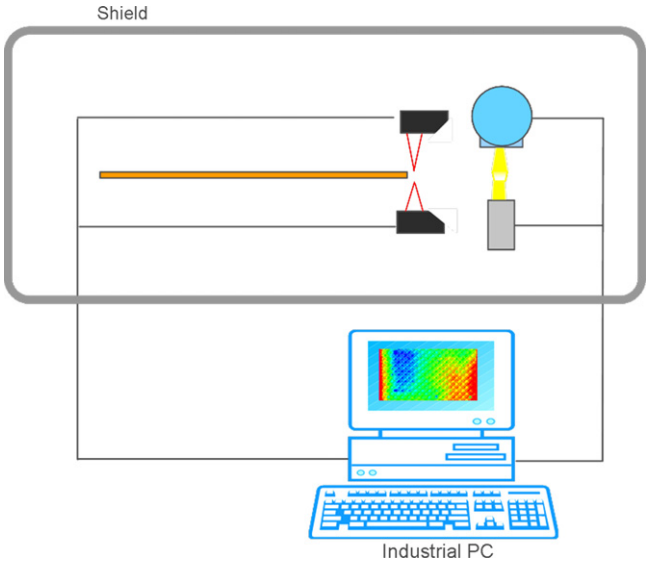

Fig. 1. Schematic illustration of the density measurement system by X-ray absorption.

Fig. 1 schematically illustrates the developed bulk density measurement system. The piece to be examined is set vertically in a metal frame (Fig. 2) with a travelling metal plate fitted with a telemetry system that measures the thickness of the piece, an X-ray emitter tube, and a radiation sensor. In testing, the measurement system performs successive scans from right to left, at a maximum rate of 1000 mm/s, with a minimum vertical displacement between the horizontal lines of 1 mm. As the device travels across the tile, it takes measurements of tile thickness and the intensity of the radiation that crosses it, with a sampling rate of 10 data per millimetre.

The entire movement and measurement system is closed in a lead shield that prevents any radiation leaks from the X-ray tube. The signals from the different measurement sensors and from the travelling system are fed into a PC with specially developed software for this application, which manages system movements, data processing, and graphic representation.

The fitted X-ray tube has 50 W power and the radiation sensor used is a ceramic scintillator-photodiode. The assembly has been designed to enable unfired industrial tiles with a maximum nominal size of 120 cm × 60 cm and thickness up to 20 mm to

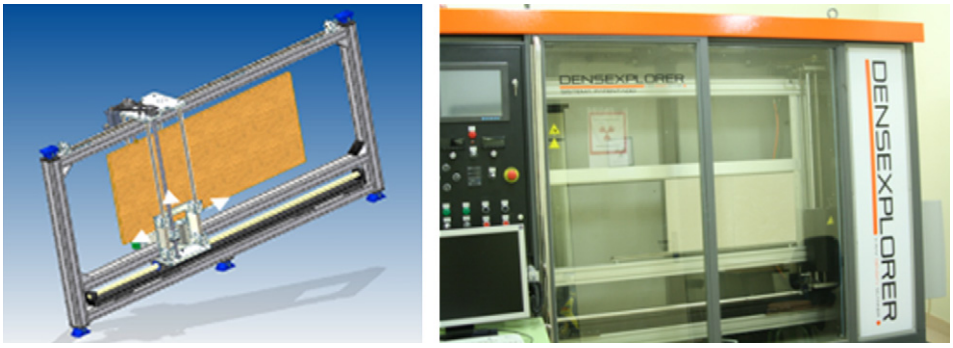

Fig. 2. Internal frame and general view of the bulk density measurement assembly.

be tested. When the test is performed at a rate of 500 mm/s, with a spacing between vertical lines of 5 mm, the analysis of a tile measuring 120 cm × 60 cm takes about 10 min.

2. Methodology and materials

The measurement of bulk density by X-ray absorption requires previous calibration to establish the relationship between the bulk density of the material being analysed and the attenuation experienced by the radiation. The developed software has a subroutine that allows calibrations to be made from a series of test pieces of the same material as that to be analysed. These test pieces must be formed under different conditions in order to obtain an appropriate range of variation of the product $h\rho$ values for testing needs.

The calibration procedure is usually carried out with cylindrical test pieces set in a holder (like the one shown in Fig. 3) for analysis. Once tube operating conditions and the test rate have been established, the different test pieces are analysed: the average test piece thickness and the intensity of the radiation transmitted through each test piece are then obtained. Using these data and the bulk densities of each test piece determined by a standard method (in this case the mercury displacement method was used), the software fits the experimental data to Eq.

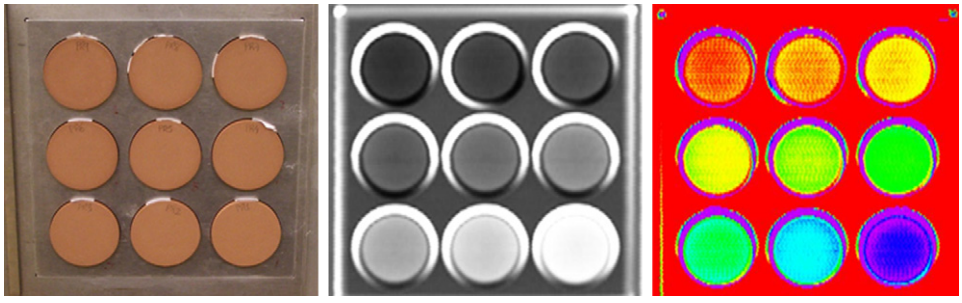

Fig. 3. Calibration test piece holder, X-ray, and bulk density map.

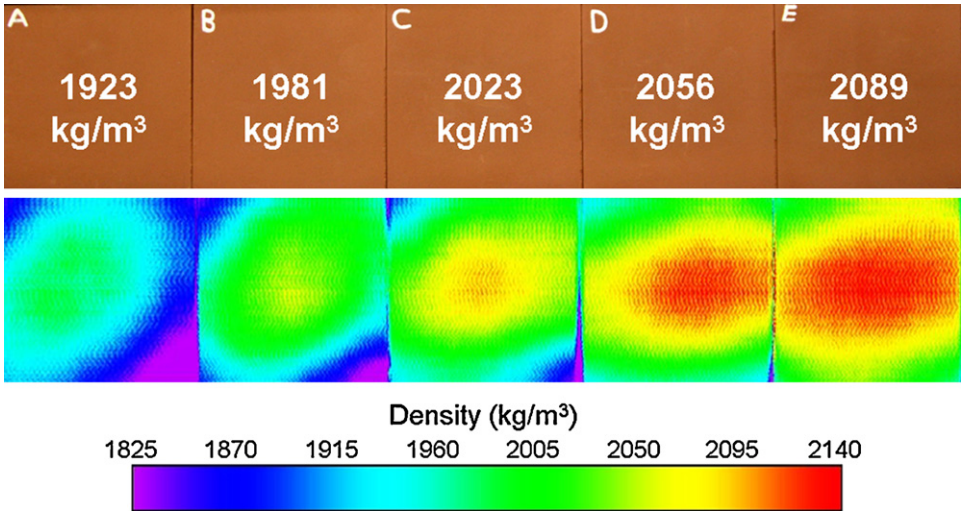

Fig. 4. Evaluation of measurement method precision. Test pieces used (top) and bulk density distribution maps (bottom).

Table 1
Average bulk densities obtained in the repeatability tests.

Ref.	Bulk density by mercury displacement (kg/m^3)	Bulk density by X-ray absorption (kg/m^3)										
		1	2	3	4	5	6	7	8	9	10	Average
A	1923	1920	1923	1923	1924	1923	1925	1924	1922	1920	1924	1923
B	1981	1978	1979	1979	1981	1981	1982	1982	1982	1982	1983	1981
C	2023	2020	2021	2021	2025	2024	2026	2026	2026	2026	2026	2024
D	2056	2056	2058	2055	2059	2059	2061	2061	2061	2061	2062	2059
E	2089	2083	2085	2083	2086	2086	2087	2086	2087	2087	2088	2086

(1), which yields the characteristic I_0 (V) and μ (m^2/kg) values of the studied material.

The test basically consists of setting the piece to be analysed inside the apparatus and then, after the shield has closed, of selecting the corresponding calibration to initiate the test with given X-ray tube and scan rate conditions.

The experiments conducted in this study were performed with three spray-dried powder compositions customarily used in ceramic tile manufacture, referenced as follows:

Stoneware tile—red-firing vitrified floor tile (Group IIa according to standard EN 87).
Porcelain tile—white-firing porcelain tile (Group I according to standard EN 87).
Wall tile—red-firing porous wall tile (Group III according to standard EN 87).

The test pieces prepared in the laboratory were formed in a hydraulic press equipped with a cylindrical die, 3 cm in diameter, or a 10 cm × 10 cm square die, depending on experiment needs. The work done on an industrial scale was carried out in an industrial hydraulic press for the production of porcelain tile of different sizes.

3. Experimental results

3.1. Evaluation of measurement method precision

In order to evaluate the precision of the density measurement method by X-ray absorption, five stoneware test pieces were prepared, measuring 10 cm × 10 cm, by pressing at five different maximum pressures. These pieces were then dried to constant weight in a laboratory oven at 110 °C. After the bulk density of the test pieces had been obtained by mercury displacement, their bulk density distributions were determined using a previously conducted calibration, under X-ray tube operating conditions of 36 kV and current intensity of 0.6 mA. This test was repeated ten times with a view to evaluating the repeatability of the measurement method.

The top part of Fig. 4 shows the five analysed test pieces, while the bottom part shows the bulk density distribution maps obtained after the first test. The bulk density values measured by the mercury displacement method and the density values estimated by X-ray absorption in each test are presented in Table 1. The agreement between both methods is observed to be very good, the maximum error (difference between the X-ray absorption measurement and the mercury displacement measurement) being 6 kg/m^3, and the mean absolute error being 2 kg/m^3.

It is interesting to note that the density measurement method by X-ray absorption not only displays the required precision, but also provides more information than other current methods. In effect, the bulk density distribution maps of the test pieces, depicted in Fig. 4, show that each piece contains a very heterogeneous bulk density distribution, which is not revealed by the mercury measurement. By way of example, the histograms of the bulk density distributions measured by X-ray absorption, corresponding to test pieces A, C, and E, together with the average bulk density values obtained by mercury displacement (solid vertical lines), are shown in Fig. 5. It can be observed that, in the case of piece C, the X-ray absorption method provides information on a variation in bulk density of approximately 260 kg/m^3, whereas the mercury displacement method only indicates that the average bulk density value of the piece is 2023 kg/m^3.

3.2. Influence of the operating variables on the bulk density measurement

3.2.1. Influence of the composition

In order to evaluate the influence of the composition of the material being analysed on the bulk density measurement, nine cylindrical test pieces with different densities were prepared from the three compositions used. After they had been dried in an oven, their bulk density was determined by mercury displacement. A calibration was then performed of each test piece

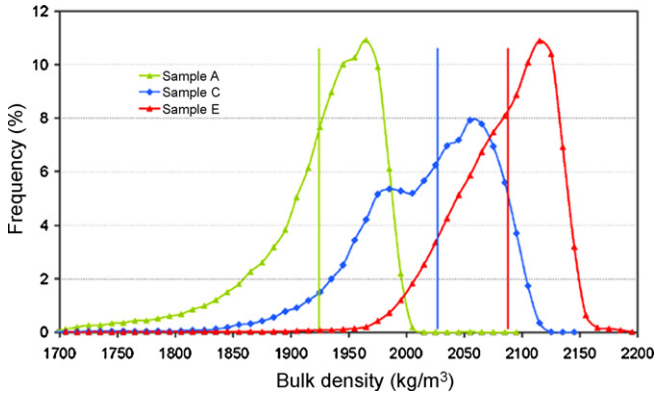

Fig. 5. Histograms of the bulk density distributions in pieces A, C, and E.

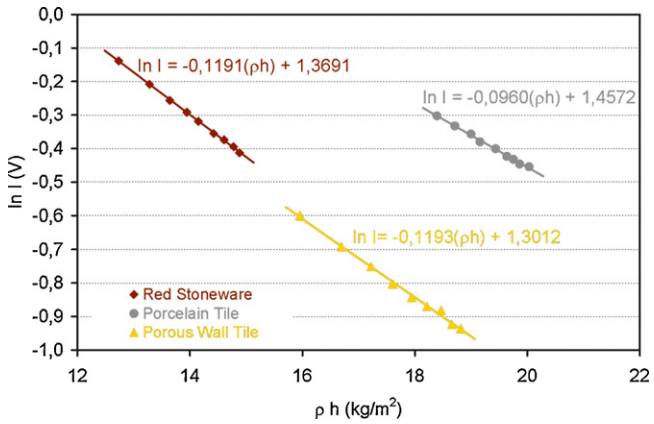

Fig. 6. Variation of ln I as a function of material surface density (ρh) for the different test compositions.

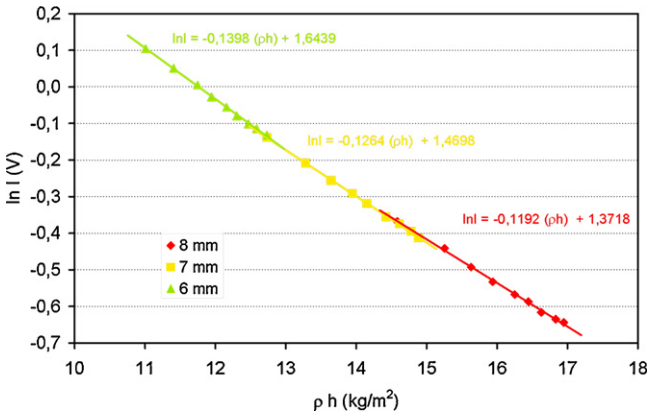

Fig. 7. Variation of ln I as a function of material surface density (ρh) for three series of test pieces of different thickness.

in the series at an analysis rate of 200 mm/s under operating conditions of 36 kV and current intensity 0.6 mA.

Fig. 6 shows the plots of the variation of transmitted radiation intensity as a function of material surface density ($h\rho$) for the three studied ceramic compositions. It may be observed that the stoneware composition displays a similar behaviour to that of the porous wall tile composition in relation to X-ray absorption. In contrast, the porcelain tile composition exhibits a slightly different behaviour, its absorption coefficient (slope of the fitted straight lines) being lower.

The differences observed in the behaviour of the analysed materials are essentially due to a small quantity of iron oxide in the stoneware and porous wall tile compositions from the clays used to prepare them, which causes the absorption coefficient to increase slightly with respect to that of the porcelain tile composition. The results of these experiments highlight the need to calibrate the apparatus for each material to be examined.

3.2.2. *Influence of thickness*

In order to study the influence of the thickness of the examined material on the density measurement, three series of nine test pieces of different densities, with thicknesses of 6, 7, and 8 mm, respectively, were prepared. After they had been dried in the oven, their bulk density was determined by mercury displacement and a calibration was performed with each series of test pieces at an analysis rate of 200 mm/s, under operating conditions of 36 kV and current intensity of 0.6 mA.

Fig. 7 presents the straight calibration lines corresponding to the test pieces. It may be observed that, though the experimental data fit a straight line for each group of test pieces, the slope and ordinate at the origin of these straight lines decrease as test piece thickness increases. This behaviour may be due to two factors: on the one hand, a non-linear response by the photodiode to incident radiation intensity and, on the other, the phenomenon known as 'beam hardening', caused by the preferential attenuation of low-energy photons in polychromatic radiation.

For this reason, in order to achieve good precision in the density measurement in tiles with significant differences in thickness (ceramic tiles with very pronounced ribs), it is necessary to perform a calibration in a sufficient wide range of surface densities ($h\rho$) in order to take the deviation of linearity, due to thickness, into account by a polynomial fit of the experimental data,

3.2.3. *Influence of the analysis rate*

The prototypes made before the construction of the apparatus presented here did not have the capacity to work at such high analysis rates as this apparatus. With a view to evaluating the influence of the analysis rate on the bulk density measurement, tests were conducted with the series of 7-mm-thick red stoneware test pieces, at two different analysis rates (100 mm/s and 500 mm/s), using as reference calibration the calibration performed at 200 mm/s.

Table 2 presents the bulk density of the test pieces measured by mercury displacement and the bulk density of the test pieces determined by X-ray absorption in these tests. The data show that though the tests were conducted at a different rate from that in the calibration used, the density estimation by X-ray absorption is observed to be very good, the maximum error being 5 kg/m^3 and the mean absolute error 2 kg/m^3. These outcomes highlight the low influence of the test rate (in standard working ranges) on the density measurement, which is very important in order to be able to examine large-sized tiles with sufficiently high speed to allow the apparatus to be used in routine production control.

3.3. *Use of the X-ray absorption technique for the study of the ceramic tile pressing operation*

3.3.1. *Detection of differences in charge, thickness, and bulk density in ceramic tiles*

With a view to evaluating the usefulness of the apparatus for studying the ceramic tile pressing operation, several profiled industrial tiles with a nominal size of 33 cm × 33 cm, obtained under different pressing conditions, were analysed. For this purpose, a series of actions were carried out in an industrial press equipped with a conventional penetrating die with four outputs, fitted with isostatic top punches intended to offset possible charge deficiencies in the die cavities. The changes in the pressing conditions, which basically consisted of the modification of the maximum pressure in the pressing cycle and/or the spray-

Table 2
Effect of test rate on bulk density measurement.

Test piece	Density by mercury displacement (kg/m^3)	Test at 100 mm/s		Test at 500 mm/s	
		Density by X-ray absorption (kg/m^3)	Error (kg/m^3)	Density by X-ray absorption (kg/m^3)	Error (kg/m^3)
1	2138	2138	0	2138	0
2	2123	2118	−5	2121	−2
3	2105	2100	−5	2103	−2
4	2081	2084	3	2086	5
5	2054	2054	0	2054	0
6	2018	2017	−1	2019	1
7	1977	1976	−1	1980	3
8	1922	1920	−2	1922	0
9	1840	1836	−4	1840	0

dried powder charge distribution in the press die cavities, are presented in Table 3.

3.3.1.1. Measurement of the surface density distribution. The surface density distributions (product ρh) corresponding to the four tiles obtained in each action, together with the average tile surface densities, are shown in Fig. 8. These representations provide information on spray-dried powder charge distribution in the press die cavities when maximum pressing pressure is applied.

It can be observed that both the surface density distribution and the average values of each tile, in the first two actions, are very similar since the only difference between them is the maximum pressing pressure attained, the powder being charged under the same conditions. In contrast, the analysis of the distributions corresponding to the tiles in action 3 reveals a considerable reduction in surface density in all the die cavities and charge displacement towards the back of the die. This change in surface density distribution was produced by modifying the charging cycle of the spray-dried powder into the press die cavities, so that the quantity of powder introduced into the back of the die cavities decreased, in relation to the spray-dried powder introduced into the front of the die cavities.

It may be noted that in all the above actions, independently of the type of charge, the tile pressed in position 4 always displayed a smaller average surface mass. It may similarly be observed that in the tiles pressed in cavities 1 and 4, there are regions with a low surface mass that remain so, to a greater or lesser extent, throughout all the actions (back right in the tiles from cavity 1 and back left in the tiles from cavity 4). This is related to the profiled fair face of the tile, and highlights the importance of controlling tile design in order to assure appropriate charge distribution.

Table 3
Changes made in pressing conditions in an industrial press.

Action	Maximum pressure (MPa)	Charge distribution
1	32.9	Conventional
2	27.9	Conventional
3	32.9	Front

3.3.1.2. Measurement of the thickness distribution. The thickness distributions obtained in the different actions are shown in Fig. 9. Comparison of these thickness maps with the mass distributions depicted in Fig. 8 shows that both variables are closely related. The regions of the tiles with the greatest quantity of material are those that are thickest when the pressing cycle ends. Examination of the thickness distributions resulting from actions 1 and 2 shows that these only differ in the average thickness values obtained. The tiles from action 1 are slightly thinner than those from action 2 (the average difference being 0.08 mm), owing to the lower applied maximum pressing pressure. Indeed, at the same quantity of spray-dried powder in the cavities (see Fig. 8) and at a constant press crosspiece travelling rate, the action 2 pressing cycle reaches the set pressure values earlier and, therefore, provides the tiles with a slightly greater thickness. With regard to the tiles obtained in action 3, these display a considerably lower average thickness than that of the tiles obtained in actions 1 and 2, as a result of the smaller quantity of material in the die cavities when the charging cycle ends.

It should be noted that, just as in the charge distribution, independently of maximum pressing cycle pressure, the thickness distributions of actions 1 and 2 are practically identical, which highlights the great robustness of the spray-dried powder charging systems used in today's industrial presses.

3.3.1.3. Measurement of the bulk density distribution. Finally, Fig. 10 presents the bulk density distribution maps of the studied tiles. It may be observed that the density distributions are directly related to the powder distribution in the die cavities. In contrast, the average bulk density of the tiles obtained only depends on the maximum pressing pressure. This occurs because, for a spray-dried powder with a given composition and particle size distribution, the bulk density reached during forming depends, exclusively, on powder moisture content and the maximum pressure applied in the pressing cycle. The relationship between these three variables constitutes the so-called compaction diagrams, which are widely used in pressing operation control. In the studied case, the moisture content and particle size distribution of the spray-dried powder introduced into the different die cavities may be considered constant, the only variable that changes being pressing pressure.

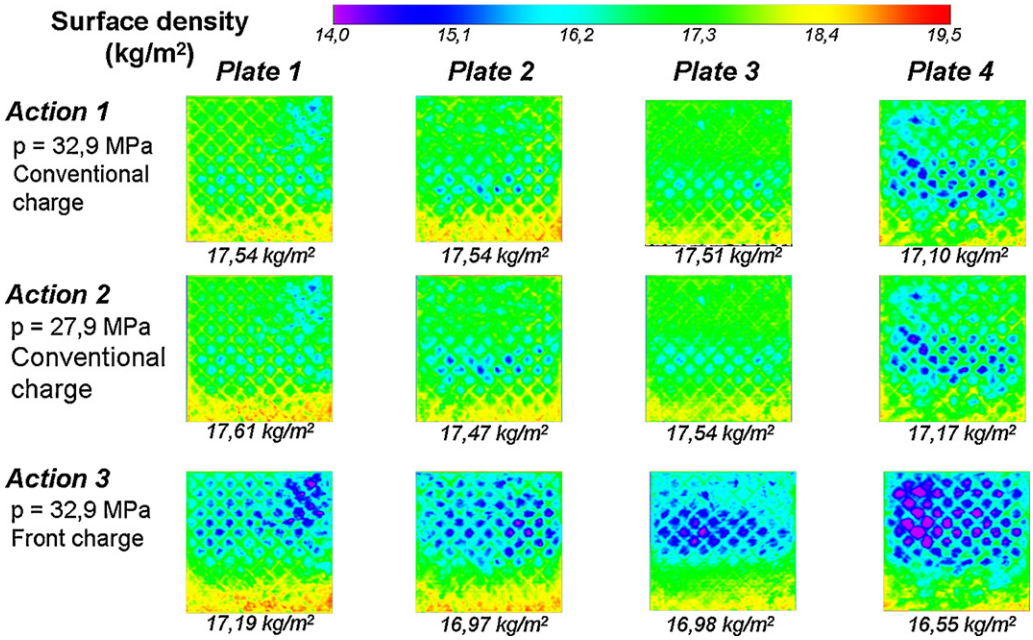

Fig. 8. Surface mass distributions in tiles obtained under different pressing conditions.

The results of these actions indicate that the detailed analysis of the bulk density distributions by X-ray absorption would allow it to be established if a given tile was liable to exhibit manufacturing defects relating to inappropriate powder distribution in the press die cavity.

3.3.1.4. Identification of pressing and firing defects in large-sized ceramic tiles. Fig. 11 shows the thickness and bulk density distributions of two smooth porcelain tiles with a nominal size of 45 cm × 67 cm, obtained in an industrial press fitted with a die with a double matrix, with isostatic bottom punches. The tile pressed in cavity 1 exhibits a homogeneous bulk density distribution; in contrast, the tile pressed in cavity 2 displays a heterogeneous bulk density distribution, owing to inappropriate operation of the isostatic punch, which led to wedging defects in the end-product.

The interpretation of the wedging defect can be completed with the information provided by the thickness distributions in both tiles. It may be observed that tile 1 displays very pronounced differences in thickness, related to the compensation of pressures applied by the isostatic punch on the powder bed. The effect of the isostatic punch led to a density distribution with average maximum differences in the tile of less than 20 kg/m^3. When tile 2 was pressed, however, this compensation did not occur, a

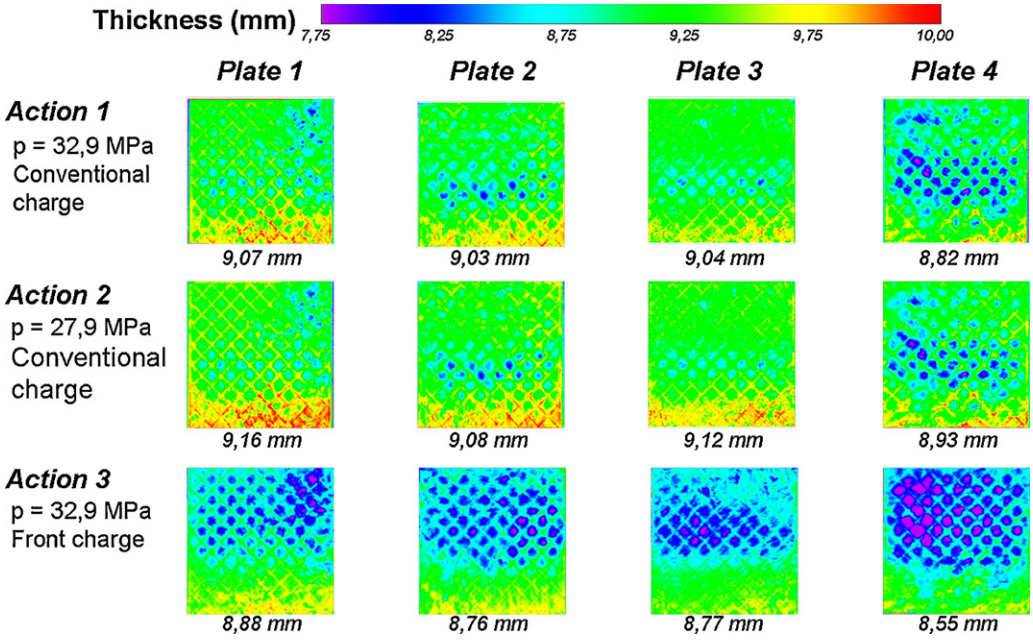

Fig. 9. Thickness distributions in tiles obtained under different pressing conditions.

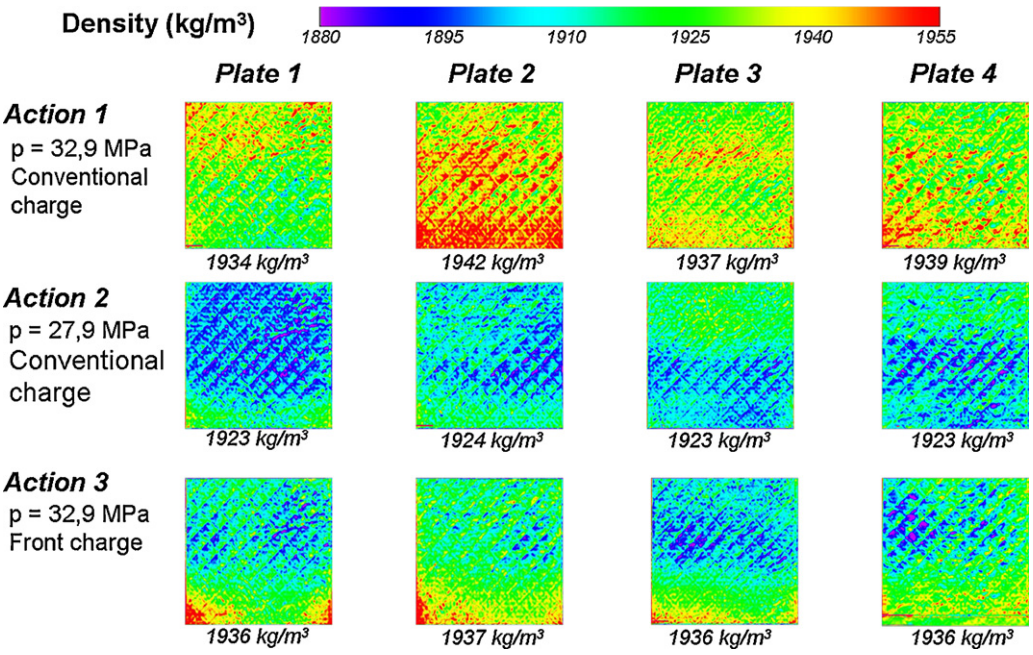

Fig. 10. Bulk density distributions in tiles obtained under different pressing conditions.

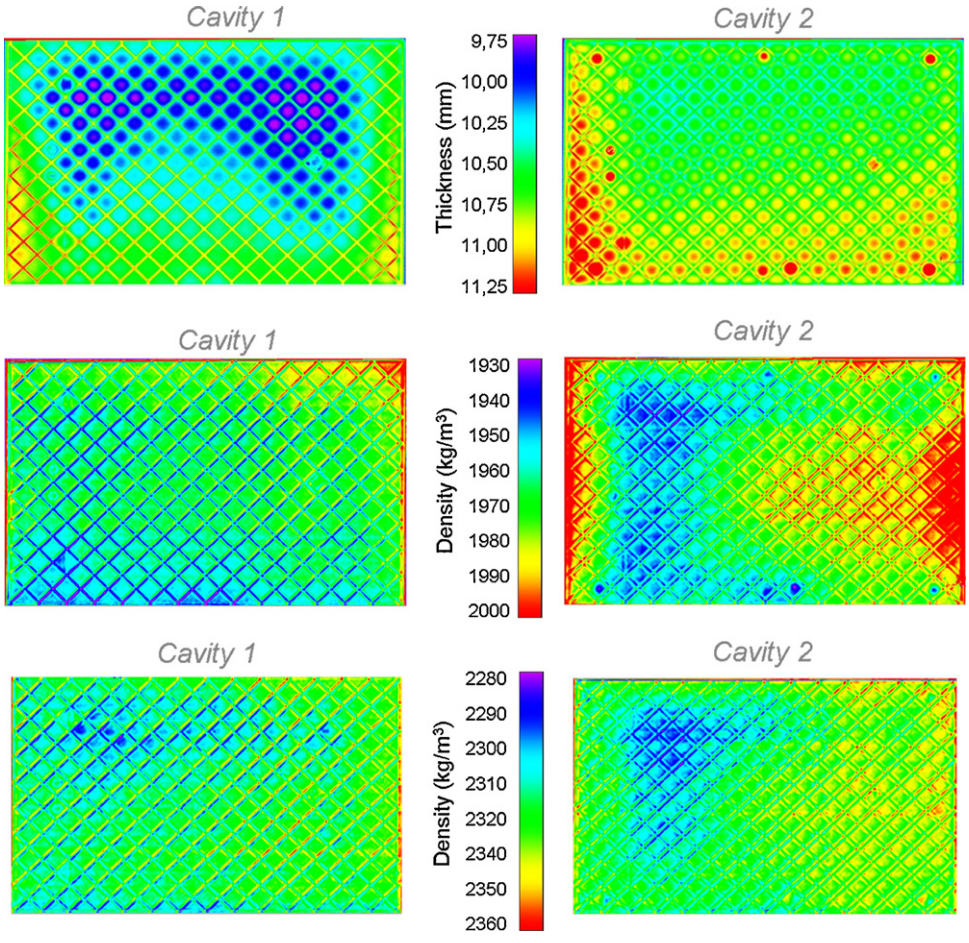

Fig. 11. From top to bottom, distribution of dry thickness, dry bulk density, and fired bulk density of two porcelain tiles.

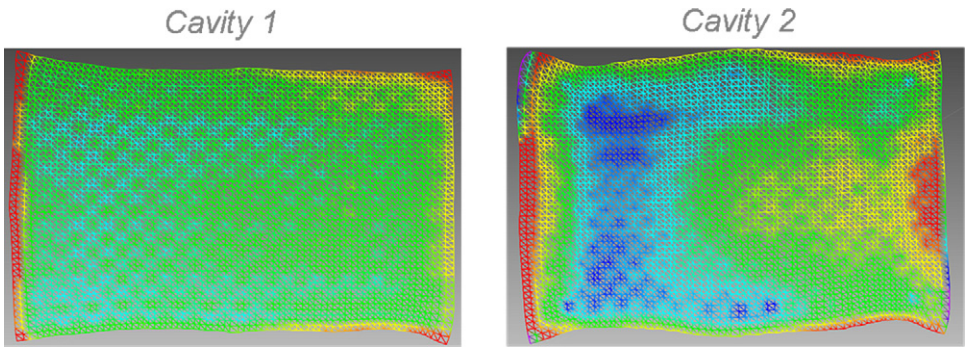

Fig. 12. Qualitative maps of strain estimated from the bulk density measurements.

tile being produced with an average bulk density very similar to that of the tile obtained in die cavity 1, but with a higher average thickness and a bulk density distribution with average maximum differences of about 50 kg/m^3.

The non-destructive character of the method enabled the same tiles to be analysed after they had been subjected to an industrial firing cycle with a peak temperature of 1200 °C, and the influence of the sintering process on end-product properties thus to be evaluated. The fired bulk density distributions of the two studied tiles are shown in Fig. 11. The fired density distributions show that the heterogeneities produced in pressing in piece 2 remained present after firing, albeit to a lesser extent, as a result of the minor influence of dry density on the fired density exhibited by porcelain tile compositions at high firing temperatures.[11]

The example provided demonstrates the usefulness of the X-ray absorption method in identifying, among other matters, the origin of manufacturing defects relating to lack of dimensional stability in the end-product. Indeed, the possibility of characterising the same tiles, before and after firing, enables it to be established whether a given defect is due to an inappropriate pressing operation or, in contrast, is caused by inappropriate kiln settings.

3.3.1.5. Estimation of the strains undergone during firing from the map of dry bulk densities. As a first step for future studies, the strains were estimated that the tiles examined in the foregoing section would undergo during firing, taking into account that porcelain tile compositions display a firing shrinkage that is practically independent of peak firing temperature.[12] In order to obtain the tile strain through the density map, mechanical calculations were made using the finite element method (FEM). The FEM model used assumes that the tile behaves as linear elastic solid, which is constructed by means of a regular mesh of small rectangular elements. Each element can undergo isotropic shrinkage (or expansion) proportional to the density that is experimentally determined by X-ray absorption. The imposition of this field of elementary isotropic strains produces a pre-stressed profile inside the tile, and provides the overall tile strain.

Fig. 12 shows, qualitatively, the strains experienced by the meshes corresponding to the two studied tiles, overlaid on the dry bulk density maps of each tile. It may be observed that the tile with the greatest differences in bulk density (tile 2) displays strains that are more heterogeneous. The model used needs to be improved and validated with experimental data in order to be able, quantitatively, to estimate the strains that the tiles will undergo during firing, based on the density and thickness distributions of the unfired tile.

4. Conclusions

The following conclusions may be drawn from the study:

- An apparatus has been designed and built, based on the X-ray absorption technique, which allows bulk density distributions to be precisely and non-destructively measured in large-sized ceramic tiles.
- The apparatus provides complementary information on thickness and surface mass distribution, which is of great interest for the optimisation of the ceramic tile forming process.
- It has been verified that the apparatus enables defects relating to heterogeneous bulk density distribution caused by an inappropriate pressing operation to be detected.
- The non-destructive character of the apparatus allows fired tiles, which had previously been characterised in an unfired state, to be examined with a view to evaluating the effect of the firing and pressing steps on end-product properties.
- A simple behaviour model has been developed that allows the shrinkages that porcelain tiles will undergo during firing to be qualitatively estimated from the tile dry bulk density distribution.

Acknowledgements

The authors of this paper wish to thank IMPIVA for its financial support in conducting the study, through the programme for the support of R&D in technology centres of the Impiva network.

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