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# A non-destructive method to assess delamination of ceramic tiles

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**Abstract**

Delamination is a typical defect of ceramic tiles. It originates from the presence of trapped air during pressing, and elastic recovery of the pressed material, which prevents the consolidation of the pressed material. Although delamination greatly reduces the quality of the final product, standard tests for ceramic tiles are not currently available.

In this work the problem of detecting delamination of green and fired ceramic tiles is approached by using ultrasonic pulse velocity measurement, a non-destructive technique normally used to assess concrete structures and the density of ceramic tiles.

It was assumed that an increase in travel time for a given thickness (i.e., decrease in travel velocity) would indicate the presence of delamination, as the sound wave has to travels around the flat pore.

The problems connected with measurement uncertainty and repeatability are discussed in the paper, in particular concerning the standardisation of the measurement.


*Keywords:* Ceramic tiles; Pressing

## 1. Introduction

Delamination cracks are well known in heavy clay extruded products, due to particle orientation and formation of slip lines.[1] However delamination occurs also in wet pressed granular bodies, due to air being trapped while pressing which prevents consolidation.

The major causes of entrapped air in the pressed tiles are believed to be:[2]

- fast pressing cycles (not enough time given for the entrapped air bubbles to escape);
- poor filling of the die (creation of voids or turbulence);
- decreasing granulate size and low moisture content (higher amount of entrapped air);
- elastic recovery during ejection (especially if low amount of plastic material or organic binder is used).

Delamination often appears at the beginning of each pressing cycle (when dies are changed), during the set up of new technologies (press or spray dryer or mill) or by testing new powder compositions.

Changing dies can take 8 h. Much of this time lost is associated with the trial and error adjustments to achieve consistent pressed tiles. According to Bendanti,[3] the economical loss due to long die changes could be 2100 $m^2$ double pressed tiles (=105 k€, if selling price is 50 €/$m^2$) or 3360 $m^2$ once fired tiles. If the cost of delamination is 1% of the total cost associated with the conventional die changes, then the cost of delamination can be as high as 1000 € every time the format is changed.

Non visible delamination is also believed to be the first cause of increase the wastage whilst cutting and installing tiles and flaking of the surface of tiles.

A short inquiry has been carried out among press producers, asking about the real incidence of delamination during the production of new tiles.[4] They said that delamination is a historical problem for ceramic tiles and it is still persisting; it implies costs due to the not sold defective tiles and to their production costs. The operators still use a destructive method to assess the defect presence each time a pressing parameter is changed, checking the first tiles coming out from the press. During the producing

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cycle usually periodic tests are carried out in order to keep the quality under control.

Although the cost of delamination of ceramic tiles can be considerable, at the moment no standard tests are available to measure the presence of delamination in ceramic tiles.

Quality control inspections of typical glaze and tile's body defects are usually carried out by destroying the tiles.[5] However, demand for product performance reliability and escalating cost for statistics derived by destructive evaluation are the main reason why non-destructive testing techniques should be considered for assessing delamination and other defects in tiles.

Thus, this article proposes to use ultrasonic pulse velocity (UPV) as a non-destructive test to detect delamination. In particular, this article contains the steps followed to establish a quantitative method to detect delaminated tiles, but with a cost lower than the delaminated costs.

## 2. Background on ultrasonic testing

The velocity of ultrasonic pulses, travelling in a solid material, depends on the density, elastic properties and on the different phases present inside the material, thus the quality could be related to its measurement. Ultrasonic testing is applied to measure elastic stiffness, mechanical properties and defects' presence in concrete, refractories and ceramic materials, etc.[6–9]

The ultrasonic pulse velocity (UPV) technique requires an electronic instrument and a pair of ultrasonic transducers. The instrument generates a high voltage, short duration (up to 1200 V for 1.5 μs) electrical pulse which is converted by one transducer into an ultrasonic signal. The other transducer detects the ultrasonic signal and converts it back into an electrical signal. The instrument then measures the time taken (known as "transit time" and usually measured in microseconds) for the pulse to travel between the two transducers. This test relies on the fact that the speed of the ultrasound is much higher in the test material than it is in air. Any air in the signal path (due to cracks or voids, etc.) causes a reduction in the pulse velocity and thus an increase in transit time. In non defective material, the ultrasonic signal travels in a straight line from the transmitting transducer to the receiving transducer. Testing known good samples establishes a reference measurement. If a much larger transit time is measured on the same sample it is easy to say that the signal has had to travel around or through some defects. Drawbacks of the method are:

- It requires a good mechanical 'bond' between the test material and the transducers. Failure to achieve this can result in completely erroneous test results. The effect of even a tiny pocket of air between the test material and the transducers is the same as crack in the product. To overcome this, coupling material is not possible to use because this would imply a loss of speed in testing big amount of tiles every day. For this application small transducers with rubber tips are available.
- Some variations between operators can be experienced due to the different pressure applied by them to the transducers. This has the effect of varying the thickness of the rubber tips and so reducing the distance travelled by the ultrasonic pulse. Too high pressure might damage the rubber tips or deform them; too low pressure might not assure a good adherence between sample and transducer. This will be overcome with a rig able to apply a steady pressure between the transducer and currying out the most suitable working pressure.
- Other variations may depend on the correct alignment between the transducers; this has the effect of varying the path the pulse has to travel across the sample, increasing the crossing time. This problem will be overcome using the same rig described before.
- The choice of the transducer is also an important consideration. Transducer frequencies for use with the instrument used in this work range from 24 kHz to 1 MHz. Frequencies used for ceramics are generally in the 150–500 kHz range which corresponds to wavelengths of around 200–16 mm. The wavelength determines the size of the smallest defect that can be detected. The physical size of the transducer may also be a factor in some applications. To overcome this problem 220 kHz transducers, with 6 mm diameter tip, will be used.

All the values carried out in this work require an accuracy of some tens of microseconds, thus having a steady measure is an essential requisite.

## 3. Experimental procedure

### *3.1. Materials*

Two bodies used in the delamination tests were supplied in granular form by two tile manufacturers:

1. unglazed floor tiles BIIa (water absorption between 0.5% and 3%),
2. porcelain floor tiles BIa (water absorption between 0.01% and 0.5%).

A two-stage pressing was used to prepare the delaminated samples. A first tile powder layer (20 g) has been pressed at about 100 kg/cm$^2$. Then 0.10 g of alumina powder positioned in a central position without spreading + 20 g of tile powder have been added and pressed at 200 kg/cm$^2$. The same procedure has been followed to produce samples with 0.20 and 0.30 g of alumina powder.

Samples with no alumina have also been prepared.

Alumina was added to simulate the delamination of the tiles. Addition of a small amount of alumina powder is expected to impede the contact of the two clay-based layers during firing. As for delamination, alumina will act as a flat defect that delays the passage of the sound wave across the tile. SEM pictures of the cross-section of the tiles were taken to confirm the delamination of the tiles induced by alumina addition.

The second set of delaminated samples (porcelain tiles formulation BIa) were obtained similarly, although the sample were pressed at 400 kg/cm$^2$.

The pressed samples (discs of about 4.6 cm diameter and 0.8 cm thickness) were dried overnight at 110 °C and sintered. The sintering cycle was for both tiles: 20 °C/h from room temperature until 110 °C, dwell for 4 h, then 100 °C/h until 1140 °C and dwell for 5 h, natural cooling until room temperature.

The fired samples were characterised by water absorption and mercury density.

The water absorption test has been carried out following the European standard (EN ISO 10545-3). The density with mercury was measured at controlled temperature (19 °C) and equilibration time of 30 min before the weight was recorded.

### 3.2. Ultrasonic pulse velocity (UPV) technique

The instruments used in this work were the PUNDIT and PUNDIT PLUS (CNS Farnell, Borehamwood, UK). The name PUNDIT derives from the initial letters of the full title of "Portable Non-destructive Digital Indicating Tester".[10]

The apparatus generates low frequency ultrasonic pulses and measures the time taken for them to pass from one transducer to the other through the material interposed between them. The range of time measurement used in this work was 0.1–999.9 μs in units of 0.1 μs. To avoid loss of acoustic signal at the transducer/tile interfaces, rubber tips were used to promote a good adherence. With the rubber tips, the last digit oscillates between two adjacent values. A standard reference with an accurately known transmission time of about 26 μs (26.3 μs in this case) was constantly used to check the reproducibility of the measurements.

The transducers consist of ceramic piezoelectric elements mounted in a stainless steel cases. The shock excitation of the pulse generator causes the transducer to oscillate mechanically at its own frequency depending on the size and stiffness of the whole transducer assembly. Thus, the elements were tightly mounted on the inside face of the case to provide highly efficient acoustic transmission.

The transducers were used in the transmission mode, i.e., facing each other and the sample in the middle. The transducers are connected to the PUNDIT by two coaxial cables.

### 3.3. Pressure managing rig

The measurements with the PUNDIT are usually taken by manually applying the transducers to the samples. The manual procedure is usually accurate for detecting cracks in concrete and other similar aggregate materials. However, it was soon verified that the manual alignment of the transducers and to rubber tips elasticity introduced unacceptable errors in the measurements of delamination of ceramic tiles, due to the small thickness of the tiles and reduced size of the defects. Therefore, a pressure managing rig was constructed to avoid operator error in applying the transducers (Fig. 1). The rig also enabled the transducers to be aligned and avoid unwanted displacement during the measurement. Furthermore, the rig applied a pre-selected constant pressure, keeping the rubber tips' strain constant, thus avoiding to artificially changing the distance between the transducers.

One transducer is fixed on a high stiffness steel bar; the second one is placed at the top of a mobile metal pipe and is allowed to move only in the same direction of the first transducer. Two cables connect the bottom of the pipe holding the sample with a variable weight. It is possible to change the pressure between the transducers by varying the weight. The second transducer and the metal pipe are kept perfectly aligned with the first transducer by a high stiffness steel pipe, fixed to the main basement by two steel bars. The operator has only to position the sample between the two transducers, shifting it up and down or left and right in order to take crossing time measurements. It is possible to move the sample retracting the plastic pipe, moving the sample and releasing the pipe, for each measurement. Good contact is ensured by the rubber tips and the pressure.

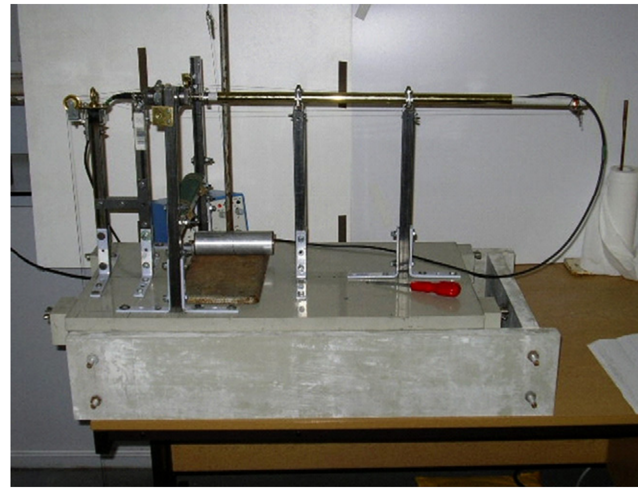

Fig. 1. Pressure managing rig.

### 3.4. Sample measuring procedure

The measuring procedure is divided into two steps: determination of the number of measurements needed for scanning the tile (meshing size) and the analysis of the UPV measurements.

The selection of the meshing size is clearly a compromise between the accuracy of the measurement and time needed to perform the measurement. Taking into account the diameter of the rubber tip (6 mm) and the diameter of the tile (46 mm), it was decided to mesh the tile in 30 elements (each of them of the same size of the rubber tip), equally scattered on the tile surface. Furthermore, no mesh elements covered the border, as in this case the presence of delamination can be checked visually.

The measurements were carried out by positioning the tile between the two probes and apply 2 kg in the rig. In general, 30 UPV measurements per tile were taken and the standard deviation of the velocity calculated.[11] It is expected that samples with no defects should show very low standard deviation values, while delaminated samples with alumina should have higher values.

## 4. Results and discussion

The pictures in Fig. 2(a) and (b) show that the introduction of alumina in the core of unglazed and porcelain tiles effectively provoked delamination. We can clearly see the delamination in the centre of the tile in the sample at the left and a defect that starts from the middle and arrives at the border in the sample at the right. In the figure the values of UPV measurements per

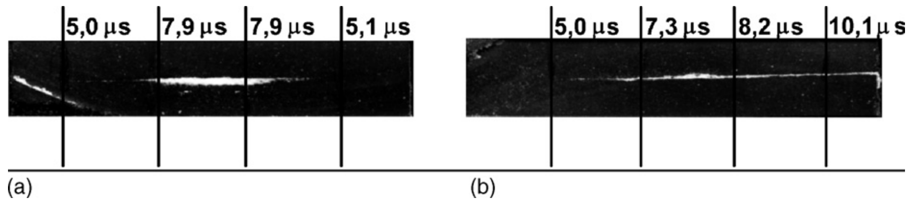


Fig. 2. SEM picture of a cross-section of delaminated porcelain tiles with (a) 0.1 g and (b) 0.3 g of alumina.

tile are shown. Qualitatively an increase of the time is clearly observable with the increase of the delamination.

### 4.1. Water absorption and density

Water absorption results are expressed through both water absorption percentage and density.

Unglazed floor tiles have shown a higher density and lower water absorption value than normal industrial production, probably due to the stronger condition of pressing and firing, carried out in laboratory conditions.

As shown in Fig. 3(a), the values are included between 0.5% and 3%, thus belonging to BIIa category.

Water absorption of unglazed floor tiles seems to slightly increases with increasing alumina content. However, some tiles without alumina showed higher water absorption. As the alumina was placed in the centre of the tiles, we should not expect any particular relationship between water absorption versus alumina content. Thus, we can conclude that water adsorption is probably able to detect delamination if a high number of BIIa tiles are tested. However water adsorption is not accurate enough to detect delamination in the single tiles.

Water absorption tests confirm that the porcelain floor tiles belonged to the BIa category. The results collected in Fig. 3(b) demonstrated that water has not been able to reach the core of the tile and the delamination gap. Therefore, water absorption is unsuitable for detecting voids or pores inside fully vitrified tiles.

Mercury density values are in good agreement with water absorption densities, confirming the results' quality. Fig. 4(a) and (b) showed that the density measure with the mercury almost overlap the value obtained with water adsorption. Again, density measurements with mercury are unable to detect delamination in porcelain tiles.

### 4.2. Ultrasonic pulse velocity (UVP)

Unglazed floor tiles show (Fig. 5(a)) a wide gap between the UPV readings of delaminated and not delaminated tiles. This difference is higher with increasing amount of alumina. The same relationship between standard deviation and amount of alumina was also observed for the porcelain tiles (Fig. 5(b)).

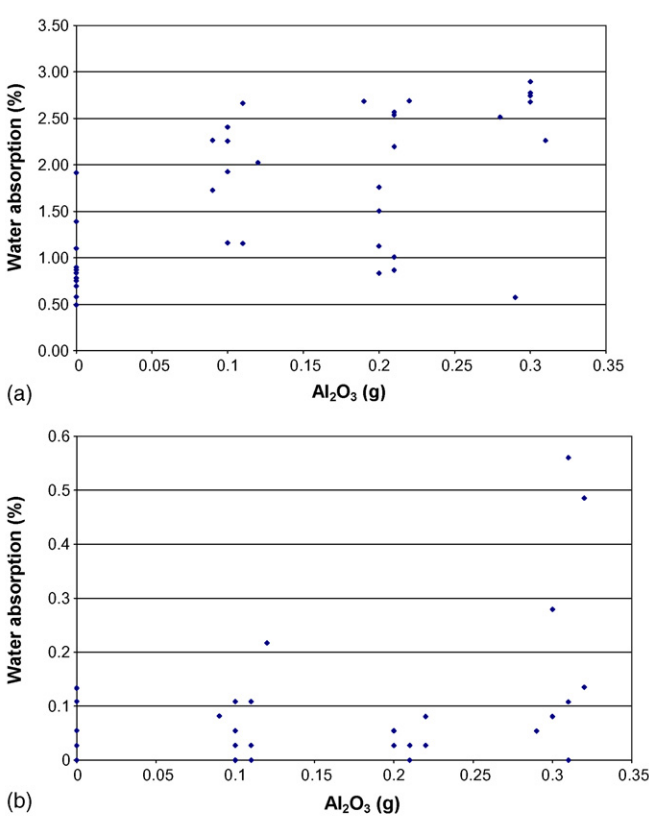


Fig. 3. Water absorption of (a) unglazed and (b) porcelain floor tiles.

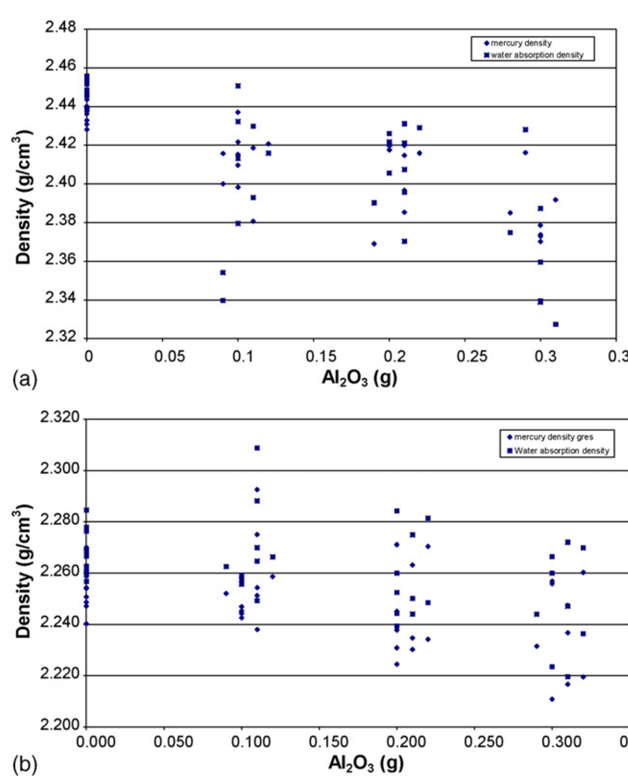


Fig. 4. Comparison between mercury density and water absorption density calculated for (a) unglazed and (b) porcelain floor tiles.

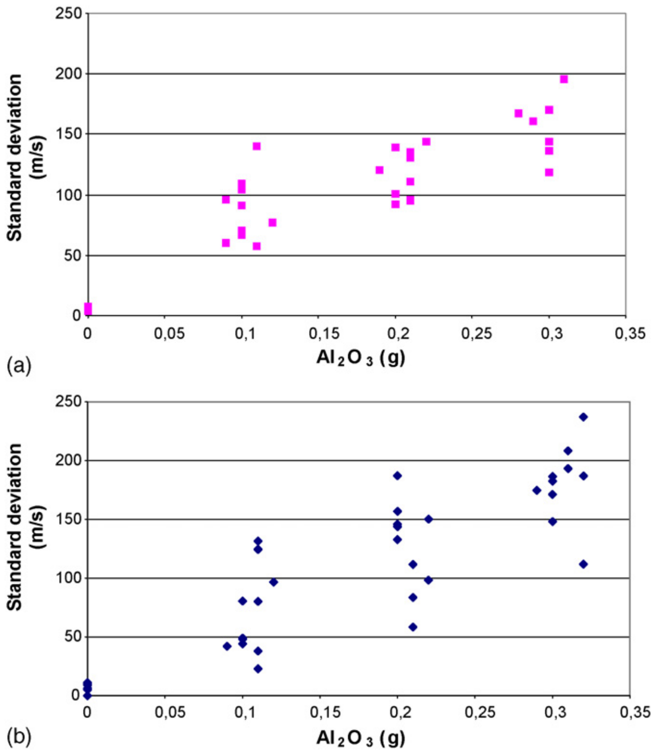


Fig. 5. Standard deviation for crossing velocity on (a) unglazed floor tiles and (b) porcelain floor tiles.

These differences in standard deviation are even higher if the background (7.3–7.5 μs) is discounted from the absolute reading of the crossing time. For each tile it is now possible to count how many speed differences are higher than 50 μs. This value has been assumed to be representative of the presence of high inhomogeneity in the tile, i.e., delamination.

Fig. 6(a) and (b) show this count, making the defected samples more visible.

In order to assess if these measure are due to real defects, their position on the surface has been checked. In all analysed porcelain tile samples, as shown in Fig. 7 (a) and (b), these measures are gathered and not equally scattered, demonstrating that they are due to flaw. In some cases, more measurements have been taken in the critical areas to confirm that they are not due to operator or instrument error. The samples shown in Fig. 7 (a) and (b) are characterized by a slightly difference between background crossing time and some particular local crossing times, but this difference (thanks to the pressure managing rig) is large enough to identify the presence of a flaw, as all the measurements are grouped in a small area.

This ability to focus on small areas is a great characteristic of UPV technique, because it permits to the operator to analyze particular parts of the tiles; besides, considering that tiles can have surfaces ranging from some tenths of centimetres square

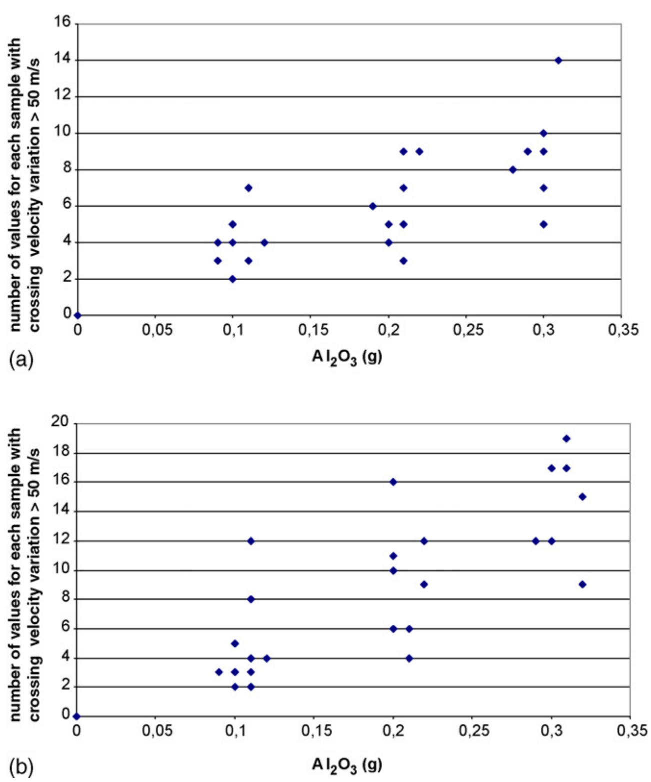


Fig. 6. Number of values for each sample with crossing velocity gap bigger than 50 μs for (a) unglazed and (b) porcelain floor tiles.

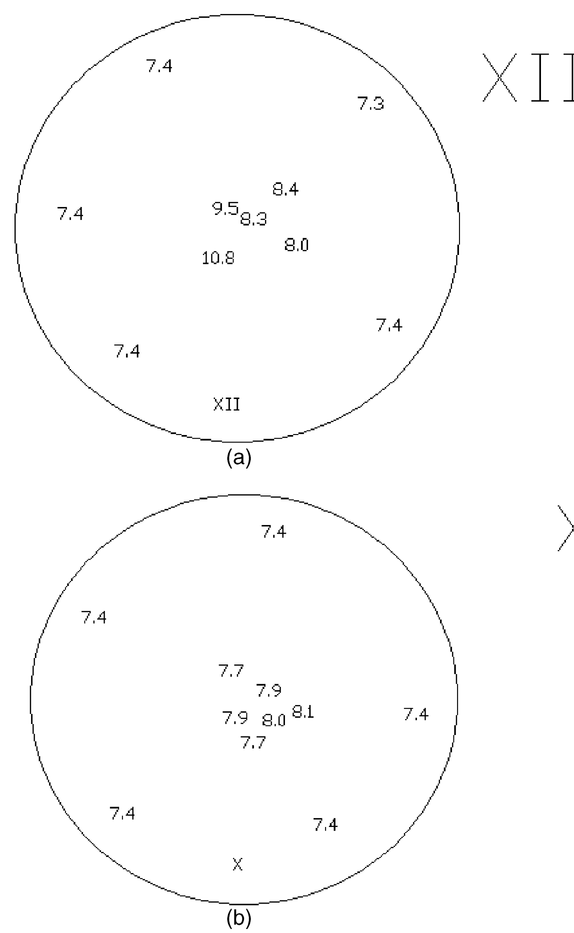


Fig. 7. Graphical representation of values distribution on (a) unglazed and (b) porcelain floor tiles.

to one metre square and more, the standard deviation could not be sufficient to assess tiles quality; thus it is possible to focus the measurements on an area close to abnormal values and carry out a local analysis of the surface as it was a smaller tile.

## 5. Conclusions

Ultrasonic pulse velocity (UPV) has been used to assess delamination on unglazed (BIIa) and porcelain floor tiles samples (BIa).

UPV seems a sufficiently sensitive and reliable technique to detect delamination in ceramic tiles. However some development work still needs to be carried out to standardise the procedure. An opportunity exists to transfer a standard test and good practice guide in use for concrete to ceramic tiles. This approach utilises existing procedures and possibly avoids having to "reinvent the wheel".

Ultimately this test can be proposed as a new standard for glazed (or non-glazed) ceramic tiles to fulfil CE marking requirements.

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