Non destructive evaluation of glaze delaminations in glazed ceramic tiles: laboratory tests

Paola Calicchia
Institute of Acoustics and Sensors "O. M. Corbino" CNR, Rome, Italy, paola.calicchia@idasc.cnr.it

Renan Guimarães Barbosa Trivelli
University of Rome "Tor Vergata", Rome, Italy, renantrivelli@hotmail.com

Sara De Simone
Institute of Acoustics and Sensors "O. M. Corbino" CNR, Rome, Italy, sara.desimone@idasc.cnr.it

Lucilla Di Marcoberardino
Institute of Acoustics and Sensors "O. M. Corbino" CNR, Rome, Italy, lucilla.dimarcoberardino@idasc.cnr.it

Patrizio Verardi
Institute of Acoustics and Sensors "O. M. Corbino" CNR, Rome, Italy, patrizio.verardi@idasc.cnr.it

SUMMARY: Glazed ceramic tile panels, azulejos, is a very distinctive character of the extraordinarily rich heritage of Portugal. Particular attention is therefore required to study the causes of the decay process, the progress of its effects, and the definition of suitable procedures concerning the conservation practice.

Issues related to the presence of glaze delamination are taken into account, attempting to find some possible solutions to the early detection of sub-surface air cavities.

In the present paper the basic approach for an extensive laboratory validation of two non contact techniques employing an acoustic excitation source is presented. Using the same acoustic excitation source, an innovative highly directive loudspeaker, the tests provided the velocity of the induced vibrations in a collection of samples by means of a Laser Doppler Vibrometer, correlated to the presence of acoustic energy absorption revealed by means of the Acoustic Absorption diagnostic system. This study compares the acoustic response of a set of historical azulejo samples and of laboratory models with artificial delaminations, and collects preliminary results useful to optimize the experimental settings for a more extensive validation of the acoustical methods.

KEY-WORDS: Acoustic Absorption diagnostics; Laser Doppler Vibrometer; Glaze delamination; Historical Azulejos; Tile models

1. INTRODUCTION

The art of Ceramic represents a characteristic element of the handicraft tradition of many countries and a legacy to preserve. The use of glazed ceramic tile panels, azulejos, as architectural finishing actually gives a very distinctive character to the extraordinarily rich heritage of Portugal. Particular attention is therefore required to study the causes of the decay process and the progress of its effects, as well as the definition of suitable procedures concerning the conservation practice.

Indeed azulejos are a durable architectural finishing; nevertheless their durability can be compromised due to manufacturing defects. The most critical of them is called "shivering" and derives from a mismatch between the coefficient of thermal expansion of the glazed layer and that of the clay body when, after the second firing, the clay contracts much more than the glaze. This aspect plays a fundamental role during the cooling phase when a compression is induced on the glaze which can locally be detached from the clay body, creating a thin cavity of air at the interface (this phenomenon is called "shivering"). Commonly this defect goes un-noticed but from a slight modification in the glaze surface flatness. When the clay absorbs moisture containing soluble salts from the wall, and if the glaze has a pore on the delaminated area, the water will evaporate and salt crystallize inside the air cavity, pushing the glaze up until it breaks and falls off. An initial lacuna in the decoration is thus formed whose area can be of several square centimetres [1], as shown in Figure 1(a). If not promptly treated, the degradation can develop from a limited lacuna till the complete loss of the glazed layer with its pictorial content.

Although some efforts have been done, at present there is no defined protocol including instrumental diagnosis to reveal glaze delamination, in particular in the early stages. On the other hand, new restoration treatments and innovative materials for preventive conservation have been developed in the framework of recent projects [1]. The development of reliable non destructive methods able to detect glaze delaminations during the early stage of the decay process, i.e. when the tiles appear not deteriorated to a visual inspection but could hide sub-surface cavities, would make any conservative action more effective and durable.

Several studies concern different diagnostic methods such as the ultrasonic pulse velocity (UPV) technique [2], that correlates the decrease in the travel velocity of the transmitted wave for a given tile thickness with the presence of delamination. When searching for non-destructive techniques applied to the industrial manufacturing process the time of flight of the ultrasonic waves is indeed widely used to detect defects in modern glazed ceramics [3], but a potential drawback if applied to historical tiles often lies in the fact that this technique does not employ contact-less transducers. More interesting for the art objects is the acoustic imaging technique using non-contact ultrasonic transducers (NCU), often applied to panel paintings [4], that needs to be validated for historical azulejos. Other non-destructive approaches include IR thermography.

Compared to other techniques, the acoustical methods are particularly effective when applied to multilayer structures such as frescoes, ceramics and panel paintings, affected by detachments and flaws. Acoustically speaking, a detachment is a sub-surface air cavity vibrating at specific frequencies when it is excited by an external acoustic pressure field. The characteristic resonance frequency of a sub-surface air cavity depends on the density of air, on the density and thickness of the surface layer, and on the cavity depth. Actually a vibrating cavity also behaves as a selective acoustic absorber, dissipating a certain amount of the incident acoustic energy into heat.

A possible solution to the localization of delaminations has been already investigated by the authors during on site diagnostics by means of an acoustic imaging technique based on acoustic absorption determination [5]. Figure 1(b) shows one of the acoustic images obtained on azulejos in the renaissance cloister of the Madre de Deus Convent, in Lisbon, hosting the Portuguese National Tile Museum (MNAz). This study indicated a potential usefulness of this technique and confirmed the relevance of the acoustical methodologies for the specific problem of glaze delamination. Although the field experimentation showed very promising results, a laboratory validation is highly recommended in order to assess the reliability of the acoustical method.

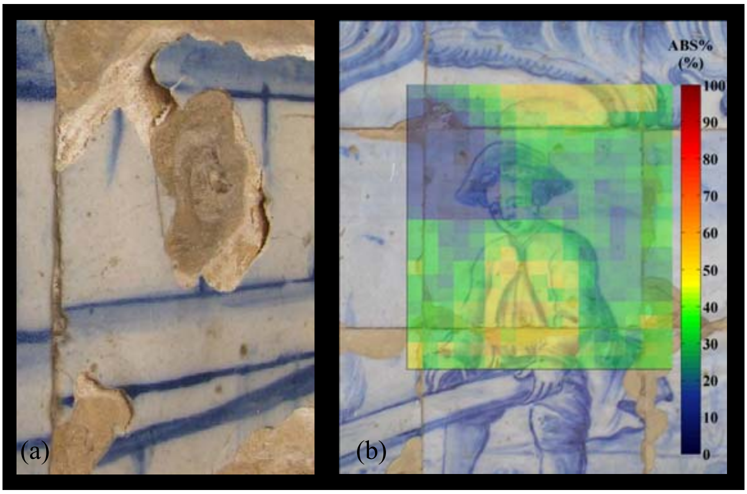


FIG.1: Example of glaze loss through efflorescence (a); acoustic absorption image on an azulejo panel (b) [5].

In the present paper the basic approach for an extensive validation of two non contact techniques employing an acoustic excitation source is presented. The Laboratory of Acoustics Research applications for Cultural Heritage (LARCH) at the Institute of Acoustics and Sensors "O. M. Corbino" of the National Research Council (CNR - IDASC) was equipped with a Laser Doppler Vibrometer (hereafter LDV) and the system denominated ACoustic Energy Absorption Diagnostic Device (hereafter ACEADD) [6]. Using the same acoustic excitation source, an innovative highly directive loudspeaker, the tests provided the velocity of the induced vibrations in a collection of samples by means of the LDV, correlated to the presence of acoustic energy absorption revealed by means of the ACEADD system. This study compares the acoustic response of a set of historical azulejo samples and of laboratory models with artificial delaminations, and collects preliminary results useful to optimize the experimental settings for a wider laboratory validation of the acoustical methods.

In the following sections the set of the investigated samples is described summarizing their main features; a brief recall of the experimental methods together with a description of the acoustic excitation source is successively provided; finally the experimental results constituting the principal objective of this preliminary study are presented, and issues concerning the accuracy and repeatability of the measurements are also tackled.

2. MATERIALS AND EXPERIMENTAL METHODS

A set of samples with different characteristics and different degree of delamination were collected among historical Portuguese azulejos. To classify the samples the presence or absence of glaze delamination, assumed by visual inspection, will be evidenced. The identification of the chemical components of the analyzed samples, the manufacturing process or the possible causes of deterioration will be not taken into account, although fundamental, since these aspects are beyond the scope of the present work.

Few laboratory models were prepared to simulate tiles with air cavities under the glazed layer. These physical models are expected to track the relationship between the presence of the cavity and its response to the acoustic excitation; besides they will be also useful in future work to determine the sensitivity of the methods to the size of the cavity and for an insight into the validation of a suitable mathematical model.

In the present investigation a restricted number of samples are presented and analyzed, in particular those considered the most representative among the entire collection of tiles.

In the following sections a description of the historical azulejo samples and the laboratory models are reported.

Successively two non destructive methods based on acoustic excitation are presented. A Single Point LDV will allow a point analysis of the vibration velocity induced by a proper acoustic source in different points on the investigated samples, thus helping the discrimination of the presence of sub-surface cavities. The ACEADD system is employed to measure the absorption of acoustic energy in the investigated samples, attempting to assess the potential discrimination of delaminated from non-delaminated tiles through a nominally low cost device.

2.1. Historical azulejo samples and laboratory models

Four Portuguese azulejos from the Lisbon area, dated between the 17th and the 18th century, constitute the set of historical tile samples studied by means of non contact acoustical methods. All the samples are single tiles or part of a tile. A number of these seem to present good adhesion of the glazed layer to a visual inspection, and will be analysed in order to classify them as suitable reference tiles.

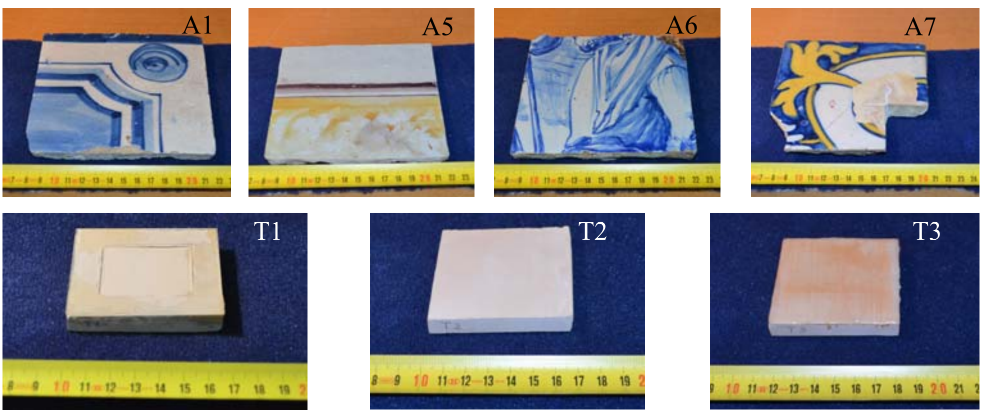


FIG.2: The set of historical azulejo samples (top) and the laboratory models (bottom).

Three laboratory models were prepared to constitute an initial and suitable set of test tiles, all built on a substrate 6.8 cm × 6.8 cm × 1.0 cm made of clay, one of them presenting an artificial delamination. The tile T2 is constituted by the simple clay substrate while the other two, T1 and T3, were assembled gluing thin glasses on the substrate. For the glazed model T1 few pieces of glass (ordinary glass for microscope, 0.8 mm thick) were cut and fixed on the substrate in order to build the side walls of a square cavity; a thin glass SCHOTT D 263 LAeco (0.7 mm thickness; 2.51 g/cm^3 density) was finally fixed as capping layer upon the previous layer. For the glazed model T3 the sole capping layer of thin glass SCHOTT D 263 was fixed on the substrate using epoxy glue on the entire surface. The preparation of the glazed models was carried out at room temperature using epoxy glue with medium drying time in order to prevent cracks during the drying process. The images of the collection of samples are shown in Figure 2, while their main features are summarized in Table 1.

Tab.1: Set of historical azulejo samples and laboratory models, with their main features.

ID	MATERIALS	SIZE (mm^2)	THICKNESS (mm)	NOTES
A1	clay body and glaze white and blue	143 × 143	11.6	Tile with no apparent delamination
A5	clay body and glaze white, dark yellow and red	140 × 140	11.3	Tile with no apparent delamination
A6	clay body and glaze white and blue	140 × 140	13.6	Tile with no apparent delamination
A7	clay body and glaze white, yellow and blue	140 × 140	14.3	Tile with significant delamination, cut sector
T1	clay body and glass	68 × 68 substrate 42 × 42 square cavity	9.65 – clay substrate 0.8 - 1st layer glass 0.7 – cap layer glass 2 layers of glue 11.15 - total thickness 1.05 – cavity depth	Model of tile with an air cavity in the centre
T2	clay body	68 × 68 substrate	9.80 – clay substrate	Reference model of substrate with no glazed layer
T3	clay body and glass	68 × 68 substrate 60 × 60 square glass	9.65 – clay substrate 0.7 – cap layer glass 1 layers of glue 11.15 - total thickness	Reference model of substrate with glazed layer

2.2. Experimental methods

When an external acoustic pressure field excites an air cavity beneath a superficial layer with a suitable frequency, this structure becomes a vibrating element. If the vibration is relevant, upon the occurrence of a resonance, also an acoustic energy absorption occurs since the vibrating element dissipates energy transforming it into heat. The present study analyses this phenomenon following the induced vibration in a collection of tiles, few of them affected by glaze delamination. Thus the vibration velocity, measured by the LDV, and the absorption of acoustic energy, measured by the ACEADD method, have been identified as significant physical quantities for describing the phenomenon in the presence of a suitable acoustic excitation source.

The basic approach for the laboratory validation foresees different phases. The first phase deals with the assessment of the reliability of the measuring procedures, obtaining important indications for the second part of the investigation. The main objective of the first phase is in the answering to the following fundamental questions:

- Is the acoustic excitation suitable to excite characteristic resonances of the objects under study? Is the frequency response of the source suitable for this task?
- Is the acoustic response of the objects under study measurable with the two acoustic methods? Is the sensitivity of two methods suitable to obtain readable data?
- Is the acoustic behaviour of reference tiles clearly recognizable and univocally classified? Is it possible to easily discriminate the acoustic behaviour of damaged from undamaged samples?
- Which are the optimal measuring conditions that emphasize these differences?
- Is the setup configuration employed in the laboratory tests appropriate to field experimentation as well? Can this basic *single tile* diagnostics already provide useful data for a first level analysis, easy to be carried out by non specialized operators?

The second phase of the laboratory validation deals with the optimization of the experimental setup in terms of best frequency tuning of the excitation source, best reference tile selection, and improved post-processing procedure to obtain a high effectiveness of the experimental techniques. A wider collection of test samples is foreseen, in particular a wider series of laboratory models showing a variety of artificial glaze delaminations with different shape and different depth of the air cavity. The main objective of this second part is the configuration of reliable tools providing acoustic images of the investigated objects. In addition the customization of few experimental configurations, to accomplish both the *single tile* and the *extended surface* analysis, is also expected. This last step could differentiate a possible procedure to study, on site, azulejos lining on walls from another possible procedure to study single azulejos after their removal from the wall.

The present paper expressly concerns the results from the first phase of the laboratory validation. These results already highlight possible measuring procedures, useful in giving indications about the glaze delamination phenomenon in an actual safeguarding process. Moreover this initial step helps to correctly plan the successive actions to accomplish an effective validation. Based on the outcomes of the first phase, the second part of the laboratory validation is presently under planning and will constitute the subject of a future work.

Although the two methods both allow non-contact and nondestructive diagnostics, the LDV is suitable for point analysis while the ACEADD device works integrating the acoustic data over a restricted area. Nevertheless this last presents the main advantage in the fact that it is a low cost device. In order to achieve a reliable integration of their results, the same excitation source was employed in the measurements with the LDV system as well as with the ACEADD device. Thus the following paragraph deals with the selected acoustic transducer, a highly directive loudspeaker, and its specific properties. In the successive paragraphs a brief description of the two experimental techniques and the definition of the physical quantities concerned in each method are presented.

2.2.1. Excitation source: the Parametric Acoustic Array

For our application an innovative acoustic source has been used as excitation source, the parametric acoustic array PAA, for its interesting features: a small sized transducer characterized by a very narrow audio beam, that are usually competing characteristics and quite difficult to have together in classical acoustic sources. The PAA generates audio waves (below 20 kHz) in air by emitting ultrasonic waves (around 40 – 60 kHz), based on the nonlinear propagation of finite-amplitude waves, thus the resulting audio beam presents a directivity pattern as narrow as the emitted ultrasonic waves [7]. This property is particularly useful to reduce a limitation of the instrumental spatial resolution due to the geometrical spreading of the acoustic beam, and makes this acoustic source particularly interesting also for on-site applications thanks to its small size. The current commercial parametric sources usually work in the so-called *self-demodulation* regime, i.e. an ultrasonic primary carrier frequency beam f_c is delivered to the transducer with a low audio frequency amplitude modulation, f_m. At a certain distance only a narrow beam carrying the audio frequency modulation, f_m, persists.

In the following tests the excitation source is a commercial PAA, the Holosonics HAS8: a flat source of 20 cm × 20 cm equipped with a control unit that delivers the composite signal to the transducer with carrier frequency f_c = 63 kHz; the control unit allows to regulate the output level and to balance the low frequency and high frequency content of the output signal [8]. After having regulated the balance of low-frequency and high-frequency output level, in order to obtain a suitable frequency response in the range 1 kHz up to 16 kHz, the HAS8 emission was studied before employing the source in the measurements on the test samples. For this purpose a ¼-inch free-field microphone with a nominal sensitivity of 50 mV/Pa was used to determine the Sound Pressure Level SPL at different frequencies, along the central axis of the source. Figure 3 shows the HAS8 frequency response revealed at two distances from the transducer surface, 20 cm and 50 cm.

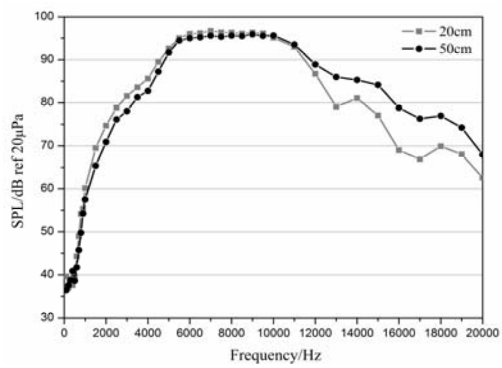


FIG.3: Sound Pressure Level measured at two significant distances from the HAS8 acoustic source along the central axis.

2.2.2. Laser Doppler Vibrometry

Vibrometers allow the non-contact measurement of surface vibrations based on laser interferometry: the superimposition of two waves is used in order to extract information about the original state of the waves. When two waves with the same frequency combine, the resulting pattern is determined by the phase difference between the two waves. The laser beam from the LVD is focused at the surface of the investigated object, scattered back from there and coupled back into the interferometer in the optical sensor head. The interferometer compares the phase ϕ and frequency f of the reflected beam with those of the internal reference beam ϕ_0 and f_0, passed through a Bragg cell producing the so called Bragg frequency shift. The vibration amplitude and frequency are extracted from the Doppler shift of the reflected beam frequency due to the motion of the surface.

The output voltage of the interferometer, V, is related to the target velocity component along the direction of the laser beam, v, as expressed in Equation 1:

$$V = K \cos[2\pi (f_B + 2v/\lambda)\, t] \quad (1)$$

where K is a constant representing the conversion efficiency, f_B is the Bragg frequency and f_D=2v/λ is the Doppler frequency.

This output voltage is then processed by the controller processor in order to extract the velocity and the displacement values. The velocity output from the controller provides an output voltage directly proportional to the velocity of the analysed object, multiplied by a constant that depends on the selected range expressed in [mm/s/V].

The LDV can provide both velocity and displacement signals independently of each other. For harmonic vibrations, the velocity and the displacement signal provide the same information according to Equation 2:

$$v(t) = 2\pi f\, x(t) \quad (2)$$

where v is the velocity amplitude, x is the displacement amplitude and f is the vibration frequency.

In this work the velocity amplitude has been measured because the resolution in velocity measurement is only limited by the background noise (higher dynamic range) and the signal-to-noise ratio is higher comparing to displacement measurements.

The system employed in this study is a Polytec Single Point Laser Doppler Vibrometer, composed of the optical sensor head OFV303 and the controller processor OFV-3001-S that allows the selection of few parameters depending on the measurement requirements.

2.2.3. Acoustic Energy Absorption Diagnostic Device

The acoustic method ACEADD is based on the determination of the acoustic energy absorption coefficient, using a non-contact setup. Under the action of an acoustical excitation, those objects affected by detachments and flaws start to vibrate and consequently to absorb a certain amount of the incident acoustic energy. Thus the method localize the defects where the acoustic energy is highly absorbed or, equivalently, the back reflected energy is low.

The device automatically scans an area, while an acoustic source S radiates towards the surface under investigation an acoustic wave with audible frequency content. A microphone M, aligned with the source S, records both the incident pressure wave $p_i(t)$ and the reflected wave $p_r(t)$, with a delay time τ due to the difference of the two acoustic paths. Both the reflection and the absorption coefficient are calculated from the acoustic impulse response $h_S(t-\tau)$ of the analysed surface. For each i-th point, the result is expressed in terms of the total reflected energy Σ_i (equation 3a), and in terms of the absorbed energy percentage $ABS\%_i$ (equation 3b)

$$\Sigma_i = \int_W \left| h_S(t-\tau) \right|^2 dt \qquad\qquad ABS\%_i = (\Sigma_R - \Sigma_i)/\Sigma_R, \qquad (3a, 3b)$$

this last calculated with respect to a properly selected reference Σ_R. This reference can be chosen among the most reflecting points belonging for instance to the analysed area, or to an external reference material. The two indicators are finally displayed as acoustic profiles and images, as shown in Figure 1(b). Furthermore the two indicators are also extracted as functions of frequency, providing an insight into many aspects regarding the object under study. An example of the frequency resolved acoustic images, obtained in four different frequency bands, is shown in Figure 4.

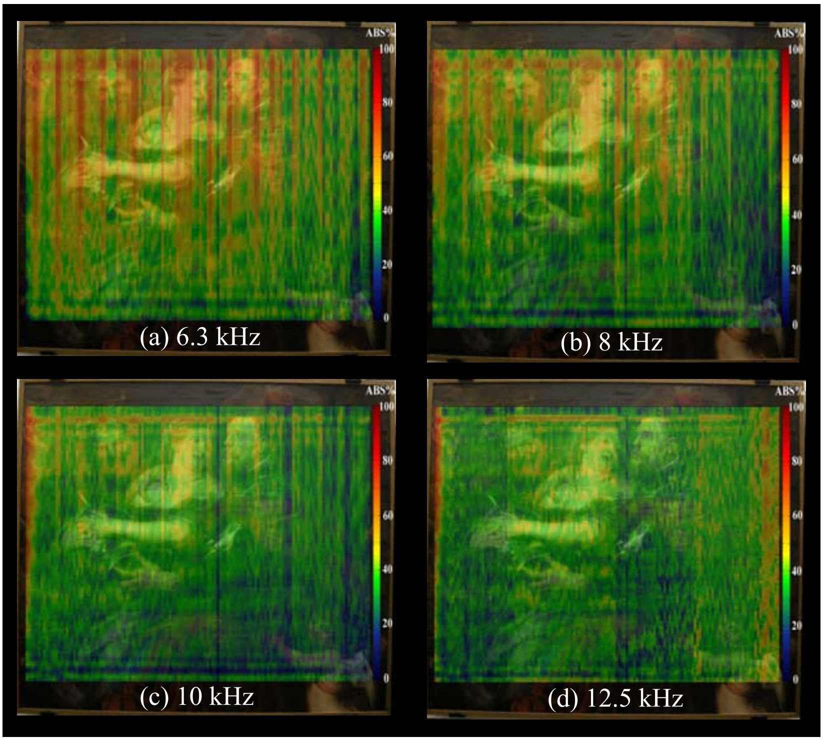


FIG.4: Acoustic images of the panel painting *Venus and Mars* by P. P. Rubens, obtained in four frequency bands whose central frequency is reported at the bottom of each image [9].

The images display the *ABS%* indicator measured on the panel painting *Venus and Mars*, by P. P. Rubens [9]. The images at the two lower bands, (a) and (b), evidence a weakness in the upper half of the panel disclosing an unexpected periodic structure, maybe ascribed to an uneven adhesion of the oak substrate to the cradle structure on its back. As frequency shifts towards higher values in the two higher bands, (c) and (d), a narrow horizontal flaw becomes more and more evident in the upper part of the painting. The frequency analysis may evidence different elements, where high frequency bands are related to a higher ability to reveal defects of smaller size, meaning that the spatial resolution improves with frequency.

This last issue was investigated measuring few total reflected energy Σ_i profiles across an abrupt interface between two materials of opposite acoustic properties: a polyurethane pyramid Akustik®-Foam panel as highly absorbing material, and a closed-cell polystyrene panel as reflecting material [10]. An indication of the spatial resolution at different frequencies is obtained from the frequency resolved Σ_i profiles and their derivative, as shown in Figure 5.

Analysing the falling edge of the curves, it can be stated that the transition from 10% to 90% occurs in about 8 cm depending on the frequency band, as shown in Figure 5a. Actually the instrument clearly perceives the effect of a different acoustic response in about 1-2 cm from the boundary, in particular at the high frequency bands, as evidenced by the derivative in Figure 5b. As expected, the spatial resolution improves as the content of the excitation source moves towards the higher audible frequency bands.

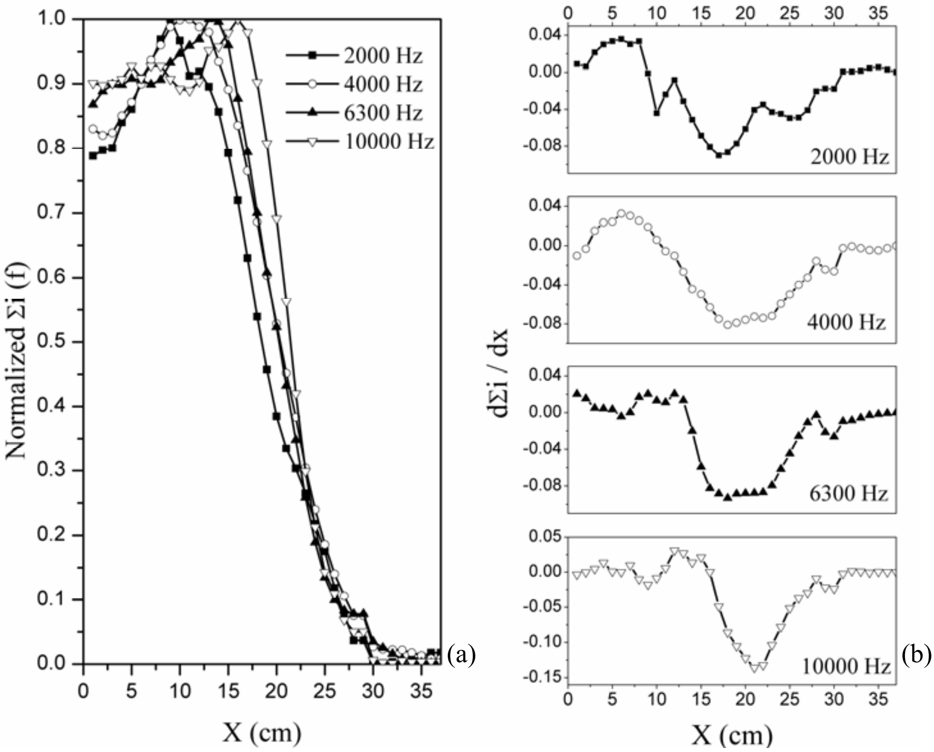


FIG.5: Total reflection profiles (a) in the most significant frequency bands displayed as 1/3 octave bands, crossing the border between two materials, and their derivative (b).

3. EXPERIMENTAL RESULTS

Leaving the acoustic source setting unchanged, the geometrical configuration adopted in the two kinds of measurements was adapted to the specific operational requirements of the experimental instrumentations, as described below. A National Instruments 16 bit multifunction data acquisition board completes both the measuring systems for the signal generation, data acquisition and signal processing.

The optical sensor head of the LDV operated in the horizontal plane, with the HAS8 and the analysed sample placed in the vertical plane; the acoustic source next to the laser sensor head was located at 60 cm from the surface of the ceramic sample, as presented in Figure 6. The analysed sample was mounted on a vertical support placed on an anti vibration table. The source was controlled in order to emit pure tones, with duration 0.375 s, scanning frequencies in the interval 1k Hz up to 16 kHz; the voltage from the velocity output of the controller processor was acquired and filtered by one third octave-band Butterworth filters to reduce the background noise, and the rms value was calculated over a steady portion of the acquired signals. The LDV ranges used in these tests were 5 mm/s/V for frequencies up to 10 kHz, and 10 mm/s/V for higher frequencies. The two ranges allowed us to extract the vibration velocity from the output voltage, in V, and express it in mm/s. Finally the vibration velocity was displayed as function of frequency, as shown in the next paragraph.

FIG.6: The laboratory equipment for LDV measurements (a); the HAS8 – sample – LDV configuration (b).

A horizontal configuration of the ACEADD system was adopted with the transceiver unit, constituted by the HAS8 transducer and an omni-directional microphone, mounted on a suitable support in order to vertically orient the acoustic beam towards the sample.

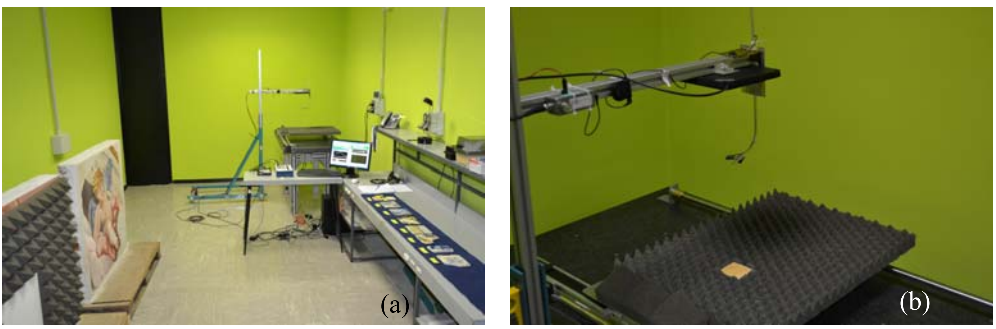


FIG.7: The laboratory with the ACEADD equipment (a); the transceiver unit with the test sample on the plane of the horizontal scanning system.

The analysed sample was embedded into a matrix made of a highly absorbing material, a polyurethane eggshell Akustik®-Foam panel 50 mm thick, where a square window has been created to locate the ceramic sample. This polyurethane panel with the sample was placed on a moving plane, fixed to an anti vibration table, as shown in Figure 7, allowing the automatic scanning along the X axis. The HAS8 source was fed with a sine wave signal with variable frequency content (logarithmic chirp signal) in the interval 1 kHz to16 kHz, naming this wide band as B1. In order to better highlight the potential differences in the acoustic response of different samples, the measurements were repeated using chirp signal with narrower bandwidth named B2 (1 kHz to 8 kHz), B3 (3 kHz to 10 kHz), B4 (5 kHz to 12 kHz) and B5 (8 kHz to 16 kHz). In this first stage of laboratory validation, the Σ indicator has been preferred to the *ABS%* indicator to display the experimental results because it does not need to be expressed with respect to any reference value, as contrarily the *ABS%* indicator needs. Indeed the reference tiles are not definitely identified yet, and they constitute part of the investigated samples the same way as all other test tiles. Thus in this study the results are preferentially expressed in terms of total reflected energy Σ_i profiles along the X axis, determined for the different samples, as shown in the next paragraph.

The experimental results are presented firstly for attempting to classify which samples can constitute the proper reference tile, both among the azulejo samples and among the laboratory models. Secondly the results shall indicate the main differences evidenced in the acoustical response of the reference tiles and the delaminated samples. Finally the integration of the LDV and of the ACEADD outcomes shall orient the optimization of the experimental settings for the laboratory validation of the acoustical methods, assuming these ones as useful tools for an early stage glaze delamination detection to conduct both in laboratory and on site.

3.1. Single point frequency analysis by LDV

Figure 8 reports the vibration velocity revealed in the central point and in a lateral point of the three laboratory models, by scanning the excitation source frequency between 1 kHz and 16 kHz. In the T1 model the central point lies upon the air cavity, while the lateral point corresponds to one of the side walls of the cavity.

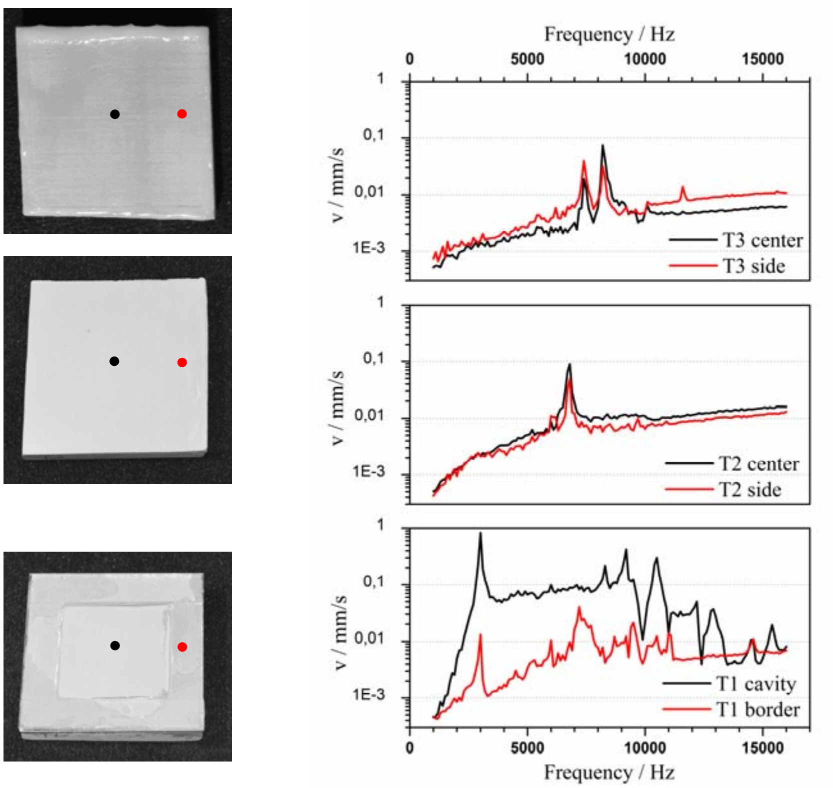


FIG.8: The laboratory models and their characteristic resonances revealed by LDV.

The velocity measured on the air cavity of T1 is clearly higher than the velocity in all other points, it presents a clear resonance at about 3 kHz and others at higher frequencies. T3 and T2 also shows their characteristic resonance, but the velocity values are evidently low. Beside its reduced vibration, T3 can be considered the best choice as reference tile since its capping layer is of the same material as for the T1 sample with the artificial air cavity, while T2 is expected to present additional acoustic energy absorption for porosity.

Figure 9 gathers the vibration velocity revealed in the central point and in two lateral points, on the left side and on the right side, of the three azulejo samples that may be considered good reference for the set of historical tiles. The characteristic resonances are visible, with relatively low velocity values. A1 seems to be a better reference than A5 and A6, at least up to about 10 kHz where its resonance occurs.

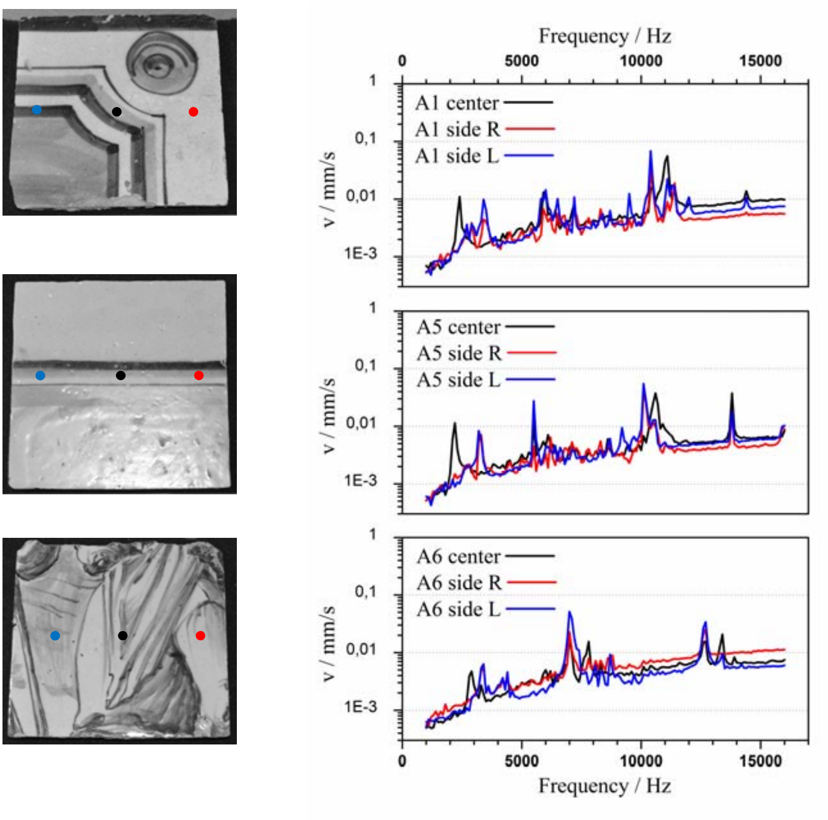


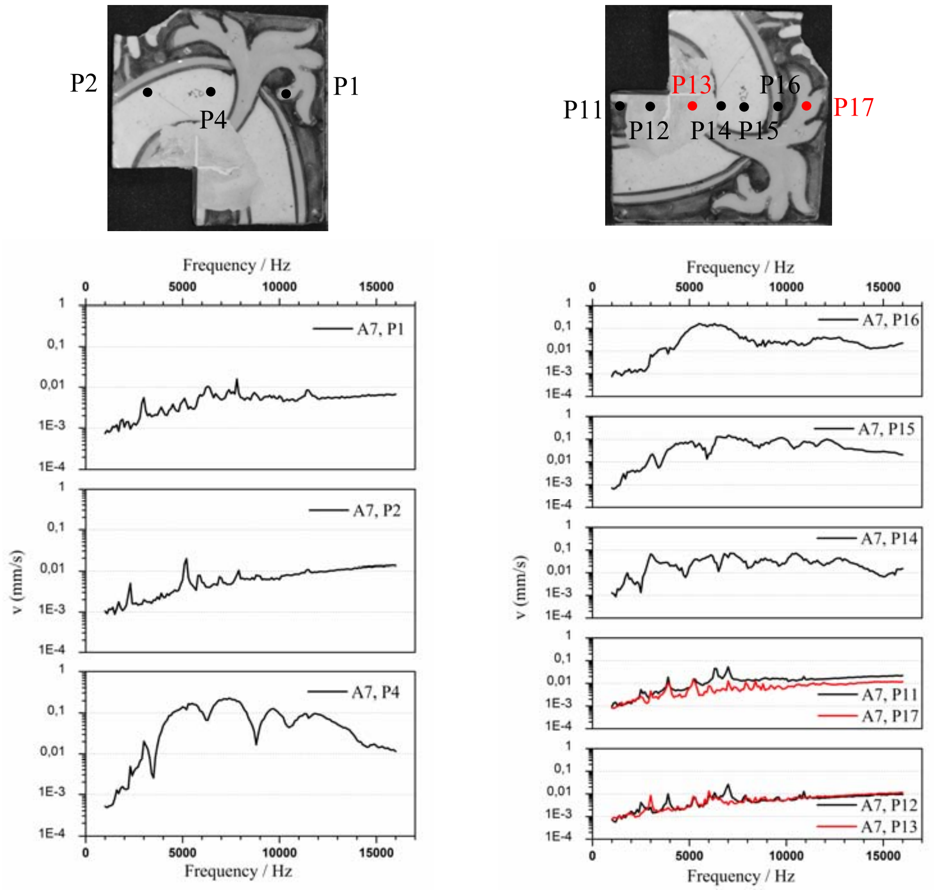
FIG.9: The historical azulejo samples A1 – A5 – A6 and their characteristic resonances revealed by LDV.

The complex state of the A7 sample was studied by analysing ten points on areas with good glaze adhesion (P1, P2, P11, P17), on lacunas (P12, P13), and on the evidently delaminated portion (P4, P14, P15, P16). Figure 10 reports the position of the investigated points and the corresponding vibration velocity curves. Similarly to the laboratory models, significant vibration occurs on the delaminated areas, while minor resonances with low velocity values are visible both on non-delaminated areas and on lacunas. It can be observed that the main differences approximately fall in the interval (3 – 12) kHz.

Based on the LDV measurements on both laboratory models and azulejos, it can be assessed that the HAS8 source seems to present suitable features as excitation source because is able to excite the characteristic resonances of all the samples. Indeed it is possible to find some important resonances in the frequency range 1 kHz to16 kHz.

The high values of the vibration velocity revealed on the artificial cavity of T1 model and on the delaminated portion of the A7 sample, in comparison with all the other analysed points, suggest that the single point analysis by LDV gives a useful indication on the presence of a sub-surface cavity. Its response can be easily discriminated from that obtained on points, with good glaze adhesion, showing lower values of vibration velocity.

In addition a distribution of points, over the entire surface of the sample, that homogeneously show low values of vibration velocity is the requirement to classify this tile a suitable reference tile as for the A1, A5 and A6 samples.



(a) (b)

FIG.10: The azulejo sample A7 and the characteristic resonances revealed by LDV at different points, as indicated by the dots on the sample image at the top.

3.1.2. ACEADD profiles

Total reflected energy profiles were obtained by ACEADD measurements on T1 and compared with those obtained on the reference model T3. Figure 11 presents the measured profiles in the different frequency bands B1 to B5.

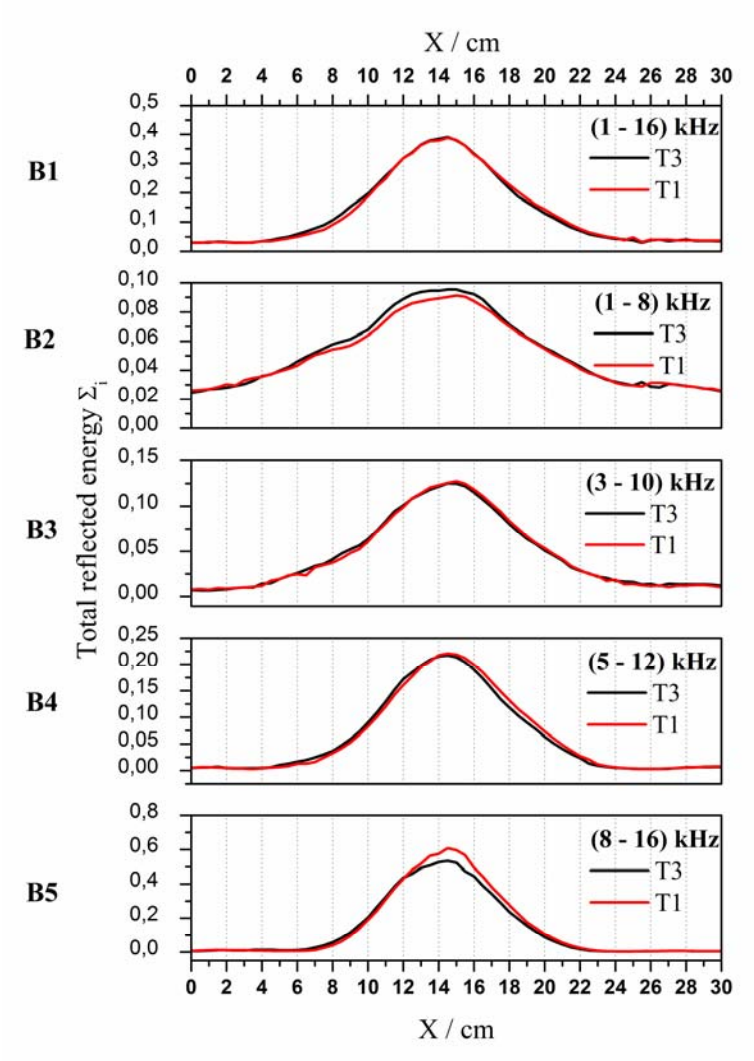


FIG.11: Profiles of the total reflected energy obtained on T1 and the reference T3.

It is worthwhile to note that the bell shape of the profiles depends on the fact that the matrix used to embed the samples is made of polyurethane foam, that works as highly absorbing material for frequencies higher than 4 kHz and as medium absorbing material for frequencies lower than 4 kHz (visible in B2 band). The behaviour of the samples is evidenced in the central part of each profile: in the B2 and B5 bands the main differences between T1 and the reference T3 appear.

For the models the repeatability of the measurements was verified by means of a number of repetitions in the B2 and B5 bands. The repetitions were carried out alternating the measurements on T1 and on T3, mounting and dismounting the sample at each repetition with particular attention to the horizontal placement of the surface of the sample exposed to the acoustic beam. This procedure allowed to assess the high reliability of the measuring configuration and procedure, able to evidence differences in the acoustic response as small as few percent, as shown in Figure 12.

The comparison of T1 and T3 models indicates that the central part of the two profiles (Figure 12, left), with the uncertainty displayed as error bars, are well distinguished. The frequency dependence of the reflection coefficient, measured in the central point of the samples (Figure 12, right), helps to understand at which frequency the main differences lie. In the B2 band the T1 model presents a reduction of the Σ_i indicator due to the presence of the air cavity, that absorbs the acoustic energy up to the 6.3 kHz octave band. In the B5 band an opposite situation occurs, mainly due to an absorption at frequencies around 12.5 kHz in the T3 sample.

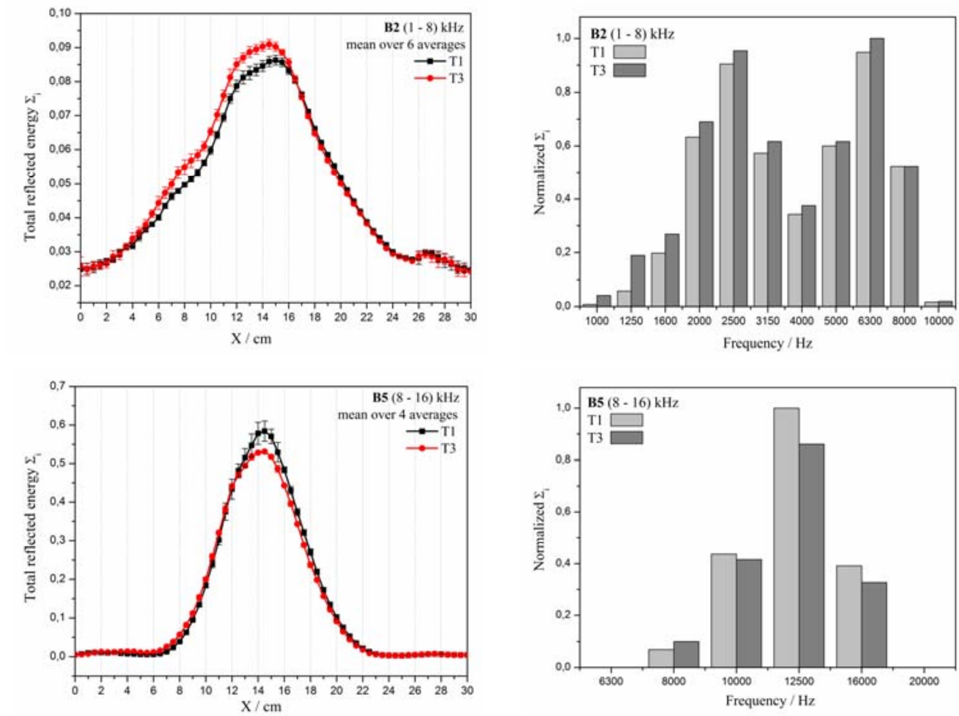


FIG.12: Normalized reflection coefficient vs frequency, expressed in 1/3 octave band, in the central point (right) of the corresponding profile (left). Data refer to the B2 and B5 bands and are displayed as average values with standard deviation.

The results obtained on the historical azulejo samples are presented in Figure 13. In this case the major differences are found in the B2 and B3 bands: the A7 profiles always show the higher distance from the A1 profiles; A1 works well as reference tile showing the highest reflection values in most frequency bands; only in the B5 band A1 presents an uneven behaviour lowering its profile below those of the other samples. This feature is likely consistent with its characteristic resonance around (10 – 12) kHz. It is worthwhile to note that the higher spatial resolution of the B5 band allows the border recognition and the outline of the analysed tile with a suitable accuracy.

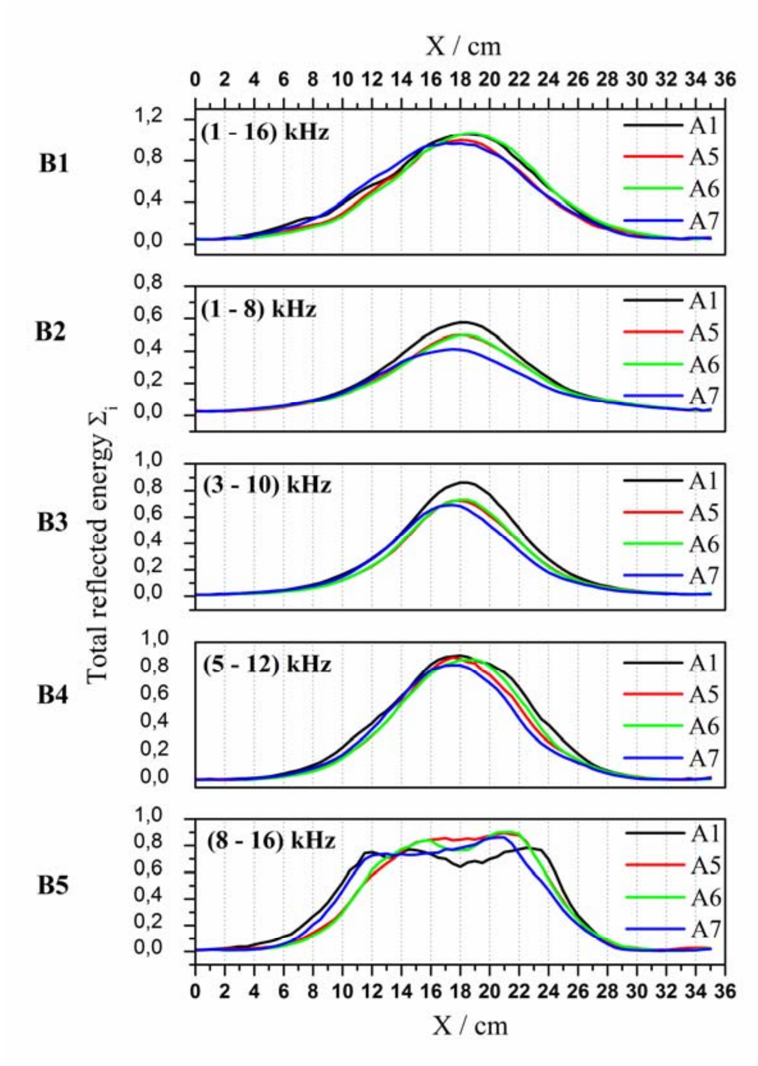


FIG.13: Profiles of the total reflected energy obtained on the four azulejo samples, taking A1 as reference.

For both the laboratory models and the azulejos, it can be assessed that the absorption data in the five bands show a good agreement with the position of the resonances revealed by the LDV. The response obtained by the acoustic absorption method is read with an appropriate sensitivity, which can be enhanced by accurately selecting the frequency band of the excitation source. The B2 band appears as the most relevant, able to emphasize the differences between the samples.

The *Σ* profiles allow to identify a reference tile as the object showing an acoustic energy absorption as low as possible. In addition, the *Σ* profiles allow, with a suitable accuracy, to discriminate the tiles having some critical points from the undamaged samples.

A simplified experimental setting, similar to that employed in these laboratory tests, and the procedure, based on the direct comparison with a reference tile, can be also adopted for a straightforward *single tile* diagnostics in a field experimentation. For example, in those cases requiring the removal of the azulejos from the wall for successive restoration actions, the ACEADD feature of integrating over a small area of the investigated surface may even be an advantage. A small number of *single point* measurements in the central part of the tile may immediately indicate the conservation state of the whole tile, requiring no elaborate post-processing of the experimental data.

4. CONCLUSION

In the LARCH laboratory two non-contact and non-destructive methods were employed in order to undertake their experimental validation for glaze delamination in ceramic tiles. A Laser Doppler Vibrometer provided the vibration velocity, induced by an acoustic source, in a collection of samples, and the Acoustic Energy Absorption Diagnostic Device revealed the presence of acoustic energy absorption in the same samples. The investigated samples include both historical azulejos and laboratory models with artificial delaminations, some of them selected as reference tiles.

The investigation evidenced that point analysis by LDV recognizes sub-surface cavities due to the higher vibration velocity values with respect to the areas with good adhesion.

Although not used for point analysis, the ACEADD device can reveal acoustic energy absorption in samples with air cavities, discriminating them from the reference tiles. Some experimental requirements also enhance the accuracy, the sensitivity and the reliability of the ACEADD method: the way the tested objects are sequentially analysed inside the same measuring session; the tuning of the frequency band of the excitation signal; the reproducibility of the sample positioning; the execution of suitable repeatability tests.

The approach to the two measurements, both based on the direct comparison with a proper reference tile, brings to experimental results easy to be interpreted also by non specialized operators. The modular structure of the measuring apparatus allows to conduct both laboratory tests and on site investigations, letting the operator chose the most appropriate experimental configuration. These aspects guarantee that the results, presented in this paper, may constitute an immediate and, for the ACEADD technique, low cost evaluation of the conservation state of glazed ceramic tiles.

Furthermore, the outcomes of the present work shall orient the optimization of the experimental settings for a more extensive validation, whose scope comprises an appropriate application of acoustic imaging techniques.

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