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Best Selection of Glue for Restoration of Archaeological Glasses Through the Modal Analysis of its Acoustic and Vibrational Response

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Highlights

1. The restoration and preservation of Roman Glasses have been studied
2. Modern copies of original roman glasses have been measured applying up to date acoustic and vibrational methods, including laser vibrometer, following the indication of the restorers
3. The glasses have been broken and reassembled using different resins, with the help of a professional Restorer
4. After the restoration the same methodology has been applied as described in point 2
5. The new measurements have been analysed and compared with the previous one in order to check the effectiveness of different resins



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ABSTRACT

The success of conservation related to archaeological glass depends on several factors, including the techniques used and the types of adhesives applied during the reconstruction process. This paper investigates the characteristics of glass conservation by using glass produced with techniques developed by ancient Romans and analysing the modal response of the vibrating surface under airborne and vibrational perspectives. In particular, the conservation process of archaeological glass has been explored, encompassing steps such as cleaning, debris removal. Adhesive usually employed in glass conservation have been tested in many ways (accelerate ageing, cohesion power, etc) but they were never tested for their vibrational behaviour. Acoustic and vibrational measurements were carried out to scientifically evaluate the performance of two types of adhesives selected from a range of products available on the market. The measurements were conducted under three specific scenarios: (1) on intact glass before any intervention, (2) after a fracture repaired using Araldite® resin 2020 Huntsman, and (3) after a fracture using epoxy resin (Hxtal Nyl-1) B. The results were processed using the transfer function and the cross-correlation of vibrational and airborne values, focusing on the intensity of acoustical radiation (IAR). The findings indicate that the first adhesive B outperforms adhesive A, as the values of the resonance peaks related to the restored glass are closer to the peaks related to the glass measured before the rupture. The tests were performed on a perfect replica of a roman glass made with experimental archaeology.

Keywords

Glass conservation; Roman glasses; laser vibration; acoustic measurements; glass heritage; intensity of acoustical radiation

1 Introduction

Glass is a material that was first discovered in the late Bronze Age [1]. Techniques of firing were adopted to make this material clearer and more transparent [1]. Although early samples of ancient glass, as revealed by archaeologists, were glossy and almost opaque, sometimes coloured with natural pigments, the glassblowing technique developed by the ancient Romans marked a significant innovation for its time. Using a blowing rod and metal tools, they shaped the incandescent mix with silica from a type of pottery kiln. The addition of pigments aimed to mimic the appearance of precious stones known in antiquity.

Research on the chemical composition of ancient spans nearly all historical periods, from the 14th century BC to recently discovered samples from the 18th century [2]. Analyses of some samples indicate conservation of ancient glass has been practiced for centuries, using natural glues derived from plants or animal products [3]. In modern times, conservation techniques often draw on knowledge from other fields, like dentistry, for the innovative materials like resins and foams.

Ancient glass conservation remains a specialist discipline, with its success largely determined by the integration of specific expertise regarding the material's characteristics, such as its composition percentages when combined with other substances, density, and application methods. This underscores the necessity of carefully designing all phases of glass conservation process [4].

2 Research aim

Modal analysis and acoustic radiation are scientific methodologies widely applied in musical acoustics to study the vibro-acoustic behaviour of instruments [5]. Previous research studies have established a correlation between sound radiation and the resonance frequencies of vibrating bodies or objects, particularly membranes [6]. These methodologies have been identified as the most appropriate techniques for explaining the vibro-

acoustic characteristics of ancient glass under external excitation, to assess the behaviour of the conservation treatment. Unlike studies on musical instruments, which focus on frequency ratios and relative harmonics, this research aims to scientifically translate the empirical methods used by conservators to evaluate the intervention on glass. Specifically, it seeks to understand how conservators classify the quality of reconstruction based on the mechanical excitation applied to glass using their fingers [7]. The application of a technique widely used in musical acoustics to the sector of conservation is a novelty, especially for ancient glasses.

3 Materials

3.1. Production of glass in ancient time

The development of glass production dates back to the 3rd-2nd century BC, pioneered by the Phoenicians and Egyptians. Early glass objects were not transparent because the fusion temperatures required for silica, calcium carbonate, sodium and potassium were not high enough for the capability of ancient times. As a result, the natural colour of glass was a light blue, with variations tending toward light green or yellow when copper or iron oxides were added [8].

Glass objects were formed by placing the vitreous mass at the end of a rod and shaping it with metal tools and stone plates. Using this technique, the earliest glass products were essentially ornaments, pendants and similar decorative items.

One of the most important phases of glassmaking is colouration, which involves the addition of oxides (either individually or in combination). Oxides of copper, cobalt, tin, antimony, lead, manganese, and iron were used to achieve the full range of glass colours, ranging from water-clear to intensely opaque. These colours were often employed to imitate the appearance of precious stones known in antiquity [9].

During the Roman Empire, one of the most important innovation innovations in the art craftsmanship was glassblowing. This technique involved blowing air into molten glass straight from the furnace to create a glass bubble. This was made possible when the glass mixture was heated to a temperature beyond its incandescent state, bringing it to a liquid form [10].

Glassblowing allowed glass to be used not only for ornaments but also for interior design in residential palaces, including the production of vases and other objects previously created in ceramic. Over time, glass was also employed for medical and cosmetic purposes and to create jewellery [11].

Roman glassblowing technique was later revived during Renaissance in Italy and remains highly valued today. Among the regions where this tradition continues, Murano (Venice) stands out for its large-scale production of glassblowing, which is considered a highly prized craft.

3.2. State of the art and challenges of conservation procedures for ancient glass objects

When archaeological discoveries bring to light precious glass specimens from the Roman era, there are many challenges to face for their conservation, influenced by various factors. The first step is to assess the natural transparency of glass, which may be obscured by aggregates such as encrustations that result from prolonged burial. The preservation state of glazed archaeological objects is undoubtedly affected by the material's thickness, which is often very thin and can lead to reticular fractures [12].

It is considered best practice to gather extensive information before proceeding with any conservative intervention. In case artefacts have undergone to previous interventions, if possible, it's useful to identify the substances used and collecting samples to analyse the types of aggregates on the surface of the object. Such analyses can also help identify the products used in previous treatments by characterizing glues, fillers, and additives [13]. For instance, common filling materials used in ancient times included animal glues or vegetable gums, whose organic components were often combined with inert materials and pigments.

When previous interventions involving gluing, and gap-filling are applied, they could be characterized by the appearance of a white, compact paste along the fractures' profiles. In other cases, missing parts could be filled with irrelevant glass fragments, either of a similar colour to the original or painted to match [14].

It is essential for a complete intervention to remove any residues from prior treatments, as these can sometimes weaken final results. The cleaning phase is therefore crucial for removing incrustation without interfere with altered or detached layers of glass.

One of the techniques for reassembly involves the use of metal bridges or clamps to secure the fragments in place before injecting the epoxy resin along fractures [15]. The missing parts are reconstructed using a double-valve counter form system, into which resin is injected. The resin is often tinted in a subdued tone to blend subtly with the original glass, forming a replacement for the missing sections.

3.3. Principles of glass conservation

3.3.1. General

Conservation is a practice that gives back shape and legibility to damaged objects through the application of appropriate treatments. These treatments are designed to avoid altering object's original state while preserving it for future generations [17].

Glass, a material discovered several centuries before the Egyptian era, holds a significant place in history, with the earliest known samples providing valuable insights. Due to its versatility, the treatment of glazed objects varies depending on their intended use and original purpose [11]. Initially, glass was used to produce drinking and eating vessels, later becoming an integral part of churches and residencies as windows.

The primary component of glass is silica (sand), followed by alkaline-terrain metals. In some compositions, lead and barium are added to enhance the material's aesthetics properties [2]. Before undertaking any conservation work on glass objects, conservators evaluate any potential risks to ensure the integrity and to prevent any further deterioration. The principles of minimum intervention and reversibility should guide any conservator choice even if due to the particular nature of glass, it's often necessary to come to a compromised solution.

3.3.2. Conservation project

The process of assembling pieces of a glazed object begins with cleaning surfaces, which involves the removal of dust, aggregates, or grease. Cleaning operations are typically carried out by moistening the material to be removed with warm water or other solvents, applied through localized infiltration using syringes or cotton swabs. This technique facilitates the softening of the residues and aids in mechanical detachment action.

The adhesive commonly used for glass include polymers such as acrylics and epoxy resin, applied along the edges of each piece.

Selecting the appropriate class of adhesive is a critical decision for conservators, as it influences the final quality of the intervention. Factors that affect the choice include the following [14]:

- the refraction index should be similar to the glass one.
- high stability to light and heat, without altering the physical, chemical, or chromatic properties.
- chemical compatibility with glass.
- permeability to vapours and gases.
- low level of toxicity for the operator and the environment.

Some epoxy resins with low viscosity have physical and chemical characteristics that differ significantly from those of glass, and their stability over time can vary. This kind of resin are the most used in glass conservation. From a chemical point of view, these compounds are usually obtained through a condensation reaction of epichlorohydrin, a derivative of propylene oxide, with bisphenols or other similar compounds. This reaction leads to the formation of epoxy functional groups. These groups are then involved in further reactions between the preformed polymers and other reagents – amines or basic acids – to form a more structured network. The use of these products in conservation is usually not recommended because once catalysed they become hardly reversible, making any future disassembly invasive. They tend to yellow and form very strong bonds which ensure that, in the event of stress, any breakages occur more easily in the walls of original objects rather than along the existing fractures. Despite this, epoxy have been widely used, especially in glass and metal conservation, as they make excellent adhesives and, thanks to their dimensional stability, and suitable to create shapes through casting into moulds. The choice to use epoxy resins is ideal when an adhesion with high mechanical properties is required and in the case of very thin or transparent glass, given their optical properties. Unlike other adhesives, in fact they usually have a refractive index very similar to glass. The resins selected are both epoxy but with slightly different composition that leads to different behaviors. Hxtal Nyl-1 is an American product, especially projected for glass conservation and it has good results in ageing tests without any optical and chemical degradation. The refractive index is similar to glass and its curing time is approximately 72 h-one week due temperature and U.R. Araldite® resin 2020 Huntsman is widely available and its' curing time is 24 h and it tends to yellow in time, the refractive index is also very similar to glass. On the other hand, acrylics diluted in acetone, when the solvent evaporates create micro-air bubbles along fractures that could interfere aesthetically with the final result.

3.3.3. Reconstruction process

The first step before applying epoxy resin by capillarity along fractures, is creating a preliminary and temporary reconstruction using small strips of paper tape across connections., as shown in Figure 1(a). The next step to

prevent the glass pieces from shifting is applying small metal clamps to the surface of the glass with a cyan-acrylic adhesive, as shown in Figure 1(b). The use of small metal clamps is not recommended for very thin or damaged glass, nor for decorative surfaces.



Figure 1. Application of paper tape stripes (a) and metal clamps (b) to the glass.

3.3.4. Final fixing

After the preliminary assembly if epoxy resin is chosen as adhesive, it can be applied along junction where it penetrates by capillarity. The clamps and excesses of adhesive can be removed mechanically only after the resin has completely cured, after having softened the cyan-acrylic with small poultices of acetone. The curing time depends on the kind of resin it spans from 24 hours for 2020 to one week for Hxtal Nyl-1 and also from environmental U.R. and temperature. All these procedures must be undertaken under a chemical hood for safety reason, although the average quantity necessary as an adhesive is usually really, small about 1 ml (resin + hardener) for a medium size object. There is no need to add pigments to adhesive as the thickness of glass wall is minimum (around 2-3 mm) and it does not interfere optically.

3.3.5. Gap-filling

The missing parts of an archaeological glass should be reconstructed only when objects need static support or when it is needed to regain their shape. Generally, the concept of conservation involves highlighting the rebuilt parts in relation to the base to which they are applied. However, some theories completely oppose this approach, preferring to camouflage the integration. There are different techniques to make the integrated parts identifiable:

- Apply a slightly different pigment to the resin.
- Make the integrated parts opaque or more polished, especially when the glass lacks colour.

It is a very complex step that could be significantly invasive, so it has to be carried out just when it is necessary.

3.3.6. Reconstruction test

After the reconstruction, conservators check the quality of the intervention by manipulating and tapping the object surface and listening to the response sound of the glass.

However, the response to this procedure is highly subjective, which is why modal analysis, in terms of sound and vibration, has been conducted to objectively quantify the magnitude and the frequency of the resonance.

4 Methods and Procedures

4.1. Measurements and equipment

The airborne soundwaves were measured with a B-format microphone (i.e. Soundfield MK-V), while the vibrations were measured with a laser (i.e. Polytec PDV 100). The distance between the glazed object and the microphone was 300 mm, corresponding to 1/4 of the average wavelength of the soundwave over the full spectrum bandwidth (between 50 Hz and 10 kHz). Additionally, a small hammer (Brüel & Kjær Type 8203) was used to create the excitation on the glass [20].

The measurements were conducted in a typical environment (semi-anechoic), similar to the conditions a restorer would encounter, with the presence of furniture and standard surfaces typically found in a common office room. The acoustic tests could have been conducted in an anechoic chamber, as an ideal and artificial environment where all the boundary conditions are under control. The choice of conducting the measurements in a common office space is to understand all the problematics that a common restorer would encounter during the fixing process.

The glass object was placed on a table in the centre of the target point, while the microphone and laser were placed 300 mm from the target point. The employment of laser in combination with the microphone was extensively used by authors in other case studies, also applied to architecture.

Given the dimensions of glass, with 50 mm diameter and round shape, the phenomenon of diffraction can be excluded from the bandwidth subject to this analysis, which is comprised between 100 Hz and 8 kHz. For medium-low octaves, the glass is considered acoustically invisible.

All audio files were recorded using a soundcard (i.e. Motu Traveler) [21] set to 96 kHz.

4.2. Data analysis

All data related to vibrational and acoustic measurements have been analysed by considering different procedures that are explained in the following subsections.

4.2.1. Elimination of extraneous information

The first step in the post-processing analysis involved the elimination of the ambient noise from the audio signals, while ensuring that important spectral information for comparison was preserved.

Since the laser is highly sensitive to any perturbation, it was difficult to obtain an uncorrupted soundwave. However, the laser also captured some extraneous, undesirable information, which was subsequently removed. The selection of the audio signals was performed using Adobe Audition 3.0. All files were sampled at a rate of 96 kHz with a 32-bit depth. The portion of the files been removed due to extraneous information was determined based on the following criteria:

- The number of the first sample from which the cut was executed.
- The number of the last sample removed.
- The total number of sample removed.

The same criteria were applied to both audio and vibrational files, considering all positions where the hammer was placed on the glass and the state of the glass's integrity (i.e. intact glass, application of Glue A or Glue B after the rupture).

4.2.2. Transfer function and Phase

The software SpectraLAB [22] was used to calculate the transfer function. The settings in SpectraLAB were the following:

- Complex transfer function (Left/Right channel).
- Spectrum and phase for each file.

The file containing the vibration response was analysed by assigning the signal obtained with the laser to the Left channel and the impulse response from the hammer to the Right channel. For the airborne response, two files were recorded for each measurement: one with two channels (Laser(L)-Hamm(R)), and the other with 4 channels (X-axis, Y-axis, Z-axis, W-axis). This organization was maintained for all files related to the 7 measuring positions.

After computation, the data were exported to Microsoft Excel to analyse the frequency-domain values as a function of dB levels.

Each glass was measured before any action was taken on the material. After the rupture, the measurements were taken at the same positions, but the glazed object was reconstructed using glue A and glue B. A total of 21 measurements were made, divided into three sets of 7 measurements.

The comparison between the results from the 3 sets of measurements was carried out by analysing the amplitude of the transfer function [23]. From a scientific perspective, when the peaks of the airborne soundwave coincide with the peaks obtained from the vibrational measurement, the gluing process can be considered of high quality. The higher the difference in peaks between the soundwave and vibrational values, the poorer the quality of the gluing.

4.2.3. Computation of the cross spectrum under the concept of Intensity of Acoustical Radiation (IAR)

The concept of acoustic radiation was introduced to measure the effectiveness of a vibrating surface excited by sound power. The commonly used parameter [24] is the ratio between the sound pressure measured in the near field (p) and the velocity measured on the vibrating plate (v), known as the complex ratio (p/v). However, the limitation of this method is that, in most cases, resonance frequencies are not correlated with the airborne acoustic emission, as demonstrated in previous literature.

The parameter that best represents the correlation between the acoustic and vibration response of a radiating plate is the intensity of acoustic radiation (IAR) [6]. The IAR is the spaced-average amplitude of the cross spectrum between the airborne sound pressure of the oscillating surface and the velocity of the vibration. Thus, the amplitude and phase of the transfer function (p/v) and the cross spectrum ($p \cdot v$) were compared with the IAR results. It is worth noting that IAR shows good correlation with the frequency response function (FRF) [25].

4.3. Limitations of the post-processing analysis

There are several potential mistakes that can occur during a post-processing elaboration, which represent the limitations of this research. These issues could be overcome with a more optimised method of automating the parameters [26]. The list of potential mistakes is as follows:

- *Cut errors.* This type of mistake is common during data elaboration but is easy to detect by the sample length in a WAV file, which may not match the others taken at the same position.
- *Hammer noise signal.* This noise is detectable at frequencies below 1 kHz and above 10 kHz. It is caused by the hammer impact. A pass-band filter can be applied to eliminate such hammer noise. This method is effective, as the peaks corresponding to other phases of the measurements remain clear.
- *Erroneous compression of files.* This mistake can be detected when importing files into SpectraLab because the values are completely out of phase compared to the data of a correctly compressed file that are supported by the same software.
- *Erroneous comparison of data in Microsoft Excel.* This mistake is not easy to be detected and can as the values will be completely out of phase compared to those of a properly compressed file supported by the same software.

5 Results

The results presented in this section summarize the steps explained previously. The initial graphs display the average values of all 7 positions recorded by the microphone before any rupture, specifically focusing on the transfer function along X-axis, Y-axis, Z-axis, and the omnidirectional W, as indicated in Figure 2. However, the results along Y-axis and Z-axis are not relevant to this study, which focuses on surface radiation (calculated at omnidirectional level, W) and the distance between the vibrating plate and the receiver (along X-axis). Therefore, only W and X-axis results are taken into consideration for the airborne analysis.

Introduction the X-axis values represents a novelty compared to previous studies, which relied solely on the W response. The frequency range for this study is between 100 Hz and 8 kHz, with the upper range limit of 8 kHz chosen because peaks beyond this frequency band are not clearly visible or detectable.

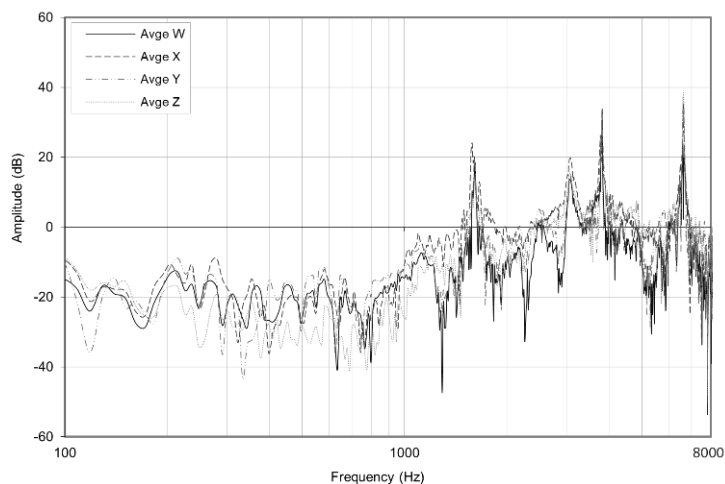


Figure 2. Airborne results related to the average of all 7 positions, measured before rupture.

Figure 2 shows that the first peak, common to all four curves, occurs at around 1600 Hz, followed by a second peak around 3050 Hz. The distance between the second and third peak is shorter than that the distance between the first and second peaks; the third peak for all four curves is detected at 3810 Hz. The last common peak within the considered bandwidth is observed at 6600 Hz. These four peaks represent the main resonance frequencies of the glazed object measured in this experiment.

The average value for each direction (X, Y, Z, W) are calculated from the transfer function averages across all 7 positions, which are summarised in Figure 3, focusing specifically on the X and W directions.

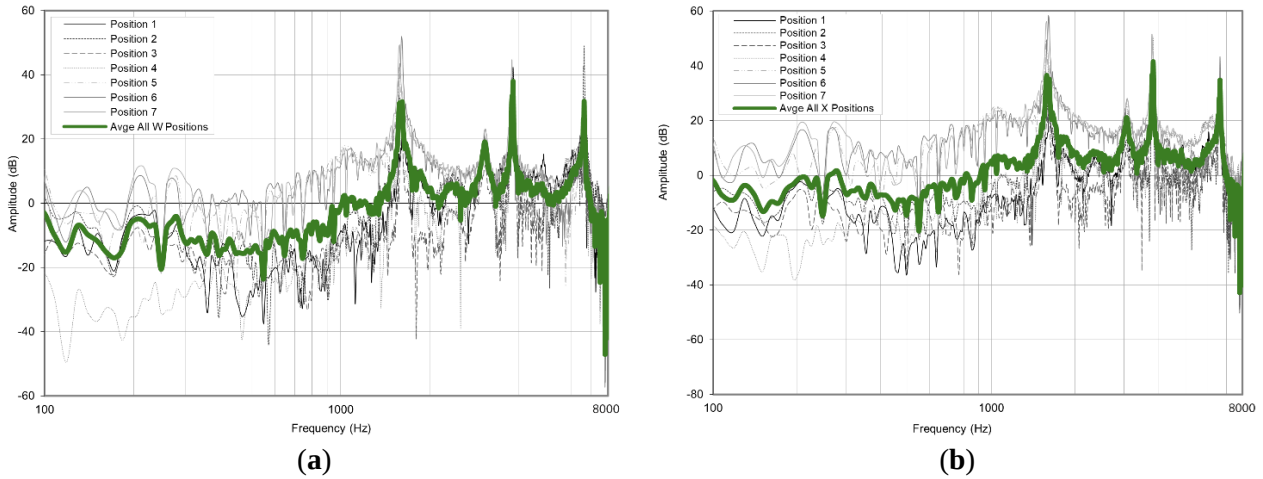


Figure 3. Airborne results (transfer function) related to all 7 positions, measured before rupture: W (a) and X-axis direction (b).

Figure 3 shows that the four peaks analyzed above are visible at all positions measured on the glass before rupture and for both considered directions (W and X). Similarly, the same peaks are observed in the measurements conducted after rupture using glue A and B, as shown in Figures 4 and 5, respectively. While the peak positions occur within the same frequency bands, the magnitude varies across all the graphs

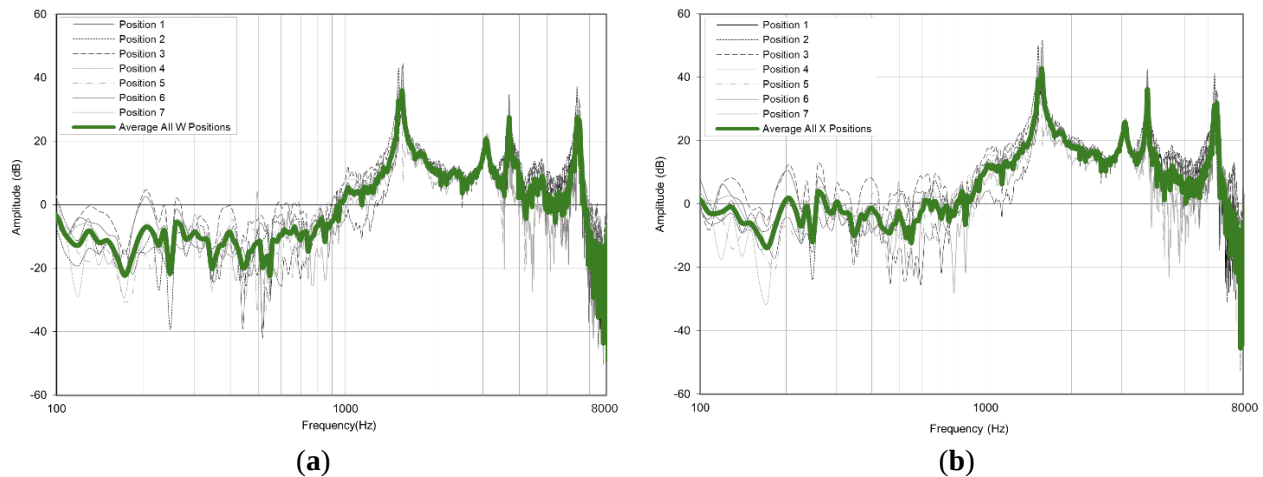


Figure 4. Airborne results (transfer function) related to all 7 positions, measured with the application of glue A: W (a) and X-axis direction (b).

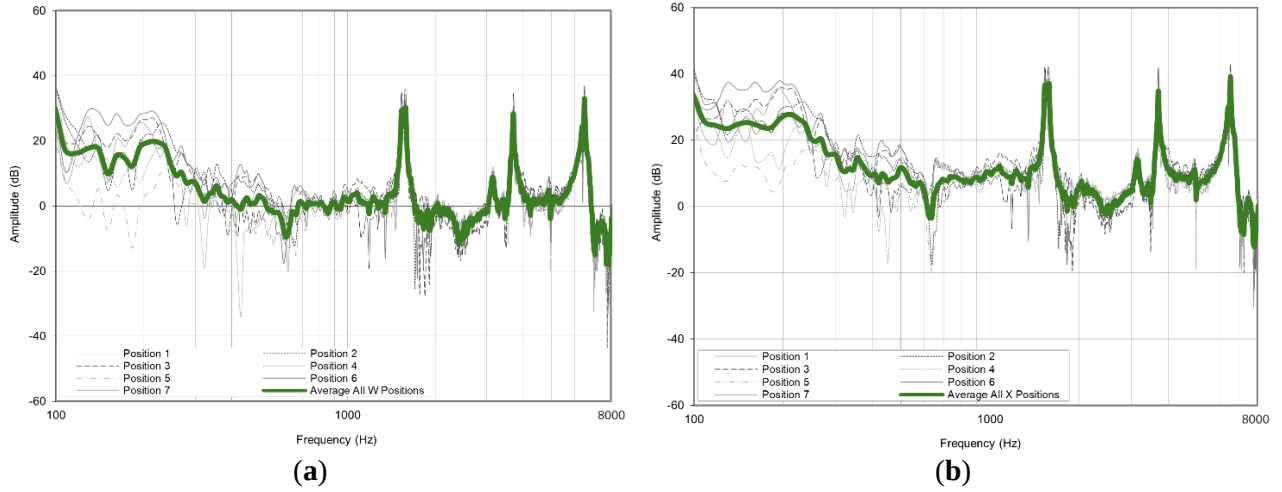
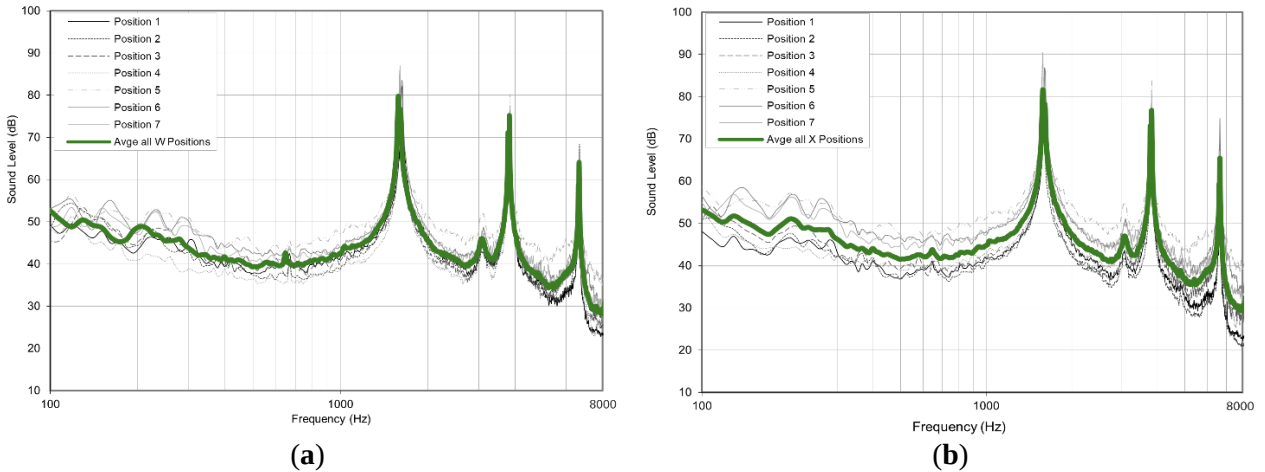


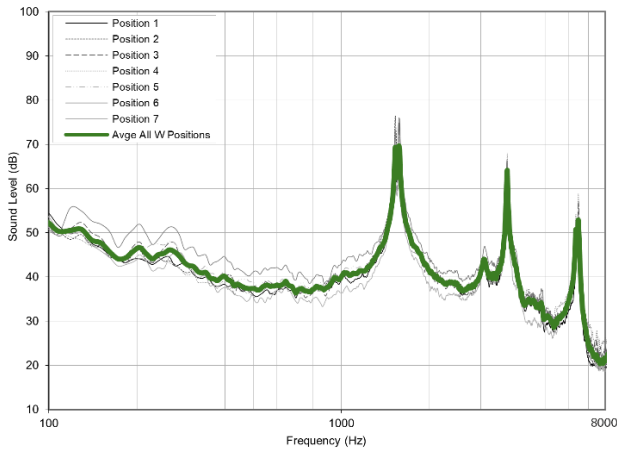
Figure 5. Airborne results (transfer function) related to all 7 positions, measured with the application of glue B: W (a) and X-axis direction (b).

As discussed in the previous section, the IAR parameter is adopted to correlate airborne results with the vibrating surface values [6]. This reference parameter was applied to evaluate the shift in peak positions caused by the application of glues compared to the response obtained before the glass was broken. The cross-spectra IAR calculations are plotted in Figure 5, representing the values before the glass rupture and with the application of glues A and B.

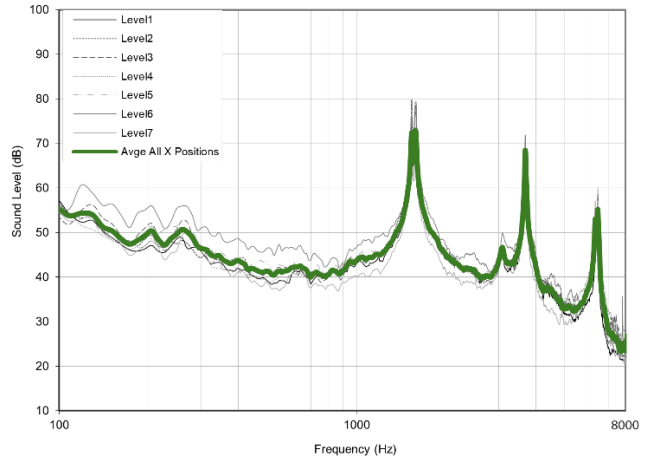
The graphs in Figure 5 demonstrate a strong correlation between the airborne and vibrational responses of the vibrating glass surface across the three selected scenarios. In addition, the results for the W and X-axis directions are very similar to each other, differing only in the magnitude of each peak.

The final step of data analysis examines the difference in sound level between the response of the glass vibrating in its intact condition and after reconstruction with glues A and B. These differences are represented in Figure 6. Notably, the analysis focuses on a narrower frequency bandwidth, ranging between 1000 to 8000 Hz, to emphasize distinctions that would otherwise be subtle or imperceptible.

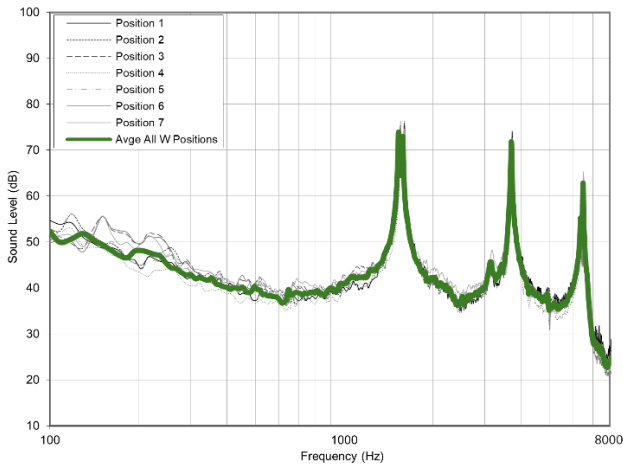




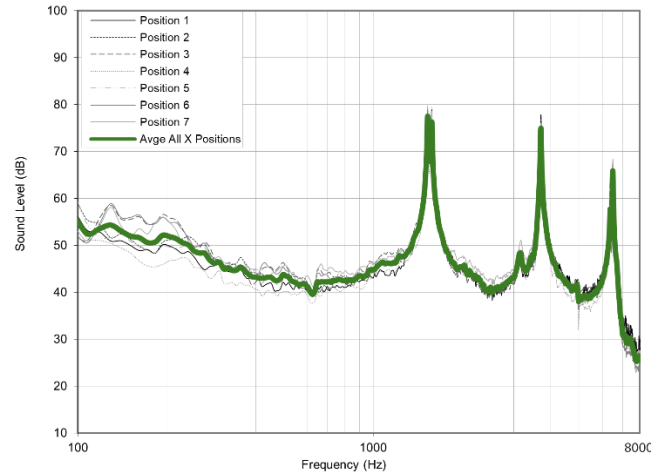
(c)



(d)

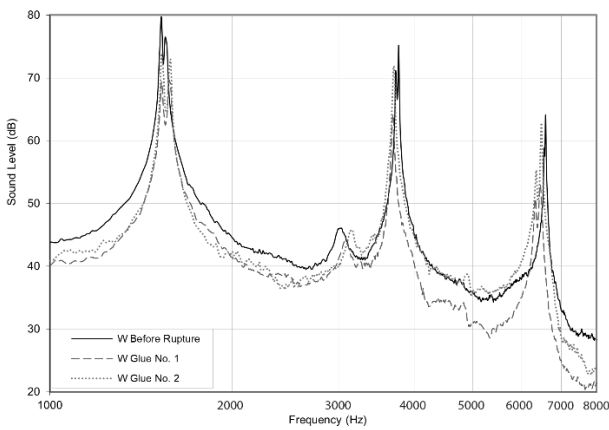


(e)

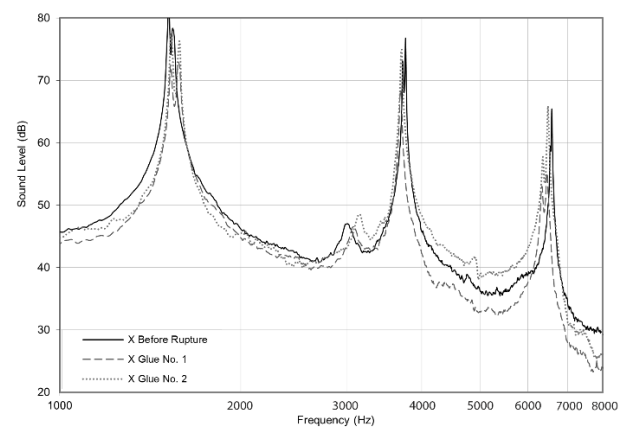


(f)

Figure 6. Cross correlation results (IAR) related to all 7 positions: W before rupture (a), X-axis before rupture (b), W application of glue A (c), X-axis application of glue A (d), W application of glue B (e), X-axis application of glue B (f).



(a)



(b)

Figure 7. Cross correlation (IAR) difference between the averaged values calculated in relation to the status of before rupture, after the application of glue A and B: W (a) and X-axis direction (b).

Graphs in Figure 7 shows that the application of glue B make the resonance of glass object closer to the integrity before rupture. The difference with respect to glue A is about 6 to 10 dB, which is a consistent amount that with the empirical method of ringing the glass with the fingers would not be possible to detect.

6 Discussions

This paper highlights the role of acoustic science in enhancing the success of glass conservation. While different adhesives behaviour has been indagated in other ways that include their resistance in time to UV and high temperature and their chemical compatibility with glass, this study highlights that IAR analysis enables a more precise evaluation of the glues used for unifying fragments, even when the differences are subtle. The results indicate that glue B outperforms adhesive A in terms of cohesion, as the resonance frequency peaks after the glass rupture are closer to those recorded when the glass was intact.

7 Conclusions

Advancements in technology have made professional tasks more precise and effective. Glass conservation, an art practiced since ancient times, initially emerged out of necessity due to the limited availability of handcrafted objects. Over time, the reconstruction of missing or fragmented glass pieces has become more specialised with the evolution of adhesive materials. From traditional animal gums and vegetal resins, the variety of modern solvents has expanded to include also foams for support, and other advanced bonding agents.

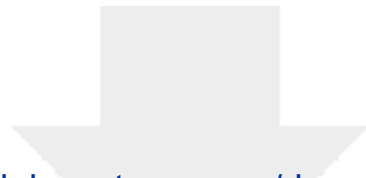
The research to find the best adhesive for glass conservation has, in the last decades investigated polymeric resins in many ways focusing on their behaviour in time, through artificial ageing in climatic chambers, but the vibrational behaviour of these substances has never been tested.

The methodology herein investigated has the potential to be further developed for conserving other artifacts, such as ceramic, porcelain, and terracotta. An ideal comparison of material responses can be achieved creating identical objects in various materials with the same shape and size. This approach, widely used in the reproduction of musical instruments for restoration purposes, underscores how technology continues to enhance the quality of professional restoration tasks.

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