ELIMINATION OF XYLOPHAGES FROM WOOD THROUGH MICROWAVE TREATMENT: MICROSTRUCTURAL EXPERIMENTS

Cesare Crova*
Istituto Superiore per la Conservazione ed il Restauro, Ministero dei beni e delle attività culturali e del turismo
Rome, Italy

Francesco Chiadini
Dipartimento di Ingegneria Industriale, Università degli Studi di Salerno
Salerno, Italy

Luciano Di Maio
Dipartimento di Ingegneria Industriale, Università degli Studi di Salerno
Salerno, Italy

Luigi Guerriero
Dipartimento di Architettura e Disegno Industriale, Università della Campania
Naples, Italy

Luca Pescione
Dipartimento di Ingegneria Civile, Università degli Studi di Salerno
Salerno, Italy

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1. Introduction

The relevant literature points to the effectiveness of microwave treatment in reducing or eliminating instances of deterioration in various types of materials caused by biological attacks. There are reports, for example, of the positive effects regarding the eradication of herbaceous vegetation from the stone facing of architectonic monuments, as well as annotations on the limits to be observed when utilising this technique, so as to avoid structural alterations in the materials [1]. In the same way, the effectiveness of microwave heating in disinfesting wooden structures under attack from xylophage insects has also been confirmed with a series of advantages, compared to previously used methods of chemical disinfestation: rapidity and effectiveness of the treatment; minimisation of health risks for the operators; absence of toxic residues and VOC emissions. In fact, trials have been conducted on the devitalisation of colonies of xylophages in wooden elements (floor and roofing frames, window and door fixtures) through exposure to microwaves for a period of approximately 3 minutes at a modest level of average absorbed power, 20 W (quantifiable as the difference between the average

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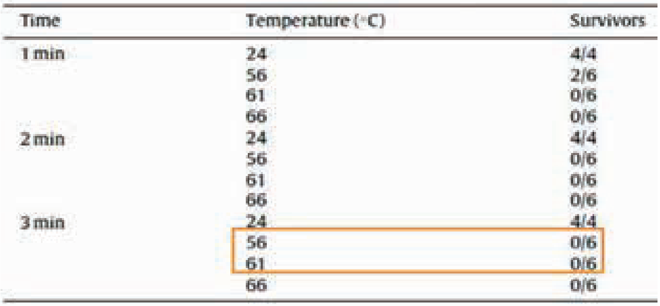
* Corresponding author: cesare.crova@beniculturali.it

incident power of 23÷25 W and the average reflected power of 3÷5 W), with the insects, no matter what their stage of development (egg, larva, pupa), being heated to the lethal temperature of approximately 60° C (Table 1; Figure 1). Although this technology is widely used for the restoration of antique furniture (placed in large-scale reverberation chambers and exposed to microwave treatments whose duration and intensity/power is set on the basis of empirical data, meaning the experience of the operators), trials have yet to be run to assess its effects on the microstructure of wood. The need for similar assessment is made even more urgent by the development of high-performance instruments [2, 3] and by the increasing use of on-site treatment techniques made possible by the introduction on the market, of microwave horn antennas and open application devices [4, 5] that can be used to radiate floor beams or roof trusses.

This study has assessed the potential for application of microwave disinfestation to wooden elements dating from the past (affected by low humidity and various forms of deterioration) through instrumental analyses designed to evaluate any microstructural changes in samples of chestnut wood (*Castanea sativa*) subject to microwave radiation for pre-set periods of time at controlled levels of power.

Table 1. Surviving xylophages based on the duration of treatment [6].



Time	Temperature (°C)	Survivors
1 min	24	4/4
	56	2/6
	61	0/6
	66	0/6
2 min	24	4/4
	56	0/6
	61	0/6
	66	0/6
3 min	24	4/4
	56	0/6
	61	0/6
	66	0/6

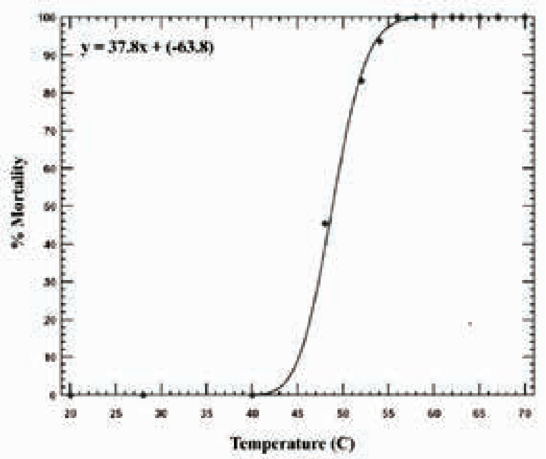


Figure 1. Percentage of xylophage mortality based on the temperature applied [7].

2. Objectives of the study

As is widely known, microwave heating is the result of diathermy: the application of an electromagnetic field to a material causes the water molecules in the material to rotate. This rotation is explained by the fact that the water molecules constitute a dipole with a momentum that is not nil, given that, as we know, the H^+ and O^- atoms are not aligned. The resulting oscillation generates kinetic energy that heats both the material and the xylophages found inside it. Seeing that the amount of heat produced is determined by the level of humidity in the wood, the areas most responsive to the treatment (those that heat up first) are the ones found further inside the structure, where the percentage of humidity is higher than in the outside layers.

There is evidence in the literature of the effectiveness of heating in terms of disinfestation and for reducing humidity and increasing permeability, but the consequences on the chemical/performance-related features of the wood treated are still up for discussion and there is no experimental evidence regarding the consequences of the treatment on the structure of the wood. In the case of new wood, assessments carried out to date have focused solely on performance and offer contrasting results: in some instances, the heating reduced the wood's properties of mechanical resistance [8,9], while in others, these properties were found to have increased [10].

3. Experimental trials

Experiments were carried out on samples of chestnut wood taken from beams (rounded beams simply stripped of their bark and found in the main frame) and from the "*chiancarelle*" (trunks of young chestnut wood smaller in diameter, split in half to form "*ginelle*" and used in the secondary frame) of the floors of Palazzo Lancellotti di Durazzo in Casalnuovo, near Naples [11]. A number of the samples date from the 18th century, while others are from the 19th century; all have been extensively exposed to biological attack (Figure 2). The samples chosen, therefore, consisted of wood of the same species, employed for structural uses but dating from different periods, and which had not undergone any previous treatment of preservation. To obtain comparative data, similar samples of recent wood were also subjected to the same experiment.

The analyses were carried out on two types of samples, one consisting of thin elements (7.2×4.0×3.4 cm), the other of thicker pieces (7.2×7.0×3.4 cm), all of them cut from the structural elements procured on-site, so as to render the dielectric measurements both convenient and suitable for comparison.

The heads of the beams showed evidence of fungus attack from *basidiomycetes* ("cavities" with deep lines of cleavage) and from *cerambycidae* xylophage, as demonstrated by the deep larval tunnels found in the alburnum. The "*chiancarelle*" trunks, on the other hand, showed evidence of attack by common woodworm (*anobium punctatum*), leaving the typical small-diameter holes (1-1.5 mm) from which the insects have emerged (Figure 3).

Figure 2. Samples of chestnut wood taken from the beam heads (above) and from the "chiancarelle" boards (below) originally found in structures of the 18th century (left) and the 19th century (right).

Figure 3. Longitudinal sections of beam heads showing the damage produced by large cerambycidae larvae, alternated with undamaged pieces. Larvae tunnels reaching the alburnum are visible amidst portions of undamaged wood.

The wood samples were exposed to microwaves in an aluminium reverberation chamber with an edge measuring 120 cm, powered by a 2-kW generator (magnetron) and an Alter SM 745 switching system. The power output of the magnetron is controlled remotely by a voltage signal of 0-10 V sent to the switch-mode power supply. The waveguide, equipped with a WR340 flange, consists of a circulator/isolator and a trunk waveguide with a directional coupler capable of providing readings of incident and reflected power using an *Anritsu ML 2438 A* dual-channel power metre outfitted with *MA 2472 D* sensors. The temperature is gauged by a *FISO TMI 4* thermometer with fibre-optic sensors. The fibre-optic probe is inserted into each sample through a hole with a diameter of 1.54 mm, down to a depth of approximately 35 mm, maintaining adequate contact with the wood sample, so as to keep air gaps from causing inaccurate temperature readings during the heating.

The samples were heated to a temperature of roughly 60 °C for a period of exposure of at least 3 minutes, at levels of average absorbed power of 20 W. These levels were chosen as being characteristic of effective treatment, with the percentage of surviving cerambycidae proving to be zero, in accordance with the literature on the subject [6].

An initial, readily apparent effect of the heating was a decrease in the mass of the samples. In light of the temperatures, which never exceeded 100°C, it can be concluded that this reduction is tied to the loss of unbonded water from the samples. An analysis of the temperatures shows that as the temperature increased in a nearly constant (linear) fashion for both the historic and the new samples, differences were observed in the reduction of the mass, which proved greater in the samples of historic wood (Figure 4). This observation, together with the fact that the density of the historic samples was significantly lower than that of the recent wood, makes it possible to conclude that different quantities of unbonded water are involved (or different levels of hygroscopicity), with the reduced quantity of water appearing to be tied to the deterioration at work in the older samples.

Pi = Power Incident = 23 ÷ 25 W ; **Pr =** Power Reflected = 3 ÷ 5 W
$\mathbf{P}_{\text{adsorbed}}$ **= 20 W** ($\mathbf{P}_{\text{adsorbed}}$ **=** $\mathbf{P}_{\text{incident}}$ **–** $\mathbf{P}_{\text{reflected}}$)
$\mathbf{T}_{\textbf{operating max}}$ = 100 ° C; **t** = 540 s ÷ 660 s;

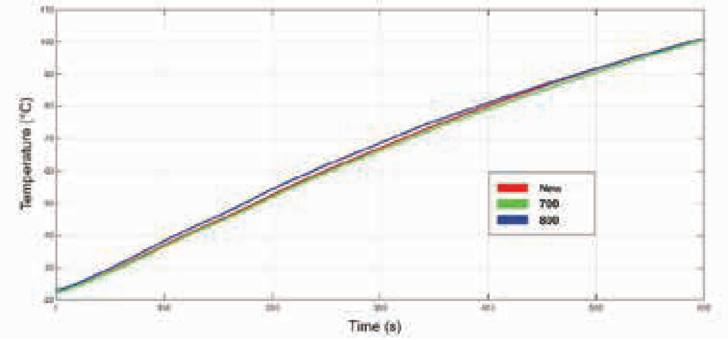


Figure 4. Temperature readings during the microwave heating of samples of chestnut wood from the 18th and 19th centuries, as well as modern samples, with an incident power of 24W, up to a maximum operating temperature of 100°C.

Both before and after the microwave treatment, the following experimental evaluations were carried out on the samples: dielectric readings, thermogravimetric analysis (TGA), infrared spectroscopy analysis (FT-IR); optical microscope examination.

Readings of complex permittivity were taken with an HP85070 dielectrometer connected to an HP85107 vector network analyser. These readings were taken to assess both the real portion, ε', and the imaginary portion, ε'', of the material's permittivity, given that wood is a non-magnetic, non-conductive medium. In taking the readings of dielectric permittivity, the samples were alternately gauged along the fibre and across the fibre to determine whether the anisotropy and the lack of uniformity of the material, influence the heating or the other parameters that come into play. Various readings were taken on wood samples with similar characteristics to arrive at average values for the dielectric constants. Table 2 shows the figures for the ε' and ε'' of the different samples:

Table 2. Dielectric constants of wood: ε' (real portion), ε" (imaginary portion).

Chestnut specimens	ε_1	ε_2
18th century chestnut specimens on fibre	2.5 ÷ 3.5	0.3 ÷ 0.7
18th century chestnut specimens cross-fibre	2.3 ÷ 3.6	0.25 ÷ 0.5
19th century chestnut specimens on fibre	2.5 ÷ 2.6	0.15÷0.35
19th century chestnut specimens cross-fibre	2.1 ÷ 2.5	0.15 ÷ 0.3
New chestnut specimens on fibre	2.2 ÷ 2.6	0.2 ÷ 0.38
New chestnut specimens cross-fibre	2 ÷ 2.6	0.15 ÷ 0.33

The readings of permittivity show that in the case of all the wood samples, regardless of their age, the loss tangent ε''/ε' was in the order of $10^{-2} \div 10^{-1}$, which represents an acceptable value for effective heating with microwaves. Given that the decrease in bonded water resulting from the microwave treatment corresponds to a variation in the electromagnetic characteristics of the materials, a comparison was made of the permittivity values and the loss tangent as measured both before and after the treatment, leading to the conclusion that for the materials exposed to the microwaves the heating was efficient.

The average values of the coefficients of the real portion and the imaginary portion were calculated, having been determined on the basis of the loss tangents of the comparative figures for all the samples exposed. The following table shows the average values as calculated, broken down by the different ages of the wood samples:

Table 3. Evaluation of the loss tangent tan δ for each category of samples and the related axis alignments.

Specimens on fibre	**Loss factor**
	$\varepsilon_2 / \varepsilon_1 = \tan\delta$
New	0.12
18th century	0.15
19th century	0.10

Specimens cross-fibre	**Loss factor**
	$\varepsilon_2 / \varepsilon_1 = \tan\delta$
New	0.11
18th century	0.13
19th century	0.10

The point of the thermogravimetric analysis (TGA) is to determine the typical temperatures of deterioration (pyrolysis) of the different constituent elements of the wood. The trial was carried out on wood dust taken from each sample, in the vicinity of the alburnum-duramen zone, both before and after heating. A comparison of the curves shows a greater loss of weight by the recent wood, as compared to the 18th and 19th century samples which presented a lower quantity of lignin, a reduction attributable to the deterioration at work. The analysis was performed in a temperature range of Tamb – 900 °C, with a heating speed of 10 °C/min in nitrogen, at 50 mL/min, for samples weighing between 8 mg and 10 mg [12]. The use of microwaves did not produce noteworthy divergences in the gravimetric curve, apart from a lowering of the curve between 50 °C and 100 °C, which points to the greater loss of unbonded water, while the main components (hemicellulose, cellulose and lignin) remained unchanged (Figure 5).

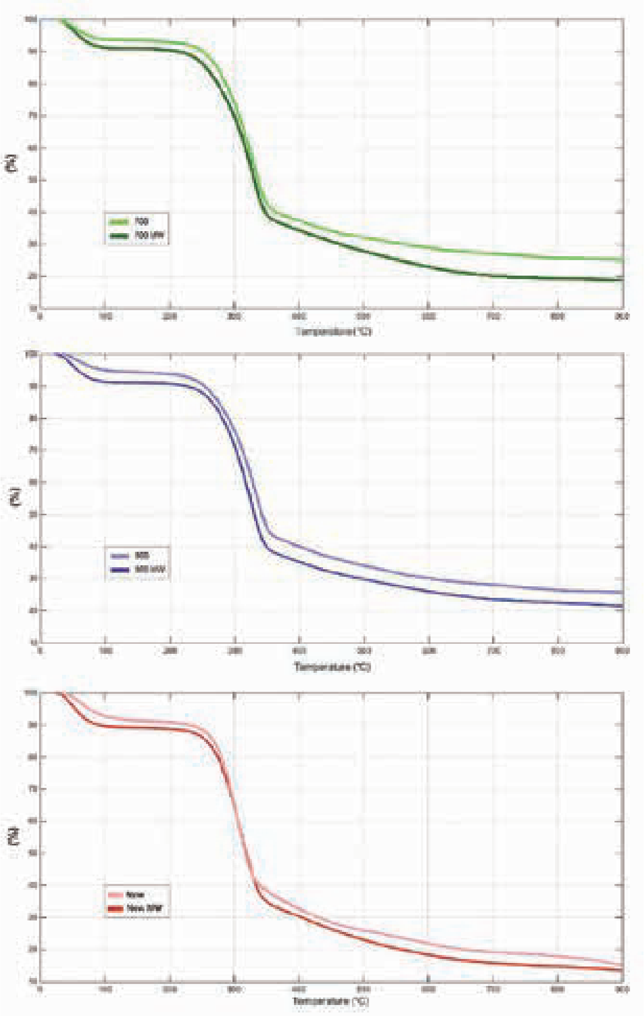


Figure 5. Overlapping thermogravimetric curves of the 18th century (1700s) specimen before and after the MW treatment (green curves - top), of the 19th century (1800s) specimen before and after the MW treatment (blue curves - centre) and of the new specimen before and after the MW treatment (red curves - bottom). The pre- and post-treatment curves regard the same samples. The lone difference is the clearly visible downward shift in the post-treatment curves, evidence of the greater loss of unbonded water by the samples that undergo microwave treatment, as shown by the downward inflection in the section extending up to 150 °C.

In accordance with the relevant literature [13,14], the graphs of the resulting curves can be subdivided into four primary sections: from Tamb – to approximately 150 °C: evaporation of the water and of the volatile light compounds; from approximately 150 °C to 260 °C: deterioration of the hemicellulose; from approximately 260 °C – 400 °C: deterioration of the cellulose and start of the deterioration of the lignin; from approximately 400 °C – Tsup: decomposition of the lignin.

Of particular interest is the infrared spectroscopy analysis (FT-IR), carried out with an interferometry spectroscope using the Fourier Transform method (FT), which serves to characterise different species of wood, though the approach is semi-destructive. The behaviour observed in the radiation wavelength range from 4000 to 500 cm^{-1} was similar for both the historic and recent materials, producing spectroscopic diagrams that resembled each other closely and proved comparable to those found in the literature [15]. The spectroscopic analysis did not reveal any noteworthy changes in the (minimum) peaks at any point along the length of the spectrum: for each wavelength of infrared radiation, the readings of the polymer components were the same, on account of the components' stability, even after the microwave treatment. The only change was the intensification of these peaks following heating (higher minimums), traceable to a morphological change, as confirmed by the relevant literature [16,17]. In light of the above, it can also be concluded that the microwave treatment did not result in modifications of the wood or of its constituent elements (hemicellulose, cellulose and lignin) (Figure 6).

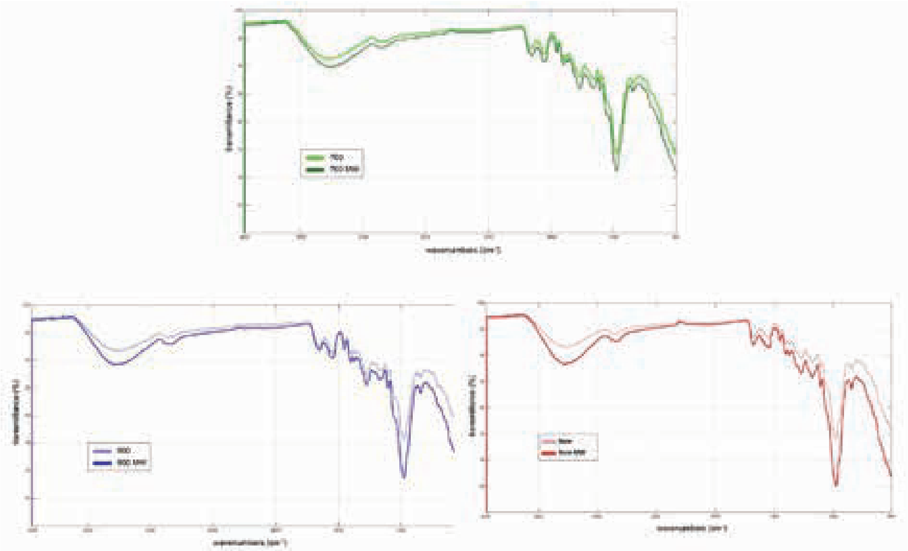


Figure 6. Overlapping FT-IR spectrums of the 18th century (1700s) specimen before and after the MW treatment (green curves - top), of 19th century (1800s) specimen before and after the MW treatment (blue curves - left) and of the new specimen before and after the MW treatment (red curves - right).

Table 4 below presents the wave peak number associated with the polymeric component evaluated, together with the status of the peak:

Table 4. Identification and attribution of the peaks of the infrared spectrums.

Characteristic peaks (cm^{-1})	**Peak level**	**Polymeric component**
3600-3200	*intense*	Stretching OH
1715	*medium*	Xylan (hemicellulose)
1607	*medium*	Lignin
1453	*weak*	Lignin
1323	*intense*	Cellulose
1216	*medium*	Lignin
1157	*medium*	Lignin
1030	*intense*	Polysaccharide (hemicellulose and cellulose)
895	*weak*	Polysaccharide (hemicellulose and cellulose)

The first conclusion to be reached from the FTIR analysis is that the spectrums of the three wood samples, though originating from different periods, present the same peaks, indicating a nearly identical composition. This translates into an absence of deterioration which, on the other hand, would have been present, had differences in the types and numbers of peaks found along the spectrum been observed. At the same time, it can be noted that, in the case of all three samples, the spectrums generated by the analysis carried out following the microwave treatment present a slight difference in intensity: one that proves greater for the treated samples. The difference is more acute in the case of the new wood, as opposed to the old, with this heightened intensity seeming to point to a slight increase in the density of the sample following the microwave treatment, corresponding, as was shown by the TGA analysis as well, to a gradual, heightened loss of unbonded water on the part of the more recent samples, as opposed to those from the more distant past.

The observations made with an optical microscope, by means of transmitted light, also provide confirmation of the feasibility of using microwaves for xylophage disinfestation; in fact the samples, viewed both before and after treatment - and regardless of whether they were historic or new, even when viewed at different levels of enlargement - did not present visual differences, preserving the same configuration and the same dimensions of the constituent elements of the parenchymatous tissue, just as was observed for the dimensions of the primary constituent parts (Figure 7).

In the image shown in Figure 7, obtained with an optical microscope at an enlargement of 5x, the main constituent parts of the structure of the chestnut wood can be identified, with highlights including portions of the early wood (darker in colour, with veins of larger diameter) and portions of late wood (more lightly shaded, with veins of smaller diameter). The change from late wood to early wood points to the start of a new growth ring, indicating the dating, and thus the age, of the element. The arrangement of the parenchymatous radii can be observed in the image, as shown by the continuous black horizontal lines and the thick array of points distributed uniformly throughout the section, coinciding with the wood fibres.

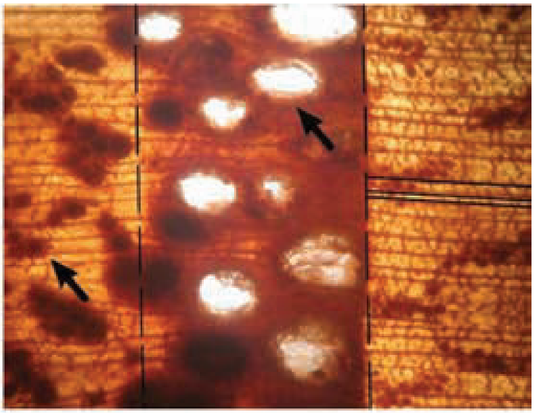


Figure 7. Transversal section examined under optical microscope (5x).

Figure 8. Radial section examined with optical microscope, pre-treatment (on left, 5x), post-treatment (on right, 20x).

4. Discussion

The experimentation carried out on the use of microwaves to disinfest chestnut wood under attack from xylophages has shown that the utilisation of this technique does not lead to modifications in the structure of the wood or in its primary constituent elements (hemicellulose, cellulose and lignin), whereas it does give rise to variations in hygroscopicity and permeability. These effects are more apparent in the historic wood samples, which showed reduced quantities of bonded water, while at the same time, no secondary effects of deterioration in the structure of the wood was observed.

5. Conclusions

In the future, there will be other fields of research to explore involving the use of microwave heating. Of particular importance will be ongoing efforts to evaluate the effects of this technology on wood structures of cultural interest, with a special focus on: permeability to water; evaluation of the properties of transport within the wood structure; confirmation of the morphology of the main constituent elements through specific analyses (X-ray diffraction); the compatibility of the treatment for wood elements decorated with tempera paint or covered with "overlays" (determining whether the painted portions would be damaged or if there is a risk of the overlays detaching from the wood support).

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Biographical notes

Cesare Crova graduated in Architecture; he is a specialist in Restoration of Monuments and has a Ph.D. in Conservation of Architectural Heritage; he is a professor in Architecture Pathology at ISCR SAF. He has carried out teaching and research at IUAV, the "Sapienza" University of Rome, the University of Molise and the Cultural Heritage Department at the University of Padua. He works in the field of cultural heritage conservation, where he is part of several working groups at MiBACT, in addition to planning and directing restoration work. He is the National Vice President of Italia Nostra and a member of the CDA of the Circeo National Park; he is a committee member of the UNI Technical Corps at UNI / CT 033 / SC 01 / GL 01 "Guidelines and terminology" and a member of MiBACT Unite 4 Heritage (MiBACT H4U), "Blue Culture Helmets ".

Francesco Chiadini is an electronics engineer, Ph.D. in Information Engineering and researcher at the Department of Industrial Engineering at the University of Salerno. He has been Chairman of many international congress sessions; Research Scholar at Pennsylvania State University (PA-USA); Science Monitor at Livingston's Laser Interferometer Gravitational Wave Observatory (LA-USA); Member of the International Doctoral Program for the assignment of the Doctor of Research at the Fakultät für Mathematik und Informatik at the FernUniversität in Hagen; Member of the Local Workshop IV Workshop on Metamaterials and Special Materials for Electromagnetism and Telecommunication (MMSM 2008). He is currently a member of the Program Committee for the Optics + Photonics Conference: Nanostructured Thin Films and the Smart Structures / NDE Program Committee - Bioinspiration, Biomimetics, and Bioreplication Conference. He has been elevated to the rank of Senior Life Member of the SPIE Scientific Society "in recognition of the significant contribution given to the Scientific Community in Optics and Photonics".

Luciano Di Maio, engineer of Industrial Technologies at Chimico-Food, Ph.D. in Chemistry and New Materials Research. From 1999 to 2004, he was a researcher of Principles of Chemical Engineering. Since 2005, he has been an Associate Professor of Science and Technology of Materials, Faculty of Engineering, University of Salerno, where he teaches courses for the Degree in Chemical Engineering, Civil Engineering, Degree courses in Food Engineering and the Single Cycle Degree in Architecture Engineering. He is a Member of the Board of Directors of the Interdepartmental Research Center at the University of Salerno "Nanomaterials and Nanotechnologies (NANO - MATES)"; member of the selective exam committee for university researchers; chairman and member of commissions for the conferment of research grants; referee of the international magazine "Polymer Engineering & Science" and a member of various scientific associations; an aggregate member of the State Examinations Committee for the license to practice as an engineer; chairman of the guidance and tutoring committee of the Academic Council for Chemical Engineering.

Luigi Guerriero graduated in Architecture; he has a Ph.D. in Conservation of Architectural Heritage. Since 2000 he has been an associate professor of Restoration at the Faculty of Architecture of the II University of Naples. He has organized and directed research groups. He has coordinated national scientific initiatives. He has been a member on the scientific council for national meetings and exhibitions. His research concerns the theories and history of restoration, particularly regarding the protagonists and interventions of the mid-twentieth century, the mensiochronologic characterization of traditional constructive elements, with the related protocol of analysis of deterioration and of structural modeling and the methods and techniques of urban restoration.

Luca Pescione graduated in Construction Engineering and Architecture at the University of Salerno; his studies cover the experimental field of Chemistry and Technology for the Restoration and Conservation of Materials, with particular regard to microwave processing in the restoration of eighteenth-century wooden elements. He is mainly engaged in architectural design and restoration, both as a freelancer and in specialized companies.

Summary

Samples of chestnut wood taken from modern age structures in the Campania Region were subjected to chemical and physical analysis in order to examine the microstructural variations in the material as a result of exposure to microwaves with the aim of devitalising colonies of xylophages. Results showed that the use of microwaves does not lead to alterations in the structure of the wood but does bring about variations in its hygroscopicity and permeability.

Riassunto

Campioni di legno di castagno, estratti da strutture campane di età moderna, sono sottoposti ad indagini chimiche e fisiche, allo scopo di evidenziare le variazioni microstrutturali indotte sul materiale dall'esposizione alle microonde finalizzata alla devitalizzazione di colonie di insetti xilofagi. I risultati sperimentali mostrano che l'impiego delle microonde non de-termina alterazioni nella struttura del legno, ma induce variazioni di igroscopicità e di permeabilità.