MICROWAVE TREATMENT OF STONE SURFACES FOR THE DEVITALIZATION OF BIODETERIOGENS

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Corrected in date 2020-04-03, see the erratum:
https://doi.org/10.6092/issn.1973-9494/10735

Keywords: disinfestation, pyroclastic lithotypes, stone materials, structural analysis

1. Introduction

The state of cultural heritage is increasingly exposed to a continuous risk of biological degradation caused by the attacks of photosynthetic biodeteriogenic agents or heterotrophic organisms. The damage caused is both aesthetic and structural, and requires an innovative, non-invasive cleaning method. Through the controlled use of microwave (MW) irradiation on stone materials disinfestation is obtained without altering the original state of conservation and greatly reduces risks for the operators [1]. This protocol, already proven to be effective on paper, cloth and wood, was executed on the building materials typical of the architectural tradition found in the region of Campania. The materials included yellow and gray tuff samples taken from a natural quarry in the geographical area of the Agro Nocerino-Sarnese, together with samples of cotto and lime mortar taken from an abandoned and unused ruin located in Sant' Eustachio in the area of Avellino (AV) (Figure 1 and 2). The experimentation consisted of exposing portions of pyroclastic lithotypes, infested with biodeteriogens such as molds, mosses, lichens and herbaceous vegetation, to microwaves for different lengths of time and using different power outputs. Analyses were carried out on each individual sample, before and after exposure, and again after powdering, to ensure uniformity [2].

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Figure 1. Example of "bozzette" (smaller-sized blocks) masonry of Neapolitan yellow tuff (18th century).

Figure.2. Example of "block" masonry of gray Campanian tuff (19th century).

This procedure allowed us to establish the basis of a new protocol that included colorimetric analysis, thermal analysis, spectroscopic analysis, X-ray diffraction, SEM, evaluation of transport properties and compressive strength, so as to guarantee the chromatic and microstructural integrity of the material at the end of the cleaning process [3].

Preserving the authenticity of historical materials is one of the main objectives of restoration. It is therefore necessary to ensure their proper conservation using increasingly innovative methods.

The aggressiveness of the substances used, treatment times, penetration depth, and risks to operators are the limits of the currently available techniques for cleaning stone surfaces affected by biodeteriogens, it is therefore necessary to experiment with a new protocol that is able to overcome these issues [4].

The new protocol to which reference is made is microwave cleaning (MW), which guarantees the cleaning of the material (yellow and gray tuff) without altering the microstructure of the stone and avoiding direct interaction with the material.

The tests were carried out on the untreated yellow and gray tuffs from a natural quarry of the Agro Nocerino-Sarnese, performing the microwave treatment only on a part of the collected material, so as to be able to compare the material treated with microwaves with the untreated material.

The use of this technique on the wall portions of the architectural assets, in general in stone affected by infestation, proved to be effective thanks to the selective microwave heating based on the dielectric characteristics of the materials, and to act only on the biological infesting organisms due to the fact they consist mainly of water [5].

2. Materials and methods

The methodology used for experimentation on the stone materials consists of two phases: in the first phase the samples are placed in the reverberating chamber of the microwave laboratory of the University of Salerno, in the second phase they are subjected to chemical-physical analyses. This procedure is carried out first on an intact sample of material, placed in the reverberation chamber and subjected to the isotropic exposure process. The chamber is an aluminum cube (120 cm in length) equipped with two stirrers, with rotation modes that work at different speeds (0.4 and 1.1 rps); a magnetron powered by 2.45 GHz, 2 kW CW with a switching power supply. The system is completed by data acquisition software that measures the incident and reflected power at the entrance of the chamber. With the obtained data, it was possible to establish a range of values for the power and exposure time parameters, which were modified in order to establish whether there are threshold values beyond which changes in the structure of the stone material are found. This system allows the magnetron to operate at different power levels, from a few watts to 2 kW. Measurement of the incident and reflected power at the input of the reverberation chamber is performed using a directional coupler. The samples are treated with an incident power of 400 ÷ 900 W for 60 ÷ 180 s (Figure 3) [6].

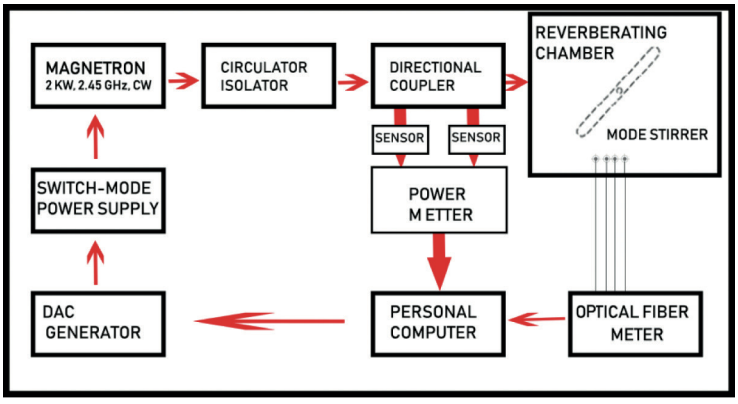


Figure 3. MW exposure system.

Figure 4. Grinding of yellow tuff.

In order to evaluate the chemical composition and the effect of the treatment with MW, FT-IR diffraction and X-ray diffraction analyses were performed on both the treated and untreated tuff samples, followed by colorimetric analysis to verify possible aesthetic alterations, and a water absorption test through full immersion and capillary ascent, and uniaxial compression resistance to verify both the transport properties and the durability of the tuff samples [7].

The same analyses were then carried out on samples of material reduced to a powder form by manual grinding in a mortar and subsequently homogenized to guarantee uniformity between the samples (Figure 4).

The material was then divided into 36 vials and subjected to 4 levels of exposure cycles of microwave treatment by increasing the power output as follows: 400W, 500W, 600W, 700W. For each passage at the respective temperatures, 9 samples were exposed, of which 3 with an exposure time of 60 seconds, 3 with an exposure time of 90 seconds and 3 with an exposure time of 120 seconds. The samples to be subjected to the irradiation consisted of glass tubes with a total weight of 13.5gr, which were labeled and then filled with just over one gram of tuff powder (Figure 5).

The samples were weighed with precision scales to one thousandth of a gram before and after the treatment to evaluate the percentage of weight loss. The data acquired by the software show the incident and reflected power, evaluated at the input of the camera (in correspondence with the waveguide); by comparing these data with the previously acquired data, the absorbed power is obtained. After performing microwave treatment on all the samples, each one was then analyzed chemically and physically, to evaluate the triggering of any alteration processes. The following investigations were carried out: thermogravimetric analysis (tGa), X-ray diffraction, infrared spectrophotometry and differential scanning calorimetry (DSC) (Figure 6).

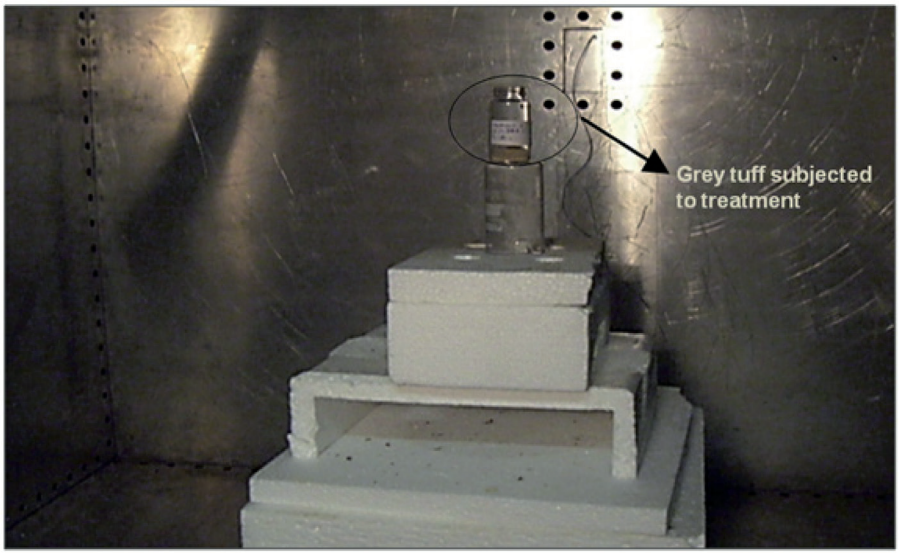


Figure 5. Reverberating chamber used for the exposure of the tuff samples to microwaves.

More specifically, the tests performed for both treatments, that is, on the whole tuff sample and on the powdered tuff sample were:

X-ray diffraction, which is used to verify the detection of "collapses" of the crystalline phases in amorphous phases; this is a process in which an X-ray beam, incident on a crystal sample, is diffused by the electrons of the latter (scattering process). From the study of the angular distribution of the diffracted radiation we go back to the shape, the size and the orientation of the elementary cell.

X-ray diffraction is commonly realized with samples consisting of microcrystalline powder of the material, in which the image produced by X-ray diffraction is recorded and analyzed to reveal information on the nature of the lattice and on any components in a mixture of polycrystals. The ground sample is placed on the support and inserted into the diffractometer. This instrument consists of two arms: on one arm is the source from which the x-ray beam is sent; on the other is mounted the counter which, by rotating, reveals the diffracted rays. These rays, once captured, are processed by a computer. The image returned by the software is a diffractogram characterized by a series of peaks, each of which represents the diffraction event. The height of the peaks is directly proportional to the intensity of the diffracted radiation. The analyses were carried out using a Bruker D8 advance diffractometer and then comparing the results of the untreated tuff with those of the same material subjected to the various treatments.

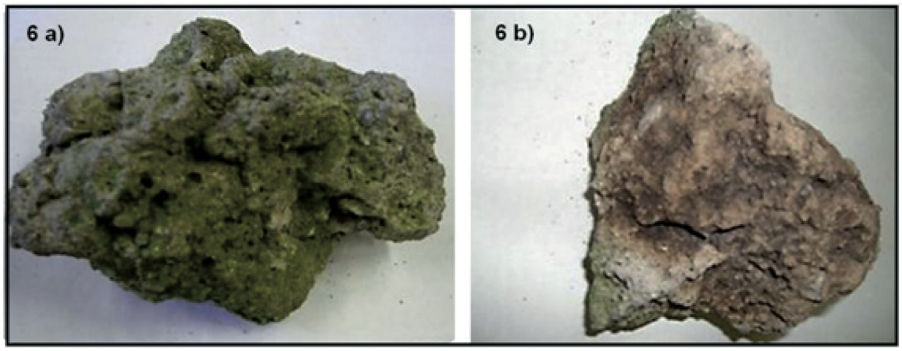


Figure 6. a) Gray tuff infested with lichens before the treatment with MW; b) gray tuff infested with lichens after the treatment with MW.

Thermogravimetric analysis, which is useful to evaluate the processes resulting from the heating suffered by an element for which the mass variation is constantly recorded. Depending on the temperature of the sample under examination it is possible to identify the chemical-physical processes it has undergone. In fact, for each element, by virtue of the weight loss and the corresponding temperature, it is possible to identify the transformation it has undergone (evaporation of water, dissociation of compounds, oxidation processes, etc.). These data are expressed by a thermogram (tGa curve) which shows the temperature or time on the x-axis, and the weight variation on the y-axis (Figure 7).

For tuff, as in general for inorganic materials, the water present can be classified in relation to the interaction with the structure: free water, hydration water, crystallization water or strongly bound water. The instrument used for thermal analysis consists of a microbalance, a gas purge system that guarantees an inert environment, the furnace that can be heated up to 1000 °C, and a data acquisition and processing system. The gas usually used to control the atmosphere around the sample is nitrogen, which prevents oxidation processes (which could cause a weight variation). Before carrying out the analyses, the capsules are previously cleaned in a flask at 1000 °C, then inserted into the thermo-gravimetric cell and the calibration process is carried out. The analyses were performed using a Netzsch thermal analyzer, and nitrogen was used as purge gas. During the test the sample was equilibrated at a temperature of 20 °C and heated to a final temperature of 600 °C, with a heating rate of 10 °C per minute [8].

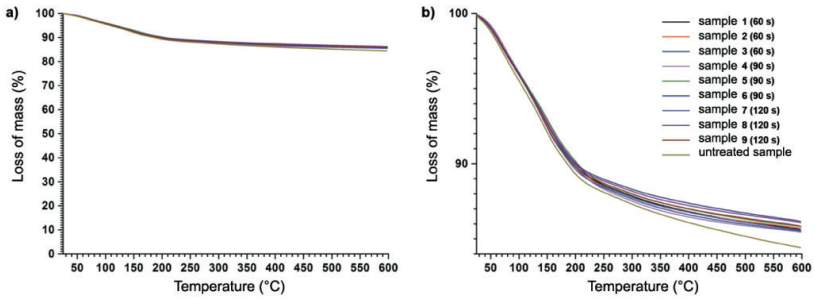


Figure 7. a) Overlap of thermogravimetric profiles of yellow tuff samples treated and not treated with microwaves at the power of 400 W with corresponding magnification; b) exposure times are shown in brackets.

Infrared spectrophotometry, which is an analytical technique used for the identification of organic and, in some cases, inorganic materials; it allows information to be obtained on the functional groups of the molecules of the analyzed substance [9]. The FTIR technique (Fourier Transform Infrared Spectroscopy) measures the wavelength range of the infrared spectrum that is absorbed by the material. The absorption of the infrared radiation produces in the molecules some characteristic vibrational motions, defined as stretching and bending. The stretching is the result of the continuous variation of the bond distances between two atoms and can be symmetric or asymmetric; the bending, on the other hand, refers to the modification of the binding angle on the same plane or outside the plane on which the angles are considered. The amount of energy needed to produce each vibrational motion depends directly on the strength and polarity of the bonds between the atoms of the analyzed molecule. The test consists in subjecting the sample to an electromagnetic radiation that interacts with the matter. In the case of solid substances, it is necessary to reduce the material to a powder and arrange a quantity equal to about 5 mg in a small mortar; the sample is then homogenized with KBr up to an amount of 100 mg. The resulting sample is placed in a special holder, which must then be pressed to obtain a disk with a thickness of a few μm. To be analyzed, it is inserted into a metal support with a suitable hole. Before testing the material, the empty support must be inserted as a reference for the analysis. The recognition of substances by analysis is often based on the comparison of the test spectrum with that of known substances or with spectra reported in the literature (Figure 8) [10].

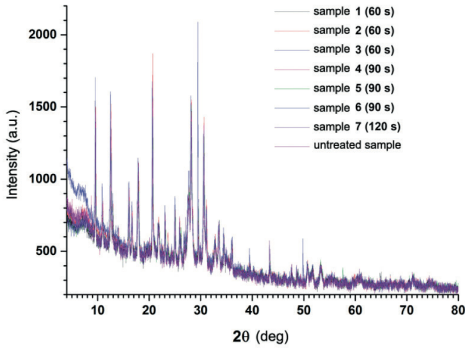


Figure 8. Superimposition of WAXD diffractograms of yellow tuff samples treated with microwaves at the power of 400 Watt; exposure times of the samples are shown in parentheses (LEGEND: deg= Temperature degrees; a.u.= Arbitrary Unit; s= Seconds).

Differential scanning calorimetry is a thermal analysis which, together with thermogravimetric analysis, provides information on some physical properties of a sample subjected to a controlled thermal cycle. Differential scanning calorimetry (DSC) allows information to be obtained on some physical properties of a sample subjected to a controlled thermal cycle, providing information on the heat reaction of the material. This information is taken from a differential measurement of the thermal flux between the test sample and a reference sample. In fact, when the sample undergoes an endothermic process (heat absorption), the system must provide a higher quantity of heat in order to maintain the same temperature, equal to that of the reference sample. Before carrying out the test, the sample is placed in a crucible, net weighed up to the fourth decimal digit, and closed using a small press. It is then inserted into the analysis instrument together with an empty crucible that will serve as a reference for the differential measurement. Three "lids" then allow to hermetically insulate the test cell from the outside.

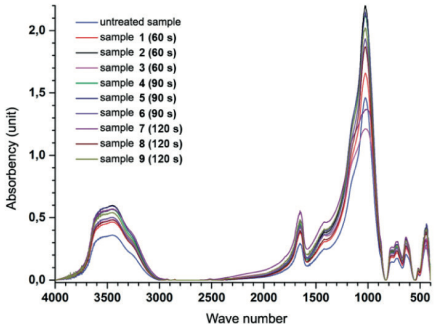


Figure 9. Overlap of IR spectra of yellow tuff samples treated and not treated with MW. The power of the treatment is 400 Watt; the exposure times are shown in parentheses (LEGEND: Wave number= intensity of the waves expressed in cm; Absorbency= absorbency of the material expressed in arbitrary unit; s= Seconds).

A continuous and uniform flow of nitrogen is created inside the furnace, which avoids any oxidation which could distort the result. During the test, the heat given off by the furnace heats both the sample and the reference crucible in the same way, therefore any variation in temperature between the two is due to phenomena that arise in the material to be analyzed: there will be a rise in temperature due to exothermic reactions or a lowering due to endothermic reactions.

Throughout the whole experiment, a thermocouple-based system collects the temperature data and sends it to a computer where it is processed, generating output using a special software (Figure 9-10) [11].

3. Results and discussion

The results of the analyses obtained by comparing the treated and the untreated material, in the form of “pieces” and “powder”, showed that in both cases there were no changes in the structure of the material as a result of the microwave treatment. For the experimentation, several treatment cycles were carried out, progressively increasing the incident power and the exposure time. In this way we tried to show whether by making the treatment more “aggressive” there were structural alterations in the material. The results of the analysis obtained from the comparison between the treated and the untreated material showed that after the microwave treatment there were no changes in the structure (Table 1) [12].

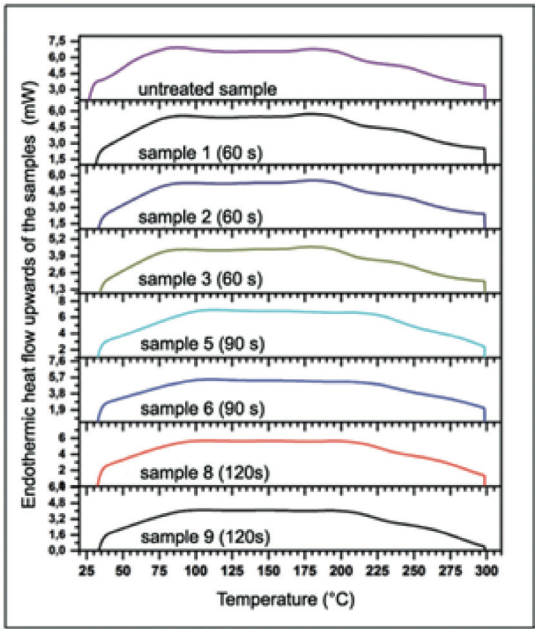


Figure 10. Overlap of DSC thermograms of treated and non-microwaved tuff samples with a power of 400 W (exposure times are shown in parentheses).

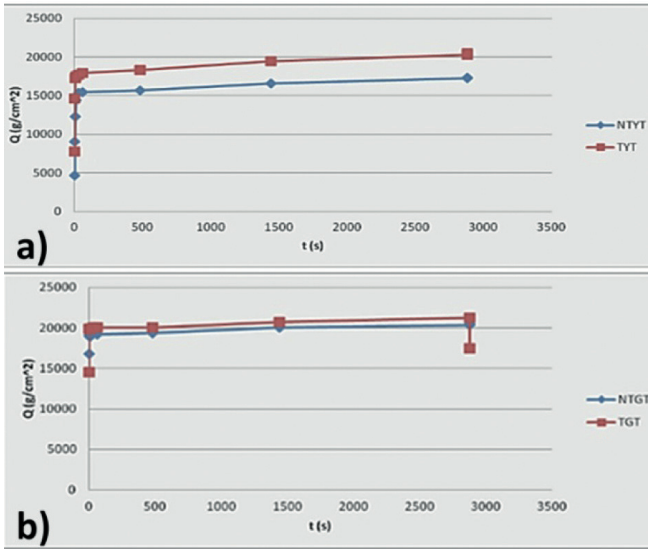


Figure 11. a) Curves of water capillary rise vs square root of time relative to yellow tuff; b) grey tuff.

Table 1. Comparative analysis (grey and yellow tuff).

	GRAY TUFF	YELLOW TUFF
Exposition Time	For this material, up to 9 minutes of treatment was reached, time for which disinfestation was obtained. This material which is "resistant" to microwaves could be treated, if necessary, for longer times.	For the yellow tuff you can not exceed 3 minutes of exposure beacuse the material would be color-altered.
Incident Power	Power of 700W were effective for the disinfestation of this materialbut in the preliminary treatments the treatment reached 1000W not showing colorimetric alterations.	It was not possible to exceed 500 W.
Reaction Of The Pest	The power and time of the treatment chosen for the disinfestation of the material were effective for both moss and lichens.	The power and time of treatment were effective only with regard to moss, for lichens the power and time of treatment should be increased.
Changes In The Microstructure	From the chemical-structural analyzes relating to the samples treated with the test protocol deemed suitable, no obvious differences were found in the material treated with respect to the material as it is.	Results similar to those obtained for gray tuff.
Aborption Capacity Of Water Afetr Treatment	The treated sample initially adsorbs more water than the untreated. This can be explained by the fact that the material has lost unbound water during treatment.	The sample absorbs more water than the gray tuff, due to the typical nature of this material.

In particular:

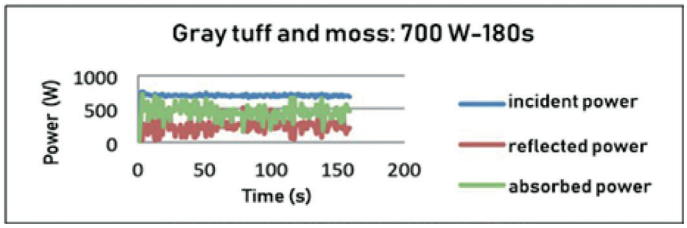
− the thermogravimetric analysis showed that when the parameters increase, the tuff loses a certain amount of water, mostly free and weakly bound water that does not influence the part linked to the structure of the material (Figure 11);

− the differential scanning calorimetry, used to complete the results of the TGA, confirms that water loss corresponds to those of free and weakly bound molecules; the colorimetric analysis shows that no perceptible chromatic alterations or aesthetic modifications occurred;

− the X-ray diffraction showed perfectly overlapping spectra and therefore no alteration of crystalline phases; the infrared spectrophotometry confirmed that there is no alteration of the structure;

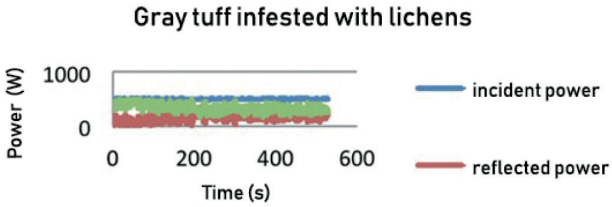
− the transport properties relative to the treated gray tuff presents an increase in water transport properties, but not to a significant extent as in the case of the yellow tuff (Table 2-3);

Table 2. Power-Time Parameters graphic (700 W-180 s).



GRAY TUFF INFESTED WITH MOSS			
WEIGHT (grams)	**POWER** (watt)	**TIME** (seconds)	**Notes**
246	700	180	
239			The sample lost 7 grams. The weed is wet
233	700	360	The sample lost 13 grams

Table 3. Power-Time Parameters graphic (500 W-600 s).



GRAY TUFF INFESTED WITH MOSS			
WEIGHT (grams)	**POWER** (watt)	**TIME** (seconds)	**Notes**
328	500	600	
317			The sample lost 11 grams

It can be observed that, according to the results of the previous experiments, there are no effects that lead us to believe that microwaves damage the tuff (Figure 12). It is important to underline the difference in the "state" of the material exposed in the chamber: in the first case, "pieces" of stone were treated, while in the second case, samples of tuff dust were exposed inside vials. This could lead one to think that, given the porous nature of the material, there may be different responses to increasing power outputs. Although temperature readings were taken during the control of the MW material, due to the limits of the instrumentation it is not possible to arrive at an accurate treatment; thus, the temperature reached during the intervention is not known. This data could have better defined the possible transformations suffered by the material, because at certain temperatures some components of the tuff collapse [13].

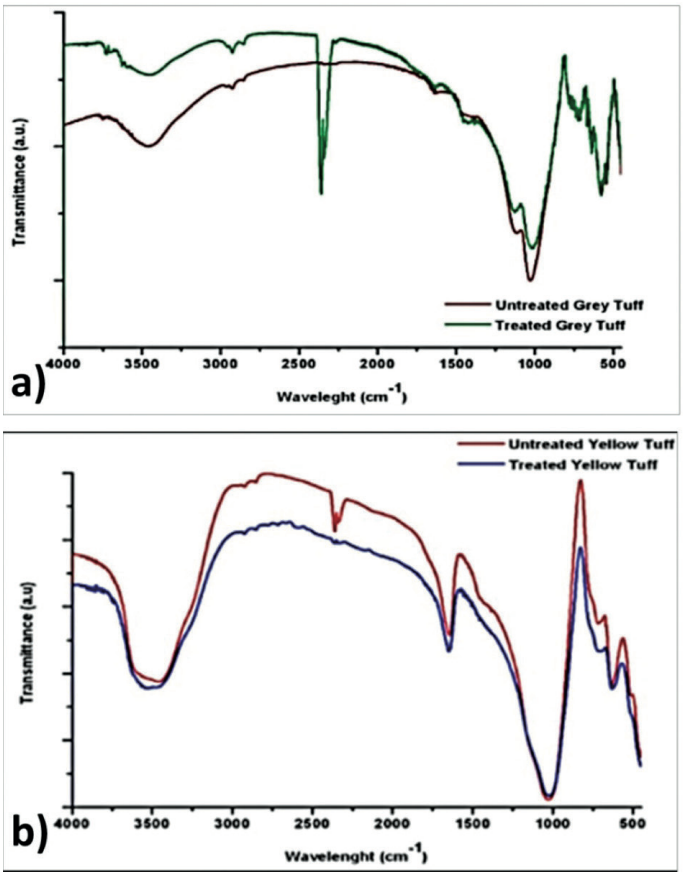


Figure 12. FT IR spectra of tuff sample: a) Gray tuff: 700 W, 9 min; b) Yellow tuff: 500 W, 3 min.

4. Conclusion

The present paper has examined a detailed range of analyses that supports the fact that the absence of structural changes in the yellow and gray tuff show that microwaves do not cause any damage. It can be concluded that the heat treatment of stone materials with microwaves effectively devitalizes pests, while the substrate of the material in question remains unchanged. This technique therefore avoids direct interaction with the tuff and does not modify its microstructure.

It would therefore be interesting to consider this technique as a possible solution in the disinfestation of architectural assets, as it aims to verify the feasibility of the method for disinfesting biodeteriogens from stone cladding (yellow tuff, gray tuff) both to preserve the chemical-structural integrity of the material, and to reduce exposure times and risks associated with the use of toxic substances. It also guarantees complete control of all operations by the operator. It is evident that the results obtained change in virtue of the material subjected to irradiation, therefore this arises as a first study from which an interesting research field for architectural restoration could develop.

Acknowledgements

E. Avallone M.D. and E. Caliendo M.D. Department of Industrial Engineering, University of Salerno, Italy, for support in reengagement of surveys; F. Chiadini M.D. and A. Scaglione M.D Department of Industrial Engineering, University of Salerno, Italy, for support in the microwave treatment; A. Fondacaro M.D. and L. Incarnato M.D. Department of Industrial Engineering, University of Salerno, Italy, for their scientific contribution in the surveys.

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Biographical notes

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 and 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 protocols for the analysis of deterioration and of structural modeling and urban restoration methods and techniques.

Margherita Cicala obtained *a magna cum* laude for the title of doctor at the University of Architecture of the II University of Naples.

Nicola Chiacchio obtained the title of doctor at the University of Architecture of the II University of Naples. He collaborates on research projects for Snecs (Social Network of the Historical Center Entities) and Chis (Cultural Heritage Information

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Bruno Bisceglia graduated in Electronic Engineering in 1984 and was appointed Assistant Professor of Electromagnetics in 1988. In 1989 he joined the Society of Jesus, and in 1996 was ordained priest. After graduating in Fundamental Theology in 1996, he defended his Doctoral thesis in Theology in 2002. He has been Adjunct Professor of Bioelectromagnetic Interaction in the University of Sannio at Benevento and Adjunct Professor of Electromagnetic Pollution and Risk Management and Mobile Communication in the University of Salerno.

Summary

The experimental work described in this paper is aimed at evaluating the possibility of applying microwave heating to a restoration project. The technique involves the application of microwaves to stone surfaces affected by biodeterioration. Results showed that the pests were completely devitalised. The materials chosen for the survey were yellow and grey tuff. Samples of both tuffs were taken in the area of agro nocerino-sarnese and samples of lime mortar and brick were taken from a ruin located in Saint Eustachio (AV). The methodology consists of two phases: in the first, samples of material are placed in a reverberation chamber and treated by varying the parameters of power and exposure time; in the second, they underwent chemical-physical analysis aimed at establishing whether the treatment had induced aesthetic and/or structural alterations. The procedure was performed both on intact samples and on powdered samples for further analysis. Specifically selected and treated samples were subjected to colorimetric analysis, thermogravimetric analysis, of x-ray diffraction, infrared spectroscopy, SEM, and analysis of transport properties and compressive strength. Results from the chemical and structural analyses relating to the samples treated with the protocol showed no differences between the treated and untreated material. The only difference between the two types of material used related to the absorption capacity of water after treatment. From this, it can be deduced that the heating of stone materials using microwaves devitalizes pests, while leaving the substrate unaltered and the microstructure unaffected by any change.

Riassunto

Il lavoro di sperimentazione illustrato è atto a valutare la possibilità di applicare il riscaldamento a microonde, nel progetto di restauro. La tecnica consiste nell'applicazione delle microonde di superfici lapidee interessate da biodeteriogeni. Tale metodo, ha mostrato come vi sia una completa devitalizzazione dell'infestante. I materiali scelti per l'indagine sono campioni di tufo giallo e tufo grigio prelevati nell'area geografica dell'agro nocerino-sarnese e campioni di malta di calce e cotto prelevati da un rudere ubicato a S. Eustachio (AV). La metodologia consta di due fasi: nella prima i campioni di materiale vengono posti nella cavità riverberante e trattati variando i parametri potenza e tempo di esposizione; nella seconda gli stessi vengono sottoposti ad analisi

di natura chimico-fisica, volte a stabilire se il trattamento avesse indotto alterazioni estetiche e strutturali. Tali processi sono stati eseguiti sia su campioni integri che su campioni ridotti in polvere per un'analisi più capillare. Nello specifico i campioni selezionati e trattati, sono stati sottoposti ad analisi colorimetrica, termogravimetrica, analisi di diffrazione di raggi X, spettroscopia infrarossa, SEM, e l'analisi delle proprietà di trasporto e della resistenza a compressione.

Dalle analisi chimico-strutturali relative ai campioni trattati con il protocollo non emergono differenze del materiale trattato rispetto al materiale tal quale, l'unica differenza sostanziale tra le due tipologie di materiale adoperato è relativa alla capacità di assorbimento d'acqua dopo il trattamento. Se ne deduce che il riscaldamento mediante esposizione a microonde di materiali lapidei devitalizza l'infestante, lasciando inalterato il substrato e non apportando modifiche della microstruttura.