

LOCALIZED MICROWAVE HEATING FOR CONTROLLING BIODETERIOGENS ON CULTURAL HERITAGE ASSETS

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Abstract

Microwave heating to control biotic agents has already been applied in several fields, in particular in the agri-food and manufacturing industries. We applied localized microwave heating at 2.45 GHz to treat biotic agents infesting wooden artifacts and stone artifacts of interest for the cultural heritage. Compared to conventional techniques and other physical methods microwave heating is safe and pollution-free. In fact, compared to biocides and mechanical removal it has a low-interaction with the material thanks to its selective action. In addition, treatment extension, color-independence, penetration depth are enhanced with respect to treatments via thermal radiation, UV, gamma rays and laser cleaning. Thus localized microwave heating treatments can be an effective alternative for controlling the development of biodeteriogens. Using microwave heating to kill micro-organisms and to prevent microbial deterioration avoids the use of the chemical formulations with biocidal action that are usually applied (before and after cleaning). The use of chemical products has recently been reviewed the European Union's Biocidal Products in order to limit the risks to the substrate and the operator, to decrease environmental pollution and to prevent the possible selection of microorganisms that are resistant to the most common biocides. We present various applications of localized microwave heating to combat biotic agent growth within wooden artifacts and on the surfaces of stone artifacts. The effectiveness of the method was studied in relation to the characteristics of the microwave system (i.e. the operative conditions, frequency, power, time and temperature of exposure), the type of biological agents infesting the support/material, and the type of support/material itself.

Keywords: Microwave heating; Localized treatment; Green conservation; Biological patina; Stone; Wooden artworks

Introduction

The controlling the biodeterioration on cultural heritage artifacts includes combatting the decay due to micro-organisms and organisms and, whenever possible, delaying re-colonization [1]. The application of biocide products, alone or in combination with protective agents or consolidants, is common practice to prevent further colonization on the surfaces of works of art. Several studies have been carried out on the use of biocides and their combination with consolidating and protective products for controlling an infestation on wood and wooden based materials [3]. Stone artefacts have also been investigated, which involves products that

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dangerous for the environment [5]. Mixtures of consolidants or water-repellent products with biocides are effective in preventing biological growth on both substrates with low bioreceptivity such as plaster and substrates with high bioreceptivity such as stone [7].

The microwave (MW) heating method is one of the physical methods recently proposed to deactivate the biological growth on stone [8], together with UV, gamma rays, laser cleaning, freeze-drying, strong solar irradiation, and jets of hot water [9]. These physical methods are designed to replace traditional methods based on chemical formulates and mechanical removal methods [14]. Microwave heating has been successfully used on wooden cultural heritage objects for disinfection from woodworm (*Hylotrupes bajulus* L. [15], *Oligomerus pitilinoidea* Wollaston [16], and *Drosophila melanogaster* [17]).

Microwave radiations, as well as the electromagnetic radiation, are able to heat only certain materials containing polar molecules with an electrical dipole moment (called “dipolar materials”) through a mechanism that is quite different from the conventional transfer mechanism, which is why the microwave facilitates selective heating. Microwave heating is due to the molecular rotation in dipolar materials, where polar molecules continuously align themselves with the oscillating electromagnetic field. Rotating molecules push, pull, and collide with other molecules, distributing the energy to adjacent molecules and atoms in the material. Once distributed, this energy appears as heat.

In the case of conventional heating methods (conduction and radiation), the material is heated through its surface and the surface temperature has to be increased far above the target temperature in order to obtain the lethal dose within the material for the infesting organisms in a quite fast and controlled way. On the other hand, with microwaves the material absorbs the electromagnetic energy, i.e. it warms itself, according to its dielectric properties. For example, a wet material hosted inside a material without dielectric losses (i.e. dried material) is heated but around the material is heated only by conduction mechanism. The selectivity of microwave radiation is well known to those using a microwave oven for food: the plate or container in which the food is placed is heated only by conduction.

Thus microwave heating is successfully used as a “disinfestation system” in several sectors. By 'disinfestation' we mean the deactivation, or a reduction of biodeteriogens attacks.

Over the past few years, the following applications have been carried out:

- works of art in wood (treatment of woodworm - larvae, pupa and eggs) [15]-[19].
- food (disinfestations of grain and other seeds from insects and parasites) [20]-[23].
- soil (sterilization and disinfestation of soil) [24].

The effectiveness of the treatment depends on accurate knowledge of the electromagnetic characteristics of the material to be treated. Successful treatment entails a preliminary study of the thermal characteristics of the material and the pathogen agents, and the consequent definition of the protocol treatment. It is fundamental to know the lethal dose for the infesting organisms (temperature and the duration of treatment, as well as the power of the system).

In this paper we describe a microwave heating system used to disinfest wooden materials from woodworm and to limit biological growth on stone surfaces. Design aspects, as well as the geometry of the applicator related to the target, are described by analyzing the electromagnetic distribution in the materials under treatment, which depends on the materials' dielectric properties. The two applications require different applicators. Firstly, disinfestation from woodworm, requires an applicator that deeply heats the material (up to 20 cm). Secondly, when treating biodeteriogens on the surfaces of stone artefacts (algae, fungi, lichens, cyanobacteria)

the microwave energy must be concentrated in the initial millimeters. Our approach was assessed by laboratory tests and on site activities [25].

Materials and Methods

The use of appropriate biocides in Europe is limited by the European Union's Biocidal Products Directive (BPD) [26], thus physical methods can be an alternative (either alone or in conjunction) to chemical treatments in order to reduce the health risk to humans, animals and the environment in general.

Localized microwave heating treatments can be an effective alternative for controlling the development of biodeteriogens. Two types of the microwave heating method are described, the first for deep infestations, such as woodworm, and the second refers to superficial colonization such as biological patina (lichens, algae, biofilms).

The design criteria for the applicator and the treatment protocol must be suitable for the specific biological agent and the support. On hygroscopic materials such as wood, it is essential that the electromagnetic energy is directed exclusively onto the target, thus preventing any unnecessary heating and consequent drying of the medium [27].

Localized MW for deep treatment

In a previous work R. Olmi et al. [28][15] demonstrated that it is possible to disinfest wooden materials from woodworm by MW heating. In a typical treatment, the infested object is exposed to microwave radiation to increase the woodworm temperature over 53-54°C for 1 minute (which we have proved is lethal for such insects [15]), while maintaining the temperature of the wood at safe levels, usually no higher than 50°C. In order for the disinfestation to be effective, it needs to obtain a quite uniform of the distribution of electromagnetic energy density inside a defined "volume" of wood. If within the volume xilophageous insects are present, they will be exposed to the lethal dose. The treated volume should be as deep as possible, for example up to about 10 cm in order to treat a timber structure of 20 cm in diameter.

An efficient control of the heating conditions is ensured by knowing the electromagnetic characteristics of the material (permittivity and conductivity). Thus, we measured the electrical properties of several types of wood in the 2÷3GHz frequency range and at different moisture contents.

The dependence of woodworm mortality on temperature (lethal dose) was investigated by heating woodworms (in particular, *Oligomerus ptilinoides* Wollaston and *Hylotrupes bajulus* L.) in a thermostatic bath for a sufficient time to maintain them at a prescribed temperature for at least one minute. The mortality curve of *Oligomerus ptilinoides* (Fig. 1, the regression logistic is in solid lines, and the experimental data are filled rectangles) is quite steep, and shows that all the woodworm died above 53.5°C. The similarity between this curve and the curves relative to *Hylotrupes bajulus* L. (Fig. 1, dashed line, and dash and pointed line) suggests a temperature response that is practically identical for both families of insects.

The equipment for the microwave heating treatment consisted of a 2.45GHz microwave generator (magnetron with adjustable power from 0 up to 250 watt), a radiative applicator and various ancillary tools to control the power emission with respect to the target temperature. The applicator was made up of a section of a rectangular waveguide suitable for operating at 2.45GHz (cross section: 7.7 x 3.7cm²) in order to satisfy the requirements of deep treatment [18]. A section of waveguide is the simplest applicator for deep heating. Other applicators, e.g. horn antennas, do not conceptually differ from an open waveguide.

A full-wave analysis of the open waveguide radiator was accomplished using a commercial finite element method (FEM) multi-physics tool [27], which also solves the associated heat conduction problem. Figure 2a shows the electric field pattern produced by the open waveguide inside a wood material in equilibrium with the environment (temperature =

20°C, moisture content = 9.3% [25]). The permittivity (averaged on the three main axis [28]) is about 2.9-0.4i, for a wood species with a density of 0.6g/cm³. Figure 2b shows the resulting temperature after 55 seconds, when the delivered power is 100W. Both pictures refer to a plane cutting the applicator aperture in the middle, parallel to the wide sides of the waveguide. The temperature distribution is almost axially-symmetric, with the same temperature distribution on the perpendicular plane (Fig. 3). The color-scale for both plots and both figures (Figs. 2 and 3) is arbitrary, increasing from blue to red.

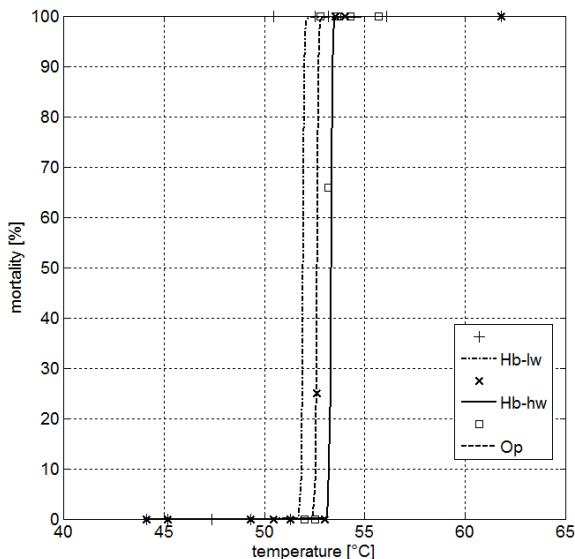


Fig. 1. Percentage of mortality vs. temperature of *Oligomerus pilinoides* (Op) and *Hylotrupes bajulus* (Hb-lw: weight < 0.1 g; Hb-hw, weight > 0.1g)

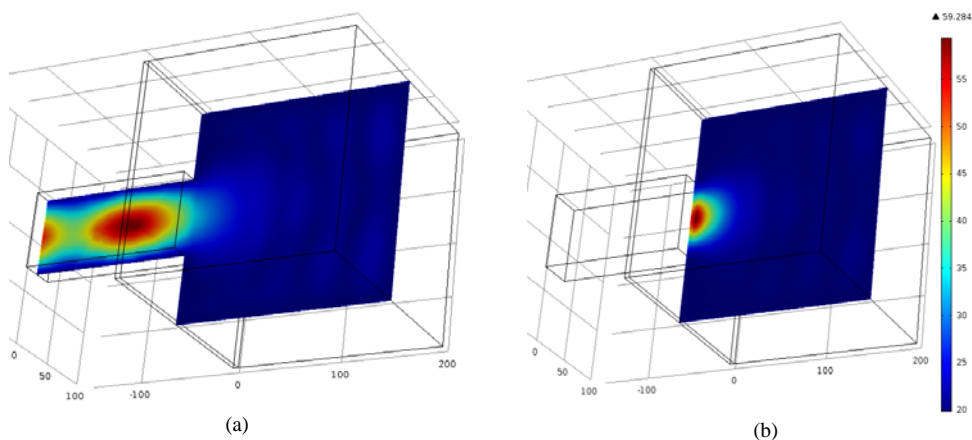


Fig. 2. (a) Electric field distribution within the waveguide and inside the material by a radiative applicator. (b) Temperature distribution in wood after 55 seconds of continuous heating.

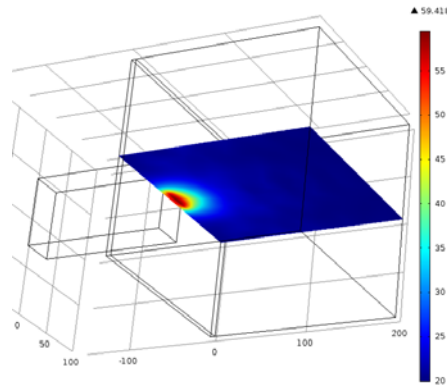
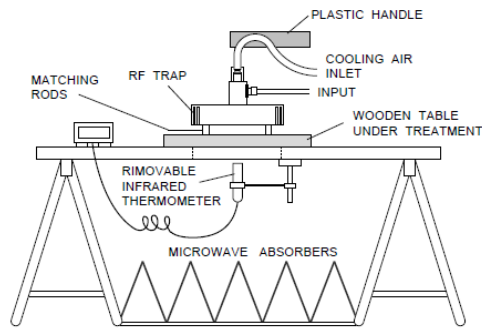
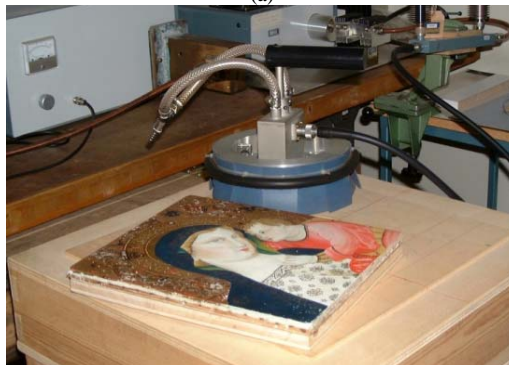


Fig. 3. Temperature distribution in wood after 55 seconds, on a plane parallel to the narrow sides of the applicator.

The treatment depth can be increased by cooling the surface. The applicator was equipped with a system for cooling the treated surface by blowing cooled air. The distance between the applicator and the wood surface is adjustable in order to optimize the power delivery. Figure 4 shows the configuration for treating a painted board. The painted layer is facing downward, with the RF power impinging on the opposite side. The temperature of this surface is controlled in real time by a non-contact infrared thermometer through a window opened in the treatment desk. Figure 4b shows the prototype of the radiative applicator to treat painted boards, wood beams and other wooden materials.



(a)



(b)

Fig. 4. (a) Laboratory set-up for microwave treating of painted boards; (b) Prototype of the microwave applicator for localized heating of painted boards.

Localized MW for biodeteriorated stone surfaces

A different approach is needed for treating infestations on surfaces, such as biological patina on stone or plaster. In these cases the heating should be limited to the initial layers where the biodeteriogens are deeply-rooted (in general up to several millimeters) [29]. In addition, the treating temperatures for lichens and fungi must be substantially higher (up to 65-70°C) – see also reference [28].

The treatment of biodeteriogens on the surface of stone artefacts, as well as lichens, algae, cyanobacteria and fungi, does not require deep heating. The treated volume should be limited to less than 1 cm in depth.

This all thus suggests rather different design criteria from those employed for developing microwave applicators suitable for volume heating. The best choice is an applicator where the EM field is bound to the emitter, i.e. a reactive applicator. A reactive applicator operates in the evanescent field, rather than in radiating mode, and the penetration depth can be easily controlled by varying the geometrical size.

The resonator can be schematized with a rectangular waveguide section, short-circuited at one side, and terminating on the slot (aperture) on the other side (Fig. 5).

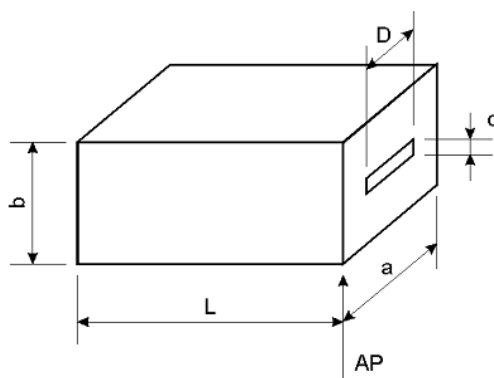


Fig. 5. The resonant applicator. AP is the plane where the slot is open-ended.

The microwave power is supplied to the applicator at the side of the short circuit by a cable-to-waveguide transition. The geometry of the slot and size of the resonant cavity with respect to the electromagnetic field distribution inside the material were calculated by numerical simulation software [27]. Figure 6 shows the electric field distribution inside the applicator and the configuration of the field defined by the standing wave, when the applicator is in contact with the material under treatment.

Figure 7 shows the image of the temperature distribution in a moistened brick (about 10% on d.b.) after 300 seconds of heating with 100 W, on the transversal section at a depth of 3 mm. The time behaviour of temperature with depth is shown in Fig. 8, from 0 to 300 seconds (in 50 second steps). The abscissa axis shows the depth in mm.

FEM simulations enable the influence to be evaluated of the geometry of the applicator on the thermal performances. Figure 9 shows the behaviour of a reactive applicator with three slot apertures that “shape” the thermal field. The temperature distribution on the two principal planes is shown as, parallel (a) and normal (b) respectively, to the wider sides of the slots.

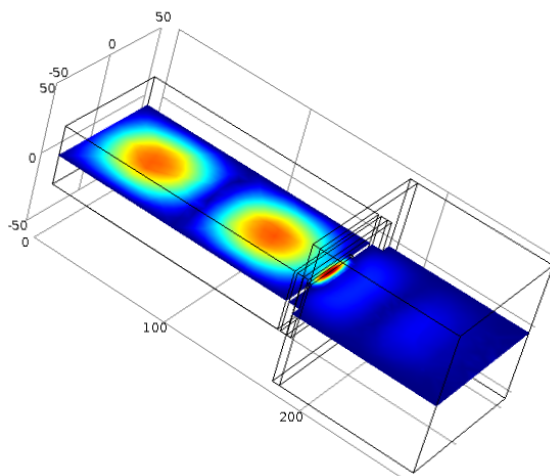


Fig. 6. The electric field distribution inside the resonant cavity.

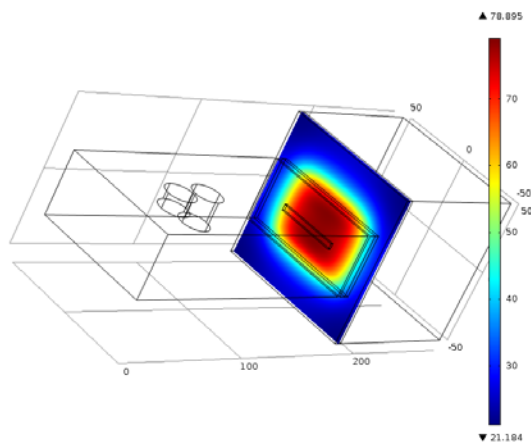


Fig. 7. Temperature distribution in moistened brick (about 10% on d.b.)

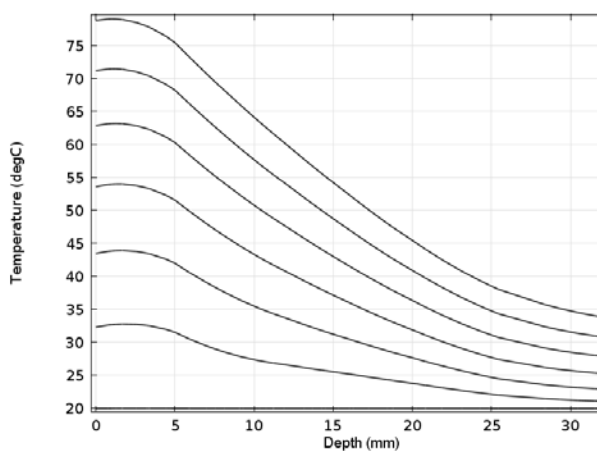


Fig. 8. Time behaviour of temperature with depth

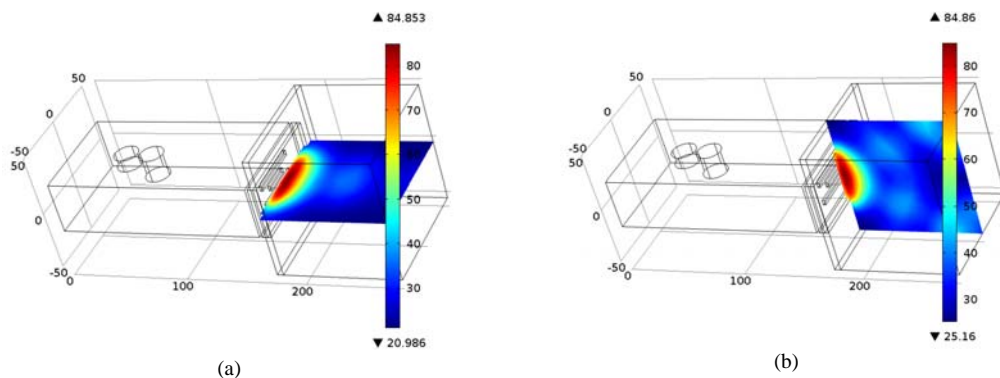


Fig. 9. Temperature distribution produced by a 3-slot applicator on a moistened brick on the parallel plane (a) and normal plane (b) to the wider sides of the slots

Results and Discussion

The criteria followed in the design of the applicator (reactive or radiative) must be suitable for the particular biodeteriogen agent and to safely treat the support. The EM power should be delivered “only” to the target, thus preventing any unnecessary heating. The intrinsic selectivity of the microwave radiation with respect to the wet materials satisfy this target. The properly design of the applicator allowed to perform heating on the delivered areas, in depth for the disinfestation of wood and wooden materials from woodworms and only in the initial layer of the support for the biocolonization on stone surfaces. In order to disinfect wooden materials from xylophagous insects (woodworms), it was necessary to obtain an uniform distribution of the lectromagnetic energy density in a volume in depth in wood. If xilophageous insects are present within this volume, they will be exposed to the microwave radiation. The lethal dose was determined in 1 minute at temperatures in the range of 50-53°C. The treatment should be as deep as possible, for example up to about 10 cm in order to a treat timber structure of 20 cm in diameter. A different approach is needed for the treatment of a biological patina on stone or plaster. The heating should be limited to the first layers where the biodeteriogen organisms are deeply-rooted [19]. In addition, the treatment temperatures for the fungal part in lichens must be considerably higher (up to 65-70°C) – see reference [20].

The above two cases: (a) deep heating of wooden works of art and, (b) superficial heating of stones, will be considered separately in the following. The microwave heating application is an effective biocide treatment with low interaction with the substrate. In fact:

- the electromagnetic energy is preferentially deposited in materials where the dielectric losses are high, i.e. in the presence of water as well as inside insects or lichens,
- after the analysis of the one-dimensional heat conduction of a plane material (see previous section).

Treatment of wooden boards

Tests on boards artificially infested by woodworms were carried out. The woodworms (*Oligomerus pilinoides*, at larvae and pupae stadium, and eggs of *Hylotrupes bajulus*) were inserted in small horizontal cavities carved in three-centimeter-thick boards, placed at depths ranging between one and two centimeters, and positioned parallel or normal to the wood grain, at varying distances (1-3cm) from the axis of the applicator aperture. The wooden boards were exposed to microwave radiation (200W delivered power), using different heating cycles and maintaining the surface temperature in the 40-50°C range.

A total of seven assays were carried out, regarding all three metamorphic stages of interest: egg, larva and pupa. Larva weights ranged between 1 and 8 mg. The conditions of the larvae were observed five minutes after the end of the microwave treatment, and were checked one day later. Pupae and eggs were followed up for ten days. All the specimens were destroyed independently of their position in the wood: larvae died, eggs did not hatch, and pupae did not develop into insects.

The effects of the microwave treatment on the painting were also investigated. Three square boards divided into four sections and painted according to ancient recipes were treated by positioning the applicator on three sections. One of the four sections of each board was not treated in order to take into account possible confounding effects (colour deterioration not due to temperature, measurement errors). A spectroscopic analysis in the visible region of the surface was carried out before and after treatment, using a portable spectrophotometer (MINOLTA, mod. ChromaMeter CM-700d). The difference between the measurement before and after the treatment was less than the detection threshold of the human eye, thus indicating the absence of damage due to the microwave treatment.

Treatment of biodeteriogens on stone surfaces

In order to plan the microwave treatment of a biological organism, the two main parameters need to be known:

- (1) the threshold temperature above which the organism dies;
- (2) the time duration needed to obtain the desired effect.

In other words, it is necessary to assess the percentage of mortality for different doses (temperature/duration).

This was achieved by heating the organisms of interest (lichens and fungi) in a thermal bath at different temperatures and time durations, followed by an analysis of the health status of the biodeteriogens. The first kind of analysis consisted in examining self and induced fluorescence of the thalli lichen had been sectioned manually or using a microtome. The fluorescence of the lichen sections were examined using a Nikon Eclipse 600 microscope equipped with different filter cubes: UV, IR, FITC (Fluorescein isothio-cyanate) and TRITC (tetramethylrhodamine isothiocyanate). Self-fluorescence of the chlorophyll and induced fluorescence of the dead cell staining were recorded using UV-2A and TRITC filters, while the induced fluorescence of living cells was recorded with an FITC filter. A Plant Cell Viability Kit, Sigma Co. was used according to manufacturer's protocols. The results are available in R. Olmi et al [29].

Another analysis was then conducted using a PAM-2100 Portable Chlorophyll Fluorometer to analyze the effectiveness of the photosynthetic system of the organism. The principle underlying chlorophyll fluorescence analysis is relatively straightforward. The light energy absorbed by chlorophyll molecules in a leaf undergoes one of three events:

- it can be used to drive photosynthesis (photo-chemistry)
- excess energy can be dissipated as heat
- it can be re-emitted as light-chlorophyll fluorescence

These three processes occur in competition, so that any increase in the efficiency of one will result in a decrease in the yield of the other two. The dark-adapted value of F_v/F_m [32] is widely used as a sensitive indicator of plant photosynthetic performance.

The vitality of biodeteriogens agents present a threshold effect with the dose (temperature/duration), at 50°C for 3 minutes the algal part of the lichen died in all the cases, while for the fungi part, the temperature must reach 65-70°C, depending on the specimen [8].

Tests were conducted on stone material infested with lichens of different species. Figure 10 shows a stone infested with a foliose and a crustose lichen, *Xanthoria* sp. and *Lecanora* sp. By controlling the power of the microwave generator (0-1kW, at the frequency of 2.45GHz), the desired temperature is maintained approximately constant at the fixed value.

The treatment procedure is as follows:

- The surface under treatment is wetted with a brush before microwave irradiation, to maximize the power absorption in the superficial layer.
- The temperature is taken continuously throughout the treatment (using a thermometer with a fiberoptic probe).
- Microwave power transmitted from the electromagnetic generator is continuously checked and regulated by a retroaction loop by the supplied temperature at the previous stage

Microwave heating treatment was effective in the impairment of these kinds of crustose lichens.



Fig. 10. (a) *Xanthoria* sp. (b) *Lecanora* sp..

Microwave leakage and safety

The MW power radiated into the environment was measured by an electric field monitor measurement system on both applicators during a disinfestation treatment. The power density was below safety levels, in both cases, in all those areas that could be accessed by the operator. This power density would be considered safe according to the most accepted standards (Directive 2004/40/EC of the European Parliament and of the Council [29/04/2004] on the minimum health and safety requirements regarding the exposure of workers to the risks arising from physical agents).

A power density of about 1-2 mW/cm² was measured close to the radiative applicator handle for wooden disinfestation. These low stray fields were due to the RF trap and to the microwave absorbers (as described in a previous section), without which there would be significantly higher power densities. Radiated power density (S) was also measured along the axis of the applicator in operative conditions, i.e. in the previously-described set-up (Fig. 4a). With an input power of 200W, a power density higher than 5 mW/cm² was present at distances of up to 115 cm when a 3 cm thick board was treated. Note that attention is needed when treating vertical objects, in order to avoid dangerous exposure.

Electric field values less than the reference standard were measured close to the reactive applicator for biodeteriorated stone surfaces, varying the power of the MW generator from 10 W to 1.0 kW (see Fig. 11). Figure 11a shows the results of the measurements performed at a distance of 50 cm from the aperture, i.e. close to handle of the applicator. Figure 11b shows the measurements performed at a distance of 1m from the applicator aperture.

Both applicators were equipped with a switch that turned the power OFF when the applicator was lifted from the surface of the object under treatment, to prevent the risk of exposing the operator to irradiation .

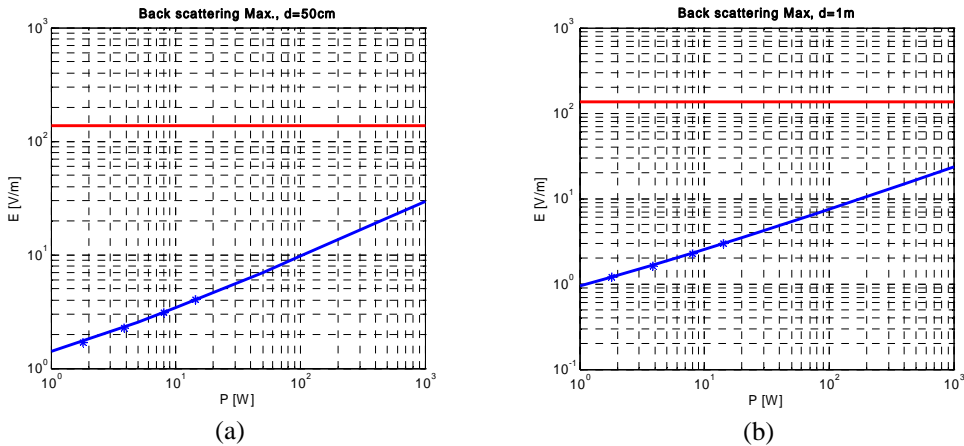


Fig. 11. Electric field values measured on the back of the applicator aperture (handle side) are plotted in a solid line blue, while the reference standard value is plotted in a red solid line. (a) Measurement performed at a distance of 50 cm from the aperture. (b) Measurement performed at a distance of 1 cm from the aperture

Conclusions

A portable microwave disinfestation device was designed and built. The system was tested to disinfest wood from insects and to deactivate biological patina on stone using an applicator.

The development of an efficient disinfestation system using microwave heating requires a careful design of the applicator in terms of transferring power from the source to the material. The choice of applicator depends on the specific biodeteriogen agent, thus information on the morphological and physiological characteristics (in particular the required time/temperature dose for their devitalization or impairment) is essential.

In addition, the characteristics of the substrata should be considered, for example, marble presents an anisotropic thermal dilatation coefficient which means that the maximum temperature of the treatment should be limited to 65-70°C. Both when volume and superficial heating are needed, the design of the applicator can greatly benefit from the availability of multi-physics simulation codes which enable the geometrical characteristics of an applicator to be related to its performance in terms of electromagnetic field and temperature distribution in the target material.

The microwave test treatments were effective against all metamorphic stages of woodworm, while keeping the wood temperature below 50 °C which ensured that there was no damage to the wood or to the surface of the painting.

The device proved to be safe according to the most accepted safety recommendations and standards concerning radiated electromagnetic fields in the environment. For the disinfection of biological agents and progressive microbiological damage, the microwave heating is also comply with the restriction introduced by the European Union’s Biocidal Products Directive (BPD) [25]. We are now focused on the cumbersome study to discover eventual limitations related to the quick heating of the support. The quick heating in wood could induce several localized stress due to the temporary dehydration of the medium. Will be necessary monitoring the effects of the stress by laboratory tests performed on several specimen of wood. On stone the rapid heating could induce similar stress than in wood, if the moisture content is not negligible. In particular on marble, where the thermal dilatation coefficient is not isotropic, the rapid heating of the substrata could be accelerate the granular disintegration phenomenon. Also in this case several tests are in progress in order to verify the appearance of

the previous phenomenon after several cycles of microwave heating from environmental temperature up to 65 °C. In addition, microwave heating should be used in conjunction with chemical treatments in order to reduce the drawbacks related to use of biocide for the health risk to humans, animals and the environment. This approach can be an effective alternative for controlling the development of biodeteriogens on stone in a localized area where the biological organisms are deeply-rooted in depth and the biocide action is not efficient.

Acknowledgments

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