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Microblasting with vegetable and cellulosic media for heritage wood cleaning: effects on surface morphology

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Abstract

In this research, microblasting with powdered vegetable particles (almond and hazelnut husks, cork saw dust and rice husks) and cellulosic fibres was used to analyse their usefulness for cleaning unpainted wooden cultural heritage and their effects on surface morphology. Tests were made on mock-ups of old pine wood from wooden boards with original soiling. Before cleaning powdered particles were characterised by SEM–EDS to analyse their morphology and elemental composition. Wood surfaces were analysed before and after cleaning by means of digital microscope (white and UV light) and SEM–EDS to evaluate cleaning effectiveness, assess effects on the wood surface, and determine eventual soiling and powdered particles larger than 1 µm that might remain in the surface after the procedure. Results indicate that microblasting with powdered vegetable and cellulosic media is a useful and safe technique to remove soiling from wood, including micrometric particles embedded in its texture, with negligible surface changes. Furthermore, residues left after the cleaning procedure are very scarce and chemically compatible with the substrate because they are mainly composed of cellulose, hemicellulose and lignin, the main components of wood.

Keywords Dry-cleaning methods, Powdered vegetable microblasting, Conservation, Surface evaluation, Sustainable materials

Introduction

Cleaning is a risky procedure when conducted over cultural heritage surfaces. Its main goal is to remove soiling deposits that are, or might be, both harmful for the conservation of cultural heritage assets or affect its aesthetic value. However, cleaning is an irreversible process in which the removal of dirt must be performed carefully because the substrate can be irreparably damaged and relevant documentary information on the object may be lost.

Based on this essential premise different techniques have been used for cleaning heritage materials; techniques that have evolved from further scientific and practical contributions [1–4]. Despite these developments, the most common available techniques used by restorers for cleaning cultural heritage are based on mechanical and chemical methods that can be carried out either independently or in combination; nevertheless, all of them have advantages and drawbacks. As reported in classic publications about cleaning [5], its effectiveness is related to material properties (such as heterogeneity, texture, cohesion and hardness, among others), soiling properties (particularly thickness and adhesion), tools or products characteristics and how they are applied. For this reason, the preliminary study of material and soiling

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as well as of the cleaning technique and their parameters are essential to perform a sensitive cleaning.

Mechanical cleaning of unpolychromed wooden supports, such as the back of altarpieces, sculptures, panel paintings or ethnological objects, is usually referred to in conferences, workshops or general publications [6–8]. Nevertheless, specific references on instruments used are generally referred to (if mentioned) in internal or unpublished reports documenting all the procedures involved for a specific intervention. In this regard, for natural soiling deposits, the method believed to be the mildest is vacuuming and, occasionally, low pressurised air. Both are often accompanied with a soft paintbrush, either of natural or synthetic bristles [9–11] to improve the results. An alternative method is the use of rubbers, sponges or pads of different toughness, from soft to extra hard [12, 13]. However, these methods have low or no effect on deep-seated natural soiling. In this situation, where hard, compacted, bonded deposits must be removed, tougher brushes, including brass or steel wire brush and steel wool, are used, although surface change is easily provoked [14, 15]. When the thickness of dirt accumulated on the wood is greater (such as accumulations of bird or bat droppings, or debris from repairs in the surroundings) the use of scrapers, trowels, blades or scalpels is mentioned [16]. In some cases, mechanical cleaning is reported to be followed by chemical cleaning using a 50:50 or 75:25 water–ethanol mixtures to remove any dirt that might have adhered to the surface [17]. And although the use of other organic solvents, surfactants or reagents have been also described to clean non-polychromed wood accompanied or not by mechanical instruments, this research focus on the physical processes of dry cleaning on wood.

All of these manual and mechanical cleaning systems are based on the use of energy to break the bonding between soiling and substrate mainly by friction or by cutting actions [1]. The former, carried out by sliding the tools on the surface, is usually considered the smoothest mechanism allowing greater control during treatment, but the latter is sometimes provoked depending on the instrument used [18].

No documented references about parameters of manual cleaning techniques are referred to in scientific literature. Probably because of the difficulty of measuring them, parameters are not usually included in studies or research, although there are some recommendations about controlling them to remove deposits properly during cleaning [1].

Manual cleaning parameters can be determined from traditional use (energy or pressure, angle, frequency of movement, vibration, rotation, etc.), and from the tool itself (size, shape, composition, etc.) according to the work formula [$W = F \times d = F \times d \times \cos\alpha$] as a physical magnitude in classical mechanics, which posits a correspondence between energy or exerted force [F], the displacement of surface deposits in this case [d], and the angle formed by the force and the displacement vectors [$\cos\alpha$] [18].

Besides these manual methods, another mechanical technique is used for heritage cleaning: the blasting or microblasting technique, depending on the equipment involved. The cleaning procedure is, in this case, carried out by particles (or abrasives) blasted to the support in a flow of compressed air to remove natural or artificial dirt deposits [19, 20].

Blasting and microblasting are based on the same processes that those involved in manual mechanical cleaning but the tools, in this case, are the abrasives. The technique is influenced by pressure, distance, angle, time, nozzle diameter, flow of particles, and specific abrasive properties (composition, size, specific weight, density, morphology, hardness, friability or toughness, etc.). It is based on the kinetic energy formula [$KE = ½m \times v^2$], where m is mass (related to abrasive properties); and v, velocity (related to pressure) [20].

As well as in manual cleaning, to break the bonding between soiling and substrate low impact, cutting or friction mechanisms are determining factors in blasting and microblasting. Friction by sliding the abrasive in different angles is usually considered the smoothest mechanism, allowing greater control during treatment. Impact or cutting is sometimes provoked depending on the abrasive used and the parameters of the technique itself, but sometimes they can be useful to clean [18, 20]. The control of the whole cleaning process through the proper selection of the abrasives and the parameters to be used is very important to avoid unwanted surface change on the surface.

Blasting and microblasting has mainly been used to remove different surface deposits in architectural heritage buildings materials or even for stripping large areas of wooden supports, including wood in heritage buildings as mentioned in classic publications on this subject [21, 22]. For cleaning building materials, natural and synthetic mineral abrasives (such as silica, calcium carbonate, aluminium silicate, aluminium oxide, glass beads, etc.) are used because they have similar properties to stones and residues that might remain after treatment would be compatible with the substrate [23, 24].

By following this approach (that is, similar properties and possible presence of residues compatible with the cleaned material) some researchers have replaced natural and synthetic mineral abrasives by other particles that can be blasted for cleaning. In these studies, powdered cellulose particles have been used for mechanical cleaning of paper documents [25, 26], the reverse side of painting canvases [27] and oil paintings [28]. Results indicate that the procedure is a suitable alternative to traditional manual cleaning on these supports because the achieved cleaning degree is higher than by using other classical mechanical cleaning techniques, no damage is provoked to the material surface and eventual residues are chemically compatible with the substrate.

In addition to those natural and synthetic mineral abrasives or cellulosic particles, the use of vegetable particles for cleaning archaeological or sculptural metals, mainly bronze and iron, [29–31] or glazed stoneware [32] have also been reported in the literature. In these works, powdered vegetable particles have been used not for their similar properties and residue compatibility with the substrate, but rather because their low hardness allows the removal of soiling without affecting the protective patina formed on those metals.

Based on all the above premises, the aim of this study is to apply, analyse, compare and evaluate the use of the microblasting technique on wood heritage using four different vegetable and cellulosic particles as an alternative to the traditional mechanical methods, taken into account that these particles have similar properties to wood and, therefore, any residues that might remain after cleaning on the surface would not have a negative effect on its preservation. Cleaning results with vegetable and cellulosic particles were compared with those obtained by cleaning with microsilica.

Materials and methods

The mock-ups selected for the study are made of pine wood and have dimensions of 2.5 × 5.5 × 1.7 cm approx. They come from old wooden boards from a rural environment with original and naturally acquired soiling.

For microblasting, three different vegetable particles from residues or sub-products of the food industry were selected from previous tests.

Aval® is a natural product obtained from the crushing of almond (*Prunus dulcis*) and hazelnut (*Corylus avellana L.*) shell, with slightly angular and polyhedral particles. It is used to reinforce bioplastics and in industrial blasting and microblasting for smooth cleaning processes, among other.

Cork is the bark of *Quercus suber L.*, the cork oak tree. The cork bark is used for the elaboration of corks for wine and sparkling wine bottles. Cork saw dust, a natural raw material by-product generated by the saws that cut the corks from the cork planks has been used, has a particle morphology fairly rounded. Cork powder is a by-product scarcely commercial for other purposes.

Oryzite® is a processed natural product made from the rice husk, the outer layer that covers the rice grain. It has slightly sub-rounded and polyhedral particles and it is used as a bio-filler for polymers in different industrial sectors.

Arbocel® was also selected as a material whose softness has been previously tested on paper and fabric supports in order to compare the results with those of vegetable particles. It is processed pure cellulose fibres, with elongated and cylindrical morphology, used as thickeners, absorbents, diluents or fillers in industrial and pharmaceutical manufacture amongst other multiple purposes. In our case, we choose the BWW40 because its fibres have a size similar to the vegetable particles.

For further comparison, some tests were carried out with silica sand (hard inorganic material), as one of the most common particles when blasting is used for woodworks cleaning in buildings, but because of the technique used, micrometric-sized.

According to manufacturer's data sheets, the main properties of these particles related to their use for microblasting are indicated in Table 1, except for cork, the information of which is taken from bibliography [33].

Cleaning tests

Cleaning tests were carried out at the Conservation-Restoration laboratories at the Faculty of Fine Arts of the University of Barcelona which have climate control (20–22 °C temperature and 50–60% humidity).

The equipment used was a foot-switch operated microblasting CTS5/B, with a straight tungsten carbide nozzle of 0.7 mm diameter. Additional equipment used include a silenced compressor of 1.5CV and a dehumidifier filter (to reduce the humidity of compressed air and the clumping of the abrasives). The treatment was made in a sandblasting cabinet equipped with an external vacuum cleaner.

Samples of vegetable particles were provided by the manufacturers in different sizes. A size fraction of ≤ 200 μm was considered for the experimental phase due to nozzle diameter. To avoid nozzle clogging when the particles did not have a suited size, the largest products (Oryzite® and Cork) were sieved with 0.125 mm standardised sieve size (ISO 3310/1), and also silica sand to have a micrometric size.

Microblasting procedure and parameters in this research were selected according to previous data in literature for other materials [34] because there are no standards for wood microblasting as well as for heritage cleaning in general. This protocol was previously tested in some mock-ups to determine if any adjustments were necessary to correctly clean the wood and it was observed that the results of previous studies in other materials were also applicable in this case.

Table 1 Properties of particles related to microblasting *(in italics data measured by the authors*)

	Arbocel®		**Aval®**		**Cork**		**Oryzite®**		**Silica sand (microsilica)**	
Material	Natural cellulose fibres (bleached cellulose, sulphite, non-coniferous)		Powdered almond and hazelnut husks		Cork saw dust		Powdered rice husks		Quartz	
Composition	~ 98.0% ~ 1.50%	Cellulose ash	35.86% 48.00% 2.76% 2.22% 1.08%	Lignin cellulose fat protein ash	50% 20–25% 20% ~ 1.5% ~ 14–18% ~ 1–2%	Suberin lignin-polysaccharides (cellulose and hemicellulose) pectinsex-tractives ash	15.00% 85.00%	Silica organic compounds (cellulose, lignin, d-xylose and traces of d-galactose)	~ 98.00% ~ 2%	Silica other silica minerals
Morphology	Elongated and cylindrical		Angular and polyhedral		Rounded		Sub-rounded and polyhedral		Sub-rounded	
Particle size (μm)	≤ 200		≤ 200		*≤ 125*		*≤ 125*		*≤ 125*	
pH	*6.8*		*5.7*		*5.5*		*6.7*		*7*	

Determination of pH of aqueous extracts (ISO 6588:1981)

Tests with each particle were made on two different pine wood mock-ups placed horizontally. Cleaning parameters were maintained constant at a 20 kPa (2,9psi) pressure and 10 cm distance from the end of the nozzle to the wood surface because larger distances are not usual or appropriate due to visual assessment cleaning loss. The angle was set at 90° because acute angles led to earlywood erosion. Nozzle pointer was fixed with a clamp to maintain distance and angle. Microblasting time was set at 5 + 5 s for Arbocel®, Aval®, Cork, Oryzite® and microsilica.

To control as far as possible the homogeneity of this treatment, testing areas were covered with cardboard until a uniform and constant blasting flow was reached. Then, the trial was initiated and timed. This procedure was necessary because when the switch is activated at first, flow is not constant depending on pressure, hose length and particle characteristics, among others. The same protective device was used to prevent the abrasive (that continues to flow after releasing the switch control) to impact the surface at the end of the treatment. During tests, an assistant was timing the treatment and protecting and uncovering the surface.

After microblasting, and before carrying out the analytical study, a portable vacuum cleaner (Museum Muntz® 555-MU-E HEPA) equipped with a flat section nozzle accompanied with a fine nylon paintbrush was used to remove any naked-eye visible remaining residues of dust and blasting particles on wood surface.

Evaluation techniques

For surface evaluation digital microscopy and scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM–EDS) were used. Digital microscopes have the advantage of being an affordable and readily available tool to conservators-restorers that can be used in the laboratory or in situ to easily verify the cleaning progress and the effects on the surface. SEM–EDS was also used to analyse elemental composition and morphology of powdered vegetable and cellulosic particles, soiling and wood surfaces.

Digital microscope (DM)

Surface evaluation was conducted by using an AM4113-FVW Dino-Lite® microscope with switchable white and UV light (395 nm). Images were taken at 65 × with direct LED white light and UV (90°) from the microscope and external grazing white light (35° and 4 cm distance) from a fiber optical illuminator (ISO 9001) regulated at position 1. All the images were processed with 2.0 Dino-Capture® software. At this magnification, texture changes can be easily appreciated, determining accurately the effects and effectiveness of the different cleaning tests.

Scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM–EDS)

A FEI ESEM QUANTA 200 attached to an EDS detector EDAX Genesis was used. Each vegetable abrasive was placed on a conductive carbon adhesive tape fixed to a SEM stub and placed unprepared into the SEM chamber. Secondary electron (SE) images, backscattered electron (BSE) images and elemental analyses were obtained under a low vacuum mode with an accelerating voltage of 20 kV. The images were taken under similar magnifications (500x, 1000x). When possible, several analyses were performed on items displaying similar features. The EDS analyses were done at the same working distance (around 10 mm) and with the same acquisition time (50 s) for each EDS spectrum. To avoid any misunderstanding with the quantifications the expression "major", "minor" and "trace" elements rather than actual numbers was used.

Powdered vegetable particles were submitted to microscopic and elemental analyses through this technique (cellulose particles were not considered for these analyses because they are already studied elsewhere [27]).

To characterise the wood surface before and after microblasting, the wood samples were also analysed by SEM–EDS. Before cleaning, each wood sample was marked twice at the surface with a stainless-steel scalpel in order to locate the area of interest once inside the electron microscope. Subsequently, the samples were introduced unprepared into the SEM–EDS and analysed following the same procedure applied to the vegetable particles. In this case, magnifications were set at 65 ×, 500 × and 1000 ×.

Results and discussion

Vegetable and cellulosic particles composition

SEM–EDS analysis highlights morphological and compositional differences between the three vegetable particles and the powdered cellulose. A summary of the results obtained by this technique is presented in Table 2.

Arbocel ®BWW 40

Selected images of Arbocel® under the SEM as well as two representative EDS spectra are displayed in Fig. 1. The sample is composed of twisted elongated fibres that are sometimes rolled up to form wool ball-like structures (Fig. 1 A, B). Both of them are composed of C and O (Fig. 1 C, D).

Aval®

Selected images of the sample under the SEM as well as representative EDS spectra are displayed in Fig. 2. It is composed of an admixture of loose, isolated, amorphous fragments probably originating from hazelnut shells as well as agglomerated highly porous polyhedral particles

Table 2 Results of SEM–EDS analyses on powdered vegetable media

Sample	Description of analysed item				Elemental composition	
	N	BSE contrast	Size (um)	Description	Major	Minor
Arbocel®	2	Dark grey	150	Wool ball-like particle	**C**	
	2	Dark grey	NA	Elongated fiber	**C**	
Aval®	6	Grey	110 × 60	Mosaic, porous particle	**C**	K, Al, Ti
	6	Grey	230 × 30	Elongated fiber	**C**, K	Al, Mg
	1	Grey	55 × 35	Low sphericity, angular particle	**C**, Ca, K	Al
	4	Light grey	NA	Wrinkled paper texture	**C**, K, Al	Ca, Si
Cork	7	Dark grey	120 × 50	Honeycomb-like particle	**C**, Ca	K
	5	White	12 × 10	Subeuhedral particle	Ca, C	K
Oryzite®	11	White	7 × 4	Subeuhedral particle	**Ca**, C, Si,	Mg, Ti, K
	3	Grey	135 × 125	Low sphericity, subangular grain	C, Si, Ca	K, Mg, Ti
	2	Dark grey	200 × 35	Elongated particle	**C**, Ca, Si, Mg	K

Elements in bold are present in a proportion equal or higher than 40%

N number of times these items were recorded. Major and minor elements constitute more than 1% and between 1.0 to 0.1% of the sample by weight respectively. Weight percentages including O and normalised to 100%. *NA* not applicable

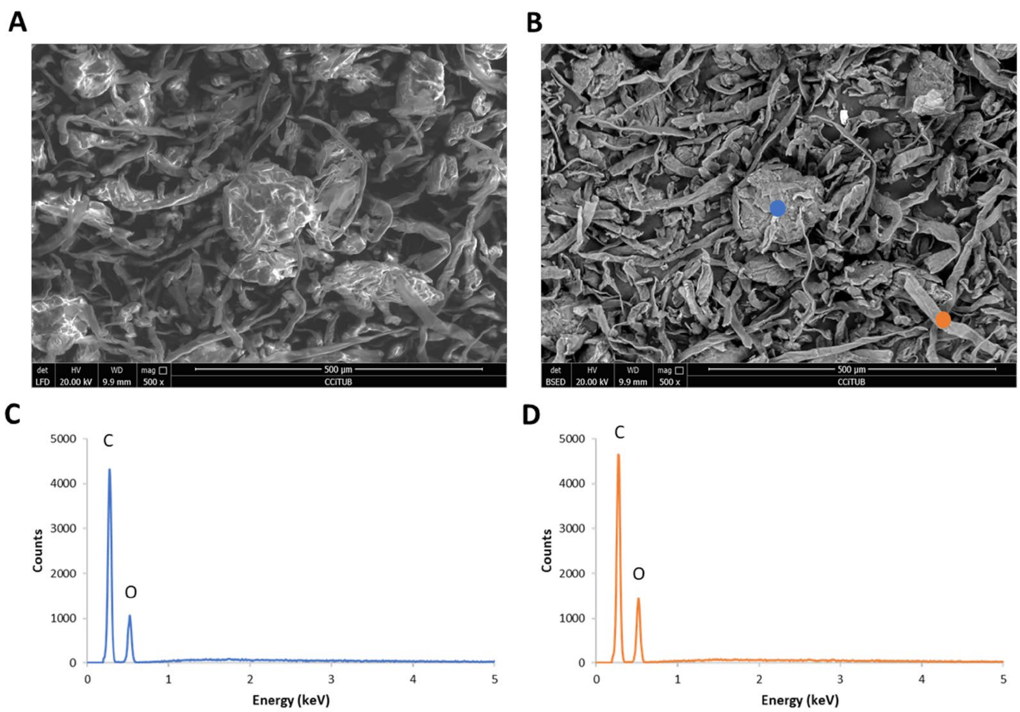

Fig. 1 Appearance of natural cellulose fibres (Arbocel®) under the SEM. **A**, **B** SE and BSE mode images at 500×. **C**, **D** selected spectra of different particles in **B**

possibly from almond shells (Fig. 2, A, C). The first ones are composed of C, K and Al (Fig. 2 D) while the second ones are mainly composed of C (Fig. 2 E).

Cork

SEM–EDS analyses of cork saw dust micro-fragments reveal its characteristic honeycomb-like structure mostly composed of C (Fig. 3 A). The BSE mode reveals, though, that the hollow cells are sometimes filled with Ca-rich inclusions (Fig. 3 B, C). As in rice husk powder, the origin of these Ca-rich subeuhedral crystals is uncertain. The chemical composition of cork has found to depend on factors such as geographic origin, climate

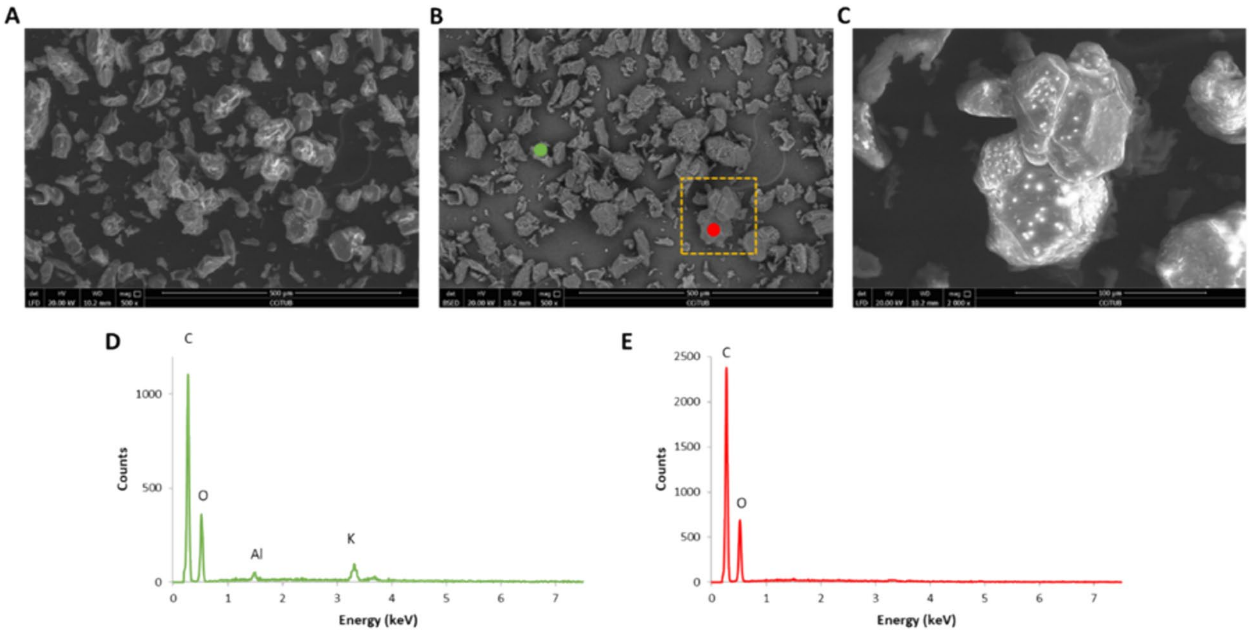

Fig. 2 Appearance of hazelnut-almond shells (Aval®) powder under the SEM. **A**, **B** SE and BSE mode images at 500×. Square in **B** indicates area enlarged in **C**; **C** close-up view of one of the particles in SE mode being, in this case, an almond shell fragment; **D**, **E** selected spectra of different fragments in **B**

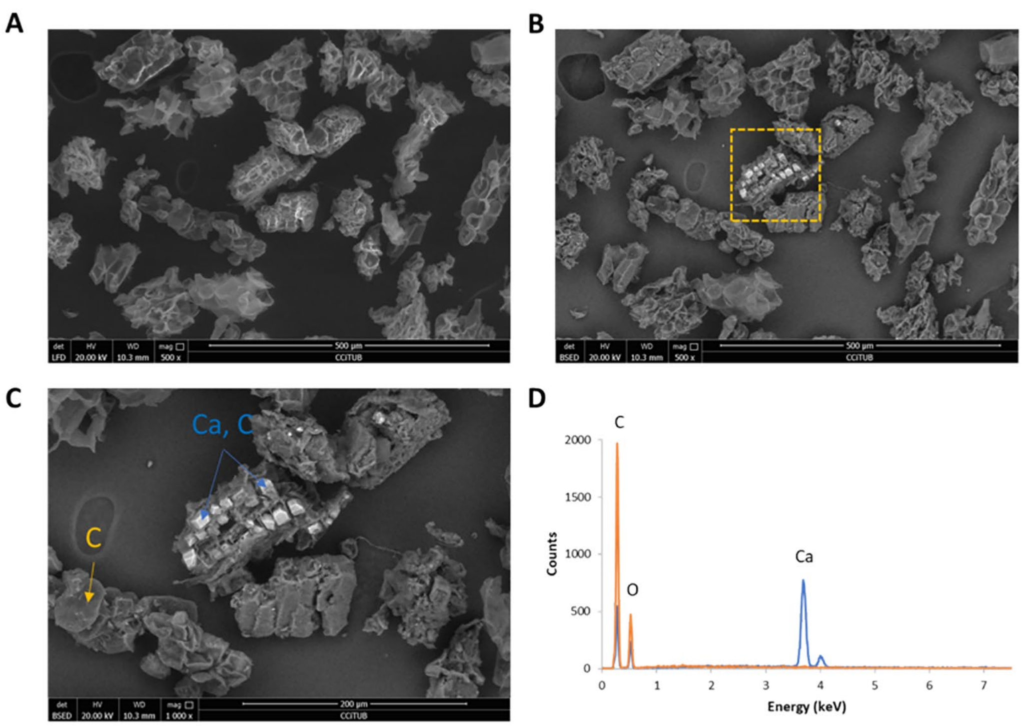

Fig. 3 Appearance of cork saw dust under the SEM. **A**, **B** SE and BSE mode images at 500×. Square in **B** indicates area enlarged in **C**; **C** close-up view of one of the particles in BSE mode. Note the highly porous structure consisting of hollow cells, some of which are filled with subeuhedral crystals (in white); **D** selected spectra of different regions in cork saw dust

and soil conditions, genetic origin, tree dimensions age and growth conditions [35].

Oryzite® RYZ-100

Representative SEM images are shown in Fig. 4 A–C. The sample appears as a rather heterogeneous material composed of agglomerates of micrometric subeuhedral calcium-rich crystals (Ca, average content of ca. 40%) associated with coarser amorphous silica-rich particles (Si, average content of ca. 15%). The EDS microanalysis also reveals the presence of carbon (C) as a major element (Fig. 4 D). It is known that rice husk is composed of a variety of components such as lignin, cellulose, and silica [36] so that the presence of C and Si is not surprising, however, the origin of Ca is still unknown.

Soiling composition

Generally speaking, the soiling adhered at the surface of the wood mock-ups is composed of metallic particles (Fe, Ti-Fe, Ti-Ba-Fe, Fe-Cu-Ni oxi-hydroxides), appearing both isolated or agglomerated, calcium-rich sulphate (Ca, S; probably gypsum), and aluminosilicates (Si, Al; mostly in the form of clay minerals, but also micas and feldspars). Spherules of different composition and sizes were also detected. All these compounds may be typically found in rural areas.

Main results concerning soiling's compositional and textural features are displayed in Table 3 and Figs. 5, 6, 7, 8 and 9.

Comparison of wood mock-up surface before and after cleaning

Coinciding with the differences observed under the digital microscope, the SEM–EDS analysis identified variations between surfaces cleaned with different vegetable particles at both, textural and elemental level.

Surfaces cleaned with Arbocel® BWW 40

DinoLite® images show a clean surface. Dust and some of the smaller dirt particles are removed. The wood surface looks well-defined and most of the raised wood fibres around the existing cracks are preserved. The use of DinoLite® with UV light is very useful to detect residues. Arbocel® emits a significant fluorescence under UV light because of its very bright white. After cleaning, and under UV light, no Arbocel® fibres are observed on the surface (Fig. 6).

SEM–EDS results concerning textural and elemental changes before and after surfaces cleaned with Arbocel®

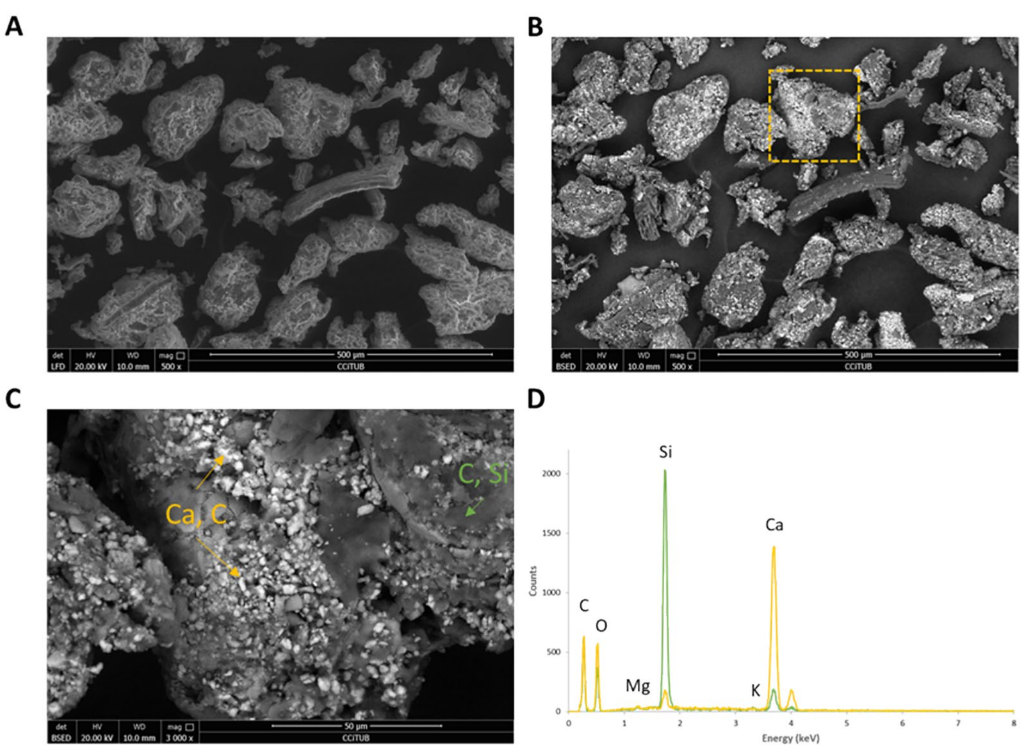

Fig. 4 SEM–EDS results obtained from rice husk powder (Oryzite®). **A**, **B** secondary and back-scattered electron (SE and BSE) mode images at 500×. Square in B indicates area enlarged in **C**; **C** close-up view of one of the particles in BSE mode. The lighter and darker zones in **B** and **C** correspond with elements of lower and higher atomic absorption. **D** selected spectra of different regions in a rice husk fragment

Table 3 Results of SEM–EDS analysis on wood mock-ups before cleaning

Sample	Description of analysed item				Elemental composition		Interpretation
	N	BSE contrast	Size (μm)	Description	Major	Minor	
2	1	Light grey	130 × 90	Isolated subeuhedral particle	Fe, C, Si, Al, Mg, Ca, S	K, Cl, S	Iron oxi-hydroxide
	5	White	NA	Micrometric subeuhedral particles agglomerated in a compact matrix	Ba, Ti, Ca, Si, Fe, S, Zn, Al, Mg	K, Cl, S	Barium-rich sulphate + titanium/iron-rich oxide + aluminosil.
	2	Light grey	30 × 20	Isolated subeuhedral particle	Fe, Si, Al, Ca, Mg, S	K	Iron oxi-hydroxide
	5	Light grey	NA	Agglomerate of micrometric particles	Ca, Si, S, Fe, Al, K	Mg, Ti, P	Calcium-rich sulphate + aluminosilic.
	1	White	10	Spheric particle	Fe, Si, Ca, Al, S	Mg, K	Iron-rich spherule
3	7	Light grey	NA	Agglomerate of micrometric particles	Ca, Si, S, Al, Mn, Fe, Mg, K	Ti, Na	Calcium- rich sulphate + aluminosilic.
	1	White	75 × 15	Elongated particle	Fe, Cu, Ca, Ni, Si, S, Al	Mg	Fe, Cu, Ni-rich compound
	3	White/dark grey	NA	Micrometric subeuhedral particles agglomerated in a compact matrix	Ba,Ti, Si, S, Ca, Mg, Al, Fe	K	Barium-rich sulphate + titanium/iron-rich oxide + aluminosil.
	2	White	200 × 85	Isolated subeuhedral particle	Si, Mn, Al, K, Ca, Mg, Ti, S	Cl	Manganese/titanum-rich compound
	1	White	40	Spheric particle	Si, Al, Ca, Mn, Mg, K, S	Ti, Na	Manganese-rich spherule
8	2	Light grey	35	Spheric particle	Ca, Si, S, Fe, Al	K, Mg, Ti	Iron-rich spherule
	11	Light grey	NA	Agglomerate of micrometric particles	Ca, S, Si, Al, Fe	K, Ti, Mg, Cl	Calcium- rich sulphate + aluminosilic.
	5	White	NA	Agglomerate of micrometric particles	**Fe**, Ca, Si, S, Zn, Al	Mg, K	Iron oxi-hydrox + aluminosil.
	3	Light grey	40 × 25	Subeuhedral plate	Si, Ca, Al, Fe, S, K, Al	Ti, Na	Aluminosilicate (mica?)
	3	White/dark grey	NA	Micrometric subeuhedral particles agglomerated in a compact matrix	Ba, Si, S, Ti, Ca, Mg, Fe, Al	K	Barium-rich sulphate + titanium/iron-rich oxide + aluminosil.
10	1	Light grey	35 × 25	Hexagonal plate	Fe, Si, Ca, Al, K, Mg, Ti, S		Iron oxide
	2	Grey	475 × 125	Low sphericity, rounded grain	Ca, Si, S, Al, Fe, Na	K, Mg	Aluminosilicate (feldspar?)
	9	Light grey	NA	Agglomerate of micrometric particles	Ca, Si, S, Al, Fe, K	Mg, Na, Ti, Cl	Calcium- rich sulphate + aluminosilic.
	2	Light grey	25 × 20	Medium sphericity, sub-rounded grain	Si, Ca, Al, Mg, Fe, S	K, P	Calcium-rich aluminosilicate (feldspar?)
	3	White/dark grey	NA	Micrometric subhedral particle agglomerated in a compact matrix	Ti, Ca, Fe, Si, S, Al, Mg, Na	K, Cl	Titanium/iron-rich oxide + aluminosil. + sulphate
	2	Grey	25 × 10	Subeuhedral particle	Si, Al, K, Ca, Fe, S, Mg	Ti, Na	Potassium-rich aluminosilicate (feldspar?)
	1	White	25 × 8	Low sphericity, subangular particle	**Fe**, Si, Ca, Al	Mg, S, K	Iron oxi-hydroxide

Elements in bold are present in a proportion equal or higher than 40%

N number of times these items were recorded. Major and minor elements constitute more than 1% and between 1.0 to 0.1% of the sample by weight respectively. Weight percentages including O and normalised to 100%.*NA* not applicable. Interpetation is referred to the main analysed item and it is inferred from element association

are displayed in Fig. 7. SEM images feature a clean surface. The larger dirt particles (> 10 μm) have almost entirely disappeared together with the smaller loose ones (between 10 and 1 μm), but compacted dirt particles that were present between wood fibres still remain in some areas. During cleaning the surface has been preserved, although some wood fibres have been moved. The larger cracks have been filled with the blasted powder. The cleaning process is progressive from 5 to 10 s and controllable.

The comparison of the pre-cleaning and post-cleaning stages (Fig. 7) also illustrates the changes in elemental composition of surfaces during the cleaning process EDS microanalysis reveals that Arbocel® is able to remove most of the soiling particles (in this case Fe, Ca, K, Si, Al and Mg) without damaging the wood surface (C).

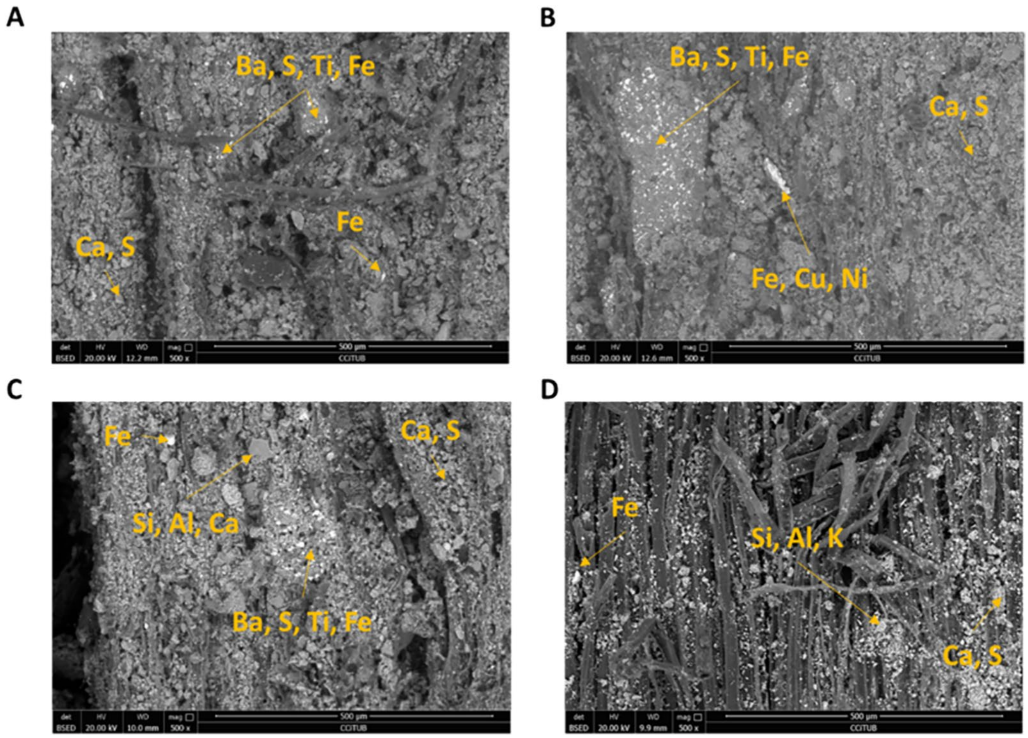

Fig. 5 Selected SEM images of wood mock-ups before cleaning (BSE mode, 500×). **A** mock-up n. 2; **B** mock-up n. 3; **C** mock-up n. 8; **D** mock-up n. 10

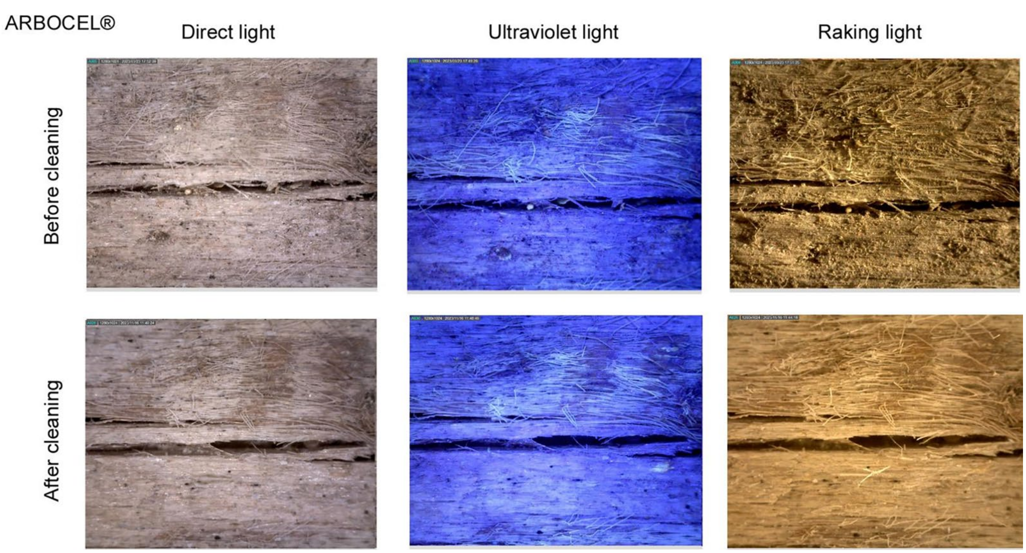

Fig. 6 Surfaces cleaned with Arbocel®. Digital microscope images 65×. Cleaning effects (direct, ultraviolet and raking light)

Surfaces cleaned with Aval®

Digital microscope images before cleaning with Aval® show a group of disordered and broken wood fibres (lower area). After 5 s of microblasting these fibres disappear from the surface. White spots that were underneath the superficial dirt can be now observed. No remarkable difference is observed after 10 s under direct visible light (Fig. 8).

Hazelnut and almond shells do not show fluorescence under UV light; thus, UV light does not reveal significant information on Aval® residues left on the surface.

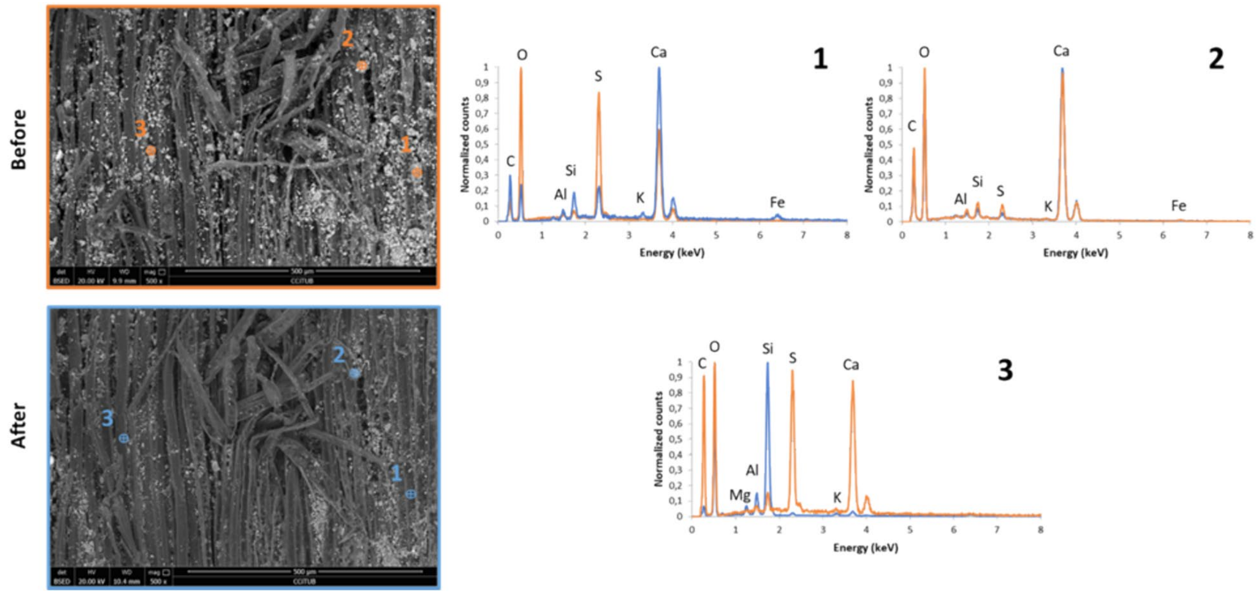

Fig. 7 SEM–EDS results obtained from mock-up n. 10. Left: BSE mode images at 500× from before (above, orange) and after (below, blue) the cleaning test with Arbocel®. 1 to 3 points indicate the analysed items. Right: EDS spectra corresponding to these points

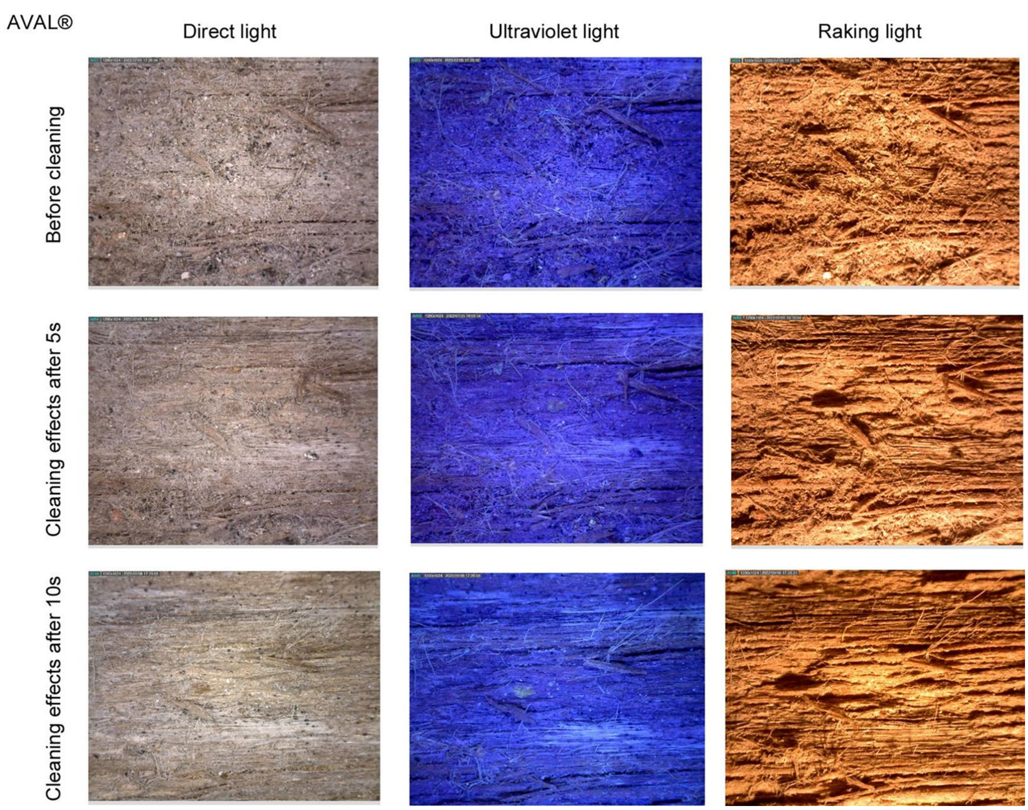

Fig. 8 Surfaces cleaned with Aval®. Digital microscope images 65×. Cleaning effects after 5 s and 10 s (direct, ultraviolet and raking light)

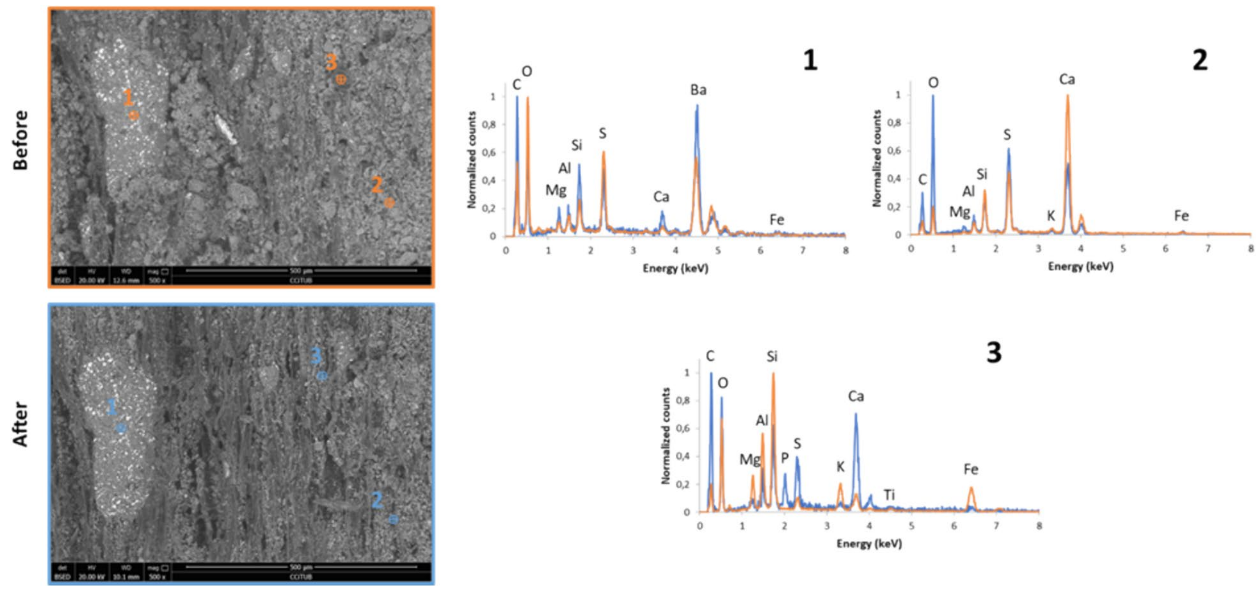

Fig. 9: SEM–EDS results obtained from mock-up n. 3. Left: BSE mode images at 500 × from before (above, orange) and after (below, blue) the cleaning test with Aval®. 1 to 3 points indicate the analysed items. Right: EDS spectra corresponding to these points

Raking light under a digital microscope clearly shows the evolution of the cleaning progress. After 5 s a significant cleaning effect is observed, that is improved after 10 s. Wood surface is preserved and no polishing, nor edge rounding effect is observed.

SEM–EDS results concerning textural and elemental changes before and after surfaces cleaned with Aval® are displayed in Fig. 9. SEM–EDS images show a significant change in surface texture before and after cleaning. In the image of the unclean surface (before), it can be seen the accumulation of soiling particles of different morphology that appear both isolated and forming more or less compact agglomerates. After 10 s of Aval® microblasting, a large amount of these particles has been removed, and only the more compact agglomerates remain. The preserved wood fibres can be clearly seen after the cleaning process.

Regarding to the elemental composition, the EDS microanalysis reveals that after the Aval® microblasting there is a very clear decrease of Fe-rich particles, and also of some of what would probably be calcium sulphates (Ca, S) and aluminosilicates (K, Si, Al, Mg). Also, after cleaning, the carbon (C) peaks corresponding to wood clearly appear.

Surfaces cleaned with Cork

Digital microscope images show that the cleanliness results after 5 s are evident. The structure of the wood is quite well preserved. There is no loss of wooden material. No surface changes are observed. The transition of the cleaning level between 5 and 10 s is progressive (Fig. 10).

Cork do not show fluorescence under UV light; thus, UV light does not reveal significant information of cork saw dust residues left on the surface. White fluorescent spots of indeterminate origin appear under the surface dust, which become more defined as the degree of cleanliness increases.

Raking light under a digital microscope clearly shows that the wood surface has been preserved and retains its morphology without surface changes.

SEM–EDS results concerning textural and elemental changes before and after surfaces cleaned with cork saw dust are shown in Fig. 11.

SEM analyses reveal a surface featuring clean and well-defined fibres, devoid of both large (> 20 µm) and small (between 20 and 5 µm) particles of dirt. With the SE mode it can be observed that the original dirt is made up of particles of diverse morphology, more or less compacted, and by the presence of randomly distributed filamentary structures. The cracks are rounded due to the effect of the deposits covering the sample.

EDS microanalysis reveals that the dirt covering the surface is characterised by the presence of Fe, Ti, Ca, K, S, Si, Al and Mg. After cleaning, a second layer of dirt appears below the first one, which could correspond to barium-rich sulphate (Ba, S; spectra 1 and 2). The most intense Ca, S and Ti peaks also appear, possibly from removed particles of calcium-rich sulphate and

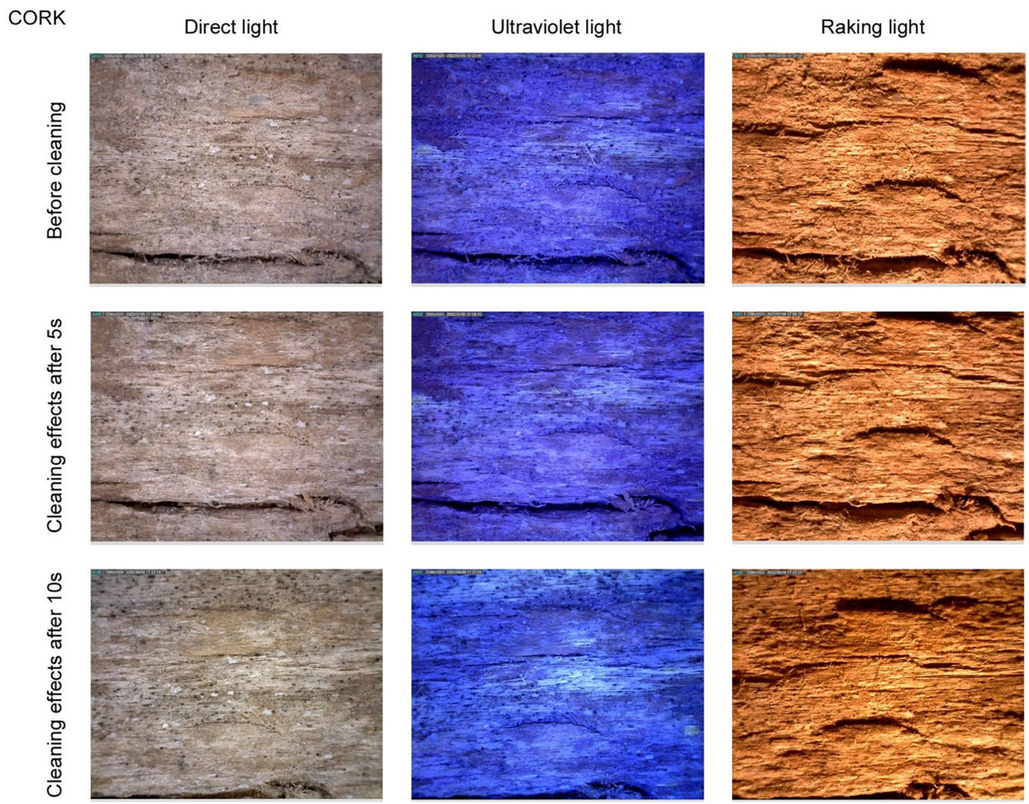

Fig. 10 Surfaces cleaned with Cork. Digital microscope images 65×. Cleaning effects after 5 s and 10 s (direct, ultraviolet and raking light)

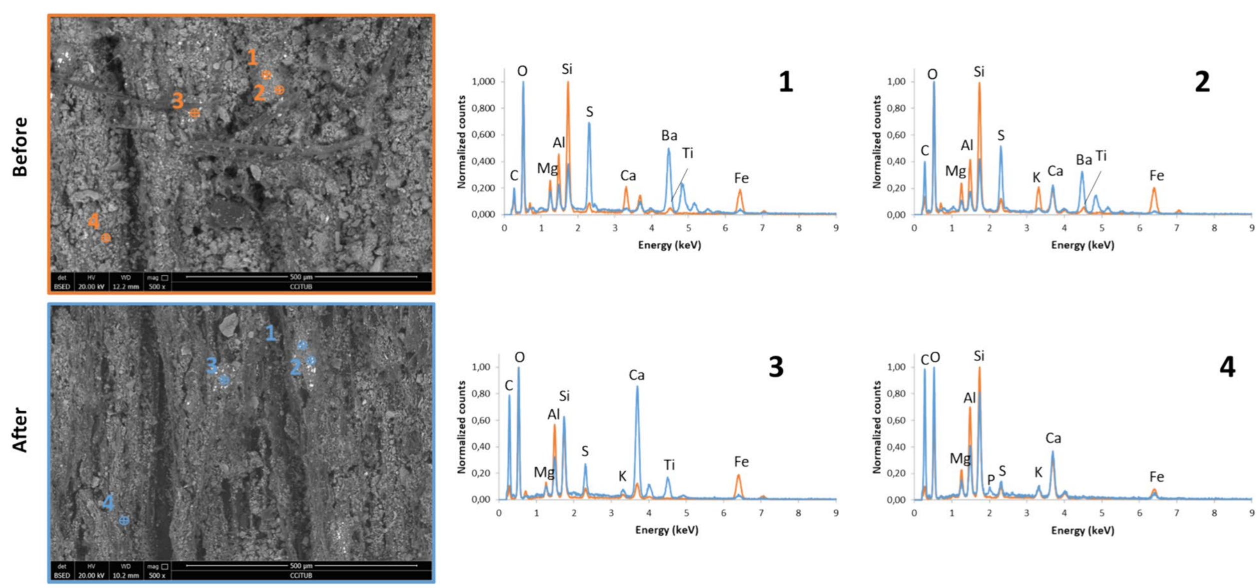

Fig. 11 SEM–EDS results obtained from mock-up n. 2. Left: BSE mode images at 500× from before (above, orange) and after (below, blue) the cleaning test with cork dust. 1 to 4 points indicate the analysed items. Right: EDS spectra corresponding to these points

titanium-rich oxide. In all cases, after cleaning, the carbon peak (wood) increases considerably.

The cleaning process is progressive from 5 to 10 s. At 5 s, part of the dirt has already disappeared and the surface appears quite clean, with the cracks appearing defined. After 10 s of cork microblasting, dirt has practically disappeared, leaving only some of the more compact agglomerates. It can be observed that the cracks acquire a certain width and definition due to the removal of the accumulated dirt.

Surfaces cleaned with Oryzite® RYZ 100

Digital microscope magnification after 5 s of microblasting shows that Oryzite® powder has removed a very significant amount of dirt. Nevertheless, initial signs of wood surface changes can be identified. The white spots that were visible before cleaning are now worn away. The arrangement of the fibres in the upper part of the image show a characteristic fibre breakage. This arrangement at the 5 s image looks cleaner and slightly rounded (top right image, rounded edges). At 10 s, the rounding of edges and loss of relief is clearer and many of the fissures have been filled by Oryzite® powder (Fig. 12).

With digital microscope UV light, the remains of Oryzite® are visible inside the fissures (they fluoresce due to their whitish colour). The crack at the top, sharp and cutting in origin, looks very clean at 5 s, but the profile is smoother and has lost fibres. At 10 s it is clearly polished/rounded and has lost the sharpness.

Under raking light, the rounding of the edges cannot be seen, but a surface change of the less hard fraction of the growth rings of the wood is observed. The spring wood, being softer and less lignified, suffers from surface change, consequently the raking light shows how the surface relief is slightly accentuated after treatment.

SEM–EDS results concerning textural and elemental changes before and after surfaces cleaned with rice husk powder (Oryzite®) are displayed in Fig. 13.

SEM analyses show a slightly worn surface. Both large (>20 μm) and small (between 20 and 5 μm) particles of dirt have been removed, but some wood fibres have also been eliminated. The surface after cleaning appears smoother and more homogeneous. The edges of the cracks are rounded, and the smaller fissures have been filled with the finer microblasted particles. The cleaning process is very fast and therefore more difficult to control.

The EDS spectra before and after cleaning confirm the results observed with SEM. Except in some specific cases (spectrum 1), the soiling (Fe, Ca, K, Al, Mg, Na)

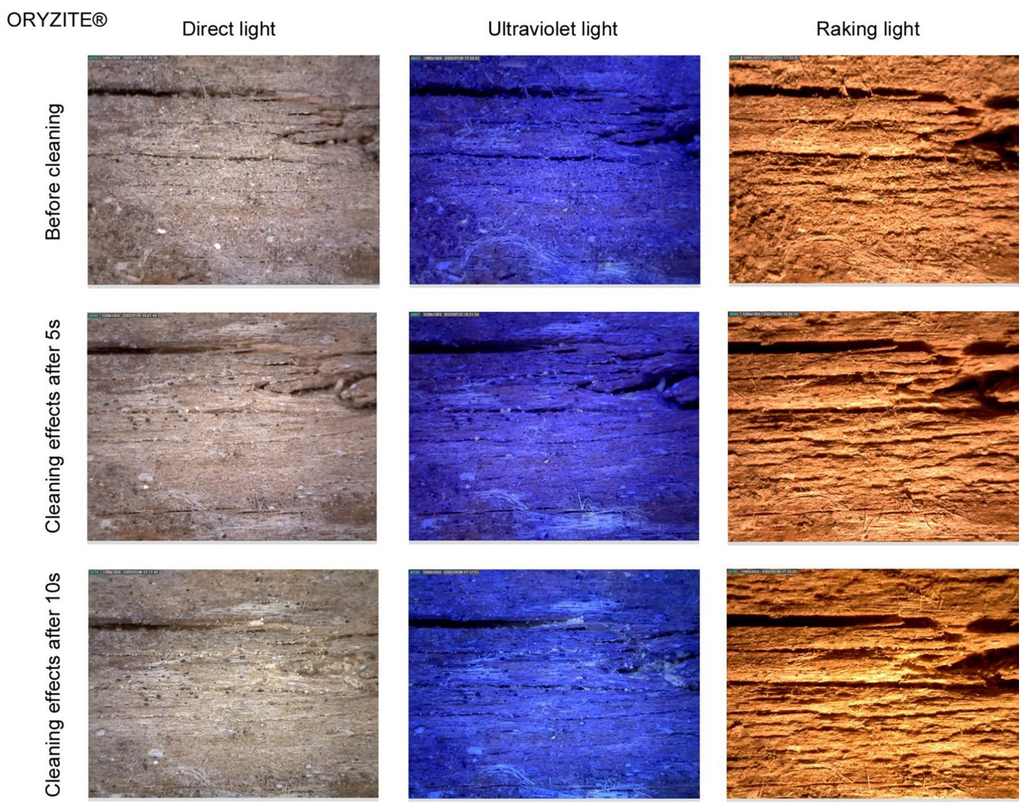

Fig. 12 Surfaces cleaned with Oryzite. Digital microscope images 65×. Cleaning effects after 5 s and 10 s (direct, ultraviolet and raking light)

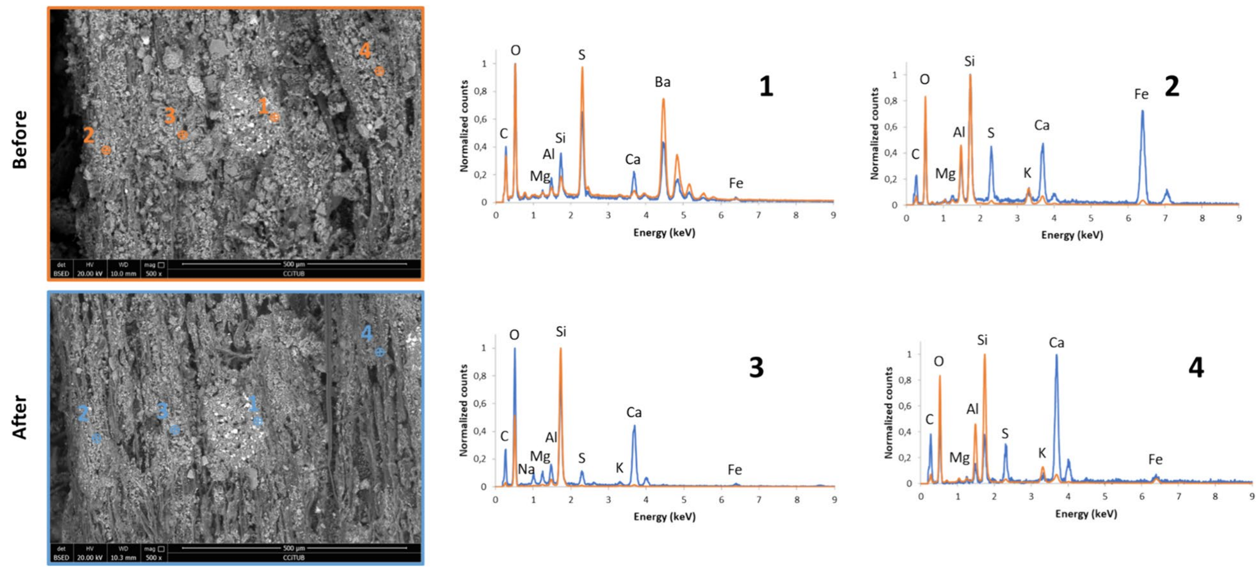

Fig. 13 SEM–EDS results obtained from mock-up n. 8. Left: BSE mode images at 500× from before (above, orange) and after (below, blue) the cleaning test with Oryzite®. 1 to 4 points indicate the analysed items. Right: EDS spectra corresponding to these points

disappears very quickly. However, the analyses show that the peak of Si increases systematically with cleaning (also that of Al in some cases) and, on the other hand, the rest of the elements decrease. A possible explanation is that this Si peak originates from the rice husk powder, which may be linked to the observed filling or compacting effect of the surface at lower magnifications. Another possibility might be the presence of an aluminosilicate soiling appearing under the first soiling layer, which would also explain the presence of Al.

Surfaces cleaned with microsilica

Digital microscope magnification shows that microsilica has removed a significant amount of dirt. Nevertheless, signs of wood surface changes can be easily identified. The arrangement of the fibres shows a characteristic fibre breakage. Wood surface looks clean but rounded and damaged. The rounding of edges and changes in surface relief and texture are clearly visible as well as residues of microsilica between the fibres and within the holes (Fig. 14).

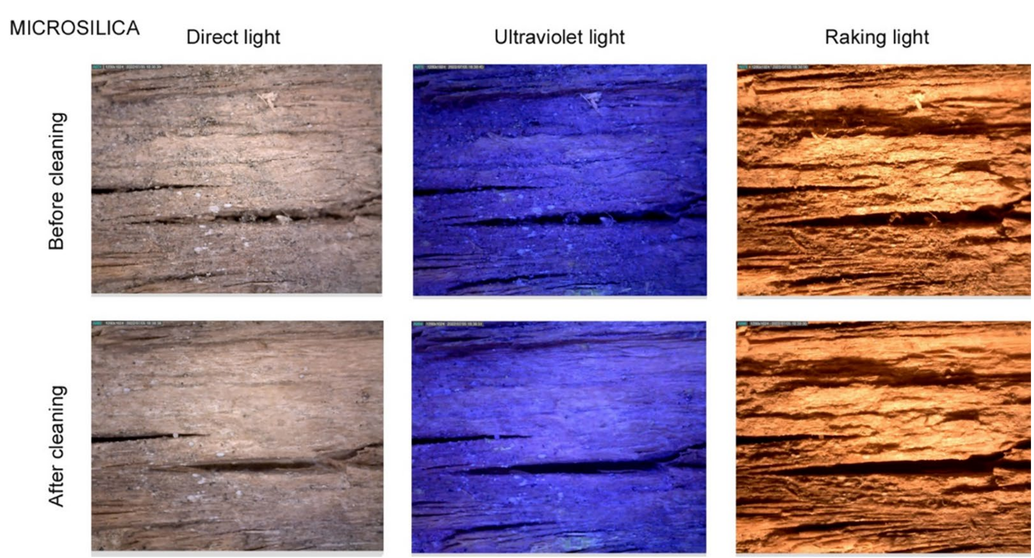

Fig. 14 Surfaces cleaned with microsilica. Digital microscope images 65×. Cleaning effects (direct, ultraviolet and raking light)

With digital microscope UV light, the remains of microsilica are not visible inside the fissures. The cracks have a profile smoother and have lost surface fibres. Under raking light, surface changes of the less hard fraction of the growth rigs of the wood is observed. The spring wood, being softer and less lignified shows further damage, consequently the raking light shows how the surface relief is more pronounced after treatment.

Cleaning results comparison

The results depicting textural variations between surfaces before and after cleaning with different vegetable particles and the cellulosic one are presented in Figs. 15, 16, 17, 18 and 19 and Table 4.

The four microblasted vegetable particles proved to have an effect on dirt removing both large and small particles larger than 1 µm.

As for their effect on the surface of wood samples once cleaned, none of the samples show significant surface changes. Only slight wood fibre movements and small breakages with Aval® and Oryzite® are provoked, unlike the samples cleaned with Arbocel® and Cork, which remain unaltered after cleaning.

Regarding to the control of the microblasting process, all three vegetable particles and the cellulosic one could be considered gentle and controllable for cleaning. However, when comparing them, the samples cleaned with Aval® and Oryzite® already show significant cleaning effects after 5 s, while the samples cleaned with Arbocel® and Cork require more time (10 s) to consider the surface cleaned. From a conservator-restorer point of view, 5 s is a very short time lapse for cleaning. Thus, having some more seconds to control the procedure could be an advantage.

Concerning the amount of cleaning residues left on the surface, some particles are observed between the fibres and within the holes in samples cleaned with Arbocel® and Orycite®. In contrast, no significant

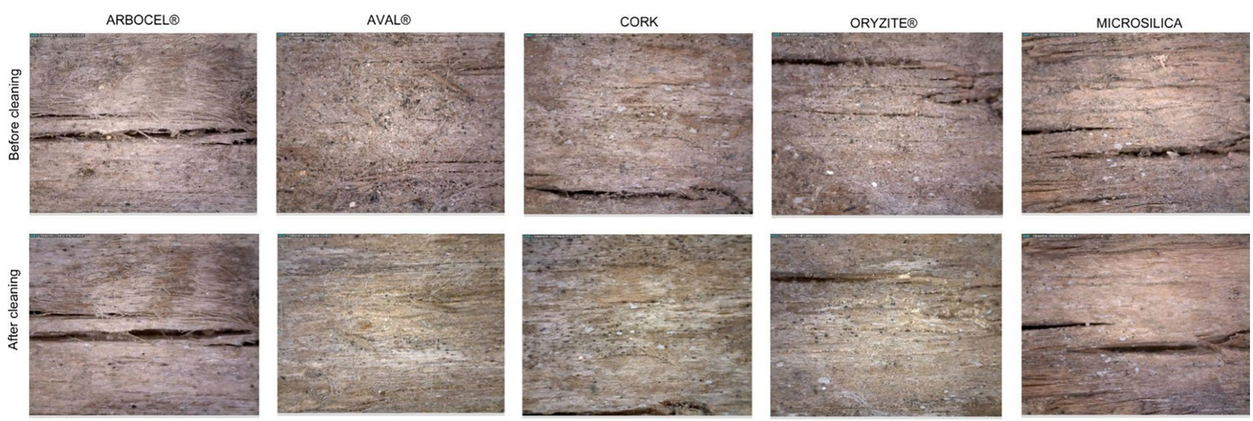

Fig. 15 Selected digital microscope images (65×). Visual summary of the results showing textural variations between surfaces before and after cleaning with different vegetable, cellulose and microsilica particles

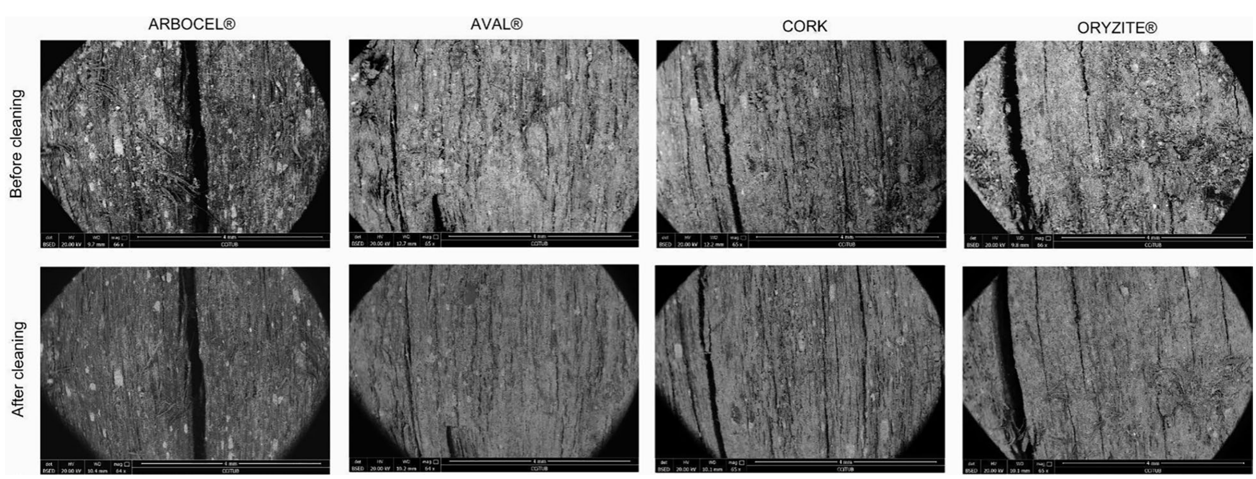

Fig. 16 Selected SEM images in BSE mode of wood mock-ups before and after cleaning. Magnification at 65×

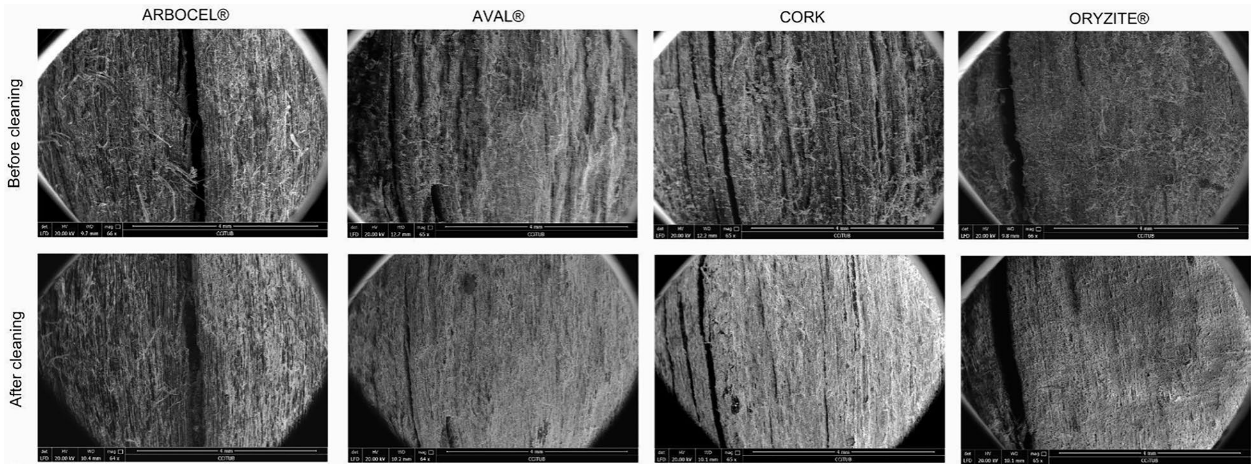

Fig. 17 Selected SEM images in SE mode of wood mock-ups before and after cleaning. Magnification at 65×

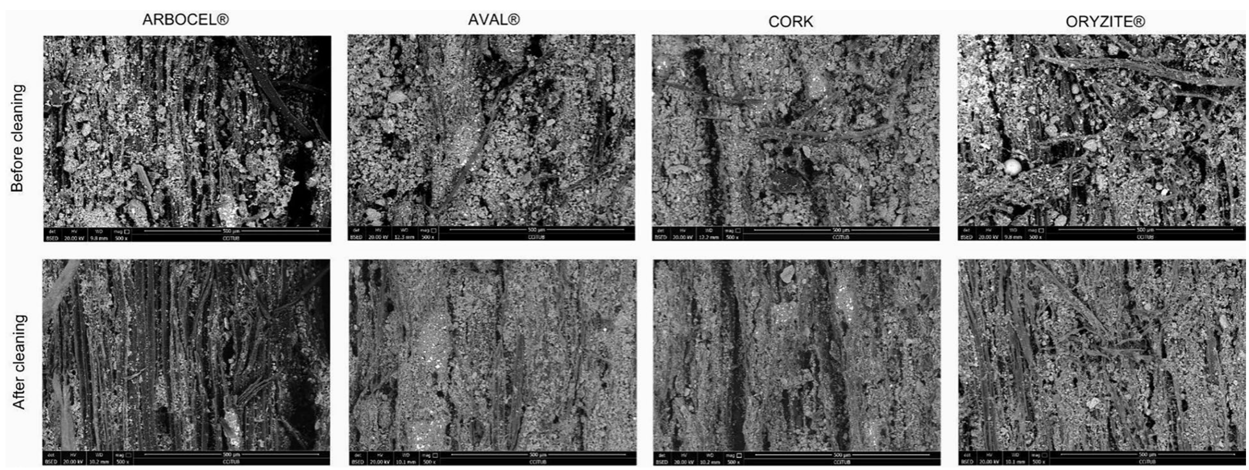

Fig. 18 Selected SEM images in BSE mode of wood mock-ups before and after cleaning. Magnification at 500×

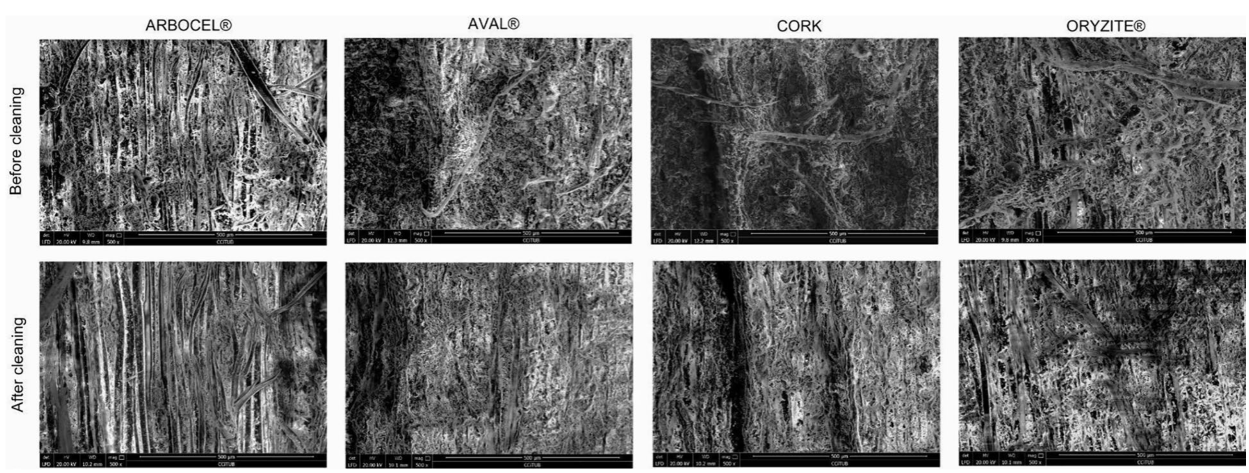

Fig. 19 Selected SEM images in SE mode of wood mock-ups before and after cleaning. Magnification at 500×

Table 4 Main results concerning textural variations between surfaces cleaned

Microblasting	Particles size < 200 µm	Arbocel®	Aval®	Cork	Oryzite®	Silica
Effects on soiling	Removes large and small soiling particles (> 1 µm)	X	X	X	X	X
	Does not remove soiling particles					
Effects on wood surface	Surface changes with wood losses					X
	Slightly changes on wood surface with breakage or movement of fibres		X		X	
	Does not modify the wood surface	X		X		
Cleaning process	Fast cleaning at 5 s		X		X	X
	Progressive cleaning from 5 to 10 s	X		X		
Cleaning material residues	Some residues are left between the fibres and inside the cracks	X			X	X
	No residues visible with DM and SEM		X	X		

residues are detected in samples cleaned with Aval® and Cork.

In comparison, the sample cleaned with microsilica shows evident surface changes. In this case the wood fibres are displaced and broken, ultimately presenting a completely different appearance after cleaning. Cleaning control using silica is difficult because of the faster erosion on the wood surface; at 5 s the surface is clearly damaged. Additionally, visible residues of microsilica remain between the fibres and within the holes.

The main results concerning textural variations between surfaces cleaned are displayed in Table 4.

Conclusions

This research proves that microblasting with powdered vegetable and cellulosic media is a useful and safe technique to remove soiling from wood, including micrometric particles larger than 1 µm embedded in its texture, with negligible surface changes.

Microblastig cleaning with the tested vegetable and cellulosic particles is a fast procedure. Cleaning effectiveness at 5 s with Aval® and Oryzite®, and from 5 to 10 s in samples treated with Cork and Arbocel® can be seen.

None of the three vegetable particles nor the cellulosic one have caused noticeable damage on wood mock-up surfaces. However, among the tested vegetable particles, Aval® and Oryzite® have to be applied bearing in mind that they might move, or even break, lifted or weaken fibres depending on wood conservation condition.

The residues of the vegetable and cellulosic powdered particles left after cleaning are very scarce. In the event of residues remaining on the surface, they are chemically compatible with the substrate because they are mainly composed of cellulose, hemicellulose, lignin and other vegetable compounds, and thus some of the main constituents of wood.

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Author contributions
M.A.I.C., A.N.T., A.P.M., I.B.M. conceptualized, investigated and interpreted the data obtained in this research and all of them also wrote this article (original draft preparation, review and editing). M.A.I.C., A.N.T., A.P.M. defined de methodology for cleaning tests and evaluation. M.A.I.C., A.N.T. performed the cleaning tests; A.P.M., I.B.M. were in charge of data curation. M.A.I.C., A.N.T., A.P.M., I.B.M. contributed to funding acquisition; I.B.M., A.N.T. held project administration. All authors read and agreed to the manuscript.

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Declarations

Ethics approval and consent to participate
Not applicable.

Consent for publication
Not applicable.

Competing interests
The authors declare that they have no competing interests.

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