NEW APPLICATIONS IN THE USE OF CELLULOSE PULP FOR THE INTEGRATION OF WOODEN SUPPORTS

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1. Introduction

The plastic reintegration of historical-artistic wooden artworks in the field of restoration has been somewhat neglected by scientific research and mostly relegated to the sector of traditional handicrafts. In the course of time, restorers have used a wide range of traditional products, such as chalk and rabbit glue, wax, and other more common synthetic materials. However, these materials have not always been appositely designed for the restoration of wooden artworks. The listed materials have also often been mixed together in an arbitrary way, without taking into account whether they were compatible with the wooden structure and to what degree. So far, few attempts have been made to identify new and more appropriate materials that might be used for this purpose [1, 2]. Modern materials for wood restoration have quite good characteristics (easily applicable, speed of drying, good workability), but these positive aspects often overshadow the compatibility of the products with the wood.

The present study shows, also in terms of materials compatibility, several products that are the most suited for wood restoration, as well as demonstraing the potential use of cellulose pulp as being well-suited for the restoration of wooden artifacts also from a chemical point of view, as it is the main component of wood (Figure 1).

Cellulose pulp is a material that is already used in the field of restoration for other purposes and here it is suggested in a new role as a stucco for wood, mixed with different adhesives selected from the wide range of present-day products [3]. By analysing the cellulose pulp it was possible to investigate this material in-depth and evaluate its original properties, as well as consider the susceptibility of cellulose material to microbial colonization.

Moreover, the samples of cellulose pulp underwent artificial aging and solubility and reversibility tests using known polarity solvents in order to evaluate the potential use of cellulose pulp as a stucco in the reintegration of wooden artifacts, as an alternative to materials utilized today.

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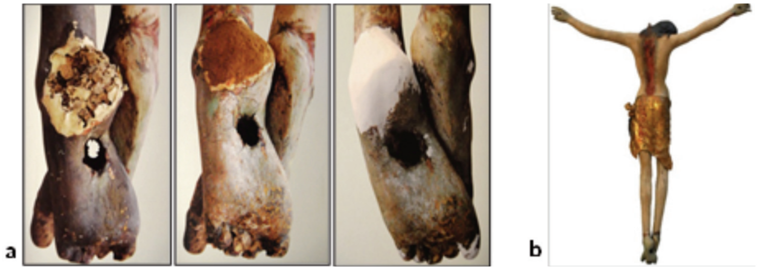

Figure 1. a) Different steps in gap-filling on a papier-mache crucifix (16th century) stored in the Diocesan Museum in Palermo. Detail of the Christ's heel before restoration (on the left); reintegration with pigmented cellulose pulp+acrylic resin (center); refining in gypsum+ rabbit glue (on the right); b) Crucifix (verso).

2. Materials and methods

In order to give the cellulose pulp-based stucco good filler properties, research was carried out on adhesives with fairly good elasticity, malleability and ease of preparation, minimal water intake and that were also lightweight, fast drying, and strong [4].

Considering these specific aspects, we assayed three adhesives based on: *i*) proteins (rabbit glue); *ii*) cellulose ethers (Klucel-G, Tylose MH300P); *iii*) synthetic polymers (BEVA-D8S, PLEXTOL-B500, AQUAZOL 200).

Attention then focused on the materials made of cellulose ethers because, up to now, they have been considered the most suitable in terms of non-toxicity, reversibility and biodegradability, although they have not yet been scientifically tested as adhesives for fillers. The adhesives assayed in this work are listed below:

– RABBIT GLUE: a water-soluble animal glue, characterized by high adhesive power, created by prolonged boiling of animal connective tissue. The glue was assayed at different concentrations (1:5; 1:7; 1:10; 1:14). In particular, the concentration equal to 1:14 was considered the most appropriate to use, because the dough was more flexible during the drying step. However, its excessive water content can limit its application in wooden artifacts.

– TYLOSE MH300 P: hydroxyl-methylcellulose is a synthetic polymer. Its adhesive power is very low, it is thus, easily reversible. Generally, Tylose solutions are utilized for paper restoration as 1-2.5%. For the stucco preparation Tylose MH300P was used at 5% in H_2O or at 5% in H_2O/EtOH (1:1).

– KLUCEL G: hydroxyl-propylcellulose has high surfactant properties. It is utilized as a fixative in mural paintings or in paper restoration at low concentrations. For the stucco preparation, it was used at 5% in H_2O or at 5% in H_2O/EtOH (1:1).

– AQUAZOL 200: thermoplastic polymer is made of two poles (2-ethyl-2-oxazoline). It is used as a consolidating agent in paint layers; it is soluble in water and in high / medium polarity-solvents. It is biodegradable and has good thermal stability and low toxicity. In this study, Aquazol 200 was utilized at 25% in EtOH.

– PLEXTOL B500: thermoplastic acrylic resin, with good viscosity in aqueous dispersion. Acrylate and methacrylate copolymer with good chemical resistance. It is utilized for relining paintings and for re-adhesion of paint layers. In this study, it was utilized pure to obtain a very structural filler stucco.

- BEVA D8S: consists of an ethylene vinyl acetate emulsified by polyvinyl-alcohol. It is utilized mainly for canvas waterproofing. In this study it was utilized pure to obtain a very structural filler stucco.

All stuccos were prepared maintaining the ratio of adhesive / cellulose powder (4 ml of adhesive/1 g of cellulose) constant. Several samples were designed for each type of stucco in order to carry out the analysis below:

1. *Weight loss (%):* to evaluate effective stucco weight
2. *Shrinkage*: analysis of sample, dimensions decrease during drying (Figure 2)
3. *Artificial aging*
4. *Reversibility test*: to evaluate the removal of material, both mechanically or using solvents with increased polarity
5. *Microbiological susceptibility*: to determine the microbial susceptibility of cellulose.

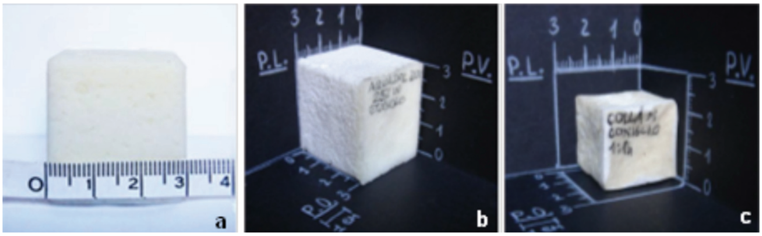

Figure 2. a) wet sample; b) cellulose pulp + Aquazol 200 dry sample, without shrinkage during drying; c) cellulose pulp + rabbit glue with shrinkage during the drying.

2.1 Shrinkage and weight loss in percentage

For each adhesive, we prepared two samples using a silicon cubic mould (3 cm^3). One sample was constantly monitored in order to establish the decrease in weight over one day, finally calculating the total weight loss of the sample. The other sample was utilized to find the decrease in volume during the drying process.

2.2 Artificial aging

In order to evaluate product performance over time and the ease with which they can be removed, the stuccos were artificially aged. The assays were carried out on the previously described stucco samples; stucco was applied on the wooden samples (20´15.5´2 cm) from an ancient spruce girder (18th century). On each sample, a lacuna was obtained (7´5´2 cm). Insulating material was layered on the wood surface before applying the stucco, with the aim of preventing the adhesive from penetrating the wooden fibers.

Finally, the samples colored by water colors were dried, in order to detect any alterations after the aging process. Artificial aging was carried out in an aging chamber under UV rays, WEISS TECHNIK 500SB (T=22°C, RH=60-65%) for 2 months.

2.3 Reversibility

Solubility tests were carried out on artificially aged samples using three different solvents, white spirits (ethers), acetone (ketones) and ethanol (alcohols), to find the lowest polarity necessary to solubilize and remove the material.

2.4 Microbiological susceptibility

Microbiological tests were performed (Figure 3) in order to verify the microbial susceptibility of cellulose pulp, and evaluate if the artificial aging process would increase microbial development in the stucco [5].

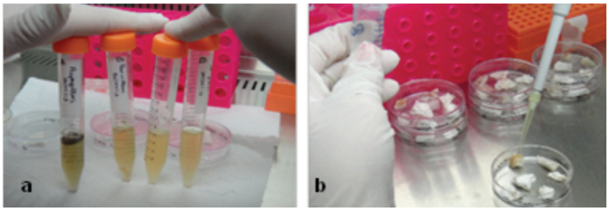

Figure 3. Preparation of microbial suspension in Nutrient Broth (a); inoculum on stucco sample (b).

2.4.1 Microbiological assay n.1

In order to determine susceptibility of the stucco to microbial colonization, we compared new stuccos with artificially aged stuccos.

Aliquots of microbial culture were inoculated into the new cellulose pulp stucco samples, prepared using different adhesives (rabbit glue, Klucel G, Tylose MH300P, Plextol B500, Beva D8S) [6, 7]. The same procedure was also carried out on the artificially aged samples, in order to observe potential differences in microbial growth between the new and aged samples. For each typology of stucco, five unaged samples and five aged samples were prepared. All samples were put into empty Petri dishes (9 mm in diameter). All tests were performed in a microbiological hood (laminar flow hood) in order to limit external contamination (Figure 4). Aliquots of 5 ml of different microbial suspensions, *Aspergillus* sp., *Penicillium* sp., *Micrococcus* sp. or *Bacillus* sp. (in Nutrient Agar liquid culture), were inoculated into each stucco sample. The samples were then incubated at 30°C for 14 days. Negative controls using stucco samples without inoculum were prepared.

Micrococcus sp. and *Bacillus* sp. (bacteria), and *Aspergillus* sp. and *Penicillium* sp. (fungi) were tested, since these microbial species are frequently isolated on artwork surfaces.

2.4.2 Microbiological assay n.2

In the second test, we assessed the role of stucco components in microbiological colonization. For this purpose, six samples of cellulose, adhesive and stucco were prepared separately or mixed together (Figure 4). Aliquots of 5 ml of bacterial (*Micrococcus* sp., *Bacillus* sp.) or fungal (*Aspergillus* sp., *Penicillium* sp.) suspension were inoculated into all samples and then incubated at 30°C for 14 days under wet conditions (Figure 5).

The assay was also proposed for the 18th century spruce girder samples, previously used for reversibility tests and subjected to artificial aging. The wooden samples (with microbial inoculum) were incubated at 30°C for 14 days.

Figure 4. Different components used in the preparation of the stucco, cellulose and rabbit glue (on the left); dry stucco (on the right).

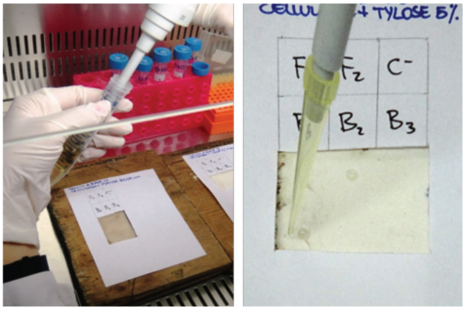

Figure 5. Inoculum of microbial suspension on wooden sample.

3. Results and Discussion

The macroscopic features were investigated, during preparation, application, drying and mechanical finishing for each stucco sample and are summarized in Table 4.

In terms of speed of drying, lightness and superficial finishing, the stuccos based on cellulose ethers showed the best qualities, regardless of the solvents in which they were dissolved. The advantage is the possibility to reduce and even eliminate the water content of the stucco without changing its features.

The best materials are the stuccos based on cellulose ethers dissolved in the mixture of 50% water and ethanol (Klucel G and Tylose MH300 P). The doughs made of acrylic adhesives cannot be adopted for use in the finish because they are difficult to work mechanically once dried. Moreover, they can be better used for reintegration of lacunae, in place of Araldite and Balsite.

3.1 Shrinkage and weight loss in percentage

The group of acrylic adhesives showed a moderate reduction in weight and no shrinkage. On the other hand, the loss of weight in the cellulose ethers samples was significant, equal to more than half of their initial value, while shrinkage was less or equal to 0.5 cm (Table 1).

The acrylic adhesives, therefore, are characterized by low shrinkage; however, their weight should be taken into account, as adhesives are also used to fill large lacunae; the stuccos made of cellulose ethers have opposite features. Table 2 reports the weights of both the wet and dry samples. Data show the weight loss of each sample following solvent evaporation, thus confirming the lightness of some adhesives with respect to others.

The weight loss in the samples was different to that in the wooden supports, which was greater because of the adhesive absorption of the wood material.

In particular it was observed that the lighter values in terms of weight loss (%) refer to cellulose ethers (72-78%), while the heavier values refer to acrylic resins (39%).

For the samples made of cellulose ethers dissolved in ethanol, rapid weight loss was observed in 2 h. Weight loss involved in samples with major water content (rabbit glue or cellulose ethers) was slower, but these samples were lighter once dried.

Stucco made of rabbit glue or Tylose MH300 P dissolved in water has high shrinkage and is lower in the sample prepared in water and ethanol (vol 1:1).

Table 1. Average shrinkage of dry samples.

Stucco	Shrinkage (cm)
RABBIT GLUE in H_2O (1:14)	0.8
TYLOSE MH300 P at 5% in H_2O	0.5
TYLOSE MH300 P at 5% in H_2O/EtOH (1:1)	0.3
KLUCEL G at 5% in EtOH	0.4
KLUCEL G at 5% in H_2O/EtOH (1:1)	0.3
AQUAZOL 200 at 25% in EtOH	0.0
PLEXTOL B500 (pure)	0.1
BEVA D-8-S (pure)	0.1

Table 2. Weight loss in dry samples.

Stucco	Weight wet sample (g)	Weight dry sample (g)	Weight loss (%)
RABBIT GLUE in H_2O (1:14)	36.1	10.4	73%
TYLOSE MH300 P at 5% in H_2O	31.1	8.3	73%
TYLOSE MH300 P at 5% in H_2O/ EtOH (1:1)	33.6	8.6	74%
KLUCEL G at 5% in EtOH	34.0	7.4	78%
KLUCEL G at 5% in H_2O/EtOH (1:1)	33.2	9.3	72%
AQUAZOL 200 (at 25% in EtOH)	37.0	14.7	60%
PLEXTOL B500 (pure)	42.4	25.8	39%
BEVA D-8-S (pure)	40.8	25.0	39%

3.2 Artificial aging

After the aging process, the differences between the samples were observed; some samples were characterized by cracking and detachments from lacuna edges and shrinkage of a few millimeters. The rabbit glue stuccos maintained their stability, remaining compact and planar, as in the Aquazol 200 stuccos. Some fissures, probably due to less elasticity during the artificial aging, were observed in the Tylose MH300 P stuccos.

3.3 Reversibility

The removal both of Tylose MH300 P and Aquazol 200 stuccos from lacunae in wooden samples was prompt in ethyl alcohol. Plextol B500 and Beva D8S stuccos were difficult to remove using solvents and it was necessary to remove them using a mechanical action. Instead, removal of the rabbit glue stucco was easy and was removed completely with the help of a scalpel (Figure 6). All solubility test results are reported in Table 3.

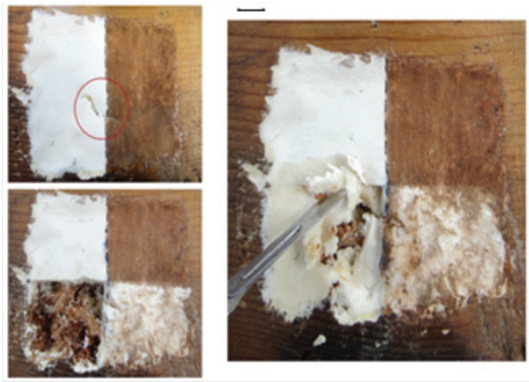

Figure 6. Tylose MH300P stucco removal.

Table 3. Solubility tests of adhesives after artificial aging.

	Rabbit glue in H_2O (1:4)	TYLOSE MH300 P at 5% in H_2O	TYLOSE MH300 P at 5% in H_2O/EtOH (1:1)	AQUAZOL 200 at 25% in EtOH	PLEXTOL B500 (pure)	BEVA D-8-S (pure)
White spirit	1	1	1	2	1	1
Acetone	2	2	2	4	3	3
Ethanol	4	4	4	4	2	2

1- No removal; 2- low removal; 3- partial removal; 4- good removal

3.4 Microbiological susceptibility

In microbiological assay n.1, microbial colonies developed on both aged and new samples after 14 days of incubation. In particular, extensive microbial growth was detected on rabbit glue samples, also due to the lack of a biocide agent. As a result, it appears that the aging did not favor microbial development. In the case of the acrylic resins, no microbial growth was detected (Figure 7b) in the new samples, while the aged ones were characterized by some microbial colonization (Figure 7c) [8].

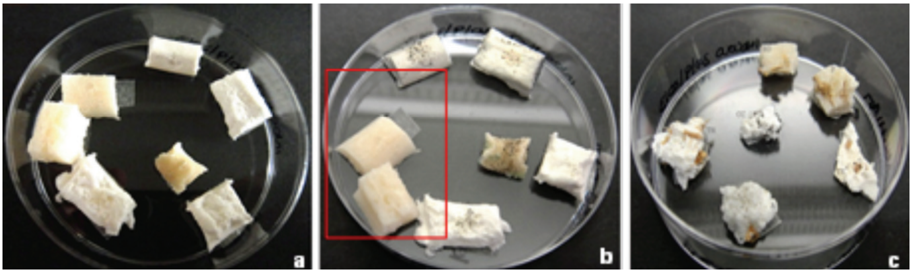

Figure 7. Microbiological assay n.1. No artificial aged samples (a) before incubation and (b) after 14 days, Aspergillus colonies were developed. Inside red square: acrylic resin stuccos; c) Aspergillus spp. growing on aged samples.

For microbiological assay n.2, no visible microbial growth was detected on the cellulose samples. On the rabbit glue samples, after 7 days we observed the presence of *Aspergillus* colonies and after 14 days we also detected *Penicillium* colonies, in addition to bacterial growth (Figure 8). Those samples containing both materials (i.e. stucco) were characterized by a slower growth (Figure 9) of *Aspergillus* and *Penicillium* colonies [9].

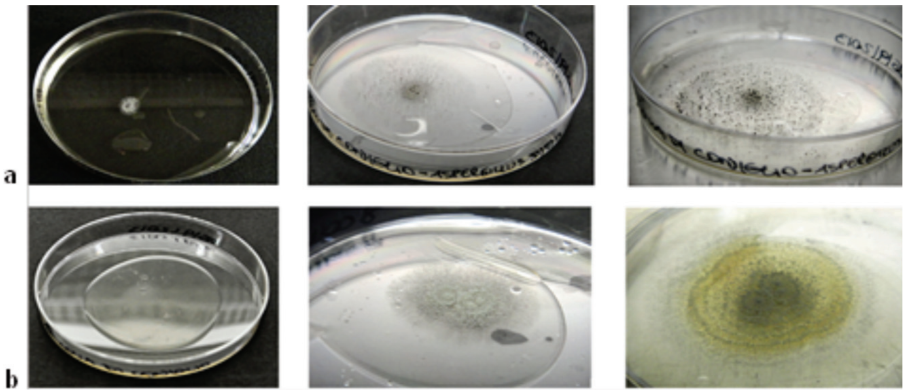

Figure 8. a) Monitoring of Aspergillus spp. growth in adhesive from 7 days to 21 days; b) monitoring of Penicillium spp. growth in adhesive from 7 days to 21 days.

These results highlight the susceptibility of glue to microbial colonization (due to its protein nature), while the cellulose was not subject to biodegradation.

No biological growth was detected in the 18th century spruce girder samples after 14 days and until today. The absence of microbial colonies is probably due to the absorption of humidity that let the sample dry (Figure 10).

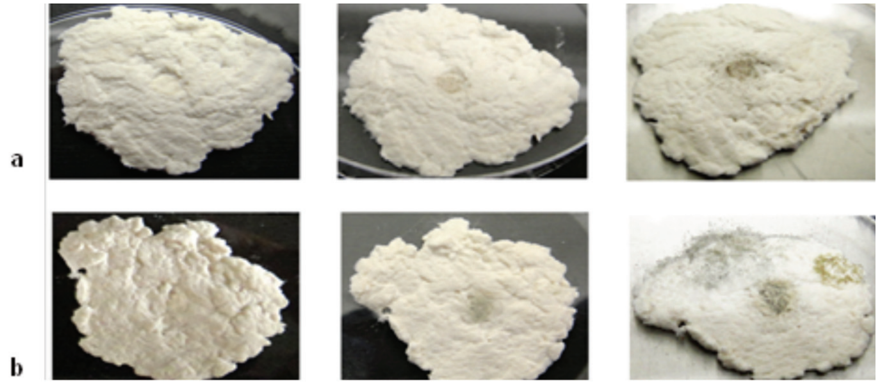

Figure 9. a) Monitoring of Aspergillus spp. growth on stucco sample (cellulose + adhesive) from 7 days to 21 days; b) monitoring of Penicillium spp. growth on stucco sample (cellulose + adhesive) from 7 days to 21 days.

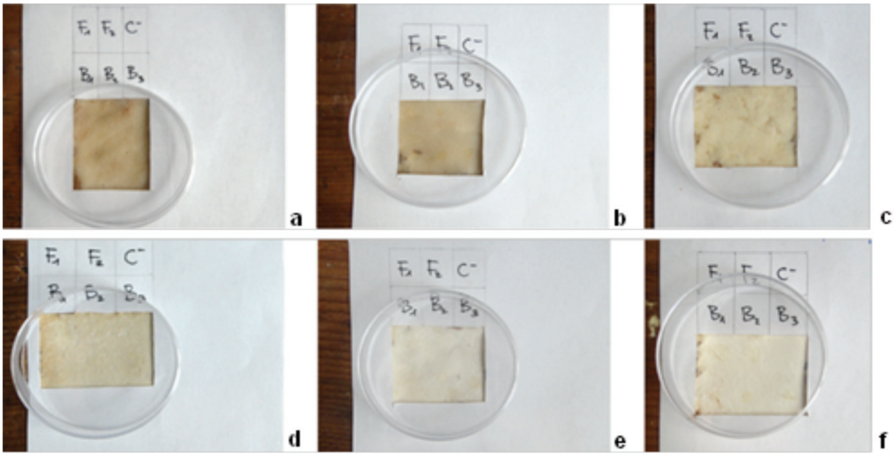

Figure 10. Stuccos based on cellulose and different adhesives used for wood reintegration, inoculated with microbial suspension: a) Plextol B500 (pure); b)BEVA D8 (pure);c) Aquazol 200 (25% in EtOH); d) Rabbit glue (1:14); e) Tylose (5% in H2O); f) Tylose (5% in H_2O/EtOH [1:1]). F1 Aspergillus spp.; F2 Penicillium spp; B1 Bacillus spp.; B2 Micrococcus spp; B3 other bacterium; C-control.

The microbiological tests focused on the microbial susceptibility of cellulose-based stuccos. The cellulose does not influence microbial colonization, which depends only on the adhesive utilized to make the stucco. Evidently, adhesives based on protein materials, such as rabbit glue, are more susceptible to microbial colonization than acrylic ones. Moreover, the wooden tablets were not characterized by microbial colonization. This represents an advantage if the stucco is utilized in reintegrating parts of an artwork exposed in a museum, with optimal thermo-hygrometric conditions (Table 4).

Table 4. Evaluation of microbial growth.

Material + Microbial inoculum		1st week	2nd week	3rd week
CELL	CELLULOSE + F1	0	0	0
	CELLULOSE + F2	0	0	0
	CELLULOSE + B1	0	0	0
	CELLULOSE + B2	0	0	0
RB	RABBIT GLUE + F1	1	3	3
	RABBIT GLUE + F2	0	2	3
	RABBIT GLUE + B1	0	1	1
	RABBIT GLUE + B2	0	0	1
CELL+RB	STUCCO (CELL+RB) + F1	0	1	1
	STUCCO (CELL+RB) + F2	0	1	1
	STUCCO (CELL+RB) + B1	0	0	0
	STUCCO (CELL+RB) + B2	0	0	0

0-No growth; 1- trace of growth <10%; 2- low growth 10-30%; average growth 30-60%; high growth (> 60%). F1- Asperigillus; F2- Penicillium; B1- Micrococcus; B2- Bacillus

4. Conclusions

An important goal of this study is to demonstrate that aged cellulose pulp is not more susceptible to microbial colonization than new cellulose pulp. Another important aspect is that the microbial growth is attributable to the presence of the adhesive product.

Some stuccos, such as rabbit glue and ether-based cellulose, have good superficial qualities, such as a good response to color. Acrylic-based stuccos, which are more compact and heavier, are recommended when the support is friable.

Cellulose-based stuccos represent a valid alternative to commercial ones: cellulose has a lower elastic module than wood, avoiding the “wedge effect” in the structure, it is easy to work mechanically and can be utilized as a superficial stucco thanks to its high compatibility with the material.

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Biographical notes

Fulvia Bartolone, Restorer of paintings and wooden sculpture. Master's Degree in Conservation and Restoration of Cultural Heritage at the University of Palermo, awarded with 110 cum laude. She has worked in the Diocesan Museum of Palermo, the Pepoli Museum of Trapani and churches and museums in different cities in Italy and Europe. In 2015 she worked as restorer for the scientific collections of the University of Palermo. In the same year she won the fellowship "*500 Giovani per la Cultura*" of the Ministry of Cultural Heritage and Activities and Tourism (MIBACT) to work in the Historical Archive of Palermo.

Mauro Sebastianelli is a restorer. He trained at the Central Institute for Restoration in Rome. Since 1993 he has carried out maintenance, preservation and restoration work both at home in Italy and abroad. Since 2003, he has taught in the field of conservation and restoration, for the educational service of the Superintendence dedicated to the Roman network of Museums, at the educational workshop in the Galleria Borghese in Rome. Since 2004 he has been a consultant for the conservation and restoration of the Archdiocese and the Diocesan Museum of Palermo and since 2007, the Regional Museum of Palazzo Mirto. Since 2007 he has also been a contract Restorer with teaching activities on the degree course in Conservation and Restoration of Cultural Heritage at the University of Palermo.

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Bartolomeo Megna is a senior researcher of Science and Technology of Materials at the DICAM Department of Palermo; he has always been interested in the theme of conservation of cultural heritage; he has a PhD with a thesis on materials for the recovery of wooden artifacts.

Summary

Cellulose pulp, mainly utilized as a support for cleaning stone material, in this study, is proposed as a filler for wooden supports. In the last 10 years, pulp has been used in many restoration works carried out in the laboratory of the Diocesan Museum (MDP) in Palermo, including both wooden sculptures and panel paintings. These artworks, exhibited in the MDP, are constantly monitored in order to evaluate the effectiveness of cellulose pulp as a filler, assessing its durability and bio-receptivity. In particular, structural characteristics, drying time, shrinkage, loss in weight, affinity to the pigments, etc., were simultaneously evaluated. Tests were also carried out on artificially aged cellulose pulp samples, in order to appraise if cellulose could be a source of nourishment for microbial growth.

Riassunto

Il presente studio propone l'analisi e l'approfondimento di un materiale già noto nel campo del restauro: la polpa di cellulosa, utilizzata da sempre come supportante per impacchi di pulitura su materiale lapideo, qui analizzata in veste di stucco per supporti lignei. Il materiale è stato utilizzato in alcuni interventi di restauro eseguiti nel laboratorio del Museo Diocesano di Palermo nel corso degli ultimi 10 anni su sculture lignee e dipinti su tavola. Le opere conservate in sale espositive costantemente monitorate hanno permesso, fino ad oggi, di valutare l'efficacia dell'intervento. Lo studio propone alcuni test eseguiti su campioni di stucco di cellulosa realizzati con leganti di origine diversa, valutando le caratteristiche di ciascuna tipologia, i tempi di essiccamento, ritiro, perdita in peso, affinità al colore, etc. Inoltre sono stati effettuati test d'invecchiamento, reversibilità e test microbiologici per determinare la resa della cellulosa all'invecchiamento e valutare se il substrato in cellulosa possa costituire un supporto alla colonizzazione microbica.