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Abstract

Bread-made artifacts are the products of an ancient creative tradition in some parts of Sardinia. While early objects were intended for pagan events (e.g., grain harvest), later artworks were specifically made for Catholic celebrations such as Christmas and Easter. Unfortunately, the cultural heritage linked to these objects is endangered by Sitophilus granarius (L.), a tiny insect that causes irreparable damage such as ruptures and galleries through an intense boring action. In this work, we have evaluated the potential for the coating technology to protect bread-made artifacts from entomatic attack. Within this scope, a nanocomposite coating and an active coating were prepared, and the coated objects were characterized in terms of optical, mechanical, and insect-resistant properties. Overall, the deposition of the coating did not negatively impact the appearance of the objects, although some differences were detected instrumentally in terms of color and gloss. In addition, both coating formulations decreased the Young's modulus of the samples subjected to a flexural test, which was attributed to the plasticizing effect of the poly-methyl methacrylate and deltamethrin. The entomatic tests revealed that the nanocomposite coating was the most effective for preventing the wheat weevil attack, with no damages detected on the samples and high mortality of the insects due to hunger. The approach proposed here can be successfully extended to other art objects (e.g., museum collections) susceptible to insect attacks.

Keywords	active coating; bread artifacts; entomatic attack; nanocomposite coating; Sardinia
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Corresponding Author	Stefano Farris
Corresponding Author's Institution	University of Milan
Order of Authors	Carlo Alessio Cozzolino, Cesare Rovera, Claudia Anchisi, Sara Savoldelli, Gaetano Campanella, Eugenio Muroni, Massimo Meloni, Stefano Farris
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Graphical Abstract

Preservation of bread-made museum collections by coating technology

Carlo A. Cozzolino[a], Cesare Rovera[a], Claudia Anchisi[a], Sara Savoldelli[a], Gaetano Campanella[a],

Eugenio Muroni[b], Massimo Meloni[b], Stefano Farris[a]*

[a]*DeFENS, Department of Food, Environmental and Nutritional Sciences, University of Milan, Via Celoria 2 – 20133 Milan, Italy*

[b]*PROLOCO Olmedo, Via Europa 20 – 07040 Olmedo (Sassari), Italy*

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*Corresponding author. Tel.: +39 0250316654; Fax: +39 0250316672

E-mail address: stefano.farris@unimi.it (S. Farris)

ABSTRACT

Bread-made artifacts are the products of an ancient creative tradition in some parts of Sardinia. While early objects were intended for pagan events (e.g., grain harvest), later artworks were specifically made for Catholic celebrations such as Christmas and Easter. Unfortunately, the cultural heritage linked to these objects is endangered by *Sitophilus granarius* (L.), a tiny insect that causes irreparable damage such as ruptures and galleries through an intense boring action. In this work, we have evaluated the potential for the coating technology to protect bread-made artifacts from entomatic attack. Within this scope, a nanocomposite coating and an active coating were prepared, and the coated objects were characterized in terms of optical, mechanical, and insect-resistant properties. Overall, the deposition of the coating did not negatively impact the appearance of the objects, although some differences were detected instrumentally in terms of color and gloss. In addition, both coating formulations decreased the Young's modulus of the samples subjected to a flexural test, which was attributed to the plasticizing effect of the poly-methyl methacrylate and deltamethrin. The entomatic tests revealed that the nanocomposite coating was the most effective for preventing the wheat weevil attack, with no damages detected on the samples and high mortality of the insects due to hunger. The approach proposed here can be successfully extended to other art objects (e.g., museum collections) susceptible to insect attacks.

Keywords: active coating; bread artifacts; entomatic attack; nanocomposite coating; Sardinia

1. Introduction

Italian cultural heritage not only includes famous artworks that are glorified worldwide, but also a huge number of local creations that are largely unsung. However, the existence of these local creations is of utmost importance, especially from a local perspective. In fact, they reflect the tradition of a territory, and have kept the historical memory alive over the years. In addition, the survival of these local activities has an economic relevance for the domestic communities, because these creations, often grouped in small museum collections, are tourist attractions. Examples of minor artworks include laceworks, weavings, rugs, basketry, paintings, and pottery. All of these creations, although widespread across the entire Italian territory, can be found much more easily in those regions that, due to either geographical or cultural reasons, still have a well-rooted identity and a strong link with the past. Sardinia is one example that brings together ancient traditions and renowned handicrafts.

Among the large number of artworks, bread-made collections represent a peculiar type of Sardinian artwork that is widespread in some areas of the region. Local communities are engaged in the manufacturing of any sort of handicraft using bread dough, which, due to its plasticity, allows for the production of amazing art objects. In most cases, the production of bread-made artifacts is linked to specific periods of the year, either to celebrate religious festivities (e.g., Christmas, Easter, etc.) or social events (e.g., the grain harvest). Examples of these artworks are shown in Figure 1. As with many local handicrafts, bread-made artworks are at risk of disappearing. Although social habits play a role in this (e.g., the youngest are less prone to perpetuating these traditions), the main reason for such risk is not directly related to humans, but rather to stored-product insects.

The wheat weevil, or *Sitophilus granarius* (L.) (Coleoptera, Dryophthoridae), is a common pest in granary goods [1]. The females lay many eggs inside the wheat matrix, and the larvae (white with a tan head) feed and grow until pupation, eating the immediate surroundings using robust chewing mouthparts. The adults (reddish-brown color, 3–5 mm long) emerge from the inside the wheat matrix by drilling a hole with their elongated snouts. The life cycle takes from 5 to 15 weeks, depending on the season and local temperatures, whereas the adults can live up to 8 months after leaving the food [2]. Wheat weevils' attack on the bread-made artifacts cause severe damage, especially due to the action of the larvae. Broken parts and tiny galleries inside the artwork represent irreparable aesthetic issues that also impair the mechanical stability. As a result, each piece keeps its integrity only for few months—one year in the best cases. For this reason, local artisans must set the bread-made collections every year in order constantly to replace the damaged objects. In addition to being a time-consuming process, it does not allow for the preservation of the oldest pieces, causing the subsequent loss of cultural and historical heritage objects. Therefore, finding a solution to the entomatic attack of the grain weevil is highly necessary. Although prevention and monitoring are, in principle, the most effective approaches to prevent the attack, they are not feasible in all circumstances. Cleaning, even if thorough, is not enough to avoid the pest, and monitoring by pheromone traps is not affordable by many. Hence, strategies for treating the final artwork can be a complementary tool for efficiently preventing insect attack and, thereby, for preserving the admirable handwork of artisans. Although the use of protective boxes made of glass or plastic polymers (e.g., polycarbonate) possibly in combination with a controlled atmosphere was considered as a valid option, the negative impact on the visitors' in terms of overall perception of the artifacts boosted us to look for a less invasive strategy. The use of protective layers developed according to nanotechnology approaches is an established strategy to preserve our cultural heritage [3,4].

In this work, we propose for the first time the use of a coating technology to protect handmade artworks made of bread dough against the attack of the wheat weevil. Two multidisciplinary approaches are presented: on one hand, a nanocomposite coating has been conceived as a "passive" physical barrier against the boring action of the insect, thus preventing the eggs deposition; on the other hand, an "active" coating with insecticide activity has been designed as a chemical shield around the object. Although restricted to a specific regional context, we believe that the findings arising from this work can be extended to a number of similar applications where damages from entomatic attack represent the main cause of the deterioration of the artwork, eventually leading to its loss.

2. Materials and methods

2.1. Materials

2.1.1. Bread samples

Rectangular specimens (15 × 10 cm^2) were prepared by the artisans of the tourist association (Pro Loco) of Olmedo (Sassari, Italy) using the same wheat dough that is used to manufacture of the handmade artworks. A metallic template was used to print the final shape on the dough. Once obtained, the specimens were stored at room temperature for 24 hours and then transferred to a desiccator for two additional weeks before the experiments. The residual moisture content, which was measured with a moisture analyzer HB43–S (Mettler Toledo, Greifensee, Switzerland), was 1.42 ± 0.35% and the average thickness was 0.8 ± 0.04 mm. Three-dimensional objects were also prepared to simulate the artworks in the museum collections. These samples were used for the entomatic tests.

2.1.2. Chemicals

Methyl methacrylate (MMA) and sodium sulphate were purchased from Sigma–Aldrich (Milano, Italy). Altuglas® BS 520 (a poly-methyl methacrylate-*co*-ethyl acrylate copolymer) was kindly provided by Altuglas International (Rho, Italy) in the form of round beads that were 150–200 μm in diameter. Commercial wheat gluten (WG) powder was kindly supplied by Lantmännen Reppe AB (Växjö, Sweden). According to the supplier, the gluten protein content was 77.7 wt% (dry basis) (modified NMKL Nr 6, Kjeltec, N×5.7, www.NMKL.org), the moisture content was 6.9 wt% of the total weight (NMKL 23, 1991), and the starch content was 5.8 wt% (dry basis) (Ewers, polarimetric method). The concentration of fats was 1.2 wt% (dry basis) (Soxtec, Lidfett.OA.19, tecator AN 301), and the residual ash content was 0.9 wt% (dry basis) (NMKL 173 2nd ed). The wheat gluten particle size distribution was as follows: 10% between 160 and 250 μm, 20% between 100 and160 μm, 55% between 50 and 100 μm, and 15% less than 50 μm [5]. Cloisite® 20A (Southern Clay Products Inc., Rockwood Additives, Gonzales, TX), a natural montmorillonite modified with a quaternary ammonium salt, was used as the nanobuilding block (NBB) for the nanocomposite coating preparation. In particular, the use of these nanoparticles was primarily aimed to modulate the optical properties of the final artifacts (more specifically, depressing the high gloss provided by the PMMA), although a synergistic effect with PMMA to prevent the drilling action of the wheat weevil was also expected. According to the supplier, the particle size was as follows: 10% less than 2 μm, 50% less than 6 μm, and 90% less than 13 μm; the density ρ was 1.7 g cm^{-3}, whereas the *d*-spacing was 2.42 nm. K-Othrine® 7.5 (Bayer CropScience), a pyrethroid insecticide for civilian use based on deltamethrin, was selected as an active component due both to its efficacy against weevils and to its long-lasting action. Anhydrous acetic acid (purity: 98%) was purchased from Sigma-Aldrich (Milano, Italy). Milli-Q water (18.3 MΩ cm) was used during the preparation of the WG-based coating formulation.

2.1.3. Experimental insects

Experiments were performed in 2015 at the University of Milan in the entomological laboratory. Unsexed, mixed-age adults that were 1-2 weeks old were used in all tests. They were obtained from a population of *Sitophilus granarius* that emerged in the laboratory from whole wheat and had with no history of exposure to insecticides. This population was reared on wheat at 26 ± 1 °C and 65 ± 5% relative humidity (RH).

2.2. Methods

2.2.1. Coating preparation and deposition.

Two coating formulations were prepared, coded as coating *F1* and coating *F2*, whereas the uncoated samples (control) were coded as *F0*.

For the preparation of the coating *F1*, a solution of poly-methyl methacrylate (PMMA) in its monomer (MMA) at a concentration of 30.3 wt% by weight was first prepared. Separately, 2 g of Cloisite® 20A were dispersed in 20 mL of MMA using an UP400S ($power_{max}$ = 400 W; frequency = 24 kHz) ultrasonic device (Hielscher, Teltow, Germany) equipped with a cylindrical titanium sonotrode (mod. H14, tip Ø 14 mm, $amplitude_{max}$ = 125 μm; surface intensity = 105 W cm^{-2}) under the following conditions: 0.5 cycles and 50% amplitude, for 10 min. Ultrasonication has been demonstrated to be a powerful approach in the top down generation of nanoparticles, such as exfoliated plates of MMT [6]. This physical process generates sound waves (more frequently, ultrasound waves), which promote deagglomeration and reduction in size of the particles as a result of the mechanical effects of the phenomenon called cavitation, i.e., the formation, growth, and implosive collapse of bubbles in a liquid [7]. The MMA-nanoclay dispersion was then gently added to the PMMA solution to obtain the final PMMA nanocomposite dispersion.

The coating *F2* was prepared according to the procedure by Türe et al. [8], with some modifications: 7 g of wheat gluten and 0.02 g of sodium sulfite (reducing agent) were added to 23 g of distilled water under magnetic stirring at 500 rpm to prevent the wheat gluten powder from lumping. Afterwards, acetic acid was added dropwise to adjust the pH value of the solution to 4 and hence to achieve complete solubilization of the wheat gluten. Finally, 5 g of the insecticide (K-Othrine® 7.5) was added to the main wheat gluten water dispersion, eventually leading to a final concentration of 0.1 wt% of deltamethrin.

The deposition of the coating dispersions *F1* and *F2* on the 2-D and 3-D samples was carried out by dipping. In particular, five dipping cycles were carried out for the formulation *F1*, whereas only one dipping cycle was performed for the formulation *F2*. Each coating deposition was followed by a drying cycle in which a constant and perpendicular flux of mild air (25.0 ± 0.3 °C for 2 min) was applied at a distance of 40 cm from the specimen. After this, dry coatings of nominal thickness of ~ 2.0 and 5.0 μm for the coating formulations *F2* and *F1*, respectively, were obtained. After the last drying cycle, samples were stored in a desiccator for an additional 1 week before analysis.

2.2.2. Color measurement.

The color of the 2-D samples was measured at room temperature using a reflection colorimeter (Minolta Chroma Meter mod. CR 210, Osaka, Japan) with a measuring surface area of 23.75 cm^2. The *CIE* (Commission Internationale de l'Èclairage) $L^*a^*b^*$ colorimetry system [9] was used for the color quantification of the sample surface, using D65 illuminant/10° observer. L^* represents the degree of lightness of a color, and its values range from 0 (an ideal black object) to 100 (an ideal white object); a^* represents redness/greenness of a color (positive values of a* represent red, whereas negative values of a^* represent green); and b^* represents yellowness or blueness, with positive values of b^* representing yellow and negative values of b^* representing blue [10,11]. The final data are the average of at least five replicates.

2.2.3. Gloss measurement

Gloss is the percentage of incident light that is reflected at an angle equal to the angle of the incident light (e.g., 45°) [12]. It corresponds to the specular reflectance of a surface. From a practical point of view, gloss indicates how well a surface reflects light in a specular (mirror-like) way. The higher the surface reflectance, the higher the gloss. A surface with specular reflectance approaching zero is said to be matte. The factors that affect gloss are surface topography (roughness and irregularities) and the possible presence of scratches. In this work, the determination of the gloss aimed to quantify any possible variation in the coated surfaces from the original value of the uncoated samples. This was a specific requirement of the Sardinian museum in order to preserve the matte appearance of the statues, thereby avoiding any potential appearance of manipulation.

Gloss measurement was performed using a UV-VIS spectrophotometer (Lambda 650, PerkinElmer, Waltham, MA, USA) coupled with a 150 mm integrating sphere, which allows for of the diffuse transmitted light to be trapped. Specular reflectance (R_s) was obtained after measuring the total reflectance (R_t) and the diffused reflectance (R_d), according to the following equation:

$$R_s\,(\%) = R_t - R_d \tag{1}$$

All of the reflectance values were measured at a wavelength of 550 nm, namely the wavelength representing the maximum sensitivity of the human eye. The final data are the average of five replicates.

2.2.4. Mechanical analysis

A three-point bending flexural test was carried out on 2-D samples to evaluate their ability to resist deformation under a load. Within this scope, a Zwick Roell dynamometer (model Z005, Ulm, Germany) fitted with a 100 N load cell was used. One 2-D sample at a time was placed on two supports (each one equipped with an 8.0 mm-diameter horizontal rod at the tip) separated by a distance of 10 cm. A third compressing bar, also equipped with an 8.0 mm diameter horizontal rod at the tip, was driven down between the two supports at a speed of 10 mm min^{-1}, bending each specimen until it snapped. Two parameters were gathered from the tests by a software-assisted procedure (TestXpertV10.11 Master), namely the modulus of elasticity (E_f) and the flexural stress (σ_{max}) defined by the following equations, respectively:

$$E_f = \frac{L^3 m}{4bd^3} \tag{1}$$

$$\sigma_{\max} = \frac{3FL}{2bd^2} \tag{2}$$

where: L is the support separation (mm), m is the gradient (i.e., slope) of the initial straight-line portion of the load deflection curve (N mm^{-1}), F is the load at a given point on the load-deflection curve (N), and b and d are the width and the height of the specimen, respectively. For each parameter, the final results are the mean of five replicates.

2.2.5. Transmission electron microscopy (TEM)

TEM images were captured to visualize the extent of deagglomeration and exfoliation of the Na$^+$-MMT clays after the ultrasonication treatment. To this purpose, 5 μL of a 0.5 wt.% water dispersion were deposited onto a Formvar-coated Cu grid (400 mesh). Observations were made after 24 hours (i.e., the time required to allow solvent evaporation) using an LEO 912 AB energy-filtering transmission electron microscope (EFTEM) (Carl Zeiss, Oberkochen, Germany) operating at 80 kV. Digital images were recorded with a ProScan 1K Slow-Scan CCD camera (Proscan, Scheuring, Germany).

2.2.6. Entomatic attack test

The resistance of the bread made samples against the *S. granarius* was evaluated by placing 4 replicates of each sample (*F0*, *F1*, and *F2*) in a closed jar with a ventilated lid at 26 ± 1 °C and 65 ± 5% RH. Each jar contained 20 unsexed, 1-2 week-old starving adults, which were in contact with the specimens for 40 days. At the end of this period, the efficacy of the coating was assessed by visual inspection—that is, by observing the presence of mechanical damages (perforations, breakages, etc.) as well as the presence of living insects.

2.2.6. Statistical analysis

Statistical significance of differences between mean values was determined by one-way analysis of variance (ANOVA) using Statgraphic Plus 4.0 software. The mean values, where appropriate, were compared by Student's *t*-test with a significance level of $p < 0.05$.

3. Results and discussion

3.1. Colorimetry analysis

The data obtained from the colorimetry analysis are displayed in Table 1. The statistical analysis showed that there was no statistically significant difference between the samples *F0*, *F1*, and *F2* in terms of brightness (L^*), whereas the deposition of the coating led to a significant increase of the a^* parameter (i.e., a subtle shift toward red was observed). Concurrently, the samples coated with the formulation denoted as *F2* experienced a slight increase of b^* (yellowness), which can be attributed to the gluten component in the formulation. However, it is worth pointing out that these instrumental differences did not correspond to any appreciable differences to the naked eye.

3.2. Gloss measurement

The results of the spectrophotometric determination of gloss at 550 nm are reported in Table 1. The bare sample (*F0*) can be considered totally opaque (the negative value is within the sensitivity of the instrument), whereas the sample *F1* was slightly glossy, which is linked to the inherent optical features of PMMA. However, it should be noted that the inherent high gloss of PMMA was depressed due to the presence of the clays dispersed in the main polymer matrix. In the absence of clays, the coated samples were high glossy, which would have made the use of PMMA unsuitable for the targeted application. An example of PMMA-coated artifact is displayed in Figure 2. The sample *F2* had an R_s value in between. It is worth noting that significant differences between samples can be only detected instrumentally. As discussed above for the color analysis, the three samples were deemed equally opaque, with no appreciable differences to the naked eye.

3.3. Mechanical analysis

The flexural properties of the 2D bread-made samples were evaluated by a three-point bending test. The coating deposition induced a decrease in the elastic modulus compared to the bare (uncoated) sample, especially for the formulation *F1* (Table 1). This observation can be explained by the plasticization effect of the polymeric PMMA, which very likely percolated in the gluten-based matrix of the specimens. The same trend, although to a lesser extent, was observed for the coating formulation *F2*, in which the decrease of Young's modulus could be attributed to the plasticizing effect of deltamethrin, which somehow compensated for the inherent stiffness and rigidity of the gluten. As for the flexural stress (i.e., the maximum stress borne by the samples during the test), it can be noticed that the deposition of the PMMA-based coating led to an increase of $\sigma_{\max}$. This result supports the finding that the *F1*-coated specimens were more flexible compared to both the bare and *F2*-coated samples, and thus they were able to withstand more stress during bending.

3.4. Entomological test

The efficacy of the coating against the attack of the *Sitophilus granarius* (L.) was tested on 3-D samples in order better to simulate the real exposure of the artifacts (e.g., statues) to insect attack. Figure 3 provides a visual summary of the entomological test carried out on the bare objects, and on the same objects coated with the coatings denoted as *F1* and *F2* (Figure 3d). The uncoated samples clearly showed tiny channels and small bites (Figure 3a), indicating the drilling action inside the sample (by larvae) and intense chewing activity from the outside of the sample (by adults), respectively. Moreover, the mortality of the insects was extremely low, meaning that almost all of the adults were still active, as clearly shown in Figure 3b. The deposition of the coating formulation

F1 provided the samples with total protection against the wheat weevil, as demonstrated by the absence of any kind of mechanical damage to the object (Figure 3c). In addition, the initial population of the insects was strongly affected in terms of mortality, supporting the fact that the PMMA-montmorillonite nanocomposite coating acted effectively in preventing the perforations made by the insects. Not only the inherent hardness of PMMA, but also the impermeable nature of the individual MMT sheets contributed to preserve the bread made objects. As can be seen in Figure 4, ultrasonication allowed the obtainment of individual platelets from the original macro-size MMT particles. Each platelet dispersed in the polymer can be thereby thought as a reinforcing "brick" acting a physical barrier against the insects, in synergy with the main PMMA polymer matrix. The active coating based on gluten and deltamethrin (Figure 3d) was likewise effective in counteracting insects attack. However, in this case we noticed subtle scratches on the sample surface, probably attacks attempted by the insects. Because these light damages were located in the areas of the 3-D samples that were less accessible to the coating dispersions, it is likely that the amount of the insecticide in these parts of the object was insufficient for keeping the insects away. In addition, it should be recalled that the deposition of the coating *F2* took place in only one pass, whereas the coating *F1* was laid on the 3-D sample in five passes.

4. Conclusions

This work demonstrated that coating is a suitable and feasible approach for tackling the long-standing issue of the entomatic attack on bread-made artifacts that widely affects museums and collections of Sardinia. The development of sophisticated coating formulations has been shown to be the key for the successful protection of the samples. In particular, the combination of polymer science and nanotechnology knowledge provided the samples with a super-resistant surface that was able to stand the drilling action of the insect. Concurrently, the development of an active coating with insecticide features has been proven a potential solution for deterring the wheat weevil, though some improvement is needed (e.g., increasing the number of coating depositions). Interestingly, neither the optical nor the mechanical properties of the coated samples were worse after the coating deposition. Next investigation should consider the long-term performance of the coatings, especially in terms of optical properties, which might change due to the exposure to the surrounding environment. The findings arising from this work can be profitably exploited for other applications that are beyond the restricted and local example of the bread-made objects from Sardinia.

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Figure 1. Examples of bread-made artifacts prepared for Easter (a) and Christmas time (b and c).

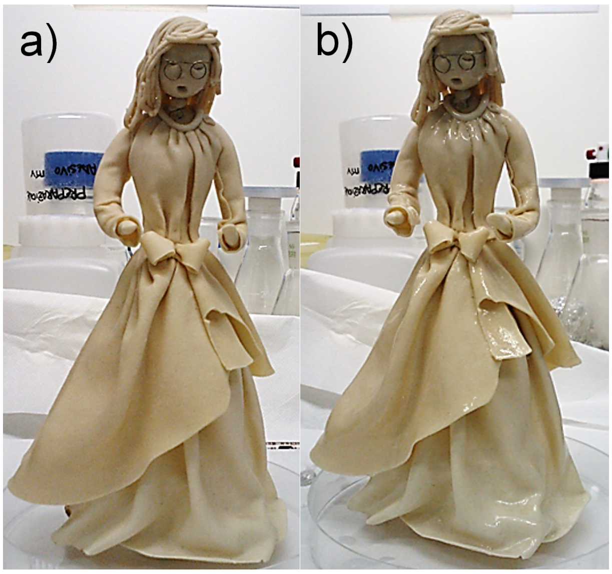

Figure 2. Bread-made artifact before (a) and after (b) the deposition of a pure PMMA coating (no nanoclays were added to the formulation). The high-gloss surface of the coated sample is clearly observed on the right.

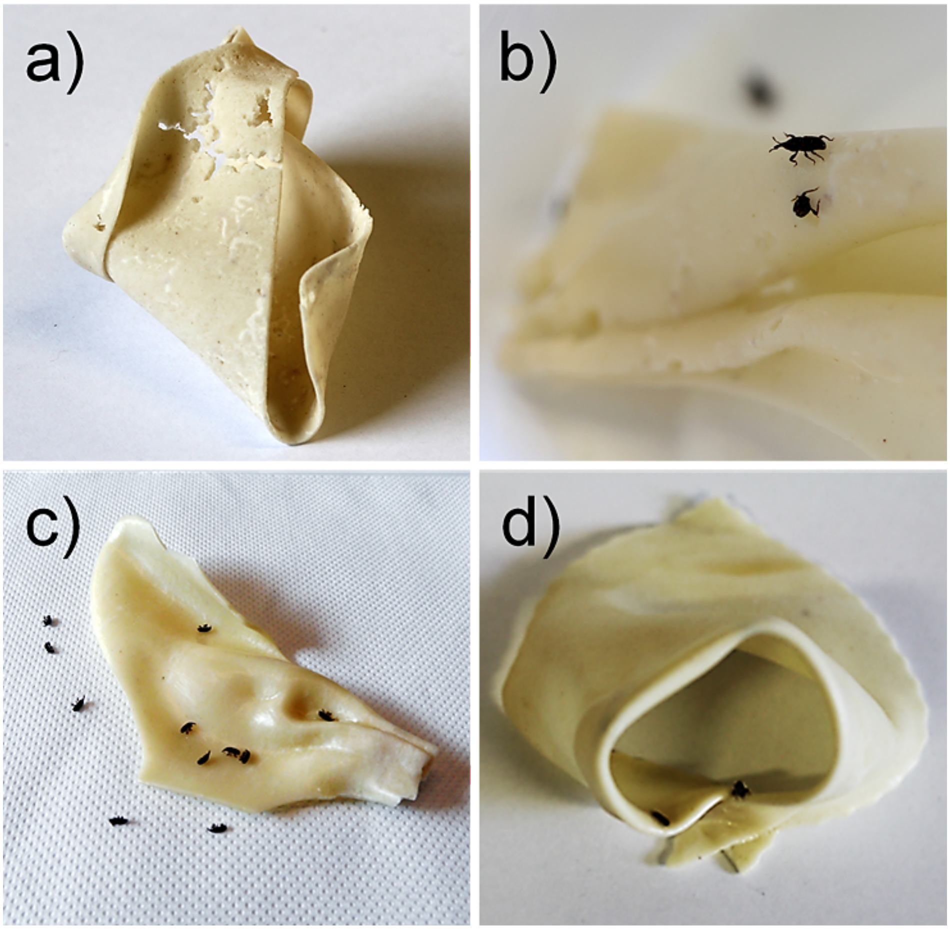

Figure 3. Visual outcome of the entomatic test on the bare (uncoated) samples (a and b) and coated samples F1 (c) and F2 (d).

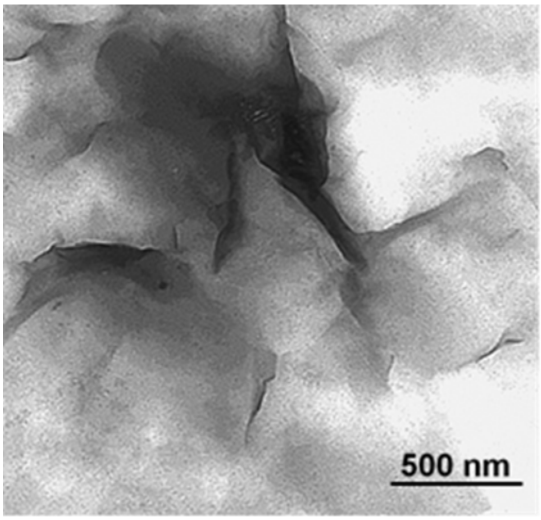

Figure 4. TEM image of a MMT water dispersion (0.5 wt.%) after ultrasonic treatment (10 min, 0.5 cycles, and 50% amplitude). Individual MMT sheets randomly oriented can be seen as darker regions.

Table 1. *CIE* $L^*a^*b^*$ color coordinates, reflectance, and mechanical parameters of uncoated (*F1*) and coated (*F2* and *F3*) 2-D samples.

Sample	L^*	a^*	b^*	R_t (%)	R_d (%)	R_s (%)	E_f (MPa)	σ_{max} (MPa)
F0	83.34^{a} (± 0.54)	-1.99^{b} (± 0.17)	17.06^{d} (± 0.67)	52.26^{a} (± 1.27)	53.61^{ab} (± 2.08)	-0.09^{a} (± 0.02)	232.46^{ab} (± 174.62)	6.38^{c} (± 1.76)
F1	82.82^{a} (± 0.42)	-1.74^{c} (± 0.14)	17.71^{de} (± 0.59)	55.28^{b} (± 2.31)	54.59^{c} (± 2.46)	0.69^{b} (± 0.18)	158.66^{a} (± 52.99)	4.92^{c} (± 0.78)
F2	82.88^{a} (± 0.23)	-1.59^{c} (± 0.11)	18.34^{e} (± 0.32)	53.61^{ab} (± 2.08)	53.22^{c} (± 2.14)	0.39^{c} (± 0.17)	327.19^{b} (± 106.99)	6.56^{c} (± 1.43)

Results are expressed as mean values and standard deviation. Different superscripts within a group (i.e., within each parameter) denote a statistically significant difference ($p < 0.05$).

- The grain weevil attack is a long-standing issue for bread-made museum collections
- Coating technology was evaluated as a potential approach to face this problem
- A nanocomposite coating and an active coating were developed and tested
- Coating deposition did not impair the appearance of the original samples
- The nanocomposite coating was more effective in preserving the bread-made artifacts