Scanning electron microscopy in monitoring the aging of alternative materials for plastering of canvas manufactured products

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Summary
During the restoration of manufactured canvas, the plastering of recoverable lacunae is generally carried out by means of dehydrated gesso, rabbit-skin glue or, more rarely, by using already made up products. Being gesso-animal glue mixture totally comparable with canvas constitutive materials, it must be considered the strong drying correlated shrinkage, the poor mechanical resistance and the possibility to be a substrate for deteriorating biological agents, in particular environmental conditions. Similarly, ready-to-use products, such as Modostuc and Polyfilla, undergo a dramatic shrinkage and show a poor mechanical resistance. Aquazol®, BEVA® gesso and Balsite®, new products commonly used for different purposes, have been here analyzed. Their behavior has been studied by careful light microscopy observation of their chromatic changes and by scanning electron microscopy of their surface details. An artificial aging condition was also induced to better characterize their response to the environmental damage progression. The performed studies on these materials provided very positive results and their potential use in the place of common use fillers has been suggested.

Key words: heritage, restoration, aging, canvas, lacunae filling.

Introduction
Materials used for lacunae filling in textile paintings must be closely comparable with constituents, in terms of chemical composition, plasticity, hardening speed, elasticity, painting appropriate surface and progressive shrinkage. Moreover, they must show a relative resistance to possible mechanical damage, microbial contamination, aging or sun radiations.

Currently few materials, considered appropriate for lacunae filling, are available and animal glue, Modustic and Polyfilla are now commonly utilized. The use of a filling plaster, composed by a proteinaceous binder and an inert (rabbit-skin glue-gesso), has been now also consolidated in the common practice. Its characteristics are indeed closely comparable to manufactured product ones. Nevertheless, in the last years it has been demonstrated to be less flexible and more susceptible of microorganism contaminations (Fuster-Lopez et al. 2006; Appolonia et al. 2009, 2010; Coppola et al. 2011).

In this study some relatively new products have been studied by scanning electron microscopy (SEM) in basic control condition and after the experimental laboratory induction of an artificial accelerated aging.

Materials and Methods
Materials
In this study, we have analyzed Aquazol®, BEVA® gesso and Balsite®, products generally used for different purposes (adhesive or fillers), even if in the same field of art manufactured prod-
ucts. In previous studies such substances had been set up on textile supports to submit them to specific tests of workability of the fillers, dimensional shrinkage, suitability to the reintegration painting and varnishing, mechanical strength, reversibility and finally resistance to the most common biodeteriogens agents (Calore et al. 2011; Viti et al. 2011; De Luca et al. 2012, 2013).

Sample aging conditions

Critical conditions of temperature and humidity have been experimentally applied to the materials. For that A Solar Box 1500e RH, (Erichsen, Italy), purchased from Erichsen (Germany), was used for the artificial aging (A). The aging conditions were as follows: 50°C, excitation with a Xenon lamp (wavelength 280-400 nm), radiation power 650W. The behavior of the materials has been studied after three weeks of warm/moist conditioning cycles (90% RH for 72h), alternated with 144 h at 50% atmospheric humidity. A Soda-lime glass UV filter was used to simulate indoor exposure.

SEM analysis

The dimensional shrinkage of tested products has been evaluated by means of SEM (Philips 515), with the aim to study surface changes after aging chamber.

To exclude alterations due to technical procedures, no critical point drying, nor gold-sputtering have been carried out (Burattini et al., 2013). Therefore, original uncoated samples have been directly mounted on graphite/adhesive tap-coated stubs. All pictures (1cm x 1cm) have been performed at the same magnification, at the same high tension (15 kV) and by taking care in maintaining the same sample tilting before and after aging procedure, even if different in the four samples. Particular reference structures were chosen to ensure the correct comparison of the two experimental conditions.

SEM analysis of microbiological contamination has been performed after air drying and gold-sputtering (Salucci et al., 2013).

Monitoring of microbiological contamination

The observations of samples has been carried out by means of a Zeiss Axiolab Hbo 50/AC, with a 40x objective, at a final 400x magnification, as well as by SEM.

Results

Figure 1. Chromatic changes before (A,B,C,D) and after (A’,B’,C’,D’) artificial accelerated aging of 15% Aquazol® 200 (A,A’), 15% Aquazol® 500 (B,B’), BEVA® gesso (C,C’) and 2% Balsite® (D,D’); only in Balsite® specimens a chromatic changes can be revealed (*).
Compared to results obtained in the previous experiments, the products do not show a further dimensional shrinkage due to artificial aging and most of them, except for Balsite®, have no significant color changes (Figure 1). When plasters were obtained with 15% Aquazol® 200 or 15% Aquazol® 500, the samples appear fragile and strongly affected by sun irradiation or thermohygrometric damage. A number of surface deformations or signs of material breaks appear indeed (Figures 2 and 3). No chromatic changes before and after artificial ageing appear (Figure 1A, A' and 1B, B'). With regard to BEVA® gesso, both consistence and aspect appear good: in fact, the ready-for-use product made with EVA resins does not reveal aging-related deformations, fractures (Figure 4) or significant chromatic changes (Figure 1D, D'). The best results are observable in samples plastered with 2% ethanol Balsite® mixed with 15% white titanium pigment. SEM observations show that signs of mechanical changes are absent. Moreover, if compared to initial conditions of the pure product, no dimensional reduction or changes due to pigment addition, appear (Figure 5). However, the plaster shows a moderate chromatic alteration after the experimental induction of aging, revealing an amber-like tone (Figure 1C, C').

Despite its aging resistance, an unexpected behavior was observed with Balsite® fillers, which showed an important fungal growth (≥ 60),

**Figure 2.** SEM of samples plastered with 15% Aquazol® 200, before (A) and after (B) artificial aging: localized surface swellings (***) appear after the treatment. A,B, Bar=0.4 μm.

**Figure 3.** SEM of samples plastered with 15% Aquazol® 500, before (A) and after (B) artificial aging: a localized surface swelling (**) and occasionally breaks (→) appear after the treatment. A,B, Bar=0.4 μm.

**Figure 4.** SEM of samples plastered with BEVA® gesso before (A) and after (B) artificial aging: no effect is evident. A,B, Bar=0.4 μm.

**Figure 5.** SEM of samples plastered with 2% ethanol Balsite® and stained with 15% pigment, before (A) and after (B) artificial aging: no effect appears. A,B, Bar=0.4 μm.
in which microscopic analysis identified *Aspergillus niger* and *Aspergillus spp* (Figure 6). SEM observations confirmed the presence of a fungal contamination (Figure 7).

A possible cause of this problem are phloem fibers, hemp or flax, added to the products in order to increase their lightness, and potentially creating a more favorable environment to fungal growth. To this regard, the manufacturer of Balsite® has changed the plaster formulation by inserting a suitable biocide, the efficacy of which was reported by De Luca *et al.*, 2013.

**Discussion**

These observations demonstrate that Aquazol®, BEVA® *gesso* and Balsite® evidence the most significant results. However, for 200 and 500 Aquazol® products, a screen obtained by means of a special layer (back and front side consolidant application) is necessary, as generally used when the materials are utilized as consolidants. Moreover, a careful evaluation of preservation conditions, both from thermo-hygrometric or sun damage, is strongly recommended. If compared to ready-to-use Modostuc and Polyfilla commercial products, but also to common plasters obtained with *gesso* and animal glue, here described products show high quality mechanical, chemical and physical characteristics. Therefore, they can be considered excellent and ecologically compatible substitutes of animal glue.

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