Historical glazes: Enhancing their value through reproduction and characterisation

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ABSTRACT

This text presents a visual study of historical glazes made with lake pigments and dyes, applied on colour gradients used historically between the 15th and 17th centuries. This pictorial reproduction was carried out using materials and methodologies no longer in use, seeking methods of reproduction as close as possible to the pictorial processes used in the 15th and 17th centuries. This made it possible to evaluate the different results offered by the glazes applied to the basic colours from a visual and empirical point of view. For this study, several test pieces were made using nine pigments in the base layers on which different glazes were applied in several superimposed layers. To prepare the glazes, different types of lake pigments and dyes were used, all of them with different concentrations/thicknesses (from one to four coats). They were used to obtain different chromatic nuances that allow us to evaluate material, visual or perceptual aspects, highlighting the extent to which the glazes can alter, shading or accentuating the underlying colour. These data were quantified and assessed by means of homogeneity coefficients, colouring power, hiding power and particle visibility.

KEYWORDS: lake pigments, dyes, transparencies, glazes.

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1. Introduction

The concept of glazing has been interpreted throughout history in different ways. In fact, its current meaning is relatively recent and, therefore, the term itself is not found in ancient treatises or recipe books[1] Mentions or allusions are found to the 'act of veiling' in these historical sources, describing techniques that suggest that the application of translucent layers of colour over a more opaque one, in order to alter, shade, change or accentuate the underlying colour, was known and commonly used as early as the 12th century (Gollini, 2020). As they are very thin layers, glazes are usually very fragile and delicate, and due to the organic nature of their materials they are more unstable in the presence of light and humidity, which causes them to degrade and even dissolve (Matteini, 2001). For this reason, as they are often mistaken for repainting or layers of deteriorated varnish, they are frequently removed. The insufficient study of glazes has sometimes led to their complete removal in the restoration and cleaning processes of many artworks. As a result, there are numerous facts that reveal the lack of knowledge about the technical and material aspects of the work linked to glazes, leading to their systematic elimination.

In addition to the well-known texts by Cesare Brandi (Brandi, 1995; Brandi, 1949), it is worth mentioning that of René Huyghe (Hernández, 2019), art historian and curator of the Louvre since 1937, who, in response to the controversy raised regarding the excessively radical restoration criteria of the National Gallery, questioned the cleaning methods proposed by this institution, pointing out that a "total" cleaning of the work may not return it to its original state, but could even remove glazes that played an essential role in the final appearance of the artwork.

Faced with this lack of historical consideration, on the one hand, and on the other, the inherent fragility that characterises both the material and the technique of the glaze, the aim of this article is to investigate this element through experimentation and reproduction with pigments (as a base paint), lake pigments and dyes that formed part of the pictorial palette from the 15th to the 17th century. The aim has been to describe and characterise glazes from a visual point of view, considering factors such as tone, colouring and covering power, homogeneity of the layer and visibility of the particle.

2. Materials and methods

With the aim of investigating the nature and characteristics of the glazes used between the 15th and 17th centuries, this work proposes the study of pictorial superimpositions made with lacquers and dyes, on chromatic bases made with different earth pigments (ochre, raw sienna, raw umber, burnt umber, *Herculaneum* red and green earth), lead white, vine black, azurite and vermilion.

For this purpose, twelve glazes were reproduced using dyes and lacquers, applied on the nine chromatic bases already mentioned, using 35x35 cm slabs prepared with calcium sulphate ($CaSO_4$), calcium carbonate ($CaCO_3$) and rabbit skin glue. Nine squares coloured with pigments agglutinated with linseed oil, progressively reducing their saturation by adding lead white, were applied to each tablet (Figure 1). All the pigments were ground, refined and agglutinated, reconstructing the historical procedures recovered from the sources. The following pigments were used for the nine chromatic bases on which the resulting glazes were applied[2] :French Ochre (40010, Kremer), Lead White (46000, Kremer), Raw Sienna (0263, CTS), Raw Umber (0266, CTS), Burnt Umber (0261, CTS), Herculaneum Red (0316, CTS), Vermilion (42000, Kremer), Vine Black (47000, Kremer), Green Earth (0264, CTS) and Azurite (10200, Kremer).

Fig. 1. Application of the chromatic bases on the boards

The twelve glazes were produced using lacquers and dyes, all supplied by Kremer: carmine naccarat (42100), *lac dye* (36020), madder lake coral (372051), dark red (372141), madder lake violet (37218), reseda (36262), aloe (38010), *stil de grain* (37394), indigo (36002), sap green (37391), *atramentum* (12030) and sepia (12400).
As already noted in previous studies (Bomford, et al., 1995), for the application of these pigments in the form of glazes it was necessary to use a varnish (oleoresin) as a binder and thus guarantee an adequate ratio of absorption and drying between the oil and the colouring matter, thus obtaining the desired visual result. There are many sources that refer to the use of varnishes as binders, particularly for pigments that dry with difficulty. Among some examples, the 16th century *Marciana Manuscript* describes how varnish is an excellent binder both for oil painting and for other painting techniques (Merrifield, 1849b). Also, in the same century Giovanni Battista Armenini, suggests adding "[...] oil and a little common varnish, because this varnish is of such a quality that it gives strength and helps all colours that suffer from drying [...]"[3] (Armenini, 1587).

In this work, a varnish made from linseed oil and mastic resin was used as a binder for the dyes and lake pigments to be applied as glazes. Mastic resin was widely used in the production of paintings from the 9th century to the end of the 19th century. It is a soft triterpenoid resin of natural vegetable origin obtained from the mastic tree *(Pistachia lentiscus. L),* which is abundant on the Mediterranean coasts (Zalbidea Muñoz, 2014). On this occasion, the varnish used consisted of two parts linseed oil to one part resin, thus reproducing the recipe described by Theophilus in the 12th century in his treatise *De diversis artibus* (Theophilus, 1847; Zalbidea Muñoz *et al.,* 2017; Zalbidea Muñoz *et al.*, 2022).

Once the lake pigments and varnish had been bound together, superimposed layers of glazes were applied on top of the base colours, thus generating a gradation in the saturation of shades (Figure 2). When preparing these mixtures, due to the different properties of the lacquer pigments, it was necessary to carry out tests on the proportions of lacquer and varnish needed to apply the glazes, considering their hiding power, ease of application and dissolution of the particle in the medium.

The glazes reproduced in this study are detailed in the next sections.

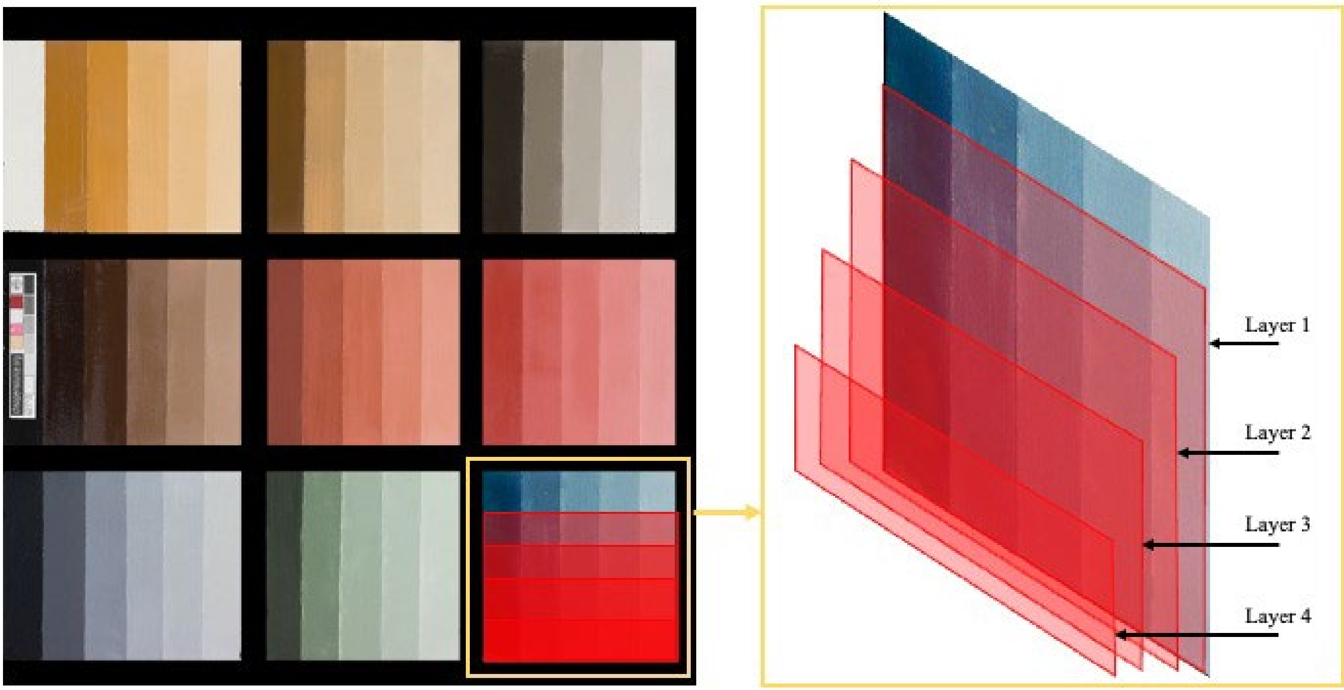

Fig. 2. Graphical representation of the application of layers of glazes. On the left, the table with the nine base pigments and their different degrees of saturation is shown. On the right, the application of the different layers of glaze superimposed on one of the base pigments is shown.

2.1. *Reds*

Historically, lakes (or lake pigments) of red tone were obtained from the madder roots (Rubia tinctorum L.), or from insects such as *Kermes Vermilio* L., known as *Kermes Vermilio Planch* L. (López, 2002), but also from parasites of *Quercus coccifera* L. (Kirby *et al*., 2014) and *Coccus Ilicis* L., which infest respectively the leaves and branches of oaks and holm oaks such as *Quercus ilex* L. (Gettens & Stout, 1966). These reds have been widely used throughout history for their application as glazes in oil painting (Leona, 2009). The use of madder as a dye for fabric dyeing and lakes production has been known since ancient Egypt, Rome and Greece (Mayer, 2005). The proportions of alizarin and other anthraquinone present in the root of the plant determine the shade of dye obtained, as does the different way in which they are processed (even the temperature of the water has an influence), giving rise to shades ranging from red to purple (Hofenk de Graaff, 2004). In this way, three shades have been obtained from the madder that have been applied as glazes: madder lake coral, dark red and madder lake violet. As far as red lacquers of animal origin are concerned, carmine naccarat is obtained from the American variety of cochineal called *Coccus Cacti* L. This variety began to be imported from the Americas in the 16th century, progressively banishing the lac obtained from *Kermes Vermilio* L., a parasite of *Quercus coccifera* L.

(Zalbidea Muñoz & Herrero-Cortell, 2022). As for lac dye, it also originates from the secretion of another insect similar to the cochineal called *Coccus Lacca* L. or *Kerria Lacca* L. Its colour is similar to those obtained from cochineal and kermes, but warmer. As Scott points out, although it is more permanent than cochineal, it is less glossy, and the glaze is denser than that obtained with cochineal (Scott, 1885).

2.2. *Yellows*

Among the most lightfast natural dyes, yellow lakes have been widely used in the textile dye market, where they are also used as a complementary shade to obtain other colours such as orange or green (Herrero-Cortell, 2019). Reseda (also known as *arzica*) has stood out as one of the most widely used yellow dyes throughout the history of art and despite its low colouring power compared to other yellow dyes, it provides a very intense tone (Gettens & Stout, 1966). As in the case of red lakes, the obtaining of reseda lake is subordinated to the addition of essential compounds such as, for example, alum. But also, the addition of small amounts of lead white gives it a higher opacity (Bomford *et al.,* 1995). A second example of yellow dye used in the study is *stil de grain*. Merrifield (1849a) notes that in France this term describes the range of lake pigments from pure yellow to green-tinted yellows. It is a dye derived from the processing of the unripe berries of the *Rhamnus* L. plant (Eastaugh *et al*., 2008). Water and soda are used to extract the colouring matter which, when precipitated on clay, was used in ancient times as a yellow lake (Doerner, 2001) and which, according to Watin (1773), can be used as a transparent glaze when alum is added. Another material used to produce yellow lakes is aloe. For its preparation it was not necessary to use mordants. It is known to be used as a supplement in varnishes and glazes (Merrifield, 1849a) or as a pigment for the preparation of glazes (Eastaugh *et al*., 2008) to accentuate or enliven the shades on which it was applied.

2.3. *Blues*

Used since ancient times, indigo has been applied throughout Europe since the 14th century as a complement to azurite and ultramarine (Doerner, 2001). It is not uncommon to find it combined with red lacquers to obtain violet shades and its use to highlight shaded areas is already mentioned in the medieval Strasbourg manuscript (van Eikema Hommes, 2002). Insoluble in water, it is obtained from the plant *Indigofera* L. A second common variety in Asia is pastel blue or glasto, extracted from the leaves of *Isatis tinctoria* L., which is widely used for pictorial purposes in Europe (San Andrés *et al.,* 2010). Indigo together with lead white was applied to reduce the intensity of the blue or was fixed to fillers (lime or white clays) to create pigments with more body (Herrero-Cortell, 2019), and in this way very colourful blue mixtures were obtained, emulating azurite.

2.4. *Greens*

Green lakes have not been a regular part of the pictorial palette, as this tone could be obtained from a mixture of yellows and blues. The fact that green earths bound with oil generate a translucent mixture and that *cardenillo* also has transparent properties determines the low popularity of green lakes in artistic palettes (Herrero-Cortell, 2019). However, de Mayerne (1620) describes this colour as suitable for shading other greens, even when used in several layers. The same author mentions obtaining greens with yellow, *massicot* and cobalt blue lakes (Eastlake, 1847). In this experiment, glazing with sap green has been applied. In the *Manoscritto Bolognese (De Fiendis viridibus),* among other recipes, it is described how to obtain this lake pigment from crushed buckthorn berries, exposed to the sun and with the addition of alum (Merrifield, 1849b).

2.5. *Blacks*

The composition and nature of *atramentum* is not yet defined (Zalbidea Muñoz, 2014). Vitruvius in his *De Architectura* (1st century BC) describes its preparation from the soot produced by the combustion of resin or the embers of resinous wood. Pliny (*Storia Naturale* I century AD) also relates this term to a final varnish or glaze with a protective as well as aesthetic intention (Giannini, 2008). The *atramentum* provided by Kremer Pigmente comes from the tannic acid of oak galls, produced by the bite of an insect that leaves its eggs in the tree stems (generally of the genus *Quercus L*.). In most cases, this ink was used to outline the figures and to darken specific areas (Herrero-Cortell, 2019).

On the other hand, sepia colour is obtained from the ink sacs of some cephalopods. It provides a very dark and semi-transparent colour and is therefore excellent for use in watercolour techniques (Mayer, 2005). It has a great colouring power and is very suitable for working in both aqueous and oily media, although watercolour allows for a multitude of shades and tones (Terry, 1893).

3. Results and discussion

After application of the glazes, the behaviour of each of the lakes and dyes applied was observed. The results obtained according to the above-mentioned criteria are described below:

3.1. *Reds*

As mentioned above, the colour tone of the lakes from madder can vary depending on the mordant used, but also on the proportions of alizarin, purpurin or pseudopurpurin contained in the root from which the colouring matter is extracted (San Andrés *et al.,* 2010; Schweppe & Winter, 1997). Likewise, red lakes of animal origin provide different shades depending on the mordant used or the insect of origin. The translucent property of carmine naccarat (Figure 3) makes it suitable for use as a glaze (Schweppe & Roosen-Runge, 1997). This characteristic is observed in the test tube made with this lake, giving a very intense magenta colour. Lac dye and madder lake violet have a high colouring power from the first coat of application (Figures 3 and 4), with the dark red glaze generating the least saturation and colour intensity (Figure 4). The coats applied with carmine naccarat, lac dye and madder lake coral were more homogeneous, with madder lake violet being the glaze that generates very visible particles (Figure 4 and 5). Most of these lakes had a high/medium hiding power which, in some cases (carmine naccarat and lac dye), almost hid the underlying base pigment in the fourth glaze layer.

Fig. 3. Glazes made with carmine naccarat (1) and Lac Dye (2)*

Fig. 4. Glazes made with madder lake coral (1), dark red (2) and madder lake violet

3.2. *Yellows*

The yellow lakes have little covering and colouring power, although in the case of *stil de grain*, the superimposed layers of glaze generate changes in the underlying tonality. In all cases except for the reseda, the dye particles are visible, forming an inhomogeneous layer (Figures 5 and 6). As for aloe, at least three coats of polish are needed to create changes in the underlying shade.

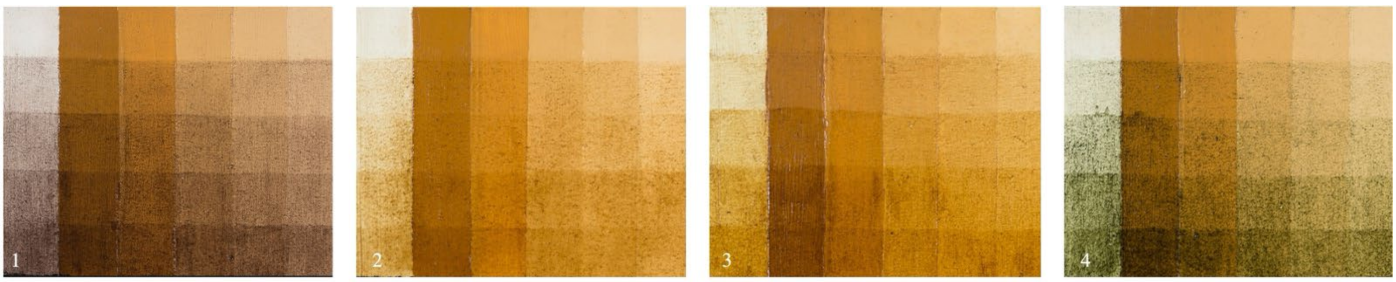

Fig. 5. Details of particle visibility in applied glazes with madder lake violet (1), aloe (2), stil de grain (3) and sap green (4)

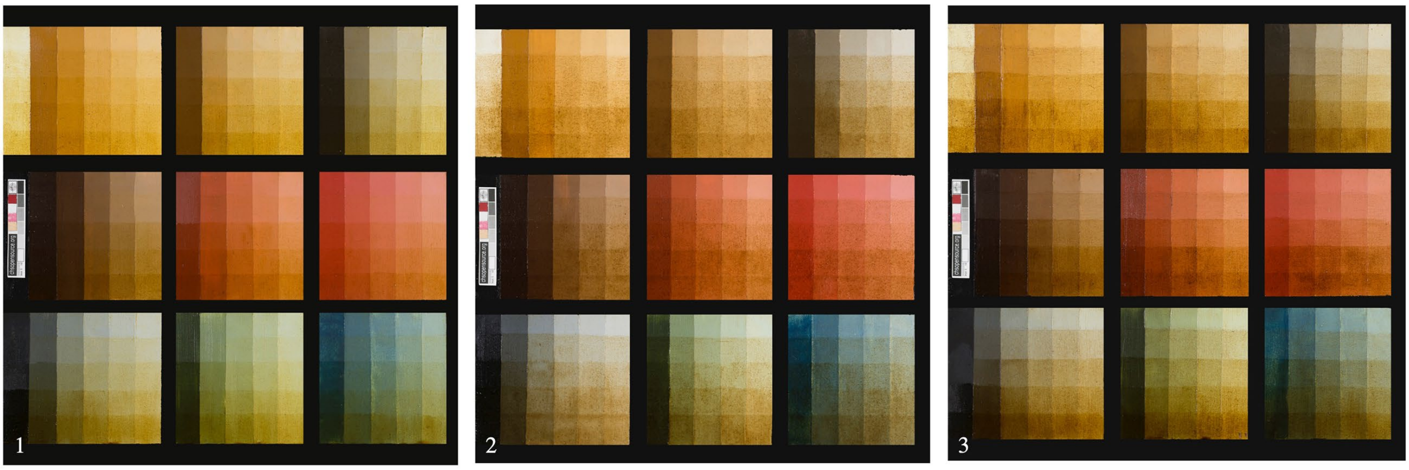

Fig. 6. Glazes made with reseda (1), aloe (2) y stil de grain

3.3. *Blues*

As shown in Figure 6, indigo has an unattractive shade, although as Ball (2012) notes, it becomes more pleasing when mixed with white. The dye particles are moderately visible. It has a high colouring and hiding power and produces homogeneous and uniform glazes. This quality was optimal for creating shadows on fabric folds (Artoni *et al*., 2019).

3.4. *Greens*

The glaze applied with sap green (Figure 7) has a dark green tone, with little colouring and hiding power and in which the presence of particles of the dye is evident (Figure 5). The layer is not very homogeneous in its application, so it is very likely that it was not used in extensive glazes. Therefore, it was very common to apply glazes with copper resinate based on a mixture of *verdigris* with resin, as a more homogeneous and saturated result was obtained than with sap green (Doerner 2001).

3.5. *Blacks*

Atramentum has a brown colour, very similar to madder lake violet, but unlike the latter, it behaves very uniformly in all applications and has a high hiding power (Figure 7). It can be seen how this glaze on the vine black creates a chromatism similar to that produced by the base pigment. Also characterised by a very homogeneous and uniform film in which the dye particles are barely perceptible, sepia provides a brown shade similar to *atramentum* although it shows more opacity, high hiding power and colouring (Figure 7).

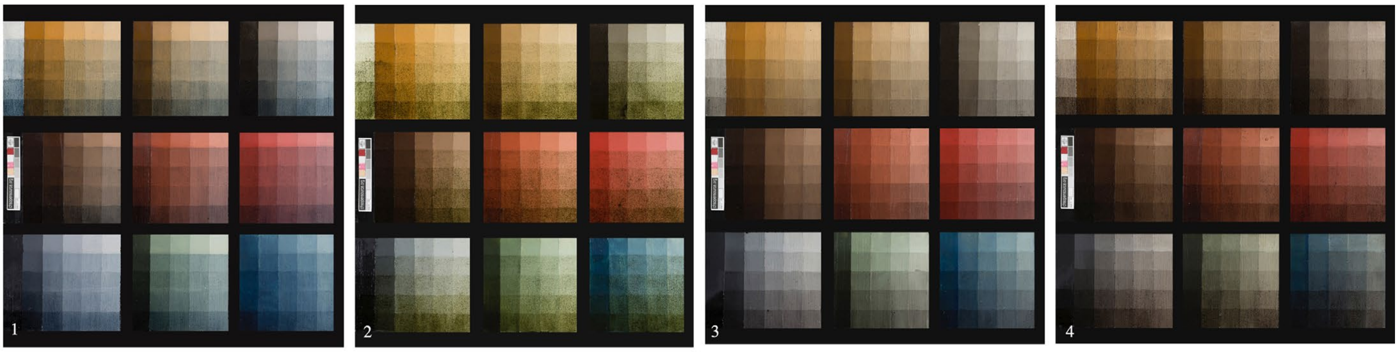

Fig. 7. Glazes made with indigo (1), sap green (2), atramentum (3) and sepia (4)

4. Conclusions

After application of the glazes on all the test pieces, relevant conclusions were drawn. As the literary sources show, the range of red dyes generates multiple shades. The origin of these lakes (vegetable or animal), as well as the mordant used for their production, leads to shades ranging from deep red to dark brown. The red glazes applied with carmine naccarat, lac dye and madder lake coral have generated a uniform layer, creating very covering films with high colouring power. As for the yellow tones applied with reseda, homogeneous glazes have been created, but with low covering and colouring power. Even so, authors such as Herrero-Cortell (2019) recall that this lake has been widely used to achieve other complementary colours. Both *stil de grain* and aloe have created more saturated layers of colour in successive applications, but the dye particles are quickly visually perceptible. In the range of blue shades made with indigo, very homogeneous glazes with high covering power were created, thus confirming the words of Artoni (2019) who stated that its properties were optimal for providing depth and shadows on fabrics. As Ball stated, it was also observed that indigo glazes applied on white tones generate softer shades (Ball, 2012). In this test tube, the dye particles are also visible to the naked eye, although in other experiments they may not be visible, due to the milling process used (Figure 7).

The sap green has a dark shade. As can be seen in the test tubes, this lake does not offer homogeneous results, being difficult to handle and bind by hand. As pointed out by several authors (Herrero-Cortell, 2019; Mayerne, 1620; Eastlake, 1847), the existence of other formulas for obtaining the green colour means that this lake has not been popular for application as glazes. Given its lack of homogeneity, it is very likely that it was not used for large or extensive glazes, but only in limited or localised areas.

As for black tones, both *atramentum* and sepia, usually applied to accentuate shadows, have generated very homogeneous glazes of high chromatic intensity.

5. Conflict of interest declaration

The authors wish to state that no financial or personal interests have affected the objectivity of the study, and that no conflicts of interest exist.

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7. Short biography of the author(s)

Laura Fuster-López - Professor at the Conservation Department in the Universitat Politècnica de València (Spain). After several research fellowships at Europe and the United States, she got her PhD in Conservation from UPV where she coordinates the area on the study of the mechanical and dimensional properties of cultural materials since 2007. She has been involved in numerous international R+D projects always with a special focus on understanding the mechanisms involved in paintings behavior and degradation.

Mª Antonia Zalbidea-Muñoz - Professor in the Department of Conservation and Restoration of Cultural Heritage at the UPV, field in which she holds a doctorate degree. She has directed her research on the procedural, material and conservative aspects of mural painting, as well as parts related to varnishes and dyes in painting. At present she is also a collaborator in the work team of the project: Byzantine illuminated manuscripts in Spain: work, context and materiality - MABILUS (MICINN-PID2020-120067GB-I00).

Miquel Àngel Herrero-Cortell - He holds a PhD in Art History. He has a degree in Fine Arts from the Polytechnic University of Valencia (UPV) and a degree in Art History from the University of Valencia (UV). He holds a Master's Degree in Conservation and Restoration of Cultural Heritage and a Master's Degree in Artistic Production. He has developed his work as a researcher focusing on the field of materials and painting techniques, as well as on painting diagnosis. He is currently teaching at the Universitat Politécnica de València.

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Notes

[1] In the Middle Ages, the monk Theophilus in his treatise *De diversis artibus* refers to glazes, without including the term, but alluding to "translucent" paintings (Theophilus, 1847). Also, in his *Treatise on Painting*, Leonardo Da Vinci talks about transparent colors (De Vinci and Alberti). But Giovanni Battista Armenini (1587) is the first author to use the term glazing in his book *De veri precetti della pittura* (Armenini 1820).

[2] For more information, please consult the websites of the pigment suppliers: https://www.kremer-pigmente.com/ and https://shop-espana.ctseurope.com/

[3] The original text saids: "...añadiendole aceite y un poco de barniz común, porque este barniz es de tal calidad que da fuerza y ayuda a todos los colores que sufren al secarse…" (Armenini, 1587).

Licensing terms

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