Optical Characterisation of Oil Painting Films with Drying Pigments under the Microscope: Cobalt Blue

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ABSTRACT

Traditionally, oil painting techniques have used certain additives to improve and adjust the rheological characteristics and properties of oil paints. In addition to the main components of oil paints, such as pigments and oils, additives have been used, among which driers, mainly composed of metallic salts such as lead, zinc, cobalt, manganese, copper or iron, play a crucial role in accelerating the drying process of the paint.

This paper focuses on the study of oil paint films made with cobalt blue pigment. The main objective is to observe and characterise this pigment using various non-invasive techniques. Specifically, it focuses on the microscopic observation of mock-ups made with cobalt blue in order to detect possible changes in the morphology of different pigments caused by accelerated ageing. This article aims to complement the characterisation of these mock-ups by colorimetry, the results of which were presented at the XVIII Conferenza del Colore in Lecco, 2023 (Llácer Peiró et al., 2023).

KEYWORDS drying pigments, cobalt blue, surface microscopy, artificial ageing

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1. Introduction

Oil painting is characterised by being a technique that employs ground pigments and/or dyes bound in a drying oil. The technique of oil painting is closely related to the use of additives to accelerate the drying process. The effectiveness of drying agents depends on the quality, purity, and application of the materials used (Zumbühl and Zindel, 2022). Although compared to other mediums, drying oils facilitated the rapid drying of paint, allowing artists to work more quickly (Eastlake, 1847; Laurie, 1926), at times the drying time was not fast enough, especially when working with multiple layers of paint. For this reason, other additives were incorporated as catalysts to further accelerate the drying process (San Andrés et al., 1996). Drying materials commonly used in oil painting include:

- Binders, typically oxidative drying oils. These are the drying oils mentioned above (linseed, walnut, or poppyseed oil) (San Andrés et al., 1996; Matteini and Moles, 2001).
- The pigments themselves, especially those of metallic nature, such as litharge or lead white. These metals act as driers by accelerating the drying process, reducing the induction period, increasing the formation and breakdown of peroxide, and finally polymerisation (Girard et al., 1965; Honzíček, 2019; Bieleman, 2002).
- Metallic salts, added to the oil as liquid driers to catalyse drying, such as Cobalt siccative, Harlem siccative, or Courtrai siccative (Villarquide, 2004).
- Natural (mastique, rosin) or synthetic (alkyd) resins, substances widely used as drying agents (Armenini, 1587; Eastlake, 1847; Merrifield, 1849; Doerner, 1998).

Although there was empirical knowledge of how certain materials affected the drying of paints, detailed studies of drying agents did not appear until the 1840s, when zinc oxide began to replace lead white (Bieleman and Lomölder, 2000). Analysing how these pigments can affect alteration, deterioration or pathology in a work is crucial for accurate diagnosis. The identification of these pigments can serve as a starting point for the characterisation of the artwork. This paper focuses on the study of oil paint films containing cobalt blue pigments, both as single paint films (without ground layer) and in interaction with the underlying ground layer. Specifically, it presents the microscopic analysis carried out to detect possible morphological changes caused by accelerated artificial ageing. This study complements the colorimetric analysis of these pigments, the results of which were presented at the XVIII Conferenza del Colore in Lecco (Llácer Peiró et al., 2023). The data and conclusions derived from this study may contribute to the understanding of the role of cobalt pigments in the modification and/or alteration of oil paintings. In addition, these results could be compared withcase studies to identify possible similarities and differences.

2. Objectives

The main objective of the present study is to identify and investigate the morphological changes that can occur in the paint film as a result of artificial ageing processes using a xenon lamp. Specifically, the study will investigate to what extent the use of cobalt blue pigments either on a traditional chalk-based ground layer and on an inert surface (glass), combined with different drying oils (linseed oil or walnut oil), can influence these phenomena. In this way, a deeper understanding is sought of how these aspects can contribute to the dimensional variations observed.

3. Materials and methods

3.1. Mock-ups Preparation

Three types of cobalt blue pigments (Kremer Pigmente) were used for characterisation: medium cobalt blue, dark cobalt blue, and cerulean blue, manually bound with two drying oils (linseed oil and cold-pressed walnut oil) (Table 1). Sources consulted on the appropriate proportions for binding oil and pigment showed discrepancies (Mayer, 1993; Myers, 2013). For this reason, the measures provided by Kremer Pigmente were followed to achieve the optimal quantitative ratio between pigment and binder (known as Critical Pigment Volume Concentration - CPVC-) [1] (Table 2).

Table 1. Materials used in the experimental

Pigment	*Ref. Kremer*	*Color Index*			*Chemical formulation*	
Cobalt cerulean blue	45730	PB 35. C.I. 77368			$CoO\text{-}nSnO_2$	
Cobalt blue medium	45710	PB 28. C.I. 77346			$CoAl_2O_4$	
Cobalt blue dark	45700	PB 74. C.I. 77366			$(Co,Zn)_2SiO_4$	
	Ref. Kremer	**Fatty acid composition (%)** (*Izzo, 2011*)				
Binder		**Palmitic**	**Stearic**	**Oleic**	**Linoleic**	**Linolenic**
Linseed oil, cold-pressed	45730	4-10	2-8	10-24	12-19	48-60
Walnut oil, cold-pressed	45710	3-8	0,5-3	9-30	57-76	2-16

Table 1. Ratio of pigments and binders.

Table 2. Proportions of pigment binder used

Pigment	*Linseed oil ratio*	*Walnut oil ratio*	*Pigment ratio*
Cobalt cerulean blue	16,67%	16,67%	83,3%
Cobalt blue medium	27.01%	27.01%	72.99%
Cobalt blue dark	15.25%	15.25%	84.75%

Table 2. Proportion of binder/pigments used in the preparation of the mock-ups.

The mixtures obtained were applied to two types of support: on wood (Medium Density Fiberboard, MDF) boards, measuring 2.5 x 8 cm, prepared with a chalk ground made of sulphate, calcium carbonate and animal glue (Santos, 2005); and on glass slides, measuring 2.5 x 7.5 mm (Figure 1). Both materials are dimensionally stable. The glass allowed for the evaluation of the interaction between the pigment and the oil, avoiding interferences with other ground layers. The MDF board was used to observe the interaction between the pigment and the ground layer. This choice of a rigid support was aimed to prevent the usual dimensional changes observed in canvases. These mock-ups were prepared under constant environmental conditions of relative humidity (RH) of 49% and temperature (T ºC) of 20 ºC.

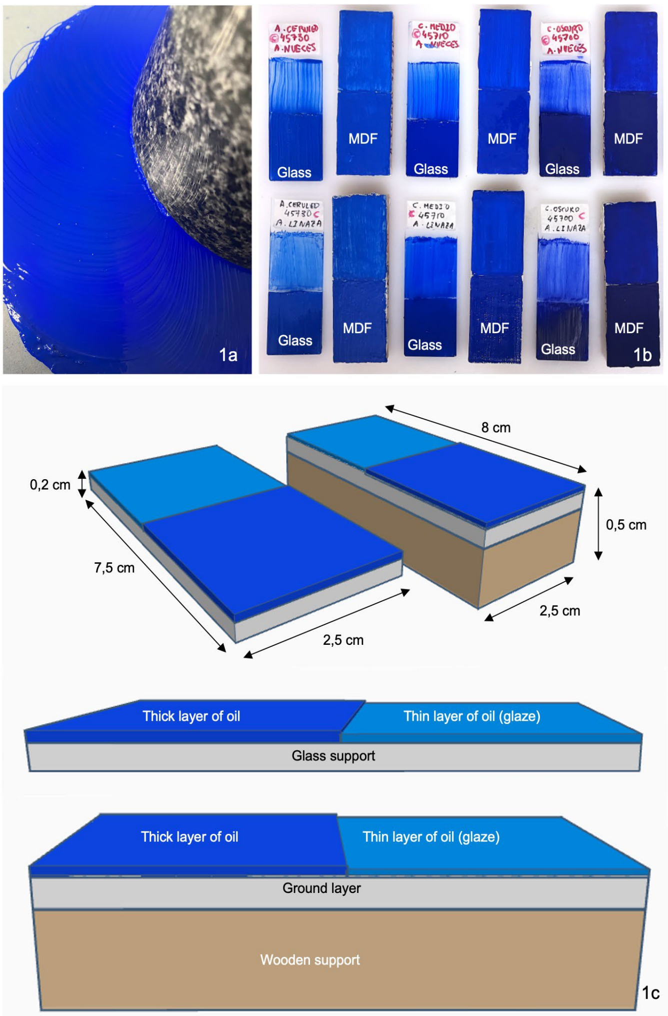

Fig. 1. Grinding cobalt blue pigment in linseed oil (1a), mock-ups made on the two substrates (1b), and diagrams of the different substrates used in this study (1c).

3.2. Artificial ageing

The mock-ups were subjected to ageing under artificial light provided by a xenon lamp (Suntest CPS+ Atlas) to evaluate changes in their properties caused by sunlight and temperature. This phase was carried out according to the ASTM D4303-03 standard, supported by previous research on the ageing of artistic materials (Sánchez and Micó, 2010; De los Reyes et al., 2015; López-Montes et al., 2016; Cavaleri et al., 2019). The specific process conditions were as follows:

- Filter: Window glass red. IR
- Wavelength (nm): 300-800 nm
- Irradiance (W/m^2): 500
- Temperature BST: 50º
- Chamber air temperature: 34º
- Duration time (h): 250+250 = 500 Total

In Figure 2, images of the mock-ups before and after artificial ageing are shown.

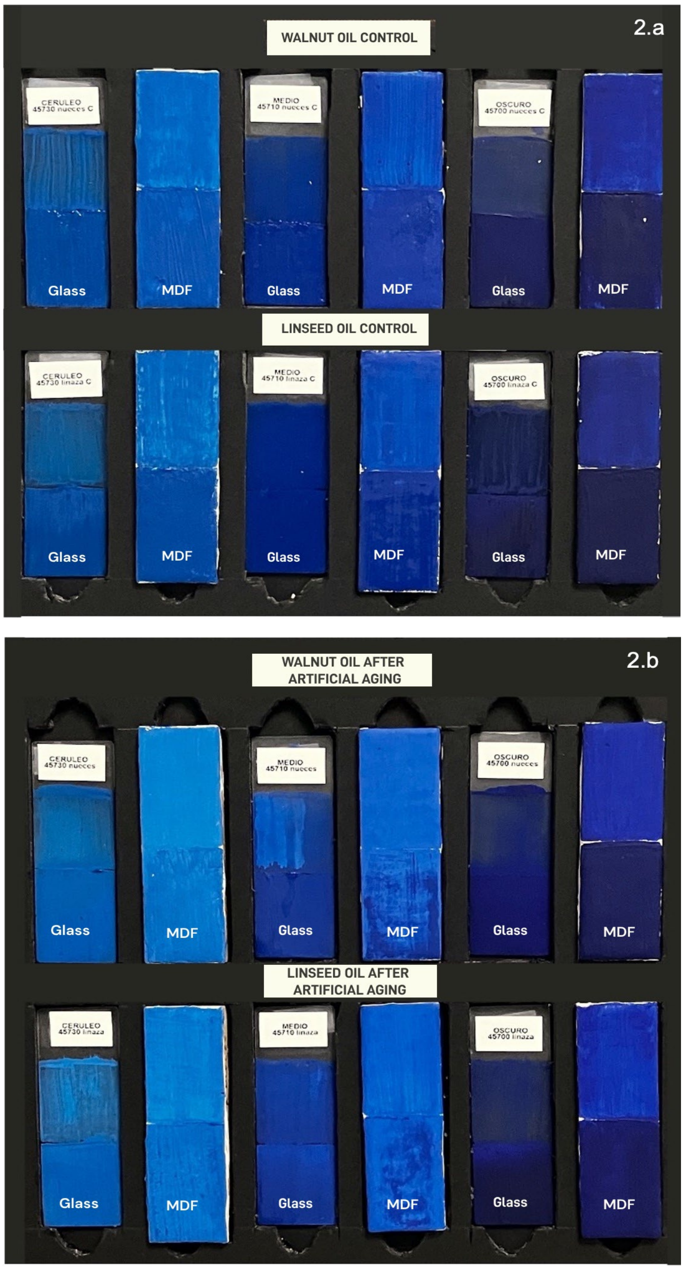

Fig. 2. 2a: Mock-ups before accelerated ageing; 2b: Mock-ups after ageing

.3.3. Microscopy

Stereomicroscopy is a technique that allows precise observation and characterisation of materials, allowing morphological studies through visual analysis of particle size, dispersion or distribution of pigment particles. The study used the stereomicroscope Leica® MZ APO and Leica® M165 120x. Representative areas were selected and sampled in all mock-ups (Figure 3) in order to obtain maximum information about the changes in the paint film. To facilitate the interpretation of the results, Table 3 shows the abbreviations used to identify the mock-ups, including the type of substrate, binding media, sampling area and pigment used.

Table 3. Acronyms used to identify mock-ups

Support	***Binder***	***Zone***	***Pigment***
W= wood G= glass	LI= linseed NU=walnut	Z	Cerulean Medium Dark

Table 3. Acronyms used for mock-ups identification

In this way, the image named "W-LI-Z1-Cerulean 1" corresponds to microscopy performed on the board support (W), with linseed oil as binding media (LI), in sampling area 1 (Z), using cerulean blue pigment.

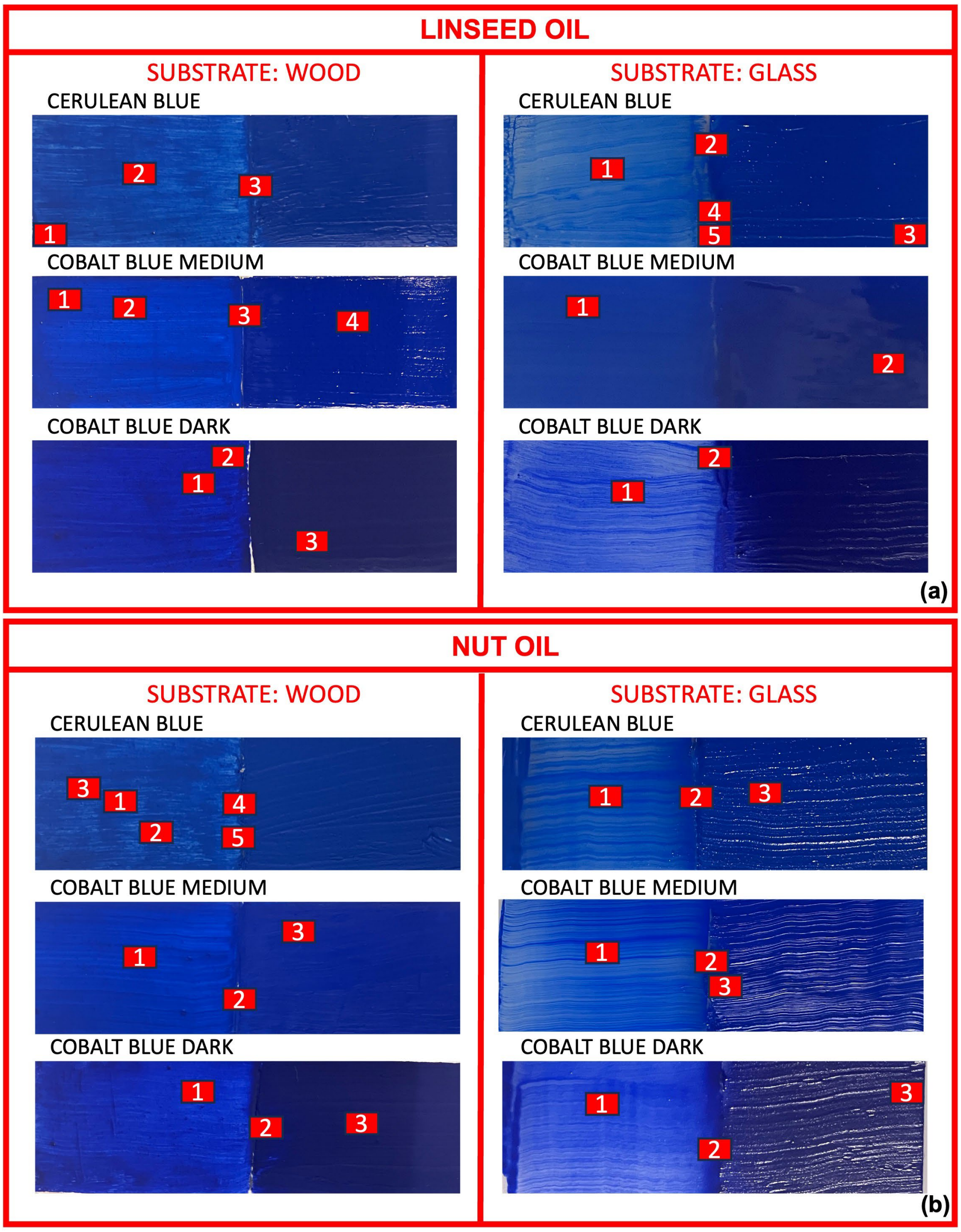

Fig. 3. Location of the sampling points where microscopy was performed: mock-ups with linseed oil (a) and with walnut oil (b).

4. Results

After subjecting the mock-ups to artificial ageing, widespread loss of the initial brushstrokes was observed in most of the mock-ups on wooden supports. This loss is attributed to the levelling index of the binder. When a brushstroke is applied, the viscosity of the binder decreases, improving its flowability. When the binder rests, it returns to its original viscosity, causing surface levelling (Zalbidea, 2014; Alonso Felipe, 2016). The porous nature of wood allows it to absorb some of the binder. Conversely, the non-porous nature of glass prevents the paint from penetrating the substrate, resulting in more defined brushstrokes and preserving some of the texture created by the brush.

4.1. Cerulean Blue

After applying the pigment to the wood panel using both oils, a smooth and homogeneous surface with a velvety appearance is observed. The pigment plus binder mixture has a medium tinting strength and, as it can be observed in the pictures, it is the only cobalt blue pigment without a violet hue [2] (Gettens & Stout, 1966).

- Results on the wooden panel: after 250 hours of accelerated ageing, the mock-ups show a more heterogeneous appearance due to the presence of spots and small craters on the surface of the paint layer caused by air entrapment. After 500 hours, the surface becomes rougher with persistent spots (Figures 4D, 4E, 4F). After 250 hours of accelerated ageing, the binder in the paint remains visible on the surface (Figure 4B). However, after 500 hours, the binder is not visually perceptible, resulting in the formation of small cavities or holes in the paint surface (Figure 4C). After 500 h, a surface fracture is observed in the areas of intersection between the thick brushstrokes and those applied as a glaze (Figure 4I). This phenomenon had not manifested prior to the artificial ageing process (Figure 4G). No significant morphological differences are observed between the two types of oil used.

- Results on the glass slide: a finer, more uniform and less opaque surface is produced on the glass support. This phenomenon is attributed to the refractive index of glass (1.5), which is very similar to the refractive index of both oils (1.484 for linseed oil and 1.46 for walnut oil) (Knut, 1999), resulting in a more translucent appearance compared to the wooden support (Figures 4P and 4R). After ageing, irregularly distributed dark spots are observed on the surface. These spots, which are rounded in shape and vary in size (Figure 4Q), are caused by pigment accumulation. No significant morphological differences were observed between the two types of oil used.

4.2. Medium Cobalt Blue

The medium cobalt blue has a more purplish hue than cerulean blue [3]. As described by Roy (2007), it has a cloudy and powdery appearance. Like cerulean blue, the surface is initially homogeneous and uniform, a characteristic that changes after ageing and acquires a more irregular texture (Figures 5C and 5I).

As the refractive index of medium cobalt blue (1.74) is lower than that of cerulean blue (1.84) (Knut, 1999), the former becomes a less opaque pigment (Figures 5K and 5L). It is important to distinguish between 'hiding power' and 'opacity'. Although these terms are closely related and are often used interchangeably, they have different nuances in technical meaning. Opacity refers to the ability of the pigment to hide the colour of the substrate to which it is applied, whereas hiding power refers to the ability of the pigment to block the passage of light through it. Opacity is closely related to refractive index, and opacity is proportional to the difference between the refractive index of the pigment and that of the binder in which it is dispersed (Abel, 1999; Doménech, 2020).

- Results on the wooden panel: in mock-ups where the pigment is bound with walnut oil, voids caused by air retention are visible on the surface (Figures 5G-5I). Around these voids, early shrinkage cracks formed without reaching the primer layer. These cracks occur as the surface layer of paint dries (Knut, 1999). The role of cobalt as a drying agent may contribute to the formation of these surface cracks (Soucek et al., 2012; Fuster-López et al., 2019).

Figures 5E and 5F show that in areas where a thin layer of pigment has been applied there are no dark spots caused by the binder after 500 hours of ageing. The reason is the thickness of the applied film, another factor that influences the oxidation process of the oil. Thicker paint films require longer drying times (Mallégol et al., 2000).

- Results on the glass slide: brushstrokes are visible, but unlike cerulean blue on glass, there are no dark spots due to pigment accumulation (Figures 5O-5Q), probably because of the small particle size (between 2 and 40 μm) compared with that of cerulean blue (Roy, 2007).

4.3. Dark Cobalt Blue

- Results on the wooden panel: with regard to the dark cobalt pigment applied to the wood panel, the surface shows a relatively uniform distribution of pigment with some areas showing greater clustering, an effect that persists after 500 hours of ageing (Figures 6A, 6B and 6C). Small scattered dark spots are evident, which may indicate areas of pigment accumulation.

Cratering can be seen in Figures 6D, 6E and 6F, an effect noted by Damato (2014). Bright white spots can be seen in some mock-ups (Figures 6F, 6I and 6L). In future studies, elemental analysis using SEM-EDX will be performed to obtain further information on these mock-ups. It can be observed that before the mock-ups are subjected to the ageing process, cratering has already occurred, surrounded by a brighter and diffuse halo, although they do not have cracks around their perimeter (Figures 6D, 6E and 6F). This pattern persists even after 500 hours of ageing.

In some areas of the mock-up prepared with dark cobalt pigment and walnut oil, the surface is extremely rough with numerous irregularities, as shown in Figures 6J, 6K and 6L.

- Results on the glass slide: after 500 hours of ageing, a reduction in the opacity of the paint is observed, even in areas where thicker layers have been applied (Figures 6Ñ and 6Q). This decrease in opacity is attributed to the similarity between the refractive indices of glass and the oils used as binders (Knut, 1999). Because the refractive index of glass is close to that of the oils, light passes more directly through the material, reducing the paint opacity.

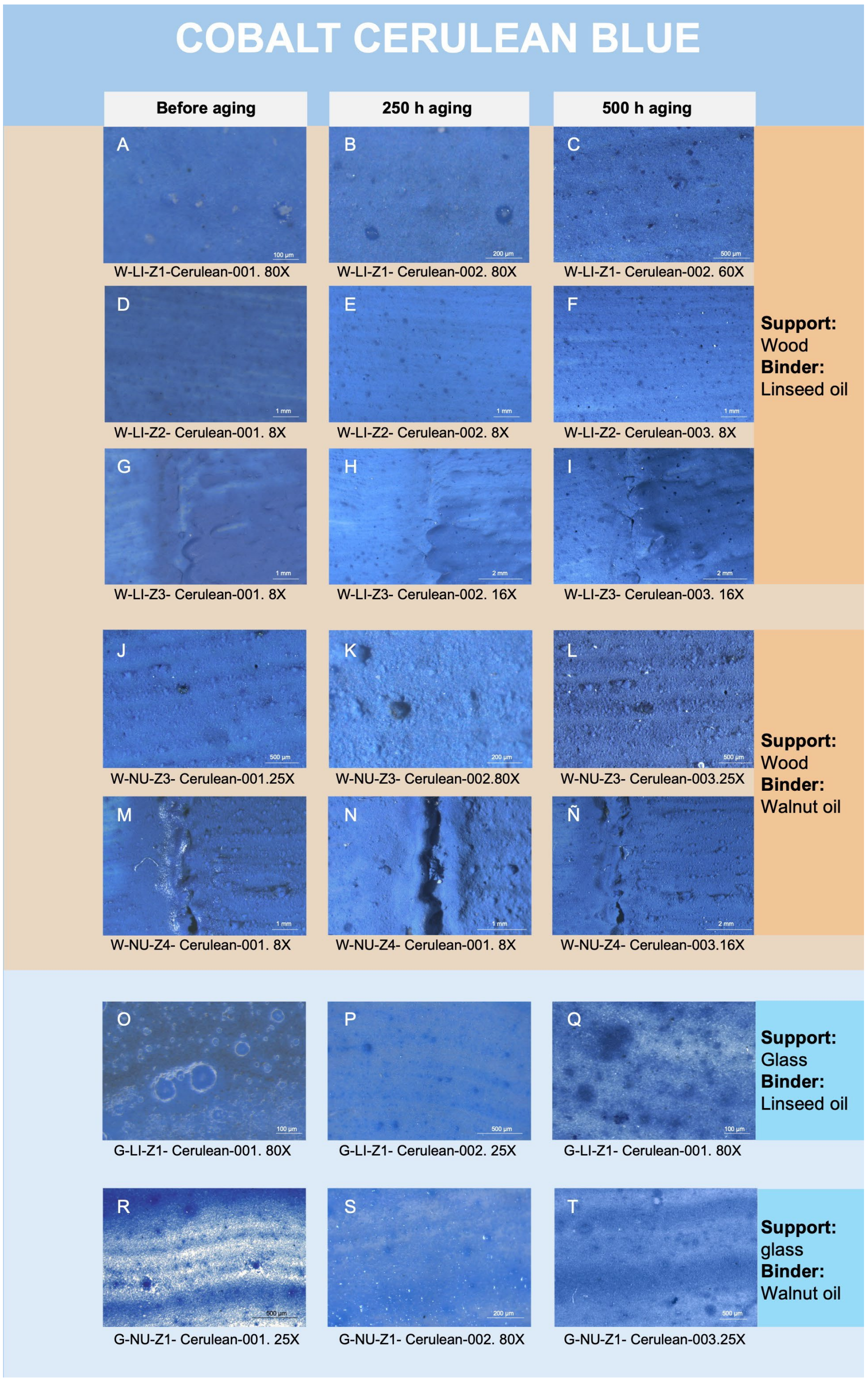

Fig. 4. Microscopic images of cerulean blue pigment bound with linseed oil and walnut oil and applied on MDF board and glass.

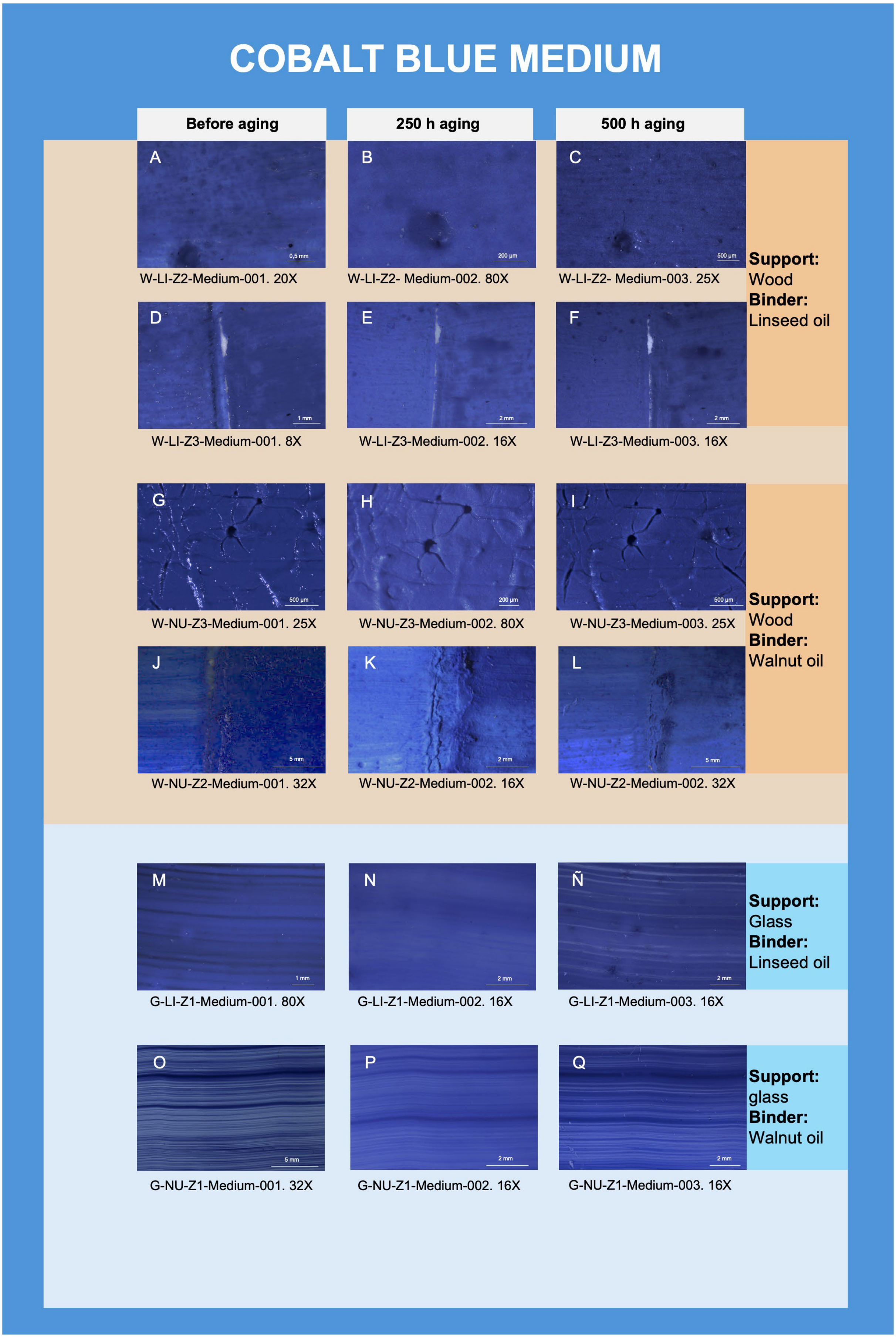

Fig. 5. Microscopic images of cobalt blue medium pigment bound with linseed oil and walnut oil and applied on MDF board and glass.

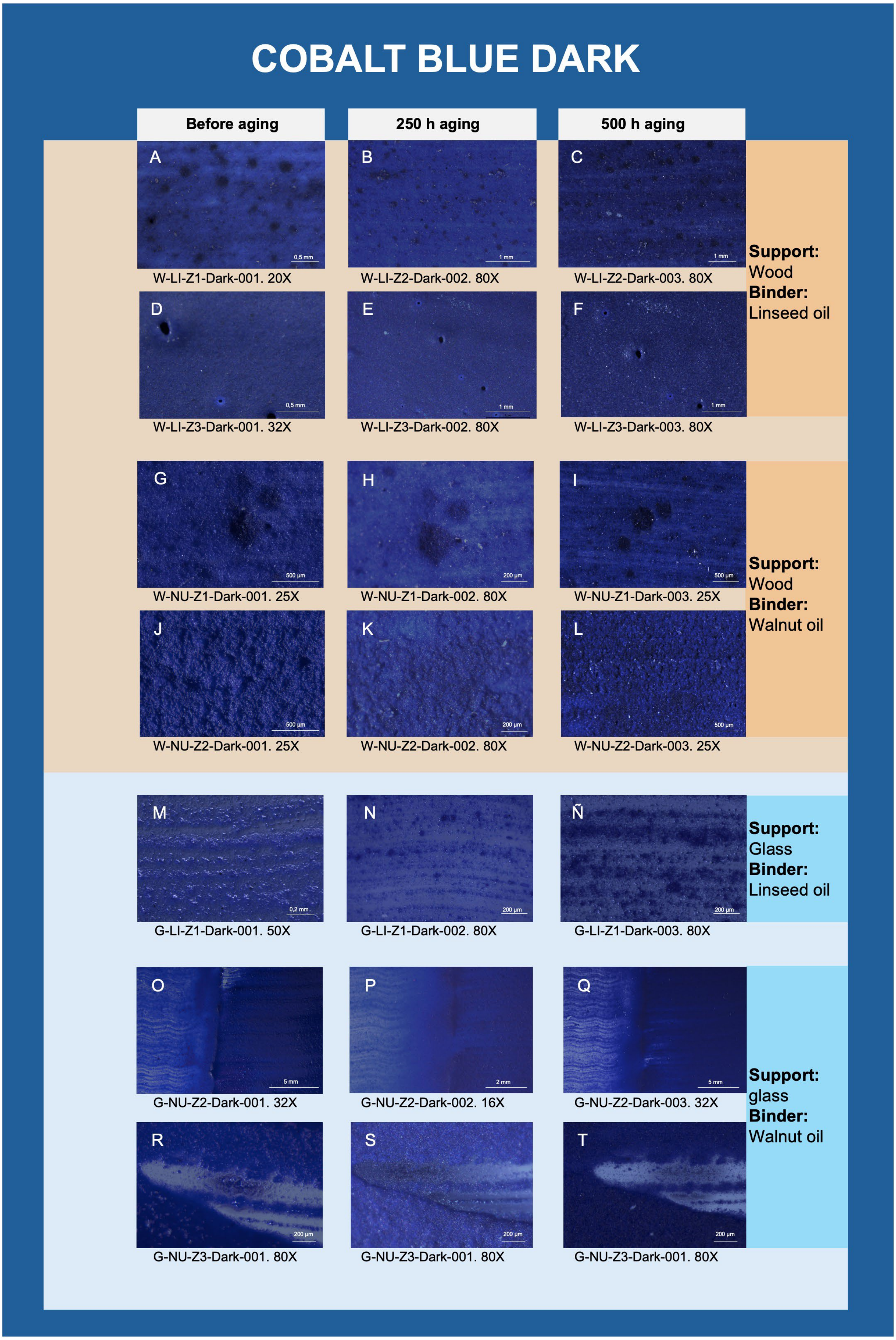

Fig. 6. Microscopic images of cobalt blue dark pigment bound with linseed oil and walnut oil and applied on MDF board and on glass.

5. Conclusions

This paper presents an experimental study for the characterisation of oil paint films containing cobalt blue, cerulean blue, and dark cobalt blue pigments using optical microscopy. Particular attention was paid to morphological and optical changes induced by accelerated artificial ageing in mock-ups prepared on different supports.

Cobalt belongs to the category of primary or active driers (along with manganese and lead, among others). They act as oxidation catalysts and cause surface drying. However, their role as driers can contribute to the formation of surface cracks.

Examination of the mock-ups by stereoscopic microscopy revealed significant changes in the structure and appearance of the oil paint films with all three cobalt pigments after accelerated ageing.

With regard to the support used, the nature of the support directly influences the morphology of the oil paint film and affects its dimensional variations. The ground layer applied on the MDF board absorbs part of the binder, resulting in a loss of brushstroke definition and increased opacity of the paint layer. In contrast, non-porous glass retains sharper brushstrokes but has less natural adhesion, resulting in surfaces with less pigment accumulation.

After artificial ageing, it was observed that mock-ups prepared on glass tended to retain a more uniform and less opaque surface due to the similarity in refractive indices between glass and the oils. Mock-ups prepared on wood, on the other hand, developed a rougher surface.

The presence of pigment aggregates was observed in all the mock-ups prepared on wood support, due to the roughness of the ground layer surface, which allows pigment particles to settle in the depressions. This resulted in a less uniform pigment distribution and affected the surface morphology. No significant difference was observed between the two types of binder used.

Brushstroke thickness also influenced the morphological changes of the oil paint film. Areas with thicker brushstrokes showed the presence of dark spots of heterogeneous sizes and shapes. The thickness of the paint layer affects the oil oxidation process. Thicker layers increase the drying time. This factor, combined with the superficial drying property of cobalt, results in an inefficient drying depth, slowing oxygen diffusion within the film volume.

After artificial ageing, a decrease in opacity was observed in the samples, attributed to a change in the refractive index of the binders.

The results obtained through microscopy and colorimetry (Llácer Peiró et al., 2023) allow for a better interpretation of the data, offering a more comprehensive view of the observed effects. The colorimetry data indicated that ageing affects the mock-ups differently depending on the support used In the samples made on MDF board, an increase in saturation was observed after ageing, while the opposite trend was noted with glass mock-ups (Llácer Peiró et al., 2023). This effect is confirmed at the microscopic level. It is due to the porosity of the ground layer on the MDF board, which facilitates greater pigment accumulation, potentially increasing saturation, while the uniformity of the film on the glass could explain the decrease in saturation.

This study has provided a more detailed understanding of the morphological changes caused by ageing in oil paint films containing cobalt blue pigment. These findings can provide valuable insights for the evaluation and study of artworks in which this pigment is used, as well as for the development of diagnostic strategies.

6. 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.

7. Funding source declaration

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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.

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

Notes

[1] The uniformity of a paint film depends on several factors, such as the grinding of the pigments and the absorption and wetting properties of the binder. When these materials are mixed, the binder first coats the paint particles (binder absorption) and then the air fills the spaces between the particles, becoming impregnated with the binder (interstitial binder). These two stages are essential to achieve a compact and uniform film (Zalbidea, 2014).

[2] The colorimetric data obtained from the samples, the results of which were presented at the XVIII Conferenza del Colore in Lecco, 2023 (Llácer Peiró et al., 2023), indicate the following values for cerulean blue: L*= 36.10; a*= -1.58; b*= -39.53; C* = 39.56; H* = 267.73, confirming that this pigment does not have a violet hue, as stated by Gettens & Stout.

[3] The colorimetry data from samples shows the following results for medium cobalt blue: L*= 29,84; a*= 10,51; b*= -45,94; C* = 47,13; H* = 282,91.

Licensing terms

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